# New insights into RF and microwave drying of foods

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# Abstract

This work reports a fundamental study of the science and economics of microwave and RF drying of foods, addressing current knowledge gaps and introducing new techniques to aid the development of new microwave and RF drying processes.

The present study has discovered that a correlation exists between the points of inflection of the moisture dependant dielectric properties and the moisture dependant water activity. Hence the moisture dependant dielectric properties are governed by the state of the water as defined by the sorption isotherms.

Water activity equations were mathematically modified in this study to successfully describe moisture dependant dielectric properties. This is a valuable contribution to science as there are currently no recognised standard equations for describing the moisture dependence of dielectric properties. These new proposed equations are of great value as they can be used in microwave drying models. This will enable engineers to optimise microwave and RF processes, leading to a reduction in the trial and error approach that currently prevails in industry. Improved optimisation may ultimately lead to more successful microwave and RF applications.

It is extremely important to be able to monitor key parameters such as porosity, oil and water content, when introducing a new food processing technique. To this end, a new and novel technique for quantifying the porosity of thin irregularly shaped food has been established. This can be used as a diagnostic tool to assess and optimise processing changes such as the introduction of new microwave or RF drying processes. A good understanding of how RF/MW processes affects porosity can be used to optimise the drying process, leading to a greater probability of success.

Sorption isotherm measurements carried out in this study showed that the gelatinisation of starch in potato has a negligible effect on the isosteric heat of sorption. This is important for drying applications, as the isosteric heat of

sorption is very significant compared to the latent heat at low moisture contents.

Dielectric properties were measured for potato crisps, biscuits, and pasta particulate at 915MHz, and were used to gain new insights into moisture levelling behaviour through analytical analysis. Contrary to common belief, it was found that moisture levelling can be more effective *below* the critical moisture content, at lower moisture contents. Although moisture levelling limits the variability of the final moisture content, it was found that fine control of the final moisture content can only be achieved by minimising variation in electric field exposure.

Feasibility studies of microwave applications showed that finish drying microwave applications were far more practical and cost effective compared to applications that have to remove large quantities of water. Dry food is generally quite thin so that it is eatable, or for rehydration purposes. As a consequence conventional food drying processes tend to be sufficiently energy efficient so that even microwave and RF finish drying processes are comparatively expensive with respect to energy usage. Hence, microwave and RF food drying must be justified by improvements in quality.

# Affirmation

The work reported in this thesis is solely the work of the author and has not been published elsewhere except for the following publications:

## **Published Journal papers**

Renshaw, R. C., Robinson, J. P., Dimitrakis, G. A., Bows, J. R., Kingman, S. W., 2016. Characterisation of potato crisp effective porosity using Micro-CT. *Journal of the Science of Food and Agriculture*, Volume 96 (13), pp.4440-4448

# **Conference proceedings**

Renshaw, R. C., Przybyla, J. S., Kingman, S. W., *Multiphysics modelling of microwave assisted mineral extraction using Micro-CT scan data*. Mechelen Belgium, Skyscan User Meeting 2010

Renshaw, R. C., 2009, *Multiphysics Modelling of Food Dehydration during RF Exposure*. Milan Italy, Comsol European Conference 2009

# **Planned journal papers**

Renshaw, R. C., Robinson, J. P., Dimitrakis, G. A., Kingman, S. W., The relationship of dielectric response and water activity in food

Renshaw, R. C., Robinson, J. P., Dimitrakis, G. A., Kingman, S. W., Empirical description of moisture dependent dielectric properties using adapted water activity equations

Renshaw R. C., Robinson J. P., Dimitrakis G. A., Kingman S. W., Moisture sorption behaviour of potato crisps and biscuits

Renshaw R. C., Dimitrakis G. A., Robinson J. P., Kingman S. W., Reduction of acrylamide in potato crisps by microwave finish drying, and measurement of potato crisp dielectric properties

Renshaw R. C., Dimitrakis G. A., Robinson J. P., Kingman S. W., Microwave cooking of potato crisps as an alternative to frying

Renshaw R. C., Dimitrakis G. A., Robinson J. P., Kingman S. W., Moisture dependant dielectric properties of biscuits at 915MHz and assessment of selective heating for microwave finish drying.

Renshaw R. C., Dimitrakis G. A., Robinson J. P., Kingman S. W., Moisture dependant dielectric properties of pasta at 915MHz and assessment of microwave drying of pasta

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# **Chapter 1 - Introduction**

The world's biggest industry is the food industry. The packaged food industry has been valued by Euromonitor international at \$1.6 trillion. The world food bank puts the food and agricultural sector at 10% of global gross domestic product, which, taking the bank's 2006 estimate for global GDP, would make the sector worth \$48 trillion (Murray, 2007).

There are four key drivers that are common to most large company's aims and objectives, which are as follows:

- 1. Reduce energy consumption and increase profit
- 2. Reduce greenhouse gas emission
- 3. Produce healthier food
- 4. Improve product quality

Given the focus on reduction in costs and energy consumption it is sensible to focus innovation on areas which have the potential to deliver significant savings in both of these areas, although they are also inextricably linked of course. Drying accounts for a significant amount of energy consumption and operational cost in the food processing industry.

The food industry currently uses a large number of drying processes. Almost every food product is dried at least once at one point of its preparation (Sokhansanj & Jayas, 2006). Drying of food typically occurs as part of the cooking process, where cooking is required for sterilisation and to produce the desired taste characteristics. Dehydration is also required specifically for food preservation. The reduced weight and bulk of dehydrated products and their dry shelf stability reduces product storage and distribution costs (Toledo, 2007). A dry product flows easier, is less sticky, and mixes with other materials more uniformly compared to a wet product. Dry foods can be used in gravity feed systems for food processing as well as in transportation (Sokhansanj & Jayas, 2006). Lowering the moisture content decreases the water activity, which decreases waters mobility and its ability to host microbial growth (Barbosa-Cánovas. & Juliano, 2007; Al-Muhtaseb, et al., 2004). Generally, growth of micro-organisms cease to occur at water activities less than 0.6, and pathogenic material does not grow above 0.86 water activity (Roos, 2007; Labuza & Altunakar, 2007). The unfavourable oxidative and enzymatic reactions that shorten the shelf life of food are also reduced with lower moisture contents (Sokhansanj & Jayas, 2006). Long shelf life is desirable for both shops and consumers as it minimises waste and is convenient for the consumer. The elevated temperatures that are commonly involved in drying can also be used as a means of food sanitation. Insects and other microorganisms can be destroyed during the application of heat and moisture diffusion. Sanitation is temperature-time dependant and can be achieved with 60°C for 3 to 5 minutes, or 48°C for 24 hours or longer (Sokhansanj & Jayas, 2006; Sokhansanj & Wood, 1991).

Microwave drying has been shown to be able to drastically reduce drying times, reduce energy consuption, and reduce factory footprint when compared to traditional air drying methods. Microwave and RF drying is volumetric, and can therefore overcome the heat and mass transfer limitations of air drying, especially in foods that are thick and dense. Pasta drying is often cited as a microwave application that provides huge improvements in drying times compared to the conventional air drying technique. Berteli & Marsaioli Jr. (2005) found that drying time of Penne Pasta could be reduced by more than 10 times using a continuous pilot scale microwave assisted hot air rotary dryer. Altan & Maskan (2005) found that microwave drying of macaroni improved drying times by 87.3% compared to the hot air method. Schiffmann (2001a) reported similar reductions in Pasta drying time, where the microwave process took 1.5 hours compared to the 8-12 hour conventional process. There were major floor savings, improved sanitation and product quality, and less energy usage.

Increasing profitability is generally the prime objective of any food company. Improving food processing energy efficiency has the potential to improve profit whilst reducing the company's impact on the environment. Kellogg's (revenue \$14.6 billion (Kellogg's, 2015a)) for instance has made a commitment to reduce energy consumption by 15-20% from 2005-2015. They are now committing to reduce energy consumption by a further 15% (per metric tonne of food produced) from their 2015 performance by 2020 (Kellogg's, 2015b). Food companies consistently spend money on research and development to improve process efficiency in a drive to increase profits.

Greenhouse gas emissions are of global concern, and most food companies have very aggressive targets to reduce their  $CO_2$  footprint. Achieving these targets requires large amounts of investment in technology. PepsiCo, a company with revenue of over \$66 billion (PepsiCo, 2015a), have stated that they want to make their UK operations and manufacturing fossil free by 2023 (PepsiCo, 2015b). They have already achieved a 20% reduction in carbon emissions since baseline. Similar to its energy commitments, Kellogg's made a commitment to reduce greenhouse gas emissions by 15-20% from 2005-2015. They are now committing to reduce greenhouse gas emission by a further 15% (per metric tonne of food produced) from their 2015 performance by 2020. This will lead them to increase the number of plants using low-carbon energy to 50% (Kellogg's, 2015b). Nestlé (revenue 88.8 billion Swiss francs (Nestlé, 2015a)) has reduced they're direct greenhouse gas emissions by 40% since 2005, achieving an absolute reduction of 11.4% and exceeding their 2015 target by 5% (Nestlé, 2015b). Barilla (revenue €3.4 billion (Barilla, 2015)) has reduced their CO<sub>2</sub> emissions by 27% between 2008 and 2013 (Barilla, 2013). United Biscuits (revenue £1.1 billion (Marlow, 2014)) have achieved a 30% reduction in carbon emissions between 1995 and 2005, with a goal of 40% reduction by 2020 (United Biscuits, 2015).

Microwave energy is potentially clean, as it is delivered by vacuum electron tubes that are fed by electricity. Electricity can be produced with zero carbon emissions by nuclear, wind, solar, or tidal power plants. RF and microwave energy is an efficient method of converting electrical energy to heat. The heat is delivered directly to the drying products via volumetric heating. Microwave energy is typically delivered by magnetrons which can be 88% efficient (e2v BM100L, 915/896MHz, 100kW), whilst RF energy delivered by triodes can be

77% efficient (e2v BW1643J2, 40MHz, 530kW). Hence, RF and microwave drying can help food companies achieve their ambitious targets for reducing greenhouse emissions.

Most food manufacturers are focused on improving nutrition and providing healthier snacks due to commercial demand. There is increasing government regulatory pressure for healthier food in the form of labelling with the future possibility of a "fat tax". Already in the UK, schools are not allowed to sell crisps due to health concerns (UK Goverment, 2015). The food industry has to pre-empt further government legislation to ensure that they are well placed commercially for any changes in regulation. There is also increasing public awareness of potential carcinogens such as acrylamide in food (IARC, 1994; Lineback, et al., 2012). Health scares can lead to falling sales, hence food companies are researching ways of reducing the quantity of potential carcinogens in their products. PepsiCo are currently reformulating their existing products and reshaping their portfolio to make products healthier (PepsiCo, 2015c). In support of this activity they also market and sell their products responsibly to facilitate informed choices and work in partnership with others to positively influence the UK diet. Nestlé have published an objective to reduce saturated fat by 10% in all relevant products that do not meet the Nestlé Nutritional Foundation criteria (Nestlé, 2015b). Barilla reformulated 41 of their products to improve their nutritional profile (Barilla, 2013).

With regards to reduction in fat content, it has recently been proven that microwaves can be used to replicate the explosive dehydration experienced during frying of potato crisps A patented technique (Bows, et al., 2010) has been developed to mimic the dehydration of standard continuous fried crisps using a microwave process to obtain a similar texture, with more control over the oil content. In a forced preference consumer test (n=200) between the microwaved crisp and its continuously fried equivalent, there was no statistically significant difference in preference (51% preferred the microwaved crisp). This allows the manufacturer to produce crisps targeted at consumer groups with a specific fat content. Although microwaved crisps are likely to be

expensive compared to fried crisps (Schiffmann, 2001a), they could become financially viable with the possible introduction of a fat tax.

Carcinogens generally occur in food as a result of overheating. Carbohydrate foods such as potato and cereals that have a high content of the amino acid asparagine, and a high accumulation of reducing sugars, are subject to the formation of acrylamide upon high temperature processes such as frying (Granda, et al., 2004; Tareke, et al., 2000; Lineback, et al., 2012). Acrylamide has been found to be a carcinogen and genotoxin, and is not present in native (raw) ingredients such as raw potatoes (Lineback, et al., 2012). It is formed during heat processing, and it has been found that acrylamide readily formed when potatoes were heated above temperatures of  $120^{\circ}$ C (Tareke, et al., 2002), encountered in processes such as frying, boiling, baking, roasting, grilling, and toasting (Lineback, et al., 2012). Overheating typically occurs during the final stages of drying where both heat and mass transfer to the centre of the product is difficult as the dried outer regions of the food acts as an insulator. RF and microwave heating is volumetric and does not require a temperature difference, unlike conventional heating. RF/MW heating can therefore overcome heat and mass transfer limitations experienced with conventional heat transfer during finish drying. RF and microwave heat may also selectively heat the moisture in the centre of the product, leading to moisture levelling. Volumetric heating and selective heating of moisture can help reduce the products exposure to high temperatures, and therefore lower the carcinogenic material in the product.

Product quality is imperative to all food manufacturers. It affects they're reputation, and ultimately their sales. With respect to drying, common food quality issues include poor uniformity of moisture content leading to mould growth, burning and/or over-drying of outer surfaces, and defects such as cracking. Overheating and burning of outer surfaces is common with traditional drying methods involving conduction, convection, or radiation. Heat is transferred to the material surface, which means the heat energy has to pass from the surface by conduction to the interior of the product. Often this can present a problem, because the material itself is a good insulator and as such, it is difficult to get the heat to penetrate to the centre of the product. This causes

the surface to dry out, whilst the core or centre of the food remains high in moisture. Differences in moisture content can present fissures and cracks (checking) during storage.

Post baking finish drying using RF has been proven to improve biscuit quality (Koral, 2004). RF and microwave heating is volumetric and will selectively heat regions of higher moisture content. A study by Bernussi et al. (1998) showed significant reductions in moisture gradient for microwave finish drying of bischits, from 2.16% to 0.88% (central disk to outer rim), and the incidence of cracking was reduced from 41.7% to 0%.

## 1.1 Issues with RF and microwave heating

Microwave/RF drying has some unique benefits such as volumetric heating, moisture levelling, quick and efficient drying, and small factory footprint. It also has some well understood drawbacks such as arcing, thermal runaway, penetration depth, cost, maintainability, and evenness of electric field intensity. After careful consideration of the advantages and disadvantages of RF and microwave drying versus more conventional techniques, a large number of applications in the food industry have been attempted (Metaxas & Meredith, 1993; Schiffmann, 2001a). Although some applications have succeeded, there are a number of applications that have failed at various stages of development, including laboratory stage, pilot stage and scale up stage (Metaxas & Meredith, 1993; Schiffmann, 2001a). The relatively large proportion of applications that have failed implies that there must be knowledge gaps in the understanding of RF/microwave drying that is seriously affecting they're application.

The moisture levelling effect, although consistently cited as an important benefit of microwave heating, is not well understood. Quantification of moisture levelling is difficult as the electric field distribution in the applicator is unknown in practical experiments. Quantification through fully coupled physics models is extremely challenging as drying is dependent on porosity, thermal conductivity, diffusivity, and permeability, all of which can change drastically during drying and are very difficult to quantify over the moisture content and temperature range experienced during drying. The moistue dependance of dielectric properties, which is vitally important for drying, is not particuallarily well undrstood. There are numerous models describing the frequency dependance of dielectric properties, but there are no recognised standard equations for desribing the moisture dependance of dielectric properties. Although it is appreciated that the state of the water has an effect on the moisture dpeendnce of dielectric properties, the exact relationship is not defined.

It is difficult to change a drying process though the use of a fundamentally different drying technique without changing the product properties. Any change in final product characteristics could affect customer acceptance and sales, and the reputation of the product could be irreparably damanged. There are a large number of food products that are popular with consumers that have a crispy dehydrated outer surface and a comparitivaly wet inner surface. These types of food products ovbiously lend themsleves to conventional heat transfer mechanisms, rather than volumetric heating. Common examples include chips (fench fries), oinion rings, breaded chicken and fish, toast, and crispy bacon. Even with completely dehydrate foods such as potato crisps, it is important that any alternative microwave dehydration technque replicates the drying curve to obtain the correct porosity texture and therefore taste.

A better understanding of dielectric behaviour with respect to drying, as well as suitable methods for obtaining the metrics relevenat to consumer acceptance, could lead to improved success in industrialisation of RF and microwave applications.

## 1.2 Conclusion

There are a number of knowledge gaps to be addressed by the work presented in this thesis. The moisture dependence of the dielectric properties must be better understood. This includes defining whether or not a relationship exists between the moisture dependence of the water activity, and the moisture dependence of the dielectric properties. The state of the water is typically defined by the moisture dependence of the water activity, where the state of the water defines its mobility. Both water activity and dielectric loss mechanisms are dependent on water mobility within foods, which infers that a relationship should exist. Product metrics that are important to customer acceptance must be able to be quantified. Porosity is difficult to quantify for thin, irregularly shaped heterogeneous food materials. This is a problem for applications such as the explosive microwave dehydration of potato crisps. This study will investigate viable methods for quantifying parameters critical to customer acceptance that are likely to vary significantly dependant on the dehydration technique. In order to better understand and advance microwave drying in the food industry, the following research activities will be undertaken:

- Identify four RF/microwave food drying applications that show the most promise with respect to energy saving and product quality/control. Each application must address at least one, but preferably most of the primary drivers for food companies i.e. energy saving, reduce greenhouse gas emissions, control fat content, reduce carcinogens, and improve quality.
- Determine if a relationship exists between the sorption isotherm and the moisture dependent dielectric properties. Investigate whether water activity equations can be adapted to describe dielectric properties with respect to moisture content.
- Characterise a food product microstructure for an application where the porosity of the product significantly changes during RF/microwave exposure. Porosity is extremely important for product taste, and any RF/microwave process that replaces an existing conventional process must be able to deliver the correct final porosity of the food product.

- Measure the sorption isotherms for the selected food products. This will define both the monolayer moisture content and the transition from multilayer to solution where microbial growth is supported.
- Measure the dielectric properties of the selected food products with respect to moisture content. These measurements will enable the study of dielectric moisture levelling, and can be used for RF/MW applicator design.
- Identify the RF/MW application that shows the most potential for further research and development. This will be identified based on its competitive advantage, moisture levelling effectiveness, practicality, and cost considerations. The competitive advantage will be judged against potential energy saving, reduction in greenhouse gases, control of fat content, reduction in carcinogens, and improvement in product quality.

# Chapter 2 – RF and microwave food drying theory

As well as describing the basic theory, the review detailed in this chapter aims to identify knowledge gaps in the science of microwave and RF drying of foods so that they can be addressed in subsequent chapters. Four cutting edge RF/MW drying applications are identified for further study that address the key drivers stated in Chapter 1.

# 2.1 Conventional drying

Drying occurs due to evaporation and boiling. Boiling occurs when a liquid is heated to its boiling temperature, which is the temperature at which the vapour pressure of the liquid is equal to the pressure exerted on the liquid by the surrounding environmental pressure (Knight, 2007). Evaporation is caused by the difference between the partial vapour pressure in the air,  $P_{v_air}$ , and the vapour pressure in the porous media,  $P_{v_solid}$ . The evaporation rate is presented in Equation 2-1 (Halder, et al., 2007), where K is the mass transfer coefficient which is used to describe resistance, and is a function of surface area exposed to the drying air, the shape of the solid, and the velocity of the drying air (Richardson, et al., 2002).

$$\dot{m} = K(P_{v\_solid} - P_{v\_air})$$
Equation 2-1

When the partial vapour pressure in the porous media is equal to the partial vapour pressure in the surrounding air, evaporation ceases to occur (Richardson, et al., 2002). Vapour pressure can only reach as high as the saturated vapour pressure, which is highly temperature dependant. Higher temperature air has a higher saturation point than lower temperature air, hence higher rates of evaporation can be achieved using hot air (Rogers & Mayhew, 1964).

In air-drying processes, two drying periods are usually observed: an initial constant-rate period in which drying occurs as if pure water were being evaporated, and a falling rate period where moisture movement is controlled by internal resistances. Figure 2-1 shows drying rate against moisture content,

where segment AB represents the initial unsteady-state, warming-up period, and BC the constant rate period.



Figure 2-1 Drying curve, showing moisture content as a function of drying time, adapted from Rizvi (1995)

During the constant-rate period regime, the drying surface is saturated with water, and drying occurs at the wet bulb temperature of the environment. In systems where the liquid movement is likely to be controlled by capillary and gravity forces, a measurable constant-rate period is found to exist. Where liquid movement is by diffusion, the water that is evaporated from the surface is not immediately replenished by movement of liquid from the interior of the material. Such structures are likely to dry without exhibiting any constant-rate period (Rizvi, 1995).

For materials that do experience a constant rate drying period, Point C in Figure 2-1 indicates the critical drying moisture content at which the departure from constant rate drying is first noticed ( $M_{Cr}$ ). The drying period represented between point C and D in in Figure 2-1 is termed the first falling-rate period. During this period the rate of liquid movement to the surface becomes less with respect to time and hence the rate of evaporation from the surface reduces with respect to time, and the surface becomes continually depleted in liquid water. Parts of the surface dry up until point D, where all evaporation occurs from the interior of the material (Rizvi, 1995).

Beyond point D, the path for transport of both the heat and mass becomes longer and more tortuous as the moisture content continues to decrease (Rizvi, 1995). The evaporation zone moves further within the solid as illustrated in Figure 2-2. This period is called the second falling-rate period. The thicker the material the slower the evaporation process becomes (Metaxas, 1996). Drying ceases at the end of the second falling rate period when the vapour pressure in the material reaches the partial vapour pressure of the drying air. This final moisture content, to which drying is limited, is called the equilibrium moisture content,  $M_E$ , as shown in Figure 2-1 (Rizvi, 1995).



Figure 2-2 (a) Conventional drying during (a) constant drying rate period and (b) second falling rate period

# 2.2 Describing hygroscopic behaviour

Most foods are hygroscopic, where hygroscopic materials are defined as having a clearly recognizable pore space, a large amount of physically bound liquid, and shrinkage often occurs in the initial stages of drying (Mujumdar, 2006). Adsorption occurs in hygroscopic materials, where water attaches itself to surfaces either within a porous media, or on an external surface of the host material. This adsorption process in hygroscopic material leads to the description of "bound" water. The interaction of food with water can result in the food exhibiting an equilibrium vapour pressure lower than pure water. This partly governs the drying process as evaporation is driven by the difference between the vapour pressure of the material, and the partial vapour pressure of the surrounding air.

#### 2.2.1 Water activity, sorption isotherms, and glass transition

Sorption isotherms are most commonly to describe hygroscopic behaviour. The sorption isotherm is a plot of the equilibrium water content against the water activity at a certain temperature and pressure. Where water activity is the partial vapour pressure in the material,  $P_{\nu}$ , divided by the vapour pressure of pure water at the same temperature, which is denoted as  $P_{\nu s}$  (Al-Muhtaseb, et al., 2002). The expression for water activity is shown below, where water activity can also be expressed as the equilibrium relative humidity of the air surrounding the food at the same temperature (Sahin & Sumnu, 2006).

$$a_{w} = \frac{P_{v}}{P_{vs}} = \frac{RH}{100}$$
 Equation 2-2

The water activity in a substance indicates the "quality" of the water content and is a measure of the waters availability to participate in physical, chemical, and microbiological reactions (Barbosa-Cánovas. & Juliano, 2007; Al-Muhtaseb, et al., 2004). Critical water activity values are 0.6 for the growth of any micro-organisms and 0.86 for the growth of pathogenic material (Roos, 2007). Foods dried to water activities less than 0.3 are very stable, resulting in a long shelf-life which is highly desirable. The glass transition ( $T_g$ ) of foods typically occurs in the 0.35-0.45 water activity range. Below the  $T_g$  a material is glassy or brittle/hard, whereas above it, materials show a rubbery texture, from leathery to soft to sticky (Labuza & Altunakar, 2007). Some foods are dried primarily to achieve a crunchy taste, which involves drying to below the glass transition point. Brunauer, et al., (1940) classed sorption isotherms into 5 different types. Figure 2-3 shows the shape of type II and type III sorption isotherms. The type II isotherm (sigmoid shaped) describes low sugar hydrocolloids and is the most common isotherm for foods (Yanniotis & Blahovec, 2009). Foods with high sugar content such as fruits tend to belong to class III isotherms, although some are class II isotherms (Yanniotis & Blahovec, 2009; Al-Muhtaseb, et al., 2002; Chinachoti & Steinberg, 1984).

Figure 2-3 (a) shows regions A, B, and C on the type II sorption isotherm. Region A of the curve represents a region in which the monomolecular layers are formed, although there may be multi-molecular layers in some places toward the end of region A (Mujumdar, 2006). Water in region A is strongly bound with an enthalpy of vaporisation considerably higher than that of pure water. Usually, water in this region is unfreezable, and is not available for chemical reactions or as a plasticizer (Yanniotis & Blahovec, 2009). Foods dried to water activities in Region A, are very stable and have a long shelf life, which is desirable for both consumers and retailers. Region B is a transitional region in which double and multiple layers are mainly formed and capillary condensation could also have taken place (Mujumdar, 2006).



Figure 2-3 (a) Type II sorption isotherm showing regions A, B, and C (b) Type III sorption isotherm

Moisture in Region B can still be considered to be bound water (Liu, et al., 2009), as water mobility is considered restricted. The water in this region is less firmly bound than in region A, and is available as a solvent for low

molecular weight solutes and for some biochemical reactions. In region C capillary condensation and swelling can take place and the water exhibits nearly all the properties of bulk water. The free water tends to behave as water in solution where chemical reactions and microbial growth can be supported (Yanniotis & Blahovec, 2009; Mujumdar, 2006). Maximum hygroscopicity is achieved when the solid is in equilibrium with air saturated with moisture ( $a_w = 1$ ) (Mujumdar, 2006). The approximate water activity and moisture content of various foods is presented in Figure 2-4. The diagram is partitioned into the traditional three water type regions.

Sorption isotherms can be obtained in one of two ways:

- An *adsorption isotherm* is obtained by placing a completely dry material into various atmospheres of increasing relative humidity and measuring the weight gain due to water uptake
- (ii) A *desorption isotherm* is found by placing an initially wet material into various atmospheres of decreasing relative humidity, and measuring the loss in weight (Al-Muhtaseb, et al., 2002).

It must be ensured that the sample is stable with its atmosphere for each measurement point.



Figure 2-4 Water activity and moisture content of some common foods, adapted from Schmidt, (2007)

#### 2.2.2 Temperature dependence of sorption isotherms

For most type II sorption isotherms, the quantity of water adsorbed at a given water activity decreases as the temperature increases. This signifies that these products become less hygroscopic as temperature increases as illustrated in Figure 2-5 (Lagoudaki, et al., 1993). The practical implications of the behaviour illustrated in Figure 2-5 is that for a given moisture content, the water activity increases with temperature. Hence shelf life is reduced at higher temperatures.



Figure 2-5 Temperature dependence of a typical type II isotherm created using data from Wang & Brennan, (1991)

An opposite trend is shown by some sugars (or sugar containing foods) which become more hygroscopic at higher temperatures because of the dissolution of sugars in water (Lagoudaki, et al., 1993).

## 2.2.3 Adsorption and desorption hysteresis

A characteristic of sorption isotherms of biological materials is their dependence on the previous sorption history (Ngoddy & Bakker-Arkema, 1975). Typical adsorption and desorption curves (see Figure 2-6) result in more water being held at the same water activity for the desorption curve than for the

adsorption curve (Labuza & Altunakar, 2007). At high and low water activities the moisture content can be the same for both adsorption and desorption, forming closure points of a hysteresis loop. In some instances the size of the hysteresis loop depends upon the rate and frequency of adsorption and desorption (Ngoddy & Bakker-Arkema, 1975).



Figure 2-6 Generalised water sorption isotherm showing a hysteresis loop (Labuza & Altunakar, 2007)

There are a number of possible reasons for sorption hysteresis. One of the most cited reasons is the "ink bottle effect". This phenomenon occurs due to the capillaries emptying differently upon desorption; the narrow ends of surface pores will trap and hold water internally below the water activity where it should have been released during adsorption (Wang & Brennan, 1991).

#### 2.2.4 Sorption isotherm models

After sorption isotherms have been measured, it is common to fit a sorption isotherm model to the experimental data using non-linear regression. Researchers and engineers use sorption isotherm equations in drying models to predict or characterise drying behaviour. There are a range of models available for fitting moisture sorption isotherm data, the most commonly used models are presented in Table 2-1. These include kinetic models, semi-empirical and empirical equations, with two, three, or four fitting parameters.

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$M_{db} = \frac{M_0 C a_w}{(1 - a_w)[1 + (C - 1)a_w]}$	BET equation	(Brunauer, et al., 1938)
$M_{db} = A \left[ \frac{a_w}{1 - a_w} \right]^B$	Oswin equation	(Oswin, 1946)
$M_{db} = C_1 + C_2 ln(1 - a_w)$	Smith equation	Smith (1947)
$M_{db} = M_0 \left(\frac{-A}{RT lna_w}\right)^{1/B}$	Halsey	Halsey (1948)
$M_{db} = \left[ -\frac{\ln(1-a_w)}{AT} \right]^{1/B}$	Henderson	(Henderson, 1952)
$M_{db} = \frac{M_0 C K a_w}{(1 - K a_w)[1 - K a_w + C K a_w]}$	GAB equation	(Guggenheim, 1966) (Anderson, 1946) (de Boer, 1953)
$M_{db} = a + bln(-lna_w)$	Chung-Pfost	(Chung & Pfost, 1967)
$M_{db} = \left[ ln \left( \frac{A - lna_w}{B} \right) \right] / (-C)$	Chen equation	(Chen, 1971)
$M_{db} = \left(-\frac{A}{\ln a_w}\right)^{1/B}$	Modified Halsey	(Iglesias & Chirife, 1976a)
$M_{db} = [A/ln(\gamma/a_w)]^{-1/r}$	Ferro- Fontan	(Ferro-Fontan, et al., 1982)
$M_{db} = C_1 a_w^{n_1} + C_2 a_w^{n_2}$	Peleg equation	(Peleg, 1993)
$M_{db} = A \left(\frac{1}{a_w} - 1\right)^{B-1}$	Lewicki I equation	(Lewicki & Wolf, 1995)
$M_{db} = \frac{F}{(1 - a_w)^G} - \frac{F}{1 + a_w^H}$	Lewicki II equation	(Lewicki, 1998)

Table 2-1 Sorption isotherm equations commonly used for foods

A large number of models have had to be developed as no one equation gives accurate results throughout the whole range of water activities, and for all types of foods (Al-Muhtaseb, et al., 2004). The BET equation is used to define the monolayer moisture content using a reduced water activity range, as it generally lacks accuracy at water activities above 0.35-0.45 (Iglesias & Chirife, 1976b; Al-Muhtaseb, et al., 2002; Wang & Brennan, 1991; McMinn, et al., 2007).

#### 2.2.5 Isosteric heat of sorption

The net isosteric heat of sorption,  $Q_n^{st}$ , is a measurement of the energy or intermolecular bonding between water molecules and absorbing surfaces (Mulet, et al., 1999).  $Q_n^{st}$ , is derived from the Clausius-Clapeyron equation, and is shown in Equation 2-3 (Reid, 2007).

$$\frac{Q_n^{st}}{R} = \frac{\partial \ln a_w}{\partial 1/T}$$
 Equation 2-3

The net isosteric heat of sorption can be calculated at a given moisture content by plotting  $\ln(a_w)$  versus 1/T. The slope of the graph  $\ln(a_w)$  versus 1/T equals - $Q_n^{st}/R$ . The net isosteric heat can also be calculated from the integrated form of Equation 2-3, using sorption isotherms measured at two temperatures (Wang & Brennan, 1991):

$$Q_n^{st} = R\left(\frac{T_1 T_2}{T_2 - T_1}\right) ln\left(\frac{A_{w2}}{A_{w1}}\right)$$
Equation 2-4

However, it is recommended to include as many temperatures in the range of study as possible to determine  $Q^{st}$ . This is because several assumptions are made in applying the Clausius Clapeyron equation. First, the heat of vaporisation of pure water  $(L_r)$ , and the excess heat of sorption  $(Q_n^{st})$  do not change with temperature. Secondly, the equation applies only when the moisture content of the system is constant (Labuza, et al., 1985).

The total isosteric heat of raw potato at 55°C measured by Wang & Brennan (1991) is presented in Figure 2-7. It can be seen that bonding energy is highest

at low moisture contents, where the most active sites are available (Wang & Brennan, 1991). It can also be observed that the net isosteric heat of sorption is very significant compared to the latent heat of vaporisation. Hence it is important to consider the isosteric heat of sorption when designing a drying process line. The higher isosteric heat in desorption has been noted by numerous authors (Rizvi, 1995; McMinn & Magee, 1999; Kaymak-Ertekin & Gedik, 2004; McLaughlin & Magee, 1998). This is considered indicative of the significant hysteresis presence (McMinn & Magee, 1999).



Figure 2-7 Isosteric heats of desorption and adsorption for potatoes at 55°C (adapted from Wang & Brennan (1991))

## 2.2.6 Measurement of moisture content

Moisture content must be accurately measured in order to define sorption isotherms, and is also necessary for analysing dielectric properties. Some of the most common and effective direct methods of moisture measurement are described in the following subsections.

#### 2.2.6.1 Gravimetric oven drying

Gravimetric methods are the most common and easiest procedures for analysis of moisture content. Approximately 29 of the 35 moisture methods recognized

by the Association of Official Analytical Chemists (AOAC) for nutritional labelling are gravimetric methods using some sort of drying oven (Fontana Jr., 2007). Scorching affects accuracy, and to reduce scorching vacuum oven drying and drying in the presence of desiccants can be carried out at temperatures below 100°C (Makower & Dehority, 1943; Kaymak-Ertekin & Gedik, 2004). Oven drying may not be suitable for foods containing volatiles such as oil. Mass loss and lipid oxidation can be impossible to distinguish from water loss which can lead to inaccurate results. Other factors that can introduce error are ambient relative humidity, weighing time with exposure to atmosphere, and handling of drying dishes.

#### 2.2.6.2 Gravimetric desiccants drying

Moisture content can be deduced gravimetrically by exposing a sample to air at zero water activity in a sealed desiccator using a suitable desiccant, which reduces the food sample to zero moisture content when equilibrium is reached. Desiccants are selected that are highly hygroscopic, can be used at room temperature, and do not react with other volatiles. This technique is useful for accurate gravimetric measurement of foods that contain oil. Drying at low temperatures using desiccants also avoids inaccuracy caused by scorching. However, powerful desiccants can be very hazardous to use.

#### 2.2.6.3 Karl Fischer titration

This is a potentiometric method to analyse trace amounts of water in a substance. A sample is dissolved in methanol, and titrated with a Karl Fischer reagent. The reagent contains iodine, which reacts proportionally with water. Thus, the water content can be determined by monitoring the potential of excess iodine. Poor solubility and interfering side reactions are the most common issues with the Karl Fischer reaction. However, many useful adjustments to the working conditions are possible to enable direct Karl Fischer titration for a wide variety of substances (Felgner, 2015).

#### 2.2.6.4 Dean and stark method

Dean and Stark method (ASTM D-95) can be used to determine the moisture content of foods containing volatile oils. Many difficulties may arise in the

determination of moisture by this method and it is generally not suitable for determining small amounts of water. Issues include relatively low precision of the receiving measuring device, difficulties in reading the meniscus, adherence of moisture droplets to the glass, over boiling, suitability of water in the distillation liquid, incomplete evaporation of water and underestimation of moisture contents, and distillation of water-soluble components (Peiris, 2009).

## 2.2.7 Measurement of sorption isotherms

The moisture sorption isotherm is created one of two ways:

- 1. Food samples are placed in controlled humidity chambers at constant temperature and the weight is measured until equilibrium is reached
- 2. A series of samples with varied moisture contents are established by adding or removing moisture; then water activity is measured once equilibrium is reached

The main risk for measuring sorption isotherms is the long equilibrium time and the risk of mould or bacterial growth at high relative humidity (Fontana Jr., 2007). McMinn & Magee (1999) quoted an equilibrium time of approximately 3 weeks for potato using controlled humidity chambers. To decrease equilibrium time, air inside the enclosure can be circulated and surface area of the sample can be increased. Toluene, ascorbic acid solution, potassium sorbate, crystalline thymol, and sodium azide have been used to prevent bacterial growth (Sahin & Sumnu, 2006; Rahman, 2009; McMinn, et al., 2003; Roca, et al., 2006; Quirijns, et al., 2005; Zhang, et al., 1996; Kiranoudis, et al., 1993). There is the risk that chemicals introduced to suppress microbial growth may change the mass at which the sample stabalises at a given relative humidity. The time taken to reach eqilibirum can be halved by evacuating air from the desiccators (Makower & Dehority, 1943). The lack of oxygen also helps suppress microbial growth. The use of microbalances in sealed chambers can also be used to reduce overall equilibration time.

#### 2.2.7.1 Gravimetric method using controlled humidity chamber

The gravimetric method using hygroscopicity of salts, sulphuric acid, or glycerol is most widespread for measuring moisture isotherms (Molnár, 2006). In this method, a weighed sample of food is stored in an enclosed container maintained at a certain relative humidity, at constant temperature. The sample is re-weighed from time to time until it is deduced that equilibrium is reached. Both Sulphuric acid and glycerol-water solutions can change water activity as it reaches equilibrium with the food sample, which can be a disadvantage. This can be mitigated by using large volumes of solution compared to the sample size, and by minimising the volume of the dissector. Gravimetric method using Sulpheric acid can also be hazardous (McMinn & Magee, 1999). Saturation of salt solutions can be easily maintained by adjusting either salt or water levels upon visual inspection, which in turn maintains the water activity. The disadvantage of saturated salt solutions is that each salt has a set relative humidity when saturated at a given temperature, hence a number of salts have to be used to obtain a sorption isotherm.

Unwanted adsorption or desorption can occur during the weighing of the sample, and it can take the dissector some time to re-establish its desired relative humidity. Steps should be taken to minimise the disturbance caused by weighing. The temperature of the solution used to generate the relative humidity must be as close as possible to the measured standard, typically  $\pm 1^{\circ}$ C (Fontana Jr., 2007; McMinn & Magee, 1999; Wang & Brennan, 1991).

#### 2.2.7.2 Measurement of water activity and moisture content

This method of generating a sorption isotherm involves measuring water activity. In this method, a set of samples of varying moisture contents are prepared. Dry samples are placed in a sealed container over water while wet samples are dried down or equilibrated with desiccant. The water activity and moisture content are then measured. Moisture content can be deduced at the start or end of the experiment, enabling changes in moisture content to be determined by recorded weight changes. It should be noted that this method still requires the sample to be stable and at equilibrium, i.e. the sorption isotherm will not be correct if the sample is wetter or dryer in the centre. In this method for measuring sorption isotherms the accuracy, repeatability, speed of measurement, stability in calibration, linearity, and convenience of use of measuring water activity can vary depending on the measurement method. Some of the methods for measuring water activity are listed below:

- Chilled mirror dew point hygrometer
- Electric hygrometer
- Hair or polymer hygrometer
- Thermocouple psychrometer
- Vapour pressure manometer (Fontana Jr., 2007)

# 2.3 Conventional drying techniques used in the food industry

The purpose of driers is to maximise heat and mass transfer. Energy is required to provide the latent heat for the phase change as well as the isosteric heat required to overcome hygroscopic bonds in the porous media. Mass transfer is required to remove the moisture and ensure that it does not condense back on the food after drying. There are a large number of dryers used in industry. One type of dryer may be used, or multi-stages depending on how much dehydration is required. In multi-stage drying, each drier is selected to efficiently remove moisture for a specific part of the drying curve in series, and may involve completely different types of driers, or similar dryers with different settings. Conventional dryers used in the food industry include sun drying, cabinet dryers, tunnel dryers, belt through dryers, spray dryers, freeze dryers, drum dryers, foam mat dryers, vacuum dryers, fluidized bed dryers, infrared drying, and frying. In all conventional drying approaches, achieving low moisture content in the centre of the food product whilst minimising the total drying time is a challenge. This is especially a problem for thick food materials that require an even final moisture content due to the limitations of conventional heat transfer. Common dryers that employ hot air convection heat transfer are cabinet dryers and conveyor band dryers (Sokhansanj & Jayas, 2006). Higher heat transfer rates can be achieved using infrared radiation drying, and frying, as discussed in the following subsections.

## 2.3.1 Infrared dryers

IR drying uses radiation as the source of heat transfer to dry the food. IR allows very high heat fluxes to be applied to the food surface. The equation for radiative heat transfer is shown in Equation 2-5, where  $\in$  is the emissivity,  $F_{1-2}$  is the view factor, A is the area,  $\sigma$  is the Stephan-Boltzmann constant (56.7x10<sup>-8</sup>Wm<sup>-2</sup>K<sup>4</sup> (Rogers & Mayhew, 1964)),  $T_2$  is the temperature of the radiating surface, and  $T_1$  is the temperature of the body receiving radiation (Çengel, 1998).

$$Q = \in F_{1-2}A\sigma(T_2^4 - T_1^4)$$
 Equation 2-5

Quartz lamps radiators can have an operating temperature of 2300°C (Ratti & Mujumdar, 2006). Assuming food at a temperature of 20°C with an emissivity of 0.9, a view factor of 0.9, the heat flux achieved using Equation 2-5 is 2.3x10<sup>6</sup>Wm<sup>-2</sup>. Assuming a hot air temperature of 120°C and a food temperature of 20°C, cross circulation driers only achieve heat fluxes of 2.8x10<sup>3</sup>Wm<sup>-2</sup> to 1.4x10<sup>4</sup>Wm<sup>-2</sup> (Arganbright & Resch, 1971). Gas circulation dryers consist of a continuous sheet or film of solids over which hot air is blown over one or both faces. Impingement drying can achieve heat fluxes of 1.4x10<sup>4</sup>Wm<sup>-2</sup> to 8.5x10<sup>4</sup>Wm<sup>-2</sup> to, again assuming a hot air temperature of 120°C and a food temperature of 20°C (Arganbright & Resch, 1971). Hence IR heating can produce heat transfer coefficients that are 27 times higher than impingement heating.

IR drying can be classed as a necessity when a dry crispy surface is required. IR can rapidly dry out surfaces due to the high heat fluxes that can be achieved, leaving it dehydrated and crispy. This is desirable on a wide variety of meat products such as bacon and sausages, as well as potato products and toast. IR drying in foods is versatile, simple, and has a fast transient response as radiative heat transfer is instantaneous. IR heating can be easily combined with conductive and microwave heating and can have significant energy savings. Nowak & Lewicki (2004) measured the energy efficiency of a hybrid IR and convection dryer to be between 35.2% and 45.7% for the drying of apple slices. Zbicinski, et al. (1992) and Evin (1992) measured IR heat efficiencies of 50% and 49% effectively. This compares to an energy efficency of 38% for a continuous flow air dryer as evaluated by Foster (1973). It can be highly efficient to convert electrical energy to IR (Quartz lamps have radiant efficiencies of 86% approximately (Ratti & Mujumdar, 2006)), radiation penetrates directly into the product without heating the surroundings, uniform heating of product surfaces can be achieved, IR sources are inexpensive compared to dielectric and microwave sources, and have a long service life and low maintenance. It's disadvantages are that scale up of the heaters is not always straightforward, it is essentially limited to surface drying, testing of the equipment must be carried out in the plant to assure successful design, burning and heat damage of product surfaces, and potential fire hazards must be considered in design and operation (Ratti & Mujumdar, 2006).

#### 2.3.2 Frying

Deep fat frying is one of the oldest processes to dry, to cook, and to formulate food products. Its success comes from its versatility and flexibility at the industrial and household scale. It offers several advantages that are difficult to reproduce with alternative technologies: very efficient heat transfer, crispy texture associated with rapid drying, and enhanced taste due to oil update (Achir, et al., 2009). Deep fat frying is suitable for foods of all shapes, but irregularly shaped foods or pieces with a greater surface to mass ratio tend to absorb and retain a greater volume of oil when it is removed from the fryer. Heat transfer coefficients are typically  $250-300 \text{Wm}^{-2}\text{K}^{-1}$  before evaporation of moisture from the surface begins, but subsequently increase to 800-1000Wm<sup>-</sup>  ${}^{2}$ K<sup>-1</sup> owing to the violent turbulence caused by steam escaping from the food (Fellows, 2000). For comparison, gas circulation dryers achieve heat transfer coefficients between  $30-140 \text{Wm}^{-2}\text{K}^{-1}$  while heat transfer coefficients between 140Wm<sup>-2</sup>K<sup>-1</sup> and 850Wm<sup>-2</sup>K<sup>-1</sup> can be achieved with impingement drying (Arganbright & Resch, 1971). The huge heat transfer coefficients in frying result in extremely short dehydration times, as such deep fat fried food is typically called "fast food". For instance potato crisps are dehydrated from approximately 80% moisture content to less than 2% in just two and a half to three minutes using deep fat frying (Desai, et al., 2014). The main disadvantage to frying is the oil uptake into the product and the associated health risks posed by high fat food. It is hard to control the amount of oil uptake and numerous studies have been carried out to better understand the oil uptake phenomenon (Aguilera & Gloria, 1997; Bouchon & Pyle, 2005; Dana & Saguy, 2006; Farkas, et al., 1996; Moreno, et al., 2010; Moyano & Pedreschi, 2006; Pinthus, et al., 1995 Southern, et al., 2000) and to reduce it (Mellema, 2003; Desai, et al., 2014). Other disadvantages include degradation of oil and oil usage (Blumentha & Slier, 1991), and production of potential carcinogens such as acrylamide (Kita, et al., 2004; Granda, et al., 2004; Tareke, et al., 2000; Lineback, et al., 2012) and Heterocyclic aromatic amines (Gibis, et al., 2015) at high temperatures.

# 2.4 RF and microwave heating

In RF and microwave heating, which is also called dielectric heating, heat is generated within the products by molecular friction in high-frequency alternating electric fields. RF and microwave heating are mainly applied to dielectric materials, which are poor electrical conductors. Generally, poor electrical conduction properties are associated with poor thermal conduction properties (Barber, 1983). Hence, it would take a relatively long time to heat dielectric materials conventionally using temperature gradients. Unlike conventional heating, dielectric heating is volumetric, which makes it fast because there is no time lag associated with conventional heat transfer mechanisms (conduction, convection, and radiation). (Metaxas & Meredith, 1993)

#### 2.4.1 The electromagnetic spectrum

Radio waves and microwaves are a form of electromagnetic energy, which travels in high frequency waves. Electromagnetic waves are self-propagating waves characterised by electric (E) and magnetic (H) field components oscillating in phase and perpendicular with each other and also perpendicular to the direction of propagation, as illustrated in Figure 2-8 (Metaxas & Meredith, 1993).


Figure 2-8 Electromagnetic wave components (Schulz, et al., 2013)

Electromagnetic waves obey the general wave formula given by Equation 2-6, where  $\lambda$  is the wavelength in free space (m), *c* is the speed of light which is a constant (3.0x10<sup>8</sup>ms<sup>-1</sup>), and *f* is frequency (s<sup>-1</sup> or Hz) (Metaxas & Meredith, 1993).

$$c = \lambda f$$
 Equation 2-6

The electromagnetic spectrum is split into bands, their approximate boundaries are described in Table 2-2. Although the table states that the dividing point between RF and microwave is 600MHz, the actual dividing point between the two adjacent frequency bands is imprecisely defined. The technology used to generate and transmit the high frequency electric fields can be used to distinguish them. RF systems are generally based on high power electrical valves (to produce the RF power), transmission lines (to carry the RF energy), and applicators in the form of capacitors; whereas microwave systems use magnetrons (to generate the microwaves), waveguides (to transport the microwaves) and cavities (in which the microwaves are applied) (Rowley, 2001). The frequencies allocated for RF and microwave heating are shown in Table 2-3 and are designated as ISM "Industrial, Scientific, and Medical".

Chapter 2 : RF and microwave food drying theory
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Region	Wavelength	Frequency [Hz]
X-Ray	0.01 – 10nm	$3x10^{19} - 3x10^{-16}$
Vacuum ultraviolet	10 – 200nm	$3x10^{-16} - 1.5x10^{15}$
Near ultraviolet	200 - 400nm	$1.5 \times 10^{15} - 7.5 \times 10^{14}$
Visible	400 - 800nm	$7.5 \times 10^{14} - 3.8 \times 10^{14}$
Near infrared	$0.8 - 2.5 \mu m$	$3.8 \times 10^{14} - 1 \times 10^{14}$
Fundamental infrared	2.5 – 50µm	$1 \times 10^{14} - 6 \times 10^{12}$
Far infrared	50 – 300µm	$6x10^{12} - 1x10^{12}$
Microwave	0.3mm – 0.5m	$1x10^{12} - 6x10^{8}$
Radiowave	>0.5m	$<6x10^{8}$

Table 2-2 Approximate boundaries for the electromagnetic spectrum(Brooks, 1998)

Frequency range	Band width	Centre frequency	Availability
6.765 MHz to 6.795 MHz	30kHz	6.780 MHz	Subject to local acceptance
13.553 MHz to 13.567 MHz	14kHz	13.560 MHz	Worldwide
26.957MHz to 27.283MHz	326kHz	27.120 MHz	Worldwide
40.660MHz to 40.700MHz	40kHz	40.680 MHz	Worldwide
433.050MHz to 434.790MHz	1.74MHz	433.920 MHz	Region 1**
886.000MHz to 906.000MHz	20MHz	896.000 MHz	UK
902.000MHz to 928.000 MHz	26MHz	915.000 MHz	Region 2
2.400GHz to 2.500GHz	100MHz	2.450 GHz	Worldwide

 Table 2-3 Frequency bands allocated for industrial use (\*\*Subject to local acceptance in a number of countries)

These bands were chosen by international agreement with the principle aim of minimising interference with communication devices and are defined in ITU (2012) in sections 5.138, 5.150, and 5.280. The UK differs in that it has an allocated frequency band at 896MHz rather than 915MHz (Metaxas & Meredith, 1993). It is worth noting that the bandwidth for 27.12MHz is considerably larger than for other RF frequencies. Most historical RF equipment has been made to operate at a frequency of 27.12MHz as it is much simpler to design dielectric heating equipment using a frequency with a greater tolerance allowence (Clark & Sutton, 1996).

## 2.4.2 RF and microwave frequency electromagnetic heating mechanisms

Any material can be heated using high frequency electromagnetic waves provided that the material is neither a perfect electrical conductor nor a perfect insulator. Ionic conduction and dipole rotation are the dominant mechanisms for RF and microwave heating of dielectrics (Jiao, et al., 2015), although it is also important to consider conduction heating which occurs on metallic surfaces.

## 2.4.2.1 Polarisation

Polarisation covers a number of heating mechanisms including electric polarisation, atomic polarisation, dipolar polarisation, and interfacial polarisation. Electric polarisation occurs at high frequencies (close to the ultraviolet), whilst atomic polarisation occurs predominantly at the infra-red frequency band. Interfacial polarisation occurs at frequencies lower than 10MHz in biological materials. Hence electric polarisation, atomic polarisation, and interfacial polarisation are of little significance for RF and microwave drying applications (Von Hippel, 1954; Tang, et al., 2002).

Dipolar polarisation is the most significant heating mechanism at microwave frequencies (Metaxas & Meredith, 1993). Molecules having a centre of symmetry such as methane (CH<sub>4</sub>) are non-polar and exhibit zero dipole moment (Sahin & Sumnu, 2006). However polar molecules such as water have electrical dipole moments, that is, the positive charge centres and negative centres do not coincide. When placed in an electrical field, polar molecules align themselves to the field. Negative and positive charges migrate to different ends of the molecules and lead to polarisation. If the electric field is alternating as shown in Figure 2-9, the polar molecules rotate continuously to align with the changing filed. This phenomenon is known as dipole rotation. In this among surrounding molecules process, the friction converts the electromagnetic energy into heat (Jiao, et al., 2015). In food, water is the primary component responsible for dipolar rotation (Sahin & Sumnu, 2006).



Figure 2-9 Dipolar rotation of a water molecule under the influence of an applied microwave electric field (Sahin & Sumnu, 2006)

### 2.4.2.2 Ionic conduction

Unlike polar molecules, dissociative ions in foods make a corresponding motion in an applied electric field. Similarly, the motion direction varies with alternating electric fields. Heat will also be generated within the material by friction among molecules caused by the oscillation of ions in the material (Jiao, et al., 2015). Salt, in its disassociated form, is predominantly responsible for ionic conduction in foods (Sahin & Sumnu, 2006).

#### 2.4.2.3 Conduction

If a wave is propagating in a conducting medium, the oscillating electric field interacts with free electrons, which sets up electrical currents (Grant & Phillips, 1980; Mehdizadeh, 2010). Work must be done to overcome resistance to the current, which results in some of the energy in the wave being dissipated as heat in the medium. As a plane wave progresses through a conducting media its energy is dissipated and it is consequently attenuated. The 'skin depth' is a measure of how rapidly the wave is attenuated. At RF and microwave frequencies the current is all carried in a thin outer layer of any conductor. This phenomenon is known as the skin effect. At RF and microwave frequencies, the resistance of a conductor can be estimated by assuming all the current is carried uniformly in a surface layer of thickness  $\delta$ , where  $\delta$  is the skin depth. The equation for skin depth is shown in Equation 2-7, where  $\mu$  is relative permeability,  $\mu_0$  is the permeability of free space [ $4\pi x 10^{-7}$ Hm<sup>-1</sup>],  $\sigma$  is electrical conductivity [Sm<sup>-1</sup>], and  $\omega$  is the angular frequency [rad.s<sup>-1</sup>] (Grant & Phillips, 1980).

$$\delta = \sqrt{\frac{2}{\mu\mu_0\sigma\omega}}$$

Equation 2-7

## 2.5 Dielectrics properties of foods

A dielectric material is an insulating material (with high resistivity and with a band gap of a few eV), that is polarizable i.e. in which electrostatic dipoles exist or form under the influence of an electric field (Blaise & Treheux, 2007). Dielectric materials, unlike metals, have no free charge carriers in them (Naidu, 2010; Mehdizadeh, 2010). Most food products are dielectric materials (Jiao, et al., 2015). The dielectric response of foods dictates their heating, and the uniformity of the heating, when subject to RF or microwave radiation. Dielectric properties are therefore instrumental in understanding RF/MW heating behaviour, and can be used to decide whether a material is suitable for RF/MW heating. Dielectric response is therefore the first thing that should be studied and understood before undertaking any investment in an RF or microwave heating process. Dielectric behaviour also dictates a large number of the advantages and disadvantages of microwave heating compared to RF heating e.g. penetration depth, evenness of heating, potential for arcing etc.

The dielectric properties of foods are the parameters that determine the coupling and distribution of electromagnetic energy during dielectric heating. Dielectric properties are described in terms of complex relative permittivity,  $\epsilon$ :

$$\varepsilon = \varepsilon' - j\varepsilon''$$
 Equation 2-8

The real part of the complex permittivity,  $\varepsilon'$ , known as the dielectric constant, describes the ability of a material to store energy in a reversible manner analogous to that of a capacitor in response to an applied electric field. The imaginary part of the relative complex permittivity,  $\varepsilon''$ , known as the loss factor, describes the ability of a material to dissipate energy in an irreversible manner analogous to a resister in response to an applied electric field (Wang, et al., 2003). As such, the dissipation of energy as heat in a material is directly proportional to  $\varepsilon''$  as shown in Equation 2-9 (Mehdizadeh, 2010).

$$Pd = \omega \varepsilon_0 \varepsilon^{"} E_{RMS}^2$$
 Equation 2-9

Where  $E_{RMS}$  is the root mean square of the electric field strength [Vm<sup>-1</sup>],  $\varepsilon_0$  is the permittivity of free space [8.854x10<sup>-12</sup>Fm<sup>-1</sup>], and *Pd* is the power density [Wm<sup>-3</sup>]. The loss factor is the sum of all the dielectric loss mechanisms. As previously stated, dielectric losses in foods are dominated by dipolar rotation and ionic conduction. It should be noted from Equation 2-9 that a higher frequency results in more energy dissipation for a given electric field and loss factor. Hence, depending on the variation of loss factor with frequency, higher electric fields may have to be used at RF frequencies than at microwave frequencies for a required amount of heating. This can lead to arcing issues.

The loss tangent, shown below, is used to indicate how absorbent or lossy a material is to microwave heating (Metaxas & Meredith, 1993).

$$Loss tangent = \frac{\varepsilon''}{\varepsilon'}$$
 Equation 2-10

When considering a plane wave propagating in the z direction, the electric field parallel to the x-axis,  $E_x$ , is described as follows:

$$E_x(z) = Ae^{\gamma z} + Be^{-\gamma z}$$
 Equation 2-11

Where the constants *A* and *B* correspond to the magnitude of the waves propagating in the -z and +z directions respectively.  $\gamma$  is known as the propagation constant, it is complex, and generally written in the following form where  $\alpha$  is known as the attenuation coefficient and  $\beta$  as the phase constant (Dibben, 2001):

$$\gamma = \alpha + j\beta$$
 Equation 2-12

The attenuation coefficient and the phase constant are related to the material properties by the following expressions (Dibben, 2001):

$$\alpha = \omega \sqrt{\frac{\mu_0 \mu_r \varepsilon_0 \varepsilon'}{2}} \left( \sqrt{1 + (\varepsilon'' / \varepsilon')^2} - 1 \right)^{1/2}$$
 Equation 2-13

$$\beta = \omega \sqrt{\frac{\mu_0 \mu_r \varepsilon_0 \varepsilon'}{2}} \left( \sqrt{1 + (\varepsilon''/\varepsilon')^2} + 1 \right)^{1/2}$$
Equation 2-14

When the medium in which the wave is travelling is lossy, the magnitude of the wave decays exponentially with distance (Dibben, 2001). This leads to limitations of RF and microwave volumetric heating. The penetration depth, shown in Equation 2-15, is defined as the distance from the surface of the material at which the power drops to  $e^{-1} = 0.368$  from its value at the surface, and it is dependent on both the dielectric constant and the loss factor.

$$\delta_p = \frac{1}{2\alpha} = \frac{\lambda}{2\pi\sqrt{2\varepsilon'}\left(\sqrt{1 + (\varepsilon''/\varepsilon')^2} - 1\right)^{1/2}}$$
Equation 2-15

Where the attenuation coefficient was simplified using substitution of the following expressions:

$$c = \frac{1}{\sqrt{\mu_0 \varepsilon_0}}$$
 Equation 2-16

$$c = \lambda f$$
 Equation 2-6

In general the penetration depths at RF frequencies below 100MHz are of the order of meters and presents little problem as far as power penetration for industrial processing of food. At microwave frequencies the penetration depths are correspondingly smaller and often the size of the material to be treated, particularly when very wet, is many times larger than the penetration depth, and microwave heating could result in unacceptable non-uniformities in the temperature distribution (Metaxas & Meredith, 1993). Salt in foods increases the loss factor and can significantly decrease the penetration depth of microwaves to the extent that some applications are no longer viable (Datta & Davidson, 2000). The larger penetration depth obtained at RF frequencies is a distinct advantage over microwave frequencies. This contributes to the

popularity of RF in finish drying applications that require uniform heating and uniform moisture distribution.

Both the dielectric constant and the loss factor are dependent on frequency, moisture content, and temperature.

## 2.5.1 Moisture dependency

As previously discussed foods systems are hygroscopic and contain water in either unbound or bound states. Bound moisture is generally referred to as the moisture in the monolayer, were the isosteric heat of sorption is very high. Bound water containing weak hydrogen bonds cannot freely rotate, and cannot support ionic conduction of salts (Mudgett, et al., 1971). The loss factor is therefore highly dependent on the amount of free water in the food system that can support these two heating mechanisms, hence water mobility is extremely important for dielectric heating of foods.

There is a critical moisture content below which loss factor is not affected significantly with moisture content as illustrated in Figure 2-10 (Sahin & Sumnu, 2006). The definition of the critical moisture content  $M_c$  is generally defined as the point of inflection where the gradient of loss factor with respect to moisture content increases. The change in the slope (before and after the critical moisture content) may be quite gradual for some materials, making positive identification difficult (Mehdizadeh, 2010; Schiffmann, 2006). Metaxas & Meredith (1993) suggested that a linear equation could be used to represent behaviour above and below the curve of inflection as shown by the dashed lines in Figure 2-10. The point at which these two linear lines meet is then defined as the critical moisture content, where Region I is lower than the critical moisture content, and Region II is higher than the critical moisture content mathematically. Metaxas & Meredith (1993) suggested that Equation 2-17 could provide a good fit to experimental data.

$$\varepsilon'' = \varepsilon_0'' + \frac{AM_{wb}^2}{M_\infty - M_{wb}}$$
 Equation 2-17

Where  $\varepsilon_0$  is the intercept, A and  $M_{\infty}$  are constants, and  $M_{wb}$  is the wet basis moisture content. However, it should be noted that there are no examples in literature of this equation being used to fit to the measured moisture dependant loss factor of foods. The equation can't adequately describe loss factor decreasing with respect to moisture content, which has been is observed in a wide variety of foods including potato, wheat flour, hard red winter wheat, apples and grapes (Mudgett, et al., 1980; Holtz, et al., 2010; Kim, et al., 1998; Nelson & Stetson, 1976; Feng, et al., 2002; Tulasidas, et al., 1995)



Figure 2-10 Variation of loss factor with moisture content showing how the critical moisture content as defined by Metaxas & Meredith (1993)

The relationship between loss factor and moisture content illustrated in Figure 2-10 is important for RF and microwave drying. As water is removed the dielectric loss decreases and hence, the material heats less well. In many cases, this leads to self-limitation of the heating as the material becomes relatively transparent to RF and microwaves at low moisture content. This has great value in obtaining moisture levelling, especially in thick materials, in which the electromagnetic energy is likely to preferentially dry wetter areas.

A study by Mudgett et al. (1980) measured both the sorption isotherm and the dielectric properties of freeze dried potato at 3GHz. BET analysis of the measured sorption isotherm gave a monolayer value of approximately 0.076kg.kg<sup>-1</sup> wet basis moisture content.

The dielectric properties of freeze dried potato are shown with respect to water activity and moisture content in Figure 2-11. Mudgett et al. (1980) suggested

that the results show a critical moisture content of 0.21kg.kg<sup>-1</sup> wet basis, or 0.86 water activity, which is well above the monolayer moisture content of 0.076kg.kg<sup>-1</sup> wet basis. The critical moisture content of 0.21kg.kg<sup>-1</sup> wet basis coincides with the point at which water exists in solution as free water i.e. the start of region C of the sorption isotherm. The fall in loss factor above a moisture content of 0.60kg.kg<sup>-1</sup> wet basis is attributed to dilution of the salts.



Figure 2-11 Dielectric constant and loss factor of freeze dried potato at 3GHz with respect to (a) water activity and (b) moisture content (Mudgett, et al., 1980)

This same behaviour was observed by Martin-Esparza et al. (2006) for apples vacuum impregnated with an isotonic solution, in that the critical loss factor moisture content occurred well above the monolayer moisture content. There are very few studies that present both dielectric and sorption properties, as a consequence their relationship is poorly understood.

## 2.5.2 Frequency dependency

The dielectric properties of most materials vary considerably with the frequency of the applied electric fields. Polar rotation generates a dispersion in the dielectric constant and a peak in loss factor with respect to frequency, the peak in loss factor occurs at the "relaxation frequency" (Nelson & Datta, 2001). A mathematical formula was developed by Debye to describe this process for pure polar materials. The Debye equation for real and imaginary parts is shown in Equation 2-18 and Equation 2-19 respectively (Nelson & Datta, 2001).

$$\varepsilon_{d} = \varepsilon_{\infty} + \frac{\varepsilon_{s} - \varepsilon_{\infty}}{1 + \omega^{2} \tau^{2}}$$
 Equation 2-18

$$\varepsilon_{d}^{*} = \frac{(\varepsilon_{s} - \varepsilon_{\infty})\omega\tau}{1 + \omega^{2}\tau^{2}}$$
 Equation 2-19

Where  $\varepsilon_{\infty}$  represents the dielectric constant at frequencies so high that molecular orientation does not have time to contribute to the polarization.  $\varepsilon_s$  represents the static dielectric constant, i.e., the value at zero frequency (dc value), this is illustrated in Figure 2-12 (Nelson & Datta, 2001).  $\tau$  is the relaxation time, it is defined as the time required for preferentially oriented molecules, under a static external electric field, to relax back to 1/e (or 36.8%) of the original condition on sudden removal of the external field. The relationship between relaxation frequency and relaxation time is expressed in Equation 2-20 (Nelson & Datta, 2001).

$$f_c = \frac{1}{2\pi\tau}$$
 Equation 2-20

The Debye equation provides a good fit for the frequency dependant dielectric behaviour of water (Kaatze, 1989). Using Debye parameters presented by Nelson & Datta (2001), the relaxation frequency of water is 17GHz.

There are many modifications of the Debye equations to better represent materials that are not purely polar, and materials that have a distribution of relaxation times. The Cole-Cole equation is used to more accurately describe the dielectric response of polar substances; the Double-Debye, Debye- $\Gamma$ , and the Davidson-Cole equation are used to describe materials with two relaxation frequencies.



Figure 2-12 Graphical representation of the Debye dielectric relaxation for water

Salt is typically present in both finished food products, usually with added sodium chloride, and in many raw food materials, which have significant salt content in the form of "ash". The "ash" content is usually dominated by potassium, hence it is sometimes referred to as "potash". The potassium content in sweet potato is 78.8% of the total ash content (Woolfe, 1992).

Salts bind water molecules when dissolved and decrease their ability to reorient themselves in response to the changing electromagnetic field direction, which suppresses the dielectric constant (Sipahioglu & Barringer, 2003). The supression of the dielectric constant due to ionic conduction occurs to a lesser extent at frequencies higher than the relaxation frequency disperion as shown in Figure 2-13a. Salt content has a far stronger effect on the loss factor, which increases drastically at lower frequencies due to ionic conduction as shown in Figure 2-13b. The effect of ionic conduction is so large that the dipolar rotation peak at 17GHz is barely visible inFigure 2-13b. Figure 2-13b shows that food can generally considered to be far more lossy at RF frequencies. Salt content can greatly reduce the penetration depth due to the large increase in loss factor.



Figure 2-13 (a) Dielectric constant and (b) loss factor of water with different salt concentrations, varying with respect to frequency

As well as water, most foods typically contain large quantities of starch, protein and oil. These items can be considered to be dielectrically inert compared to water at common ISM frequencies with each having a loss factor of 0.5 or less (Bansal, et al., 2015; Ndife, et al., 1998; Ahmed, et al., 2008;

Cataldo, et al., 2009; To, et al., 1974). RF and microwave drying is ideally suited to foods as the loss factor of water is typically very high compared to other food constituants, so moisture can be selectively heated.

The complex permittivity is typically made up of a combination of ionic conduction and dipolar rotation in the RF and microwave frequency ranges. In heterogeneous materials, a number of relaxation frequencies may be apparent. Each peak in loss factor relates to the relaxation frequency of an individual constituent of the heterogeneous body. Figure 2-14 shows a typical trend for the dielectric constant and loss factor over a frequency range for a heterogeneous material.

Mashimo & Kuwabara (1987) measured a wide range of living materials and found absorption peaks around 100MHz and 20GHz which was attributed to bound water and free water respectively. The loss factor of bound water in potato and chicken are shown at ISM band frequencies in Table 2-4. Although there is no ISM band near the relaxation frequency of bound water, the loss factor dispersion is very broad, which makes it relatively easy to excite.



Figure 2-14 (a) dielectric constant for heterogeneous material (b) Loss factor due to dipolar rotation for a heterogeneous material (c) Loss factor due to both dipolar rotation and ionic conduction for a heterogeneous material (Roussy G and Pearce J.A., 1995)

ISM band	Potato bound water loss factor	Chicken muscle bound water loss factor
13.56MHz	3.4	3.3
27.12MHz	6.6	6.3
40.68MHz	9.2	9.0
433.92MHz	7.5	8.9
896MHz	3.8	4.6
915MHz	3.7	4.5
2.45GHz	1.4	1.7

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 Table 2-4 Bound water loss factor for potato and chicken at ISM band

 frequencies

## 2.5.3 Temperature dependency

It can be observed from Figure 2-15 that the dispersion peak in loss factor decreases with temperature for free water, as well as moving to higher frequencies (Tang, et al., 2002). The change in dispersion peak, or relaxation frequency, means that the loss factor may either increase or decrease with increasing temperature, depending on whether the operating frequency is higher or lower than the relaxation frequency (Nelson & Datta, 2001). It can be seen that the dielectric constant decreases with respect to temperature before the region of dispersion i.e.  $\varepsilon_s$  decreases with increasing temperature. The decrease in  $\varepsilon_s$  is a result of increased Brownian movement at higher temperatures. During the region of dispersion the dielectric constant will increase with temperature due to the shift in relaxation frequency (Tang, et al., 2002).

The decrease in water viscosity with increasing temperature facilitates an increase in ion mobility, which leads to more frictional heating. At higher temperatures, water activity increases, ions become more mobile and not tightly bound to water, and thus the loss factor from the ionic loss component increases with temperature (Sahin & Sumnu, 2006; Wang & Brennan, 1991). A generalised plot showing the temperature dependency of ionic and dipolar loss mechanisms is illustrated in Figure 2-16.



Figure 2-15 Effect of temperature on  $\varepsilon$  and  $\varepsilon$  of water (Ryynanen S., 1995)



Figure 2-16 Contribution of different dispersion mechanisms in biological materials as affected by frequency and temperature (Tang, et al., 2002)

Ionic conduction causes the dielectric constant to increase with increasing temperature. This is the opposite of the effect of dipolar rotation, which decreases the dielectric constant with increasing temperature at frequencies lower than the relaxation frequency of water as shown in Figure 2-15. Due to

the salt content in food, and the dominance of ionic conduction at lower frequencies, there are many examples of the dielectric constant rising with temperature at lower frequencies (Nelson, 2005; Guo, et al., 2008).

Increasing the temperature provides energy to free some of the bound water molecules. If there is a high ratio of bound water compared to free water in the food, then an increase in temperature can increase both the dielectric constant and the loss factor if sufficient energy is provided to free some of the bound water (Sahin & Sumnu, 2006; Nelson, 1991a; Guo, et al., 2008).

An increase in loss factor with respect to temperature can lead to thermal runaway. This is a mechanism where loss factor increases with respect to temperature, leading more energy dissipation which in turn leads to further increases in loss factor. This can lead to overheating which can in turn lead to charring, smoke, arcing and fires. Thermal runaway is rarely an issue for food drying at intermediate moisture contents as any increase in loss factor due to temperature is tempered with a decrease in loss factor due to dehydration. Thermal runaway can become a problem when drying food with low moisture content.

## 2.5.4 Models used to describe dielectric behaviour of foods

A number of attempts have been made to generate predictive models for the dielectric behaviour of foods. These attempts have been hampered by the quality of literature dielectric data, which shows much variability arising from the inaccuracy of different measuring techniques, changes in composition, etc. Although the data depend strongly on composition, such as water and salt content, detailed composition data often do not accompany the dielectric property data (Sun, et al., 1995).

Mudgett et al. (1971) and To et al. (1974) attempted to describe dielectric behiavour of aqueous solutions of nonfat dried milk according to its chemical composition using the theory of unsaturated aqueous salt solutions as described by the Hasted-Debye model (Hasted, et al., 1948). However, the loss factor

was too low, by approximatley 50% in some instances, this was attributed to bound or dissociated ions and solute-solvent interaction.

To et al. (1974) did successfully calculate the dielectric properties of meat juice which had a high moisture content of 93.1%. An average prediction error of 7.5% was achieved at different frequencies and temperatures. The method was truly predictive, but it did rely on being able to measure the electrical conductivity, which is not possible for lower moisture content foods. There has been considerable success in modelling the dielectric properties of fluid mixtures. Mudgett et al. (1974) showed that the dielectric properties for oil-water emulsions of varying composition at 3GHz, 25°C were predicted by the Fricke (1955) model, whilst Buck (1965) and Roebuck et al. (1972) showed that the dielectric properties of alcohol-water mixtures could be predicted using the Maxwell model.

Mudgett et al. (1977) proposed a distributive model which was used to describe pototato dielectric properties with a maximum error of approximately 10% at the three frequenices of 300MHz, 915MHz, and 3GHz. The distributive model is shown below where  $\varepsilon_c^*$  is the complex permittivity of the aqueous ionic fluids,  $\varepsilon_s^*$  is the complex permittivity of the solid food and colloidal solids,  $V_s$  is the volume fraction of the solids, and  $V_c$  is the volume fraction of the fluids.

$$\varepsilon^* = \varepsilon_c^* V_c + \varepsilon_s^* V_s$$
 Equation 2-21

However, this model was only used to predict dielectric behaviour of potato with 85% moisture content. It should also be noted that the dielectric constant measured for potato by Mudgett et al. (1977) was approximately 70, whereas Pace et al. (1968) mesasured approximately 80 at the same frequencies. In a later study, Mudgett et al. (1980) measured the dielectric properties of potato with respect to moisture content, and compared the results to distributive model predictions. The model was only able to describe dielectric behaviour above a moisture content of 50%. Dielectric properties were far lower than predicted below this moisture content due to reduced water and ion mobility caused by binding.

Dielectric behaviour with respect to moisture content is very complex, and is dependant on the biology of the food. Most studies that desicribe dielectric properties in terms of moisture content, generate models that are specific to the food using regression fits of measured dielectric data, such as a study carried out by Prakash et al. (1992) on hydrocolloids. Such equation fits are food specific, and non-predictive. General equations have been successfully created for fully hydrated foods. Sun et al. (1995) fitted an equation to meats, while and Sipahioglu & Barringer (2003) fitted an equation to fruits, and an equation to vegetables, and Guan et al. (2004) fitted an equation for mashed potato. However, these studies do not capture the full range of moisture dependance

The research carried out to date suggests that it is not possible to generate a general prediction model for moisture dependant dielectric properties of food. The moisture and temperature dependence of dielectric properties is dependant on complex interactions with the food constituants. Hence predictive models cannot be used to predict the moisture dependant dielectric properties of a reasonably complex food from properties of its constituants.

Mixture models have however been developed which predict the dielectric properties of packed food or particulates. Nelson et al. (1991b) found that the Bottcher, Landau and Lifshitz, Looyenga equations agreed very closely in their estimates of the permittivity of the solid material, whereas the Bruggeman-Hanai, Rayleigh, and Lichenecker equations gave increasingly large values in that order. The mixture equations are based on volume fractions which are unfortunately difficult to obtain (Sun, et al., 1995).

## 2.5.5 Dielectric measurement techniques

Although many techniques have been developed for dielectric property measurement, the selection of the appropriate technique depends on the nature of the dielectric material, the frequency of interest, the degree of accuracy required, and the availability of measurement equipment (Datta, et al., 2005). For frequencies of less than 100MHz, parallel plate bridge and Q-metre methods are employed but are not used for measurement of microwave frequencies (Bussey, 1967; Metaxas & Meredith, 1993). A selection of

methods used for dielectric measurement of foods at microwave frequencies is described in the following subsections.

#### 2.5.5.1 Waveguide and coaxial transmission line methods

The loss factor and the dielectric constant can be determined by measuring the phase and amplitude of microwave signals reflected from or transmitted through a sample of material. For a waveguide structure, rectangular samples that fit into the dimensions of the waveguide at the frequency being measured are essential. For coaxial lines, an annular sample is required (Datta, et al., 2005). This method is useful for accurately measuring packed foods or particulates. The measurement technique only allows a relatively short bandwidth of frequencies to be measured (for example 900-1000Hz) and calibration can be time consuming.

#### 2.5.5.2 Short-circuited line technique

A terminating face such as a metallic barrier is placed in a coxial transmission line causing the transmitted electromagnetic wave to be reflected back to the source. The field strength at any given point within the transmission line is simply a vector sum of the strengths of the incident and reflected waves. The standing wave ratio (SWR) is defined as the ratio of the vector sum of the strengths at maximum to that of minimum. The insertion of a dielectric material into the transmission line in contact with the short-circuit termination causes changes in the position and the width of the standing wave nodes. The changes in position of the node and the SWR are used to calculate the dielectric constant and loss factor of the inserted dielectric material. The technique can be used to measure the dielectric properties of liquid, powder, or solid samples. The limitation of the technique when dealing with a solid sample lies in the sample preparation (Datta, et al., 2005). An annular sample has to be prepared, which may be time consuming, or it may not be possible in instances where food structure must be retained. At lower frequencies coaxial lines can be more practical than waveguides with regards to size (Datta, et al., 2005).

#### 2.5.5.3 Open ended coaxial line

The coaxial probe method, used for broadband measurement of high loss dielectric mateirals, circumvents many disadvantages of the transmission line technique (Datta, et al., 2005). The method, using an open-ended coaxial-line probe with network and impedence analysers, measures the reflection coefficient with a dielectric sample in contact with the probe and calculates permittivity (Nelson, 2010). The probe consists of an inner conductor, an outer conductor forming a flange at the right end and an insulator filling between the two conductors as shown in Figure 2-17.



Figure 2-17 Flanged coaxial probe (Al-Harasheh, 2005)

This method is well suited for braodband measurements on liquid and semisolid high loss materials with which problematic air voids between the tip of the probe and the sample can generally be avoided (Nelson, 2010). Care must be taken that the tempeature used for calibration (typically air, water and short circuit) is the same as the temperature of the dielectric sample, and that there has been no temperature drift between calibration and measurement of the dielectric sample. A single coaxial probe can typically operate over a frequency range of about 30:1 with uncertainty for real permittivity in the order of  $\pm 3\%$  for suitable materials. The frequency range depends on the diameter of the coaxial aperture, for example a coaxial probe with 7 mm aperture can operate from 200 MHz to 6 GHz. The best measurement accuracy and resolution is achieved around the centre frequency of this band. Air gaps between the specimen and the sample introduces error, and can be difficult to avoid with hard solid specimens (Krupka, 2006). The sample measured is relatively small, so measurents issues can arise due to the homegenity of granular or particulate materials (Nelson & Bartley, Jr., 1998).

## 2.5.5.4 Cavity perturbation techniques

Methods based on the electromagnetic field perturbation of a resonant cavity by insertion of a small sample have been used extensively for measuring the dielectric properties of low loss materials. This necessitates measuring the shift of the cavity resonant frequency away from the original unperturbed value and the change in the cavity Q-factor before and after sample insertion. The dimensions of the sample must be small compared with the size of the cavity, resulting in a small frequency shift and ensuring the validity of the perturbation theory, which assumes the basic electric field configuration of the cavity remains relatively intact (Metaxas & Meredith, 1993; Mehdizadeh, 2010). Furthermore, correct positioning of the sample in the cavity is important to preserve symmetry (Metaxas & Meredith, 1993). As small samples are used, this method may require grinding the food sample. This method can only be used for specific frequencies, and may not be suitable if retention of the food structure is deemed important.

## 2.5.5.5 Free space transmission technique

In free space transmission technique a sample is placed between a transmitting antenna and a receiving antenna, and the attenuation and phase shift of the signal are measured. The results are used to compute the material dielectric properties (Datta, et al., 2005). Although typically less accurate, this method may be easily implemented in industrial applications for continuous monitoring and control.

## 2.6 Application of RF and microwave for drying of foods

# 2.6.1 Advantages and disadvantages of RF and microwave drying of foods

## 2.6.1.1 Advantages

## • Volumetric heating

This can result in the product being heated more evenly than in conventional heat transfer processes. This is especially important in the falling rate drying period and can drastically reduce drying times (Metaxas, 1996; Erle, 2005 Chandrasekaran, et al., 2013; Schiffmann, 2006). When the moisture can be

heated to the boiling point of the liquid phase, the pores are filled with vapour and the moisture flow is now governed by the total pressure. Moisture is "pumped" out of the food matrix and into the surrounding air (Metaxas, 1996). However, the degree of volumetric heating depends on the penetration depth, penetration depth is greater at RF frequencies due to the longer wavelength.

#### • Direct and instantaneous heating of food

MW/RF heating in the food occurs instaneously. There is no intermediate material necessary to facilitate the heating of food, unlike with conduction, convection and radation heat transfer mechanisms. Hence RF and MW heating is faster as there is no transient heating associated with intermediate materials. As there is no intermediate material necessary to facilitate the heating of the food, RF and microwave heating is more efficient than conventional heating.

#### • Moisture levelling

RF and microwave energy selectively heats water in foods, improving efficiency, and in some instances producing a more even moisture distribution of the finished product. This can improve quality and preservation of the product. This effect is complicated by the fact that water and salts lose mobility and become bound at lower moisture contents. Schiffmann (2006) was of the opinion that miosture levelling would be quite effective above the critical moisture content, but not so effective below it. As it is easier to achieve large even fields at RF due to its lower wavelength, and penetration depth is greater at RF frequencies, RF can be better suited to certain moisture levelling and finish drying applications. (Mehdizadeh, 2010; Koral, 2004).

#### • Reduced burning

RF and microwave heating can be used to dry food more aggressively than conventional processes as water is selectively heated. Water requires latent heat to convert to steam and free water is limited in temperature to its boilding point of 100°C. Hence selective heating of water can limit the temperature reached and reduce burning. Sensorial and nutritional damage caused by long drying times or high surface temperatures can be prevented using RF/microwave drying (Shaheen, et al., 2012).

• Increase in production

As RF/microwave energy can reduce drying times through fast, selective, volumetric heating, drying time is decreased which leads to in an increase in production. RF/microwave energy can be used either as a temporary boost to production (giving the manufacturer flexibility) or as a permenant step up in production.

• Decrease in factory footprint

RF/microwave ovens typically have smaller factory footprints than their conventional alternatives (Schiffmann, 2001a). Unlike RF/MW dryers, conventional dryers are designed to provide required contact/surface area for heat transfer between the product and a medium.

#### 2.6.1.2 Disadvantages

• Uniformity of heating

Uniformity of volumetric heating can be an issue and is caused by insufficient penetration depth, wavelengths that are small relative to the size of the applicator, applicator design, and geometry of the heated material. Lack of uniform heating is a problem experienced predominantly at microwave frequencies, where wavelengths and the associated penetration depths are small. This problem can be especially acute during scale up, where larger applicators are employed using the same frequency as for the much smaller pilot plant.

#### • Arcing

Arcing is a high voltage discharge which can occur in high electric fields. Breakdown in air is seen at a value of 30kV/cm, at standard temperature and pressure. The breakdown level is affected by dielectric load conditions and occurs at lower electric field values if the pressure is reduced (sometimes vacuum is preferred for various processes) (Reader, 2001). Generally, very high electric field strengths are required at RF frequencies to achieve a reasonable rate of temperature rise in materials with a loss factor below  $10^{-2}$  (Holtz, et al., 2010). Arcing headroom is the difference between the highest electric field in the applicator during normal operation, and the electric field at

which arcing occurs. When arcing is the limiting factor there is motivation to reduce the arcing head room as much as possible in order to maximise power input, which improves throughput and minimises the footprint of the dryer. This can lead to arcing issues when there are unusual variations in the input material, such as variation in moisture content, foreign matter, or burnt carbonised material (Clark, 1997; Schiffmann, 2001b). As arcs can damage the product, the conveyor belt, and possibly lead to some unforeseen downtime, the RF industry has introduced arc suppression measures, utilising phenomena such as pre-arc frequency shift (Clark D.C., 1997; Metaxas A. C., 1996).

#### • Thermal runaway

Thermal runaway occurs when an increase in loss factor is experienced with an increase in temperature. This leads to increased non-uniform heating, and eventually carbonisation or burning. Burning or carbonisation does not typically occur when RF/MW heating high to intermediate moisture content foods as the temperature is limited by free water, which cannot rise above 100°C. The risk of burning due to thermal runaway is highest when high intensity electric fields are used to dry product to very low moisture contents, although measurements by Mudgett et al. (1974), Nelson (1991a), and Guo et al. (2008) showed that dehydrated food is not very sensitive to changes in temperature. Hence, the risk of thermal runaway in food processing may be considered relatively low. When thermal runaway does occur, it invariably degenerates into arcing and carbon formation, which produces profoundly undesired flavours (Gard, et al., 2014).

#### • Cost

RF/MW equipment is capital intensive and the economics of a particular application must be thoroughly examined before any equipment is installed in industry. Furthermore, taking the efficiency of converting prime fuel to electricity to be about 30% and a mains electricity to heat into the product efficiency of 65% gives an overall fuel efficiency of about 20%. However, many conventional heating drying processing plants exhibit efficiencies considerably less than 20% (Metaxas & Meredith, 1993).

Vacuum electron tubes have been known to have reliability issues, have a limited life and are therefore treated as consumables. The major consumable cost with any industrial RF or microwave generator is the cost of the triode tube or magnetron. Kilowatt for kilowatt, the cost of an industrial triode is significantly lower than the cost of an industrial magnetron. Also, the average life of a triode in a RF heating system is around 20,000 hours (3 years or more), compared with a typical magnetron life of 3000 to 6000 hours (Koral, 2015).

#### • Scale up

Scale up of RF and microwave dryers from experimental pilot dryers to viable commercial propositions can prove problematic (Koral, 2015). Key issues include cost, limitations in power sources, heating uniformity, and final product quality. In some applications, multiple vacuum electron tubes may be necessary to achieve the required throughput, which increases cost and complexity compared to a pilot plant. On a more practical level, single RF heating systems can be acquired at much higher power levels than that at microwave frequencies (Mehdizadeh M., 2010). Conveyor belts and associated parts can experience excessive heating as a result of scale up, especially if the food being dried is low loss, as is the case for finish drying (Koral, 2015). Careful attention of applicator materials is required during scale up, as the additional power may render some materials unsuitable. The selection of materials compatible with a large scale RF/MW oven can increase costs.

• Food texture

RF and microwave heating is volumetric, and an external crust region is not generated during RF/microwave heating. Hence, RF/microwave heating cannot be used to replace conventional cooking techniques in instances where external outer crusts are desirable on the food.

#### • Effect on food structure

If the solid matrix is not rigid, the excess pressure can cause the material to expand, thereby increasing the pores size (Metaxas, 1996). This can change the

taste of the product, increase its bulk which can be undesirable for transport and packaging, or result in a product that is not aesthetically pleasing.

## 2.6.2 Main components of a microwave system

The main components of a microwave system are: the magnetron, the circulator, the waveguide, and the applicator. Figure 2-18 shows a block diagram of a typical microwave system. The magnetron is the preferred source of electromagnetic waves due to its efficiency and size, and they are currently capable of delivering up to 100kW of power (Meredith, 1998; E2V Technologies Limited, 2002).



Figure 2-18 Generic microwave heating block diagram (Reader, 2001)

Matching techniques are used to minimise the amount of reflected power and the circulators function is to prevent reflected energy from damaging the magnetron. Waveguides are typically used to transmit energy from the magnetron to the applicator (Saltiel & Datta, 1999).

The applicator transfers the electromagnetic energy into the workload. The size and shape of the applicator depends on the operating frequency, the dielectric properties, and the shape and size of the processed material (Saltiel & Datta, 1999). Common microwave applicators include travelling wave applicators, multimode and single mode resonant cavities (Metaxas & Meredith, 1993).

Multimode resonant cavities are by far the most commonly used form of high frequency applicator (Mehdizadeh, 2010; Metaxas & Meredith, 1993). In essence, any metal enclosure a few times larger than the wavelength can serve

as a basic multimode microwave applicator. Such devices, unlike many other high frequency applicator systems, do not need a skilled operator or sophisticated tuning devices (Mehdizadeh, 2010). Mode stirrers and randomisers improve heating non-uniformity in batch ovens, while in continuous or conveyor belt multimode ovens, product movement accomplishes the same purpose (Reader, 2001). Key features of multimode ovens include:

- Suitability for bulk processing applications
- Oven dimensions that are often determined by product dimensions
- Moderate to high efficiency
- Adaptability to batch or continuous product flow
- Performance that is less sensitive to product position or geometry
- Good uniformity that may require motion of product or hybrid heating (National Research Council, 1994)

If the matching system is effective, a multimode cavity will always input a certain amount of heat. This can be problematic if there is any abnormalities in the dielectric load, or if the feed is interrupted. In order to prevent overheating the food, interlocks can be added to multimode applicator systems to switch the power off in instances were the flow of the load is interrupted. Temperature sensors can also be added to the microwave oven that can be used to trigger the system (Ferrite Microwave Technologies, 2017).

Meander travelling wave applicators can be of interest for finish drying applications as high levels of electric field uniformity can be achieved. In this applicator, the feed travels at right angles to the waveguide. The waveguide is arranged in a serpentine shape so that a number of passes can be achieved. The waveguide terminates at a matched water load. If there is an interruption in the feed, then extra heat is lost in the water load. However, this type of applicator is not suitable for low loss materials that have a loss factor approaching 0.1 as the number of passes required for an even electric field would not be practical (Metaxas & Meredith, 1993). This limits the use of meander travelling wave applicators for food finish drying applications.

#### 2.6.3 Main components of an RF system

In general RF applicators are connected to the oscillatory circuit generating the power (the tank circuit) via an inductive loop and other circuit elements which constitute the matching circuit. The vacuum triode or tetrode tube is the main device for the production of radio frequency energy in high power industrial applications (Metaxas, 1996).

There are two distinct braches of industrial RF heater design, free-running oscillators (FRO), or 50-ohm technology. In the food industry, virtually 98% of all industrial sized RF applications actually running in a production situation are based on the FRO. In free-running oscillator (FRO) systems the frequency is not stabilised by crystal control or other means. The achieved frequency stability depends upon the specific design characteristics of the circuit. Modern designs use a "high-Q" transformer-coupled circuit that greatly improves the frequency stability and performance, making it possible, with careful design, to maintain the frequency within the target ISM frequency band. The main advantages of the FRO system are cost, simplicity and flexibility. FRO systems have the ability to handle a wide range of adjustment for different load dimensions and dielectric loss factor variations. The main disadvantages of FRO systems are lack of frequency stability and harmonic output. The free running triode oscillator circuit has a very significant advantage in drying, as it can be dynamically matched to the load. The power absorbed by the load is then automatically controlled by the load itself. An RF dryer designed in this way is uniquely suited to moisture control. It is able to profile the moisture content of the product passing through the dryer. If there is high moisture in the product, then more RF power is drawn automatically. If there is less moisture in the product, or if the feed is interrupted, the power is then stored in the high power source and not drawn out (Koral, 2015).

The main basic types of RF applicators are parallel plate, fringing field, and staggered throughfield applicators. Parallel plate applicators are used mostly for relatively thick materials such as meat blocks, woollen bales, and textile packages (Metaxas A. C., 1996). Fringing field applicators can achieve better

electrical coupling between the material and the electrode array compared to capacitor plate applicators, which means less chance of arc formation (Roussy G and Pearce J.A., 1995; Rowley A.T., 2001). The major disadvantage of fringing field applicators is that only relatively thin layers of product can be used, otherwise there will be an electric field variation throughout the product thickness (Roussy G and Pearce J.A., 1995). Staggered throughfield applicators also have good coupling, and are used for intermediate thickness products, typically up to 10mm in thickness, in RF post-baking applications such as biscuits (Metaxas A. C., 1996).

# 2.7 Applications for RF and microwave drying in the food industry

Growth in the microwave heating industry has been slow since its inception in the 1950s (Metaxas & Meredith, 1993; Thostenson & Chou, 1999). Schiffmann (2001a) estimated that there are less than 1000 RF/MW driers inistalled in the USA, with a total sales value of \$500 million, and a total power of 100MW. This compares to a total power of 100,000MW for the total power of domestic microwave ovens in the USA. The estimated number of installed microwave systems and the status of microwave applications in the food industry are shown in the table below. Most of the applications listed below operate at 915MHz. It can be seen that the most successful microwave application is meat tempering, which is not a drying application. A large number of microwave drying applications have fallen into disuse in the food industry as cab be seen in Table 2-5 (Schiffmann, 2001a).

Process	Installed systems	Status
Potato crisp drying	>100	None operating
Pasta drying	>20	Very few operating
Donut proofing	>20	None operating
Donut frying	5-10	None operating
Meat tempering	>400	Very sucessful
Bacon cooking	>25	Very sucessful
Low fat potato crisps	>100	None operating

Table 2-5 Status of microwave applications in the food industry

Post baking drying is the predominant application at RF frequencies. This includes, biscuits, puff pastry, choux pastry (i.e. elcairs and pofiteroles) and sponges. Post baking of biscuits is the oldest and best established application of RF heating in the food industry, with around 400 or so installations worldwide (Awuah, et al., 2015). Based on the numbers discussed, it can be seen that RF frequencies are most commonly used for drying applications due primarily to the large number of post baking finish drying applications. The vast majority of post baking RF dryers operate at 27.12MHz (Koral, 2004).

Although it may appear that relatively few MW/RF applicators are operating in the food industry, MW/RF drying is perhaps being used more than is realised as sucessful processes are rarely disclosed by industry (Clark & Sutton, 1996). There may be hundreds of besoke microwave drying systems operating, most under a cloud of secrecy (Schiffmann, 2001a). There are many reasons for its limited use including cost, technical difficulties, and lack of expertise (Metaxas & Meredith, 1993; Schiffmann, 2001a). Schiffmann (2001a) has listed a number of formerly sucessful applications that have been discontinued which would suggest that there may even be a decline in the use of microwave drying in the food indsutry. Among the discontinued RF/MW applications cited were finish drying of potato crisps, pasta drying, snack drying, cake baking, doughnut processing, and the finish drying of biscuits and crackers. It is apparently not always the microwave process itself, but rather changes in the circumstances of production that make competing techniologies more successful.

Four RF/MW applications has been identified for further study as a result of a literature review, and of communication with experts in the food industry. The two new applications identified are MW/RF finish drying of potato crisps to reduce acrylamide, and explosive MW dehydration of potato crisps to control fat content and reduce carbon emissions. With respect to established applications, RF/MW finish drying of biscuits, and MW drying of pasta has been identified for further study. As already discussed, finish drying of biscuits is probably the most successful application of RF/MW drying in the food industry, it would be useful to cross check relevant new findings in this study

using this application. Pasta drying is of interest as is one of the most successful applications in the food industry with respect to cited reduction in drying times (Schiffmann, 2001a).

## 2.7.1 RF/microwave drying of potato crisps post frying

Potato crisps are one of the most popular snack products in the world and are by far the largest single category of savoury snack food in the UK with annual sales in excess of £2billion (Wu, et al., 2013). They are also among the products that have been reported to have the highest levels of acrylamide (Kita, et al., 2004), where acrylamide is a known carcinogen (IARC, 1994; Lineback, et al., 2012).

Carbohydrate foods such as potato and cereals that have a high content of the amino acid asparagine, and a high accumulation of reducing sugars, are subject to the formation of acrylamide upon high temperature processes such as frying (Granda, et al., 2004; Tareke, et al., 2000; Lineback, et al., 2012). Acrylamide is not present in native (raw) ingredients, such as raw potatoes, it is formed during heat processing. Capuano & Fogliano (2011) stated that acrylamide begins to form at temepratures above 100°C and it has been found that acrylamide forms readily when potatoes are heated above temperatures of 120°C (Tareke, et al., 2002). These temperatures are encountered in processes such as frying, boiling, baking, roasting, grilling, and toasting (Lineback, et al., 2012). Because acrylamide is a potential carcinogen for humans, several works have been devoted to the study of the mechanism of its formation and factors influencing its formation. Still, there are few solutions on how to decrease acrylamide creating during processing (Kita, et al., 2004).

The critical point when acrylamide is formed during crisps processing is frying. It has been shown that the most important factors influencing acrylamide formation is temperature and time of frying. The final moisture content of potato crisps is 0.01-0.02kg.kg<sup>-1</sup> wet basis (Schiffmann, 2001a; Desai, et al., 2014; Bows, et al., 2010). Kita et al. (2004) assessed acrylamide levels with reduced frying temperatures or reduced frying times, introducing a post-frying

air drying process at 105°C to obtain the correct final moisture content. Kita et al. (2004) reported a 70% decrease in acrylamide content after 2 minutes of frying at 185°C and 75 minutes of post-frying drying with 105°C air. A larger decrease of more than 80% was achieved by frying for 3 minutes at 160°C and 75 minutes of post-frying drying with 105°C air. However, the 185°C frying process described took 4.5-5 minutes to achieve the desired final moisture content of less than 0.02kg.kg<sup>-1</sup> without post-drying with air. This frying time is long compared to the standard continuous fried process, which takes 2.5 to 3 minutes at 185°C to achieve a final moisture content less than 0.02kg.kg<sup>-1</sup> (Bows, et al., 2010; Granda, et al., 2004). This would suggest that the frying carrying out by Kita et al. (2004) may have had poor temperature control, and the actual frying temperature may have dropped lower than 185°C. Frying for 2 minutes at 185°C resulted in a moisture content of approximately 0.22kg.kg<sup>-1</sup> wet basis in the Kita et al. (2004) study. Frying for 2 minutes at 185°C on a standard continuous frying process results in a moisture content of approximately 0.04kg.kg<sup>-1</sup> wet basis as presented by Bows et al. (2010). Frying for 3 minutes at 160°C resulted in a moisture content of approximately 0.29kg.kg<sup>-1</sup> wet basis in the Kita et al. (2004) study. The major problem with the suggested reductions of acrylamide in the study conducted by Kita et al. (2004) is the moisture contents at the end of the frying process. The glass transition of potato is approximately 0.04 to 0.05kg.kg<sup>-1</sup> wet basis at 185°C (Mizuno, et al., 1998). Hence, potato crisps leaving the fryer at moisture contents of 0.22kg.kg<sup>-1</sup> or 0.29kg.kg<sup>-1</sup> wet basis would be exiting the frying process above the glass transition point. As there is still a large amount of water in the potato crisps, and the crisp would still be above the glass transition in the fryer, the potato crisp will not have undergone all the structural changes associated with the standard continuous fried process. Hence the crisps porosity will not be fully developed, as this is an explosive process and a slow finish drying process from 0.22kg.kg<sup>-1</sup> wet basis down to less than 0.02kg.kg<sup>-1</sup> is likely to result in a very crunchy crisp that may not be desirable to the consumer.

The standard drying curve for a continuous fried potato crisp is shown in Figure 2-19. This was adapted from the patent submitted by Bows et al. (2010)

which replicated the frying curve using a microwave technique. The continuous frying curve for potato crisps shows that the drying rate is very slow after about 90 seconds. The moisture content at 90 seconds is approximatley 0.04kg.kg<sup>-1</sup>, and it takes a further 90 seconds in the fryer to achieve the final moisture content below 0.02kg.kg<sup>-1</sup>. After the initial 90 seconds, the potato crisp structure should be set as it is below the glass transition at 0.04kg.kg<sup>-1</sup> wet basis at 185°C, and there is no more explosive dehydration left to occur that could potentially change the structure. A study by Granda et al. (2004) showed that arcylamide content was 980ppb after 3 minutes frying at 180°C, this reduces to 450ppb after 90 seconds of frying. Hence frying for 90 seconds at 185°C to a moisture content of 0.04kg.kg<sup>-1</sup> should result in a reduction in acrylamide of approxiamtely 50%. There is a lack of data points presented by Granda et al. (2004) for acrylamide versus time at given frying temperatures, and the trendline can be debated. Measured data points after 90 seconds of frying at 185°C is required to confirm the exact benefits in acrylamide reduction.



Figure 2-19 Drying curve for continuous fried potato crisp, adapted from Bows et al. (2010)

Frying at 185°C for 90 seconds followed by a finish drying process limited to 105°C is attractive as acrylamide levels could potentially be reduced by 50% whilst retaining the desired structure of a continuously fried potato crisp.

Finish drying with air has advantages in that the temperature could be very carefully controlled so that the required temperature of 105°C is not exceeded. However air drying is very slow as the heat transfer depends on temperature difference. Evaporation is limited at moisture contents lower than 0.04kg.kg<sup>-1</sup> as most of the water is bound as indicated by the isosteric heat of potato shown in Figure 2-20. The potato crisp temperature can be over 100°C when drying from 0.04kg.kg<sup>-1</sup> down to less than 0.02kg.kg<sup>-1</sup> with air at 105°C. The low temperature difference results in very low heat transfer rates as convection, conduction, and radiation are dependent on temperature difference. Hence the high levels of isosteric energy shown in Figure 2-20 required to dry potato to less than 0.02kg.kg<sup>-1</sup> will take a long time to deliver with 105°C air. Evidence of long drying times required to finish dry with 105°C air to below 0.02kg.kg<sup>-1</sup> was presented by Kita et al. (2004). In this study it took 75 minutes to dry potato crisps from 0.22kg.kg<sup>-1</sup> down to less than 0.02kg.kg<sup>-1</sup> using 105°C air.



Figure 2-20 Total isosteric heat of desorption for potato – adapted from Wang & Brennan (1991)

Microwave finish drying can supply the total isosteric energy required to dry the potato crisps independent of any temperature difference. Microwave finish drying has been successfully used in an industrial scale with production rates as high as 900kg/h in the 1960s (O'Meara, 1968). At one point there was more than one hundred 915/896MHz operational microwave potato crisp finish drying systems. The microwave finish drying process was used to reduce browning that occurred due to the Maillard reaction when high sugar content potatoes were fried to produce potato crisps (O'Meara, 1973). However all these systems were shut down by the mid-1970s due to improvements in storage techniques, which practically eliminated the problem of high-sugar potatoes (Goldblith, 1966; Decareau, 1968). This was a very similar application to acrylamide reduction, where a reduced temperature is required. Moisture content was reduced from 0.08-0.06kg.kg<sup>-1</sup> down to less than 0.02kg.kg<sup>-1</sup> at a temperature of 100°C with a dwell time of just 2-3 minutes (Goldblith, 1966; Decareau, 1968). This shows that microwave finish drying can be used on an industrial scale to reduce acrylamide levels and is a far more attractive and practical solution than air finish drying as the dwell time is just 2-3 minutes. Production rates could potentially be doubled using microwave finish drying as the proposed dwell time in the fryer is halved.

Hence, this proposed RF/microwave finish drying application would dehydrate partially fried potato crisps from 0.04kg.kg<sup>-1</sup> wet basis to less than 0.02kg.kg<sup>-1</sup> wet basis. RF/microwave finish drying could become a necessity once again if the government were to restrict the quantity of acrylamide in food.

#### 2.7.2 Microwave processing of potato crisps

Schiffmann (2001a) reported that a new application had been developed for micrwave drying of potato crisps. This was used to produce nonfat and low-fat nonfried potato crisps. The sliced potatoes were conveyed through a hot air predryer, and then through a "high-intensity microwave field" that rapidly converted the potato moisture to steam, causing the slice to puff and the surface roughened so it resembled that of a traditional potato crisp. Then the surface was coated with flavoring and a small amount of oil and finally through a second microwave applicator where they were dried to the final desired moisture content and crispness. Schiffmann (2001a) noted that microwaves are an extremely expensive way to evaporate water by comparison to frying, high-velocity hot air, or infrared. While there was a demand for reduced fat potato

crisps, the processing cost had to be high, making this an expensive product. The market for low fat products then seriously declined, affecting the viability of this product.

There is now renewed interest in low fat potato crisps. In the UK, schools are not allowed to sell crisps due to health concerns (UK Goverment, 2015). A traffic light system has been introduced in the UK to warn customers of high fat and salt content. More than 60% of the food sold in the UK uses this traffic light system, inluding large snack manufacturers such as Mars UK and PepsiCo UK (UK Goverment, 2013). A new "sugar tax" is being introduced in the UK and there is speculation that a "fat tax" could follow (UK Goverment, 2016). The new legislation has had an impact on the potato crisp industry with potato crisp sales falling between 2015 and 2013 in the UK (Mintel, 2016). The current drive to reduce both child and adult objscity in the developed world has renewed R&D spend on microwaved potato crisps. Microwaved crisps give crisp manufacturers control of the fat content to produce targeted lower fat products. Microwave processing also enables companies to lower their carbon footprint, by replacing a gas fired process with an electrical process. PepsiCo have stated that they want to make their UK operations and manufacturing fossil free by 2023 (PepsiCo, 2015b). The only way they can realistically achieve this is by replacing their gas fired fryers with an electrical alternative.

A new microwave process has been invented by Bows et al. (2010). This microwave process replicates the time-temperature profile of continuous fried potato crisps so that potato crisps can be produced with controlled oil content. Crisps produced using this microwave process (with a similar fat content of continuous fried crisps) were preferred by 51% of participants in a forced preference taste. The survey was carried out with 200 people who were selected to be balanced across gender, age (<35, >35), socio-economic grouping, and eating frequency. This allows us to conclude that consumers equally preferred microwaved potato crisps and standard fried crisps. This microwave technique is operating on a small prototype line with the aim of scaling up to full production.
### 2.7.3 RF drying and moisture levelling of biscuits post baking

Radio Frequency (RF) finish drying of cookie, cracker, and biscuits has been successfully and extensively used in bakeries for over 40 years now (Koral, 2004; Schiffmann, 2001a). There is a wide variety of biscuits and their formulations, weight, diameter and other factors directly influence the baking process. In general, when cookies are baked using conventional oven heating methods, in ovens of 80m to 110m using universal conveyor belts, after approximately 3.5 to 4 minutes they exit the oven with 0.05-0.08kg.kg<sup>-1</sup> wet basis moisture content. They are then subsequently cooled to achieve a final content of 0.35-0.04kg.kg<sup>-1</sup> wet basis. If the moisture gradient between the border and the centre of the biscuit is too high (greater than 0.015kg.kg<sup>-1</sup>), it can present fissures and cracks (checking) during storage, due to expansion and contraction of the cookie (Chang, et al., 2011). Conventional bake-ovens utilise indirect heating methods. Heat is transferred to the material surface only, by virtue of conduction, convection or radiation. This means the heat energy has to pass from the surface by conduction to the interior of the product. Often this can present a problem, because the material itself is a good insulator and as such, it is difficult to get the heat to penetrate to the centre of the product. Therefore the surface dries out, whilst the core or centre of the piece remains high in moisture. RF post baking is a proven solution to this problem (Koral, 2004). RF and microwave heating is volumetric and will selectively heat regions of higher moisture content.

Bernussi et al. (1998) studied the effects of microwave heating biscuits after conventional baking with respect to moisture gradient and product quality. The study showed that biscuits prebaked in a conventional oven at 240°C for 4 minutes and subsequently heated with a microwave oven set at 2450MHz and magentron power output of 617.27W for 29 seconds showed significant reductions in moisture gradient, from 2.16% to 0.88% (central disk to outer rim), and the incidence of cracking decreased from 41.7% to 0%. So the association of conventional and microwave heating processes for baking cookies minimised biscuit cracking by reducing the moisture gradient and

preserved normal characterisitics of the product including colour, texture, flavour, and linear dimensions (Chang, et al., 2011).

One of the major reasons that RF dryers are used in the biscuit industry is to boost production. Finish drying using RF allows the manufacturer to increase the belt speed of their conventional dryer, thus increasing throughput. The RF dryer has a small footprint being only 2 or 3 meters long, with the baking oven being 20 or more meters long (Schiffmann, 2001a). This can allow the manufacturer to boost production at relatively low capital cost to meet market demand without having to increase the size of the factory. Manley & Clark (2011) stated that using post-oven dielectric drying can increase the output of a conventional oven by up to 33%, whilst Chavan & Chaven (2010) stated that baking speed was increased between 30-50%.

Schiffmann (2001a) noted that all the finish drying systems used in industry are RF systems, rather than microwaves, even though microwaves finish dry equally well. The reason is that the conveyors tend to be quite wide – 1 or more meters – and it is much easier to achieve uniform drying over this width with RF. Manley & Clark (2011) noted that post-oven RF dryers are now quite common.

#### 2.7.4 Microwave assisted drying of pasta

Pasta is made by hydrating semolina, mixing it, kneading it, extruding it through a die to obtain the desired shape, and then drying it (Abecassis, et al., 1994). In dry pasta products, drying is the most critical step. Pasta is extremely difficult to dry because it is so dense that moisture moves slowly to the surface. The product moisture content must be reduced from approximately 0.3kg.kg<sup>-1</sup> wet basis down to 0.12-0.13kg.kg<sup>-1</sup> wet basis and the drying temperature in the traditional process is kept low – approximately 35°C in order to attain a uniform rate of water removal and avoid case hardening of the surface (Schiffmann, 2001a; Marchylo & Dexter, 2001). Case hardening of the surface hinders natural moisture diffusion and sets up undesirable stresses within the product (Marchylo & Dexter, 2001). The greatest quality defects in pasta products are due to problems with this step (Chang, et al., 2011). If drying is

too fast, fissures or ruptures can occur; in which case the starch exuded during cooking will cause the pasta to stick together. As a result, the conventional lower temperature (LT) drying times are long, typically 8 to 12 hours. The slow drying time can result in accelerated microbiological and enzymatic deterioration of the product. The warm, moist conditions are an ideal breeding ground for microbial growth, especially Salmonella – a serious problem in the production of pasta containing egg as an ingredient (Chang, et al., 2011; Schiffmann, 2001a).

Hot air is, by itself is relatively efficient at removing free water at or near the surface, whereas the internal moisture in pasta is difficult to remove as it is driven by diffusion and not by capillary forces due to the lack of porosity (Hills, et al., 1997). Hence internal moisture transport is slow in pasta. Microwave energy solves this problem by providing a positive moisture flow towards the surface. The unique pumping action of microwave energy provides an efficient way of removing internal free water. Conventional air drying (especially high temperatures and longer drying times) can cause serious damage to flavour, colour and nutrients and can reduce bulk density and rehydration capacity of dried food product (Altan & Maskan, 2005).

One of the most successful industrial applications of microwaves in the food industry is the microwave assisted drying of pasta. Berteli & Marsaioli Jr. (2005) cited an experiment where the microwave process was compared to the conventional LT process which found advantages for both the product quality and the process. Pasta re-hydration or cooking time was reduced to half providing advantages for the product. Advantages for the process included shorter production time. This reduced the area occupied by 90%, in spite of doubling the final capacity. The higher temperatures allowed by the processes granted a sharper pasteurisation effect on the final product. The operational costs reduced by 26% in relation to conventional drying. A typical microwave process employed by industry for pasta drying was described by Schiffmann (2001a) to have three stages:

Stage 1: Hot air pre-drying at 82°C for 35 minutes, where the product is dried from 0.3kg.kg<sup>-1</sup> wet basis moisture content down to 0.18kg.kg<sup>-1</sup> wet basis.

Stage 2: Microwaves and hot air at 82°C for 12 minutes, where the product is dried from 0.18kg.kg<sup>-1</sup> wet basis moisture content down to 0.13-0.135kg.kg<sup>-1</sup> wet basis.

Stage 3: A final equalizing stage in which there is no heat or air movement, where the product typically exits at 0.12kg.kg<sup>-1</sup> moisture content.

Humidity control is important during the drying process. Relative humidity is kept low, at 15-20% in the first two drying stages; and high at 70-80% in the final equilibrium stage (Schiffmann, 2001a). The improvements in drying time of the stage 2 microwave process has been assessed by a number of authors. Berteli & Marsaioli Jr. (2005) observed a reduction in drying time from 6.5 hours to 20 minutes, Altan & Maskan (2005) stated that drying times were improved by 87.3%, and Schiffmann (2001a) observed a reduction in drying time from 8-12 hours to 1.5 hours. However, it should be noted that Berteli & Marsaioli Jr. (2005) measured pasta temperatures as high as 104°C during the microwave drying process, which is not equivalent to the conventional LT process. Schiffmann (2001a) stated that drying was carried out using large multimode resonant cavities operating at 915MHz.

Microwave technology has been used for pasta drying since at least the 1970s (Berteli & Marsaioli Jr., 2005), during that time the conventional air drying process has seen numerous innovations. The most imporatant advances in air drying is due to high temperature (HT) and ultra high temperature (UHT) drying. Before the 1970s drying was performed at relatively low temperatures (LT) up to a maximum of 60°C, and drying times for long goods were 18 hours or more. HT drying at temperatures of 60-85°C was introduced to the inidsutry in the 1970s and early 1980s. The initial drive behind the devleopment of HT drying was improved bacterial control for egg products. This resulted in drying cycles of approximately 8 hours for long goods and 5 hours for short goods. This permitted more compact drying lines, reduction in high capital costs, reduction in plant space, and improved cooking quality and colour of the pasta. HT quickly baecame the process of chioce for most pasta manufacturers

worldwide. In the late 1990s the application of ultra-high drying has become common using drying temperatures of 85-110°C. Drying times are 4-5 hours for long goods and 2-3 hours for short goods. UHT drying reporedly produces pasta products with cooking quality and colour equal to or better than that obtained with HT drying. However, with higher temperature drying, particularly UHT drying, there is the potential for the development of a brown or reddish colour due to excessive non-enzymatic browning i.e. the Maillard reaction or the "burning" of pasta. This can have a negative impact on protein nutritional quality (Marchylo & Dexter, 2001).

The advances in air drying technology is likely to have had an impact on the use of microwave drying of pasta. Schiffmann (2001a) stated that microwave drying of pasta was another example of a microwave application that was falling into disuse. He claimed that there were few, if any microwave pasta dryers still in operation. It should also be noted that this reference pre-dates Berteli & Marsaioli Jr. (2005) who stated that microwave assisted drying of pasta is one of the most successful industrial applications of microwaves in the food industry. The Berteli & Marsaioli Jr. (2005) paper discusses a pilot plant as well as the planned scale up. Shaheen et al. (2012) assertained that microwave drying of pasta is still being carried out in Italy.

### 2.8 Conclusion

An overview of the theory and current state of the art application of RF and microwave drying has been provided. Two new microwave drying applications have been identified for further study:

- Microwave explosive dehydration of potato crisps
- Microwave finish drying of potato crisps

With regards to the key drivers identified in Chapter 1, Microwave explosive dehydration of potato crisps addresses reduction in greenhouse gas emission, and the production of healthier food. This application enables the control of oil content, and gas fired fryers can be replaced with electrically driven microwave systems. Microwave finish drying of potato crisps addresses the production of healthier food. This finish drying process has the potential to significantly reduce carcinogenic content by finish drying efficiently at a lower temperature. Two established applications have also been identified for further study:

- Microwave pasta drying
- MW/RF finish drying of potato crisps

Microwave drying of pasta is consistently cited as having the most impressive reductions in drying time compared to conventional drying. This application is seen to reduce energy consumption and increase profit. It also addresses improvements in product quality by reducing the risk of microbial growth during drying. RF finish drying of potato crisps is by far the most successful RF/MW drying application in the food industry, with approximately 400 installations cited. This application addresses improvement in product quality, as cracking of the product is reduced by obtaining a more even distribution of moisture through volumetric heating. Although these applications are well established, they are interest to this study for two main reasons. It has been reported that microwave drying of pasta has fallen into disuse, and the reasons for this must be understood and discussed. Secondly, study of biscuit finish drying is important for this study to check and verify any new science presented, especially with respect to the understanding of moisture levelling.

As well as identifying four MW/RF applications for further study, the following knowledge gaps have been identified. These knowledge gaps are addressed in the subsequent chapters of the present study.

• There is a lack of sorption isotherms available for cooked foods. It is unclear whether chemical changes, such as the gelatinisation of starch, has an effect on the hygroscopy of foods. Isosteric heat is quantified using sorption isotherms and is required for commercial feasibility calculations due to its significance at low moisture contents.

- It is standard practice to define the state of the water in food using the points of inflection of the sorption isotherm. Like water activity, dielectric response is also known to be dependent on moisture mobility. However, there are no studies that establish the relationship between moisture dependant dielectric response and water activity.
- Although there are numerous models for frequency dependent dielectric behaviour, there are no recognised equations for moisture dependent dielectric behaviour. Hence modelling of MW processes is currently limited, leading to a trial and error approach in MW drier development.
- It is noted that it is very difficult to introduce a new drying process without adversely affecting some of the critical food parameters. One of the critical parameters for potato crisps is porosity. There is currently no satisfactory method for quantifying porosity of thin heterogeneous food structures containing oil.
- Moisture levelling is cited as a major advantage for MW/RF drying. Conversely, uneven heating is cited as a major disadvantage to MW/RF drying. There are no studies available that quantify the moisture levelling mechanism with respect to variation in exposure to electric field intensity.

# Chapter 3 – Analysis of dielectric and water activity behaviour in foods

### 3.1 Introduction

There is evidence to suggest that both water activity and dielectric response are dependent on water mobility. Sorption isotherm plots (water activity against moisture content) are used to define the transition from monolayer to multilayer, and the transition from multilayer to solution as shown in Figure 3-1. The first point of inflection defines the monolayer moisture content, which is a transition from monolayer moisture to multilayer moisture (Yanniotis & Blahovec, 2009). Water interacts directly with other chemical groups of molecules through dipole-dipole forces, dipole-ion interactions, van der Waals forces, and hydrogen bonding. Associated with this binding is the so-called monolayer moisture content, which theoretically assumes that each hydrophilic group has a water molecule associated with it (Labuza & Altunakar, 2007). Below the monolayer moisture content the moisture is tightly bound, with an enthalpy of vaporisation considerably higher than that of pure water (Yanniotis & Blahovec, 2009; Wang & Brennan, 1991). Foods that are dehydrated below the monolayer moisture content have a maximum shelf-life (Labuza & Altunakar, 2007). Examples of foods dried below the monolayer moisture content are potato crisps, crackers and biscuits (Schmidt, 2007). The next point of inflection is the transition from multilayer to solution. Once the moisture is in solution there is enough water mobility to support microbial growth and chemical reactions (Labuza & Altunakar, 2007). As described, the sorption isotherm defines the state and mobility of the water via its point of inflections. The target shelf life of a food product is defined by the final water activity of the dried product (Sahin & Sumnu, 2006), hence sorption isotherms can be used to define drying regimes.





Figure 3-1 Typical sorption isotherm of a carbohydrate food showing points of inflection (adapted from Reid (2007))

It is also known that dielectric loss mechanisms are dependent on water mobility. Studies show that the loss factor does not increase much with respect to moisture content until a "critical dielectric moisture content" is reached, after which a rapid increase in loss factor with respect to moisture content can be observed as shown in Figure 3-2 (Schiffmann, 2006; Metaxas & Meredith, 1993; Mudgett, et al., 1980; Tulasidas, et al., 19951 Guo, et al., 2008). The point of inflection at which the loss factor increases rapidly with respect to moisture content can be thought of as the point at which water is free to respond to electromagnetic waves. The critical dielectric moisture content has been observed in foods at both RF and microwave frequencies, suggesting that mobility is important for both ionic conduction, and dipolar rotation (Sacilik & Colak, 2010). A second point of inflection has been observed at high moisture contents in numerous foods (Mudgett, et al., 1980; Kim, et al., 1998; Feng, et al., 2002; Tulasidas, et al., 1995). This second point of inflection shows a decrease in the rate of change of loss factor with respect to moisture content as shown in Figure 3-2, and it is caused by reduced ionic conduction. This reduction in ionic conduction occurs due to dilution of the salts (Mudgett, et al., 1980). The second point of infection can be significant for microwave applications such as the explosive dehydration of potato crisps, which dries the food from its saturated state.







The relatively flat dielectric response below the critical dielectric moisture content shows that the dielectric response is being restricted by interaction between the water and the food. Both dipolar rotation and ionic conduction require water mobility, and both NMR and water activity studies show that water mobility is limited at lower moisture contents (Schmidt, 2007). Hence it can be concluded that water mobility is extremely important for dielectric behaviour and therefore for microwave drying.

It is important that the moisture dependence of the loss factor is not completely flat at low moisture contents, as moisture levelling requires a positive gradient of loss factor with respect to moisture content. The risk of burning increases as microwave heating ceases to be selective to wet food, and dry food can be heated at the same rate, or a higher rate than wet food in the applicator (Schiffmann, 2006). Burning is especially a risk without moisture levelling when it is considered that the electric field is likely to be uneven, which can result in excess heating of dry food compared to wet food. Uniformity of heating one of the major problems associated with microwave heating (Chandrasekaran, et al., 2013). It is a continuing concern in microwave heating and there are no clear insights gained regarding which guidelines, if any, in microwave-oven design yield superior uniformity in general (Edgar & Osepchuk, 2001). Lack of moisture levelling can lead to wet regions being left in the final product, which can result in reduced shelf life and cracking in the final product (Koral, 2004; Marchylo & Dexter, 2001). Cracking, or "checking" as it is called in the industry occurs due to internal stresses caused by moisture differential within the product (Koral, 2004).

As the critical dielectric moisture content is dependent on water mobility, and sorption isotherms are used to define water mobility, their relationship is of interest. Of particular interest, is whether the critical dielectric moisture content corresponds with the transition from monolayer to multilayer, or if it coincides with the transition from multilayer to solution. There are very few examples in literature where the points of inflection on sorption isotherms are compared to the critical dielectric moisture content. Dielectric property studies are typically completely separate from sorption isotherm studies, as they belong to different physics disciplines. The two main objectives of this chapter are as follows:

- Determine the relationship between the critical dielectric moisture content and the points of inflection of the sorption isotherms using published data.
- Determine whether water activity models can be adapted to describe dielectric behaviour with respect to moisture content. There is currently no standard model used to fit dielectric response with respect to moisture content, and a standard model would be of use for modelling applications.

## 3.2 Relationship between loss factor and sorption isotherm point of inflections

### 3.2.1 Method – Relationship between loss factor and sorption isotherm point of inflections

A literature review was carried out to identify foods for which the dielectric response with respect to moisture is published, as well as the corresponding sorption isotherms. Moisture dependant dielectric response and sorption isotherm data was obtained for nine foods: Potato (3GHz at 25°C, and 2.8GHz at 22°C), wheat flour (27MHz at 22°C), chickpea flour (27MHz at 20°C, 30°C and 40°C, 1.8GHz at 20°C, 30°C and 40°C), yellow dent field corn (20MHz at 24°C), carboxymethyl cellulose (2.45GHz at 20°C), hard red winter wheat (28MHz, 100MHz, 1GHz, and 12.1GHz at 24°C), apples (915MHz and 2.45GHz at 60°C) and grapes (2.45GHz at 25°C). The sorption isotherm measurement was taken at exactly the same temperature as the dielectric measurement, except for wheat flour, yellow dent corn, hard red winter wheat, and grapes. For yellow dent corn and hard red winter wheat the dielectric measurement was taken at 24°C and the sorption isotherm was measured at 25°C, while dielectric measurement was taken at 25°C and the sorption isotherm was measured at 30°C for grapes. The dielectric measurement for wheat flour was taken at 22°C while the sorption isotherm measurement was taken at 20°C.

The monolayer moisture content was calculated by fitting the BET sorption isotherm model to the experimental data using non-linear regression, where BET stands for Brunauer, Emmet, and Teller, who are the authors that derived the sorption equation (Brunauer, et al., 1938). This is a well-established method for defining the monolayer in the for the food industry (Al-Muhtaseb, et al., 2002). The equation is shown below were  $M_{db}$  is the moisture content dry basis,  $a_w$  is water activity,  $M_o$  is the monolayer moisture content which acts as a fitting parameter, and *C* is also a fitting parameter. Chapter 3 : Analysis of dielectric and water activity behaviour in foods

$$M_{db} = \frac{M_0 C a_w}{(1 - a_w)[1 + (C - 1)a_w]}$$
Equation 3-1

The non-linear regression fitting of the BET model was carried out over a water activity range of 0-0.45, as the BET model is only effective at representing sorption isotherms at lower water activity ranges (McMinn, et al., 2007). Non-linear regression fitting of the BET equation was carried out using Minitab 16 software (Minitab Inc., Pennsylvania State University, USA), which is a statistical analysis software package. The best fit of the non-linear regression was ascertained using residual sum of squares, which is described as follows:

$$RSS = \sum (u_e - u_p)^2$$
Equation 3-2

Where  $u_e$  is the experimental value, and  $u_p$  is the value predicted by the model. There is no recognised mathematical method for determining the transition from multilayer to solution. This transition was determined by inspecting the sorption isotherm curve for an increase in the rate of change of water activity with respect to moisture content.

For each graphical comparison of the points of inflection, moisture content wet basis was plotted agianst water acitvity, and loss factor was added to a second X axis. Water content was plotted as wet basis, as this is convention for the reporting of dielectric properties. The moisture content of the points of inflection of loss factor and water activity were recorded in a comparison table for all the instances analysed.

### 3.2.2 Results and discussion – Relationship between loss factor and sorption isotherm point of inflections

The desorption isotherm and corresponding loss factor for freeze dried potato is shown in Figure 3-3.



Figure 3-3 Desorption isotherm and corresponding loss factor of freeze dried potato at 3GHz taken from Mudgett et al. (1980)

The data presented in Figure 3-3 was taken from Mudgett et al. (1980) were both measurement sets were taken on a desorption basis. The BET monolayer moisture content was calculated to be 0.076kg.kg<sup>-1</sup> wet basis. The loss factor was measured at a frequency of 3GHz at 25°C, which is the same temperature at which the sorption isotherm was measured. It can be seen that the loss factor does not change either before the monolayer, or for a period afterwards. The first point of inflection occurs at approximately 0.12kg.kg<sup>-1</sup> wet basis, which is only after a significant amount of multilayer moisture is present. There is a jump in loss factor from 0.1 to 0.9 at 0.12kg.kg<sup>-1</sup> wet basis, after which the loss factor is constant until a moisture content 0.16kg.kg<sup>-1</sup> wet basis. The loss factor starts increasing significantly at a point of inflection of approximately 0.16kg.kg<sup>-1</sup> wet basis, which is just before the water starts behaving like a solution at 0.2kg.kg<sup>-1</sup> wet basis. When the moisture starts acting like a solution it has enough mobility to support chemical reactions and the growth of microorganisms (Labuza & Altunakar, 2007). For the case of potatoes, Figure 3-3 provides evidence that the mobility required to support micro-organism growth is similar to the mobility required for moisture dependant dielectric loss mechanisms. Microwave moisture levelling is dependent on having a positive gradient of loss factor with respect to moisture content. Hence, microwave moisture levelling may be limited in the monolayer region as the response appears to be flat. However, there is a lack of resolution on the low moisture content loss factor measurements, there may in fact be a positive gradient of loss factor with respect to moisture content. The low loss factor at moisture contents below the multilayer-solution transition provides complications with respect to microwave heating. Low loss materials are more difficult to heat using microwave radiation (Bradshaw, et al., 1998).

After the critical dielectric moisture content, the next significant point of inflection for the loss factor occurs at approximately  $0.31 \text{kg.kg}^{-1}$  wet basis. This point of inflection constitutes a decrease in  $d\varepsilon'/dM_{wb}$ , where  $\varepsilon''$  is loss factor and  $M_{wb}$  is moisture content wet basis. This decrease in  $d\varepsilon''/dM_{wb}$  is caused by a dilution of the salts which reduces the ionic conduction component of the loss factor. The dilution continues to negatively affect  $d\varepsilon''/dM_{wb}$  until the loss factor decreases. The zero point of  $d\varepsilon''/dM_{wb}$  is approximately 60% wet basis, after which the loss factor decreases until saturation is reached.

Holtz et al. (2010) carried out a similar measurement to Mudgett et al. (1980) of potato at 2.8GHz as shown in Figure 3-4. The BET monolayer was calculated to be 0.050kg.kg<sup>-1</sup> wet basis using the data presented by Holtz et al. (2010).





Figure 3-4 Desorption isotherm and corresponding loss factor of potato at 2.8GHz taken from Holtz et al. (2010)

It can be seen that, similar to the Mudgett et al. (1980) data, there is no change in dielectric response at the monolayer. There is not enough data points to detect the jump in loss factor from 0.1 to 0.9 which occurred at 0.12kg.kg<sup>-1</sup> in the Mudgett et al. (1980) study. Also similar to the Mudgett et al. (1980) data, the point of inflection due to solution at 0.21kg.kg<sup>-1</sup> wet basis closely coincides with a large increase in  $d\varepsilon''/dM_{wb}$ . The point of inflection due to dilution of the salts is difficult to decipher in the Holtz et al. (2010) data due to the spread of data points. However, with the trendline used, it would appear that dilution of the salts begins to lower  $d\varepsilon''/dM_{wb}$  at a moisture content of approximately 0.5kg.kg<sup>-1</sup> wet basis, which is higher than the Mudgett et al. (1980) value of 0.3kg.kg<sup>-1</sup>. However, the zero point of  $d\varepsilon''/dM_{wb}$  is approximately 0.6kg.kg<sup>-1</sup> wet basis, which agrees with the Mudgett et al. (1980) data.

The moisture dependance of loss factor at 27MHz and water activity for wheat flour is shown in Figure 3-5. It loss factor profile looks very similar to that observed in potato by Mudgett et al. (1980) and Holtz et al. (2010). There is an initial change in  $d\varepsilon''/dM_{wb}$ , which occurs just after the monolayer of 0.065kg.kg<sup>-1</sup> wet basis, then there is a very significant rise in  $d\varepsilon''/dM_{wb}$  at

0.26kg.kg<sup>-1</sup> wet basis, followed by a decrease caused by dilution of the salts, with the rate of change turning negative at approximately 0.65kg.kg<sup>-1</sup> wet basis.



Figure 3-5 GAB fit Desorption isotherm at 20°C taken from Moreira et al. (2010) and loss factor at 22°C, 27MHz taken from Kim et al. (1998) for wheat flour-water mixtures

The large increase in  $d\varepsilon'/dM_{wb}$  at 0.26kg.kg<sup>-1</sup> wet basis is higher than the 0.19kg.kg<sup>-1</sup> wet basis point of inflection of water activity where the water starts to behave as a solution. This lag of loss factor response did not occur in the potato measurements presented by Mudgett et al. (1980) and Holtz et al. (2010). This may be due to the diference in frequency, where the Mudgett et al. (1980) and Holtz et al. (2010) potato studies were carried out at 3GHz and 2.8GHz respectively, whereas the wheat flour study carried out at 27MHz by Kim et al. (1998). There is a possibility that ionic conduction requres more water mobility than dipolar rotation. However chickpea flour was measured by Guo et al. (2008) at both RF and microwave frequencies, and in each instance the critical dielcetric moisture content occurred at the ponit at which the water started to behave as a solution. Therefore, the lag in dielectric response for wheat flour may be due to other factors such as experimental error, or differences in food behaviour. The dielectric study was carried out by Kim et

al. (1998) and the sorption study was carried out by Moreira et al. (2010), and they may have used a different type of wheat flour. This could result in a poorer coellation in results compared to data presented by Mudgett et al. (1980) and Holtz et al. (2010), each of which carried out both sorption and dielectric studies themselves, ensuring the food type was common between dielectric and sorption studies.

The loss factor and water activity of chickpea flour is presented in Figure 3-6, Figure 3-7, and Figure 3-8 at temperatures of 20°C, 30°C, and 40°C respectively. Each figure contains plots of the loss factor at 27MHz and 1.8GHz compared to the sorption isotherm. A similar plot is shown for the desorption isotherm and loss factor of yellow dent corn in Figure 3-9, where the loss factor was measured at 24°C, 20MHz, and the desorption isotherm was measured at 25°C. Although none of these plots contain loss factor measurements at low moisture contents that encompasss the monolayer region, it is clear from the loss factor profile that there is only one large point of inflection. In each instance this large point of inflection in the dielectric response corresponds very closely with the second point of inflection of the sorption isotherm, which is defined as the transition from multilayer to solution. Up to this point, the loss factor does not change significantly and is very low.



Figure 3-6 Desorption isotherm at 20°C taken from Durakova & Menkov (2005) and loss factor at 20°C taken from Guo et al. (2008) of Chickpea flour at frequencies of (a) 27MHz and (b) 1.8GHz



Figure 3-7 Desorption isotherm at 30°C taken from Durakova & Menkov (2005) and loss factor at 30°C taken from Guo et al. (2008) of Chickpea flour at frequencies of (a) 27MHz and (b) 1.8GHz



Figure 3-8 Desorption isotherm at 40°C taken from Durakova & Menkov (2005) and loss factor at 40°C taken from Guo et al. (2008) of Chickpea flour at frequencies of (a) 27MHz and (b) 1.8GHz





Figure 3-9 Chung and Pfost fit Desorption isotherm at 25°C taken from Samapundo et al. (2007) and loss factor at 24°C, 20MHz taken from Nelson (1991c) for yellow dent corn

RF/moisture levelling requires a positive gradient of loss factor with respect to moisture content, and Figure 3-6 to Figure 3-9 shows that similar to potato and wheat flour, the loss factor may not vary with respect to moisture content below the monolayer moisture content. More data points with suitable resolution are required to determine whether or not the loss factor has a positive gradient with respect to moisture content at the monolayer transition. Although the trends are similar for 27MHz compared to 1.8GHz for chickpea flour, the loss factor is higher at 27MHz. The higher loss factor observed at 27MHz is indicative of the presence of ionic conduction as the contribution of ionic conduction to the loss factor is higher at lower frequencies (Tang, et al., 2002). Conversly the contribution of dipolar rotation to the dielectric loss is less at 27MHz compared compared to 1.8GHz, as 27MHz is further away from the 17GHz relaxation frequency of water (Nelson & Datta, 2001). Despite ionic conduction being the dominant loss mechanism at 27MHz, and dipolar rotation being the dominant loss mechanism at 1.8GHz, the point of inflection is the same. This potentially suggests that a very similar amount of water mobility is required for dipolar rotation and ionic conduction to occur in chickpea flour, and that both loss mechanisms occur when the moisture starts to behave as a solution. It is interesting to note that the loss factor point of inflection at 0.11kg.kg<sup>-1</sup> wet basis stays relatively constant when comparing the plots at 20°C, 30°C, and 40°C for chickpea flour. However, both the transition from monolayer to multilayer and the transition from multilayer to solution decrease in terms of moisture content for a given water activity as temperature rises (Lagoudaki, et al., 1993). The transition from monolayer to multilayer is better defined, in that it is typically caulcatued using the BET model (Labuza & Altunakar, 2007). However there is no model defined for calcualting the moisture content at the transition from multilayer to solution, which makes its identification more subjective. It is therefore difficult to make exact statements as to how much the transition from multilayer to solution is changing with respect to temperature compared to the critical dielectric moisture content. The monolayer moisture content is 0.062kg.kg<sup>-1</sup> wet basis at 20°C, 0.053kg.kg<sup>-1</sup> wet basis at 30°C, and 0.050kg.kg<sup>-1</sup> wet basis at 40°C as calcualted using the BET model.

The loss factors plotted from Figure 3-6 to Figure 3-9 show no decrease in the rate of change of loss factor at higher moisture contents. However, this is not to say that dilution of ions does not occur, the point of inlfection associated with dilution of ions may occur at moisture contents that are higher than the moisture content range presented.

The sorption isotherm and corresponding loss factor for carboxymethyl cellulose is shown in Figure 3-10. This plot includes loss factor measurements below the monolayer moisture content. Similar to the previous plots, there is a point of inflection in the loss factor that directly corresponds to the point at which the moisture starts behaving as a solution. Hence, after the multilayer there is a large increase in loss factor. No reduction of  $d\varepsilon''/dM_{wb}$  due to ionic dilution is observed, although this may occur at higher moisture contents which is beyond the range measured by Nelson et al. (1991d).



Chapter 3 : Analysis of dielectric and water activity behaviour in foods

Figure 3-10 GAB fit adsorption isotherm at 20°C taken from Torres et al. (2012) and loss factor at 20°C, 2.45GHz taken from Nelson et al. (1991d) for carboxymethyl cellulose

The desorption isotherms and loss factor for hard red winter wheat is shown in Figure 3-11 at 28MHz, 100MHz, 1GHz, and 21.1GHz. The plot at 28MHz is similar to the plots previously discussed where the main point of inflection of the loss factor roughly aligns with the transition from mulitlayer to solution, albeit the loss factor point of inflection occurs at a lower moiture content. The loss factor trend is relatively straight at 100MHz, while the loss factor profile conforms to the sorption isotherm shape at 1GHz, and to a lesser extent at 12.1GHz. This implies that at 1GHz and 12.1GHz the loss factor increases rapidly during the multilayer region, and then slows during the solution region. This is unusual behaviour as any reduction in the rate of change in loss factor at high moisture contents is usually attributed to dilution of salts (Mudgett, et al., 1980). Ionic conduction tends to be dominant at lower frequencies, yet no reduction in the rate of change in loss factor is observed at high moisture contents at 28MHz and 100MHz. A reduction in loss factor is observed at high moisture contents at 1GHz and 12.1GHz even though dipolar rotation should be dominant (Tang, et al., 2002). The trend of loss factor with respect to moisture content at 1GHz and 12.1GHz is in contrast to all the other trends discussed thus far, such as potato, chickpea, and yellow dent corn, where in each instance the critical dielectric moisture content coincided with the multilayer-solution transition moisture content. For hard red winter wheat at 1GHz and 12.1GHz, the critical dielectric moisture conent coinsides with the monolayer rmoisture content. It should be noted that the trendline at 100MHz may be misleading, more datapoints may indicate a straight line rather than a sigmoid curve.



Figure 3-11 Desorption isotherm at 25°C taken from Sun & Woods (1994) and loss factor at 24°C taken from Nelson & Stetson (1976) of hard red winter wheat at frequencies of (a) 28MHz (b) 100MHz (c) 1GHz, and (d) 12.1GHz

It can be seen from Figure 3-12 that the relationship between water activity and loss factor is different for apples than for potato, wheat flour, chickpea flour, yellow dent field corn, and carboxymethyl cellulose. It has been noted by Sipahioglu & Barringer (2003) that vegetables had different dielectric behaviour than fruits.



Figure 3-12 Desorption isotherm at 60°C taken from Kaymak-Ertekin & Gedik (2004) and loss factor at 60°C taken from Martin-Esparza et al. (2006) of apples at frequencies of (a) 915MHz and (b) 2.45GHz

For apples, both the trend lines and the points of inflection are very closely aligned for loss factor and water activity. The rate of change of loss factor with respect to moisture content increases during transition from monolayer to multilayer, and decreases during transition from multilayer to solution. The decrease in  $d\varepsilon'/dM_{wb}$  during the transition from multilayer to solution could be attributed to dilution of the salts, but it is more pronounced at 2.45GHz than at 915MHz despite the fact that ionic conduction would be more dominant at 915MHz. If the decrease in  $d\varepsilon'/dM_{wb}$  is due to dilution of salts, it would imply that salt dilution becomes significant in apples as soon as the water acts as a solution. The large increase in loss factor, which defines the critical dielectric moisture content, coincides with the monolayer moisture content. This is in contrast to potatoes and other vegetables assessed in the present study where a large increase in loss factor coincided with the transition from multilayer to solution. This contrast in dielectric behaviour would suggest that the multilayer to solution.

in apples which contain sugar, than the multilayer in potatoes and other low sugar foods with high hydrocolloid content.

The desorption isotherm and loss factor for grapes at 30°C is shown in Figure 3-13 at 25°C, 2.45GHz. The loss factor follows the trend of the sorption isotherm in the same way as was observed for apples in Figure 3-12. This would suggest that fruits do behave differently from most low sugar foods that have high hydrocolloids content, where a large increase in loss factor occurs during the multilayer, and dilution of the salts occur when the moisture is in solution. In fruits such as grapes and apples the water predominantly interacts with sugar and pectin, whereas in vegetables such as potato, water predominantly interacts with starch carbohydrates and cellulose (Kaymak-Ertekin & Gedik, 2004; Maroulis, et al., 1988; McLaughlin & Magee, 1998). The representative sugar (glucose + fructose + sucrose) contents of grapes and apples are 0.44-0.45kg.kg<sup>-1</sup> wet basis and 0.43kg.kg<sup>-1</sup> wet basis respectively. Apples also have a pectin content of 0.065kg.kg<sup>-1</sup> wet basis. Potatoes are characterised by high starch content and contain 0.02kg.kg<sup>-1</sup> wet basis of sugars, and 0.44-0.46kg.kg<sup>-1</sup> wet basis starch (Kaymak-Ertekin & Gedik, 2004). The dielectric behaviour of grapes and apples would suggest that there is enough mobility to support dielectric loss mechanisms in the multilayer region, whereas low sugar foods with high hydrocolloids content can only support dielectric loss mechanisms when the water acts as a solution. The loss factor is generally higher in fruit in the multilayer region compared to low sugar food with high hydrocolloid content, making it easier to couple microwave energy into the food for dehydrating down to the monolayer moisture content. There is a risk that the loss factor response is flat for part of the multilayer region in low sugar, hydrocolloid rich foods, with moisture not being selectively heated as a result. However, better resolution is required for dielectric measurements of low sugar foods with high hydrocolloid content in the low moisture content multilayer region to determine if the loss factor response is truly flat. Microwave heating below the monolayer content appears challenging for both fruits and vegetables as the loss factor is extremely low. Microwave finish drying of potato crisps at 915MHz is an example of a low loss material that proved difficult to process due to arcing/throughput. Schiffmann, (2001a) reported that fires were not uncommon when finish drying potato crisps at at 896/915MHz due to the low dielectric loss of nearly dry potato crisps. This finish drying process dried potato crisps from 0.06-0.08kg.kg<sup>-1</sup> wet basis down to less than 0.02kg.kg<sup>-1</sup> wet basis, and the drying equipment required fire detection and control systems.



Figure 3-13 Desorption isotherm at 30°C taken from Kaymak-Ertekin & Gedik (2004) and loss factor at 25°C taken from Tulasidas et al. (1995) of grapes at a frequency of 2.45GHz

A comparison between the monolayer moisture content observed in the sorption isotherm, and a corresponding rise in loss factor denoted by the critical dielectric moisture content is shown in Table 3-1. It can be seen that all the low sugar foods analysed that had high hydrocolloid content had no rise in loss factor corresponding to the monolayer moisture content, with the exception of hard red winter wheat at 1GHz and 12.1GHz. The fruit analysed, apples and grapes, did have a large rise in loss factor corresponding to the monolayer moisture content. Conversely, Table 3-2 shows the correlation between the critical dielectric moisture content and the transition from multilayer to solution. This shows that low sugar foods with high hydrocolloid content experience a large increase in loss factor at the transition from multilayer to solution whereas fruits do not. Again hard red winter wheat at 1GHz and 12.1GHz is the exception of this trend where no rise in loss factor was observed during the transition from multilayer to solution. In general however, there was a distinct difference in behaviour observed in sugary fruits compared low sugar foods with high hydrocolloid content.

Food	Temperature	Frequency	Dielectric reference	Water activity reference	Monolayer moisture content ( <i>wb</i> ) from sorption isotherm [kg.kg <sup>-1</sup> ]	Critical dielectric moisture content ( <i>wb</i> ) corresponding to monolayer moisture content [kg.kg <sup>-1</sup> ]
Freeze dried potato	25degC	3GHz	Mudgett, et al., 1980	Mudgett, et al., 1980	0.076	No rise in loss factor
Potato	22degC	2.8GHz	Holtz, et al., 2010	Holtz, et al., 2010	0.050	No rise in loss factor
Wheat flour	22degC (dielectric) 20degC (sorption)	27MHz	Kim, et al., 1998	Moreira, et al., 2010	0.068	No rise in loss factor
Chickpea flour	20degC 30degC 40degC	27MHz, 1.8GHz	Guo, et al., 2008	Durakova & Menkov, 2005	0.062 0.053 0.050	No measurements
Yellow dent field corn	24degC (dielectric) 25degC (sorption)	20MHz	Nelson, 1991c	Samapundo, et al., 2007	0.068	No measurements
Hydrocolloids	20degC	2.45GHz	Nelson, et al., 1991d	Torres, et al., 2012	0.079	No rise in loss factor
		28MHz				No rise in loss factor
Hard red	24 (dielectric) 25 (sorption)	100MHz	Nelson & Stetson, 1976	Sun & Woods, 1994	0.068	No rise in loss factor
winter wheat		1GHz				0.07
		12.1GHz				0.10
Apples	60degC	915MHz, 2.45GHz	Feng, et al., 2002	Kaymak-Ertekin & Gedik, 2004	0.117	0.127
Grapes	25degC (dielectric) 30degC (sorption)	2.45GHz	Tulasidas, et al., 1995	Kaymak-Ertekin & Gedik, 2004	0.13	<0.15

 Table 3-1 Critical dielectric moisture content corresponding with the monolayer moisture content observed in the sorption isotherm

Food	Temperature	Frequency	Moisture content ( <i>wb</i> ) of transition from multilayer to solution [kg.kg <sup>-1</sup> ]	Critical dielectric moisture content ( <i>wb</i> ) corresponding to the transition from multilayer to solution [kg.kg <sup>-1</sup> ]
Freeze dried potato	25°C	3GHz	0.20	0.16
Potato	22°C	2.8GHz	0.21	0.21
Wheat flour	22°C (dielectric) 20°C (sorption)	27MHz	0.19	0.26
Chickpea flour	20°C 30°C	27MHz,	0.11 0.095	0.11
	40°C	1.8GHz	0.09	
Yellow dent field corn	24°C (dielectric) 25°C (sorption)	20MHz	0.17	0.15
Hydrocolloids	20°C	2.45GHz	0.16	0.16
		28MHz		0.16
Hard red	24°C (dielectric)	100MHz	0.19	0.21
winter wheat	25°C (sorption)	1GHz	0.19	No rise in loss factor
		12.1GHz		No rise in loss factor
Apples	60°C	915MHz, 2.45GHz	0.30	No rise in loss factor
Grapes	25°C (dielectric) 30°C (sorption)	2.45GHz	0.30	No rise in loss factor

 Table 3-2 Critical dielectric moisture content corresponding with the transition from multilayer to solution observed in the sorption isotherm. Source of data is shown in Table 3-1.

Table 3-3 compares the point of inflection of loss factor that corresponds to dilution of salts with the transition from multilayer to solution. It can be seen for all low sugar foods with high hydrocolloid content that dilution of salts only affect the loss factor at moisture contents that are at least 50% higher than the transition from multilayer to solution, if any reduction in loss factor due to dilution of salts is observed at all. For grapes and fruits, and for hard red winter wheat at 1GHz and 12.1GHz, a decrease in  $d\varepsilon'/dM_{wb}$  is observed at the exact transition from multilayer to solution. This shows that sugary fruits tend to have a different dielectric behaviour from low sugar foods with high hydrocolloid content at high moisture contents.

Food	Temperature	Frequency	Moisture content ( <i>wb</i> ) of transition from multilayer to solution [kg.kg <sup>-1</sup> ]	Moisture content ( <i>wb</i> ) at point of inflection of loss factor typically attributed to dilution of salts [kg.kg <sup>-1</sup> ]	
Freeze dried potato	25°C	3GHz	0.20	0.30	
Potato	22°C	2.8GHz	0.21	0.50	
Wheat flour	22°C (dielectric) 20°C (sorption)	27MHz	0.19	0.50	
Chickpea flour	20°C	27141	0.11		
	30°C 27MHz 1.8GHz		0.95	No measurements	
	40°C	1.00112	0.9		
Yellow dent field corn	24°C (dielectric) 25°C (sorption)	20MHz	0.17	No measurements	
Hydrocolloids	20°C	2.45GHz	0.16	No measurements	
		28MHz		None observed	
Hard red winter wheat	$24^{\circ}$ C (dialoctric) $25^{\circ}$ C (countiers)	100MHz	0.10	None observed	
	24°C (dielectric) 25°C (sorption)	1GHz	0.19	0.15	
		12.1GHz		0.17	
Apples	60°C	915MHz, 2.45GHz	0.30	0.30	
Grapes	25°C (dielectric) 30°C (sorption)	2.45GHz	0.30	0.40	

Table 3-3 Point of inflection of loss factor typically attributed to a dilution of salt compared to the transition from multilayer to solution observed in the sorption isotherm. Source of data is shown in Table 3-1.

### 3.3 Proposed dielectric loss factor equation

There is no standard equation currently being used to represent dielectric properties of food with respect to moisture content. Previous studies have attempted to build predictive models, however these models had limitations due to the complexity of water and salt interaction with food constituents (To, et al., 1974; Mudgett, et al., 1977; Mudgett, et al., 1980; Prakash, et al., 1992; Sun, et al., 1995; Sipahioglu & Barringer, 2003). There has been a recent increase in multiphysics modelling using Finite Element Analysis with fully coupled RF and heat transfer physics (Ni, et al., 1999; Datta, 2007b; Dinčov, et al., 2004; Aversa, et al., 2007, Renshaw, 2009; Chen, et al., 2006). The analysis is typically carried out at a single frequency, for which the applicator is specifically designed. It would be advantageous to have a general equation to which experimental moisture dependant dielectric response data could be fitted for use in such analyses.

The previous sub-section shows that the relationship between moisture content and loss factor has the same sigmoid shape as the relationship between moisture content and water activity, with the exception occurring when salt dilution causes  $d\tilde{\varepsilon}'/dM_{wb}$  to become negative. The phenomena of salts causing a fall in loss factor was observed with wheat four at 27MHz by Kim et al. (1998) and with potato at 3GHz by Mudgett et al. (1980). Hence a reduction in loss factor due to dilution of salts can occur at high ISM band frequencies as well as low ISM band frequencies. However, these phenomena only occur at very high moisture contents for some foods. There is a relationship between the points of inflection of water activity and loss factor with respect to moisture content. In vegetables, the monolayer-multilayer transition generally has a negligible effect on  $d\varepsilon'/dM_{wb}$ , the multilayer-solution transition causes a large increase in  $d\varepsilon'/dM_{wb}$ , and dilution of salts causes a decrease in  $d\varepsilon'/dM_{wb}$ , resulting in a sigmoid shape of loss factor with respect to moisture content. In fruits, the monolayer-multilayer transition causes a large increase in  $d\vec{\varepsilon'}/dM_{wh}$ , and the multilayer-solution transition causes a decrease in  $d\vec{\varepsilon'}/dM_{wb}$ , again resulting in a sigmoid shape of loss factor with respect to moisture content.

Hence, regardless of individual behaviour, the shape is sigmoid except at very high moisture contents where  $d\varepsilon'/dM_{wb}$  can become negative.

This enables us to adapt water activity equations to describe loss factor behaviour. As previously discussed in Chapter 2, the GAB equation is used to fit sorption isotherms for a wide variety of foods and can provide good fits for the entire water activity range (Al-Muhtaseb, et al., 2002). The GAB equation is presented below, where  $M_0$  is the monolayer, and C and K are constants. Hence it is a three parameter fitting water activity equation.

$$M_{db} = \frac{M_0 C K a_w}{(1 - K a_w) [1 - K a_w + C K a_w]}$$
Equation 3-3

The GAB equation can be re-arranged in quadratic form as shown below:

$$a_w^2 (M_{db} K^2 - M_{db} K^2 C) + a_w (M_{db} K C - 2M_{db} K - M_0 K C) + M_{db} = 0$$
  
Equation 3-4

Water activity can then be made the subject using the general quadratic equation shown below:

$$a_w = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$
 Equation 3-5

Substituting the re-arranged GAB equation parameters into the quadratic Equation 3-5 yields the following relationship:

$$a_{w} = \frac{1}{2} \left( \frac{-2M_{db} + M_{db}C - M_{0}C + \sqrt{4M_{db}CM_{0} + C^{2}M_{db}^{2} - 2M_{db}M_{0}C^{2} + M_{0}^{2}C^{2}}}{M_{db}K(C-1)} \right)$$
Equation 3-6

As the trend is the same, the water activity term can simply be replaced with the loss factor. However, unlike water activity, the loss factor does not equal zero at zero moisture content. Therefore another term,  $\varepsilon_{om}$  is added, which is the loss factor at zero moisture content. Also, the loss factor shape is more of a

typical sigmoid shape when expressed in terms of moisture content wet basis, rather than dry basis. The modified expression is shown in Equation 3-7.

$$\varepsilon'' = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon''_{0m}$$
Equation 3-7

However, this equation does not express the fall in loss factor which can be caused by dilution of the salts at high moisture contents i.e. when  $d\varepsilon'/dM_{wb}$ , becomes negative. To express this phenomenon, an extra term has been added as shown in Equation 3-8 where U and S are additional fitting parameters. This additional term can generate a sharp decrease in loss factor that only affects the expression during the relevant moisture content range. In instances where  $d\varepsilon''/dM_{wb}$  does not turn negative, U returns to zero.

$$\varepsilon'' = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon''_{0m} - UM_{wb}^S$$

#### Equation 3-8

Other water activity equations can be adapted for loss factor using a similar approach. Some water activity equations are only suitable for returning values up to 1, as the maximum value for water activity is 1. In these instances, the adapted equation has to be factored up for loss factor using an additional parameter. The adapted water activity equations are presented in Table 3-4. These equations are intended to be used to curve fit experimental moisture dependant dielectric responses using non-linear regression at a single frequency. Therefore, the physics of the dielectric response is not important (i.e. whether dipolar rotation or ionic conduction is dominant), rather the adapted water activity equation's ability to curve fit the dielectric response is what is important.

Name	Water activity equation	Adapted loss factor equation		
GAB	$a_{w} = \frac{1}{2} \left( \frac{-2M_{db} + M_{db}C - M_{0}C + \sqrt{4M_{db}CM_{0} + C^{2}M_{db}^{2} - 2M_{db}M_{0}C^{2} + M_{0}^{2}C^{2}}}{M_{db}K(C-1)} \right)$	$\varepsilon^{\prime\prime} = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2 M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon_{0m}^{\prime\prime} - UM_{wb}^S$		
Oswin	$a_w = (M_{db}/A)^{1/B} \left(1 + (M_{db}/A)^{1/B}\right)^{-1}$	$\varepsilon'' = C(M_{wb}/A)^{1/B} \left(1 + (M_{wb}/A)^{1/B}\right)^{-1} + \varepsilon''_{0m} - UM^{S}_{wb}$		
Henderson	$a_w = 1 - e^{-ATM_{db}^B}$	$\varepsilon^{\prime\prime} = C\left(1 - e^{-ATM_{wb}^B}\right) + \varepsilon_{0m}^{\prime\prime} - UM_{wb}^S$		
Chung- Pfost	$a_w = e^{-e^{\frac{M_{db}-a}{b}}}$	$\varepsilon^{\prime\prime} = C\left(e^{-e^{\frac{M_{wb}-a}{b}}}\right) + \varepsilon^{\prime\prime}_{0m} - UM^{S}_{wb}$		
Chen	$a_w = e^{A - Be^{-CM}db}$	$\varepsilon^{\prime\prime} = D\left(e^{A-Be^{-CM_{wb}}}\right) + \varepsilon_{0m}^{\prime\prime} - UM_{wb}^{S}$		
Ferro- Fontan	$a_w = \frac{\gamma}{e^{AM_{db}r}}$	$\varepsilon^{\prime\prime} = \frac{\gamma}{e^{AM_{dw}r}} + \varepsilon_{0m}^{\prime\prime} - UM_{dw}^S$		

 Table 3-4 Sorption isotherm equations adapted for loss factor

### 3.4 Fitting of proposed dielectric loss factor equations

The equations presented in Table 3-4 were fitted to measured data presented in literature of loss factor varying with respect to moisture content. The best fit was calculated using non-linear regression where the fitting criteria used was the residual sum of squares, which is described as follows:

$$RSS = \sum (u_e - u_p)^2$$
 Equation 3-9

Where  $u_e$  is the experimental value, and  $u_p$  is the value predicted by the model. The non-linear regression was carried out using Minitab 16 software (Minitab Inc., Pennsylvania State University, USA), which is a statistical analysis software package. Although the Residual Sum of Squares is an effective way at determining the best equation fit for a given set of data, it cannot be used to compare the fit of different data sets as the RSS figure is dependent on the number of data points. The Mean Relative Error is useful for comparing equation fits of different data sets and is presented in Equation 3-10.

$$MRE = \frac{100}{N} \sum \left| \frac{u_e - u_p}{u_e} \right|$$
Equation 3-10

It is generally understood that a good fit is obtained when MRE<10% (McMinn, et al., 2007; McLaughlin & Magee, 1998). However, care must be taken when using MRE as it can be highly affected by the fit of low value points. For instance, at low moisture content, the loss factor is typically extremely low and experimental error is high. A lack of fit with these very low loss factor points at low moisture content can lead to a high MRE.

The parameters used to obtain the best RRS fits are presented in Appendix A. The best fit equation using RRS is presented in Table 3-5 for each food analysed. Table 3-5 also presents the average MRE, and lowest MRE fits.
Food	Best fit adapted model using RSS	Best fit adapted model using MRE	Average MRE	Lowest MRE
Freeze dried potato	GAB	Oswin	9.78	7.65
Potato	Henderson	Oswin	38.75	15.65
Wheat flour	GAB	Henderson	13.41	8.61
Chickpea flour, 27MHz, 20degC	Ferro-Fontan	Ferro-Fontan	2.16	0.05
Chickpea flour, 27MHz, 90degC	Ferro-Fontan	Ferro-Fontan	0.55	0.02
Chickpea flour, 1.8GHz, 20degC	Ferro-Fontan	Ferro-Fontan	0.63	0.38
Chickpea flour, 1.8GHz, 90degC	Ferro-Fontan	Ferro-Fontan	0.26	4.8e-4
Yellow dent field corn, 20MHz, 24degC	Ferro-Fontan	Ferro-Fontan	3.13	1.46
Carboxymethyl cellulose, 2.45GHz, 20degC	Chen	Chen	7.24	2.30
Hard red winter wheat, 18MHz, 20degC	GAB	GAB	5.54	4.73
Hard red winter wheat, 100MHz, 20degC	Ferro-Fontan	Ferro-Fontan	8.26	3.76
Hard red winter wheat, 1GHz, 20degC	Chen	Ferro-Fontan	9.64	6.34
Hard red winter wheat, 12.1GHz, 20degC	GAB	GAB	2.18	1.49
Apples, 915MHz, 60degC	GAB	Chung-Pfost	3.01	2.06
Apples, 2.45GHz, 60degC	Chen	Henderson	4.50	3.16
Grapes, 2.45GHz, 25degC	Owen	Chung-Pfost	0.44	0.01

## Table 3-5 Best fit adapted equations and error for moisture dependant lossfactor using experimental data from literature

It can be seen that the average MRE of the adapted water activity is below 10 for all but potato and wheat flour which were 38.8 and 13.4 respectively. The MRE for potato is misleading as the data points had a very large spread, which

is a symtpom of high experimental error. The equation fits for potato was actually very impressive considering the dataspread. The adapted Henderson equation fit for potato loss factor is shown in Figure 3-14. Table 3-5 shows that all the adapted water equations were generally effective at desribing loss factor moisture depenance. The lowest MRE equation fits were all below 10 with the exception of potato, due to the experimental data as shown in the figure below.



Figure 3-14 Adapted Henderson equation best fit for potato at 22°C, 2.8GHz, using loss factor measured by Holtz et al. (2010)

The adapted Ferro-Fontan had the largest number of RSS best fits for the data analysed, followed by the GAB equation. Out of the 6 adapted water activity equations analysed, 4 were best fits using RSS, and all 6 had best fits using MRE. A large number of sorption isothem equations exist because no one equation gives accurate results throughout the whole range of water activities, and for all types of foods (Al-Muhtaseb, et al., 2002). As both water activity and loss factor is dependent on water mobility, which is dictated by the complex interaction of the water with the food matrix, it stands to reason that both water activity and loss factor require a number of equations to accurately describe the moisture dependent behaviour.

Most of the equation fits that do not have to describe a negative  $d\varepsilon'/dM_{wb}$  are so good that there is no visible difference between the experimental trendline and the fitted trendline. An example of equation fits to dielectric measurements that do not contain a negative  $d\varepsilon'/dM_{wb}$  is shown in Figure 3-15. The equation fits were also excellent for dielectric measurements that contained decreasing  $d\varepsilon''/dM_{wb}$  to the point at which it reached zero, as shown in Figure 3-16. Error bars shown in Figure 3-15 and Figure 3-16 represent the standard deviation of the loss factor measurements, although no standard deviation was available for yellow dent field corn.



Figure 3-15 Adapted Ferro-Fontan equation best fit for (a) chickpea flour at 1.8GHz, 20°C using loss factor measured by Guo et al. (2008) and (b) Yellow dent field corn at 20MHz, 24°C, using loss factor measured by (Nelson, 1991c)



Figure 3-16 Best fit for apples at 60°C using loss factor measured by Feng et al. (2002) at frequencies of (a) 915MHz (adapted GAB fit) and (b) 2.45GHz (adapted Chen fit)

It is more difficult to perform a good non-linear regression when the extra mathematical term is required to describe a negative  $d\varepsilon'/dM_{wb}$ , which occurs when there is a decrease in loss factor at high moisture contents. Examples of equation fitting to dielectric loss measurements that contain a negative  $d\varepsilon'/dM_{wb}$  is shown in Figure 3-17. The difficulty in fitting the trendline arises from the number of fitting parameters used. It is therefore problematic to ascertain whether the comparatively worse MRE experienced for potato and wheat flour is due to limitations in the equations ability to fit, or due to limitations of the non-linear regression.



Figure 3-17 Adapted GAB equation best fit for (a) freeze dried potato at 25°C, 3GHz, using loss factor measured by Mudgett et al. (1980) and (b) wheat flour at 22°C, 27MHz, using loss factor measured by Kim et al. (1998)

# 3.5 Temperature dependence of proposed dielectric loss factor equations

There have been attempts to build temperature dependence into water activity equations. The equation that has been most successful in describing temperature dependence is the GAB equation. This was used by Kiranoudis et al. (1993) to describe the temperature dependence of potatoes, carrots, tomotaoes, green pepper, and oinion at  $30^{\circ}$ C,  $45^{\circ}$ C, and  $60^{\circ}$ C. Kiranoudis et al. (1993) achieved an MRE of 4.1%, 9%, and 15.8% for the moisture dependence of water activity of potato at  $30^{\circ}$ C,  $45^{\circ}$ C, and  $60^{\circ}$ C respectively.

The GAB equation is typically written as follows:

$$M_{db} = \frac{M_0 C K a_w}{(1 - K a_w) [1 - K a_w + C K a_w]}$$
Equation 3-3

Where  $M_0$  is the monolayer moisture constant; *C* and *K* are variables related to the energies of interaction between the first and further molecules at the individual sorption sites (Al-Muhtaseb, et al., 2002). To keep the error of monolayer estimation within ±15% the value of *C* must be greater than 5.5 (Lewicki, 1998). Theoretically *C* and *K* are related to the sorption enthalpies:

$$C = c_o exp\left[\frac{H_m - H_n}{RT}\right] = c_o exp\left[\frac{\Delta H_c}{RT}\right]$$
Equation 3-11

$$K = k_o exp\left[\frac{H_1 - H_n}{RT}\right] = k_o exp\left[\frac{\Delta H_k}{RT}\right]$$
Equation 3-12

Where  $c_0$  and  $k_0$  are entropic accommodation factors;  $H_m$ ,  $H_n$ , and  $H_1$  are the molar sorption enthalpies of the monolayer, multilayers and bulk liquid respectively [J mol<sup>-1</sup>], R is the universal gas constant [J mol<sup>-1</sup>K<sup>-1</sup>], and T is temperature [K]. The temperature dependence of the monolayer can be represented with the following equation, where  $\Delta H_X$  and  $X_{m0}$  are constants (Quirijns, et al., 2005):

$$M_0 = X_{m0} exp\left[\frac{\Delta H_X}{RT}\right]$$
 Equation 3-13

This temperature dependance can be applied to the GAB equation as previously adapted for moisture dependant loss factor (Equation 3-8). The resultant loss factor equation which is both temperature and moisture dependant is shown below in Equation 3-14, where the loss factor at zero moisture content,  $\varepsilon''_{0mT}$ , becomes a temperature dependant variable.

$$\varepsilon'' = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon''_{0mT} - UM_{wb}^S$$

Equation 3-14

This equation aims to act as curve fitting equation, rather than an apptempt to represent the physics of the dielctric response. The loss factor for chickpea flour was measured at temperatures from 20°C to 90°C in 10°C intervals by Guo et al. (2008). Non linear regression were carried out at 20°C and 90°C i.e. the extreme temperature of the range measured. The parameters *C*, *K*, and *M*<sub>o</sub> at 20°C and 90°C were then used to solve simultaneous equations to obtain values for  $c_0$ ,  $\Delta H_c$ ,  $k_0$ ,  $\Delta H_k$ ,  $X_{m0}$ , and  $\Delta H_x$ . However, the results were unsatisfactory, returning an average MRE of 43 over the temperature range. Hence the temperature dependence of loss factor cannot be described using the same GAB mathematical expressions used to describe the tempeature dependence of sorption isotherms. Bespoke mathematical expressions would have to be built into the adatpted water activity equations for loss factor. The adapted GAB equation temperature and moisture dependent fit of Chickpea loss factor is shown in Figure 3-18.



Figure 3-18 Adapted temperature dependant GAB equation fit for chickpea flour at 27MHz, using loss factor measured by Guo et al. (2008)

#### 3.6 Fitting of proposed dielectric constant equations

The proposed adaptions of water activity equations for loss factor can also be applied to the dielectric constant as presented in Table 3-6. The equations for dielectric constant are however, less complicated. There is no need for the additional term used to describe a fall in loss factor. The dielectric constant always increases with moisture content, making it easier for the water activity equation to adequately describe its moisture dependence. For example, the adapted Ferro-Fontan fit for potato moisture dependent dielectric constant is shown in Figure 3-19. Unlike the loss factor, it can be seen that there is no decrease in dielectric constant at high moisture content due to dilution of the salts. In fact salts suppress the dielectric constant, so any dilution should simply lead to further increases in the dielectric constant. The adapted Ferro-Fontan fit shown in Figure 3-19 has an MRE of 7.0%, which is considered a good fit (McMinn, et al., 2007; McLaughlin & Magee, 1998).



Figure 3-19 Ferro-Fontan equation fit for freeze dried potato at 25°C, 3GHz, using dielectric constant measured by Mudgett et al. (1980)

Name	Water activity equation	Adapted dielectric constant equation		
GAB	$a_{w} = \frac{1}{2} \left( \frac{-2M_{db} + M_{db}C - M_{0}C + \sqrt{4M_{db}CM_{0} + C^{2}M_{db}^{2} - 2M_{db}M_{0}C^{2} + M_{0}^{2}C^{2}}}{M_{db}K(C-1)} \right)$	$\varepsilon' = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon'_{0m}$		
Oswin	$a_w = (M_{db}/A)^{1/B} \left(1 + (M_{db}/A)^{1/B}\right)^{-1}$	$\varepsilon' = C(M_{wb}/A)^{1/B} \left(1 + (M_{wb}/A)^{1/B}\right)^{-1} + \varepsilon'_{0m}$		
Henderson	$a_w = 1 - e^{-ATM_{db}^B}$	$\varepsilon' = C\left(1 - e^{-ATM_{wb}^B}\right) + \varepsilon'_{0m}$		
Chung- Pfost	$a_w = e^{-e^{\frac{M_{db}-a}{b}}}$	$\varepsilon' = C\left(e^{-e^{\frac{M_{wb}-a}{b}}}\right) + \varepsilon'_{0m}$		
Chen	$a_w = e^{A - Be^{-CM}db}$	$\varepsilon' = D\left(e^{A - Be^{-CM_{wb}}}\right) + \varepsilon'_{0m}$		
Ferro- Fontan	$a_w = \frac{\gamma}{e^{AM_{db}r}}$	$\varepsilon' = \frac{\gamma}{e^{AM_{dw}r}} + \varepsilon'_{0m}$		

Table 3-6 Sorption isotherm equations adapted for dielectric constant

### 3.7 Conclusion

This study has analysed data from disparate sources to discover that a correlation exists between the points of inflection observed for water activity with respect to moisture content, and for the points of inflection observed for loss factor with respect to moisture content. The monolayer-multilayer transition is not always apparent in loss factor, however the transition from multilayer to solution provides a significant point of inflection for both water activity and loss factor that is highly correlated. The dielectric behaviour with respect to sorption isotherm points of inflection was found to be distinctly different for low sugar foods containing high levels of hydrocolloids compared to fruits.

Excluding hard red winter wheat, which had mixed behaviour, no change in loss factor was generally observed at the transition from monolayer to multilayer moisture for low sugar foods with high hydrocolloid content. A large increase in loss factor corresponding to the critical dielectric moisture content was found to occur at the transition from multilayer to solution at RF and microwave frequencies of 20MHz, 27MHz, 1.8GHz, 2.45GHz, 2.8GHz, and 3GHz.

For sugary fruits, a large increase in loss factor was observed at the transition from monolayer to multilayer moisture. The large increase in loss factor observed during the monolayer-multilayer transition in fruits suggests that water in the multilayer is more able to dialectically respond in sugary fruits compared to low sugar foods with high hydrocolloid content. A decrease in  $d\varepsilon'/dM_{wb}$  was found to occur at the transition from multilayer to solution.

Water activity equations have been successfully adapted to accurately describe moisture dependant behaviour of both the loss factor and the dielectric constant. These expressions for moisture dependant dielectric behaviour represent an important contribution to science, as they can be used to model microwave drying processes, which are solved at a single frequency. Currently, microwave and RF applications are optimised through trial and error, which can be costly and ineffective. Being able to model microwave and RF drying processes with the aid of these new equations will enable the optimisation of applicators through modelling, which could reduce costs and improve the probability of success. The equations could potentially be adapted further in future studies to include temperature dependence as well as moisture dependence.

## Chapter 4 – Porosity analysis

### 4.1 Introduction

RF and microwave applications are being proposed in the food industry to replace existing processes using conventional heat transfer. Conventional drying processes are very successful in terms of product taste, as they have typically been developed for decades to become a commercial success e.g. biscuits, crisps, pasta, breakfast cereals e.t.c. (Owens, 2001). When any new dying process is implemented, it is vital that it does not change the taste of the commercially successful product. Important food parameters that affect taste are porosity (Rodríguez-Ramíez, et al., 2012), moisture content (Roos, 2007), and fat content (Achir, et al., 2009). Porosity in particular is an important quantitative parameter that has a significant effect on mechanical, textural, and quality characteristics of dried material (Rodríguez-Ramíez, et al., 2012).

New methods for dehydrating potato slices to produce potato crisps are currently being developed with the aim of improving productivity, and be able to control the fat content to meet the demand for healthier snacks (Bows, et al., 2010). Quantification of porosity is important in order to ensure that new processing techniques for potato crisps do not compromise the product structure, and that the taste of commercially successful products is retained.

Typically, crisps are fried using either a continuous fryer (regular crisps), or a batch fryer ("kettle" crisps). In the continuous method, sliced and washed potatoes are continuously introduced into a vat of frying oil at a temperature of about  $185^{\circ}$ C or higher. They are conveyed through the oil by paddles or other means, and removed from the oil about two and a half to three minutes of frying by a conveyor belt when the moisture content of the crisps has been reduced to about  $0.02 \text{ kg kg}^{-1}$  by weight or less (Desai, et al., 2014). It is important that the final moisture content is less than  $0.02 \text{ kg kg}^{-1}$  to obtain a low water activity, and hence an acceptable shelf life. It is also important that the moisture content is below the glass transition point, which can be broad and

ill defined (Zeleznak & Hoseney, 1987), so that its texture is perceived by the consumers as crisp. Carbohydrates and proteins are plasticized by water, a state which is defined by their glass transition point, - lower moisture contents result in the carbohydrates and proteins acting similar to a solid-like glass (Roos, 2007). The glass transition for potato crisps at 20°C is approximately 0.22kg.kg<sup>-1</sup> wet basis (Mizuno, et al., 1998). An extremely low final moisture content of less than 0.02kg.kg<sup>-1</sup> wet basis is desirable to maximise the time taken for the food to turn soft when exposed to ambient air. The continuously fried potato crisp generally has texture and flavour characteristics which are usually recognisable by consumers as typical commercially produced continuous process potato crisps (Baas, et al., 2007; Bows, et al., 2010; Desai, et al., 2014; Henson, et al., 1997). Potato crisps produced by batch processes in kettle fryers are generally harder and crunchier than continuously fried crisps and have a flavour that some consumers find more appealing than typical continuously fried commercial crisps (Desai, et al., 2014; Henson, et al., 1997). As the name implies, batch process kettle frying of potato crisps involves placing a batch of unwashed potato slices in a kettle of hot oil, at an initial temperature of about 150°C. The cooking oil temperature follows a generally U-shaped time versus temperature profile where the temperature of the oil typically drops quite rapidly upon introduction of the potato slices into the oil by as much as 28°C or more. The oil temperature continues to fall more gradually to a low point of about 116°C after approximately 4 minutes. Heat to the kettle is then quickly increased and the temperature of the oil begins to gradually rise, finally reaching the initial temperature of about 150°C (Desai, et al., 2014). Batch frying of potato crisps usually takes about 13 minutes and the temperature-time profile can be found in a patent written by Henson et al. (1997). It is believed that the U-shaped temperature-time profile imparts the intense flavour that some consumers find more appealing than typical continuously fried commercial crisps (Desai, et al., 2014). However Henson et al. (1997) stated that the initial dehydration rate was the most important factor in providing the crunchy taste, which allowed the temperature-time curve to be reduced to 8 minutes using a more efficient kettle fryer. Kettle fried potato crisps also have a final moisture content of 0.02 kg kg<sup>-1</sup> or less (Henson, et al., 1997). Traditional kettle fryers are significantly constrained by burner capacity

and heat transfer capacity. The oil volume, initial fry temperature, and the potato batch weight need to be tightly controlled in order to obtain the correct U-shaped temperature-time profile. Production of kettle crisps is therefore less economical and more suited to small scale operations with fryer capacities ranging from as few as 60 pounds per hour up to 500 pounds per hour, compared to continuous fryers capable of producing 1000 to 5000 pounds per hour of finished product (Baas, et al., 2007).

The oil content of potato crisps has come under focus due to health reasons. However, while low oil content is desirable for nutritional reasons, excessively low oil content levels result in poor taste and texture. The trend in the snack food industry has been to provide consumers with healthier alternatives to traditional snack foods as there is great demand for healthier snacks (Desai, et al., 2014). As well as having a crunchier taste, kettle crisps typically have lower oil contents, typically ranging between 28% and 30% compared to continuous fried crisps which typically have between 32% and 38%. Kettle crisps are sliced 1.5mm to 1.7mm thick, compared to continuous fried crisps which are usually sliced to around 1.3 to 1.4mm thick. Bernard (1985) discovered that thicker crisps have lower oil uptake.

A patented new continuous cooking process has been developed by Baas et al. (2007) that mimics the U-shaped temperature-time profile produced by a batch kettle fried potato crisp operation. Potato slices are placed into hot oil in a flume portion of a continuous fryer. The potato slices exit the flume into an upstream portion of the fryer. Cooling oil is injected to the upstream portion of the continuously achieve the trough portion of the U-shaped temperature-time profile. The potato slices are then further fried in the downstream portion of the fryer in a region having an increased hot oil temperature. The increased hot oil temperature occurs by injecting hot oil into the downstream portion. The temperature of the injected hot oil can be controlled by the exit moisture content of the potato slices. The oil content of the process produces crisps with oil content of 20%-23% by weight which is significantly lower than the traditional kettle-style crisp (Baas, et al., 2007).

The conventional continuously fried potato crisp still holds a dominant proportion of the potato crisp market. The demand for healthier snacks with lower oil content has provided motivation for the recent development of potato crisps produced using microwave processing. A patented technique (Bows, et al., 2010) involving a microwave process has been developed to mimic the dehydration of standard continuous fried crisps to obtain a similar texture, with more control over the oil content. The potato slices are first blanched and then oil is added to the level desired for the final dehydrated product. The potato slices are then routed to a microwave oven for explosive dehydration, mimicking the rapid dehydration process that occurs in frying. Finish drying then occurs using microwave heating were dehydration occurs at a slower rate. Although microwaved crisps are likely to be expensive compared to fried crisps (Schiffmann, 2001a), they could become financially viable with the possible introduction of a fat tax. Patents, such as the microwave process described by Bows et al. (2010) are being generated by snack manufacturers to protect their commercial positions in the event of a fat-tax introduction. Quantifying porosity, moisture content, and oil content is important for these new processing developments to optimise the cooking process and to produce high quality targeted products.

The most challenging parameter to quantify is the potato crisp solid volume fraction due to the presence of oil, closed pores, and irregular shape. The potato crisp is made up of oil, water, solid material, and air (Rodríguez-Ramíez, et al., 2012), as presented in Equation 4-1 where  $v_s$  is the volume fraction of the solid,  $v_g$  is the volume fraction of the gas,  $v_w$  is the volume fraction of the water, and  $v_o$  is the volume fraction of the oil.

$$1 = v_s + v_g + v_w + v_o$$
 Equation 4-1

Porosity is defined as the "void" volume divided by the encapsulating "apparent volume" and the relationship between porosity,  $\phi$ , and solid volume fraction is shown in Equation 4-2.

$$\phi = 1 - v_s - v_w - v_o$$
 Equation 4-2

Porosity is an important physical property that is linked to sensory attributes such as firmness, crispiness, and crunchiness (Guessasma, et al., 2011). In the instance where a food product is completely saturated, and all the pores are full of water, it can be seen from Equation 4-2 that the food will have a porosity of zero. In most drying applications the physical space available for fluid movement is of interest. Ni et al. (1999) defined effective porosity for modelling purposes as shown in Equation 4-3. This is the total volume fraction of the gas, oil, and liquid in all the pore space (closed and open), or put another way, the space not occupied by solid structure. Effective porosity can be regarded as a more useful term than porosity as it directly indicates the amount of solid material, and the amount of space that is available for transport phenomena. The effective porosity presented in Equation 4-3 has been described as the "apparent porosity" by Datta (2007b) and as "net porosity" in an experimental investigation carried out by Pinthus et al. (1995).

$$\phi_{eff} = v_g + v_w + v_o = 1 - v_s$$
 Equation 4-3

When analysing porosity it is of course important to understand how porosity is generated. Uncooked potatoes typically contain 70–80% water and 16–24% starch. They consist of tightly packed parenchyma cells containing cytoplasm fluid, starch granules, mitochondria, golgi apparatus, amyloplasts, generative cells, and lipid droplets (Bordoloi, et al., 2012). The pore structure of potato crisps is created during the frying process where tissue disruption/separation occurs owing to the pressure caused by the expanding water vapour. As the middle lamella pectic substances between adjacent cell walls become softened and partially solubilized by the cook treatment, the cells are forced apart (Reeve & Neel, 1960; Costa, et al., 2001). The Reeve & Neel (1960) study showed that there are only small blisters, if any, in thin cut 1.1mm thick potato crisps (approx 6 to 8 cells deep), whereas blisters formed readily when cut to normal commercial 1.4mm thickness (approx 10-12 cells deep). Reeve &

Neel<sup>14</sup> hypothesised that for thicker crisps, the starch in the outer cells gel and become dehydrated more rapidly than those cells in the centre of the slices. Starch gelatinisation starts at approximately 57°C when swelling becomes irriversible, and may stop at approximately 95°C (Costa, et al., 2001). Expanding steam is thus trapped due to the gelling of the outer surface. At first the expanding steam is principally trapped in the tiny intercellular spaces. The internal pressure increases leading to the consequent build up of swollen pockets, or blisters. Water vapour is released by the bursting of a few localised sites that break under the stress caused by pressure.

A number of studies were able to calculate the porosity of foods using measurements of true density using various pycnometric techniques, and apparent density using displacement methods (Krokida, et al., 2000; Pinthus, et al., 1995). The true density is shown in Equation 4-4, where  $m_T$  is the total mass,  $V_s$  is the solid volume,  $V_w$  is the water volume,  $V_o$  is the oil volume, and  $V_t$  is the true volume.

$$\rho_t = \frac{m_T}{V_s + V_w + V_o} = \frac{m_T}{V_t}$$
Equation 4-4

The apparent density includes the air volume,  $V_g$ , as shown in Equation 4-5, and can also be expressed as the total mass,  $m_T$ , divided by the total volume,  $V_T$ .

$$\rho_T = \frac{m_T}{V_s + V_w + V_o + V_g} = \frac{m_T}{V_T}$$
Equation 4-5

Porosity is then calculated using Equation 4-6 (Krokida, et al., 2000; Pinthus, et al., 1995).

$$\phi = 1 - \frac{\rho_T}{\rho_t} = 1 - \frac{V_t}{V_T}$$
 Equation 4-6

Where  $V_T$  is the apparent volume, and  $V_t$  is the true volume.

Pinthus et al. (1995) conducted a study that measured moisture content and oil content as well as total mass enabling calculation of the oil and water volumes. Measurements of true and apparent densities enabled the calculation of the true and apparent volumes using Equation 4-4 and Equation 4-5. This enabled Pinthus et al. (1995) to quantify effective porosity using Equation 4-7 below:

$$\phi_{eff} = 1 - \frac{V_t - V_w - V_o}{V_T}$$
 Equation 4-7

Where  $V_T$  is apparent volume,  $V_t$  is true volume,  $V_w$  is water volume, and  $V_o$  is oil volume. As a result, Pinthus et al. (1995) was able to plot oil pick up versus effective porosity. An oil content of 30% (wet basis) corresponded to an effective porosity of approximately 0.43 for a fried restructured potato product. The techniques used to measure oil and moisture content was destructive.

The porosity can also be calculated using apparent density if the mass fraction and density of the constituents is known as described in Equation 4-8.

$$\phi_{eff} = \frac{\rho_T \left( 1 - f_w - f_o + \rho_g \frac{f_w}{\rho_w} + \rho_g \frac{f_o}{\rho_o} \right) - \rho_s}{\left( \rho_g - \rho_s \right)}$$
Equation 4-8

Where  $\rho_T$  is apparent density,  $f_w$  is the mass fraction of the water,  $f_o$  is the mass fraction of the oil,  $\rho_g$  is the air density,  $\rho_o$  is the oil density, and  $\rho_s$  is the solid density. The proof for this equation is shown in Appendix B.

The measurement of apparent density of potato crisps using displacement methods is problematic. Using liquid as a displacement medium can produce errors due to adsorption. Free flowing solid methods for measuring apparent density have potential issues relating to voids between the particulate and the food, the packing density of the particulate, and the size of the particulate in relation to sample thickness and the pore size. X-ray micro-computed tomography, or X-ray Micro-CT can be used to visualise the solid geometry of the potato crisp and can provide a non-intrusive and non-destructive technique for accurately deducing effective porosity defined in Equation 4-3 (Lin, et al., 2010; Lim & Barigou, 2004).

X-ray micro-CT is a combination of X-ray microscopy and tomographical algorithms. Its principle is based on contrast in the X-ray images being generated by differences in X-ray attenuation (absorption and scattering), arising principally from differences in density within the specimen. X-rays are passed through an object along many different paths in many different directions, thus, yielding an image which displays differences in density at thousands of points in a 2D slice through the specimen. Many contiguous slices, each of a certain finite thickness, are generated in this way (Lim & Barigou, 2004). Finally, a computerised reconstruction is carried out. The reconstruction of the samples is based on a mathematical formalism known as the Radon transform and its mathematical framework. After processing, the CT produces a spatial description of the object under analysis where the field of view is divided into elemental digital units known as voxels. Each voxel is characterised by the attenuation coefficient of the material inside it, which is related to density. This spatial digital characterisation of the sample under analysis results in a 3D distribution of the material density within the object which allows for further digital processing of the sample (Lin, et al., 2010).

Lim & Barigou (2004) carried out a Micro-CT study on aerated chocolate bar, strawberry mousse, honeycomb chocolate bar, chocolate muffin, and marshmallow were spatial cell size distribution, cell wall-thickness distribution, connectivity, and air cell volume fraction were quantified. Van Dalen et al. (2007) studied the internal structures of dry crackers, coated biscuits shells, and soup inclusions. The effectiveness of moisture barrier systems was assessed using porosity data from the Micro-CT analysis to provide inputs to a moisture transport model. Esveld et al. (2012) built on work by Dalen et al. (2007) using Micro-CT scan data of crackers to produce 3D CAD geometry of the pore network for a diffusion transport model. Lassoued et al. (2007) analysed scan data of bread to deduce porosity, and was able to

analytically calculate density as a result. Lassoued et al. (2007) found that bread had an open cellular structure, and was able to quantify mean cell size and mean wall thickness.

In order to assess porosity, subject matter is cropped so that the empty volume surrounding the porous medium is not considered in the Micro-CT porosity analysis. It is important that empty space external to porous media is not included as part of the void volume, as this would result in an incorrect calculation of porosity. Unfortunately, potato crisps are too thin and irregularly shaped to analyse a meaningful cropped volume. Sometimes, the crisp is only two pores thick. The present study uses a novel technique to determine the effective porosity of three types of potato crisp using Micro-CT scan data. Standard continuously fried crisps, continuously fried kettle crisps, and proprietary microwaved crisps were analysed. As means of comparison, the present study also calculated porosity for continuous fried potato crisps using apparent density measurements, where apparent density was measured using displacement of rapeseed, ceramic beads, and sunseed oil.

The microwave process described by Bows et al. (2010) was designed to replicate the continuous frying process to produce a potato crisp with the same structure and taste, whilst retaining control of the oil content. While the Micro-CT study was used to check the similarity of the potato crisp porosity, a forced preference test was used to check if the taste of the continuous fried potato crisp had been replicated using the microwave process.

#### 4.2 Materials and method

#### 4.2.1 Micro-CT porosity analysis

In the Micro-CT study, six crisps were scanned using a high resolution desktop X-ray Micro-CT system, Skyscan 1172 (Skyscan, Belgium), which consists of a microfocus sealed X-ray tube operating at a voltage of 60 kV and current of 163  $\mu$ A. The X-ray detector consists of a 1024 x 1024 pixels 12-bit digital cooled CCD-camera. The micro-CT system used is shown in Figure 4-1, and is

schematically represented in Figure 4-2. The six potato crisps scanned were provided by Walkers (PepsiCo Inc., UK) and consisted of three different potato crisp types, two of each type. The three types of potato crisp scanned were standard fried potato crisps, microwaved potato crisps, and continuous kettle fried potato crisps.



Figure 4-1 Skyscan 1172 Micro-CT system (Skyscan, Belgium)



Figure 4-2 Schematic of Micro-CT system

Each crisp sample scanned was approximately 7mm x 5.8mm x 1mm. The scanned samples were wrapped in parafilm to prevent oil from contaminating the sample cavity. The images were reconstructed using hierarchical InstaRecon<sup>®</sup> software to produce a stack of 2D images, each  $2.52\mu m$  apart (voxel size). One of the reconstructed images is shown in Figure 4-3.



Figure 4-3 Re-constructed image forming part of a 2D image stack were each notch on the horizontal axis is  $250 \mu m$ 

The image stack was then imported into Mimics<sup>®</sup> (Materialise, Belgium), an image processing program that can be used for segmentation and generation of 3D models from Micro-CT data. Each crisp had to be split into 4-5 sections along the 5.8mm dimension for data handling purposes. The greyscale range of the solid potato material was selected to produce a mask, or binary image of the solid phase. This was selected manually using the clear distinction between solid and fluid greyscale caused by the large difference in density. When assessing a particular product constituent using Micro-CT, it is important to first assess whether the material of interest has a sufficiently different density from the other constituent materials to permit separation by greyscale. In the case of potato crisps, the density of the solid material was measured to be 1434kgm<sup>-3</sup> (see 4.2.6 for method), which is significantly higher than the two other constituent materials, oil, which was measured to be 910kgm<sup>-3</sup> (Rogers & Mayhew, 1964).

The mask can include specs floating in space that are not connected to the main body. This is caused by noise captured within the greyscale threshold. To remove noise and artefacts the main body of the mask was selected and separated from areas of the mask that were not connected. In some instances the mask had to be manually edited to deselect pixels connecting the crisp from the parafilm wrap. Because the oil surrounds the crisp and is not part of the mask, there were instances where the parafilm wrap was only joined to the crisp by one pixel. In these instances, the mask was eroded by 1 pixel, the crisp could then be separated from the parafilm wrap, and the mask of the crisp was then dilated back by 1 pixel to its original size. A mask generated on one of the 2-D image slices is shown in Figure 4-4. Each notch on the horizontal grid in Figure 4-3 and Figure 4-4 corresponds to 250µm; hence the horizontal length of the image is 7.75mm.



Figure 4-4 Mask applied to 2D image stack to represent the solid potato structure, each notch on the horizontal axis is 250µm

The solid potato mask was then used to produce 3D CAD geometry of the potato crisp segment, as shown in Figure 4-5. Typically, effective porosity would be calculated by subtracting the solid volume from the total cropped volume, and then dividing by the total cropped volume. However, in this study the cropped scan of 2D slices includes large areas of air that sit outside of the potato crisp. Therefore a new method had to be devised to calculate the porosity. The CAD geometry of the porous medium was exported into 3-Matic<sup>®</sup> (Materialise, Belgium) where a "wrap" operation was carried out to determine the apparent volume of the potato crisp segment. Wrap operations are available in a number of commercially available CAD programs. The operation encapsulates the geometry and is analogous to wrapping the geometry in a blanket to cover up gaps. It is usually used to create water tight volumes from CAD geometry that may otherwise contain small gaps or imperfections. The maximum size of gap that is bridged by the wrap operation can be specified, which in this case corresponded to the maximum opening pore size on the external potato crisp surface. This ensures that the wrap covers the external surface only, and was determined approximately using a linear measurement tool on 2D slices in Mimics<sup>®</sup> (Materialise, Belgium). The wrap operation was visually inspected once it was generated to make sure that all the largest pore openings on the external surface had been bridged. The wrap operation carried out for this potato crisp segment is presented in Figure 4-6. As the operation creates a smooth surface, regions of the wrap are concave, where it covers large pores, and other regions of the wrap encapsulate too much volume as it covers sharp peaks in the solid.



Figure 4-5 Three dimensional CAD geometry of a segment of solid potato crisp structure generated from Micro-CT scan data



Figure 4-6 Micro-CT geometry wrapped to deduce the apparent volume

The volume of the solid (Figure 4-5) and the apparent volume of the same potato crisp (Figure 4-6) now exist in CAD as separate watertight volumes. These volumes can be quantified by the CAD package using standard functionality. Nearly all CAD packages are able to deduce volumes of CAD geometry accurately, especially in this case due to the number of vertices contained within the geometry. The effective porosity can be calculated using the following formula, where  $V_c$  is the apparent volume of the potato crisp and  $V_s$  is the volume of the solid porous potato structure.

$$\phi_{eff} = (V_c - V_s) / V_c$$

Equation 4-9

## 4.2.2 Apparent density measurement using free flowing solids

As comparison to the Micro-CT technique, the apparent density of continuous fried potato crisps was measured using displacement of free flowing solids,

initially with rapeseed of 2mm diameter, and then with ceramic beads of 0.5mm diameter. Six measurements were carried out using each free flowing media. The displacement measurements were carried out using the method described by Segnini et al. (2004). In each instance the mass of the free flowing solid was measured without potato crisps to assess repeatibility of packing density. When using ceramic beads as the displacement medium, it was important to break the crisps to smaller sizes of approximatley 5x5mm. This drastically improved repeatability of the experiment by reducing the potential for voids that can occur under the irregularly shaped crisp during measurment. This was required because the ceramic beads did not flow as freely as rapeseed. It also enabled the ratio of crisp to free flowing solid to be increased which decreased experimental error. The standard deviation using full size crisps was 7817kgm<sup>-3</sup> (4 measurements) while the standard deviation using broken up crisps was 94kgm<sup>-3</sup> (6 measurements).

#### 4.2.3 Apparent density measurement using sunseed oil

The apparent density was also measured using sunseed oil displacement as a comparison to the Micro-CT technique. Although this technique avoids any issues regarding packing density that occurs when using particulate solids, oil can be absorbed into the porous potato crisps which needs to be taken into consideration. The oil absorption can be deduced by weighing the potato crisps before the experiment, and afterwards when they have been removed from the bottle and the surface oil has drained off. Four measurements were taken using approximately 21g of crisps in each instance. Each measurement used a separate bag of potato crisps.

A stainless steel bottle was manufactured as shown in Figure 4-7 to measure the density of potato crisps using oil displacement. The bottle was made from two parts of stainless steel which screwed together, and a smaller diameter glass tube at the top. Marks were etched into each side of the bottle so that the bottle could be screwed together to the same point during measurement to ensure repeatability. The small diameter glass tube was designed to provide fine measurement of the fluid level. A mark was etched onto the glass to define a repeatable volume.



Figure 4-7 Cross section of bottle used to measure potato crisp density using oil displacement

The utilised volume of the bottle could be deduced using water. First, the mass of the empty bottle was measured. The bottle was then filled with water using a funnel until the water level reached the marking on the glass tube. The mass of the bottle containing water was then measured. This process was repeated three times and the mean volume was calculated using Equation 4-10 where  $m_{Tw}$  is the mass of the bottle containing water,  $m_B$  is the mass of the bottle, and  $\rho_w$  is the density of water.

$$V_B = (m_{T_W} - m_B) / \rho_w$$
 Equation 4-10

/

The oil density was also calculated using the bottle. The clean and empty bottle was filled with sunseed oil using a funnel until the oil level reached the marking on the glass tube. The mass of the bottle containing oil was then measured and the process was repeated three times. The density of sunseed oil was calculated using Equation 4-11 where  $m_{To}$  is the mass of the bottle containing oil,  $m_B$  is the mass of the bottle, and  $V_B$  is the utilised volume of the bottle.

$$\rho_o = (m_{To} - m_B) / V_B$$
 Equation 4-11

For measuring the density of potato crisps, crisps were first packed into the bottom half of the bottle and then filled with oil. As many potato crisps were packed in as possible in order to minimise experimental error, typically 20 grams of crisps were used per measurement. The top half of the bottle was then screwed on and filled up to the glass marking with sunseed oil. The mass of the full density bottle was measured 3 times to an accuracy of 0.01 grams and the average taken.

The oil was then carefully poured out of the density bottle and the top half of the bottle was screwed off. The equation used to calculate density does not account for excess surface oil, which extends beyond the envelope of the "apparent volume". To reduce this source of error, the crisps were then placed into a plastic container on top of a wire gauze. Blotting paper was located below the wire gauze to absorb the excess oil. It is important that the blotting paper does not come into direct contact with the potato crisps as it has the potential to pull the oil from the pores of the potato crisps. A saturated potassium hydroxide salt solution, which generates a similar water activity as packaged crisps, was contained in an open glass container beside the crisps to prevent moisture deviation. The saturated salt solution has a water activity of 0.093 at room temperature (Greenspan, 1977), while potato crisps nominally have a water activity of 0.1 (Schiffmann, 2001a). The mass of the potato crisps was measured each day until the mass stabilised. New blotting paper was used under the wire gauge after each mass measurement. The mass of the potato

crisps was then used to calculate the potato crisp density using Equation 4-12, where  $m_c$  is the mass of the potato crisps,  $V_B$  is the utilised volume of the bottle,  $m_{TOD}$  is the total mass of the full bottle containing oil and crisps,  $m_B$  is the mass of the bottle,  $\rho_o$  is the density of the sunseed oil, and  $m_{c\_ab}$  is the mass of the oil absorbed into the potato crisps. The derivation of Equation 4-12 is described in Appendix C.

$$\rho_{c} = \frac{m_{c}}{V_{B} - \frac{m_{TOD} - m_{c} - m_{B}}{\rho_{o}} + \frac{m_{c} - m_{c_{ab}}}{\rho_{o}}}$$
Equation 4-12

#### 4.2.4 Water mass fraction

Measurement of the water mass fraction (moisture content wet basis) of potato crisps is complicated by the presenece of oil. Moisture content is typically less than 0.02kg.kg<sup>-1</sup> (Schiffmann, 2001a; Ziaiifar, et al., 2008), while Walkers (PepsiCo, UK) potato crisp packaging data shows that oil content constitutes 32.3% of the total potato crisp mass. Oven drying is therefore not a satisfactory method for determining moisture content as oil oxidation and evaporation occur at elevated temperatures. Hence, the water mass fraction of continuous fried potato crisps was measured gravimetrically through exposure to 4 litres/min of dry nitrogen at room temperature in a desiccator until mass equilibrium had been obtained. This method avoids the use of hazardous desicants such as Phosphorus Pentoxide (P2O5). Phosphorus Pentoxide is highly hygroscopic chemical that can cause severe burning and has a Threshold Limit Value of 1 mg.m<sup>-3</sup> (Merck, 2010). It has been used by Ertekin & Gedik (2004) and Perdomo et al. (2009) to achieve zero moisture content in various foods through continuous exposure in an enclosed volume. For the dry nitrogen technique proposed, a control dish containing oil was included in the desiccator to check that the oil mass remained stable throughout the measurement process. The mass measurement interval was 2 weeks and twelve sample dishes were measured in the desiccator, each containing approximately 2.4 grams of potato crisp. The rig used for moisture measurement is shown schematically in Figure 4-8. A picture of the desiccator loaded with sample dishes is shown in Figure 4-9.



Figure 4-8 Dry nitrogen experiment setup for drying samples to zero water activity



Figure 4-9 Desiccator loaded with sample dishes for dehydration using dry nitrogen

Dean and Stark method (ASTM D-95) was used as a quick, non-hazardous, and approximate check for the final moisture content obtained by the proposed dry nitrogen technique. Although Dean and Stark method (ASTM D-95) is less well suited to measuring low moisture contents where an accuracy of +/-0.5% wet basis is desirable, there was no other established technique suitable for carrying out the check due to the presence of oil and health and safety. Three replicates were measured. This method involved reflux distillation of samples in the presence of toluene, a solvent which is immiscible with water, and separation of the water phase. For the comparative study, both Dean and Stark and the proposed dry nitrogen technique was used to measure the moisture content of potato crisps that had been supplied by a potato crisp manufacturer, placed in a large sealed 5 litre bag, and allowed to equilibrate for 4 weeks before measurement. The dry nitrogen technique was also used to measure the

moisture content of standard unsalted continuous fried crisps that had been packaged as per the normal commercial procedure.

#### 4.2.5 Oil mass fraction and density

Oil mass fraction was provided by the manufactuer and is published on the potato crisp packaging. Oil used for frying was provided by the manufacturer, and the density of the oil was measured by transferring it into a measuring beaker to a set volume, and weighing the gain in mass.

#### 4.2.6 Solid potato density

The solid potato density was calculated using Equation 4-13, which can be used for uncooked potato, where  $\rho_T$  is the apparent density,  $f_w$  is the mass fraction of the water, and  $\rho_w$  is the density of water. The apparent density of uncooked potato was measured to be 1055kgm<sup>-3</sup> through water displacement of peeled potatoes (3 replicates), and the mass fraction of water was measured to be 0.822kg.kg<sup>-1</sup> by oven drying at 105°C for 24 hours (3 replicates). The proof for Equation 4-13 can be found in Appendix D.

$$\rho_s = \frac{1 - f_w}{\frac{1}{\rho_T} - \frac{f_w}{\rho_w}}$$

Equation 4-13

#### 4.2.7 Forced preference consumer test

The forced preference consumer test was carried out by Walkers (Pepsico, UK) with the microwaved potato crisps and the continuous fried potato crisps. The object of the survey was to determine how successful the new proposed microwave manufacturing process is as replicating the taste of continuous fried potato crisps whilst retaining control of the oil content. The test was carried out with 200 people who were selected to be balanced across gender, age (<35, >35), socio-economic grouping, and eating frequency. The consumers saw two products in a randomised order, to avoid ordering effects. For each product in turn, the consumer was asked how much they liked the product across a

number of attributes on a 9 point scale as well as some diagnostic questions on a 5 point scale. Once they answered all the questions, they were asked which product they preferred.

### 4.3 Results

Parameter			Number of
measured	Method	Samples	replicates
		Standard continuous	10
Effective	Micro-CT	fried unsalted potato	(2 crisps, 5
porosity		crisps	segments each)
	Micro-CT	*	8
Effective		Microwaved unsalted potato crisps	(2 crisps,4
porosity			segments each)
Effective porosity	Micro-CT	Continuous kettle	10
		fried thick unsalted	(2 crisps, 5
porosity		potato crisps	segments each)
Apparent	Rapeseed	Standard continuous	6
Apparent density	displacement	fried unsalted potato	(approximately
defisity	displacement	crisps	10 grams each)
Apparent density	Ceramic beads	Standard continuous	6
	displacement	fried unsalted potato	(approximately
density	uispiacement	crisps	19 grams each)
Apparent density	Sunseed oil	Standard continuous	4
	displacement	fried unsalted potato	(approximately
activity		crisps	21 grams each)
Apparent	Water displacement	Uncooked potato	3
density			(approximately
	F	~	45 grams each)
	~	Standard continuous	
Water mass	Dean and stark	fried unsalted potato	3 (approximately
fraction	(ASTM D-95)	crisps non-standard	16 grams each)
		packaging	
		Standard continuous	
		fried unsalted potato	12 for each
Water mass fraction	Gravimetric using	crisps non-standard	12 for each
	dry nitrogen exposure	packaging	product
		Standard continuous	(approximately 4.3 grams each)
		fried unsalted potato	4.5 grains each)
		crisps	
		Chops	3
Water mass	Oven drying at	Uncooked potato	(approximately
fraction	105°C for 24 hours	encoured polato	14 grams each)
			i i grunns cucil)

This section will present the results of the experiments presented in Table 4-1.

Table 4-1 Summary of experiments carried out

#### 4.3.1 Micro-CT effective porosity

The effective porosity results for each segment of the six potato crisp samples analysed using Micro-CT is presented in Table 4-2, along with the average effective porosity and standard deviation.

The average effective porosity of the standard fried potato crisps is 0.63 with a standard deviation of 0.032. A reasonably large standard deviation is expected for fried potato crisps. There is temperature variations in the fryer and each crisp will be subjected to different frying conditions as it finds a different path through the continuous fryer.

The microwave process is designed to replicate the frying process, so it is important that the effective porosity, and the taste of the microwaved crisp, does not deviate too much from the continuous fried potato crisp. The effective porosity of the microwaved potato crisp is 0.653, which is very similar to the effective porosity of 0.627 for the continuous fried potato crisp. This similarity in effective porosity helps demonstrate the success of the microwave process. The results of the forced preference survey showed that the microwaved dehydrated product was preferred by 51% which allows us to conclude that consumers equally preferred microwaved potato crisps and standard fried crisps. The microwave process was designed to imitate the dehydration process in the continuous fryer, and it can be deemed to be very successful in terms of final crisp effective porosity is lower for the microwaved crisp at 2.8% of the mean as opposed to 5.1% of the mean for fried crisps.

Type of crisp	Effective porosity of segments from crisp sample 1	Effective porosity of segments from crisp sample 2	Average effective porosity of potato crisp segments	Standard deviation of effective porosity of potato crisp segments	Standard deviation of effective porosity as percentage of the mean
Standard packaged fried potato crisps	0.615	0.627			
	0.606	0.671		0.032	5.1%
	0.613	0.68	0.627		
	0.604	0.654			
	0.581	0.615			
Packaged microwaved potato crisps	0.659	0.625	0.653	0.018	2.8%
	0.656	0.651			
	0.679	0.629			
	0.664	0.664			
Packaged continuous Kettle fried thick potato crisps	0.641	0.558			
	0.622	0.59			
	0.538	0.56	0.543	0.067	12.3%
	0.441	0.493			
	0.45	0.533			

 Table 4-2 Effective porosity obtained from Micro-CT analysis

The continuous fried "Kettle" crisps, manufactured using a process that mimics batch frying, have a markedly lower effective porosity at 0.543 compared with the standard continuous fried crisps (0.627), and the microwaved crisps (0.653). It can be seen visually from the Micro-CT scan images in Figure 4-10 that the continuous kettle fried crisp has a larger solid volume fraction, and that the standard continuous fried crisp has a higher effective porosity. The frying time of kettle crisps is longer at 7-9 minutes, and the oil temperature is lower throughout the process at 150°C to 110°C, compared to 2.5 to 3 minutes at 185°C for continuous fried crisps (Desai, et al., 2014; Baas, et al., 2007). A longer frying time at lower temperature would result in less explosive dehydration. It stands to reason that less explosive dehydration seen in frying of kettle crisps would produce a lower effective porosity. A lower effective porosity signifies more solid material which is likely to be a driving factor behind the crunchy taste for which kettle crisps are known. The lower oil uptate of thicker crisps observed by Bernard (1985) may be evidence of lower porosity. Most oil uptake occurs post frying (Bouchon & Aguilera, 2001), and it is intuitive that a lower void volume would suck in less oil.



Figure 4-10 Micro-CT generated 2D image of (a) continuous fried kettle crisp, and (b) standard continuous fried crisp, each notch on the horizontal axis is 250µm

The standard deviation of the effective porosity for the kettle crisp is 12.4% of the mean, which is a lot higher than for the other two crisp types. The kettle frying process is inherently more variable than the standard continuous frying technique due to large variations in temperature during frying, which is likely to cause more variation in porosity. The start of the kettle frying process is particularly likely to cause variation in temperature as a large batch of raw unwashed potato slices is introduced into a hot kettle of oil causing a U shaped temperature profile (Desai, et al., 2014). Conversely continuous fried crisps enter the fryer on a continuous conveyor and the frying temperature is kept constant to  $\pm/-2^{\circ}C$ .

## 4.3.2 Apparent density using displacement of free flowing solids

The measured apparent density of standard continuous potato crisps using rapeseed displacement was 377kgm<sup>-3</sup> with a standard deviation of 82kgm<sup>-3</sup>, which is 22% of the mean. This result is similar to the apparent density measured by Segnini et al. (2004) using rapeseed displacement, which ranged from 380kgm<sup>-3</sup> to 450kgm<sup>-3</sup> for a variety of commercial potato crisps. The measured apparent density using ceramic beads displacement was 935kgm<sup>-3</sup> with a standard deviation of 94kgm<sup>-3</sup>, which is 10% of the mean. The variation in packing density for the rapeseed was measured to be 0.2% whilst it was only 0.1% for the ceramic beads. Inaccuracies are likely to be caused by the rapeseed diameter, which at 2mm is twice that of potato crisp thickness. Not only is this an issue with respect to thickness, but it means that the rapeseed can only contact the potato crisp every 4mm, which is not enough to take into account some surface features. The ceramic beads result is likely to be more accurate, however the size of the beads is 0.5mm, and study of the Micro-CT images generated in this study shows that some of the open pores on the surface of the crisp are of a similar size. Hence there is the risk that some beads could lay within the potato crisp during measurement, which could result in an artificially higher apparent density. Hence, measurement of apparent density using displacement of free flowing solids is inherently inaccurate if the pore size is significant in relation to the thickness of the food.

#### 4.3.3 Apparent density using displacement of sunseed oil

The average potato crisp apparent density was 785kgm<sup>-3</sup>, with a standard deviation of 53.8 kgm<sup>-3</sup>, which is 6.8% of the mean. This standard deviation is lower than the 22% and 10% from the mean obtained through rapeseed and ceramic bead displacement respectively. This shows that using oil displacement leads to more repeatable results compared to free flowing solids. A relatively high standard deviation is expected as potato crisps are inherently variable products. The calculations associated with the oil displacement method account for oil absorbed in the potato crisp during the experiment. However, there is a risk that the additional surface oil picked up during the displacement measurement may offset the density result. Although surface oil was drained from the potato crisps until stability was reached, this may not have been completely effective due to oil viscosity. If excess oil is retained on the surface after the experiment, the resultant density calculated would be artificially high. It is therefore interesting to note that the calculated apparent density of 785kgm<sup>-3</sup> obtained with oil displacement is lower than the apparent density of 935kgm<sup>-3</sup> calculated using ceramic bead displacement. This may suggest that the method used for draining off the surface oil is effective. Although this is uncertain as the density obtained using ceramic bead displacement could be artificially high due to the possibility of ceramic beads entering the internal potato crisp pore network. The two apparent density measurements can be used to calculate effective porosities which can be compared to the porosity results obtained using the novel Micro-CT method, providing some verification of the new method.

#### 4.3.4 Water mass fraction

For the comparative study, the Dean and Stark measurement extracted water corresponding to a moisture content of 0.0223kg.kg<sup>-1</sup> wet basis with 0.0033 kg.kg<sup>-1</sup> standard deviation, whilst the dry nitrogen technique extracted water corresponding to a moisture content of 0.0262kg.kg<sup>-1</sup> wet basis with 0.0010kg.kg<sup>-1</sup> standard deviation. Hence the dry nitrogen technique extracted more moisture, and provided more consistent results. The dry nitrogen

technique was also more accurate as the mass of the dishes could be measured to an accuracy of 0.0001 grams, with an average moisture removal of 0.11 grams per sample. Hence the measurement accuracy of the dry nitrogen technique is approximately 0.1% of the water measured. The extracted moisture from the Dean and Stark experiment was measured to the nearest 0.05ml and the average water extracted for each experiment was 0.9ml. Hence the measurement accuracy of the Dean and Stark measurement is approximately 6% with respect to the water removed. Although the Dean and Stark method is not ideal for such low moisture content measurements, it provides a useful non-hazardous check of the dry nitrogen technique as none of the typical measurement techniques employed is suitable/desirable due to the presence of oil and health and safety. The fact that the stabilisation of the drying curve is actively monitored using the dry nitrogen technique gives confidence that the method has been given sufficient time to remove as much water as is feasible using the technique. The disadvantage of using dry nitrogen is that it takes a long time, the drying curve presented in Figure 4-11 shows that the drying time is 27 weeks. Gravimetric determination of water content using dry nitrogen was selected as the preferred method for deducing moisture content due to its certainty and accuracy.



Figure 4-11 Dry nitrogen gravimetric measurement of potato crisp moisture content (potato crisps samples supplied by manufacturer in large 5 litre bag)
The moisture content of standard continuous fried potato crisps were consequently measured using the gravimetric dry nitrogen technique. These potato crisps were commercially packaged as per the standard method, unlike the crisps used in the comparative study. The extracted water resulted in a moisture content of 0.0173kg.kg<sup>-1</sup> with 0.0009kg.kg<sup>-1</sup> standard deviation after 22 weeks stabilisation. Schiffmann, (2001a) and Ziaiifar et al. (2008) both stated that commercial potato crisps have a moisture content lower than 0.02kg.kg<sup>-1</sup>. This is in agreement of the dry nitrogen moisture measurements, and provides futher verification of the technique.

#### 4.3.5 Analytical calculation of effective porosity

Analytical calculations of effective porosity were made using the apparent density measurements for comparison with the porosity measurements carried out using Micro-CT. The mass fraction of oil,  $f_o$  is 0.323kg.kg<sup>-1</sup> as specified on the potato crisp packaging, the water mass fraction  $f_w$  was measured to be 0.017kg.kg<sup>-1</sup> using the dry nitrogen technique, the density of oil,  $\rho_o$ , was measured to be 910kgm<sup>-3</sup>, and the density of solid potato was measured to be 1434kgm<sup>-3</sup>. The measured density of the solid potato material is very close to the value of 1419kgm<sup>-3</sup> presented in literature (Datta, 2007b; Rahman, 2005). The density of water is 998.2kgm<sup>-3</sup> and the density of air is 1.2kgm<sup>-3</sup> (Rogers & Mayhew, 1964). Equation 4-8, which is shown below for convenience, was used to calculate the effective porosity using the apparent density of 935kgm<sup>-3</sup> measured using ceramic bead displacement. The effective porosity was calculated to be 0.639 using the apparent density of 785kgm<sup>-3</sup> measured using oil displacement.

$$\phi_{eff} = \frac{\rho_T \left( 1 - f_w - f_o + \rho_g \frac{f_w}{\rho_w} + \rho_g \frac{f_o}{\rho_o} \right) - \rho_s}{\left( \rho_g - \rho_s \right)}$$
Equation 4-8

The effective porosity value of  $0.627\pm0.032$  obtained through Micro-CT analysis is higher than the effective porosity value of  $0.570\pm0.043$  calculated using the apparent density measured using ceramic bead displacement. The porosity calculated using the apparent density obtained via ceramic bead displacement is likely to be slightly low due to the experimental error that could be caused by some of the ceramic beads entering the potato crisp open pores during density measurement. The ceramic beads have a diameter of 0.5mm and some of the external pore entries were measured to be approximately 0.6mm on the Micro-CT 2D images.

The Micro-CT value of  $0.627\pm0.032$  for effective porosity is only slightly lower than the effective porosity of  $0.639\pm0.025$  calculated analytically using the apparent density obtained from oil displacement. The difference between Micro-CT effective porosity, and the analytically calculated effective porosity of 0.0639 is 0.012, which is well within the standard deviation of both methods. This provides verification for the new Micro-CT technique for quantifying porosity.

The apparent density of potato crisps can be calculated analytically using the porosity measured in the Micro-CT analysis. This analytical calculation can be carried out by rearranging Equation 4-8 to make apparent density the subject, as shown below. Using an effective porosity of 0.627 for continuous fried potato crisps, the apparent density is 812kgm<sup>-3</sup> using Equation 4-14.

$$\rho_T = \frac{\rho_s \left(1 - \phi_{eff}\right) + \rho_g \phi_{eff}}{\left(1 - f_w - f_o + \rho_g \frac{f_w}{\rho_w} + \rho_g \frac{f_o}{\rho_o}\right)}$$
Equation 4-14

### 4.4 Conclusion

A new technique has been developed in this study for accurately quantifying the effective porosity of thin, heterogeneous, irregularly shaped food containing both surface oil, and oil within the pores of the food structure. The method uses manipulation of Micro-CT scan data and a CAD wrap operation. The present study shows how this novel method can be used to gain insights into product quality. The effective porosity, and variation in effective porosity, can be used to quantify the effect different cooking processes have on the product structure. The effective porosity data obtained reflects development of cutting edge production techniques, controlling the temperature-time curve and dehydration rate to deliver specific product attributes. "Kettle" crisps produced using a continuous fryer that mimicked the batch frying process, have a low effective porosity of 0.54 which corresponds to the desired crunchy taste of batch fried "kettle" crisps. The effective porosity had a standard deviation of 0.067 which was high compared to standard continuously fried crisps. High variation is indicative of the traditional batch frying process due to the highly variable oil temperature in the cooking process. Fried potato crisps and potato crisps microwave dehydrated to mimic the drying profile of fried potato crisps had very similar effective porosities of 0.63 and 0.65 respectively. In a forced preference consumer test (n=200), there was no statistically significant difference in preference (51% preferred the microwaved crisp).

# Chapter 5 - Sorption isotherms

### 5.1 Introduction

A number of RF and microwave applications for pasta, biscuits and potato crisps have been identified, and their success depends on the dielectric behaviour of the product over the range of moisture contents during the drying process. The nature of the adsorbed water can vary significantly within different materials. For instance, food such as potato crisps and biscuits must be dried below the monolayer moisture content to obtain a shelf life longer than a month (Labuza & Altunakar, 2007; Wang & Brennan, 1991; McMinn, et al., 2007; Schiffmann, 2001a). Sorption isotherms relate the water activity within the foodstuff to the water content, and can be used to understand the physical nature of the adsorbed water. The monolayer is defined by the sorption isotherm using the BET monolayer formulation (Mazza & LeMaguer, 1978). Other drying applications involve drying below a critical water activity of 0.6, below which microbial growth ceases, and water transitions from acting as a solution to multilayer behaviour (Labuza & Altunakar, 2007). The moisture content corresponding to the transition from solution to multilayer is also defined by the sorption isotherm (Mujumdar, 2006). As well as defining the monolayer and transition from solution to multilayer, the sorption isotherm can also be used to define the isosteric heat (Wang & Brennan, 1991). The isosteric heat must be supplied in order to remove bound moisture. The energy levels of isosteric heat can be comparable to latent heat at low moisture contents (Wang & Brennan, 1991), and must be taken into account when carrying out feasibility studies of microwave finish drying applications.

This chapter presents a combination of literature data and experimental observations to obtain sorption isotherms for pasta, biscuits and potato crisps, and will subsequently be compared with dielectric property data in Chapter 6.

#### 5.1.1 Sorption behaviour of potato crisps

There are numerous publications of sorption isotherms for raw potato (McLaughlin & Magee, 1998; Wang & Brennan, 1991; Makower & Dehority, 1943; Kaymak-Ertekin & Gedik, 2004; McMinn & Magee, 1999). Published desorption isotherms at 30°C and 60°C for potato are shown in Figure 5-1. All the curves were produced using best fit sorption equations with the exception of Kiranoudis et al. (1993), which is measured data complete with standard deviation error bars. It can be seen there is broad agreement at 30°C, however there is a significant varance in the desorption isotherms reported by the authors at 60°C. On average, the standard deviation observed by Kiranoudis et al. (1993) for dry basis moisture content measurement is 2.1 times higher at 60°C compred to 30°C. This would suggest that either the experimental proceedure becomes less accurate at higher temperature, or potato moisture content is perhaps more variable at high temperature for a given water activity.



Figure 5-1 Desorption isotherms for potato at (a) 30°C and (b) 60°C

Although there is a lot of data available for uncooked potato, there is a lot less data available for cooked products as noted by McMinn et al. (2007). To date, there has been no published sorption data for potato crisps. Moisture measurement techniques are more difficult for potato crisps due to the large amount of oil present. For instance the Walkers salt and shake potato crisps used in the present study has an average oil content of 0.323kg.kg<sup>-1</sup> wet basis

as reported on the potato crisp packaging. This may be one of the reasons for the lack of published sorption isotherm data. Although the solid mateiral of the potato crisp has the same basic chemical composition as uncooked potato, Vullioud, et al. (2004) reported that foodstuffs with similar chemical composition but different physical characteristics had different sorption isotherms. Iguedjtal, et al., (2008) furthered this argument to say that if the same variety and the same species of foods are dried or processed differently, they will have a different moisture content for the same water activity. Consequently sorption isotherms were measured for potato crisps.

#### 5.1.2 Sorption behaviour of biscuits

Sorption isotherms of biscuits have been measured by McMinn et al. (2007), Guillard et al. (2004) and Palou et al. (1997) as shown in Figure 5-2. It can be seen from Figure 5-2 that there are significant differences in the sorption behaviour of the biscuits and cookies presented in literature. Unfortunately none of the biscuits measured in Figure 5-2 could be sourced for the present study. The present study requires sorption isotherm and dielectric measurements of the same food species for comparison. Hence digestive biscuits were selected for sorption and dielectric response measurement as digestive biscuits could be locally sourced.



**Figure 5-2 Desorption isotherms for biscuits** 

#### 5.1.3 Sorption behaviour of pasta

Adsorption properties for spaghetti were measured by Lagoudaki et al. (1993) at 22°C, 30°C, 37°C and 45°C as shown in Figure 5-3. Lagoudaki et al. (1993) reported a comparison with sorption results of macaroni measured by Pixton & Warburton (1973). The comparison showed that the results differed by an average of 3%. It can therefore be assumed that the sorption data presented by Lagoudaki et al. (1993) is valid for penne pasta, and can be used for comparision with dielectric measurement of penne pasta. Pixton & Warburton (1973) found that there was no hysteresis with adsorption and desorption for macaroni pasta. The results presented by Lagoudaki et al. (1993) for pasta at 22°C could be used for comparison to dielectric measurement results, hence no measurements were required for pasta sorption. The BET monolayer was

calculated to be 0.0704 dry basis through non-linear regression of the 22°C sorption data of pasta reported by Lagoudaki et al. (1993). Pasta is dried to a moisture content of 0.136-0.149kg.kg<sup>-1</sup> dry basis (Schiffmann, 2001a; Marchylo & Dexter, 2001), which is above the monolayer moisture content. A moisture content of 0.149kg.kg<sup>-1</sup> dry basis corresponds to a water activity of 0.6 at 22°C using the GAB parameters presented by Lagoudaki et al. (1993). It can be seen from Figure 5-3 that there is a point of inflection at a water activity of 0.6 at 22°C which corresponds to the transition from multilayer to solution. Hence, the moisture content of finished pasta is just below the critical water activity of 0.6 (Labuza & Altunakar, 2007).



Figure 5-3 Adsorption isotherms for spaghetti pasta measured by Lagoudaki et al. (1993)

### 5.2 Materials and method

#### 5.2.1 Sorption isotherm measurement

Sorption isotherms were measured for biscuits and continuous fried potato crisps due to the lack of available data within the literature for these foodstuffs.

McVities Digestive biscuits and Walkers salt and shake continuous fried crisps were purchased from a local supermarket. The biscuits consist of 0.213kg.kg<sup>-1</sup> fat of which 0.101kg.kg<sup>-1</sup> are saturates, 0.629kg.kg<sup>-1</sup> carbohydrates of which 0.166kg.kg<sup>-1</sup> is sugar, 0.036kg.kg<sup>-1</sup> fibre, 0.072kg.kg<sup>-1</sup> protein, and 0.013kg.kg<sup>-1</sup> <sup>1</sup> salt. The potato crisps consist of 0.323kg.kg<sup>-1</sup> fat of which 0.026kg.kg<sup>-1</sup> are saturates, 0.522kg.kg<sup>-1</sup> of carbohydrate of which 0.004kg.kg<sup>-1</sup> are sugars, 0.04kg.kg<sup>-1</sup> of fibre, and 0.062kg.kg<sup>-1</sup> protein. The Walkers salt and shake potato crisps were measured in an unsalted state. Salt is added to potato crisps after the drying process. Hence unsalted measurements of the sorption isotherm provide more relevant information for the drying process.

Sorption isotherms were measured gravimetrically using saturated salt solutions. This method is well established and has been used and presented by a large number of authors including Kaymak-Ertekin & Gedik (2004), Kiranoudis et al. (1993), McLaughlin & Magee (1998), McMinn & Magee (1999), and Zhang, et al., (1996). Salt solutions were preferred over sulphuric acid as it is easier to maintain a consistent relative humidity with salt solutions. The moisture content of the solution will change as it interacts with the food sample during the equilibrating time. The solution will also interact with the outside environment when the container is opened for mass measurement of the sample. It is easy to visually check that salt granules are visible in the salt solution, signifying saturation. If there are very few visible salt granules, then more salt could be added to the solution. In such an instance, the salt solution was removed from the plastic container and placed into a fume cupboard where more salt was added. If there was very little liquid in the solution, then more deionised water was added.

The stability of the oil with respect to temperature needed to be assessed in order to select a suitable range of temperatures to measure sorption isotherms. Oil may be unstable at higher temperatures due to either oxidation or evaporation, and this may invalidate the gravimetric measurement technique used to generate the sorption isotherm.

Experiments were carried out to assess oil stability at different temperatures. Three temperatures were assessed, 30°C, 40°C and 55°C, with two dishes of oil measured for each temperature. Each dish of oil was enclosed in a sealed air tight container with magnesium chloride to ensure stable moisture content, and an empty control dish. The container was placed in an incubator, which controlled the temperature to  $\pm 1^{\circ}$ C. The 30°C oil dish was very stable and it's mass varied by  $\pm 0.05\%$  whilst maintaining a consistent average. The oil mass fraction is on average 0.323kg.kg<sup>-1</sup> according to the potato crisp packaging data. Hence a variation of  $\pm 0.05\%$  equates to 0.00017kg.kg<sup>-1</sup>, which was deemed acceptable with respect to the measurement of water content.

Oil samples at 40°C and 55°C gained 4% to 5% of mass before stabilising after approximately 80 days. A similar experiment was carried out by Crapiste et al. (1999) were sunflower oil mass stabiliy was studied at 37°C, 47°C, and 67°C. Crapiste et al. (1999) observed a slow decrease in mass between 0.02-0.1% during the initial stages, and then a continuous increase in mass due to oxidation. The intial decrease in mass was attributed to partial evaporation of humidity and other volitile components. As the oxidation processs proceds, oxygen is incorporated into unsaturated fatty acids and more hydroperoixides are formed. There was also dimerization and polymerization reactions and oxygen uptake is faster, producing a weight gain (Crapiste, et al., 1999). Weight remained relatively constant at 37°C where little or no oxidation took place, while sunflower oil samples at 47°C and 67°C gained 0.4% and 0.8% mass respectively after 100 days. Stabailisation was not achieved at the end of the experiment carried out by Crapiste et al. (1999), with oil at 47°C, and 67°C showing steep increasses in mass at 100 days. The surface to volume ratio was more than 5 times higher in the present study compared to the study carried out by Crapiste et al. (1999) leading to faster stabilisation times. The gain of mass by the oil at 40°C and 55°C observed in the present study would skew sorption isotherm results due to potato crisps and biscuits having fat contents of 0.323kg.kg<sup>-1</sup> and 0.213kg.kg<sup>-1</sup> respectively. Therefore, it was decided to measure sorption isotherms at 20°C and 30°C to ensure oil stability.

Salt solutions were selected that would provide a range of relative humidity measurement points at reasonably spaced intervals for sorption isotherm measurement. Sorption isotherms were not measured above a water activity of 0.8 as spoiling would occur before equilibrium could be reached. No chemicals were used to suppress mould growth as there was a conceived risk that chemicals could interact with the food and affect the sorption properties. The relative humidity for each salt solution at a given temperature is presented in Table 5-1 (Greenspan, 1977). Each salt solution was created using deionised water, a suitable quantity of the relevant salt, and a high walled glass dish. A fume cupboard was used when transferring salt from their containers to the salt solutions.

Saturated salt solution	Water activity at 20°C	Water activity at 30°C
Potassium Hydroxide	0.093	0.074
Potassium Acetate	0.231	0.216
Magnesium Chloride	0.331	0.324
Potassium carbonate	0.432	0.432
Magnesium Nitrate	0.544	0.514
Sodium Chloride	0.755	0.751

Table 5-1 Saturated salt solutions used for sorption measurement(Greenspan, 1977)

Plastic dishes with lids were used as sample holders. Samples had to be removed from the container for mass measurement, and the lids were used to limit sorption interaction outside of the controlled relative humidity environment. Sample dishes and lids were labelled using engraving, as paper labels would sorb water and skew the results. Sample dishes were first cleaned and then dried using the dry nitrogen rig shown in Figure 5-4.

Each sample dish was used to hold approximately 4.3g of biscuit samples, or 2.2g of potato crisp samples. For adsorption, samples were initially dried to zero water activity by continually exposing the samples to dry nitrogen in a desiccator using the apparatus shown in Figure 5-4. Drying to zero water activity and moisture content was also used to quantify the moisture content of the packaged potato crisps and digestive biscuits. The dry nitrogen flow rate was 4 litres.min<sup>-1</sup> and drying was carried out at room temperature. For

desorption, samples were initially saturated by continual exposure to saturated nitrogen in a desiccator. The nitrogen was saturated using a drechsel bottle filled with deionised water, and passed over the sample at 4 litres.min<sup>-1</sup> at room temperature. The apparatus used to saturate the samples is shown in Figure 5-5.



Figure 5-4 Test rig used for obtaining zero water activity samples using dry nitrogen



Figure 5-5 Test rig used for obtaining saturated samples using saturated nitrogen

Four replicates were used for each sorption measurement. For a sorption measurement, two airtight sealed plastic containers, each containing two sample dishes, were used to equilibrate the food samples with salt solutions. The containers were put into incubators to maintain the desired temperature to  $\pm 1^{\circ}$ C. The dish lids were used when the samples were taken out of the container for mass measurement. This minimised interaction between the samples and the uncontrolled relative humidity of the lab. The mass of the

samples was measured once a week, although this was increased to twice a week when using Sodium Chloride saturated solution. Stability needed to be deduced sooner when using Sodium Chloride saturated solutions due to the risk of spoilage. For desorption, the samples were deemed to be in equilibrium when mass measurement indicated an increase in mass. Similarly for adsorption, the samples were deemed to be in equilibrium when the mass measurement indicated a decrease in mass. This reversal in moisture sorption trend was typically less than  $\pm 0.001$ kg.kg<sup>-1</sup> dry basis for both biscuits and potato crisps.

The measured sorption isotherms were fitted with sorption isotherm models using non-linear regression. The non-linear regressions were carried out using Minitab<sup>®</sup> 16 (Minitab Inc., USA). The fit was assessed using mean relative error (MRE) presented in Equation 5-1 (McMinn, et al., 2007; McLaughlin & Magee, 1998; Wang & Brennan, 1991)

$$MRE = \frac{100}{N} \sum \left| \frac{u_e - u_p}{u_e} \right|$$
Equation 5-1

Where  $u_e$  is the experimental value, and  $u_p$  is the value predicted by the model. It is generally understood that a good fit is obtained when MRE<10% (McMinn, et al., 2007; McLaughlin & Magee, 1998).

The GAB model has been found to exhibit a good description of sorption behaviour of almost all foods from a water activity of zero to 0.9 (Al-Muhtaseb, et al., 2002). However, this study is focused specifically on potato and biscuit sorption isotherms, so it was important to select relevant equations for curve fitting. A literature review resulted in the selection of sorption models presented in Table 5-2 that were used for curve fitting (Iglesias & Chirife, 1995; Iglesias & Chirife, 1976a; Ferro-Fontan, et al., 1982; Wang & Brennan, 1991; McLaughlin & Magee, 1998; Kaymak-Ertekin & Gedik, 2004; Iglesias & Chirife, 1978; Peleg, 1993; Lewicki, 1998; McMinn, et al., 2004; Lewicki, 2000; McMinn, et al., 2007; Palou, et al., 1997). It has been shown in leterature that the BET model is only effective at representing sorption isotherms at lower water activity ranges, and is typically utilised to curve fit sorption isotherms at

water activies less than 0.45 in order to define the monolayer moisture content	t
(McMinn, et al., 2007).	

Equation	Name of equation	Reference
$M_{db} = \frac{M_0 C a_w}{(1 - a_w)[1 + (C - 1)a_w]}$	BET equation	(Brunauer, et al., 1938)
$M_{db} = A \left[ \frac{a_w}{1 - a_w} \right]^B$	Oswin equation	(Oswin, 1946)
$M_{db} = \frac{M_0 C K a_w}{(1 - K a_w) [1 - K a_w + C K a_w]}$	GAB equation	(Guggenheim, 1966) (Anderson, 1946) (de Boer, 1953)
$M_{db} = \left[\frac{A}{\ln\left(\frac{\gamma}{a_w}\right)}\right]^{1/r}$	Ferro- Fontan	(Ferro-Fontan, et al., 1982)
$M_{db} = C_1 a_w^{n_1} + C_2 a_w^{n_2}$	Peleg equation	(Peleg, 1993)
$M_{db} = \frac{F}{(1 - a_w)^G} - \frac{F}{1 + a_w^H}$	Lewicki II equation	(Lewicki, 1998)

Table 5-2 Water activity equations used in the present study for fitting sorption isotherms

# 5.2.2 Calculation of isosteric heat of sorption

The net isosteric heat for potato crisps and biscuits was deduced using the measured sorption isotherms at 20°C and 30°C. The net isosteric heat,  $Q_n^{st}$ , was calculated using Equation 5-2 (Wang & Brennan, 1991), where  $T_1$  and  $T_2$  are the temperatures of two sorption isotherms, and  $A_{w1}$  and  $A_{w2}$  are the water activities of the two sorption isotherms at a given moisture content.

$$Q_n^{st} = R\left(\frac{T_1 T_2}{T_2 - T_1}\right) ln\left(\frac{A_{w2}}{A_{w1}}\right)$$
 Equation 5-2

The total heat of sorption,  $Q^{st}$ , was then calculated using Equation 5-3 (Wang & Brennan, 1991), where  $Q_n^{st}$  is the net isosteric heat, and  $L_r$  is the latent heat of vaporisation. The units for isosteric heat are Jkg<sup>-1</sup>.

$$Q^{st} = Q_n^{st} + L_r$$
 Equation 5-3

When calculating isosteric heat, it is important that fitted sorption isotherm models are not used to extend the range above or below the measured sorption isotherm data points as this can lead to inaccurate results.

The moisture content for commercially packaged potato crisps and biscuits was lower than the moisture content range of desorption isosteric heat that could be calculated from the experimental data. Hence, the range of isosteric heat was extrapolated using an empirical exponent correlation proposed by Tsami et al. (1990). The relationship is shown below, where  $Q_0$  and  $m_0$  are correlation parameters and  $m_{db}$  is the dry basis moisture content. This relationship was successfully used by McMinn & Magee (1999) who was able to plot predictive and experimental desorption behaviour for potato.

$$Q^{st} = Q_0 e^{\left(-m_{db}/m_0\right)} + L_r$$
 Equation 5-4

A non-linear regression fit was necessary to fit the predicted isosteric heat to the experimental results using fitting parameters  $Q_0$  and  $m_0$ .

For calculating the net isosteric heat for pasta, Equation 5-5 was used due to the availability of four sorption isotherms at 22°C, 30°C, 37°C and 45°C (Lagoudaki, et al., 1993).

$$-\frac{Q_n^{st}}{R} = \frac{\partial \ln a_w}{\partial 1/T}$$
 Equation 5-5

Using more than two sorption isotherms has been known to improve the accuracy of the predicted isosteric heat (Labuza, et al., 1985). The slope of  $\ln(a_w)/(1/T)$  was calculated for each moisture content point, which equates to  $-Q_n^{st}/R$ .

## 5.3 Sorption isotherm results

The moisture content of the packaged products were obtained when the biscuits and potato crisps were initially dried to zero water activity prior to the adsorption measurements. The moisture content of the packaged continuous fried potato crisps was measured to be 0.017kg.kg<sup>-1</sup> wet basis, while the moisture content of packaged digestive biscuits was measured to be 0.025kg.kg<sup>-1</sup> wet basis. The measured sorption isotherm results for continuous fried potato crisps and digestive biscuits are presented in Table 5-4 along with the standard deviation of the four samples measured for each data point. An adsorption-desorption hysteresis pattern was observed for each sorption isotherm as shown in Figure 5-6 and Figure 5-7 for potato crisps and digestive biscuits respectively, where the error bars represent the standard deviation of the 4 replicates. These are typical hysteresis loops that have closing points similar to the general hysteresis loop described by Labuza & Altunakar (2007). The sorption isotherms measured in this study demonstrate a hysteresis that occurs before the first measurement point at 0.09 water activity. There was less hysteresis observed in digestive biscuits than for potato crisps.

All the sorption isotherms measurements conform to a Type II sigmoidal Brunauer classification (Brunauer, et al., 1940). This classification is commonly found in carbohydrate foods (Iguedjtal, et al., 2008; Al-Muhtaseb, et al., 2002).

Water activity	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
_	Potato crisps ac	dsorption at 20°C	Potato crisps o	lesorption at 20°C	Digestive biscuits	adsorption at 20°C	Digestive biscuits	desorption at 20°C
0	0.000	0.0000	0.000	0.0000	0.000	0.0000	0.000	0.0000
0.093	0.025	0.0011	0.040	0.0018	0.027	0.0009	0.043	0.0001
0.231	0.037	0.0017	0.057	0.0033	0.042	0.0004	0.056	0.0002
0.331	0.045	0.0019	0.069	0.0043	0.051	0.0002	0.065	0.0004
0.432	0.056	0.0022	0.080	0.0048	0.064	0.0004	0.076	0.0006
0.544	0.071	0.0030	0.090	0.0050	0.081	0.0003	0.090	0.0005
0.755	0.128	0.0031	0.127	0.0037	0.138	0.0007	0.143	0.0010
	Potato crisps ac	dsorption at 30°C	Potato crisps o	lesorption at 30°C	Digestive biscuits	adsorption at 30°C	Digestive biscuits	desorption at 30°C
0	0.000	0.0000	0.000	0.0000	0.000	0.0000	0.000	0.0000
0.074	0.017	0.0010	0.029	0.0005	0.021	0.0005	0.033	0.0003
0.216	0.031	0.0017	0.049	0.0007	0.037	0.0004	0.049	0.0002
0.324	0.039	0.0019	0.060	0.0011	0.048	0.0002	0.058	0.0003
0.432	0.050	0.0022	0.070	0.0007	0.060	0.0016	0.068	0.0003
0.514	0.058	0.0025	0.077	0.0006	0.073	0.0011	0.081	0.0008
0.751	0.113	0.0034	0.117	0.0022	0.129	0.0075	0.134	0.0064

## Moisture content dry basis [kg.kg-1]

 Table 5-3 Sorption isotherm measurement results



Figure 5-6 Measurements of sorption isotherms, showing hysteresis for potato crisps at (a)  $20^{\circ}$ C and (b)  $30^{\circ}$ C



Figure 5-7 Sorption isotherm measurements showing hysteresis for digestive biscuits at (a)  $20^{\circ}$ C and (b)  $30^{\circ}$ C

The Mean Relative Error (MRE) of fitted water activity models is presented in Table 5-4 and Table 5-5 for potato crisps and digestive biscuits respectively, along with the fitting parameters obtained through non-linear regression. The BET model is only used to describe a reduced water activity range up to 0.45. It is generally thought to be inaccurate at describing sorption at water activities above 0.45, but is used up to a water activity of 0.45 to define the monolayer.

		20degC	30degC	20degC	30degC
		Potato	Potato	Potato	Potato
		crisp	crisp	crisp	crisp
Model	Parameter	adsorption	adsorption	desorption	desorption
BET	С	20.3108	13.225	31.9575	22.2231
(a <sub>w</sub> = 0-0.45)	M <sub>0</sub>	0.0336	0.0308	0.0478	0.0432
(	MRE [%]	1.47	0.76	1.21	3.33
	С	18.1522	13.3193	23.6289	19.2596
GAB	К	0.9703	0.9731	0.7352	0.765
(aw = 0-0.76)	M <sub>0</sub>	0.035	0.0313	0.0582	0.0514
	MRE [%]	1.62	0.72	1.32	1.14
	$\gamma$	1.00489	1.07071	1.53859	1.67925
Ferro-Fontan	α	0.01928	0.03058	0.05699	0.08729
(aw = 0-0.76)	R	1.31165	1.12405	1.21959	1.0307
	MRE [%]	0.23	2.90	1.51	3.51
Ocwin	А	0.067993	0.059441	0.086227	0.077083
Oswin (aw = 0-0.76)	В	0.54683	0.570521	0.339103	0.371588
(uw = 0 0.70)	MRE [%]	6.73	5.93	1.03	2.19
	$b_1$	6.5586	7.0943	10.6973	10.509
Peleg	b <sub>2</sub>	19.6909	19.8806	10.0537	21.6687
(aw = 0-0.76)	n <sub>1</sub>	0.402	0.5464	0.4201	0.4975
	n <sub>2</sub>	3.7139	4.6547	4.1345	7.4911
	MRE [%]	0.66	0.80	1.23	0.36
	F	0.061279	0.055474	0.136909	0.120095
Lewicki II	G	0.681248	0.676904	0.265895	0.291812
(aw = 0-0.76)	Н	0.266902	0.383583	0.429487	0.476488
	MRE [%]	0.21	1.78	1.02	1.81

#### Table 5-4 Optimised equation fits for potato crisp sorption isotherms

The monolayer moisture content is always predicted to be lower at higher temperature. Water molecules interact with hydrophilic components of the product, such as carbohydrates and proteins. This interaction occurs through hydrogen bonds, causing an exothermic reaction, which is not favoured with increasing temperature (Doporto, et al., 2012). It was found that monolayer moisture content is always higher for desorption than for adsorption due to hysteresis. The monolayer moisture content of potato crisps ranged from 0.031kg.kg<sup>-1</sup> to 0.048kg.kg<sup>-1</sup> dry basis. The packaged moisture content of potato crisps was measured to be 0.018kg.kg<sup>-1</sup> dry basis. The monolayer moisture content of digestive biscuits ranged from 0.038kg.kg<sup>-1</sup> to 0.044kg.kg<sup>-1</sup> dry basis. The packaged moisture content of digestive biscuits ranged from 0.038kg.kg<sup>-1</sup> to 0.044kg.kg<sup>-1</sup> dry basis. The packaged moisture content of digestive biscuits was measured to be 0.026kg.kg<sup>-1</sup> dry basis. Hence both potato crisps and digestive biscuits have packaged moisture contents that are significantly lower than their monolayer moisture contents, even at 30°C. Foods are very stable below the monolayer moisture content which results in a long shelf life.

		20degC Digestive biscuit	30degC Digestive biscuit	20degC Digestive biscuit	30degC Digestive biscuit
Model	Parameter	adsorption	adsorption	desorption	desorption
DET	С	15.7223	12.6301	72.4171	39.6716
BET (a <sub>w</sub> = 0-0.45)	$M_0$	0.0391	0.0378	0.0443113	0.0408806
(u <sub>w</sub> 0 0.13)	Error [%]	1.46	0.21	1.33	2.23
	С	13.3583	10.8696	48.2369	31.3683
GAB	К	0.929	0.9197	0.882155	0.896504
(aw = 0-0.76)	$M_0$	0.0425	0.0417	0.0482715	0.0446223
	Error [%]	2.02	1.59	0.20	0.67
	$\gamma$	1.1419	1.29661	0.956637	1.0213
Ferro-Fontan	А	0.04547	0.0814	0.00587469	0.0133703
(aw = 0-0.76)	r	1.11364	0.93019	1.90101	1.56223
	Error [%]	0.87	1.97	0.36	1.7
Oswin	А	0.075766	0.071566	0.0886213	0.0816817
(aw = 0-0.76)	В	0.522224	0.530653	0.402023	0.432197
(uw = 0 0.70)	Error [%]	4.33	2.97	5.88	5.94
	b1	7.6586	8.17	8.64516	8.30713
Peleg	b <sub>2</sub>	18.0838	16.4259	18.1811	17.4192
(aw = 0-0.76)	n <sub>1</sub>	0.4401	0.5277	0.299267	0.35392
	n <sub>2</sub>	3.3779	3.5712	3.73748	3.75545
	Error [%]	0.61	0.47	0.28	0.65
	F	0.07942	0.079544	0.0970944	0.0903996
Lewicki II	G	0.580859	0.554375	0.487294	0.500263
(aw = 0-0.76)	Н	0.400452	0.494209	0.180281	0.268248
	Error [%]	0.85	0.79	0.65	1.4

Table 5-5 Optimised equation fits for Digestive biscuit sorption isotherms

In every instance, the monolayer predicted by the GAB model was higher than that predicted by the BET model. The highest discrepancy in monolayer prediction between BET and GAB occurs in potato crisp desorption at 20°C. The GAB model predicts a monolayer of 0.0582kg.kg<sup>-1</sup> dry basis while the BET model predicts a monolayer of 0.0478kg.kg<sup>-1</sup> dry basis. This constitutes a 0.01kg.kg<sup>-1</sup> discrepancy, which is illustrated in Figure 5-8. The BET model is based on monolayer theory, and is used specifically to predict the monolayer moisture content. The GAB equation is based on multilayer theory, and is used to provide good fits to sorption isotherms for the entire water activity range. It has been noted by McMinn et al. (2007), Palou et al. (1997), and Erbas et al. (2005) that the GAB equation predicts higher monolayer moisture content than the BET equation. If the transition between monolayer and multilayer is defined as the first point of inflection in the sigmoid type II isotherm (Labuza, 1975), then Figure 5-8 shows that the BET model clearly provides a better monolayer prediction.



Figure 5-8 Monolayer moisture content predictions of BET and GAB for measured desorption of potato crisps at 20°C

The best fits for each sorption isotherm is summarised in Table 5-6. Out of the best fits, the worst sorption isotherm fit was 1.02%, where an MRE less than

10% is considered to be a good fit (McMinn, et al., 2007; McLaughlin & Magee, 1998). Hence the models that provided the lowest Mean Relative Error all provided excellent fits to the experimental data. The Peleg equation provided the best fit to 4 sorption isotherms, while the Lewiki II equation provided the best fit to 2 sorption isotherms and the GAB model provided the best fit to 2 sorption isotherms. The Peleg and Lewiki II equations are purely empirical equations, whilst the GAB equation is a derived kinematic model. The fitting results in Table 5-6 are broadly in line with published data. The GAB equation is generally considered to be the most versatile kinematic model in literature and was recommended by the European COST 90 project (Al-Muhtaseb, et al., 2002; Iguedital, et al., 2008). Lewicki (1998) fitted the GAB, Peleg, as well as his own proposed Lewicki II model to sorption isotherms of 31 food products including potato flakes and potato starch. It was found that for fits with a root mean square less than 10%, the GAB equation described less than 35.5%, Lewicki II model described 71.0%, and the Peleg model described 77.4% of the isotherms. Table 5-7 shows that the best average fit for the eight sorption isotherms measured in this study was the empirical Peleg equation, which had an average MRE of just 0.41%, followed by the empirical Lewicki II equation, followed by the kinematic GAB equation.

Sorption Isotherm	Best fit model	Best fit error [%]
20degC potato crisp adsorption	Lewicki II	0.21
30degC potato crisp adsorption	GAB	0.72
20degC potato crisp desorption	Lewicki II	1.02
30degC potato crisp desorption	Peleg	0.36
20degC digestive biscuit adsorption	Peleg	0.61
30degC digestive biscuit adsorption	Peleg	0.47
20degC digestive biscuit desorption	GAB	0.20
30degC digestive biscuit desorption	Peleg	0.65

 Table 5-6 Best fit equations for sorption isotherms measured

Water activity model	Water activity range	Average fit [%]
BET	0 to 0.45	2.86
GAB	0 to 0.76	0.89
Ferro-Fontan	0 to 0.76	0.98
Oswin	0 to 0.76	3.85
Peleg	0 to 0.76	0.41
Lewicki II	0 to 0.76	0.73

Table 5-7 Average fit of digestive biscuit and potato crisp adsorption and desorption

The temperature dependence of the sorption isotherm experimental data points is shown in Figure 5-9 and Figure 5-10, complete with error bars representing the standard deviation of the 4 replicates. The curves of the best fitted equations are also shown in these figures. It can be seen in Figure 5-9 and Figure 5-10 that the curves are an excellent fit for the experimental data points.



Figure 5-9 (a) Adsorption and (b) desorption of measured isotherms of potato crisps at  $20^{\circ}C$  and  $30^{\circ}C$ 



Figure 5-10 (a) Adsorption and (b) desorption of measured isotherms of digestive biscuits at  $20^{\circ}$ C and  $30^{\circ}$ C

Figure 5-11 compares the adsorption of digestive biscuits at 20°C to adsorption data taken from literature for biscuits and cookies. The measurements for digestive biscuits are mid-range, hence digestive biscuits can be considered to have a fairly typical hygroscopy for a biscuit/cookie. The digestive biscuits are

generally less hygroscopic than the cookies measured by Palou et al. (1997), especially considering these were measured at 25°C rather than 20°C. The digestive biscuits were more hygroscopic than the laminated cookie measured by Guillard et al. (2004), and more hygroscopic than the oatmeal biscuit measured by McMinn et al. (2007).



Figure 5-11 Digestive biscuit adsorption at 20°C measured in the present study compared to literature adsorption data at 20°C for cookies and biscuits

A best fit of literature values for desorption of uncooked potato at 30°C was found through non-linear regression using Minitab<sup>®</sup> 16 (Minitab Inc., USA). The literature values used were taken from Kaymak-Ertekin & Gedik (2004), Kiranoudis et al. (1993), McLaughlin & Magee (1998), McMinn & Magee (1999), and Zhang et al. (1996). The best fit was found using the Peleg equation which returned an MRE of 7.5%. The Peleg equation parameters used

to obtain this best fit for uncooked potato was:  $b_1 = 15.8463$ ,  $b_2 = 32.3003$ ,  $n_1 = 0.4654$ , and  $n_2 = 4.5318$ . The best fit for uncooked potato is compared to potato crisps in Figure 5-12, were both curves are desorption isotherms at 30°C. Standard deviation error bars are included for the Kiranoudis et al. (1993) measurement data points of uncooked potato, and for the data points of potato crisp desorption measured in this study.





It can be seen from Figure 5-12 that the potato crisps appear to be considerably less hygroscopic than uncooked potato. However, this may be due to the presence of oil in potato crisps, which is hydrophobic (Vácha, et al., 2011). Hence the oil is contributing to the mass, but not contributing to the hygroscopy of the potato crisps. The equation for dry basis moisture content is shown in Equation 5-6, where  $m_w$  is the mass of the water, and  $m_d$  is the mass of the dry matter.

$$M_{db} = \frac{m_w}{m_d}$$
 Equation 5-6

For raw potato, the dry mass is simply the dry potato mass. For potato crisps, the dry mass is the dry potato mass and the oil mass. The mass of potato crisps is made up of solid potato, oil which is 0.323kg.kg<sup>-1</sup> as stated on the potato crisp packaging, and water which was gravimetrically measured to be 0.173kg.kg<sup>-1</sup> using constant exposure to dry nitrogen. Hence, if the mass fraction of oil is 0.323kg.kg<sup>-1</sup> and the mass fraction for water is 0.0173kg.kg<sup>-1</sup> for finished potato crisps, then the mass fraction of solid potato is 1-0.323-0.0173 = 0.66kg.kg<sup>-1</sup>. Hence for 1kg of potato crisps  $1/m_d = 1/(0.66+0.323) =$  $1.017 \text{kg}^{-1}$ . If there was no oil, then  $1/m_d = 1/0.66 = 1.515 \text{kg}^{-1}$ . The ratio between these factors for potato crisps without oil and potato crisps with oil is 1.515/1.017 = 1.49. Hence the dry basis moisture content measured for potato crisps can be converted into an equivalent moisture content of potato by multiplying by a factor of 1.49, which factors out the oil. This ratio is calculated using the solid potato mass and the oil mass, hence the ratio does not change with respect to water content. This ratio was applied to the potato crisp desorption isotherm as shown in Figure 5-13 to factor out the oil content and make a direct comparison with raw potato sorption isotherm. It can be seen from Figure 5-13 that the adjusted desorption isotherm for potato crisps is very similar to the desorption isotherms measured for raw potato as presented in literature. The highest water activity point measured for potato crisps shows slightly lower moisture content than the uncooked potato measurements. This may be due to the gelatinisation of starch, potato type, or sorption hysteresis. The gelatinization temperature of potato starch was measured to be 61.1°C by Shiotsubo (1984). Wang & Brennan (1991) measured sorption isotherms for potato from 40°C to 70°C as shown in Figure 5-14. The Wang & Brennan (1991) data shows no significant change in sorption trend after the gelatinization temperature of 61°C. The spread in sorption isotherm measurements shown in Figure 5-13 at least in part demonstrates the different sorption behaviour of different types of potato. For instance Kaymak-Ertekin & Gedik (2004) measured the sorption of Ödemiş potatoes from Bozdağ Turkey, whereas McLaughlin & Magee (1998) measured the sorption of Pentland Dell potatoes. Hygroscopy of potato may also be affected by other variables such as climate (temperature, precipitation e.t.c.) and soil quality. The uncooked potato desorption measurements was carried out on raw potato that had no previous sorption history. The potato crisps however were initially dehydrated through frying, they were then re-hydrated close to saturation, and then desorption measurements were carried out. Sorption hysteresis is affected by sorption history, including the speed and frequency at which the sorption hysteresis loop is traversed (Ngoddy & Bakker-Arkema, 1975).



Figure 5-13 Potato crisp desorption at  $30^{\circ}$ C measured in the present study with values adjusted for oil content, compared to literature desorption data at  $30^{\circ}$ C for uncooked potato



Figure 5-14 Adsorption of uncooked potato from 40°C to 70°C (Wang & Brennan, 1991)

## 5.4 Isosteric heat results

Knowledge of the isosteric heat of sorption is of great importance when designing equipment for dehydration purposes. The heat of sorption is considered indicative of the intermolecular attractive forces between sorption sites and water vapour (Al-Muhtaseb, et al., 2002). This energy must be taken into consideration in addition to latent energy when assessing the total amount of energy required for a drying process.

The calculated isosteric heat of sorption is presented in Figure 5-15 and Figure 5-16 for potato crisps and biscuits respectively. These results show that biscuits and potato crisps exhibit similar levels of isosteric heat. They both show a large difference in desorption and adsorption, where desorption requires more energy than adsorption, this behaviour has been observed in a large number of food sorption studies (Wang & Brennan, 1991; Rizvi, 1995; McMinn & Magee, 1999; Kaymak-Ertekin & Gedik, 2004; McLaughlin & Magee, 1998).



Figure 5-15 Total isosteric heat of desorption and adsorption for potato crisps



Figure 5-16 Total isosteric heat of desorption and adsorption for biscuits

Figure 5-17 and Figure 5-18 compares the sorption properties of potato crisps to the sorption properties of raw potato measured by Wang & Brennan (1991). It can be seen from the heat of adsorption behaviour shown in Figure 5-17 that the isosteric heat for ptoato crisps and raw potato is practically the same, whilst there is an approximate offset of 0.01kg.kg<sup>-1</sup> wet basis between potato crisps and raw potato for the heat of desorption shown in Figure 5-18. Figure 5-17 shows that the cooking process has a negligable affect on the hygroscopy of the ptotato parenchyma cells, inside which sorption takes place (Bordoloi, et al., 2012; Costa, et al., 2001).



Figure 5-17 Total isosteric heat of adsorption for potato crisps and raw potato



Figure 5-18 Total isosteric heat of desorption for potato crisps and raw potato

The final moisture content of potato crisps has been measured to be 0.017kg.kg<sup>-1</sup> wet basis in the present study. It is necessary to know the isosteric heat at this water content to deduce the energy requirements for drying. However, the isosteric heat deduced using the experimental data only extends down to a moisture content of 0.038kg.kg<sup>-1</sup>. Using the exponential relationship proposed by Tsami et al. (1990) the desorption plot for isosteric heat can be extended as shown in Figure 5-19. The model predicts a total heat of sorption of 5.16x10<sup>6</sup>Jkg<sup>-1</sup> at a moisture content of 0.017kg.kg<sup>-1</sup>, where 2.44Jkg<sup>-1</sup> of the total is made up of the latent heat of vaporisation. Hence the total isosteric heat of vaporisation. This highlights how important it is to take isosteric heat into account when carrying out drying calculations, and it indicates how energy intensive it is to achieve such low moisture contents in potato. By defining the total amount of energy required for drying, the isosteric heat curve can be used to assess the economic viability of a proposed RF/MW drying process.



Figure 5-19 Total isosteric heat of desorption for potato crisps – measured and predictive

The final moisture content of packaged biscuits was measured to be 0.025kg.kg<sup>-1</sup>, but the heat of desorption was only deduced down to a moisture content of 0.043kg.kg<sup>-1</sup> dry basis. Hence, the same approach was taken for biscuits as was taken for potato crisps, where a non-linear regression of the experimental desorption data was carried out using the model proposed by Tsami et al. (1990). The resultant plot for the heat of desorption for biscuits is shown in Figure 5-20. The heat of desorption for biscuits at the packaged final moisture content of 0.025kg.kg<sup>-1</sup> is  $6.7x10^6$ Jkg<sup>-1</sup>, where  $2.44x10^6$ Jkg<sup>-1</sup> is accounted for by the latent heat of vaporisation. Hence the total heat of sorption at a moisture content of 0.025kg.kg<sup>-1</sup> is 2.7 times higher than the latent heat of vaporisation.



Figure 5-20 Total isosteric heat of desorption for biscuits – measured and predictive

The sorption behaviour of pasta shown in Figure 5-21 is markedly different from potato crisps and biscuits. At a moisture content of 0.025kg.kg<sup>-1</sup> pasta has roughly half the total heat of sorption compared to the heat of desorption predicted for biscuits. However the energy of sorption at moisture contents above 0.07kg.kg<sup>-1</sup> wet basis are higher than that observed in biscuits or potato crisps. Pasta drying occurs from 0.3kg.kg<sup>-1</sup> down to 0.12-0.13kg.kg<sup>-1</sup> wet basis (Schiffmann, 2001a; Marchylo & Dexter, 2001), and it can be seen that the energy of sorption over this range is significant despite the final moisture content of 0.12-0.13kg.kg<sup>-1</sup> wet basis being higher than the monolayer moisture content of 0.066kg.kg<sup>-1</sup> wet basis.


Figure 5-21 Total isosteric heat of sorption for pasta

### 5.5 Conclusion

This chapter quantifies sorption isotherms, final moisture contents, and isosteric heats of sorption for potato crisps, biscuits and pasta. The isosteric heat of potato crisps, biscuits and pasta have been calculated and presented, showing that the isosteric heat is extremely significant at low moisture contents when compared to the latent heat of vaporisation. For instance, this study showed that the total heat of desorption for biscuits is 2.7 times higher than the latent heat of vaporisation at a moisture content of 0.025kg.kg<sup>-1</sup> wet basis. The isosteric heat quantified in this chapter is used in the subsequent chapter to calculate the total energy required for drying. This enables an initial commercial assessment to be made of the viability of proposed microwave processes. The points of inflection of the sorption isotherms generated in this chapter are compared to the relevant moisture dependant dielectric properties in the subsequent chapter.

Sorption isotherms for pasta have already been measured by Lagoudaki et al. (1993), so no sorption isotherm measurements of pasta was required in the

present study. However, the isosteric heat of pasta is not presented in literature, and was calculated in the present study using the sorption isotherms measured at 22°C, 30°C, 37°C and 45°C by Lagoudaki et al. (1993). No published sorption isotherm data was available for potato crisps. Biscuit sorption isotherm data was available in literature, but it was found to have significant variance depending on the type of biscuit, and the biscuits measured in literature could not be obtained for dielectric measurement in the present study. Adsorption and desorption isotherms were measured for unsalted continuous fried potato crisps and digestive biscuits at 20°C and 30°C. The sorption data at 20°C is required for comparison to measured dielectric properties, and the sorption data at 30°C provides a second data set necessary for calculating the isosteric heat of sorption. Sorption behaviour of potato crisps and biscuits could not be measured gravimetrically at temperatures higher than 30°C due to the mass stability of oil. It was found that sunseed oil gained approximately 4% mass due to oxidation of oil when the temperature was raised to  $40^{\circ}$ C, which would have introduced too much error into the gravimetric sorption isotherm measurements.

The method used for measuring the sorption isotherms was novel. Drying to deduce the moisture content was carried out using continuous exposure to dry nitrogen. This was necessary as oven drying would have affected the large oil mass, skewing the gravimetric measurement of the moisture content. Saturation was carried out by passing a dry nitrogen continuously through a dreschel bottle, and then to the food. The oxygen free atmosphere supressed mould growth without the need for any hazardous chemicals.

Sorption isotherm models were fitted to the sorption data for potato crisps and digestive biscuits. The GAB, Lewicki II and Peleg equation provided the best fits for the sorption isotherms, with the Peleg equation providing the best average fit, with an average Mean Relative Error (MRE) of just 0.41%. Out of the sorption isotherms measured, the best fit equations produced a worst case MRE fit of 1.02%, where MRE below 10% are considered to be good fits.

The sorption isotherms measured for digestive biscuits had approximately midrange hygroscopy compared to biscuit sorption data published in literature. The hygroscopy of potato crisps were considerably lower than uncooked potato. This was due to the presence of oil which contributes to the mass, but is not hygroscopic. Factoring out the oil mass, the 30°C desorption isotherm for potato crisps agreed well with uncooked potato presented in literature. This shows that the gelatinisation of starch that occurs during frying has a negligible effect on sorption behaviour at low moisture contents, and the isosteric heat of the potato. This is an important discovery as the monolayer and the isosteric heat are important properties affecting drying, product stability, and shelf life.

The BET monolayer moisture content was calculated for potato crisps, biscuits and pasta by non-linear regression fitting of the BET model to the relevant sorption isotherms. The sorption monolayer of pasta was found to be 0.066kg.kg<sup>-1</sup> wet basis, with literature citing the final moisture content of packaged pasta as ranging from 0.12 to 0.13kg.kg<sup>-1</sup> wet basis. The adsorption monolayer moisture content of potato crisps was found to be 0.033kg.kg<sup>-1</sup> wet basis at 20°C, with the final moisture content of packaged potato crisps being measured as 0.017kg.kg<sup>-1</sup> wet basis. The adsorption monolayer of digestive biscuits was calculated to be 0.038kg.kg<sup>-1</sup> wet basis with the final moisture content of packaged potato crisps being measured as 0.017kg.kg<sup>-1</sup> wet basis. The adsorption monolayer of digestive biscuits was calculated to be 0.038kg.kg<sup>-1</sup> wet basis with the final moisture content of packaged digestive biscuits measured to be 0.025kg.kg<sup>-1</sup> wet basis.

### Chapter 6 - Dielectric measurements and assessment of microwave applications

### 6.1 Introduction

This chapter presents dielectric measurements of the identified MW drying applications, which are consequently used to assess moisture levelling with respect to variation in electric field exposure. The moisture dependent dielectric properties are curve fitted using the adapted water activity equations presented in Chapter 3. This enables use of the measured moisture dependent dielectrics in future microwave modelling activities. The commercial feasibility of the MW applications identified is also discussed, using the isosteric heats determined in Chapter 5.

Dielectric drying is typically carried out at ISM frequencies of 13.56MHz, 27.12MHz, 40.68MHz, 896MHz, 915MHz, and 2.45GHz (Metaxas & Meredith, 1993). ISM frequencies are reserved for industrial processing, and high power vacuum electron tubes are commercially available at these frequencies. Measurement of dielectric properties, and consequent calculations carried out in this chapter, were carried out at a microwave frequency of 915MHz. This American ISM frequency was preferred to the UK frequency of 896MHz as the food industry is much bigger in America, for instance the salty snack industry is worth \$28 billion in north America while it is only worth approximately \$4 billion in the UK (Nielson N.V., 2014; SNACMA, 2016). Finish drying of potato crisps, previously used to prevent browning of high sugar potatoes during drying, has been successfully carried out at 915MHz. There used to be 100 of these driers in operation, hence there is a lot of heritage using 915MHz (Schiffmann, 2001a). Production of low fat potato crisps using microwave explosive dehydration also has heritage of being carried out at 915MHz (Gaon & Wiedersatz, 1993). Pasta drying is carried out at a frequency of 915MHz, using large multimode resonant cavities (Schiffmann, 2001a). Microwave frequencies are less prone to arcing issues compared to RF frequencies. A higher electric field is generally required to

achieve a given power density for RF frequencies compared to microwave frequencies (Rowley A.T., 2001). Arcing is a well-documented issue for some of these applications, including finish drying of potato crisps and biscuits (Schiffmann, 2001a; Clark, 1997). The frequency of 915MHz is more practical than 2.45GHz for high power industrial scale drying due to its longer wavelength and larger depth of penetration, which facilitates treatment of larger throughputs (Schiffmann, 2001a). Although nearly all biscuit drying is carried out at RF frequencies, Schiffmann (2001a) states that MW finish drying is just as effective. The reason RF is used over MW is that the conveyors tend to be one or more meters wide, and uniform drying is easier to achieve over this width with RF. Dielectric properties of biscuits are measured at 915MHz in the present study due to the availability of dielectric measurement equipment. Dielectric measurements of potato crisps, digestive biscuits, and pasta particulate are presented in this chapter, where measurements were taken at a temperature of 20°C.

The selective heating of water is cited as a key benefit for microwave drying as it leads to moisture levelling (Schiffmann, 2006; Metaxas & Meredith, 1993). The moisture levelling mechanism is used to improve uniformity of the final moisture content (Baker, 1997). Moisture levelling occurs in instances were drier food heats less readily than wetter food in the applicator due to their dielectric properties, leading to wetter food receiving more heating. The additional heating experienced by wetter food in the microwave applicator increases the rate of moisture removal of wetter food which reduces the moisture variation of the final product. The moisture levelling mechanism is dependent on the dielectric properties, as wetter food must experience comparatively more dielectric heating in a given electric field for it to take effect. Moisture variation of the final product can occur due to uneven electric field exposure of food travelling through the microwave applicator. Selective heating of moisture due to favourable dielectric properties can help counteract any unevenness in electric field exposure. Moisture levelling can also reduce burning as well as limiting moisture variation, as very dry food receives comparatively less microwave heating for a given electric field. The moisture levelling mechanism is analysed in this chapter using the relevant moisture dependent properties for the four proposed microwave applications.

The moisture dependence of the loss factor is likely to be governed by the state of the water, as water mobility is necessary for the microwave loss mechanisms. When studying the moisture dependence of the loss factor, a major point of inflection called the "loss factor critical moisture content" is typically observed, below which the moisture is considered to be "bound", and above which the moisture is "free" (Metaxas & Meredith, 1993). The measured loss factor for potato crisps, biscuits, and pasta is compared to the state of the water as defined by the sorption isotherm. The sorption isotherm is used to define the transition from monolayer to multilayer, and the transition from multilayer to solution and their relationship with the loss factor critical moisture content is of interest. The sorption isotherms for potato crisps and biscuits were measured while the sorption isotherm for pasta was taken from literature as presented in Chapter 5.

The moisture levelling mechanism is assessed for each food by calculating the additional moisture required to obtain 10% additional heating when receiving 10% lower intensity electric field exposure. This provides some indication as to the ability of the moisture levelling mechanism to counteract uneven electric field exposure. The loss of the dielectric load is also of interest in its own right. The loss factor of foods below the critical moisture content can be deemed "microwave transparent" (Metaxas & Meredith, 1993). Low loss dielectric loads are known to be problematic with respect to microwave drying (Clark & Sutton, 1996; Ku, et al., 2001; Schiffmann, 2006).

The quality of the product dried using the proposed microwave applications is discussed. Food quality is a vital consideration for any food producer. Any microwave application designed to replace a conventional drying process should not adversely affect product characteristics, as any change in taste or quality may risk customer acceptance and future product sales. Some microwave applications may be introduced solely because they improve product quality, such as the reduction in acrylamide in potato crisps, or the reduction of "checking" (fracture) in biscuits.

The economics of pasta drying and potato crisps drying is also discussed in this chapter. This includes a detailed analysis of the cost of energy for the new proposed applications, which is the explosive dehydration of potato crisps to control fat content and reduce carbon footprint, and the finish drying of potato crisps to reduce carcinogenic acrylamide levels.

### 6.2 Method - Waveguide dielectric measurements

When selecting the most appropriate dielectric measurement technique, it was important to keep the structure of the food intact. A measurement of food packed as it would be in a microwave applicator is most relevant as this enables the loss of the dielectric load to be directly assessed. Hence it was decided that cavity perturbation would not be a suitable measurement technique as it requires the food to be crushed in order to obtain repeatable results. The open ended coaxial line technique was also deemed unsuitable as small spaces between the food and the probe can greatly affect the results and the small volume measured could lead to great variability in results and a lack of repeatability. Hence, the waveguide technique was deemed ideal, as this could give the dielectric response of a realistic load for industrial processing, with the food structure fully intact. The dielectric loss of the actual material will be far higher due to the amount of air present in the packed material. The loss of the dielectric load is important for assessing the ease at which the dielectric load can be heated. Quantification of the dielectric load is important for applicator design, as it can drive design decisions. For instance highly conductive applicator walls, microwave transparent conveyor belt, and a sensitive matching system may be required for a low loss dielectric load. A measurement of the dielectric load seen by the microwave applicator is therefore a more appropriate measurement than that of the food itself. The waveguide technique is particularly useful for low loss particulate material.

The complex permittivity of material can be measured in a waveguide using a vector network analyser (Gregory & Clarke, 2012). In this case, two waveguide ports and a load cell was used to carry out dielectric measurements of food using an Agilent E5071C Vector Network Analyser (VNA), cables with N type connectors, and Agilent Materials Measurement Software 85071E on a laptop. The laptop was connected to the VNA via a GPIB to USB cable. Dielectric measurements of potato crisps, digestive biscuits, and of pasta were carried out using the waveguide technique.

The waveguide dimensions were 247mm by 123mm and the waveguide assembly schematic is shown in Figure 6-1. The load cell includes a polycarbonate sheet fitted into a recess on the bottom of the load cell, which allows it to be packed with material. Silicone sealant was used to create a water tight seal in the load cell so that reference fluids could be measured to check the calibration. The frequency range measured using the waveguide was 850MHz to 950MHz. The load cell was bolted together using six evenly spaced bolts, and were tightened to a pre-torque of 20Nm using a torque wrench.

A two port calibration using three shorts and a through (SSST) was used. The three shorts had lengths of 55.3mm, 165.3mm, and 0mm (flush). Trigger measurements were carried out using the "Transmission Epsilon Fast" model, which was recommended by Agilent. The power selected for both calibration and trigger measurements was 5dBm. This is reasonably high, giving a powerful response to measure, whilst being below the threshold at which compression may start to occur (10dBm). Compression occurs when the output power becomes non-linear with respect to the input power, and a drop in gain is observed.

In order to verify the validity of the SSST calibration, a material with known dielectric properties was measured. Ethanol was selected as it is a good reference material of known dielectric response, where its dielectric properties have been characterised by the National Physical Laboratory in a report written by Gregory & Clarke (2012). Ethanol is a pure substance that can occupy a true

measured volume (unlike particulates). The volume of ethanol required was hazardous, so a room with a large air extraction system was used to take the measurement due to ethanol's high evaporation rate. The SSST calibration was carried out in a laboratory that had sufficient space, and was then moved to the small room with air extraction for the dielectric measurement of ethanol. This meant switching the VNA off and on, and disconnecting/reconnecting the cables etc. This process could potentially affect the accuracy of the measurement, hence the empty load cell was measured before and after the move to check for measurement drift. The loss factor of the empty cell structure was measured to be 5.49e-6 before moving, and was measured to be -0.00162 after moving. The dielectric constant was measured to be 1.000371 before moving, and was measured to be 0.997877 after moving. Considering the calibrated loss factor should have read zero, and the dielectric constant should have been one, the level of drift was very low, especially considering the reference material to be measured, Ethanol, has a dielectric constant of 13.8 and a loss factor of 10.3 at 915MHz, 20°C (Gregory & Clarke, 2012). Hence the empty load cell measurements showed an acceptable amount of error.



Figure 6-1 Waveguide dielectric measurement setup

It was found that the cell structure could not be completely filled to the brim with ethanol without spillage occurring when bolting on the waveguide. The depth of the load cell is 96mm, and it was found that the ethanol level would decrease to 92.6mm as a result of fitting and removing the studs. Hence, the cell structure was filled to approximately 86mm. The depth from the surface to the top cell structure flange was measured using a depth micrometer. The depth of material to be measured could be entered into the Agilent software. Micrometer measurements showed that the distance between the cell flange and the ethanol would stay consistent before and after torqueing the studs. There was a small amount of inaccuracy in depth measurement due to some ripples generated by the extraction system in the spray booth. However, measurements taken before and after the dielectric measurement would suggest that this inaccuracy is likely to be approximately +/- 0.1mm. This is likely to contribute to the amount of error observed in repeatability.

Three measurements of ethanol were taken. In each case the ethanol was transferred into the load cell, the studs fitted, the measurement taken, then the waveguide was dissembled and the ethanol transferred out again. Hence the error bars generated capture variably of the whole measurement setup process. The average temperature of the dielectric measurements was 22°C, which is compared to best-fit double-Debye data for ethanol at 22.5°C as presented by Sato et al. (1999) in Figure 6-2 and Figure 6-3. The comparison of the dielectric constant is shown in Figure 6-2, where the measured values are 2.28% below the best fit double-Debye data presented by Sato et al. (1999) on average. The measured values of loss factor presented in Figure 6-3 are 1.96% below the best fit double-Debye data presented by Sato et al. (1999) on average. Considering the wavegue measurement was not made in a temperature controlled room, and the depth was difficult to measure due to ripples in the fluid caused by the extraction system, the measured result shows acceptable accuracy compared with published data. As well as instabilities in room temperature, the temperature of the top surface of the ethanol in the waveguide load cell was lower than ambient due to its high rate of evaporation, while the bulk of the fluid would have been closer to room temperature.



Figure 6-2 Dielectric constant of Ethanol measured in waveguide compared to Sato et al. (1999) data



Figure 6-3 Loss factor of Ethanol measured in waveguide compared to Sato et al. (1999) data

McVities Digestive biscuits and unsalted Smith's (Walkers) continuous fried crisps were purchased from a local supermarket for dielectric measurement. These samples were also used for sorption isotherm measurement in Chapter 5 and the porosity of unsalted Smith's (Walkers) continuous fried crisps was also quantified using a novel Micro-CT method in Chapter 4. Napolina penne pasta was also purchased from a local supermarket for dielectric measurement. The sorption isotherms measured by Lagoudaki et al. (1993) is applicable to this

Food constituent	Biscuit content	Potato crisps	Pasta content
	[kg.kg <sup>-1</sup> ]	content [kg.kg <sup>-1</sup> ]	[kg.kg <sup>-1</sup> ]
Total fat	0.213	0.323	$1.5 \times 10^{-4}$
Saturated fat	0.101	0.026	3.0x10 <sup>-5</sup>
Total carbohydrates	0.629	0.522	0.72
Sugar carbohydrates	0.166	0.004	0.03
Fibre	0.036	0.044	0.030
Protein	0.072	0.062	0.12
NaCl	0.013	-	1x10 <sup>-6</sup>

dried pasta. The nutritional content for each of the foods measured is presented in Table 6-1, where this data was taken obtained from the food packaging.

Table 6-1 Nutritional content of foods which were dielectrically measured

In terms of sample preparation, Penne pasta was kept intact, whilst digestive biscuits and potato crisps were broken up to approximately 20-30mm sized pieces to improve packing density, similar to what would be achieved using a vibrating belt feed. For each food type, one batch of food was used for all the dielectric measurements taken. Multiple packets of food were required to make a batch in each instance. The waveguide load cell was first filled with one of the food types to determine the quantity of food to be dielectrically measured. The measured quantities were 433 grams of potato crisps, 793 grams of digestive biscuit, and 1095 grams of penne pasta. The dry basis packing density of the potato crisps was 146kg.m<sup>-3</sup>, for the biscuits it was 265kg.m<sup>-3</sup>, and for the pasta it was  $332 \text{kgm}^{-3}$ . The contents of the load cell were then transferred to a desiccator, through which dry nitrogen was passed at 41/min. The samples were not dried to zero moisture content as this would have taken a prohibitively long time. Instead they were dried to moisture contents below the moisture content of the finished product. The drying apparatus used to dry the samples is shown in Figure 6-4. The finished product moisture content of potato crisps and biscuits had already been quantified to be 0.017kg.kg<sup>-1</sup> and 0.025kg.kg<sup>-1</sup> wet basis respectively in Chapter 5, using the same dry nitrogen apparatus. The moisture content of packaged pasta was quantified to be

0.115kg.kg<sup>-1</sup> wet basis using an oven at 105°C with an exposure time of 107 days.



#### Figure 6-4 Test apparatus used for drying food samples

The waveguide was calibrated at the start of each day of measurements. After initial calibration the samples were transferred from the desiccator to the waveguide load cell and measured. In order to quantify the variation in dielectric measurement with respect to food packing and orientation, the food samples were emptied from the load cell into a plastic bag, and then transferred back to the load cell for each measurement. The dielectric measurement was carried out three times, each time with different packing. This ensured repeatability, which is of particular importance as there was a concern that the orientation of the particulates could affect the results. It took approximately 12 minutes to carry out each of the three dielectric measurements, including time taken to transfer the food to and from a plastic bag. Hence, the samples were in an uncontrolled humidity environment for approximately 36 minutes, in which time it could either adsorb or desorb moisture, depending on the moisture content of the food samples, and the humidity of the room. This is why the experiment was restricted to 3 repetitions, so as to limit the error in the moisture content. The dielectric measurement of food samples using the waveguide was carried out in an air conditioned laboratory, with temperature control of  $\pm 1^{\circ}$ C. The temperature control was necessary to prevent drift in the VNA. A measurement of the empty load cell was carried out before and after each set of 3 dielectric measurements. The drift in loss factor of the empty load cell before and after sample measurements was generally below 0.0002, while drift in dielectric constant was generally below 0.0007.

After 3 measurements of a food type, the mass of the food was measured using a weighing scale accurate to 0.1 grams, and was then transferred from the waveguide load cell to a plastic container. The food container was left open, without a lid, and placed inside a larger container. The larger container held two axial Multicomp fans (part number 9606149) to circulate the air, and an open container of deionised water. The lid of the larger container was then closed with an air tight seal. The box containing the samples was taken out periodically to measure the gain in mass until the samples had reached the desired moisture content for the next experiment. The mass of the samples was increased by approximately 0.01kg.kg<sup>-1</sup> dry basis for each experiment. Once the new target mass had been reached for the next experiment, the food sample box was removed from the larger container. A lid with an air tight seal was then fitted to the food sample box, and the food was left to stabilise for a minimum of two weeks. The stabilisation period was necessary to ensure that the moisture distribution was reasonably even. This stabilisation time period was deduced by observing the food stabilisation periods for sorption isotherm measurement (sorption isotherm measurements are discussed in Chapter 5).

For each food type, a moisture content below the moisture content of the packaged product was selected as the initial point of measurement. The moisture content was increased in approximately 0.01kg.kg<sup>-1</sup> dry basis increments as discussed above until mould growth started to occur. At which point no more measurements could be carried out, as deterioration of the food would affect the measurement.

### 6.3 Dielectric measurement results and assessment of proposed potato crisp microwave processes

### 6.3.1 Dielectric measurement results for potato crisps

In order to better understand the dielectric measurement results, the monolayer moisture content and the transition to solution needs to be quantified. The BET

monolayer moisture content was calculated in Chapter 5 to be 0.0336kg.kg<sup>-1</sup> dry basis (0.0325kg.kg<sup>-1</sup> wet basis). Figure 6-5 shows the adsorption isotherm at 20°C ±1°C. Labuza & Altunakar (2007) stated that water activity at 0.6 constitutes the critical point at which there is potential for growth of microbes if the moisture content goes higher. It can be seen from Figure 6-5 that a point of inflection could well occur at 0.6 given enough data points. The point of inflection at a water activity of 0.6 corresponds to transition from multilayer to solution. The ability to support microbe growth, and to support chemical reactions is seen as symptoms of the water behaving more like a solution than a multilayer (Yanniotis & Blahovec, 2009; Mujumdar, 2006). The moisture content at a water activity of 0.6 is 0.082kg.kg<sup>-1</sup> dry basis (0.076kg.kg<sup>-1</sup> wet basis).



Figure 6-5 Measured adsorption isotherm at 20°C for potato crisps, showing transition from monolayer to multilayer and the transition from multilayer to solution

The tabulated dielectric measurement results for potato crisps are shown in Appendix E. The measured loss factor and dielectric constant of potato crisps is shown in Figure 6-6 and Figure 6-7 respectively, where the monolayer and the solution transition are displayed in each instance.





Figure 6-6 Measured loss factor at 20°C, 915MHz, for potato crisps, showing BET monolayer and solution transition moisture contents



Figure 6-7 Measured dielectric constant at 20°C, 915MHz, for potato crisps, showing BET monolayer and solution transition moisture contents

The loss factor gradient is initially 0.26, which increases by a factor of 2.2 to a gradient of 0.57 after the monolayer moisture content, it then increases further by a factor of 2.4 to a gradient of 1.35 after the solution transition moisture content. The dielectric constant has a similar, if less pronounced trend. The dielectric constant initially has a gradient of 1.16, which increases by a factor

of 1.38 to 1.6 after the monolayer moisture content, and increases by a further factor of 2.18 to 3.47 after the solution transition moisture content. This shows that the transitions from monolayer, to multilayer, to solution defined by the sorption isotherm have a profound effect on dielectric behaviour. This stands to reason as both dielectric response and water activity are dependent on water mobility. Restrictions in water mobility due to water being "bound" are known to reduce both water activity and dielectric response (Labuza & Altunakar, 2007; Wang & Brennan, 1991; Schiffmann, 2006). However, what may be surprising is that there is a detectable point of inflection in dielectric response at the monolayer moisture content. Most other carbohydrate foods assessed in Chapter 3 do not have a discernible change in loss factor gradient at the monolayer moisture content. This was the case for raw potato, wheat flour, Chickpea flour, yellow dent field corn, and hydrocolloids (Mudgett, et al., 1980; Holtz, et al., 2010; Kim, et al., 1998; Moreira, et al., 2010; Guo, et al., 2008; Durakova & Menkov, 2005; Nelson, 1991c; Samapundo, et al., 2007; Nelson, et al., 1991d; Torres, et al., 2012).

Figure 6-8 shows the loss factor and water activity plotted with respect to moisture content. This plot shows that the points of inflection for the monolayer moisture content and the solution transition moisture content are aligned. As previously discussed the loss factor increases sharply after the solution transition moisture content. This behaviour was observed for most other carbohydrate foods, such as potato, wheat flour, chickpea flour, and yellow dent field corn, all of which are discussed in Chapter 3.





Figure 6-8 Measured adsorption isotherm at 20°C and loss factor at 20°C 915MHz for potato crisps, showing BET monolayer and solution transition moisture contents

# 6.3.2 Fitting equation to measured moisture dependant dielectric properties of potato crisps

Adapted water activity equations, derived in Chapter 3 were fitted to the measured loss factor and dielectric constant of potato crisps. The parameters of the fitted equations, as well as the Mean Relative Error (MRE) and the Residual Sum of Squares (RSS) are presented in Appendix F. The adapted GAB equation was found to provide the best fit to both the dielectric constant and loss factor measurements for both MRE and RSS measures. The GAB fit provided an MRE fit of 2.08% for dielectric constant, and 0.31% for loss factor as shown in **Error! Reference source not found.**. These are excellent fits as it s generally understood that a good fit is obtained when MRE<10% (McMinn, et al., 2007; McLaughlin & Magee, 1998). The GAB equation fits for potato crisp loss factor and dielectric constant are shown in Figure 6-9 and Figure 6-10 respectively. The adapted GAB equations used to describe the moisture dependant loss factor and dielectric constant are shown below for convenience, where  $M_0$ , C, and K are fitting parameters,  $M_{wb}$  is moisture content wet basis,

 $\varepsilon_{0m}^{\prime\prime}$  is the loss factor at zero moisture content, and  $\varepsilon_{0m}^{\prime}$  is the dielectric constant at zero moisture content. The equations for Mean Relative Error and the Residual Sum of Squares are also shown below, where  $u_e$  is the experimental value, and  $u_p$  is the predicted value. In this study, RSS has been used to determine the best fit, whilst MRE has been used as a method of comparing fits of different experimental data sets. Please note that the adapted water activity equations represent the moisture dependant dielectric properties at a singular temperature and frequency.

$$\varepsilon'' = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon_{0m}'' - UM_{wb}^S$$

$$\varepsilon' = \frac{1}{2} \left( \frac{-2M_{wb} + M_{wb}C - M_0C + \sqrt{4M_{wb}CM_0 + C^2 M_{wb}^2 - 2M_{wb}M_0C^2 + M_0^2C^2}}{M_{wb}K(C-1)} \right) + \varepsilon'_{0m} - UM_{wb}^S$$
Equation 6-2

$$RSS = \sum (u_e - u_p)^2$$
Equation 6-3

$$MRE = \frac{100}{N} \sum \left| \frac{u_e - u_p}{u_e} \right|$$
Equation 6-4

Model	Parameter	Potato crisps loss factor	Potato crisps dielectric constant	
M_0C		0.108224	0.125909	
		17.6525	14.6434	
	К	2.66442	0.764394	
GAB <u> ε<sub>0m</sub> <u> </u> U <u> S</u> MRE [%]</u>	0.0109861	1.20209		
	U	0	0	
	S	1	1	
	MRE [%]	2.08	0.31	
	RSS	3.54E-05	3.64E-04	

 
 Table 6-2 Best equation fit for moisture dependant potato crisp loss factor and dielectric constant



Figure 6-9 Adapted GAB equation best fit for measured potato crisps loss factor at 915MHz



Figure 6-10 Adapted GAB equation best fit for measured potato crisps dielectric constant at 915MHz

### 6.3.3 Dielectric response in relation to proposed potato crisp microwave applications

### 6.3.3.1 Dielectric response in relation to microwave finish drying of potato crisps

The proposed microwave finish drying process of potato crisps starts at 0.04kg.kg<sup>-1</sup> and finishes at 0.017kg.kg<sup>-1</sup> as shown in Figure 6-11 (where the experimental data presented is the same as that presented in Figure 6-9). This microwave drying process will potentially decrease the levels of acrylamide by 50% as discussed in Section 2.7.1, where acrylamide is a carcinogenic substance that forms readily when carbohydrate foods are heated above 120°C (IARC, 1994; Lineback, et al., 2012; Tareke, et al., 2002). The temperature of crisps rises above 120°C at the end of the conventional crisp frying process (Bows, et al., 2010). The microwave finish drying process enables finish drying at a temperature below 120°C, hence crisps can be removed from the frying process early to reduce acrylamide formation (Goldblith, 1966; Decareau, 1968). The final moisture content of potato crisps was measured gravimetrically to be 0.017kg.kg<sup>-1</sup> wet basis in Chapter 4 using continuous expose to dry nitrogen.



Figure 6-11 Measured loss factor at 20°C, 915MHz, for potato crisps, showing start and end of the proposed microwave finish drying process

Uneven moisture of particulate material being dried in a RF/MW applicator can occur for two main reasons. Either the food enters the microwave process with uneven moisture content, or uneven moisture content is generated in the microwave process as a result of uneven electric field exposure. Variability in electric field is inherent in all microwave applicators, and moisture levelling can be seen as an important effect that can counteract differences in electric field exposure. It should be noted that meander travelling wave applicators, which can produce quite even electric fields, are not suitable for this finish drying application due to the low loss factor of the dielectric load. Variability in moisture content can occur throughout a particulate, and within individual food samples. Moisture levelling within a material could be primarily driven by volumetric microwave heating of the entire food matrix rather than selective heating of the moisture phase. For instance, biscuits that have a wet inner core due to the conventional baking process can experience moisture levelling during MW/RF finish drying purely as a result of volumetric heating, where volumetric heating of the entire food matrix leads to dehydration of the inner core of the material. Selective heating of moisture can contribute to moisture levelling within a material, but it will always occur to some extent through volumetric heating. Moisture levelling of particulate, were different pieces of food have different moisture contents, is however different. Moisture levelling of particulate is completely dependent on the selective heating of moisture. A simple analysis can be carried out to assess the ability of selective heating of moisture to counteract uneven electric field exposure of particulate material travelling through the microwave applicator. Microwave heating is governed by the Equation 6-5 shown below (Mehdizadeh, 2010):

$$Pd = \omega \varepsilon_0 \varepsilon^{"} E_{RMS}^2$$
 Equation 6-5

Where  $E_{RMS}$  is the root mean square of the electric field strength inside the material [Vm<sup>-1</sup>],  $\varepsilon_0$  is the permittivity of free space [8.854x10<sup>-12</sup>Fm<sup>-1</sup>], and *Pd* is the power density [Wm<sup>-3</sup>]. This is a square law, so a small change in electric field has a large effect on heating. Hence, the loss factor has to vary significantly with respect to moisture content to counteract any variation in electric field exposure.

As particulate travels through the applicator there will be variation in the electric field, which is a function of the applicator geometry, the frequency of the energy source, and the dielectric properties of the particulate itself. As a result, some material in the particulate that travels through the applicator will be exposed to lower intensity electric field on average than other material in the particulate. Variations in electric field exposure in the particulate material can lead to variations in heating, and variation in final moisture content of the particulate. Using the moisture dependant dielectric measurements of the particulate material, the effect of variations in electric field exposure of the particulate material can be explored. More explicitly, the variation in moisture required to counteract a deficiency in electric field strength can be calculated using the moisture dependant dielectric properties of the dielectric load. Let's assume material enters the applicator at the same time, and some of the particulate is exposed to lower intensity electric field. After a period within the applicator the particulate material will become less dehydrated than average compared to all the other material that has been in the applicator for the same duration. However, the wetter material will start to heat more readily if dielectric properties are favourable, which is known as the moisture levelling effect.

The difference in moisture content required to counteract 10% lower intensity electric field exposure of the particulate material with 10% more microwave heating is calculated in scenario 1, as shown in Table 6-3. The average moisture content of the particulate is taken as 0.0285kg.kg<sup>-1</sup>, which is half dehydrated from the start moisture content of 0.04kg.kg<sup>-1</sup> to the final moisture content of 0.017kg.kg<sup>-1</sup>. The moisture content required to counteract the 10% lower intensity electric field by 10% additional heat is 0045kg.kg<sup>-1</sup> wet basis. However, this moisture content is higher than the 0.04kg.kg<sup>-1</sup> moisture content at the start of the finish drying process. Hence, the moisture levelling effect in this finish drying process is unlikely to be able to adequately correct a difference in moisture content caused by a 10% difference in electric field exposure after half the required dehydration has taken place. A small portion of the deficiency in electric field exposure of the wetter 0.045kg.kg<sup>-1</sup> potato crisp will be accounted for by the difference in dielectric constant, which is 1.7%

higher for the wetter potato crisp. This analysis shows that exposure of the dielectric load to electric field intensity must be very uniform if the tolerance for final moisture content is tight.

### Boundary conditions of the scenario 1 analysis

Average food particulate moisture content = 0.0285kg.kg<sup>-1</sup> wet basis Wet potato crisp particulate has 10% lower intensity electric field exposure

### Calculation

Extra moisture required for wet potato crisp particulate to have 10% more heating than received by potato crisp particulate with the average moisture content of 0.0285kg.kg<sup>-1</sup> wet basis

### Result

Extra 0.0167kg.kg<sup>-1</sup> wet basis required

Hence wet potato crisp particulate = 0.045kg.kg<sup>-1</sup> wet basis.

Moisture content wet basis [%]	Loss factor	Dielectric constant	Electric field of bulk material [Vm <sup>-1</sup> ]	Pd [Wm <sup>-3</sup> ]
0.0285	0.0182	1.2271	1	0.00093
0.045	0.0248	1.2478	0.9	0.00102
Difference in moisture	0.0167			
Increase in heating for	10%			

Summary of results for scenario1 is shown in Table 6-3.

increase in nearing for moisture revening –

# Table 6-3 Potato crisp particulate moisture levelling scenario 1 - 10% increase in heating for wet potato crisp particulate which has 10% lower intensity electric field exposure than average. Moisture content of average particulate material was taken as 0.0285kg.kg<sup>-1</sup> wet basis.

The loss factor at the final moisture content of 0.0173kg.kg<sup>-1</sup> wet basis is only 0.0149 for the potato crisp particulate, which is extremely low. Careful selection of low loss applicator materials would be required to heat a particulate material with a loss factor of 0.0149. For instance the conveyor belt may have to consist mostly of Teflon, and it may be necessary to make the applicator walls from aluminium to maximise energy efficiency. Selection of low loss materials for the applicator will add cost to the capital equipment.

### 6.3.3.2 Dielectric response in relation to explosive microwave dehydration of potato crisps

In the proposed explosive dehydration microwave process, potato crisps enter the fryer at a moisture content of 0.82kg.kg<sup>-1</sup>, and are explosively dehydrated down to 0.04kg.kg<sup>-1</sup> in 109 seconds as shown in Figure 6-12 (Bows, et al., 2010). The maximum temperature of the potato crisps during the microwave process is 155°C. A subsequent finish drying process, currently assumed to be an additional microwave finish drying process, dries the potato crisps from 0.04kg.kg<sup>-1</sup> to a final moisture content. Final moisture content for microwaved crisps will be the same as that required for continuous fried potato crisps, which was measured gravimetrically to be 0.017kg.kg<sup>-1</sup> wet basis in Chapter 4 using continuous expose to dry nitrogen.



Figure 6-12 Moisture content and temperature of potato crisps during rapid dehydration using microwave energy, figure adapted from patent written by Bows et al. (2010)

The loss factor of potato crisps was only measured up to 0.123kg.kg<sup>-1</sup> wet basis due to spoilage issues above this moisture content. The final moisture content of the explosive microwave dehydration process, which is 0.04kg.kg<sup>-1</sup> wet basis, is shown relative to the measured potato crisp loss factor in Figure 6-13.



Figure 6-13 Loss factor at 20°C, 915MHz, for potato crisps, showing moisture content at the end of the proposed explosive dehydration microwave process

In order to assess moisture levelling effectiveness in counteracting variation in electric field exposure near the end of the explosive dehydration process, scenario 2 was analysed as shown in Table 6-4. This scenario calculates the additional moisture required to provide 10% additional heating at a given dwell time if potato crisp particulate are exposed to 10% lower intensity electric field. The average moisture content of the particulate was taken to be 0.05kg.kg<sup>-1</sup> wet basis, where moisture levelling is important as it is close to the final moisture content of 0.04kg.kg<sup>-1</sup>. At a given exposure time, Table 6-4 shows that 0.016kg.kg<sup>-1</sup> additional moisture would need to be present to provide 10% additional heating in the event of 10% lower intensity electric field exposure for the wetter potato crisp particulate. The wetter potato crisp particulate in this scenario has 2.2% higher dielectric constant, which will account for a portion of the 10% lower electric field exposure. Further analysis carried out in scenario 3 showed that a moisture content of 0.061kg.kg<sup>-1</sup> is required to receive the same heating with 10% lower intensity electric field exposure. This shows that the microwave process cannot moisture level to within a tolerance of 0.01kg.kg<sup>-1</sup> wet basis if potato crisp particulate receives

10% variation in electric field exposure when travelling through the microwave applicator. Scenarios 2 and 3 are summarised as follows:

### Boundary conditions of the scenario 2 analysis

Average food particulate moisture content = 0.05kg.kg<sup>-1</sup> wet basis Wet potato crisp particulate has 10% lower intensity electric field exposure

### Calculation

Extra moisture required for wet potato crisp particulate to have 10% more heating than received by potato crisp particulate with the average moisture content of 0.05kg.kg<sup>-1</sup> wet basis

### Result

Extra 0.016kg.kg<sup>-1</sup> wet basis required

Hence wet crisp particulate = 0.066kg.kg<sup>-1</sup> wet basis

Summary of results for scenario 2 is shown in Table 6-4.

Moisture content wet basis [%]	Loss factor	Dielectric constant	Electric field of bulk material [Vm <sup>-1</sup> ]	Pd [Wm⁻³]
0.05	0.0271	1.2549	1	0.00138
0.066	0.0368	1.2828	0.9	0.00152
Difference in moisture		0.0158		
Increase in heating for	10%			

Table 6-4 Potato crisp particulate moisture levelling scenario 2 - 10% increase in heating for wet potato crisp particulate which has 10% lower intesnity electric field exposure than average. Moisture content of average particulate was taken as 0.05kg.kg<sup>-1</sup> wet basis

### Boundary conditions of the scenario 3 analysis

Average food particulate moisture content = 0.05kg.kg<sup>-1</sup> wet basis

Wet potato crisp particulate has 10% lower intensity electric field exposure

### Calculation

Extra moisture required for wet potato crisp particulate to have the same heating as that received by potato crisp particulate with the average moisture content of 0.05kg.kg<sup>-1</sup> wet basis

### Result

Extra 0.011kg.kg<sup>-1</sup> wet basis required Hence wet crisp particulate = 0.061kg.kg<sup>-1</sup> wet basis

Electric field of bulk Moisture content wet Dielectric Loss material [Vm<sup>-1</sup>] Pd [Wm<sup>-3</sup>] basis [%] factor constant 0.00138 0.05 0.0271 1.2549 1 0.9 0.00138 0.061 0.0335 1.2734 0.0109 Difference in moisture content = Increase in heating for moisture levelling = 0%

Summary of results for scenario 4 is shown in Table 6-4.

Table 6-5 Potato crisp particulate moisture levelling scenario 3 – Wet potato crisp particulate with 10% lower intensity electric field exposure than average has the same heating as potato crisp particulate with an average moisture content of 0.05kg.kg<sup>-1</sup> wet basis

Although potato crisps were only measured up to a moisture content of 0.123kg.kg<sup>-1</sup> wet basis due to spoilage issues, dielectric measurements taken by Mudgett et al. (1980) of uncooked potato can be used to assess the deielctric behaviour with respect to the explosive dehydration process at higher moisture contents. Measurements taken by Mudgett et al. (1980) of uncooked potato at 3GHz at 20°C is shown in Figure 6-14 with the start and end points of the explosive dehydration microwave process. It can be seen from Figure 6-14 that the loss factor actually increases as the potato is dehydrated from 0.82kg.kg<sup>-1</sup> down to 0.66kg.kg<sup>-1</sup> wet basis. This could lead to some thermal runaway and any uneveness in electric field could result in large differences in moisture content resulting from the negative loss factor gradient between 0.82kg.kg<sup>-1</sup> and 0.66kg.kg<sup>-1</sup> wet basis would have to be corrected in the positive loss factor gradient between 0.66kg.kg<sup>-1</sup> and 0.04kg.kg<sup>-1</sup>

However, a plot of the loss tangent with respect to moisture content is shown in Figure 6-15 showing a point of inflection at 0.31kg.kg<sup>-1</sup> wet basis. This shows that moisture levelling may not occur until the potato crisps have been dehydrated below 0.31kg.kg<sup>-1</sup> wet basis.



Figure 6-14 Loss factor at 20°C, 3GHz, for uncooked potato, showing moisture content at the start and end of the proposed explosive dehydration microwave process. Adapted from (Mudgett, et al., 1980)



Figure 6-15 Loss tangent at 20°C, 3GHz, for uncooked potato, showing moisture content at the start and end of the proposed explosive dehydration microwave process. Calculated using Mudgett et al., (1980) data

The dielectric constant decreases by 33% when the potato is dehydrated from 0.31kg.kg<sup>-1</sup> down to 0.25kg.kg<sup>-1</sup>, which will result in an increase in electric

field internal to the dielectric for a given external electric field. A significant increase in internal electric field in the potato crisp material with lower moisture content will counteract the decrease in loss factor. This can be analysed using moisture dependant dielectric measurements of solid potato presented by Mudgett et al. (1980). Unlike the previous analysis, which used dielectric properties of particulate material, the measurements carried out by Mudgett et al. (1980) was of solid potato at 3GHz. This allows a different analysis approach, which can take account of the effect that a different dielectric constant has on the electric field within the potato. Both loss factor and dielectric constant changes with respect to moisture content, and both properties have an effect on microwave heating. In this analysis method, a potato slice is considered to be a slab within a uniform and infinite electric field, where the electric field can be calculated using the following equation: (Fitzpatrick, 2014).

$$E_1 = \frac{E_0}{\varepsilon_1}$$
 Equation 6-6

Where  $E_{I}$  is the electric field within the dielectric [Vm<sup>-1</sup>],  $\varepsilon_{I}$  is the dielectric constant of the dielectric, and  $E_0$  is the external electric field [Vm<sup>-1</sup>]. Assuming a uniform and constant electric field, the microwave heating can be calculated for different moisture contents, where reductions in internal field are taken into account using Equation 6-6. This differs from the previous particulate analysis approach which looked at the effect of different electric fields on particulates with different moisture contents. In the particulate analysis, the difference in electric field can come about due to a number of reasons, including applicator design, and a change in dielectric constant due to moisture or temperature dependence. The particulate analysis is useful in instances where the dielectric constant does not vary much over the moisture content range of interest. Unlike the previous particulate analysis, this analysis is of an individual potato slice and the electric field intensity is not varied. Instead, the heating is calculated for different moisture contents in a uniform electric field, taking the change in dielectric constant into account. The results are shown in Figure 6-5, and it can be seen that there is slightly less heating at moisture contents of 0.30 and 0.31kg.kg<sup>-1</sup> wet basis compared to 0.25kg.kg<sup>-1</sup> for a given external electric field. This would lead to negligible moisture levelling over this moisture

content range. Hence, any difference in moisture content of particulate travelling through the applicator will not be corrected during dehydration from 0.31kg.kg<sup>-1</sup> down to 0.25kg.kg<sup>-1</sup> wet basis.

			Internal		Increase in heating
Total moisture			electric		compared to
content wet	Loss	Dielectric	field	Pd	0.25kg.kg <sup>-1</sup> moisture
basis[kg.kg <sup>-1</sup> ]	factor	constant	E <sub>1</sub> [Vm <sup>-1</sup> ]	[Wm⁻³]	content heating
0.25	4.8045	13.1	0.0761	0.00142	0.00%
0.26	5.7079	14.2	0.0706	0.00145	2.17%
0.28	7.5989	16.3	0.0613	0.00146	2.77%
0.29	8.5364	17.4	0.0575	0.00144	1.38%
0.3	9.4471	18.5	0.0540	0.00140	-0.83%
0.31	10.3220	19.6	0.0510	0.00136	-3.64%

Table 6-6 Heating in potato crisps at moisture contents of 0.25kg.kg<sup>-1</sup> wet basis and higher with a uniform and constant external electric field

The same analysis approach was taken with a base moisture content of 0.05kg.kg<sup>-1</sup> wet basis and the results are detailed in Table 6-7.

Total			Internal		Increase in heating
moisture			electric		compared to
content wet	Loss	Dielectric	field		0.05kg.kg <sup>-1</sup> moisture
basis[kg.kg <sup>-1</sup> ]	factor	constant	E <sub>1</sub> [Vm <sup>-1</sup> ]	Pd [Wm⁻³]	content heating
0.05	0.1929	4.100	0.244	0.000584	0.0%
0.06	0.2242	4.101	0.244	0.000679	16.2%
0.07	0.2577	4.105	0.244	0.000779	33.3%
0.08	0.2941	4.119	0.243	0.000882	51.1%
0.09	0.3341	4.154	0.241	0.000986	68.8%
0.1	0.3787	4.221	0.237	0.001082	85.2%



Interestingly, the mositure levelling effect is very strong over the moisture content range of 0.05kg.kg<sup>-1</sup> to 0.1kg.kg<sup>-1</sup> wet basis. An extra 0.01kg.kg<sup>-1</sup> wet basis in mositure content results in 16% more heat, while an extra 0.05kg.kg<sup>-1</sup> wet basis in mositure content results in 85% more heat. This would certainly help to correct any gross differences in moisture content resultant from uneven electric field exposure that has been exasperated by thermal runaway. The reason that moisture levelling appears to be more effective at lower moisture

contents is the moisture dependant behaviour of the dielectric constant. The dielectric constant for slab type geometry is inversly proportional to the electric field, and the heating is proporitional to the square of the electric field. Hence, for this assumed geometry, heating is more sensitive to changes in dielectric constant than to changes in loss factor, as the loss factor is direction proportional to heating, and is not squared. The dielectric constant does not change much at lower moisture contents as shown in Figure 6-16 and Figure 6-17, where Figure 6-16 presents measurements taken by Mudgett et al. (1980) of solid potato and Figure 6-17 presents measurements of potato crisp particulate material taken in the present study. This is yet another reason as to why microwave heating is best applied for finish drying. Established reasons for restricting microwave heating to finish drying is the ability for volumetric heating to pump mositure to the surface for removal, and the high cost of dehydrating large quantities of water using microwave energy.



Figure 6-16 Dielectric constant at 20°C, 3GHz, for solid uncooked potato, showing flat response at low moisture contents – adapted from Mudgett, et al. (1980)

One of the potential issues of explosive dehydration of potato crisps is arcing. The loss factor of the potato crisp particulate at the end of the process is just 0.0225, which is very low and the heating required is set and cannot be adjusted. The heating required is pre-determined as the microwave process must follow the dehydration curve of the frying process to create the desired potato crisp structure. This lack of flexibility with regards to microwave heating could be an issue if arcing is found to occur. The finite difference weighted average of potato crisp isosteric heat from 0.82kg.kg<sup>-1</sup> to 0.04kg.kg<sup>-1</sup> wet basis was calculated to be 2.54x10<sup>6</sup>Jkg<sup>-1</sup> using the Tsami et al. (1990) curve fit model of measured desorption results presented in Chapter 5 (see also Figure 2-20). The duration of the proposed explosive dehydration microwave process is just 110 seconds, hence the power required is 23.1kW.kg<sup>-1</sup>. This level of microwave heating can be used to carry out practical microwave heating experiments on potato slices to assess the risk of arcing.



Figure 6-17 Dielectric constant at 20°C, 915MHz, for potato crisps particulate, showing relatively flat response at low moisture contents

### 6.3.4 Quality of microwave dried potato crisps

### 6.3.4.1 Quality of microwave finish dried potato crisps

It is proposed that MW finish drying starts at a moisture content of 0.04kg.kg<sup>-1</sup>, after approximately 109 seconds of frying as shown in Figure 6-18.



Figure 6-18 Continuous frying stopped half way through with proposed MW finish drying step added. Continuous frying curve adapted from Bows et al. (2010)

From a quality point of view, the glass transition of potato at the frying temperature of 185°C is 0.04kg.kg<sup>-1</sup> to 0.05kg.kg<sup>-1</sup> wet basis (Mizuno, et al., 1998). Hence, the structure and porosity of the potato crisp will be set when the product leaves the fryer at a moisture content of 0.04kg.kg<sup>-1</sup> and enters the microwave finish drying process. The porosity and structure of the potato crisp has a strong influence on taste (Rodríguez-Ramíez, et al., 2012), and this will be unchanged with the proposed finish drying process. The microwave finish drying process dries the product from 0.04kg.kg<sup>-1</sup> down to 0.017kg.kg<sup>-1</sup>, which is the same final moisture content of the standard continuous frying process (Desai, et al., 2014). Hence water activity, shelf life, and crispness will be the same as the standard continuous fried process. Oil uptake should be the same as a result of the frying process and oil vaporisation during the microwave process is likely to be very low due to the temperature limit of 105°C and the short microwave exposure time of just 2-3 minutes (Goldblith, 1966; Decareau, 1968). Hence, customers should not notice any difference in taste as a result of the additional microwave finish drying process as porosity, water content and oil content should remain unchanged. This microwave process can therefore be

considered low risk with respect to customer acceptance and future sales. The key advantage of the finish drying process is the reduction in acrylamide levels, which is considered a carcnigenic substance (IARC, 1994; Lineback, et al., 2012). Frying at 185°C to a moisture content of 0.04kg.kg<sup>-1</sup> followed by a microwave finish drying porcess could result in a acrylamide reduction of approximately 50% using data presented by Granda et al. (2004). However there is a lack of data points presented by Granda et al. (2004) and further experiments are recommended to deduce a more accurate reduction in accrylamide resultant from the proposed process.

#### 6.3.4.2 Quality of explosive microwave dehydrated potato crisps

The explosive dehydration microwave process was developed to replicate the frying dehydration curve in the hope that the taste of a continuous fried potato crisp could be replicated, but with the advantage of having control of the oil content. A novel Micro-CT method has been developed in the present study to assess the porosity of potato crisps. Using this new method, the porosity of continuously fried potato crisps was deduced to be 0.627 with a standard deviation of 0.032, and the porosity of potato crisps that underwent the proposed explosive microwave dehydration process had a porosity of 0.653 with a standard deviation of 0.018. These results showed that the microwave process was successful at replicating the porosity of the continuous fried potato crisp. A forced preference consumer test was carried out with 200 people who were selected to be balanced across gender, age (<35, >35), socio-economic grouping, and eating frequency. In this survey the microwaved dehydrated product was preferred by 51% which allows us to conclude that consumers equally preferred microwaved potato crisps and standard fried crisps. This provides further confirmation that the proposed microwave process successfully replicates the frying process so that the product is acceptable to the customer and of low commercial risk with respect to sales. The ability to vary oil content independently of the product structure is a huge competitive advantage and allows the producer to offer lower fat potato crisps to targeted consumer groups.

# 6.3.5 Energy requirement and commercial feasibility for proposed potato crisp microwave processes

# 6.3.5.1 Energy requirement and commercial feasibility of microwave finish drying of potato crisps

Using the isosteric heat calculated in Chapter 5, it is possible to calculate the additional cost of energy required for the microwave finish drying process, compared to the frying process. The inputs and assumptions used are listed below. These inputs and assumptions are fully discussed in Appendix G.1, complete with references.

- Microwave finish drying starts at 0.04kg.kg<sup>-1</sup> wet basis and finishes at 0.017kg.kg<sup>-1</sup> wet basis
- Isosteric heat curve deduced from sorption isotherm measurement in Chapter 5 was used to calculate the total isosteric heat of sorption over the required moisture content
- It is assumed that the finite difference weighted average of potato crisp isosteric heat over the relevant moisture content range constitutes the energy required for drying.
- It is assumed that the microwave finish drying process will be fed from a typical continuous fryer, resulting in a production rate of 3000kg.hr<sup>-1</sup> of finished product at a moisture content of 0.017kg.kg<sup>-1</sup> wet basis
- It is assumed that the oil content does not change during microwave finish drying due to the low proposed temperature of 105°C and the short dwell time of 2-3 minutes
- It is assumed that the product enters the finish drying process at a temperature higher than 105°C, hence no warm up of material is required.
- The efficiency of the magnetron to convert electrical energy to microwave energy is assumed to be 88%
- It is assumed that 97% of the microwave energy is converted into useful heating energy
- The cost of the microwave generator equipment is calculated to give an indication of capital costs, the cost of the applicator and conveyor belt system is not considered.
- The efficiency of the fryer is approximately 65%, which is the ratio of the energy added to the food and the total energy supplied to the appliance during cooking. The continuous fryer is gas fired.
- The cost of electrical energy was assumed to be 0.21£/kWh
- The cost of gas energy was assumed to be 0.0656£/kWh
- It is assumed that production occurs for 8 hours in a day, 5 days a week, for 51 weeks in a year

Using the isosteric heat calculated in Chapter 5, it was calculated in Appendix G.1 that the microwave finish drying process would require one 100kW 915MHz generator for a production rate of 3000kg.hr<sup>-1</sup> of finished product. If amortization of the generator occurs in the first year of production, then the cost of the generator and the extra cost in energy amounts to 0.051 pence per packet of crisps. For subsequent years, the extra cost of energy for the microwave process equates to 0.014 pence per packet of crisps. The additional energy costs of the microwave process, and the prospect that finish drying can be carried out with one 100kW generator is commercially interesting. A full economic feasibility study would also have to consider reliability, development costs, scale up costs, and capital costs.

## 6.3.5.2 Energy requirement and commercial feasibility of explosive microwave dehydration of potato crisps

As in the previous section, it is possible to calculate the additional cost of energy required for explosive microwave dehydration of potato crisps compared to the existing frying process, using the isosteric heat calculated in Chapter 5. The inputs and assumptions used in this commercial analysis are the same as those presented for potato crisp finish drying, with the following exceptions:

• Explosive microwave dehydration starts at 0.82kg.kg<sup>-1</sup> wet basis and finishes at 0.04kg.kg<sup>-1</sup> wet basis

- The oil content is assumed to be 0.15kg.kg<sup>-1</sup>, which is approximately half that a "full fat" potato crisp.
- The potato crisps are heated from 20°C to 155°C in the explosive microwave dehydration process
- It is assumed that the microwave finish drying process described in the previous section is used to dehydrate the potato crisps from 0.04kg.kg<sup>-1</sup> wet basis down to a final moisture content of 0.017kg.kg<sup>-1</sup> wet basis.

The inputs, assumptions, and calculations are all fully detailed and discussed in Appendix G.2, complete with references. In order to replace one 3000kg.h<sup>-1</sup> fryer, it was calculated that ninety five 100kW 915MHz generators would be required for explosive dehydration, and one 100kW 915MHz generators would be required for finish drying. Generators with a power output of 100kW at a frequency of 915MHz are commercially available from suppliers such as Muegge (2016). The cost of a generator capable of delivering 100kW is approximately £70,000 (Smith, 2016). Hence the cost of the generators alone would be approximately £6.7 million for the full scale application.

If amortization of the generators is carried out in the first year, the additional cost of the generators and the energy amounts to 5.3 pence per packet of crisps. Subsequent years would cost 1.7 pence per packet of crisps for the additional cost of the energy. The amortization in the first year does not consider the cost of the applicator and conveyor belt system, which may be very complex in this instance due to the high number of generators required. Additional maintenance costs of the microwave system is not considered, these costs may also be significant due the number of generators. Single packs of Walkers crisps sell for 55 pence in most supermarkets, while packs sold in multipacks sell for an equivalent 33 pence each. The 1<sup>st</sup> year amortization period would be 9.6% of the price of a single packet of crisps, and 16.0% of a multipack packet of crisps. The subsequent yearly periods would cost 3.0% of the price of a single packet of crisps. Hence a fat tax of around 10% may make this process financially viable in theory, especially if the low fat crisps can be sold at a slight premium.

However, the main issue with this process is practicalities associated with the ninety six 100kW 915MHz generators required to replace the frying process. There are 95 generators required to bring the moisture content from 0.82kg.kg<sup>-1</sup> down to 0.04kg.kg<sup>-1</sup> wet basis, and 1 generator required to bring it from 0.04kg.kg<sup>-1</sup> down to 0.0173kg.kg<sup>-1</sup> wet basis. There is a lot more water removal involved in the first process despite the fact that the total isosteric energy is higher at lower moisture contents. This can be better understood by considering the dry basis moisture contents. The first process takes the moisture content from 4.56kg.kg<sup>-1</sup> down to 0.042kg.kg<sup>-1</sup> dry basis, while the second finish drying process takes the moisture content from 0.042kg.kg<sup>-1</sup> down to 0.018kg.kg<sup>-1</sup> dry basis. The 96 generators required for the overall microwave process would require a large factory footprint. Each 100kW generator would have a footprint of approximately  $3.2m^2$  (Smith, 2016). It can be assumed that this footprint would have to be doubled to allow access for maintenance, hence the total footprint of the generators alone would be  $3.2x2x96 = 614m^2$ . Connecting the generators to the applicators could be quite complex, and maintaining ninety six generators would be very expensive and require considerable expertise compared to maintaining one fryer. The energy costs calculated, and the number of generators required present significant challenges to commercialisation, even if a large fat tax levy was to come into effect. Development costs, scale up costs, capital costs, and reliability would all have to be carefully considered to assess commercial viability.

### 6.4 Dielectric measurement results and assessment of proposed biscuit microwave finish drying

### 6.4.1 Dielectric measurement results for digestive biscuits

It is desirable to assess the dielectric properties in relation to the state of the water, as the state of the water is important for stability and shelf life of the food. The state of the water is defined by the adsorption isotherm shown in Figure 6-19, where the BET monolayer moisture content is 0.0391kg.kg<sup>-1</sup> dry basis (0.0376kg.kg<sup>-1</sup> wet basis) as deduced in Chapter 5. Although the error bars are shown in Figure 6-19, they are not easily visible as the standard deviation is so small. The transition from multilayer to solution, which

corresponds to the critical water activity of 0.6 (Labuza & Altunakar, 2007), occurs at a moisture content of 0.0934kg.kg<sup>-1</sup> dry basis (0.0854kg.kg<sup>-1</sup> wet basis).

The tabulated dielectric measurement results for biscuits are shown in Appendix E. Both the loss factor and the dielectric constant measurements are presented in Figure 6-20 and Figure 6-21 respectively, with the transition from monolayer to multilayer, and the transition from multilayer to solution. The gradient below the transition from monolayer to multilayer is linear and is very low at 0.27, where the gradient was calculated using the measurement data at 0.0015kg.kg<sup>-1</sup> and 0.0363kg.kg<sup>-1</sup> wet basis. The gradient remains linear until a point of inflection is observed at a moisture content of 0.046kg.kg<sup>-1</sup> wet basis. There is an incremental increase in the gradient of loss factor above 0.046kg.kg<sup>-1</sup> in the multilayer region. Above the transition from multilayer to solution, the gradient is linear and is relatively high at 2.0, where the gradient was calculated using the measurement data at 0.028kg.kg<sup>-1</sup> and 0.12kg.kg<sup>-1</sup> wet basis.



Figure 6-19 Measured adsorption isotherm at 20°C for biscuits, showing transition from monolayer to multilayer and the transition from multilayer to solution



Figure 6-20 Measured loss factor at 20°C, 915MHz, for biscuits, showing BET monolayer and solution transition moisture contents



Figure 6-21 Measured dielectric constant at 20°C, 915MHz, for biscuits, showing BET monolayer and solution transition moisture contents

The biscuit loss factor is compared to the corresponding biscuit sorption isotherm in Figure 6-22. It can be seen that the first point of inflection of the loss factor occurs after the monolayer moisture content point of inflection of the sorption isotherm. No decrease in the rate of change in loss factor was detected, which has been observed as a result of salt dilution in the dielectric measurement of carbohydrate foods presented in literature (see Chapter 3). However, the loss factor was only measured up to a moisture content of 12.1kg.kg<sup>-1</sup> wet basis due to mould growth issues, and saturation occurs at a moisture content of approximately 0.20kg.kg<sup>-1</sup> wet basis as observed during sorption isotherm measurement. Hence the loss factor gradient may decrease with respect to moisture content at higher moisture contents.

From the data presented, it can be said that the loss factor is subdued below the monolayer moisture content. An incremental increase in loss factor is observed during the multilayer region resulting in a large positive gradient in loss factor with respect to moisture content after the transition to solution. The loss factor critical moisture content probably occurs at approximately 0.05kg.kg<sup>-1</sup> wet basis where a point of inflection can be observed. The loss factor critical moisture content therefore occurs in the multilayer region, slightly above the monolayer moisture content of 0.0376kg.kg<sup>-1</sup> wet basis.



Figure 6-22 Measured adsorption isotherm at 20°C and loss factor at 20°C 915MHz for biscuits, showing BET monolayer and solution transition moisture contents

# 6.4.2 Fitting equation to measured moisture dependant dielectric properties of biscuits

The parameters of the adapted water activity equations were fitted to biscuit loss factor and dielectric measurements using non-linear regression. The best fit parameters of the adapted water activity equations are presented Appendix F, where the adapted GAB equation provided the best fit. Figure 6-23 and Figure 6-24 show the adapted GAB equation fits, and the adapted GAB parameters are shown in Table 6-8. The GAB fit MRE for the dielectric constant and loss factor was 0.24% and 0.97% respectively. Hence the adapted GAB equation fit provides an excellent representation of the dielectric data as it is generally understood that a good fit is obtained when MRE<10% (McMinn, et al., 2007; McLaughlin & Magee, 1998).



Figure 6-23 Adapted GAB equation best fit for measured biscuit loss factor at 915MHz



Figure 6-24 Adapted GAB equation best fit for measured biscuit dielectric	ļ
constant at 915MHz	

Model	Parameter	Biscuit loss factor	Biscuit dielectric constant
GAB	Mo	0.0926863	0.100759
	С	26.1926	16.641
	K	2.34492	0.737943
	ε <sub>0m</sub> "	0.0167847	1.34637
	U	0	0
	S	1	1
	MRE [%]	0.97	0.24
	RSS	5.26E-06	3.17E-04

 Table 6-8 Best equation fit for moisture dependant biscuit loss factor and dielectric constant

# 6.4.3 Dielectric response in relation to microwave finish drying of biscuits

The final moisture content of digestive biscuits is 0.025kg.kg<sup>-1</sup> wet basis, which was measured gravimetrically through continuous exposure to dry nitrogen as discussed in Chapter 5. The prime objective of microwave finish drying of biscuits is to improve moisture uniformity and prevent cracking, or "checking" as it is called in the biscuit industry. Checking can occur if the

moisture gradient between the border and the centre of the biscuit is greater than 0.015kg.kg<sup>-1</sup> (Chang, et al., 2011). This can be improved to  $\pm 0.002$ kg.kg<sup>-1</sup> using RF finish drying (Awuah, et al., 2015). Removing the biscuit too early from the oven dryer could result in incorrect colouration and flavour development (Manley, 2000). Hence, this study proposes that finish drying should commence at a moisture content of 0.04kg.kg<sup>-1</sup> wet basis for digestive biscuits. The loss factor of digestive biscuits is shown in Figure 6-25 with respect to the start and end moisture contents of the proposed microwave finish drying process.

Similar to Scenario 1, 2, and 3 in Section 6.3.3, an analysis was carried out on the bulk particulate material, determining sensitivity to electric field exposure and moisture content. The power density was calculated using Equation 6-5 to assess a moisture levelling scenario. The adapted GAB equation for moisture dependant loss factor was used to calculate the power density, where the GAB parameters are presented in Table 6-8.



Figure 6-25 Loss factor at 20°C, 915MHz, for biscuits, showing start and end of the proposed microwave finish drying process

In this moisture levelling scenario, the biscuit particulate is half way through its dehydration process with an average moisture content of 0.0325kg.kg<sup>-1</sup> wet

basis, where it enters the microwave process at 0.04kg.kg<sup>-1</sup> and leaves at 0.025kg.kg<sup>-1</sup>. The scenario assess how much extra moisture is required to provide 10% extra heating in an instance where the biscuit particulate will be exposed to 10% lower intensity electric field in the applicator. Some of the biscuit particulate could on average experience 10% lower intensity electric field as it travels through the applicator as a result of a number of factors including applicator design, frequency, and dielectric properties. The additional moisture required for 10% extra heat when exposed to 10% lower intensity electric filed was calculated to be 0.018kg.kg<sup>-1</sup>. This requires a moisture content of 0.051kg.kg<sup>-1</sup> wet basis which is larger than the moisture content proposed for the start of the microwave process. The dielectric constant of the particulate biscuit is 2.5% higher at a moisture content of 0.051kg.kg<sup>-1</sup> compared to the dielectric constant at 0.0325kg.kg<sup>-1</sup> moisture content, which will account for a portion of the 10% lower intensity electric field exposure. Similar to potato crisp finish drying, this calculation shows that this process is very sensitive to electric field exposure. The moisture levelling mechanism is unlikely to keep the final moisture content tolerance within  $\pm 0.01 \text{kg}.\text{kg}^{-1}$  wet basis if electric field exposure of the biscuits varies by more than 10%.

#### **Boundary conditions of analysis**

Average food particulate moisture content = 0.0325kg.kg<sup>-1</sup> wet basis Wet biscuit particulate has 10% lower intensity electric field exposure

### Calculation

Extra moisture required for wet biscuit particulate to have 10% more heating than received by biscuit particulate with the average moisture content of 0.0325kg.kg<sup>-1</sup> wet basis

### Result

Extra 0.0184kg.kg<sup>-1</sup> wet basis required Hence wet biscuit particulate = 0.51kg.kg<sup>-1</sup> wet basis

Moisture content wet basis [%]	Loss factor	Dielectric constant	Electric field of bulk material [Vm <sup>-1</sup> ]	Pd [Wm <sup>-3</sup> ]
0.0325	0.0251	1.3826	1	0.00128
0.051	0.0342	1.4172	0.9	0.00141
Difference in moisture content =				0.0184
Increase in heating for moisture levelling =				10%

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Table 6-9 Biscuit moisture levelling scenario - 10% increase in heating for wet biscuit particulate which has 10% lower intensity electric field exposure than average. Moisture content of average particulate material was taken as 0.0325kg.kg<sup>-1</sup>, wet basis.

The scenario presented in Table 6-9 shows that evenness of electric field exposure is critical to the success of this application. It would also suggest that the main benefit of RF/MW finish drying is volumetric heating, which can be used to aid the removal of moisture from the centre of biscuits and help avoid heat damage to the surface of the biscuit which can occur in conventional air drying. The lack of moisture levelling greatly increases the risk of burning due to uneven electric field. This may be one of the reasons why finish drying of biscuits is almost exclusively carried out with RF dryers rather than microwave dryers (Schiffmann, 2001a). It is easier to obtain a uniform electric field at RF frequencies due to the longer wavelength, especially over wide conveyor belts. Awuah et al. (2015) reported that a moisture tolernace of  $\pm 0.002$ kg.kg<sup>-1</sup> can be achieved using RF heating, which suggests that food travelling through an RF applicator can receive very even electric field exposure. This may be especially true for biscuits as they are typically processed on a monolayer.

### 6.4.4 Quality of RF/MW finish dried biscuits

Improvement in quality is the main motivation for RF finish drying biscuits. Traditional oven baking can lead to large moisture gradients from the centre of the biscuit to the outside of the biscuit. These moisture gradients cause stresses to build up in the biscuit structure which leads to fractures, or "checking" as it is called in the biscuit industry (Koral, 2004). Checking is known to occur when the moisture gradient in the biscuit is more than 0.015kg.kg<sup>-1</sup> (Chang, et al., 2011). The variance in moisture content has been reported to be brought

down to 0.002kg.kg<sup>-1</sup> through RF finish drying (Awuah, et al., 2015). As stated in the previous section, electric field uniformity is critical to this process, an observation also made by Dev & Raghavan (2015). It is therefore important that food travelling through the applicator receives uniform electromagnetic field exposure.

### 6.5 Dielectric measurement results and assessment of proposed pasta microwave process

### 6.5.1 Dielectric measurement results for pasta

As with biscuits and potato crisps the dielectric properties of pasta were assessed in relation to the state of the water, as the state of the water is important for stability and shelf life of the food. The state of the water is defined by the adsorption isotherm shown in Figure 6-19, where the BET monolayer moisture content is 0.070kg.kg<sup>-1</sup> dry basis (0.066kg.kg<sup>-1</sup> wet basis), and the transition from multilayer to solution corresponding to a water activity of 0.6 occurs at a moisture content of 0.151kg.kg<sup>-1</sup> dry basis (0.131kg.kg<sup>-1</sup> wet basis). The sorption data was taken from Lagoudaki et al. (1993), where the sorption properties of pasta was measured at 22°C.



Figure 6-26 Sorption isotherm at 22°C for pasta adapted from Lagoudaki et al. (1993), showing transition from monolayer to multilayer and the transition from multilayer to solution

The tabulated dielectric measurement results for pasta are shown in Appendix E. Both the loss factor and the dielectric constant measurements are presented in Figure 6-27 and Figure 6-28 respectively, with the transition from monolayer to multilayer, and the transition from multilayer to solution. The gradient of the loss factor with respect to moisture content below the monolayer is 1.0. The gradient increases to 2.2 between the monolayer and the solution transition. The gradient then increases steeply to 5.6 above the solution transition.



Figure 6-27 Measured loss factor at 20°C, 915MHz, for pasta, showing BET monolayer and solution transition moisture contents

The loss factor is plotted together with the water activity in Figure 6-29. This plot clearly shows that there is a correlation between the points of inflection of the loss factor and water activity. This is particularly noticeable at the transition from multilayer to solution where there is a large increase in both water activity and loss factor. This shows agreement with published carbohydrate food dielectric behaviour, where a large increase in loss factor was observed at the transition from multilayer to solution (Mudgett, et al., 1980; Holtz, et al., 2010; Kim, et al., 1998; Guo, et al., 2008)



Figure 6-28 Measured dielectric constant at 20°C, 915MHz, for pasta, showing BET monolayer and solution transition moisture contents



Figure 6-29 Measured adsorption isotherm at 22°C and loss factor at 20°C 915MHz for pasta, showing BET monolayer and solution transition moisture contents

# 6.5.2 Fitting equation to measured moisture dependant dielectric properties of pasta

Adapted water activity equations, derived in Chapter 3 were fitted to the measured loss factor and dielectric constant of pasta as shown in Appendix F. Figure 6-30 and Figure 6-31 show the GAB equation fits, which was found to be the best fit by RSS for both loss factor and dielectric constant, the parameters are shown in Table 6-10. The GAB fit MRE for the dielectric constant and loss factor is 1.07% and 4.46% respectively, which is below the 10% threshold generally defined as a good fit (McMinn, et al., 2007; McLaughlin & Magee, 1998).



Figure 6-30 Adapted GAB equation best fit for measured pasta loss factor at 915MHz





Figure 6-31 Adapted GAB equation best fit for measured pasta dielectric constant at 915MHz

Model	Parameter	Pasta loss factor	Pasta dielectric constant
	Mo	0.232982	0.23671
	С	140	233.051
	К	0.0454407	0.00815148
GAB	ε <sub>0m</sub> "	0.0272279	1.4497
	MRE [%]	4.46	1.07
	RSS	2.68E-03	1.33E-02

Table 6-10 Best equation fit for moisture dependant pasta loss factor and dielectric constant

# 6.5.3 Dielectric response in relation to microwave drying of pasta

The microwave drying process for pasta starts at a moisture content of 0.18kg.kg<sup>-1</sup> and finishes at a moisture content of 0.13kg.kg<sup>-1</sup> as reported by Schiffmann (2001a). The start and end points of the microwave drying process is shown relative to the loss factor in Figure 6-32. The loss factor was only measured up to 0.17kg.kg<sup>-1</sup> wet basis due to moulding issues at higher moisture contents.



Figure 6-32 Loss factor at 20°C, 915MHz, for pasta, showing start and end of the proposed microwave drying process

Using the same methodology as Scenario 1, 2, and 3, presented in Section 6.3.3, the ability for the moisture levelling mechanism to counteract uneven electric field exposure was analysed for particulate pasta using the adapted GAB equation fit for loss factor presented in the previous section. It was calculated how much extra moisture is required so that pasta particulate receiving 10% lower intensity electric field exposure would receive 10% more heating due to moisture levelling, where the dwell time was such that on average 50% of the required microwave dehydration has taken place. The additional moisture required was calculated to be 0.0177kg.kg<sup>-1</sup> as shown in the Table 6-11. Hence, for an average moisture content of 0.155kg.kg<sup>-1</sup> at a given dwell time, a moisture content of 0.173kg.kg<sup>-1</sup> is required to obtain 10% additional corrective heat for pasta particulate that is exposed to 10% lower intensity electric field strength. However, unlike the finish drying applications for potato crisps and biscuits, the dielectric constant of particulate pasta varies significantly over the moisture contents analysed. Pasta particulate with 0.173kg.kg<sup>-1</sup> wet basis moisture content has a dielectric constant which is 15.8% higher than pasta particulate with 0.155kg.kg<sup>-1</sup> wet basis moisture content. The higher dielectric constant will have a significant effect on the electric field relative to the 10% difference in electric field intensity analysed. Moisture dependant dielectric measurements of the solid pasta material, rather than the bulk particulate material, would enable better microwave heating analysis to be carried out. This would enable differences in dielectric constant to be taken into account, using the analysis approach discussed in Section 6.3.3.2, where the internal electric field was quantified using dielectric measurements of solid potato taken by Mudgett et al. (1980) (results are presented in Table 6-6 and Table 6-7). However, the bulk pasta particulate analysis shown in Table 6-11 presents a best case in terms of moisture levelling, and highlights that moisture levelling cannot be used to counteract large differences in electric field exposure to achieve tight tolerances less than  $\pm 0.01$ kg.kg<sup>-1</sup> of the final moisture content. Although this analysis highlights the care that must be taken in applicator design to maximise uniformity of electric field exposure, the duration of microwave exposure is relatively long at 12 minutes, and there is also a stabilisation period after microwave heating of approximately 45 minutes (Schiffmann, 2001a). The stabilisation period provides time for the moisture to distribute more evenly in the pasta particulate. Further stabilisation of moisture content can of course occur after packaging if no spoilage/deterioration occurs in the higher moisture content pasta during the stabilisation period. Large scale industrial drying of pasta cited in literature is carried out at microwave frequencies, which suggests that a tight enough tolerance of final moisture content can be achieved using microwave applicators (Metaxas & Meredith, 1993; Schiffmann, 2001a).

### **Boundary conditions**

Average food particulate moisture content = 0.155kg.kg<sup>-1</sup> wet basis Wet pasta particulate has 10% lower intensity electric field exposure

### Calculation

Extra moisture required for wet pasta particulate to have 10% more heating than received by pasta particulate with the average moisture content of 0.155kg.kg<sup>-1</sup> wet basis.

### Result

Extra 0.0177kg.kg<sup>-1</sup> wet basis required Hence wet pasta particulate = 0.173kg.kg<sup>-1</sup> wet basis

Moisture content wet basis [%]	Loss factor	Dielectric constant	Electric field of bulk material [Vm <sup>-1</sup> ]	Pd [Wm <sup>-3</sup> ]
0.155	0.3234	2.4180	1	0.0165
0.173	0.4392	2.8000	0.9	0.0181
Difference in moisture content =				0.0177
Increase in heating for moisture levelling =				10%

Table 6-11 Pasta particulate moisture levelling scenario - 10% increase in heating for wet pasta particulate which has 10% lower intensity electric field exposure than average. Moisture content of average particulate material was taken as 0.155kg.kg<sup>-1</sup> wet basis

Bradshaw et al. (1998) classed low loss materials as having a loss factor less than 0.01, and referred to these materials as poor candidates for microwave heating applications. Pasta has a loss factor higher than 0.44 at the start of the proposed microwave process, with a loss factor of 0.2 at the end of the process and should therefore be an easy dielectric load to heat.

### 6.5.4 Quality of microwave dried pasta

A study by Altan & Maskan (2005) showed that starch was not completely gelatinized during microwave drying, an observation that was also noted by De Pilli et al. (2009). De Pilli et al. (2009) found that conventionally dried pasta cooked in 10 minutes, while micorwave dried pasta took 15 minutes to cook. The increase in cooking time of pasta dried by microwaves could be attributed to the delay of starch swelling, and, subsequetnly, a longer time for gelatinization was required during cooking (De Pilli, et al., 2009). A longer rehydration time is obviously undesirable for consumers, and could significantly affect sales of microwaved pasta. Berteli & Marsaioli Jr. (2005) found that microwave drying of pasta had no negative affect on the appearance or quality of the final product. The microwave process is however usually compared to the low temperature (LT) traditional technique. High tempeature (HT) conventional processing, which was introduced in the 1970s and early

1980s quickly baecame the process of chioce for most pasta manufacturers worldwide. Ultra high temperature (UTH) air drying was introduced in the late 1990s and has become common. UHT drying reporedly produces pasta products with cooking quality and colour equal to or better than that obtained with HT drying. However, with higher temperature drying, particularly UHT drying, there is the potential for the development of a brown or reddish colour due to excessive non-enzymatic browning i.e. the Maillard reaction or the "burning" of pasta. This can have a negative impact on protein nutritional quality (Marchylo & Dexter, 2001). Dexter et al. (1981) found that HT drying applied to the latter stages of drying produced spaghetti that was equal to or better than LT spaghetti in colour quality while also exhibiting improved strand strength and superior cooking properties.

# 6.5.5 Energy requirement and commercial feasibility of proposed pasta drying process

It has been reported by Schiffmann (2001a) that pasta can be dried in 1.5 hours by utilising microwave drying. Microwave drying of pasta is nearly always compared in literature to the traditional low temperature (LT) process, which takes 8-12 hours (Schiffmann, 2001a; Berteli & Marsaioli Jr., 2005; Altan & Maskan, 2005). The long drying time of the traditional process leads to very long driers which would have had a very large external surface. The large external surface area would have contributed to thermal inefficiencies. These inefficiencies meant that the microwave drying process was able to reduce operational costs by 26% compared to LT air drying (Berteli & Marsaioli Jr., 2005).

The efficiency of a microwave magnetron can be 88%, with 97% of the heat being applied to the food (E2V Technologies Limited, 2002; Industrial Microwave Systems, L.L.C., 2012). However, electricity is 3.2 times more expensive than gas. Hence, the fuel efficiency of a conventional air drying system would have to be less than 27% for conventional drying to cost more than a microwave system in terms of energy usage. It is certainly possible that a large continuous dryer processing food with an exposure time of 8-12 hours would have an efficiency of less than 27%, contributing to the 26% improvement in operational costs quoted by Berteli & Marsaioli Jr. (2005).

Whilst the drying time for LT is 12 hours for long goods and 9 hours for short goods, high temperature (HT) air drying reduces the processing time to 8 hours for long goods and 5 hours for short goods, while ultra high temperature (UHT) processing reduces the processing time to 4-5 hours for long goods and 2-3 hours for short goods (Marchylo & Dexter, 2001). The reduction in drying time alone would have greatly improved the efficiency of the conventional air drying process. Modern thermal insulation will also have an effect at improving efficiency. Pavan pasta dryers utilise 70mm of Polyurethane foam and 30mm of horizontal fibre rock wool sandwich between two 1mm layers of stainless steel (Pavan Group, 2016). Conveyor driers typically have an efficiency of 40-60% (Strumiłło, et al., 2006), and it is therefore highly likely that the modern HT and UHT pasta dryers have an efficiency over the breakeven threshold of 27%. An important general observation is that most dry foods are thin so that they are edible, or they are thin to reduce rehydration times. Hence, the advantage of microwave volumetric heating can be limited, as it should be possible to dry most foods conventionally with an efficiency higher than 27% using modern technology (Zbicinski, et al., 1992; Evin, 1992; Foster, 1973; Fisher, et al., 2002). This would go a long way to explaining why microwave pasta drying has fallen into disuse (Schiffmann, 2001a).

Pavan have built UHT pasta production lines that deliver 6,500kg.h<sup>-1</sup> of finished product. The number of 100kW generators required can be calculated using the same methodology used to calculate the number of generators for potato crisp processing (Section 6.3.5 and Appendix G). Assuming that microwave pasta dries the product from 0.18kg.kg<sup>-1</sup> down to 0.13kg.kg<sup>-1</sup> wet basis, six 100kW generators are required for a production rate of 6,500kg.h<sup>-1</sup> of finished product. In order to calculate the energy required for warm up, the heat capacity of zero moisture pasta was taken as 532Jkg<sup>-1</sup>K<sup>-1</sup>, which was calculated using data presented by De La Peña & Manthey (2014). The six

generators alone would cost approximately £400,000 (Smith, 2016), the applicator would incur additional capital costs.

In conclusion, the running cost of microwave drying is likely to be higher than HT and UHT, purely based on the cost of energy. Conventional drying must be less than 27% efficient for microwave drying to be more cost effective, and conveyor driers typically have an efficiency of 40-60% (Strumiłło, et al., 2006).

### 6.6 Conclusion

The moisture dependant dielectric response of potato crisps, digestive biscuits, and pasta have been measured. The measured moisture dependant dielectric responses were fitted to the adapted water activity equations presented in Chapter 3, and the best fits were presented. In each case the adapted GAB equation provided the best fit. The Mean Relative Error was no higher than 4.5% for each adapted GAB equation fit, while the average adapted GAB fit had a Mean Relative Error of just 1.5%. These equations can be used in microwave drying models for optimisation of the relevant applicator. This should provide a big improvement compared to the current trial and error method for applicator development.

For potato crisps, biscuits, and pasta, the moisture dependent dielectric behaviour was compared to the state of the water as defined by the sorption isotherm. As with the published data of hydrocolloids with low sugar content analysed in Chapter 3, there is a strong increase in loss factor associated with the transition from multilayer to solution. This helps to re-enforce the novel conclusions reached in Chapter 3, that there is a correlation between the points of inflection of the loss factor and the state of the water as defined by the sorption isotherm. There is however, more of a transition in loss factor observed during the multilayer than observed in published data on hydrocolloids with low sugar content.

Analysis showed that the moisture levelling mechanism is weak compared to variations in exposure to electric field intensity. Hence, for finish drying applications, a uniform electric field exposure is vital, and requires careful applicator design. This conclusion concurs with industry practice, where the most successful RF/MW drying application by far is RF finish drying of biscuits which has approximately 400 installations. An even field is relatively easy to achieve using RF driers. What was perhaps surprising regarding the moisture levelling analysis was that selective heating can actually be stronger below the critical loss factor, contrary to common belief. This again concurs with the results seen in industry, where moisture levelling is deemed to be very effective at low moisture contents, which are below the critical moisture content.

The finish drying of potato crisps looks to be a future application with real promise. Finish drying can commence at a moisture content of 0.04kg.kg<sup>-1</sup>, which is below the glass transition point and the structure will be set. This should enable a reduction in carcinogenic acrylamide of 50%. It can also be deemed to be financial viable, with the commercial feasibility analysis showing that it can be done with one 100kW 915MHz generator. The extra energy cost would only be 0.014p per packet of crisps. It would cost 0.051p per packet for 1 year that includes amortization of the generator. Other considerations such as applicator and upkeep costs as well as reliability are required for a full commercial analysis. The loss factor of finished potato crisps is only 0.0149, which would require careful applicator design.

Potato crisp production using explosive microwave dehydration from 0.82kg.kg<sup>-1</sup> down to 0.04kg.kg<sup>-1</sup> wet basis was also assessed. There is risk of thermal runaway through the initial stages of dehydration. However, the biggest problem was that it would require 96 100kW 915MHz generators to replace one 3000kg.h<sup>-1</sup> fryer. Even if a fat tax was able to cover the considerable costs (the generators alone would cost £6.7 million), the proposed drying solution would have severe practical limitations including a large footprint, complexity, and maintenance.

Pasta drying was previously regarded as one of the most successful industrial applications of microwaves in the food industry. There are now few, if any microwave pasta dryers still in operation. The reasons for this appear to be twofold. The first reason is that conventional technology has seen considerable advancement with the instruction of HT and UHT conventional drying which reduced drying rates from 9 hours to 5 hours and 2-3 hours respectively for short goods. Hence the 26% saving in operational cost made by the conventional LT process is likely to be eroded to the point that microwave processing is now more expensive than conventional techniques. The second reason is that the microwave process dries at a relatively low temperature, so gelatinisation does not fully occur. This leads to rehydration times of 15 minutes compared with a rehydration time of 10 minutes pasta dried using state of the art conventional air drying processes. Hence, it is perhaps not surprising that this microwave process has fallen into disuse.

## **Chapter 7 Conclusion and future work**

## 7.1 Conclusion

This study identified two state of the art microwave applications for investigation:

- Microwave explosive dehydration of potato crisps to replace frying
- Microwave finish drying of potato crisps.

Two existing RF/MW were also analysed:

- RF/MW finish drying of biscuits
- Microwave pasta drying

Although this thesis focused on the four microwave applications listed above, on a general basis, this thesis attempts to improve the understanding of industrial microwave and RF drying of food. There have been a large number of microwave and RF applications in the food industry that have either failed, or fallen into disuse, and a better understanding of the science is required. This study gives new insights into the science of microwave drying, presents a novel Micro-CT method for assessing the porosity of irregular shaped thin heterogeneous foods, and presents some of the practical and commercial limitations of microwave drying.

The new and novel aspects presented in this thesis, as well as the advancements in understanding of the physics of microwave drying of food is summarised as follows:

• The proposed microwave finish drying of potato crisps is a new and novel application. Acrylamide levels could be reduced by approximately 50% and production could be doubled.

- The present study discovered that moisture dependant dielectric properties are dependent on the state of the water as defined by the sorption isotherms. This discovery was established by analysing disparate data of moisture dependant water activity (sorption isotherms) and moisture dependant dielectrics to find that a correlation exists between the points of inflection. It was found that the relationship between the state of the water and the dielectric behaviour is different for fruits compared to low sugar hydrocolloids.
- Water activity equations have been modified to successfully describe moisture dependant dielectric properties. This is a very significant advancement in science, as these equations can be utilised in microwave drying models to improve optimisation of RF and microwave drying processes. Currently RF and microwave drying application solutions are developed on a physical trial and error basis. The development of these moisture dependant dielectric equations should ultimately reduce development costs and improve the likelyhood of success.
- A new technique was developed for drying and saturating food, which can be used for both water activity and dielectric measurements. For drying, continuous exposure to dry nitrogen is used, and for saturation a continuous nitrogen supply is passed through deionised water in a dreschel bottle before being exposed to the food. The oil content is kept stable using this technique (vital for gravimetric moisture measurements) and mould growth is suppressed due to the lack of oxygen.
- A new and novel technique for quantifying the porosity of thin irregularly shaped food has been established. This was a Micro-CT technique that made use of a CAD wrap operation to determine the encapsulating volume. This technique can be used as a diagnostic tool to assess processing changes. Porosity of kettle and continuous fried

crisps were quantified accurately for the first time using this method. The lower porosity of the kettle fried crisp results in the crunchy taste for which it is renowned.

- Sorption isotherm measurements of potato crisps have shown that the gelatinisation of starch has a negligible effect on the stability of the food at low moisture contents. This is a significant discovery as food stability and shelf life is of prime commercial importance.
- The isosteric heats calculated using the measured sorption isotherms were used to deduce the cost of energy of the proposed potato crisp and pasta microwave processes, and to deduce the total number of generators required. The isosteric heat is very significant compared to the latent heat and must be considered when calculating energy requirements for drying.
- The moisture levelling effect has been quantified with respect to variation in exposure to electric field intensity. It was found that the moisture levelling effect is relatively weak compared to undesirable variations in electric field exposure. Hence it is of vital importance for finish drying applications that the applicator is able to provide an electric field that provides even exposure to product passing through. It was found that the moisture levelling effect can be stronger below the critical dielectric loss factor, rather than above it, contrary to common belief. Dielectric properties have been measured for potato crisps, biscuits, and pasta. Knowledge of these dielectric loads is important for the design of the relevant microwave applicators. The dielectric properties of finished potato crisps were particularly low, and the design of the corresponding finish drying applicator would require careful attention.
- Cost analysis was carried out using the isosteric heat calculated from the measured sorption isotherms. This cost analysis showed that microwave applications that remove large quantities of water have

limited viability due to the large number of generators required. For the microwave explosive dehydration of potato crisps, 96 100kW generators would be required to replace one 3000kg/hr fryer. Not only would this be expensive, but it would be a very impractical drying solution requiring a large factory footprint. As a rule of thumb, cost analysis has shown that conventional drying systems need to be less than 27% efficient for microwave drying to be a cost effective alternative. It is thought that modern hot air pasta dryers are now above this threshold, leading to the decline in microwave pasta drying. It can generally be assumed that microwave drying in the food industry is more expensive than the equivalent conventional process, both in capital equipment and running costs

As well as the novel science listed, there were some general observations of note. Firstly, it is very difficult to replace a conventional drying process with a microwave drying process without adversely affecting an important food characteristic. This is evident in pasta drying, where the starch is not gelatinised due to the low drying temperature, which leads to long rehydration times. The use of RF/MW in the food industry has been problematic, and is seen as a "high tech solution" requiring high levels of expertise, resulting in extreme caution regarding new potential applications. In a lot of instances RF/MW has been used as a temporary solution, and has been phased out wherever possible in preference to lower cost conventional drying technologies. Most very dry foodstuff is thin so that it is edible, eroding some of the competitive advantage of volumetric heating. The use of RF/MW drying can only really be justified by improvements in product quality.

### 7.2 Future work

There are a number of aspects arising from this study that require further investigation:

- The RF/MW finish drying of potato crisps is the application studied that most warrants further research and development. It is a novel application that can potentially reduce carcinogenic acrylamide levels by 50% whilst doubling production, and is commercially viable and relatively simple to implement. The ability to achieve potato crisp finish drying limited to 105°C with a 2-3 minute dwell time was deduced from a previous MW finish drying application to reduce browning of high sugar content potatoes. The 50% reduction in acrylamide levels was deduced by published studies relating frying time and temperature to acrylamide levels. However the data presented in literature did not contain enough data points to get an accurate figure on the reduction in acrylamide that can be achieved with a reduced frying time. Experiments using an RF dryer with a feed of partially fried potato crisps should be carried out to properly quantify the reduction in acrylamide levels that can be achieved using the proposed process of frying to 0.04kg.kg<sup>-1</sup> wet basis, and RF drying to 0.017kg.kg<sup>-1</sup> wet basis.
- Water activity equations were adapted in this study to describe moisture dependant dielectric properties using curve fitting. It would be very useful for modelling purposes for these equations to be extended to include temperature dependence.
- All the moisture dependant dielectric measurements carried out in this study of potato crisps, biscuits, and pasta were carried out at room temperature. There are very few examples in literature of moisture dependent properties being measured at different temperatures. Measuring the dielectric properties over relevant moisture content ranges at different temperatures would help improve understanding of

dielectric behaviour. This data could be compared to sorption isotherm measurements carried out at different temperatures. The ability to selectively heat dielectric material to counteract variation in electric field exposure should be assessed at high temperatures. Temperature dependant measurements would also allow further assessment of thermal runaway risks.

• One of the potential issues of explosive dehydration of potato crisps is arcing. The loss factor at the end of the process is just 0.022. The heating required is pre-determined as the microwave process must follow the dehydration curve of the frying process to create the desired potato crisp structure. This lack of flexibility with regards to microwave heating could be an issue if arcing is found to occur. The present study has calculated that the power required for this explosive microwave dehydration process is 23.1kW.kg<sup>-1</sup>. Analysis and practical experiments should be carried out to assess whether or not arcing is an issue with these power levels at 915MHz.

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<u>325g?langId=44&storeId=10151&krypto=J9SL2bOPEzbq7h4C2OeYqYym0G</u> wqyPciZQniB8TOgWOHhueYFIK3u6tVtcXKkHWYjwXQ%2BslAUAeX2H T2RSrZ11%2F6yR9AxoaHt%2FRloXfxix3YJuHgGvzx [Accessed 5 October 2016].

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# Appendix A – Fitting dielectric equations to experimental data presented in literature

		_			Chickpea	Chickpea
	D	Freeze			flour,	flour,
Model	Param-	dried potato	Potato	Wheat flour	27MHz,	27MHz,
woder	eter	•			20degC	90degC
	M <sub>0</sub>	0.2394	0.376197	0.413	0.148977	0.144337
	С	81.6922	39.9102	11.558	313.984	347.941
	К	0.0222	0.004811	0.0012788	0.447778	0.001272
GAB	ε <sub>0m</sub> "	0.06	0.05	0.673	0.157093	1.621
	U	23.5457	139.253	477.754	0	0
	S	2.5409	2.182	1.425	1	1
	MRE [%]	7.83	126.95	15.03	1.10	0.94
	RSS	2.03	346.64	11.89	3.21E-05	0.07
	А	0.3185	0.4658	0.497	0.428069	0.173985
	В	0.1911	0.1784	0.256	0.179429	0.105527
	ε <sub>0m</sub> "	0.1	0.05	0.673	0.16	4.27
Oswin	С	22.1806	61.9981	164.826	35.6566	286.905
OSWIII	U	11.2911	67.6714	158.232	0	0
	S	5.1281	3.2355	2.931	1	1
	MRE [%]	7.65	15.65	10.29	4.72	0.98
	RSS	2.89	339.56	13.32	2.81E-03	0.03
	А	228.852	36.0194	12.701	48.7627	7570170
	В	4.902	4.9485	3.535	5.00766	9.07084
	ε <sub>0m</sub> "	0.1	0.1	0.673	0.16	4.27
Henderson	С	20.372	47.0835	103.459	33.496	245.395
nenderson	U	9.525	51.8995	105.974	0	0
	S	7.203	3.9995	3.98	1	1
	MRE [%]	7.73	23.12	8.61	4.95	1.13
	RSS	4.06	323.10	12.25	1.31E-03	0.03
	А	0.289	0.4211	0.432	0.25209	0.177623
	В	-0.0951	-0.1334	-0.181	-0.09187	-0.04331
	Β ε <sub>0m</sub> "	-0.0951 0.1	-0.1334 0.05	-0.181 0.673	-0.09187 0.14725	-0.04331 4.27
Chung-						
Chung- Pfost	ε <sub>0m</sub> "	0.1	0.05	0.673	0.14725	4.27
-	ε <sub>0m</sub> " C	0.1 22.715	0.05 67.7702	0.673 172.732	0.14725 3.25663	4.27 396.106
-	ε <sub>0m</sub> " C U	0.1 22.715 11.8636	0.05 67.7702 74.3803	0.673 172.732 169.631	0.14725 3.25663 0	4.27 396.106 0

		Freeze			Chickpea flour,	Chickpea flour,
	Param-	dried		Wheat	27MHz,	27MHz,
Model	eter	potato	Potato	flour	20degC	90degC
	А	-0.2	-0.2	-0.348	-0.2	-0.2
	В	20.9033	23.4976	19	27.7133	60.403
	ε <sub>0m</sub> "	0.1	0.05	0.673	0.16	4.27
	С	10.5181	7.4968	7.497	16.0276	23.0884
Chen	D	27.7441	82.7747	177.709	2.0996	483.805
	U	11.8636	74.3803	120	0	0
	S	4.5917	3.0704	3.07	1	1
	MRE [%]	9.53	22.72	18.08	0.12	0.14
	RSS	3.04	351.84	106.39	4.92E-07	5.36E-0
	γ	31.6472	90.5004	271.993	7.16737	694.493
	Α	0.0755	0.1591	0.403	0.23706	0.01932
	r	-2.2729	-2.381	-1.568	-1.47887	-2.5496
Ferro-	ε <sub>0m</sub> "	0.1	0.05	0.673	0.16	4.27
Fontan	U	18.5434	87.7898	189.657	0	0
	S	2.9831	3.2677	3.235	1	1
	MRE [%]	14.17	21.36	17.01	0.05	0.02
	RSS	3.10	369.21	58.29	8.42E-08	9.23E-0
Lowe	st RSS	2.03	323.10	11.89	8.42E-08	9.23E-0
		<b>C</b> A D		<b>C</b> A <b>D</b>	Ferro	Ferro
Lowest R	ss model	GAB	Henderson	GAB	Fontan	Fontan
Lowes	t MRE	7.65	15.65	8.61	0.05	0.02
l owest M	RE model	Oswin	Oswin	Henderson	Ferro Fontan	Ferro Fontan
	ine model	0.50011	0.500	i chaci son	rontan	i ontan

Table A-1 Best fit equations and error for moisture dependant loss factor of freeze dried potato, potato, wheat flour, chickpea flour

		Chickpea	Chickpea	Yellow dent	Carboxymethy
		flour,	flour,	field corn,	cellulose,
	Paramet	1.8GHz,	1.8GHz,	20MHz,	2.45GHz,
Model	er	20degC	90degC	24degC	20degC
	M <sub>0</sub>	0.15495	0.115617	0.2458	0.254672
	С	154.075	480.983	75.5168	30.83
			0.050152		
	K	0.814818	7	0.1287	0.346563
GAB	ε <sub>0m</sub> "	0.187	1.4576	0.3176	0.0239977
	U	0	0	0	0
	S	1	1	1.425	1.425
	MRE [%]	0.99	0.38	4.87	2.94
	RSS	0.00	0.00	0.01	6.23E-05
	Α	0.236836	0.15325	0.319799	2.342
	В	0.186796	0.11282	0.182564	0.367519
	ε <sub>0m</sub> "	0.19	1.54	0.391672	0.0305853
Oswin	С	0.97446	9.41576	3.86791	172.46
OSWIII	U	0	0	0	0
	S	1	1	2.931	2.931
	MRE [%]	0.48	0.46	3.55	7.51
	RSS	0.00	0.00	0.00	6.43E-04
	Α	3387.83	1419050	322.814	2.34463
	В	5.31254	7.71968	4.99957	3.44696
	ε <sub>0m</sub> "	0.1893	1.54	0.383664	0.0299
Henderson	С	0.58593	8.85282	2.95338	16.4204
rienderson	U	0	0	0	0
	S	1	3.98	3.98	3.98
	MRE [%]	0.55	0.69	4.14	24.19
	RSS	0.00	0.00	0.00	9.11E-03
	А	0.25841	0.143628	0.3022	1.08415
	В	-0.09557	-0.029444	-0.09684	-0.508967
	ε <sub>0m</sub> "	0.18943	1.54	0.4079	0.0104272
Chung-	<u> </u>	1.7345	9.86506	4.39396	69.6228
Pfost	U	0	0	0	0
	S	1	1	1	1
		0.89	0.02	2.37	2.30
	MRE [%]	0.05	0.02	2.07	=

		Chickpea	Chickpea	Yellow dent	Carboxymeth
		flour,	flour,	field corn,	cellulose,
		1.8GHz,	1.8GHz,	20MHz,	2.45GHz,
Model	Parameter	20degC	90degC	24degC	20degC
	А	-0.2	-0.2	-0.2	-0.2
	В	14.8124	131.372	22.6635	8.41564
	ε <sub>0m</sub> "	0.19	1.54	0.4079	0.0104272
	С	10.2752	33.963	10.3267	1.96477
Chen	D	2.27264	12.0492	5.3668	85.0375
	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	0.50	0.02	2.36	2.30
	RSS	0.00	0.00	0.00	4.51E-05
	γ	328.446	11.1322	8.53813	16391.7
	А	2.83126	0.0003407	0.18407	7.86524
	r	-0.569336	-4.16019	-1.84519	-0.213432
Ferro-	ε <sub>0m</sub> "	0.19	1.54	0.41618	0.0299639
Fontan	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	0.38	0.00	1.46	4.19
	RSS	0.00	0.00	0.00	3.21E-03
Lowe	est RSS	0.00	0.00	0.00	4.51E-05
		Ferro	Ferro	Ferro	
Lowest I	RSS model	Fontan	Fontan	Fontan	Chen
		0.38	0.00	1.46	
	st MRE				2.30

Table A-2 Best fit equations and error for moisture dependant loss factor of chickpea flour, yellow dent field corn, and carboxymethyl cellulose

Ferro

Fontan

Ferro

Fontan

Chen

Ferro

Fontan

Lowest MRE model

		Hard red	Hard red	Hard red	Hard red
		winter	winter	winter	winter
	-	wheat,	wheat,	wheat,	wheat,
	Param-	28MHz,	100MHz,	1GHz,	12.1GHz,
Model	eter	24degC	24degC	24degC	24degC
	M <sub>0</sub>	0.344873	0.349546	0.403125	0.129941
	C	203.973	354.812	636.836	14.8015
	К	0.0151858	0.00852868	0.00468425	0.851807
GAB	ε <sub>0m</sub> "	0.296256	0.157668	0.112751	0.0604961
	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	4.73	14.24	15.86	1.49
	RSS	0.01	0.09	0.04	6.49E-04
	А	5.94481	128.042	200	0.185644
	В	0.39494	1.14929	1.2	0.356381
	ε <sub>0m</sub> "	0.361881	0.0290887	0.05	0.081624
Oswin	С	1733.86	150.559	114	0.829525
OSWIII	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	5.26	7.47	11.22	2.23
	RSS	0.02	0.02	0.02	7.94E-04
	А	2.1199	0.1683	0.2221	76.5329
	В	2.5511	1.3272	1.3301	2.53812
	ε <sub>0m</sub> "	0.3025	0.13	0.100043	0.0786965
Henderson	С	11.3655	22.3422	12.2164	0.640597
Tienderson	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	6.54	7.35	11.76	1.89
	RSS	0.02	0.02	0.02	6.69E-04
	А	1.482	2.036	0.101262	0.143608
	В	-0.83477	-1.063	-0.037481	-0.078064
	ε <sub>0m</sub> "	0.1283	-0.012	0.118837	0.074957
Chung-	C	58.2089	148.312	0.332885	0.755439
Pfost	U	0	0	0	0
	S	1	1	1	1
	S MRE [%]	<u>1</u> 5.05	<u> </u>	<b>6.34</b>	<u>1</u> 2.04

		Hard red	Hard red	Hard red	Hard red
		winter	winter	winter	winter
		wheat,	wheat,	wheat,	wheat,
	Param-	28MHz,	100MHz,	1GHz,	12.1GHz,
Model	eter	24degC	24degC	24degC	24degC
	А	-0.2	-0.2	-0.2	-0.2
	В	4.1705	2.7802	14.9054	6.2941
	ε <sub>0m</sub> "	0.1213	-0.4613	0.1188	0.075
	С	1.5069	0.8481	26.6804	12.8099
Chen	D	14.4146	12.5504	0.4066	0.9227
	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	6.46	7.63	6.34	2.04
	RSS	0.05	0.01	0.01	7.57E-04
	γ	756.552	47.3801	0.368	1.74091
	А	5.23	3.71787	0.00295	0.28366
	r	-0.23	-0.128881	-2.58964	-0.97448
Ferro-	ε <sub>0m</sub> "	0.31	0.0326455	0.12387	0.089
Fontan	U	0	0	0	0
	S	1	1	1	1
	MRE [%]	5.23	3.76	6.34	3.42
	RSS	0.02	0.01	0.01	1.29E-03
Low	est RSS	0.01	0.01	0.01	6.49E-04
Lowest	RSS model	GAB	Ferro Fontan	Chen	GAB
		4.73	3.76	6.34	1.49
Lowe	SUVIRE	4.75	5.70	0.54	1.45

Table A-3 Best fit equations and error for moisture dependant loss factor of hard red winter wheat

		Apples,	Apples,	Grapes,
		915MHz,	2.45GHz,	2.45GHz,
Model	Parameter	60degC	60degC	25degC
	$M_0$	0.164872	0.1197	0.149865
	C	11.9272	50.0798	114.694
	К	0.0881887	0.1151	0.0393911
GAB	ε <sub>0m</sub> "	0.475638	0.4929	0.01
	U	0	0	7.83352
	S	1	1	4.02769
	MRE [%]	1.11	3.19	0.96
	RSS	0.02	0.24	0.01
	А	0.26127	0.20605	0.2744
	В	0.45394	0.30369	0.2543
	ε <sub>0m</sub> "	0.5	0.54151	0.5
Oswin	С	9.64016	7.04807	18.8393
Cowin	U	0	0	3.7319
	S	1	1	3.8989
	MRE [%]	1.97	2.08	0.01
	RSS	0.03	0.03	0.00
	A	8.82226	28.9077	27.9119
	В	1.79028	2.352	2.8882
	ε <sub>0m</sub> "	0.46179	0.3962	0.1
Henderson	С	8.64366	6.9376	18.2055
	U	0	0	1.8519
	S	1	1	3.7274
	MRE [%]	1.65	1.68	0.01
	RSS	0.06	0.08	0.00
	A	0.18923	0.17253	0.229546
	В	-0.1362	-0.08553	-0.098623
	ε <sub>0m</sub> "	0.27428	0.49017	0.1
Chung-	C	8.98557	6.93145	18.822
-	C			
Pfost	U	0	0	3.0279
Pfost		0	0 1	3.0279
Pfost	U			

		Apples,	Apples,	Grapes,
		915MHz,	2.45GHz,	2.45GHz,
Model	Parameter	60degC	60degC	25degC
	A	-0.2	-0.2	-0.2
	В	4.0121	7.5166	9.9624
	ε <sub>0m</sub> "	0.2743	0.4902	0.1
	С	7.3419	11.6913	9.9718
Chen	D	10.975	8.4661	23.0762
	U	0	0	2.9022
	S	1	1	3.0963
	MRE [%]	1.10	1.73	0.42
	RSS	0.03	0.03	0.02
	γ	10.7284	7.38927	26.4428
	А	0.1566	0.03107	0.0968
	r	-1.2679	-1.9858	-1.7111
Ferro-	ε <sub>0m</sub> "	0.8371	0.62957	0.1
Fontan	U	0	0	9.5438
	S	1	1	2.2585
	MRE [%]	2.69	3.99	1.25
	RSS	0.07	0.14	0.11
Lowe	est RSS	0.02	0.03	0.00
Lowest	RSS model	GAB	Chen	Oswin
Lowe	st MRE	1.10	1.68	0.01
Lowest N	/IRE model	Chung-Pfost	Henderson	Chung-Pfos

Table A-4 Best fit equations and error for moisture dependant loss factor of apples and grapes

### Appendix B – Derivation of equation used to analytically calculate the porosity of potato crisps

The potato crisp consists of solid potato, oil, water, and air (Rodríguez-Ramíez, et al., 2012). Therefore the total mass of the potato crisp is described as follows where m is the mass, subscript T is the total, subscript s denotes the solid material, subscript w is the moisture, subscript o is the oil, and subscript g is the air within the porous media:

$$m_T = m_s + m_w + m_o + m_g$$
 Equation B-1

This can be expressed in terms of volume and density as shown below, where  $\rho$  is density, V is volume, and v is specific volume.

$$m_T = \rho_s V_s + \rho_w V_w + \rho_o V_o + \rho_g V_g$$
  

$$\frac{m_T}{V_T} = \rho_s \frac{V_s}{V_T} + \rho_w \frac{V_w}{V_T} + \rho_o \frac{V_o}{V_T} + \rho_g \frac{V_g}{V_T}$$
  

$$\rho_T = \rho_s v_s + \rho_w v_w + \rho_o v_o + \rho_g v_g$$
Equation B-2

If the crisp is made up of oil, water, and solid material, then the remaining volume fraction is made up of gases as described below:

$$v_g = 1 - v_o - v_w - v_s$$
 Equation B-3

Substituting Equation B-3 into Equation B-2

$$\rho_T = \rho_s v_s + \rho_w v_w + \rho_o v_o + \rho_g (1 - v_o - v_w - v_s)$$
 Equation B-4

Effective porosity is defined in Equation B-5

$$\phi_{eff} = 1 - v_s$$
 Equation B-5

Substituting Equation B-5 into Equation B-4:

$$\rho_T = \rho_s \left( 1 - \phi_{eff} \right) + \rho_w v_w + \rho_o v_o + \rho_s \left( \phi_{eff} - v_o - v_w \right)$$
Equation B-6

The specific volume of oil and water can be described in terms of mass fraction as shown in Equation B-7 and Equation B-8 respectively, where f is mass fraction.

$$v_o = f_o(\rho_T / \rho_o)$$
 Equation B-7

$$v_w = f_w (\rho_T / \rho_w)$$
 Equation B-8

Substituting Equation B-7 and Equation B-8 into Equation B-6 and then rearranging to make effective porosity the subject.

$$\rho_{T} = \rho_{s} \left( 1 - \phi_{eff} \right) + \rho_{T} f_{w} + \rho_{T} f_{o} + \rho_{g} \left( \phi_{eff} - f_{o} \frac{\rho_{T}}{\rho_{o}} - f_{w} \frac{\rho_{T}}{\rho_{w}} \right)$$

$$\rho_{T} = \frac{\rho_{s} \left( 1 - \phi_{eff} \right) + \rho_{g} \phi_{eff}}{\left( 1 - f_{w} - f_{o} + f_{o} \frac{\rho_{g}}{\rho_{o}} + f_{w} \frac{\rho_{g}}{\rho_{w}} \right)}$$

$$\phi_{eff} = \frac{\rho_{T} \left( 1 - f_{w} - f_{o} + \rho_{g} \frac{f_{w}}{\rho_{w}} + \rho_{g} \frac{f_{o}}{\rho_{o}} \right) - \rho_{s}}{\left( \rho_{g} - \rho_{s} \right)}$$
Equation 4-8

## Appendix C – Derivation of equation used for calculating potato crisp apparent density using oil displacement measurement

The mass of the oil absorbed by the potato crisp is described in Equation C-1, where  $m_c$  is the mass of the potato crisps before they are submerged in oil, and  $m_{c\_ab}$  is the mass of the crisps after they have been removed from the density measuring bottle and the surface oil has been drained off.

$$m_{o\_ab} = m_c - m_{c\_ab}$$
 Equation C-1

The volume of this absorbed oil is calculated below, where  $\rho_o$  is the oil density:

$$V_{o_{ab}} = m_{o_{ab}} / \rho_o$$
 Equation C-2

Substituting Equation C-1 into Equation C-2:

$$V_{o_{ab}} = \left(m_c - m_{c_{ab}}\right) / \rho_o$$
 Equation C-3

The apparent volume of the crisps is the difference between the marked bottle volume ( $V_B$ ) and the oil volume ( $V_o$ ), with the oil absorbed into the crisp added to obtain the apparent volume:

$$V_c = V_B - V_o + V_{o_ab}$$
 Equation C-4

Substituting Equation C-3 into Equation C-4:

$$V_c = V_B - V_o + \frac{m_c - m_{c_ab}}{\rho_o}$$
Equation C-5

The potato crisp density is the mass of the crisps divided by their apparent density, as shown below, where  $m_c$  is the mass of the potato crisps, and  $V_c$  is the volume of the potato crisps:

$$\rho_c = m_c / V_c$$
 Equation C-6

Substituting Equation C-5 into Equation C-6:

$$\rho_c = \frac{m_c}{V_B - V_o + \frac{m_c - m_{c_ab}}{\rho_o}}$$
Equation C-7

The volume of oil is expressed in Equation C-8, where  $m_o$  is the mass of the oil, and  $\rho_o$  is the density of the oil.

$$V_o = m_o / \rho_o$$
 Equation C-8

Substituting Equation C-8 into Equation C-7:

$$\rho_c = \frac{m_c}{V_B - \frac{m_o}{\rho_o} + \frac{m_c - m_{c_ab}}{\rho_o}}$$
Equation C-9

The mass of the oil used in the experiment is described in Equation C-10 where  $m_{TOD}$  is the total mass of the bottle containing crisps and oil, and  $m_B$  is the mass of the empty bottle.

$$m_o = m_{TOD} - m_c - m_B$$
 Equation C-10

Substituting Equation C-10 into Equation C-9:

$$\rho_{c} = \frac{m_{c}}{V_{B} - \frac{m_{TOD} - m_{c} - m_{B}}{\rho_{o}} + \frac{m_{c} - m_{c_{ab}}}{\rho_{o}}}$$
Equation 4-12

## Appendix D – Derivation of equation used to calculate solid potato density

The total density of potato is the total mass divided by the total volume as shown below.

$$\rho_T = \frac{m_T}{V_T}$$
 Equation D-1

Potato is made from solid material, and water, hence Equation D-1 can be described as follows, where  $V_w$  is the volume of the water, and  $V_s$  is the volume of the solid.

$$\rho_T = \frac{m_T}{V_w + V_s}$$
 Equation D-2

The water and solid volume are described in Equation D-3 and Equation D-4 respectively, where  $m_w$  is the mass of the water,  $m_s$  is the mass of the solid,  $\rho_w$  is the density of water, and  $\rho_s$  is the density of the solid material.

$$V_w = \frac{m_w}{\rho_w}$$
Equation D-3

$$V_s = \frac{m_s}{\rho_s}$$
 Equation D-4

Substituting Equation D-3 and Equation D-4 into Equation D-2.

$$\rho_T = \frac{m_T}{\frac{m_w}{\rho_w} + \frac{m_s}{\rho_s}}$$
Equation D-5

The mass of water and the mass of the solid material can be expressed in terms of mass fraction as shown in Equation D-6 and Equation D-7 respectively, where  $f_w$  is the water mass fraction, and  $f_s$  is the solid material mass fraction.

$$m_w = f_w m_T$$
 Equation D-6

$$m_s = f_s m_T$$
 Equation D-7

Substituting Equation D-6 and Equation D-7 into Equation D-5, and then rearranging to make the density of the solid the subject:

$$\rho_T = \frac{m_T}{\frac{f_w}{\rho_w} + \frac{f_s}{\rho_s}}$$

$$\rho_s = \frac{f_s}{\frac{1}{\rho_T} - \frac{f_w}{\rho_w}}$$
Equation D-8

As previously stated, it is assumed that solid potato is made from two constituents, water and solid potato material, which is described mathematically in Equation D-9

$$f_s = 1 - f_w$$
 Equation D-9

Substituting Equation D-9 into Equation D-8:

$$\rho_s = \frac{1 - f_w}{\frac{1}{\rho_T} - \frac{f_w}{\rho_w}}$$
Equation 4-13

## Appendix E – Measured dielectric properties

The measured dielectric properties of potato crisps, biscuits, and pasta are shown in Table E-1, Table E-2, and Table E-3 respectively. The standard deviation is of the three measurements taken, where the food samples were transferred from the load cell into a plastic bag, and transferred back into the load cell in each instance.

Moisture content wet basis [kg.kg <sup>-1</sup> ]	Dielectric constant	Dielectric constant standard deviation	Loss factor	Loss factor standard deviation
0.00170	1.20	0.0066	0.011	0.00087
0.01207	1.21	0.0048	0.014	0.00051
0.02006	1.22	0.0056	0.016	0.00068
0.03037	1.23	0.0094	0.019	0.00142
0.03897	1.24	0.0113	0.021	0.00161
0.04757	1.26	0.0080	0.027	0.00110
0.05617	1.26	0.0045	0.032	0.00061
0.06497	1.28	0.0055	0.037	0.00101
0.06510	1.28	0.0020	0.036	0.00066
0.07360	1.29	0.0077	0.041	0.00151
0.08474	1.33	0.0104	0.055	0.00239
0.08986	1.34	0.0084	0.059	0.00303
0.09828	1.38	0.0022	0.072	0.00166
0.10708	1.40	0.0089	0.082	0.00182
0.11479	1.42	0.0140	0.088	0.00381
0.12269	1.46	0.0108	0.106	0.00551

Table E-1 Dielectric constant and loss factor measurements for potato crisps at 20°C, 915MHz

Moisture content wet basis [kg.kg <sup>-1</sup> ]	Dielectric constant	Dielectric constant standard deviation	Loss factor	Loss factor standard deviation
0.0015	1.34	0.0023	0.017	0.00050
0.0083	1.35	0.0048	0.018	0.00112
0.0133	1.36	0.0050	0.020	0.00059
0.0190	1.37	0.0004	0.021	0.00015
0.0268	1.38	0.0042	0.023	0.00057
0.0363	1.39	0.0074	0.027	0.00081
0.0459	1.41	0.0041	0.030	0.00089
0.0541	1.42	0.0017	0.037	0.00040
0.0631	1.46	0.0056	0.044	0.00166
0.0720	1.48	0.0040	0.053	0.00282
0.0803	1.51	0.0045	0.066	0.00153
0.0883	1.55	0.0052	0.078	0.00197
0.0973	1.60	0.0075	0.096	0.00427
0.1047	1.64	0.0154	0.110	0.00361
0.1128	1.67	0.0090	0.127	0.00061
0.1207	1.72	0.0019	0.144	0.00335

Table E-1 Dielectric constant and loss factor measurements for digestive biscuits at  $20^{\rm o}C, 915 MHz$ 

	Moisture content wet basis [kg.kg <sup>-1</sup> ]	Dielectric constant	Dielectric constant standard deviation	Loss factor	Loss factor standard deviation
	0.050	1.60	0.0086	0.071	0.0013
	0.061	1.64	0.0204	0.083	0.0013
	0.067	1.65	0.0279	0.088	0.0086
-	0.076	1.71	0.0140	0.101	0.0008
	0.085	1.74	0.0097	0.113	0.0019
	0.094	1.80	0.0372	0.136	0.0121
	0.102	1.86	0.0104	0.164	0.0045
	0.110	1.93	0.0183	0.187	0.0057
	0.119	1.98	0.0375	0.206	0.0119
	0.126	2.00	0.0107	0.215	0.0040
	0.133	2.06	0.0114	0.232	0.0055
	0.141	2.15	0.0403	0.240	0.0125
	0.148	2.33	0.0377	0.286	0.0187
	0.156	2.44	0.0246	0.320	0.0166
	0.163	2.51	0.0246	0.346	0.0153
_	0.170	2.74	0.0193	0.440	0.0039

Table E-3 Dielectric constant and loss factor measurements for pasta biscuits at 20°C, 915MHz

# Appendix F – Fitting dielectric equations to measured data

The proposed adapted water activity equations were fitted to the measured dielectric properties using non-linear regression. The best fit parameters for potato crisps, biscuits and pasta are presented in Table F-1, Table F-2, and Table F-3 respectively.

There was no decrease in loss factor gradient with respect to moisture content observed in the dielectric measurements, hence U = 0 and S = 1 for all the adapted equation fits. These parameters are not shown in tables for conciseness.

Model	Parameter	Potato crisps loss factor	Potato crisps dielectric constant
	<u> </u>	0.108224	0.125909
	<u> </u>	17.6525	14.6434
GAB	<u> </u>	2.66442	0.764394
	ε <sub>0m</sub>	0.0109861	1.20209
	MRE [%]	2.08	0.31
	RSS	3.54E-05	3.64E-04
	A	5	5.62144
	B	0.493456	0.522177
Oquin	ε <sub>0m</sub> "	0.013408	1.20751
Oswin	С	172.662	378.342
	MRE [%]	6.16	0.36
	RSS	1.65E-04	5.21E-04
	А	1.14638	2.24545
	В	1.77592	2.17652
Honderser	ε <sub>0m</sub> "	0.0115607	1.2014
Henderson	C	3.07665	11.1555
	MRE [%]	4.94	0.87
	RSS	1.98E-04	2.65E-03
	А	0.231387	0.977883
	В	-0.132819	-0.444378
Chung-	ε <sub>0m</sub>	0.0085851	1.1687
Pfost	C	0.926051	276.84
	MRE [%]	2.51	0.32
	RSS	3.58E-05	3.93E-04
	А	-0.2	-0.2
	В	5.70944	6.72012
	ε <sub>0m</sub> "	0.0085851	1.17563
Chen	C	7.52902	3.53322
	D	1.13108	26.7441
	MRE [%]	2.51	0.33
	RSS	3.58E-05	4.66E-04
	γ	569269	16330600
	Â	11.3442	13.8587
Ferro-	r	-0.152271	-0.122658
Fontan	ε <sub>0m</sub> "	0.0115765	1.20007
	 MRE [%]	12.43	1.05
	RSS	3.02E-04	3.82E-03
Lowes		3.54E-05	3.64E-04
Lowest R		GAB	GAB
Lowes		2.08	0.31
	RE model	GAB	GAB

Table F-1 Loss factor and dielectric constant equation fits for potato crisps

Model	Parameter	Biscuit loss factor	Biscuit dielectric const
-	Mo	0.0926863	0.100759
	С	26.1926	16.641
	K	2.34492	0.737943
GAB	۳. 80m	0.0167847	1.34637
-	MRE [%]	0.97	0.24
	RSS	5.26E-06	3.17E-04
	А	0.209926	5.14881
_	В	0.366599	0.522201
Onuin	ε <sub>0m</sub> "	0.0192483	1.34002
Oswin -	C	0.690876	489.373
-	MRE [%]	2.81	0.66
	RSS	1.68E-05	2.17E-03
	А	129.767	0.833903
_	В	2.72837	2.14074
	ε <sub>0m</sub> "	0.0192674	1.35049
-Henderson	C	0.374949	41.9252
-	MRE [%]	2.79	0.47
	RSS	1.65E-05	1.08E-03
_	А	0.143556	0.187119
_	В	-0.0750101	-0.112018
Chung-	ε <sub>0m</sub> "	0.0178863	1.33654
Pfost	C	0.490488	2.37827
-	MRE [%]	2.15	0.30
	RSS	1.06E-05	4.76E-04
	А	-0.2	-0.2
_	В	6.77892	5.31445
	ε <sub>0m</sub> "	0.0178863	1.33654
Chen	С	13.3315	8.92711
-	D	0.599083	2.90483
	MRE [%]	2.15	0.30
	RSS	1.06E-05	4.76E-04
	γ	3.41E+07	5.41E+07
- Ferro-	А	15.021	14.8329
	r	-0.121425	-0.111969
Fontan	ε <sub>0m</sub> "	0.0191076	1.36
-	MRE [%]	2.97	0.44
	RSS	2.11E-05	9.10E-04
Lowe	st RSS	5.26E-06	3.17E-04
	RSS model	GAB	GAB
	st MRE	0.97	0.24
Lowest N	IRE model	GAB	GAB

Table F-2 Loss factor and dielectric constant equation fits for biscuits

Model	Parameter	Pasta loss factor	Pasta dielectric constant
	M <sub>0</sub>	0.232982	0.23671
GAB	С	140	233.051
	K	0.0454407	0.00815148
	ε <sub>0m</sub> "	0.0272279	1.4497
	MRE [%]	4.46	1.07
	RSS	2.68E-03	1.33E-02
	А	5	1.99761
	В	0.383364	0.529994
	ε <sub>0m</sub> "	0.0485991	1.49813
Oswin	C	2484.98	110
	MRE [%]	7.72	2.56
	RSS	4.89E-03	8.07E-02
	А	14.5373	0.220724
	В	3.2658	2.13082
	ε <sub>0m</sub> "	0.07	1.4995
Henderson	C	7.7777	217.052
	MRE [%]	7.46	1.63
	RSS	5.27E-03	4.56E-02
	А	0.819064	0.584666
	В	-0.394963	-0.264941
Chung-	ε <sub>0m</sub> "	0.00975472	1.53159
Pfost	С	66.3546	135.331
	MRE [%]	3.95	1.51
	RSS	4.27E-03	2.34E-02
	А	-0.2	-0.2
	В	7.14173	10.0529
	ε <sub>0m</sub> "	0.0342694	1.56096
Chen	С	4.10333	3.96786
	D	15.0701	230.449
	MRE [%]	4.22	1.75
	RSS	4.59E-03	3.19E-02
	γ	8.90E+07	6.46E+07
Ferro- Fontan	Å	15.6055	14.6725
	r	-0.123113	-0.118853
	ε <sub>0m</sub> "	0.0597794	1.5499
	MRE [%]	4.692840183	2.492768141
	RSS	4.33E-03	1.71E-01
Lowes		2.68E-03	1.33E-02
Lowest R	SS model	GAB	GAB
	t MRE	3.95	1.07
Lowest M	RE model	Chung-Pfost	GAB

Table F-3 Loss factor and dielectric constant equation fits for pasta

## Appendix G – Commercial feasibility calculations

#### G.1 Energy requirement and commercial feasibility of microwave finish drying of potato crisps

The finite difference weighted average of potato crisp isosteric heat from 0.04kg.kg<sup>-1</sup> to 0.0173kg.kg<sup>-1</sup> wet basis was calculated to be 4.55x10<sup>6</sup>Jkg<sup>-1</sup> using the Tsami et al. (1990) curve fit model of measured desorption results presented in Chapter 5 (see Figure 5-19). Production rates of modern fryers for potato crisp production can be as high as 3000kg.h<sup>-1</sup> finished product (Food Machinery, 2015; Heat and control, 2012). This converts to a production rate of 0.83kgs<sup>-1</sup> of finished product, with an oil content of 0.323kg.kg<sup>-1</sup> and a final moisture content of 0.0173kg.kg<sup>-1</sup> wet basis. The oil content is printed on the nutritional content of Walkers crisps packets, while the final moisture content was measured using dry nitrogen exposure as described in Chapter 4. The dry mass content is 0.9827kg.kg<sup>-1</sup>, which was calculated by subtracting the water content of 0.0173kg.kg<sup>-1</sup> from 1. Hence the dry basis product produced is calculated below:

Dry basis product produced = dry mass content x total production rate Dry basis product produced = 0.9827kg.kg<sup>-1</sup> x 0.83kgs<sup>-1</sup> Dry basis product produced = 0.82kgs<sup>-1</sup>

Potato crisps enter the microwave process with a moisture content of 0.04kg.kg<sup>-1</sup> wet basis, this can be converted to a dry basis quantity using the formula below where  $M_{db}$  is the dry basis moisture content and  $M_{wb}$  is the wet basis moisture content:

$$M_{db} = \frac{1}{\left(\frac{1}{M_{wb}}\right) - 1}$$
Equation G-1
$$M_{db} = \frac{1}{\left(\frac{1}{0.04}\right) - 1} = 0.042 \text{kg.kg}^{-1}$$

Dry basis moisture content is defined in the formula below where  $m_w$  is the wet mass and  $m_d$  is the dry mass. This can be used to calculate the initial quantity of moisture entering the microwave process. This assumes that the oil content does not change during the finish drying process.

$$M_{db} = \frac{m_w}{m_d}$$
Equation G-2  
0.042kg.kg<sup>-1</sup> =  $\frac{m_w}{0.82kg.s^{-1}}$   
 $m_w = 0.0341kg.s^{-1}$ 

This final quantity of moisture leaving the microwave process in finished product can be calculated as shown below, where  $M_{wb}$  is the wet basis moisture content,  $m_w$  is the mass of the moisture, and  $m_T$  is the total mass.

$$M_{wb} = \frac{m_w}{m_T}$$
Equation G-3  
0.0173kg.  $kg^{-1} = \frac{m_w}{0.83kg. s^{-1}}$   
 $m_w = 0.0144kg. s^{-1}$ 

The moisture to be removed by the microwave process is calculated by subtracting the final moisture mass from the initial moisture mass:

Moisture to be removed = Initial moisture mass – final moisture mass Moisture to be removed = 0.0341kg.s<sup>-1</sup> – 0.0144kg.s<sup>-1</sup> Moisture to be removed = 0.0197kg.s<sup>-1</sup>

As discussed previously the total isosteric energy to be supplied when dehydrating potato crisps from 0.04kg.kg<sup>-1</sup> down to 0.0173kg.kg<sup>-1</sup> wet basis is  $4.55 \times 10^{6}$  Jkg<sup>-1</sup>. Hence the total energy required for this process can be calculated as follows:

Total microwave energy required = total isosteric heat x water to be removed

Total microwave energy required =  $4.55 \times 10^{6}$  Jkg<sup>-1</sup> x 0.0197kg.s<sup>-1</sup> Total microwave energy required = 89.7kW

The efficiency of the magnetron to convert electrical energy to microwave energy is assumed to be 88%, this is the specified energy efficiency for the e2v 100kW 915MHz constant wave BM magnetron (E2V Technologies Limited, 2002). It is assumed that 97% of the microwave energy is converted into useful heating energy as quoted by Industrial Microwave Systems, L.L.C. (2012). This assumes, rather optimistically, that the load is of a sufficient loss that matching is near perfect i.e. negligible reflected power, and that there is negligible microwave heating of the applicator and the waveguides. The total electrical energy to be supplied to the microwave generator is calculated below:

Total electrical energy =	Total microwave energy required / (magnetron
	efficiency x conversion to useful heating energy)
Total electrical energy =	89.7kW / (0.88 x 0.97)
Total electrical energy =	105kW

The total electrical energy required can be used to calculate the cost of the energy require to finish dry the potato crisps. The generator output energy required can be calculated as follows:

Generator output =	Total microwave energy required / Conversion to	
	useful heating energy	
Generator output =	89.7kW / 0.97	
Generator output =	92.5kW	

Microwave generators are available at this output power. Muegge produce an off-the-shelf 100kW generator which operates at 915MHz, while Ferrite can supply a 125kW generator which operates at 915MHz and is highly customizable (Muegge, 2016; Ferrite Microwave Technologies, 2016). Hence microwave finish drying for a production rate of 3000kg.h<sup>-1</sup> can be carried out

with one generator. The cost of a generator capable of delivering 100kW is approximatley £70,000 (Smith, 2016).

Now the frying energy cost for finish drying can be calculated for comparison. The efficiency of a fully loaded gas burning fryer is approximately 65%, which is the ratio of the energy added to the food and the total energy supplied to the appliance during cooking (Fisher, et al., 2002). Hence the total gas energy required for frying down from from 0.04kg.kg<sup>-1</sup> down to 0.0173kg.kg<sup>-1</sup> wet basis can be calculated as follows. Please note that as this is the end of the complete frying process, oil warm up energy is not considered.

Total gas energy =Total frying energy required / fryer efficiencyTotal gas energy =89.7kW / 0.65Total gas energy =138kW

Electrical energy currently costs 0.21£/kWh (British Gas, 2016a), whilst gas energy costs 0.0656£/kWh (British Gas, 2016b). If it is assumed that production occurs for 8 hours in a day, 5 days a week, for 51 weeks in a year, the extra energy cost for the microwave process can be calculated. The cost of drying from 0.04kg.kg<sup>-1</sup> down to 0.0173kg.kg<sup>-1</sup> wet basis is calculated for the fryer and the microwave process as follows.

Cost of gas energy required for	=	Cost of gas x power x
fryer finish drying for 1 year		production time
		0.06560/1-W/h = 1201-W/

- = 0.0656£/kWh x 138kW x 2040h
- = £18,475

Cost of electrical energy required for = Cost of gas x power x microwave finish drying for 1 year = 0.21£/kWh x 105kW x 2040h = £45,035

Hence the extra yearly energy running cost for the microwave process =  $\pounds 45,035 - \pounds 18,475 = \pounds 26,560$ .

If it is assumed that amortization occurs over a 1 year period, then the additional cost relative to a single packet of crisps can be calculated. A standard packet of Walkers crisps weighs 32.5 grams, as printed on the packet. Again assuming a production rate of 3000kg.h<sup>-1</sup> occurs for 2040 hours a year, the total number of bags of crisps produced in a year = 3000kg.h<sup>-1</sup> x 2040h / 0.0325kg = 188 million packets. The cost for the first year would be (£26,460 + £70,000) / 188,000,000 = 0.051 pence per packet of crisps. The cost for subsequent years would be £26,460 / 177,000,000 = 0.014 pence per packet of crisps. The amortization in the first year does not consider the cost of the applicator and conveyor belt system.

#### G.2 Energy requirement and commercial feasibility of explosive microwave dehydration of potato crisps

The production rate of modern industrial fryers can be as high as 3000kg.h<sup>-1</sup> of finished product (Food Machinery, 2015; Heat and control, 2012). The new microwave process may ultimately be expected to match the production rate of a continuous fryer, which is the existing technology that it is proposed to replace. The capital costs, and the running costs of the microwave process compared to the existing process will be explored to determine the commercial viability of the microwave process. The energy requirements for both the microwave process, and the frying process must be quantified to assess running energy costs.

The finite difference weighted average of potato crisp isosteric heat from 0.82kg.kg<sup>-1</sup> to 0.04kg.kg<sup>-1</sup> wet basis was calculated to be  $2.54x10^6$ Jkg<sup>-1</sup> using

the Tsami et al. (1990) curve fit model of measured desorption results presented in Chapter 5 (see also Figure 5-19). The microwave heating process allows control of the oil content, which is the key benefit. This analysis will assume an oil content of 0.15kg.kg<sup>-1</sup> for the final microwaved potato crisp product, which results in roughly half the fat content of continuously fried potato crisps, which have an oil content of 0.323kg.kg<sup>-1</sup>.

In order to consider the energy requirements for the microwave process, the moisture to be removed must be quantified for a production rate of 3000kg.h<sup>-1</sup> (0.83kgs<sup>-1</sup>) of finished product. The final moisture mass of the explosive microwave dehydration process has been previously calculated in Appendix G.1 as the initial moisture mass of the microwave finish drying process. Hence, at a moisture content of 0.04kg.kg<sup>-1</sup> the moisture mass is 0.0341kg.s<sup>-1</sup>.

The mass content of solid material is 0.833kg.kg<sup>-1</sup>, which was calculated by subtracting the final water content of 0.017kg.kg<sup>-1</sup> and the final oil content of 0.15kg.kg<sup>-1</sup> from 1. Hence the dry basis product produced is calculated below:

Solid product produced = dry mass content x total production rate Solid product produced = 0.83kg.kg<sup>-1</sup> x 0.833kgs<sup>-1</sup> Solid product produced = 0.694kgs<sup>-1</sup>

Potato crisps enter the microwave process with a moisture content of 0.82kg.kg<sup>-1</sup> wet basis, this can be converted to a dry basis quantity using the formula below, where  $M_{db}$  is the dry basis moisture content, and  $M_{wb}$  is the wet basis moisture content. There is no oil in the potato at the start of the process.

$$M_{db} = \frac{1}{\left(\frac{1}{M_{wb}}\right) - 1}$$
Equation G-4
$$M_{db} = \frac{1}{\left(\frac{1}{0.82}\right)^{-1}} = 4.56 \text{kg.kg}^{-1}$$

Dry basis moisture content is defined in the formula below where  $m_w$  is the wet mass and  $m_d$  is the dry mass. This can be used to calculate the initial quantity of moisture entering the microwave process.

$$M_{db} = \frac{m_w}{m_d}$$
 Equation G-5  
4.56kg.  $kg^{-1} = \frac{m_w}{0.694kg.s^{-1}}$ 

 $m_w = 3.16 kg. s^{-1}$ 

The moisture to be removed by the microwave process is calculated by subtracting the final moisture mass from the initial moisture mass:

Moisture to be removed = Initial moisture mass – final moisture mass Moisture to be removed = 3.16kg.s<sup>-1</sup> – 0.0341kg.s<sup>-1</sup> Moisture to be removed = 3.13kg.s<sup>-1</sup>

As discussed previously the total isosteric energy to be supplied when dehydrating potato crisps from 0.82kg.kg<sup>-1</sup> down to 0.04kg.kg<sup>-1</sup> wet basis is  $2.54 \times 10^{6}$  Jkg<sup>-1</sup>. Hence the total energy required for this process can be calculated as follows:

Dehydration microwave energy required = total isosteric heat x water to be removed Dehydration microwave energy required = 2.54x10<sup>6</sup>Jkg<sup>-1</sup> x 3.13kg.s<sup>-1</sup> Dehydration microwave energy required = 7.95MW

As well as the energy required to vaporise the moisture, the energy required to warm up the product also needs to be considered. The potato crisps are ultimately heated to  $155^{\circ}$ C in the microwave process as described in the patent written by Bows et al. (2010). It is assumed that the majority of the water can only be heated to  $100^{\circ}$ C, after which it boils. Hence the heat up energy of water and solid potato must be calculated separately. The specific heat capacity of potato is 3640Jkg<sup>-1</sup>K<sup>-1</sup>, and the specific heat capacity of water is 4182Jkg<sup>-1</sup>K<sup>-1</sup>

<sup>1</sup> (Çengel, 1998). Raw potato consists of water and solid potato structure. The mass fraction of water in potato is 0.82kg.kg<sup>-1</sup>, as deduced in the present study by oven drying at  $105^{\circ}$ C for 24 hours. Hence the specific heat of solid potato can be calculated as follows, where  $C_{p\_potato}$  is the specific heat of potato,  $C_{p\_w}$  is the specific heat of water,  $C_{p\_s}$  is the specific heat of the solid, and  $f_w$  is the mass fraction of water.

$$C_{p_potato} = f_w C_{p_w} + (1 - f_w) C_{p_s}$$
Equation G-6  
3640 = (0.82)(4182) + (1 - 0.82) C\_{p\_s}  
$$C_{p_s} = 1171 J. kg^{-1} K^{-1}$$

Hence the energy required to heat the solid potato from 20°C to 155°C can be calculated using Equation G-7 (Çengel, 1998), where Q is heat,  $\dot{m}$  is mass flow rate,  $C_p$  is specific heat, and T is temperature. The solid mass is 0.694kg.s<sup>-1</sup> as previously calculated.

$$Q = mC_p(T_1 - T_2)$$
 Equation G-7  
 $Q_s = (0.694)(1171)(155 - 20)$   
 $Q_s = 110kW$ 

Similarly, the energy required to heat the oil up to 155°C can be calculated. The oil mass to be heated can be found by multiplying the final production rate of 0.83kgs<sup>-1</sup> by the final oil content of 0.15kg.kg<sup>-1</sup> which gives an oil mass of 0.125kgs<sup>-1</sup>. The specific heat of sunflower oil was taken as 2608Jkg<sup>-1</sup>K<sup>-1</sup> at 155°C as presented by Fasina & Colley (2008).

$$Q = \dot{m}C_p(T_1 - T_2)$$
 Equation G-7  
 $Q_o = (0.125)(2608)(155 - 20)$   
 $Q_o = 44kW$ 

Assuming a maximum water temperature of 100°C, the heating required for the water is calculated below. The initial water content was previously calculated to be 3.16kgs<sup>-1</sup>. The specific heat capacity of water at 100°C is 4217kJkg<sup>-1</sup>K<sup>-1</sup> (Cengel, 1998).

$$Q = \dot{m}C_p(T_1 - T_2)$$
 Equation G-7

 $Q_w = (3.16)(4217)(100 - 20)$ 

$$Q_w = 1067kW$$

Hence the total heat required to raise the temperature of the water to  $100^{\circ}$ C and to raise the temperature of the oil and the solid potato to  $155^{\circ}$ C is:

```
Warm up \ energy = 110kW + 44kW + 1067kW
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*Warm up energy* = 1.22MW

The total energy requirement is the sum of the warm up energy and energy required to vaporise the water, which is calculated as follows:  $Energy \ required = 1.22MW + 7.95MW = 9.17MW$ 

As in the previous section, it was assumed that the magnetron efficiency is 88%, and that 97% of the microwave energy is converted to useful heating energy (E2V Technologies Limited, 2002; Industrial Microwave Systems, L.L.C., 2012). The total electrical energy to be supplied to the microwave generator is calculated below:

Total electrical energy =	Total microwave energy required / (magnetron
	efficiency x conversion to useful heating energy)
Total electrical energy =	9.17MW / (0.88 x 0.97)
Total electrical energy =	10.7MW

The total electrical energy can be used to calculate the cost of the energy required to microwave process the potato crisps. The generator output energy required can be calculated as follows:

Generator output =	Total microwave energy required / Conversion to	
	useful heating energy	
Generator output =	9.17MW / 0.97	
Generator output =	9.45MW	

Hence, to replace one 3000kg.h<sup>-1</sup> fryer, 95 off the shelf 100kW 915MHz generators would be required for explosive dehydration and 1 off the shelf 100kW 915MHz generators would be required for finish drying (Muegge, 2016). The cost of a generator capable of delivering 100kW is approximatley  $\pounds$ 70,000 (Smith, 2016). The total cost for the generators alone would be  $\pounds$ 70,000x96 =  $\pounds$ 6,720,000.

The energy requirements for the frying process is slightly different, as the oil content is higher at 0.323kg.kg<sup>-1</sup> compared to the proposed oil content for microwaved crisps which is 0.15kg.kg<sup>-1</sup>. The energy required for the frying process was calculated to be 7.30MW using the same process as for the microwave energy. This is lower than the 9.17MW required for the microwave drying process. Less water needs to be removed in frying compared to microwave drying for a given mass throughput due to the higher oil content. Oil has a lower heat capacity than water, 2608Jkg<sup>-1</sup> compared to 4217Jkg<sup>-1</sup>, hence warming oil up requires less energy as described in Equation G-7 (Çengel, 1998; Fasina & Colley, 2008).

As in the previous section, the efficiency of a fully loaded gas burning fryer is assumed to be approximately 65% (Fisher, et al., 2002). Hence the total gas energy required for frying down from from 0.82kg.kg<sup>-1</sup> down to 0.04kg.kg<sup>-1</sup> wet basis can be calculated as follows.

Total gas energy =	Total frying energy required / fryer efficiency
Total gas energy =	7.30MW / 0.65
Total gas energy =	11.2MW

Therefore, although frying requires less energy due to the higher oil content of the final product, the gas enrgy required is 11.2MW, which is slightly higher than the 10.7MW of electrical energy required for the microwave process due to the superier energy efficiency of the microwave process.

Electrical energy costs 0.21£/kWh (British Gas, 2016a), whilst gas energy costs 0.0656£/kWh (British Gas, 2016b). If it is assumed that production occurs for 8 hours in a day, 5 days a week, for 51 weeks in a year, the extra energy cost for the microwave process can be calculated. The cost of drying from 0.82kg.kg<sup>-1</sup> down to 0.04kg.kg<sup>-1</sup> wet basis is calculated for the fryer and the microwave process as follows.

Cost of gas energy required for	=	Cost of gas x power x
Fryer from 0.82kg.kg <sup>-1</sup> down to 0.04kg.kg <sup>-1</sup>		production time
	=	0.0656£/kWh x 11.2MW x
		2040h
	=	£1.5 million

Cost of electrical energy required for	=	Cost of electricity x
microwave drying from 0.82kg.kg <sup>-1</sup>		power x production time
down to 0.04kg.kg <sup>-1</sup>	=	0.21£/kWh x 10.7MW x
		2040h
	=	£4.6 million

The microwave process would also require a finish drying process to bring the moisture down from 0.04kg.kg<sup>-1</sup> to 0.017kg.kg<sup>-1</sup>. The extra yearly energy running cost for the finish drying process compared to frying was calculated to be  $\pounds 26,560$  in the previous section.

Hence the extra yearly energy running cost for the microwave process =  $\pounds 4.600$ million +  $\pounds 0.027$  million -  $\pounds 1.504$  million =  $\pounds 3.12$  million.

If it is assumed that amortization of the generator costs occurs over a 1 year period, then the additional cost relative to a single packet of crisps can be calculated. A standard packet of Walkers crisps weighs 32.5 grams, as printed on the packet. Again assuming a production rate of 3000kg.h<sup>-1</sup> occurs for 2040 hours a year, the total number of bags of crisps produced in a year = 3000kg.h<sup>-1</sup> x 2040h / 0.0325kg = 188 million packets. As the additional energy cost is £3.12 million, the generators for the explosive microwave drying process costs £6.72 million, and the generator for finish drying would cost £70,000, the cost of the first year would be  $(\pounds 3, 120,000 + \pounds 6, 720,000 + 70,000) / 188,000,000 =$ 5.3 pence per packet of crisps. The cost for subsequent years would be  $\pounds 3,120,000 / 177,000,000 = 1.7$  pence per packet of crisps. The amortization in the first year does not consider the cost of the applicator and conveyor belt system. Single packs of Walkers crisps sell for 55 pence in most supermarkets (Sainsbury's, 2016), while packs sold in multipacks sell for an equivalent 33 pence each. The 1<sup>st</sup> vear amortization period would be 9.6% of the price of a single packet of crisps, and 16.0% of a multipack packet of crisps. The subsequent yearly periods would cost 3.0% of the price of the price of a single packet of crisps, and 5.0% of a multipack packet of crisps.

## Appendix H – Published peer reviewed paper

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