AN EVALUATION OF MICROWAVE AND RF TECHNOLOGY AS ENERGY AND TIME EFFICIENT ALTERNATIVES FOR THE DRYING PROCESS OF BARLEY MALT

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Abstract

The objective of this project was to explore the fundamental interactions between microwave and radio frequency (RF) energy and malt. The main goal was to develop and apply a method for the reduction of time and energy costs and the enhancement of product quality in the kilning process which is used to dry malted barley. Volumetric heating technologies such as microwaves and radio frequency heating have been applied in industrial processing for many years and have the potential to reduce both time and energy costs compared with conventional heating. Potential benefits include savings in energy and process time whilst increasing product homogeneity and thus quality. Furthermore, it was important that the treatment should not adversely affect any of the key brewing (or distilling) quality parameters which are currently specified by the malt end-users.

Firstly, the impact of moisture content on the dielectric properties of whole malt and barley grains (variety *Tipple*) at varying moisture contents relevant to the malting process (5-46% w/w) was investigated. Measurements were obtained using the transmission line waveguide technique, at room temperature (20°C) using microwave frequencies between 2.3 - 2.5 GHz. According to the results, water content was the dominant factor affecting the dielectric properties of both malt and barley grains and there was a strong similarity between malt and barley dielectric properties at a set moisture content, despite their different chemical compositions. The dielectric constant (ε') of malt increased from 2.14 to 6.54 over the range of 5 to 46% w/w moisture, whereas for barley the equivalent range in ε' was 2.21 to 6.72. The dielectric loss factor (ε'') increased with moisture content for both malt and barley from 0.03 to 1.12 and from 0.03 to 1.03 respectively. The outcomes of this work could be applied to the development of microwave processing of malt and barley whole grains.

Following the characterisation of these dielectric properties at microwave frequencies, it was decided that the penetration depth of microwaves in barley grains was unlikely to deliver sufficient throughput for an industrial process to be developed. Hence we opted next to investigate the application of radio frequencies (RF) to the drying of malt. We investigated the influence of radio frequency energy applied to malt hydrated to different moisture contents in comparison with a conventional drying process. At the end of the RF processing the moisture content was measured and the amylase activity of the bulk sample was tested. The high and medium loss samples did not maintain the required enzyme activity at the end of the drying process due to high sample temperature effects. In contrast, the low loss samples had better enzyme preservation, despite the fact that high temperatures (>90°C) still existed. This trend

could be explained by the fact that the time of the RF treatment in this experiment was less than that in the case of high and medium loss samples. Additional tests were performed to optimise the variable parameters of the RF tunnel system in order to achieve a smoother drying procedure for the malt. The observations from the RF tunnel experiments showed that more extensive study was required to control the temperature effect that was responsible for the deactivation of the amylases. Moreover an additional study on the effect of lower RF frequencies on the drying process of malt in combination with conventional drying could be more promising for the final product quality and for the decrease of time and energy costs.

Acknowledgements

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Chapter 1 Theoretical background

1.1 Introduction

The traditional industrial drying process of barley malt known as kilning includes the use of high amount of electricity energy to cover the needs of drying approx. 60 tonnes of green malt per hour in the industrial ovens by applying an air fan.

In UK malting sector consumes around 1300 GWh per year from which the 86% is used by fuels for heating purposes. Thus the energy use per tonne is quite high as it can reach the 1000 kWh (AEA, 2011). Kilning process absorbs the 78% of the heat demands and unfortunately from the energy consumed only the 20% is recovered by using glass tube heat exchangers to recover the heat.

Consequently, the necessity of designing an alternative way of drying the malt appears to be indispensable. Based on the above idea, this project tries to study the efficiency of applying electromagnetic energy as a means for the drying of malt always by trying to give a good quality final product. The theory behind this study is covered in the following section and includes all the necessary information which someone should have in mind in order to design and apply a reliable alternative drying method of malt based on the implementation of the electromagnetic energy.

1.2 Structure of barley grain

Cultivated barleys (*Hordeum vulgare* L.) have the capacity to be grown in harsh climatic conditions, although high quality malting barley is best grown in cool, temperate maritime regions (Bamforth, 2009). It is preferred to other grains for malting because it has high concentration of starch and develops relatively high amounts of the diastatic enzymes on germination which are required to ‘self-digest’ the barley starch during brewing. In addition, the husk of barley is also suited to brewing because it can act as an efficient filter bed during mash separation and also because it protects the grain against damage when conveyed around an industrial scale malting plant. Malted barley has many important functions in brewing, including the contribution of colour, flavour, essential nutrients for yeast metabolism and brewhouse functionality (Ockert, 2006).

The barley kernel consists of the embryo (baby plant) along with a starchy endosperm as a food reserve, packed within protective layers as presented in Figure 1. The

endosperm includes starch formed of large and small granules packed within a matrix of protein which is covered by a thin cell wall (Figure 2). The endosperm is completely controlled by the embryo (Bamforth, 2009) and represents the 75-80% of the dry weight of the grain (Briggs, 1998).

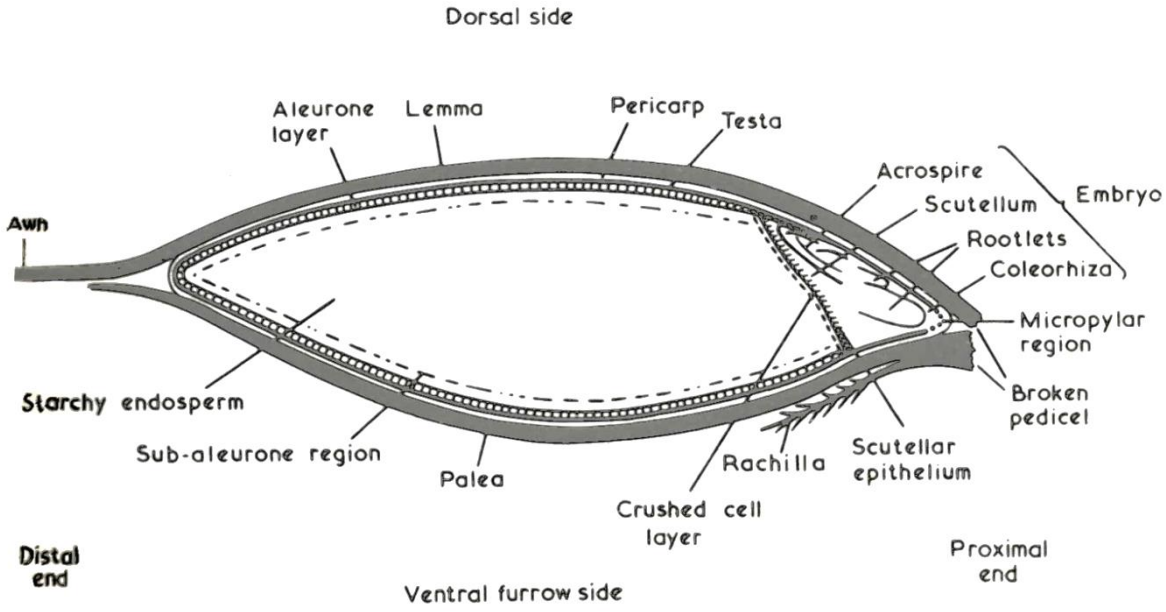

Figure 1. Schematic representation of the internal of a barley kernel in longitudinal section (Briggs and Hough, 1981).

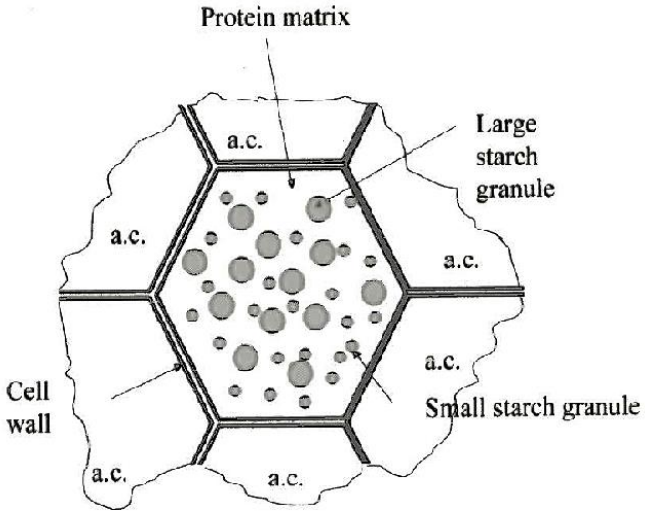

Figure 2. Schematic representation of the starchy endosperm cell of barley (a.c.) (Bamforth, 2009).

Starch is a mixture of two glucose polymers, amylose and amylopectin. These are common plant storage polysaccharides which are mixtures of high molecular weight chains of poly-(α-D-glucopyranose). The amount of amylose in British barley starches ranges between 18-26%, the remaining portion being amylopectin (Briggs, 1998).

The aleurone layer which surrounds the starchy endosperm (other than where the scutellar tissue separates the embryo from the endosperm) is the site of the enzyme production and secretion. Enzyme synthesis is controlled by gibberellin hormones which are produced by the embryo, but in order to trigger this release, the moisture content of the grain must increase to >32%. (Bamforth, 2009).

The hydrolytic enzymes that are developed during the malting process are capable of breaking down the cell walls of the starchy endosperm as well as the proteins and starches contained within (Ockert, 2006).

When barley germinates, the first external sign is the emergence of the small, white coleorhiza (root sheath) protruding at the proximal end of the grain (near to the original point of attachment to the plant). For this, several rootlets emerge and in the meantime the coleoptile (which maltsters and brewers refer to as the 'acrospire') emerges underneath the husk. In mature grain, the husk, the pericarp and the testa are considered to be dead tissues. The husk and the pericarp protect the barley grain and restrict the diffusion of water or gases. The aleurone layer cells do not grow or divide during germination, but this is a living tissue which respires and metabolizes, as well as being a key site of enzyme synthesis.

During the malting process, there are losses of dry material due to the release of substances into the steep water, due to oxidation via the respiratory process and also the physical losses of the dried (de-culmed) rootlets and dust. It is estimated that the total losses during malting range from 6% to 12% of the barley steeped dry weight (Briggs, 1998).

It is important to note that not all barleys are appropriate for use in malting. For instance, only husked barleys are suitable for the malting industry (Briggs, 1998).

1.3 Malting process

Malt is mostly produced from barley grain and every year 1.5 million tonnes of malt is produced in the UK (AEA, 2011). Malting process plays a major role in the production of malt beers. The aim of this process is to produce the enzymes which are involved in the hydrolysation of the endosperm starch of barley grain in order to prepare fermentable sugars (Briggs, 1998).

In modern industrial malting operations there are four process stages which include the import, drying and storage of barley; steeping; germination; and kilning (Bamforth, 2009).

1.2.1 Intake, drying and storage of barley

Barley is classified as winter and spring varieties which can be therefore divided into two-rowed and six-rowed types, according to how many kernels there are in the head (Ockert, 2006). In United Kingdom, maltsters and brewers normally use the two-row barleys. It also remarkable that if the seed is sown early in the year protein content in barley is lower because starch is concentrated through the whole growing period (Bamforth, 2009).

Barley raw material, at first, arrives at the malthouse and is weighed while the transport waits until a sample is tested for its germination capacity, nitrogen, protein and moisture content. Moreover, visual examination of the grain is being done for varietal purity, ergot, Fusarium infection, colouration, odour and other aspects. When the barley gets approval, it is offloaded and cleaned to remove any accompanying “rubbish” (e.g. dust, rocks) and is screened to take away thin and undersized corns before getting into the silo.

Stored barley should be dried to an overall moisture content of 10%-13% with the embryo at a somewhat higher level 18%-20%. During storage, grain, which is very attractive to insect infestation, must be protected and for that reason is ventilated. In some cases, the use of fungicides keep the barley free from diseases and helps the malting of grain (Bamforth, 2009).

1.2.2 Steeping

In this stage, barley is steeped into water in order to increase its moisture content from 11%-12% to 43%-46% over a 48 hours period; it will not germinate if its moisture is less than 32% (Briggs, 1998). For that reason, during steeping, a series of plunges or "wet stands" followed by “dry stands” is designed. During the wet phase, air is blown through the wet grain bed, while during the dry phase also known as “air rest” extraction fans are used to remove carbon dioxide (AEA, 2011).

Moreover, this process achieves the hydration of all grains to a certain level but moisture in the embryo will be higher than in the endosperm. The hydrolysis helps the grain to become friable, easily chewed and milled. The optimal temperature for steeping is between 14°C and 17°C which represents a balance between the more

rapid uptake of water as temperature is increased, versus the increased rate of microbial growth.

At first, water penetrates into the grain through the micropyle of the embryo and then is distributed through the starchy endosperm (Bamforth, 2009). Thus, the surface layers of barley are wetted rapidly, unlike the interior which is not fully hydrated for 2 to 3 days. The bulk volume of the grain increases by about a quarter across the steeping process (Briggs, 1998). In this stage, moisture content increases to 33%-37% in 6 to 16 hours. Then, the mixture is rested whilst air is sucked through the bed for 8 to 10 hours in order to expose the embryo to oxygen and remove the carbon dioxide produced by respiration. After that, another "wet" stage follows in which barley is immersed for 10 to 20 hours to reach the required moisture content level (Bamforth, 2009). During steeping, the embryo hydrates before the endosperm (Briggs, 1998).

Steeping vessels are normally made of stainless steel (Bamforth, 2009). There are two types of steeping vessels used in UK: the *conical bottomed* (Figure 3) which is more effective for increasing the moisture content of the grain and the *flat bottomed* which is more efficient for the extraction of CO_2. Few maltsters use both vessels in series (AEA, 2011).

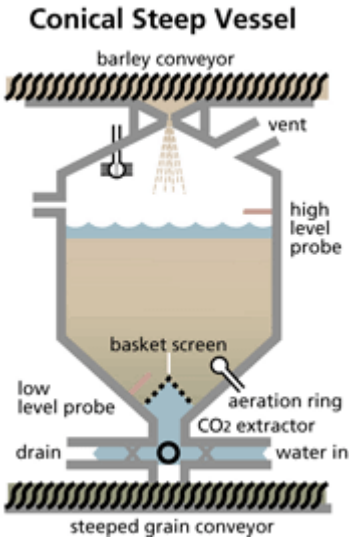

Figure 3. Schematic representation of conical bottom steeping vessel (Source: http://www.muntonsmicrobrewing.com).

1.2.3 Germination

The aim of this process is the development of the enzymes that are capable of hydrolyzing the endosperm cell walls, the protein and the starch of the grain, which results in softening of the endosperm. The temperature in this process is approximately 16°C-20°C and the procedure lasts about 4 days (Bamforth, 2009). During germination, more than 98% of the barley grains must germinate to achieve a good quality of malt (Briggs, 1998).

In order to understand that the grain is ready for germination, coleorhiza (root sheath) which disrupts through the testa and pericarp, appears at the base of the grain. Then, the bud splits and seminal roots are formed. In husked barleys, the acrospire usually appears from beneath the lemma close to the top of the grain. After saturation, starch is formed in the embryo (Briggs, 1998).

During germination it is important that the grain moisture stays close to that to which it was steeped (e.g. 45%). For this reason, the air which is circulated through the bed during germination, to remove the liberated heat which accompanies respiration, is fully saturated with water. In addition, the grain is turned periodically during germination to avoid rootlet matting. Once the whole endosperm is soft enough to be squeezed manually from the surrounding tissues-indication that 'green' malt is ready, malt is going for drying through the kilning process (Briggs, 1998).

There are two common types of germination vessels used in UK. The first one is the *circular saladin*, a circular vessel equipped with turners connected to an arm that rotates around the vessel (Figure 4).

The second one is the *saladin box*, an horizontal box fitted with turners that automatically move backwards and forwards along the length of the box. Even if it is an older method of germination, is still used at some malt plants (AEA, 2011).

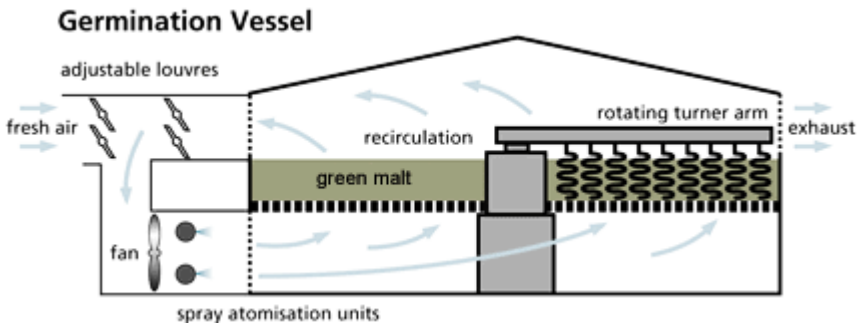

Figure 4. Schematic representation of circular saladin germination vessel (Source: http://www.muntonsmicrobrewing.com).

1.2.3.1 Biochemical activity

The enzymes produced during germination are classified based on how they act on proteins, cell walls and starches. The relative amounts and types of the enzymes of barley seed change considerably during this process. For the modification of the grain, cell walls of the endosperm must be partially degraded in order to enable access to starch degrading enzymes. Cell walls which are rich in *β*-glucan are broken down by hemicelluloses and *β*-glucanases that are generated during germination (Ockert, 2006). After 1-3 days of germination, the aleurone layer releases mineral nutrients and hydrolytic enzymes responding to gibberellin hormones originating starting from the embryo (Figure 5) and extending to the distal (awn end) of the grain. The presence of gibberillic acid increases the production and release of the enzymes (Briggs, 1998).

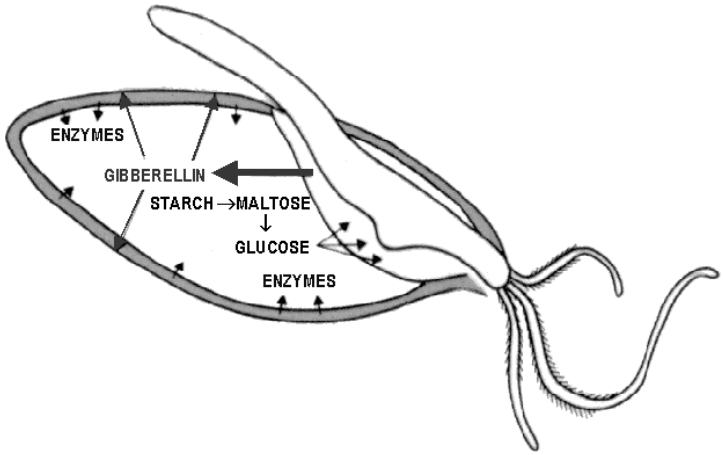

Figure 5. The production and release of hydrolytic enzymes during germination step (Source: http://www.crc.dk/flab/hydrolas.htm).

When cell walls are broken down to a certain extent, starch degrading enzymes such as α-amylase, pentosanases and cytolytic enzymes are activated (Figure 6). Those enzymes are responsible for breaking down the starch into fermentable sugars as they cause a physical modification of the structure of the endosperm and facilitate the access of diastatic enzymes to starch granules (Bewley and Black, 1994).

The most important enzymes of this type are *α*-amylase, *β*-amylase and limit dextrinase. *β*-Amylase that already exists in barley grain, increases very much during germination, while *α*-amylase does not appear in raw grain and is only generated during this process (Ockert, 2006).

α-Amylase has the capacity to break the alpha-(1→4) bond of starch producing oligosaccharides by solubilizing the amorphous and crystalline parts of the starch granules. *β*-Amylase aggresses the same bond but from the non-reducing ends of the molecules of amylose and amylopectin. Thus, because of the action of both enzymes a

variety of fermentable sugars is produced during the mashing process. As far as it is concerned limit dextrinase, this enzyme hydrolyzes the (1→6)–*α*-glucosidic branch points in low molecular weight branched dextrins (Gupta et al., 2010).

Protease enzymes are responsible for the degradation of 40-50% of the protein and its breakdown into peptides and free amino acids. These solubilized materials are important for brewery and malt extracts as they participate in the non-enzymatic browning reaction during kilning and wort boil steps, and contribute to beer foam.

A key point of the germination process is to control the end point in order to achieve the appropriate enzyme content and therefore the desired degree of malt modification (Ockert, 2006).

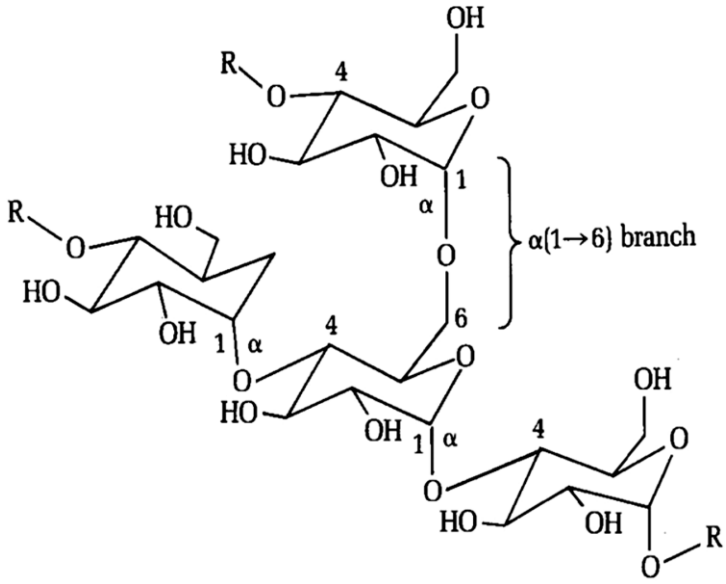

Figure 6. Starch degrading enzymes and their action in the break of α-(1→4) and α-(1→6) linkages (Cook, 2013).

1.2.3.2 Green malt

When barley is germinated and therefore is modified to a certain extent, the product is referred to as “green” malt. Following this, it is dried through the kilning process. In order to identify if the barley kernel has become “green malt” in lab scale, maltsters observe several characteristics such as the appearance and odour of the grain and the rate of its modification. To be more specific, green malt looks fresh and healthy while its odour is clean and pleasant. If barley has been modified properly, the endosperm is quite soft whereas the unmodified one is harder or more sodden. Another important

parameter that should be taken under consideration is the length and the shape of the acrospire and the rootlets (Ockert, 2006).
In industry, the germination process is controlled by time, temperature, humidity and the degree of steeping attained in the steep (steep out moisture content). According to that the desired extent of modification can be achieved.

1.2.4 Kilning

The main aim of this procedure is the drying of malt to a final stable moisture level of around 2-6% (typically 4%) without destroying the key starch degrading enzymes by using a hot air flow. The preservation of many enzymes is very important because they are used in brewing to generate fermentable sugars in the mash whilst other enzymes like lipoxygenase (LOX) should be destroyed as they have a negative impact on beer flavour stability. For that purpose, kilning starts at low temperatures with "air-on" temperatures around 50°C. The "air-on" temperature represents the air that is blown onto the bed of malt. As the warm air is blown from the bottom to the top layer of the malt bed it induces a temperature and moisture gradient throughout the depth of the bed. As the moisture evaporates and the material increases its temperature during the drying process, those gradients gradually reduce (AEA, 2011).
When enough of the water is removed, temperature can be raised to a certain level according to the type of final malt that is required. In that phase, flavours and colours are generated (Bamforth, 2009). Enzymes which are sensitive in high temperatures such as β-amylase and glucanases decrease in aggressive kilning, whilst others like α-amylase are more resistant.
The kilning process varies substantially depending on the equipment, barley quality and customers' requirements. The parameters which are usually controlled in order to give the preferable outcome in colours and flavours are the temperature, time and airflow rate (Ockert, 2006).

1.2.4.1 Design of kilns

Most modern kilns feature beds (approximately 1 m deep) of malt and are mostly circular in cross section, made from stainless steel and can be used for 400-500 tons of barley. There is a heat source that warms the incoming air and a fan that spreads the air through the bed (Bamforth, 2009). The heat source is normally used indirectly in

order to avoid interactions between the amines of malted barley and combustion by-products that may lead to the formation of nitrosamines (Ockert, 2006).

The air temperature is measured both below ("air-on") and above ("air-off") the bed on the kiln deck, by temperature probes. Malt grain is supported on a wedge-wire floor that allows air to pass through the bed, which is normally up to 1.2 m deep (Bamforth, 2009). It is known that the deeper is a kiln unit the more susceptible is to moisture and temperature variations within the malt (AEA, 2011).

Because of the energy consumption of this operation, modern kilns utilize energy conservation systems such as glass tube air-to-air heat exchangers (Figure 7). New design kilns tend also to use "indirect firing", in which fuel combustion products are expelled without passing through the grain bed (Bamforth, 2009). The circular design permits better airflow distribution and easier drying. Kilns usually have a moving screw loader with a static loading/stripping mechanism that enables the homogenous loading, and thus drying, of malt (Cook, 2013).

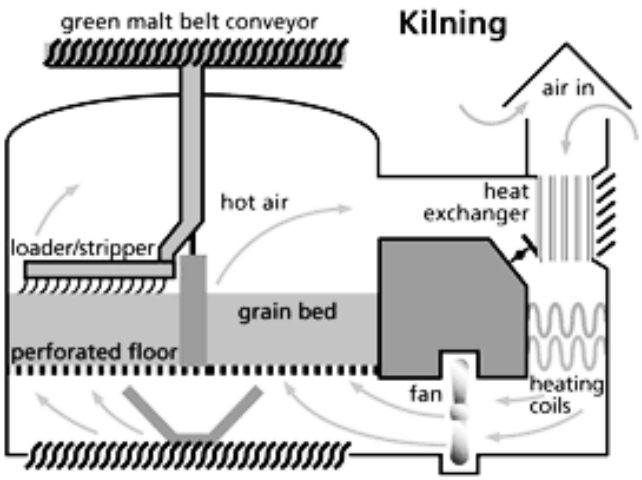

Figure 7. Schematic representation of kilning stage in circular kiln (Source: http://www.muntonsmicrobrewing.com).

1.2.4.2 Free drying phase

At first, beds of grain are dried in a current of circulating hot air in a high constant rate at low temperature between 50-60°C (Cook, 2013). In this step, drying rate is proportional to the airflow volume and malt moisture content reaches 15-20%. (Ockert, 2006). As a consequence, enzymes are protected from being destroyed and they are still active (Bamforth, 2009).

1.2.4.3 Intermediate phase

When the malt has dried to such a level that water is no longer freely available at the surface, the relative humidity of the air-off starts to drop below saturation (100%) and the drying rate drops. The mass transfer of water to the drying air is no longer controlled by the air flow rate and this is lowered (reduced fan speed) as temperatures are increased to approximately 60-80°C. This point is also known as "break point" (Cook, 2013).

This stage happens approximately 12 hours into the kilning process (Bamforth, 2009). The key point in this step is to remove moisture from the interior of the grain by heating rather than airflow. Thus, the airflow rate is reduced in order to save energy and enzyme development is stopped. In this step moisture content drops to about 10% (Ockert, 2006).

1.2.4.4 Curing phase

In this final stage, air-on temperatures are raised typically to between 80-105°C and the development of flavours and colours commence via the Maillard reactions. During this phase, pH and airflow are reduced; airflow has little effect on water evaporation, as water in the grain is tightly bound. Thus, colour formation and enzyme degradation achieve their maximum action (Ockert, 2006).

As a consequence of the Maillard reactions, volatile and antioxidant compounds such as pyrazines and polyphenols are formed, whilst amino acids are decreased due to their interaction with reducing sugars (Cook, 2013). Another effect is the partial inactivation of lipoxygenase (LOX) due to the heat treatment. The development of complex flavours depends on the style of malt required from brewers. The differentiation is in the way that malt is treating by changing time, temperature and humidity (Cook, 2013).

Additionally, during this stage dimethyl sulphide (DMS) is formed because high temperature enhances the breakdown of a precursor substance known as S-methylmethionine (developed during germination) into free DMS. Because it is a volatile compound, is easily removed during kilning and boiling of wort. It is important to note that DMS in high levels gives a creamed corn flavour to beer product that is not desirable (Ockert, 2006).

When kilning is finished the heat is switched off and the grain is cooled before its exposure to a stream of air at ambient temperature (Bamforth, 2009).

1.4 Malt quality parameters and specifications

Malt analysis is one of the most important parts involved in the malting process as it ensures the quality of the malted grains. There are two worldwide recommended methods of analysis in use: the "*Analytica-EBC*" and the "*Methods of Analysis of the ASBC*" (Briggs, 1998).

In recent years, malt quality parameters are becoming more and more demanding because of the varied and often precise requirements of brewers. Some of the most important parameters that characterize the quality of malt are moisture content, extract, free amino nitrogen (FAN), diastatic power, friability, *β*-glucan content, nitrogen content, reducing power, lipoxygenase (LOX) activity and nonenal potential (Guido et al., 2007).

1.4.1 Moisture content

The industrial standard target for malt moisture is approximately 4% w/v. The standard methods for measuring moisture contents of grains use drying ovens. The significance of malt moisture in the brewery is economic and functional as well and for that reason brewers need to know that malt meets the specification required. During malting process, grains absorb a great amount of moisture under humid conditions (Ockert, 2006).

In order to measure the moisture content, 5 g of ground barley is dried for 3 hours at 104°C. During drying, moisture is calculated by the percentage of the weight loss that occurred (Guido et al., 2005).

For the measurement of samples with a moisture content higher than 18%, in the first step the grains are dried in a rapid airflow and then are dried in the oven, treated similarly as for the kilned malt (Briggs, 1998).

1.4.2 Diastatic power

Diastatic enzymes are referred to the combined amount of *α*-amylase, *β*-amylase and other enzymes that are involved in the breakdown of the starch during germination (Ockert, 2006). Diastatic power is mostly used as an index for the determination of *β*-amylase. This parameter is 35-40 for standard Ale and can reach to 100-125 for lager malts and is measured in Windisch-Kolbach units (°W-K) according to the EBC methods. The higher the DP is the faster the degradation of starch is (IBD, 2002).

For the measurement of DP, a Skalar SAN plus analyzer can be used (in larger well equipped malt laboratories) by the automated ferricyanide procedure Malt-6C (USDA, 2011).

1.4.3 *α*-Amylase

This enzyme is absent from the early stages of barley growth and is synthesized and secreted from the scutellum and aleurone layer during germination. *a*-Amylase is responsible for the breakdown of starch and more specifically for the hydrolysis of α-glucosidic-(1-4)-glucose linkage. The degradation of starch occurs faster with the presence of *β*-amylase because those enzymes act 'in concert' (Briggs, 1998). *a*-Amylase is normally thermostable until 65°C or greater (Fox, 2010).

The determination of *α*-amylase is done by colourimetric method which is very cost effective, specific and uses a simple format (Tarr et al., 2012). More specifically, to determine the *α*-amylase, a substrate mixture is prepared by incubating soluble starch with *β*-amylase to yield a *β*-limit dextrin. Malt extract is added to that mixture and samples are taken in several time points and are mixed with iodine solution. When the colour is that of a standard, time is noted and the amount of *α*-amylase is measured as inversely proportional to the time required to reach the reference colour. The results are expressed in dextrinizing units (DU) (Briggs, 1998).

1.4.4 *β*-Amylase

The *β*-amylase acts in the non-reducing end of amylose and amylopectin by detaching the maltose. About 70% of the amylose and 50% of the amylopectin of barley starch are hydrolysed with catalysis by *β*-amylase. This enzyme is stable for temperatures not higher than 60°C (Fox, 2010).

1.4.5 *β*-Glucan

β-Glucans are polysaccharides and consist of part of the cell wall structure in the endosperm, made of unbranched chains of *β*-D-glucopyranose remnants as it is shown in Figure 8 (Briggs, 1998). Its degrading enzymes that are formed during the malting process break down this structure, allowing further modification to occur. High levels of *β*-glucan are related to slower modification of malt (Ockert, 2006). Barleys with high contents of *β*-glucan are harmful in both malting and feeding (Molina-Cano et al., 1997).

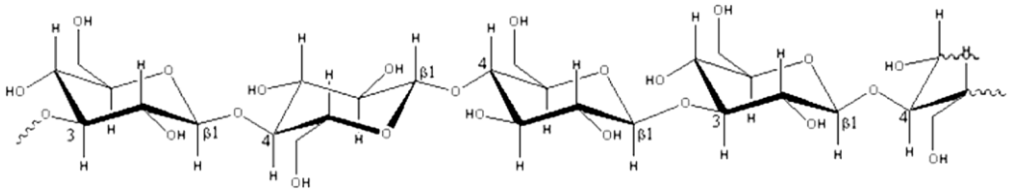

Figure 8. Linear unbranched polysaccharides of linked *β*-(1→3) and *β*-(1→4)-D-glucopyranose units (Cook, 2013).

In the following diagram there is a schematic representation of the degradation of β-glucans during germination:

***β*-glucan in cell wall**

⇩ *β*-glucan solubilase

High molecular weight soluble but viscous *β*-glucan

Barley Endo *β*-glucanase (1:3; 1:4) – greatly increased levels on malting
High activity on mixed link *β*-glucans

⇩

Endo β 1:4 glucanase
Endo β 1:3 glucanase

Smaller water soluble molecules such as β-glucan fragments

1.4.6 Friability

Malt friability represents the extent to which the endosperm has been modified during germination and is inversely related to the protein content (Guido et al., 2007).
Friability is a physical test that is used to crush the malt through equipment with a roller in rotation mode. According to this method, 50 g of malt are milled with a standard pressure in the roller (IBD, 2002). The amount of malt that is dropped down as powder is the modified (friable) part. The unfriable part represents the unmodified malt (Ockert, 2006). The regular limits for friability in malting industry vary depending on the type of malt that is used. For ale and distilling malt it should be minimum 85% while for lager style malt it should be minimum 75% (http://www.muntonsmalt.com/products/ale-malt/).

1.4.7 Nonenal potential

Nonenal potential indicates how a beer will release E-2-nonenal during storage (Guido et al., 2007). This compound is very important because of its very low flavour limit (0.035 μg/L) and the fact that it is responsible for the paper taste of aged beers (Guido et al., 2005).
The quantification of nonenal potential can be done by using the GC-MS (Guido et al., 2007).

1.4.8 Wort reducing power

Wort reducing power measures the oxidation state of beers by indicating the decolourization during a certain time period which is especially defined for fast, intermediate and slow reducing agents (Guido et al., 2007).

1.4.9 Protein content

The protein content of malting barley is proportionate to the free amino acid nitrogen content of the malt and inversely related to the malt extract content. In brief, the higher the protein level, the higher the total enzyme potential. In order to measure the total protein content, the overall amount of nitrogen is multiplied by 6.25. The total malt protein contains both soluble and insoluble proteins (Ockert, 2006).

1.4.9.1 Soluble Protein (SP)

Soluble protein represents the malt protein which is dissolved when the mashing process is finished. It can be measured by either the nitrogen content or the UV absorbance of an extract and contains soluble proteins, peptides and amino acids. This index is useful to estimate the content of proteins and large peptides that enhance the foaming ability, taste and other physical properties of beers (Jones, 2005).

1.4.10 Free amino nitrogen (FAN)

Free amino nitrogen indicates the amount of $–NH_2$ groups in an extract and is measured by their interaction with a specific reagent. The reagent has the ability to detect proteins, peptides and amino acids that contain free amino (–NH2) groups (Jones, 2005). Put another way, FAN measures the amount of soluble protein that is

further degraded into free amino acids and is an indication of the yeast nutritional value of an extract (Ockert, 2006).
There are two methods for the determination of FAN based on colourimetry. The first (and more common) method uses ninhydrin and the second trinitrobenzene sulphonic acid (TNBS) and they are both calibrated with amino acid standards (Briggs, 1998).

1.4.11 Wort Colour and Clarity

The determination of wort colour is not easy because wort varies a lot in the intensity and the nature of its colour. As a consequence, different worts can lead to a variety of absorption spectra (Briggs, 1998). Wort colour is strongly influenced by the variety of barley, the kilning process and the modification degree of malted grain (Ockert, 2006). In order to analyze the wort colour, hot water extracts are used or boiled extracts to give the boiled wort colour. The *Analytica-EBC* also uses a spectrophotometric measurement of the colour intensity of wort at 430 nm (Briggs, 1998).
Blurry wort may mean that there is a chain complexity of starch and proteins and indicates a low level of modification (Ockert, 2006).

1.4.12 Extract

The portion of malt that is solubilized during mashing to form wort is the malt extract and is measured either as fine-grind or coarse-grind extract. *Fine-grind* extract indicates the maximum extract that can be achieved whereas *coarse-grind* extract represents a brewer's expectations from the malt. The difference between fine and coarse extract known as fine-coarse difference (FCD) shows the degree of modification of malt. The lower is this indicator the higher is the modification of malt (Ockert, 2006).
The following table (Table 1) lists representative specifications for the main quality parameters of ale, lager and distilling malt, according to Muntons plc., analyzed by EBC Methods of Analysis.

Table 1. Malt specifications for ale, lager and distilling malt according to Muntons plc. malting company (Source: http://www.muntonsmalt.com/products/ale-malt/).

	Ale Malt	Lager Malt	Distilling Malt
Moisture %	≤3.5	≤5.0	≤4.5
Extract (0.2 mm), dry %	82.0		≥77.0 (sol %)
Extract (1.0 mm), dry %	81.5		≥76.0 (sol %)
Extract - Fine Grind, dry %		≥80	
Fine/Coarse difference (0.2/1.0)	0.5		
Total Nitrogen, dry %	1.40-1.60		1.40-1.65
Total Soluble Nitrogen, dry %	0.57-0.67	0.60-0.75	0.5-0.65
Soluble Nitrogen Ratio			34-40
Kolbach %	37-42	36-44	
Colour (EBC Units) 450 g Mash	4.5-7.0		
EBC Wort Colour (EBC Units)		≤3.4	
Friability %	≥85	≥75	≥85
Homogeneity %	≥95		≥97
DP WK units	≥156	≥220	
DP (°IoB)			≥63
Fermentable Extract %			≥67
NDMA ppb	≤5		≤2
EBC *β*-Glucan mg/L		≤220	
FAN (1030) mg/L			110-140
Phenol ppm			< 1.0
PSY			≥408
MC g/t			≤3

1.5 Energy consumption

1.5.1 General consumption

The malting sector in the UK consumes approximately 1,375 GWh of energy every year. This mainly derives from the use of fuels for heating processes, with annual consumption that reaches 1,176 GWh (86% of total energy usage). Moreover, specific energy consumption (SEC) during 2008-2009 was 961 kWh/tonne (AEA, 2011).
Current energy usage reaches 830-1010 kWh/t of malt produced, of which thermal gas energy represents 750-850 kWh/t, whilst electricity represents 80-160 kWh/t, which is approximately 20% of the total costs of malt production (Davies, 2006).

1.5.2 Energy consumption during kilning process

The kilning stage accounts for around 78% of the heat demands that is required for the evaporation of water for drying the malt to its final moisture content. Modern kilns utilize glass tube air-to-air heat exchangers that recover heat from saturated exhaust air in order to preheat the ambient incoming air to the kiln. During the pre-break phase, heat exchangers are capable of regaining about 20% of the energy available, whilst the remaining 80% is lost to the atmosphere (Figure 9) (AEA, 2011).

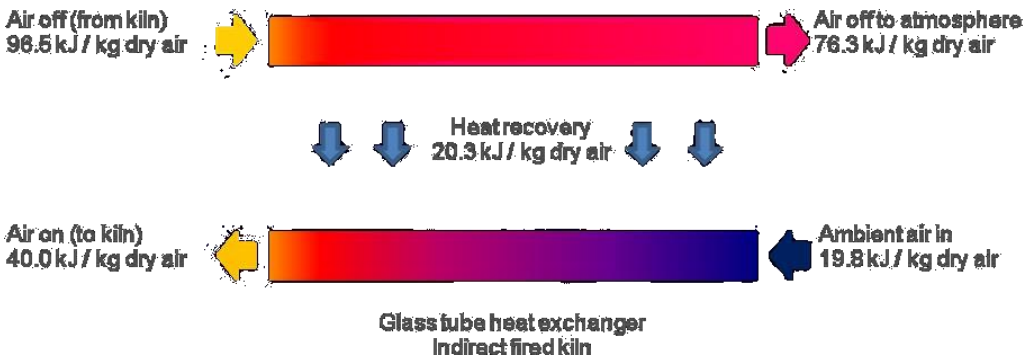

Figure 9. Typical average energy flows in a glass tube heat exchanger during pre-break kilning (AEA, 2011).

1.5.3 The potential for energy savings

Fuels utilized during malting can include fossil fuel natural gas, gas oil, kerosene, and LPG. Their partial replacement with biomass, such as woodchip, could decrease energy costs and carbon emissions.

Another way to save energy could be the use of heat pumps, as a means to enhance the temperature of low grade heat energy to a higher temperature by increasing its utility.

One solution is the application of closed cycle heat pumps after the glass tube heat exchanger, where the working fluid remains in the system. They normally use a refrigerated gas as working fluid and they can be applied as a second stage of energy recovery after the glass tube heat exchangers. Thus, it is possible to recover an additional 43% of energy. The main reason why they are not widely used is the technical viability, as there may not be enough space to fit the condenser heat exchanger.

Another solution that has been reported is the use of open cycle heat pumps, which are able to use the water evaporated from malt as working fluid to save energy.
Last but not least, the implementation of an energy efficient drying system in order to decrease the moisture of malt during the kilning process might be an efficient way to reduce energy costs associated with kilning. In this category is the evaluation of microwave heating as a fast, precise and energy efficient drying method for kilning stage of malting process. This technology could be used in combination with the existing conventional heating system (AEA, 2011).

1.6 Industrial microwave processing

1.6.1 Electromagnetic wave

The electromagnetic wave is formed by the combination of electric and magnetic field (Figure 10) and can be quantified by using the Maxwell's equations (Andrews, 2009). The electromagnetic waves are transverse waves and as they travel through the field, the up and down perturbations form the electric field whilst the side to side perturbations form the magnetic field. The electromagnetic waves differ on their frequencies or equivalently on their wavelengths or energies (Brown, 2003). They are classified depending on their frequency to radio waves, microwaves, visible region, infrared and ultraviolet radiation, X-rays and gamma rays.

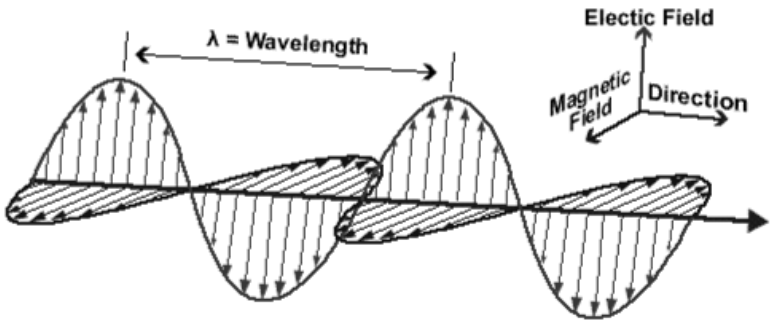

Figure 10. The electromagnetic wave showing the direction of the electric and magnetic field which is perpendicular to each other (http://www.ndt-ed.org/EducationResources).

1.6.2 Microwave and radio frequencies

Radio and microwave frequencies are part of the electromagnetic spectrum (Figure 11) and are considered as non-ionizing frequencies because their energy is below 30 eV which is approximately the energy required to ionize atoms in a human tissue (Grupen, 2010).

Radio and microwave frequencies can be applied as a means of communication in radars, satellites, television and broadcasting. It is always important to notice that any interference between the electromagnetic fields and communication systems must be avoided. Any emissions of electromagnetic energy must be controlled within specific limits for personnel safety and to avoid any interaction with other communication services (Meredith, 1998). For this reason, frequency bands are allocated as industrial, scientific and medical (ISM) bands.

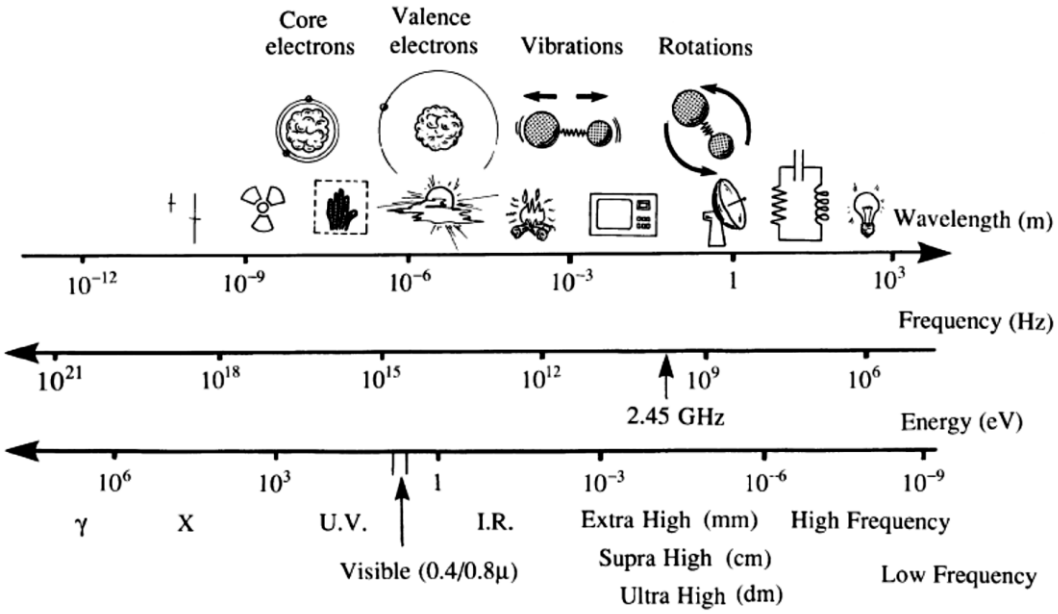

Figure 11. The electromagnetic spectrum showing the range of all wavelengths, energies and frequencies (Stuerga, 2008).

1.6.2.1 Radio frequency heating

In conduction heating, the use of voltage to pass the adequate current in high resistant materials is inhibitive for low frequencies. To outreach this problem, frequency is increased at a range 1-100 MHz and usually to 27.12 MHz and 13.56 MHz that are

ISM frequencies. Radio frequency has been used industrially since the 1930s and can be applied in plastics, wood, food and textile drying (Meredith, 1998).

In this type of heating, the sample is placed between electrodes formed like plates or rods, in high voltage at a selected high frequency (Figure 12). Thus, a capacitor is formed in which the sample is part of the dielectric and the current flows between the electrodes. The presence of the dielectric material causes polarisation that leads to the partial counteraction of the electric field and therefore decreases the electric field strength. This is observed as a drop in the voltage. The structure of the material can be considered as an ideal capacitor in series having a small resistor as a heating source into the material (Meredith, 1998).

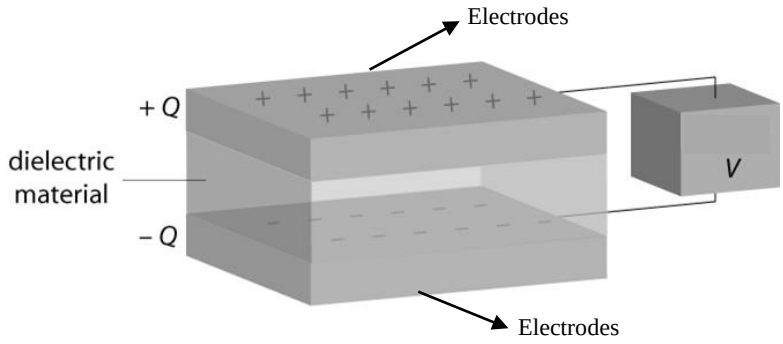

Figure 12. Parallel plate capacitor with a dielectric material between the electrodes (Source: http://www.doitpoms.ac.uk/tlplib/dielectrics/).

1.6.2.2 Microwave heating

Microwave frequencies range from approx. 300 MHz to 300 GHz with wavelengths ranging from 1m to 1mm accordingly. By applying microwaves, heat is generated internally within the material in contrast to external heating sources. As a result, the thermal gradients and flow of heat is the opposite of those in conventionally heated materials (Wheeler, 1963).

Microwave heating in industry is performed at ISM frequency bands such as 915 MHz (USA) and 2.45 GHz (worldwide), according to international agreement with main purpose to avoid interference with communication services. Microwave heating has many applications in industrial processing. Food industry (tempering, continuous baking, vacuum drying and pasteurization), ceramics, plastics and chemical industry are using a lot this technology. The advantages of microwave heating for industrial use is the volumetric and selective heating, the rapid heat transfer and the fact that it is

environmental friendly process because they do not appear any products of combustion (Metaxas and Meredith, 1993).

The absorption of microwave energy due to its propagation depends highly on the dielectric properties of the material. Thermal properties have a small thermal dependence into the heating rate comparing to the corresponding of the dielectric properties. In microwave heating, the irradiated medium acts as converter of the energy unlike to conventional heating. As a consequence, when heating is applied, thermal changes of dielectric properties may cause thermal runaway or decrease of material heating (Stuerga, 2008).

Materials with low loss factor can be heated by applying microwaves better than radio frequencies. In microwave heating, frequencies are higher than radio frequencies and as a consequence the intensity of the applied electric field (known as electric field strength) and the risk of arcing are lower (Meredith, 1998).

1.6.3 Microwave interactions with dielectric materials

Microwaves behave like a beam of light and when they reach an object reflection or transmission may occur (Feng et al., 2012). When a dielectric material is heated by microwaves, energy is either reflected or conveyed through the surface, a part of which is absorbed (Figure 13). The determination of the energy proportions for each case is defined by measuring the dielectric properties (Raghavan, 2005). The interaction process between microwaves and dielectric materials can be done by using two basic heating mechanisms; the ionic conduction and the polarization.

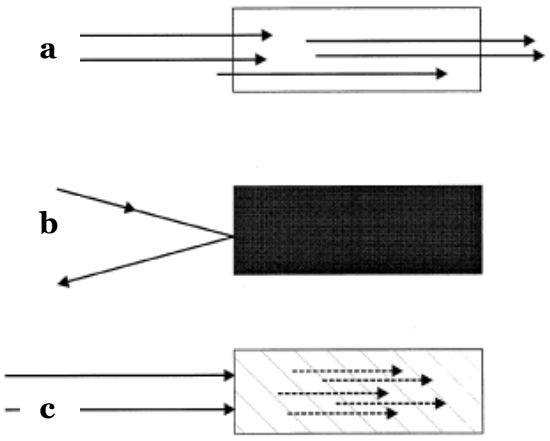

Figure 13. Microwave interaction with several types of materials: a) transparent, b) reflector and c) absorber (Haque, 1999).

1.6.3.1 Ionic conduction

In ionic conduction mechanism, hydrated ions are trying to follow the direction of the electric field (Figure 14) and as a result of this motion energy is transferred (Ryynanen, 1995).

To be more specific, the dissipated charged particles of a sample (mostly ions) oscillate back and forward with the alternating electric field caused by microwave irradiation. Hence, they collide with their neighbouring molecules or atoms and as a consequence movement is caused and heat is created. The effects of ionic conduction are mainly important when studying the thermal behaviour of ionic liquids during a microwave irradiation. The conductivity principle is much more effective than the dipolar polarization in order to generate the heat in different materials (Oliver Kappe et al., 2012).

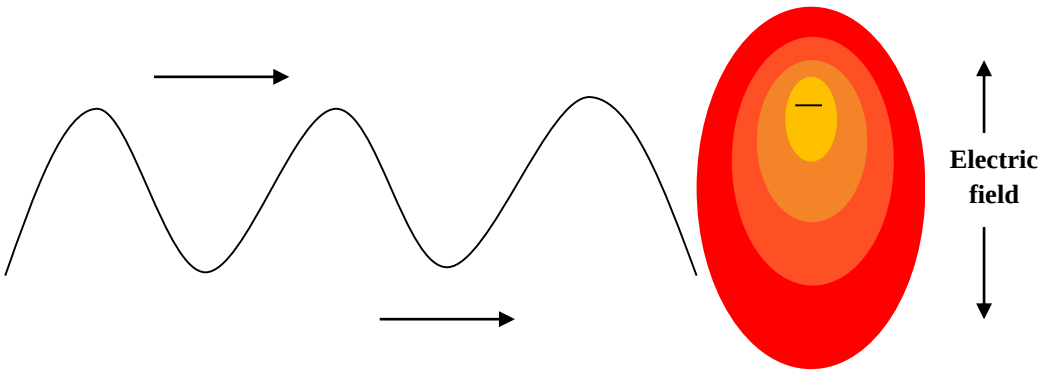

Figure 14. A schematic representation of an electron cloud moving in phase by applying the electric field in a good conductor.

1.6.3.2 Polarization mechanisms

When two opposite polar charges ($\pm q$) have a distance between them, they form an electric dipole that is represented by a vector known as dipole moment, which is pointing from the negative to positive pole (Von Hippel, 1954).

Polar molecules constitute nonzero permanent dipole moments. The constitution of dipole moments can occur instantly for non-polar molecules, due to the deformation of their electronic distributions and nuclear positions. Polarization is defined as the average dipole moment per unit volume. The four types of polarization are interfacial, atomic, dipolar (orientation) and electronic polarization as presented in Figure 15.

In *electronic polarization*, electrons can change their position regarding to the positive atomic nucleus by applying an external electric field. Hence induced dipole moments occur and cause this phenomenon (Von Hippel, 1954). This type of polarization can be found in all compounds and appears in the visible band of electromagnetic spectrum (Ryynanen, 1995).

When atoms are moved in the form of crystals or molecules, a dipolar moment is induced and the *atomic polarization* occurs which appears in the infrared band of electromagnetic spectrum. In this type, resonant frequencies are lower because greater mass has to be moved. Moreover, when both types of polarization are applied, they can give most dry solids a permittivity of $\varepsilon' < 10$. When these are the only types to be applied, materials have a low loss factor at microwave frequencies (Ryynanen, 1995).

In *dipolar polarization*, dipolar molecules in many dielectric materials form electrostatic couples to the microwave electric field and tend to orient themselves with it mechanically. When microwaves interact with dipoles the latter are rotated and heat is scattered from internal resistance to the rotation.

Thus, since microwave field is changing in time, dipoles will try to realign as the field reverses and therefore remain in a permanent state of mechanical oscillation at the microwave frequency (Meredith, 1998).

Most biological materials consist of water which is a polar molecule. When dipole polarization occurs, the energy is lost in random to the thermal movement of the water which results to an increase in temperature. For that reason, dipolar polarization is strongly affected by temperature.

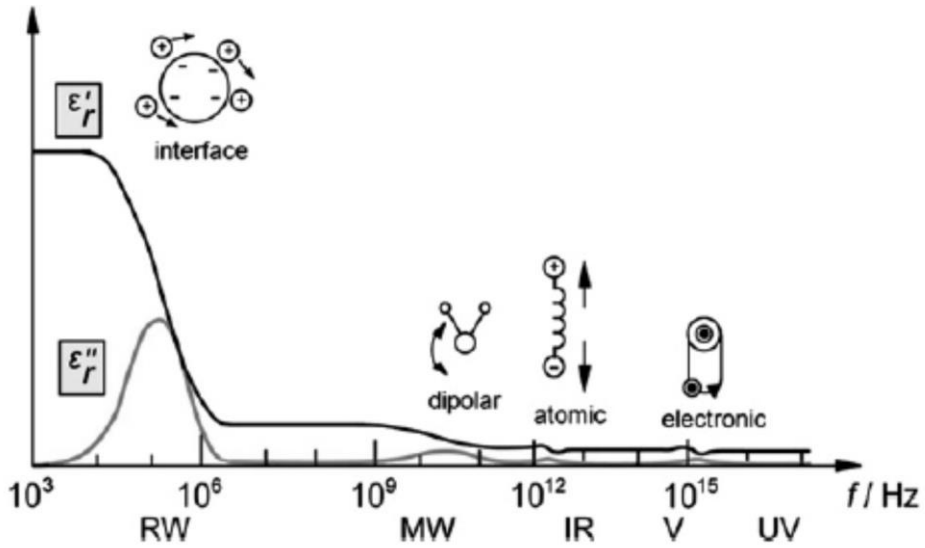

Figure 15. Dielectric permittivity spectrum of electromagnetic wave frequencies during various processes (Justice et al., 2011).

When charges that have a distance between them are obstructed in their movement either because they are caged in the material or on interfaces or because they are unable to be discharged, a distortion happens that is known as *interfacial polarization* (Von Hippel, 1954).

Dipolar polarization is the most dominant type when microwave heating is applied at frequencies above 1 GHz but it has also an impact to lower frequency bands. In contrast, for frequencies below that range ionic conductivity is the most significant type of polarisation, strongly depended to temperature (Ryynanen, 1995). Due to the fact that germinated barley consists mostly of water that needs to be dried through kilning process to produce the final malt product, dipolar polarization and ionic conductivity are the dominant mechanisms that affect the dielectric properties of malt and exist when applying the electromagnetic energy to dry the grains.

1.6.3.3 Microwave interactions with water

Water interacts in different ways with microwaves depending on its form. In dilute solutions, water molecules are either modified by the presence of ions or keep all the properties of pure water, known as bulk water. In dense solutions, bulk water does not exist and two types of water molecules appear: those that are bounded by one ion and those that are shared by cations to compose ion-water clusters. In ionic solutions, dielectric losses as well as relaxation of water are considered to be insignificant because ionic conduction is predominant and hence conduction losses appear (Stuerga, 2008).

For instance, for two samples containing equal quantities of distilled and tap water, respectively, heated by microwaves at a standard radiation power, tap water will be heated faster because of its ionic content (Oliver Kappe et al., 2012).

The permittivity of a strongly polar solvent such as water is following the Debye relation:

$$\varepsilon = \varepsilon_{\infty} + \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + j\omega\tau} \qquad \text{(Equation 1.1)}$$

ε_{∞} = real permittivity at frequency that tends to infinity

ε_s = static permittivity

τ = relaxation time

ω = angular frequency ($=2\pi f$)

According to this equation, relaxation time τ represents a time constant in the final state of polarization when polar molecules turn slowly and exponentially. At low frequencies, dipoles have a constant permittivity which has the maximum value ε_s. In terms of pure water, static permittivity at room temperature is approximately 80. Real permittivity is also permanent at high microwave frequencies where free water dipoles are unable to follow the quick alternation of the field. The permittivity for infinite frequency is not influenced by temperature. Dielectric loss factor is minor for low and high frequencies. However, at the relaxation frequency, dielectric loss factor achieves its maximum value which is half of the difference between static (ε_s) and real permittivity that tends to infinity (ε_∞) as it is represented in Figure 16.

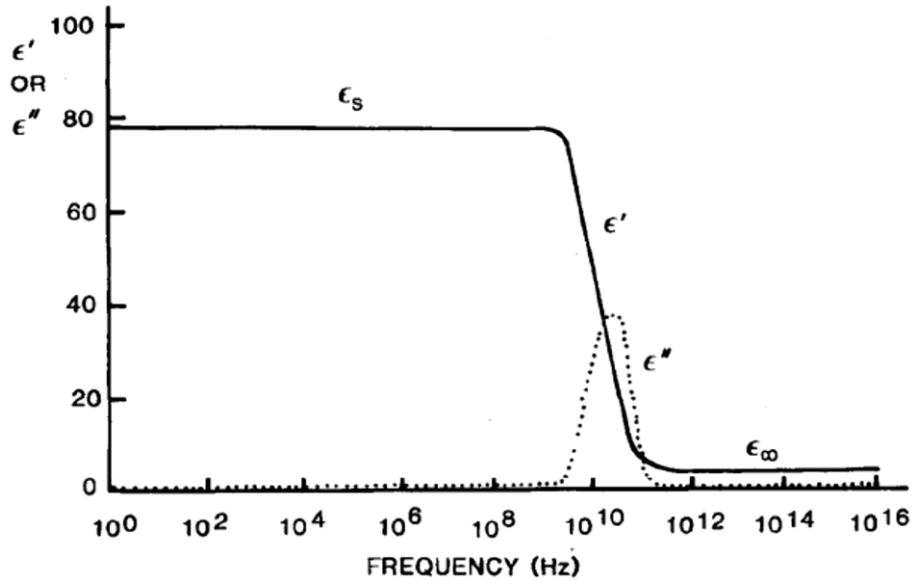

Figure 16. Diagrammatic representation of dielectric behaviour of free water at a constant temperature at different frequencies (Mudgett, 1985).

When hydrogen bonds limit the free motion of water molecules, bound water is formed which has a structure between those of ice and normal "free" water. For bound water molecules relaxation frequency is lower than for free water molecules, whereas static permittivity (ε_s) has the same value as ice (Ryynanen, 1995). In food materials, if water is strongly bonded with the proteins or carbohydrates, the contribution of bound water to the dielectric properties of the material is smaller at high moistures in microwave frequencies (Sumnu et al., 2005). However, in hygroscopic foods the effect of bound water cannot be ignored as it could be the 16-28% of the total moisture and consequently has a significant role in the drying process of the material at a level below 0.6% (Feng et al., 2012).

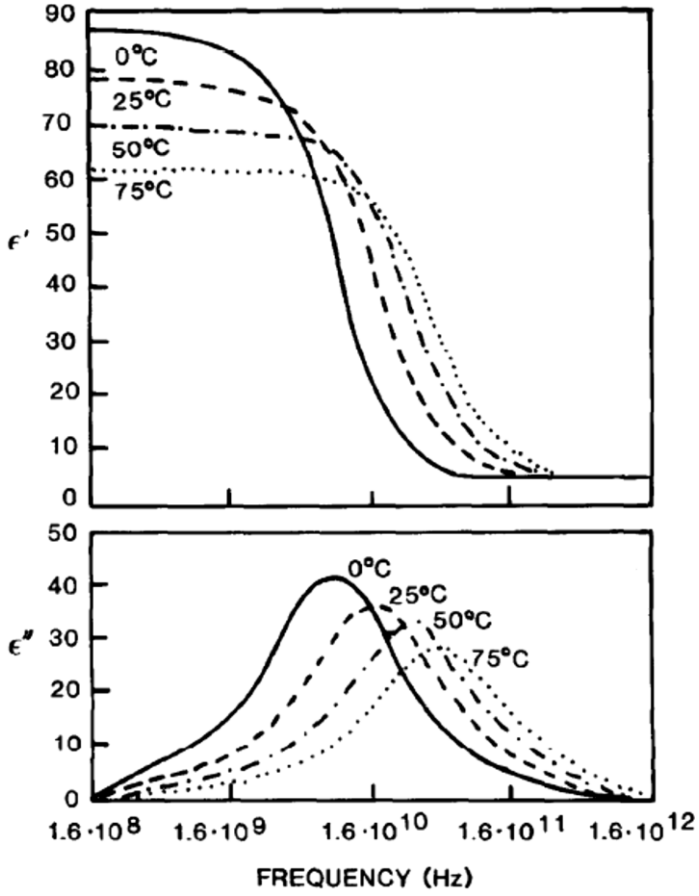

Figure 17. Temperature effect on ε' and ε'' of water (Mudgett, 1985).

During thawing of substances with a high concentration of water, both ice and water dielectric constant and loss factor are increased while temperature is rising. After thawing, these values decrease when temperature increases as showing in Figure 17 (Ryynanen, 1995).

1.6.4 Dielectric properties of materials

Dielectrics are a class of materials that are considered to be poor conductors of electricity in contrast to metallic materials (Nelson, 2010). There is a great variety in dielectric properties of materials depending on composition, density, temperature and frequency (Meredith, 1998). The knowledge of dielectric properties is primary in the design of heating systems. It predicts the power density and the associated electric field strength and is also essential for the microwave penetration depth in the material (Von Hippel, 1954).

1.6.4.1 Complex permittivity

Complex permittivity is a variable that indicates how microwaves interact with materials by describing their ability to absorb, propagate or reflect electromagnetic energy (Ryynanen, 1995).

Dielectric materials have different permittivity than that of free space. It is a complex quality represented by the following equation:

$$\varepsilon = \varepsilon' - j\,\varepsilon'' \qquad \text{(Equation 1.2)}$$

ε = complex permittivity

ε' = dielectric constant

$j = \sqrt{-1}$

ε'' = dielectric loss factor

Dielectric constant (real part) shows the ability of a material to store electrical energy, whereas dielectric loss factor (imaginary part) shows its ability to convert stored energy into heat. Thus, dielectric loss factor indicates the potential of a material to warm up using microwave or radio frequency heating (Nelson, 2010). The real part, ε', affects the transmission of waves in electric fields and is usually much bigger than the loss factor. If the imaginary part is $\varepsilon''=0$, that means that the material has zero loss factor and therefore it does not absorb energy (Ryynanen, 1995).

The ratio of the dielectric loss factor to the dielectric constant is known as the loss tangent ($\tan\delta$) and is a term that quantifies how low or high loss (Figure 18) a material can be (Yaw, 2012).

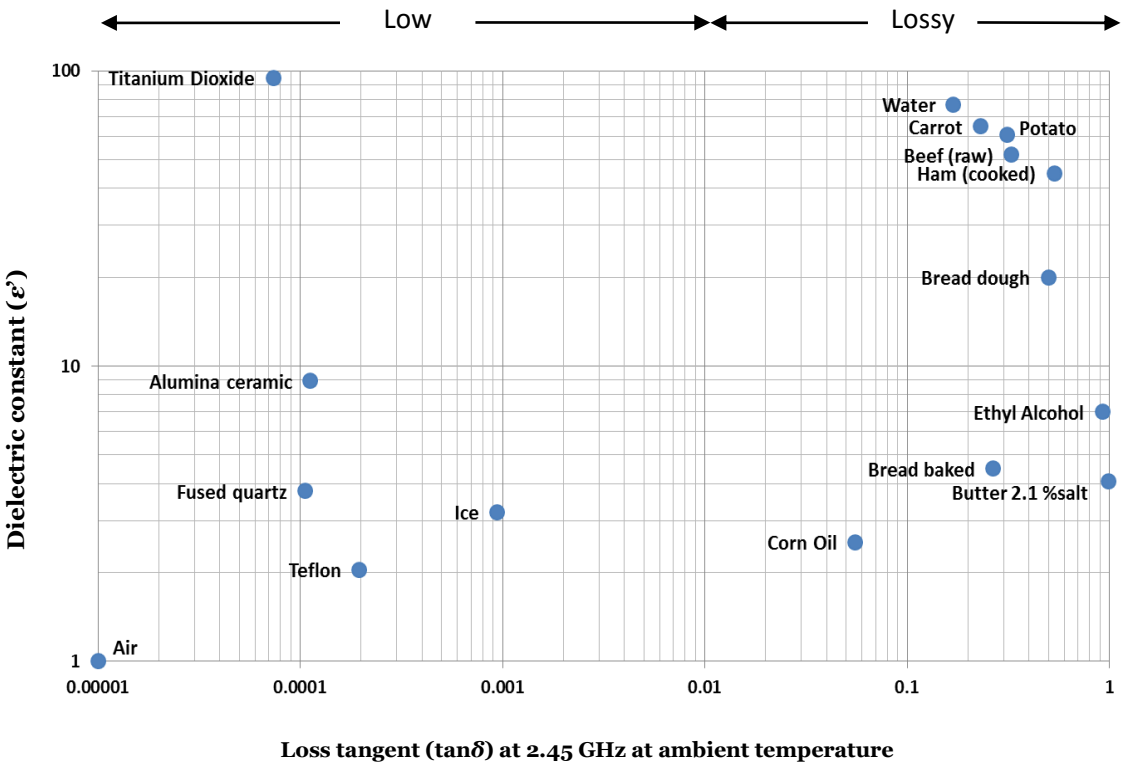

Figure 18. Graphic representation of common materials and their dielectric properties according to their loss tangent at 2.45 GHz and ambient temperature (Meredith, 1998).

1.6.4.2 Power Density

Power density (p) is a fundamental factor in order to select the appropriate frequency for usage to a certain application of radio frequency or microwave heating as it governs heating rate in each of the different phases of a material.

$$p = 2\pi f \varepsilon_0 \varepsilon'' E_i^2 \qquad \text{(Equation 1.3)}$$

p = Power density (W/m^3)
f = frequency
ε_0 = permittivity of free space (8.85 x 10^{-12} F/m)
ε''= dielectric loss factor
E_i = electric field strength (V/m)

When electromagnetic energy is propagated into a material, the latter is heated according to the dielectric and/or magnetic losses and the electric and magnetic field

strength. Thus, the estimated dissipated power density depends straight on the electric and magnetic fields and the distribution of the electric and magnetic losses (Stuerga, 2008).

Moreover, the power density propagated in a material is proportional to frequency where the other parameters are permanent. In other words, as the frequency rises, the volume of the material in the oven can be decreased leading to a more compact oven. Power density shows also a similar trend with the loss factor (Meredith, 1998).

1.6.4.3 Penetration depth

Penetration depth (D_p) is determined as the depth below the surface of a material in which microwave power is reduced to 1/e (36.8%) of its propagated value. In some cases, penetration depth is considered to be the distance at which the power of microwaves is weakened to half of its transmitted power (Raghavan, 2005).

To be more specific, the width of a wave that proceeds into a dielectric heating material decreases because of the power absorption. When reflected waves do not exist, the field intensity and its power flux density drop exponentially with distance from the surface. Power dissipation has also the same trend, as the power absorbed in a primary volume of material is proportional to the power flux density that flows through it.

Penetration depth is an essential parameter for materials, because it gives a direct indication of the heat that has been distributed into it. It is remarkable to notice that penetration depth is not an index that heat does not exist at a depth over that limit (Meredith, 1998).

Penetration depth (D_p) is given by the following equation where $\varepsilon'' \leq \varepsilon'$:

$$D_p = \frac{\lambda_0 \sqrt{\varepsilon'}}{2\pi\varepsilon''} \qquad \text{(Equation 1.4)}$$

λ_0 = free space wavelength of incident radiation

Penetration depth increases when frequency decreases. At frequencies below 100 MHz penetration distance for dielectric materials is close to meters while for pure water at frequency of 2.45 GHz is close to centimetres. Thus, in a microwave reactor filled with a high loss material the penetration depth is very small (Stuerga, 2008). These materials that have high conductivity and low capacitance are considered as

reflectors. For low loss materials, penetration depth is very large and consequently only a very small amount of energy is absorbed by the material that is transparent to microwave energy. For that reason, microwave energy is transferred more efficiently to materials with dielectric loss factors in the middle of the conductivity range as it is presented in Figure 19 (Thostenson and Chou, 1999).

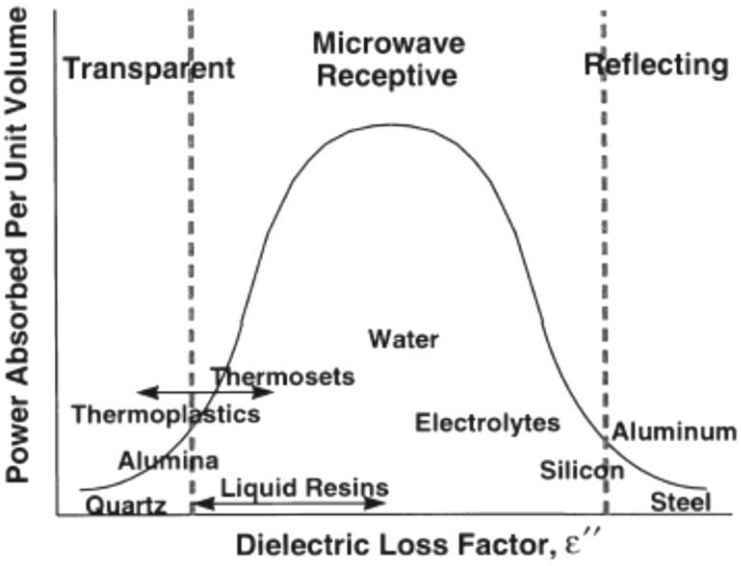

Figure 19. Schematic diagram of the relationship between the dielectric loss factor and microwave power absorbed by some common materials (Thostenson and Chou, 1999).

1.6.4.4 Factors affecting dielectric properties

The main factors that affect the dielectric properties of materials are:

- Frequency of applied electromagnetic field
- Temperature
- Moisture content
- Density and structure of the material
- Bulk density of granular materials
- Chemical composition

When polarization occurs due to the orientation of molecules with permanent dipole moments while electric field is applied, dielectric constant remains permanent at low and high frequencies compared to molecular relaxation process and the losses are negligible. At intermediate frequencies, dielectric constant is dispersed and losses are higher at the relaxation frequency which is inversely proportional to relaxation time

($\omega=1/\tau$) (Nelson, 1994). The dielectric loss factor usually fluctuates with frequency, especially in dipolar loss materials (Meredith, 1998).

In order to understand the impact of temperature to the dielectric properties of food products it is important to understand how the free water, the bound water and the ionic conduction affect the dielectric dispersion (Feng et al., 2012). In the area of dispersion, the dielectric constant rises when this parameter is increased and the loss factor fluctuates depending on the behaviour of the operating and the relaxation frequency. Below the area of dispersion, when the temperature increases, dielectric constant decreases (Nelson, 1994).

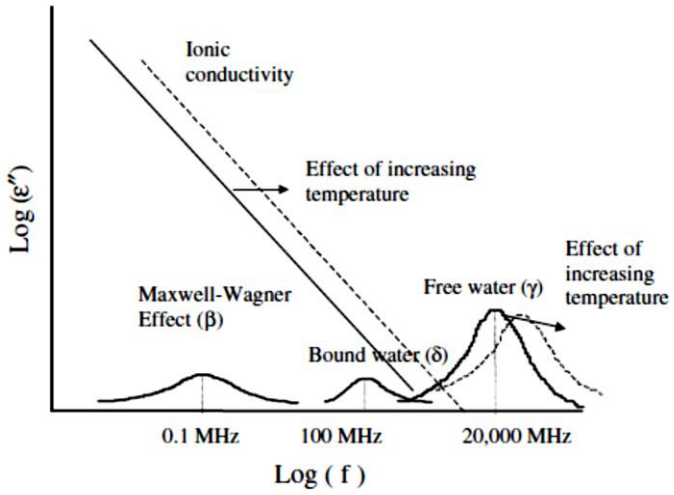

Figure 20. Frequency and temperature effects of different dispersion mechanisms on loss factor (Feng et al., 2012).

According to the Fig. 20, in the microwave frequencies, bound water, free water and ionic dispersion mechanisms can be involved in the effect of temperature and frequency. The percentage of bound and free water in a food material defines the behaviour of its dielectric properties to the temperature and frequency alterations. Ionic conductivity always has a positive effect to the dielectric properties with increasing temperature (Feng et al., 2012).

Moisture content has a strong impact on the dielectric properties of materials which it is generally observed that they increase or decrease following the respective trend of moisture content. Dielectric properties have a critical moisture content (M_c) which define the limits between free water and bound water. The water at moisture content below the M_c is known as bound while the water at moisture above this critical zone is known as free water. In the graph below (Fig. 21) a vigorous decrease in the dielectric

properties is observed following the downward trend of the moisture content due to the slow mobility of the water dipoles. (Feng et al., 2012).

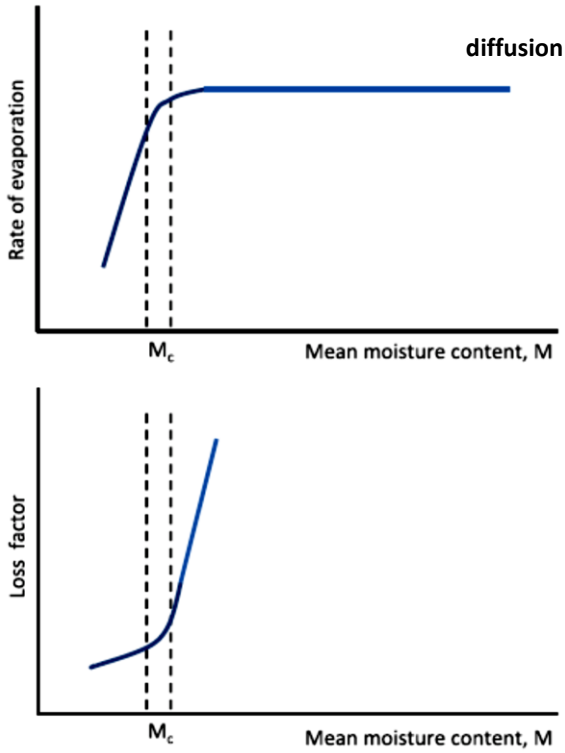

Figure 21. The impact of moisture content on the dielectric properties of food materials (Metaxas and Meredith, 1993).

Bulk density is another important parameter especially for granular and pulverized materials such as cereal grains. There is a linear relationship between the relative complex permittivity and bulk density in particulate materials (Nelson, 1994).

1.6.5 Measurement of dielectric properties

Measurements of dielectric properties include the determination of dielectric permittivity and dielectric loss factor of the materials. There are many methods to measure the dielectric properties depending on the frequencies, the materials and the applications. Some of these methods are transmission line, open-ended coaxial probe/line, free space and resonant method (Yaw, 2012).

According to studies on the measurements of dielectric properties of grains and seeds at microwave frequencies, bulk density of grain can be detected along with moisture content. For granular and pulverized materials, the permittivity depends on the bulk density of the material (Nelson, 2010).

1.6.5.1 Transmission line

This is a broadband method that allows the measurement of both permittivity and permeability of the dielectric material. In this method the sample is placed in a section of waveguide or coaxial line, while a vector network analyser (VNA) is used to measure the S-parameters (Figure 22) and then using special algorithm to extract the dielectric properties. The relevant scattering parameters known as the S-parameters are related with the complex permittivity and permeability of the sample by equations (Yaw, 2012). Waveguides are hollow tubes, usually in rectangular intersection, through which electromagnetic waves are propagated. This method includes the measurement of the reflected (S11) and transmitted (S21) signal. Prior to measurement, calibration of the connector plane should be done.

In order to convert S-parameters to complex dielectric parameters a software program is used. The sample should be usually machined before the measurement in order to fit tightly into the waveguide or coaxial line to reduce faults in measurements provoked by air gaps. In order to achieve better results, the electric field should reach a maximum level and that can be achieved by open circuited or other capacitive end.

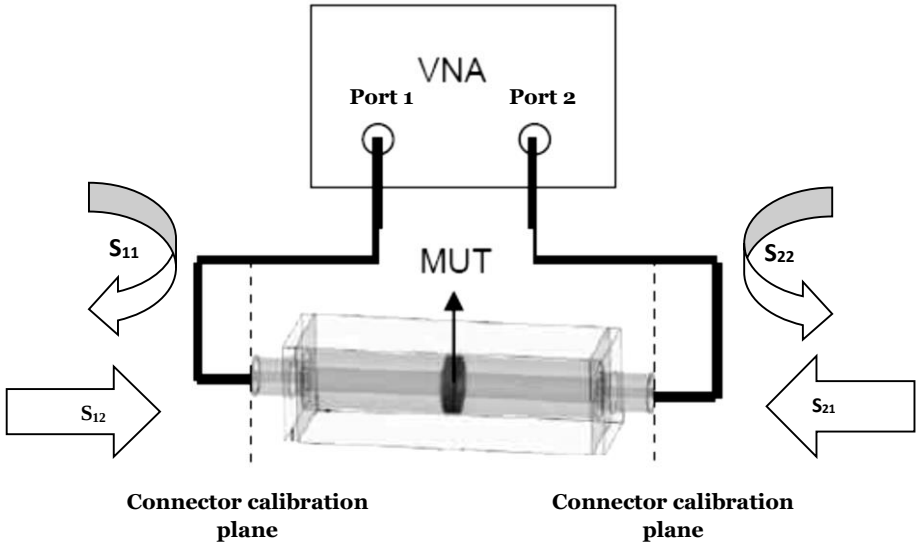

Figure 22. Measurement of the S-parameters of a material under testing (MUT) using transmission line method with a waveguide (Yaw, 2012).

This method is suitable for medium and high loss material and the measurement of both permittivity and permeability of the testing material. In contrast, one of the problems that may rise is the limited accuracy of the measurement by the effect of the

air gaps. However, the accuracy is high when the length of the sample is much bigger than one half of the wavelength within the material (Yaw, 2012).

1.6.5.2 Open-ended Coaxial line/probe

The open-ended coaxial probe technique is used as a non-destructive method (Yaw, 2012) to measure dielectric properties of materials such as agricultural products (Nelson, 2010). In order to calculate the permittivity, this method uses an open-ended coaxial probe that is pressed against a sample and the reflected signal is measured using network analysers as it is shown in Figure 23 (Raghavan, 2005).

This method is suitable for diverse measurements on fluids and high loss materials because air gaps between the probe and the material can be avoided. Thus, the sample is in close contact with the probe without being physically damaged (Yaw, 2012).

The measurement of the reflection signal can be done by using a vector network analyser (VNA). At first, VNA is calibrated in order that the reflection coefficient measurement is referenced to probe aperture plane. This can be done either by using reference liquids for direct and easy calibration at the open end of the probe or by using a combination of standard calibration for the connector plane and a simulated model of the probe to translate the connector calibration plane.

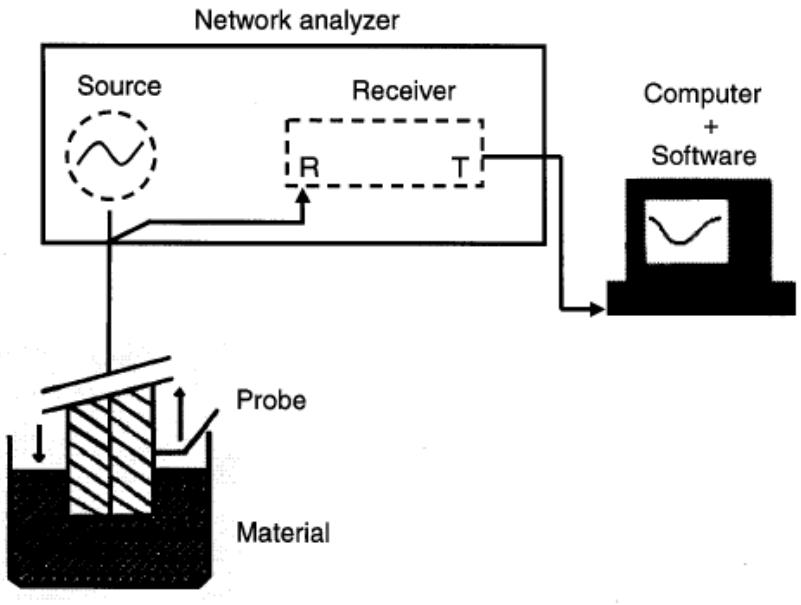

Figure 23. Measurement of liquid sample using open-ended coaxial-line probe (R=reflected power / T=transmitted power) (Raghavan, 2005).

The advantages of this method are the simple preparation of the sample and that does not require any special preparation such as machining. Dielectric properties of many samples can be measured rapidly after calibration and the measurements can be performed in ambient temperature. The fact that only reflection can be measured is a disadvantage as well as that some measurements are affected by air voids (Yaw, 2012).

1.6.5.3 Cavity perturbation

This method is suitable for determining the dielectric properties of homogenous materials especially for food services. It is a simple and accurate method, quite capable for high temperature and low dielectric loss materials (Raghavan, 2005).
Cavity perturbation can occur by either modifying the volume or entering small pieces of dielectric material. The first method is known as shape perturbation while the second is the material perturbation which is applied in the dielectric measurements.
Moreover, for dielectrics with small loss tangents, this method gives highly accurate measurements and is more beneficial in the definition of relative permittivity. A great advantage of this method is the fact that it can measure dielectric samples of small sizes and various shapes such as spheres, rods, discs and slabs (Balmus, 2006).

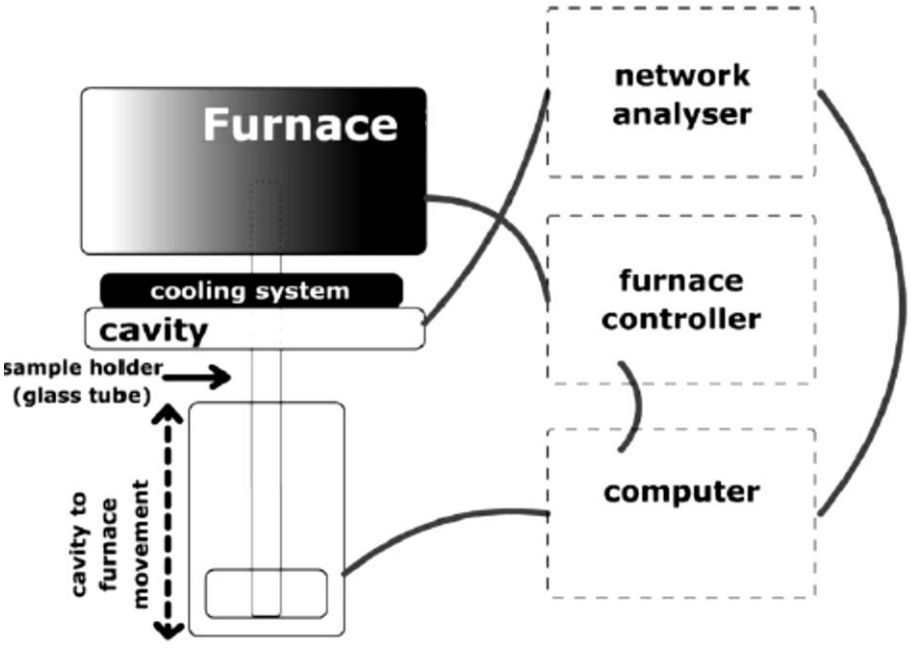

Figure 24. Schematic representation of a cavity perturbation method (Lester et al., 2006).

The propagation of the electromagnetic fields for resonant cavities is designed in the standard transverse magnetic (TM) or transverse electric (TE) mode. The method is based on the shift in resonant frequency and the alteration in absorption characteristics of a tuned resonant cavity. In order to do the measurement, the whole sample is placed through the centre of the cavity as presenting in Figure 24 (Raghavan, 2005).

In order to determine the dielectric properties, firstly the resonant frequency and the quality factor of an empty cavity are measured. After that, the measurement is repeated by filling the cavity with the testing sample. The complex permittivity and permeability are then calculated using the frequency, volume and Q-factor. The calibration of the VNA is not necessary for this method. Thus, this method is appropriate for the measurement of small and low loss samples, but it is limited to specific band of frequencies (Yaw, 2012).

1.6.5.4 Free space method

This method is broadband and non-destructive and allows the measurement of material under high temperatures or for inhomogeneous materials (Raghavan, 2005). The testing material must be of large and flat type size. For the measurement two antennas are used facing each other and they are connected to a vector network analyser.

Thus, the free space method is suitable for measurement of a wide range of frequencies including high frequency for both magnetic and electric properties. However, multiple reflections between antenna and surface of the sample may occur and diffraction effects at the edge of the sample are also possible (Yaw, 2012).

Chapter 2 Materials and Methods

2.1 Micromalting process

2.1.1 Raw Material

For the preparation of the malt samples, either spring barley (variety *Tipple*) or winter barley (variety *Flagon*) supplied by Crisp Malting Group (Gt Ryburgh, Fakenham, Norfolk, NR21 7AS) were used.

2.1.2 Steeping and Germination processes

Barley was malted using CustomLab Micromaltings equipment (Custom Laboratory Products, The Mains of Balnamoon, Banffshire, UK). The steep/germinator unit (Figure 25a) was a 2-tank system (140cm x 80cm x 153cm) where each tank was capable of holding 4 x 500 g samples of barley in cylindrical vessels (Figure 26). For the preparation of larger amounts of green malt, metallic slotted rectangular plates (one for each tank) could be rested on the rollers at the base of each malting compartment, enabling the whole compartment to be filled with barley which extends the capacity of each chamber up to 8 kg of barley. When using the latter procedure, the automatic turning of vessels is inactivated and manual mixing of malt was required through germination to avoid rootlet matting twice per day during the process.

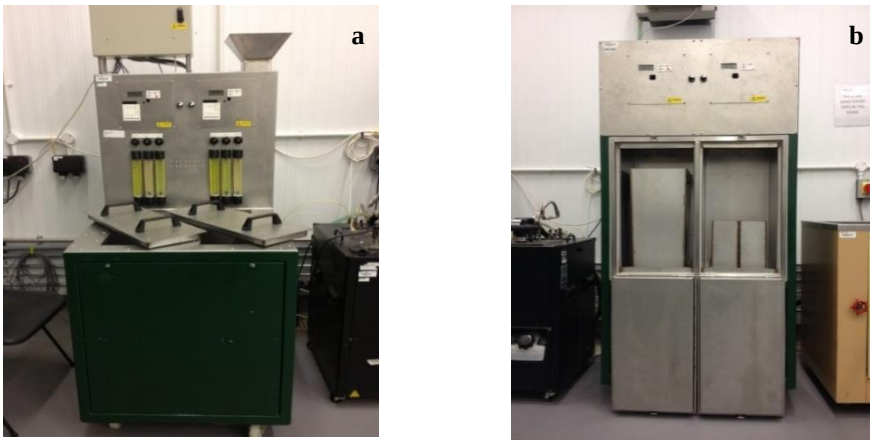

Figure 25. CustomLab micromalting equipment used in the research: a) Steeping/Germination and b) Kilning Units showing the use of either one large kiln vessel (*Left*) or 4 smaller kiln vessels (*Right*).

Figure 26. Steep/germinator cylindrical vessel used in the research containing germinated barley.

Table 2. Details of the "standard" micromalting protocol utilized in the steeping and germination process.

Steeping	**Germination**
16°C	16°C
Equilibration: 1h	Germ1: 24h
Wet1: 7h	Germ2: 24h
Dry1: 12h	Germ3: 24h
Wet2: 8h	Germ4: 24h
Dry2:12h	
Wet3: 4h	
1 day 19h	**4 days**

2.1.3 Kilning process

Two kiln compartments (Figure 25b) could be fitted with either 4 small kiln vessels (each of which takes the contents of one micromaltings cage (Figure 26)) or alternatively with one large kiln vessel, depending on the desired scale of micromalting experiment. The small kiln vessels (15cm x 30cm x 15cm) have a capacity of 4 x 0.7 kg whereas the large vessels (25cm x 50cm x 25cm) hold 8 kg of green malt each (Figure 25b). As the malt beds were static and the airflow was forced through the grain rather than around it, representative kilning was achieved. During kilning, air-on temperature, air-off temperature, humidity and airflow were continuously monitored and logged. According to the procedure, the "Standard Ale1" Malting Programme on the CustomLab Micromaltings was followed (Table 2). This standard cycle which was used for the drying of small quantities lasted for 23 h with a start temperature of 55°C and a maximum temperature of 95°C.

Table 3. Details of the "standard" micromalting protocol utilized in the kilning process.

Kilning
Kiln1: 12h, 55ºC
Kiln2: 6h, 65ºC
Kiln3: 2h, 85ºC
Kiln4: 2h, 95ºC
Cooling: 1h, atm.
23 hours

2.2 Malt analysis tests

2.2.1 Moisture content

Moisture content was measured for all samples in triplicate by using the HR83 Halogen balance analyser (Mettler-Toledo, Beaumont Leys Leicester, UK). To run the test, two methods of the analyser were used with temperature profile at 105°C for malt samples and 130°C for barley samples with switch-off criteria set to a sensitivity of 1 mg moisture loss per 90 sec. Normally, the malt method is designed to measure low moistures (20% and lower) and the barley method for higher moistures (45% to 20%). However, a comparison of both methods to a range of hydrated malt from 45% to 5% was performed to test if the application of a different method could provide significant changes in the moisture content results as presented in Section 3.4.

Thus, if the analyser cannot detect above 1 mg moisture loss in 90 secs, it recorded the final reading. For each test, 2 g of a homogeneous ground sample were used. The test was simple, rapid and accurate however it was compared to the industry standard oven drying method which is the "Analytica-EBC" 4.2 for the malt and the 3.2 for barley grains. The reason why a different method is used for malt is because the conditions of drying enhance the chemical condensations in which water is formed as a product of reaction (Analytica-EBC, 2000). The oven used for the comparison of the methods was a Genlab Model OV/100/F/DIG.

2.2.2 Analysis of α-Amylase activity

The α-Amylase analysis followed the "Megazyme" Ceralpha Method. The principle of the Ceralpha assay is presented in the following picture:

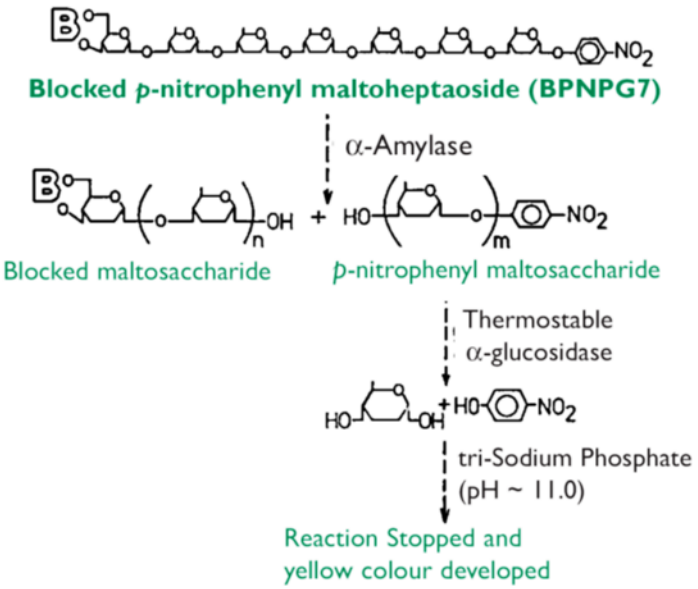

Figure 27. Schematic description of the reactions that take place to measure the α-amylase activity according to the Ceralpha megazyme method.

As shown in Figure 27, the α-amylase cleaves a bond within the blocked *p*-nitrophenyl maltosaccharide substrate, while the non-blocked reaction product which contains the *p*-nitrophenyl substituent is instantly cleaved to glucose and free *p*-nitrophenol is released by the excess quantities of thermostable α-glucosidase and free *p*-nitrophenol. The end of the reaction is signalled by the appearance of phenolate colour on addition of tri-sodium phosphate (pH ~ 11.0).

The assay procedure included the dispense of 100 μL aliquots of substrate solution (unbuffered) into the polypropylene 13 mL tubes provided and pre-incubation at 40°C for 5 min. Ten such tubes needed for duplicate measurements on five malt samples. Likewise, the eppendorfs were pre-incubated containing saline malt extracts at 40°C for 5 min. To each tube containing substrate solution, 100 μL of a specific pre-equilibrated saline malt extract added (duplicate tubes for each malt samples). The tubes were stirred on a vortex mixer and then were incubated at 40°C for exactly 10 min. When this time period was finished, 1.5 mL of the Stopping Reagent was added to each tube and the tubes were stirred again. The absorbance of the solutions at 400 nm was measured using polystyrene cuvettes, 1.6 ml, semi-micro in the JENWAY 7315 Spectrophotometer against distilled water. A single reaction blank only was normally sufficient for each batch of samples analysed. It was prepared by mixing: 1.5 mL stopping reagent, 0.1 mL substrate solution and 0.1 mL of one saline malt extract.

For the calculation of the α-amylase content, the Megazyme α-amylaze extraction method was followed:

Units (CU)/gram of milled malt:

$$\frac{\Delta E400}{\text{Incubation Time}} \times \frac{\text{Total Volume in Cell}}{\text{Volume of aliquot assayed}} \times \frac{1}{\text{EmM}} \times \frac{\text{Extraction volume}}{\text{Sample Weight}} \times \text{Dilution}$$

$$= \Delta E400 \times 376$$

ΔE_{400} = Absorbance (reaction) – Absorbance (Blank)

Incubation time (10 min) = 10

Total volume of reaction (0.1 mL extract + 0.1 mL substrate + 1.5 mL stopping solution) = 1.7

Volume of aliquot (extract) assayed = 0.1

E_{mM} of *p*-nitrophenol at 400nm in 1% tri-sodium phosphate = 18.1
Extraction volume (100 mL/ 0.5 g sample) = 100
Dilution of the original extract = 20-fold for malt extracts

For the conversion of Ceralpha Units (CU) to Dextrinising Units (DU) the equation below is used:

$$\mathrm{DU} = (0.23 \times \mathrm{CU}) + 0.61$$

For the preparation of the α-amylase reagents, the following procedures were used:
For the "Buffer A" solution, 50 mL of the concentrated "Buffer A" is diluted in 1 L ultrapure water and then the pH was set in 5.4. It was then stored in the cold room (4°C) until use.
For the substrate solution (HR reagent), the blocked *p*-nitrophenyl maltoheptaoside (BPNPG7, 54.5 mg) and the thermostable α-glucosidase (125 U at pH 6.0) were dissolved in 10 mL of ultrapure water. Then, this substrate solution was divided into 6 eppendorf tubes (1.5 mL) and stored in the freezer at -20°C until use. For the Stopping Reagent, the concentrated stopping reagent solution was diluted with 500 mL RO water and then stored in the cold room (4°C) until use.

2.2.3 Analysis of β-Amylase activity

The β-Amylase analysis followed the "Megazyme" Method. The principle of the "Betamyl-3" assay is presented in the following picture:

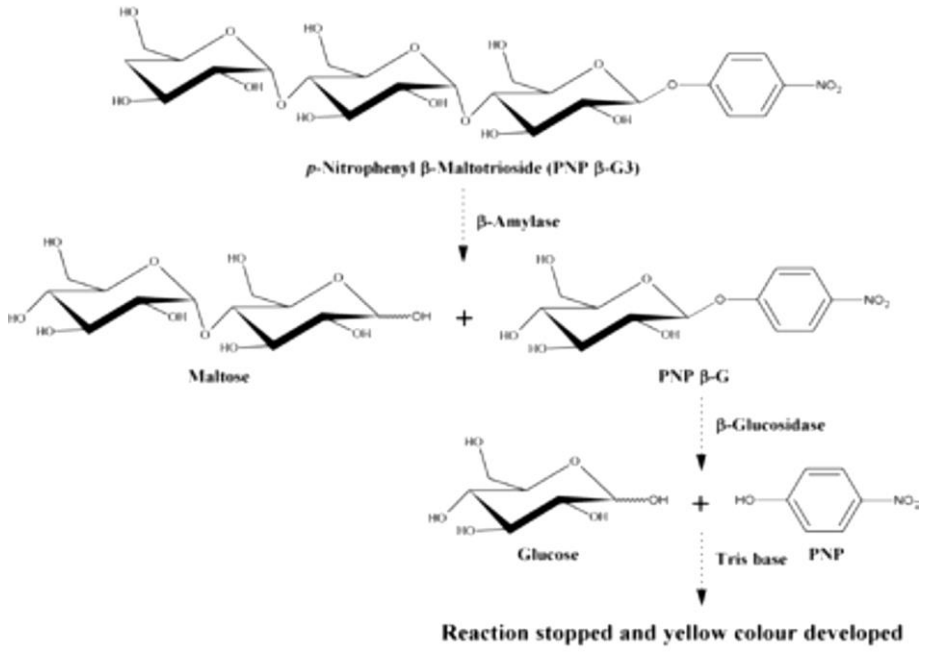

Figure 28. Schematic description of the reactions that take place to measure the β-amylase activity according to the Betamyl-3 megazyme method.

As presented in Figure 28, the blocked *p*-nitrophenyl-β-D-maltotrioside (PNPβ-G3) is cleaved to maltose and PNPβ-G. Then the PNPβ-G is rapidly cleaved to *p*-nitrophenol and glucose by the excess quantities of β-glucosidase which is an integral part of the reagent mixture. The end of the reaction is signalled by the appearance of yellow colour.

The assay procedure included the dispense of 100 μL aliquots of substrate solution (unbuffered) into the polypropylene 13 mL tubes provided and pre-incubation at 40°C for 5 min. Ten such tubes needed for duplicate measurements on five malt samples. Similarly, the eppendorfs were pre-incubated containing saline malt extracts at 40°C for 5 min. To each tube containing substrate solution, 100 μL of a specific pre-equilibrated saline malt extract added (duplicate tubes for each malt sample). The tubes were stirred on a vortex mixer and then were incubated at 40°C for exactly 10 min. When this time period was finished, 1.5 mL of the Stopping Reagent was added to each tube and the tubes were stirred again. The absorbance of the solutions at 400 nm was measured using polystyrene cuvettes, 1.6 ml, semi-micro in the JENWAY 7315 Spectrophotometer against distilled water. A single reaction blank only was normally sufficient for each batch of samples analysed. It was prepared by mixing: 1.5 mL stopping reagent, 0.1 mL substrate solution and 0.1 mL of the "MES buffer".

For the calculation of the β-amylase content, the Megazyme β-amylaze extraction method was followed:

Units (CU)/gram of milled malt:

$$\frac{\Delta A_{400}}{\text{Incubation time}} \times \frac{\text{Total Volume in Cell}}{\text{Aliquot Assayed}} \times \frac{1}{\text{EmM}} \times \frac{\text{Extraction Volume}}{\text{Sample Weight}} \times \text{Dilution}$$

$$= \Delta A_{400} \times 19.72$$

ΔA_{400} = Absorbance (reaction) – Absorbance (Blank)

Incubation time (10 min) = 10

Total volume of reaction in cell (0.1 mL extract + 0.1 mL substrate + 1.5 mL stopping solution) = 1.7

Volume of aliquot (extract) assayed = 0.1

E_{mM} of *p*-nitrophenol at 400nm = 18.1

Extraction volume (5 mL/ 0.5 g sample) = 5

Dilution of the original extract = (21 fold)

For the preparation of the β-amylase reagents, the following procedures were used:

The "MES buffer" 48 mL, 1 M, pH 6.2 plus disodium EDTA (20 mM), BSA 10 mg/mL and sodium azide (0.20% w/v) was diluted in 500 mL ultrapure water and then the pH was adjusted to 5.4. This extraction buffer was then stored in the cold room (4°C) until use.

For the substrate solution (HR reagent), the blocked *p*-nitrophenyl-β-D-maltotrioside (PNPβ-G3, 54.5 mg) and the β-glucosidase (50 U) were dissolved in 10 mL of ultrapure water. Then, this solution was divided into 6 eppendorf tubes (1.5 mL) and stored in the freezer at -20°C until use. For the Stopping Reagent, 10 g of 1% w/v Trizma base was diluted in 900 mL of ultrapure water and pH was then adjusted to 8.5. The solution was filled ultrapure water to 1 L and was stored in the cold room (4°C) until use.

The kit also contained a Tris/HCl buffer (25 mL, 1M, pH 8.0) plus disodium EDTA (20 mM) and sodium azide (0.20% w/v) stored at 4°C. 1.25 mL of this buffer was diluted in 25 mL of RO water and 0.44 g of cysteine HCl was added in order to form the extraction buffer; the pH was adjusted to 8.0 with 4 M NaOH. The extraction buffer was stable for 8h at 4°C.

2.2.4 FAN content

In order to measure the free amino nitrogen content, the "Analytica-EBC" 8.10 method was followed. For the preparation of the reagents, the following procedures were used:

1g KIO_3 (potassium iodate) was diluted in 300 mL of ultrapure water and then 200 mL of 96% v/v ethanol were added to form a 500 mL final solution.

The calibration standard needed used 0.1072 g glycine dissolved in water to 100 mL solution. It was then stored to 0-4°C until use.

For the colour reagent, 10g $Na_2HPO_4 \cdot 12H_2O$ (DHP for FAN), 6g KH_2PO_4, 0.5g ninhydrin and 0.3g fructose were dissolved in ultrapure water to 100 mL final solution. The pH was then measured in order to be between 6.6 and 6.8.

In order to do the measurement, 1 mL of wort samples or 1mL glycine standard were diluted in RO water to 100 mL. 2 mL of the diluted samples were transferred into individual glass tubes to which 1 mL of colour reagent was added. Additional tube was filled with 2 mL of the diluted glycine reagent and 1 mL colour reagent. Finally, another tube was used to put 2 mL RO water and 1 mL colour reagent. The test was being held in triplicate. All tubes were immersed in the water bath for 16 min at 100°C. After that, the tubes were put in a cold water bath for 20 min to decrease their temperature at 20°C. 5 mL of KIO_3 solution were then added to each tube.

The measurement was then done by using polystyrene cuvettes, 1.6 ml, semi-micro in the JENWAY 7315 Spectrophotometer at 570 nm against distilled water.

2.2.5 Friability test

The friability test is performed according to the "Analytica-EBC" 4.15 method. 50 g of sample was required for a test and the sample was put in the friabilimeter made by Pfeuffer for 8 minutes. During the test the dust produced by the malt crushing fell in a small vessel and this material represented the modified part. At the end of the test the malt that remained in the roller was weighed and this material was the unmodified part. The friability was calculated by the following equation:

$$\text{Friability (\%)} = 100 - [\text{Unmodified part (g)}] \times 2$$

2.3 Dielectric measurements techniques and associated methodology

2.3.1 Bulk density measurement

Bulk density is an essential parameter for the accurate measurement of the dielectric properties of malt. For that reason, a known volume of each sample was weighed to find its bulk density and therefore the exact mass that would fit in the cell of the waveguide could be calculated. The method followed was to take each sample, put it in a 1L beaker and measure its volume. Then the exact mass of the sample was measured using a weighing scale. The procedure was done in triplicate for all samples. The formula used for the calculation of the bulk density was the following:

$$\rho = \frac{m}{V}$$

where ρ = bulk density of the sample

m = bulk mass of the sample

V = bulk volume of the sample

2.3.2 Transmission line technique

In the dielectric properties experiments, two types of waveguide cells were used: the WR284 and the WR430 which differed in their size and volumetric capacity and are hollow tubes in rectangular intersection through which electromagnetic waves propagate to the sample as TE_{10} mode (Thostenson and Chou, 1999). The rectangular waveguides support electromagnetic waves only over a certain frequency band, depending on the cross-sectional dimensions. The bigger the size of the waveguide, the lower in frequency it works. Waveguides are specified in WR numbers which symbolizes the "rectangular waveguide" (IEEE, http://www.microwaves101.com/).

At first, the connector plane (Figure 22) was calibrated before taking any measurements using two offset shorts, a fixed and a sliding load designed by MEGA Industries, RF Solutions (MEGA Industries, LLC,28 Sanford DriveGorham, ME 04038) according to the equipment specifications (Hasar, 2009). All the measurements were performed at room temperature (~20°C). Two types of waveguide cells were used: the WR284 waveguide cell with dimensions of 10 x 72 x 34 mm and the WR430 waveguide cell with dimensions of 20 x 109 x 55 mm (Figure 29). Summarized information about the two waveguide cells is provided in Table 4.

The S-parameters (S_{11}, S_{12}, S_{22}, and S_{21}) were measured through the VNA. The measurements were performed at a range of 2.6 to 3.2 GHz and 2.3 to 2.6 GHz

depending on the general specifications of the cell used and the aims and objectives of each experiment. The sample should be packed tightly into the waveguide cell to reduce faults in measurements due to air gaps.

In order to convert the S-parameters to complex dielectric parameters, an iterative method was implemented via "Python" software program and a standard procedure was followed. Therefore, the dielectric constant (ε') and the dielectric loss factor (ε'') were determined.

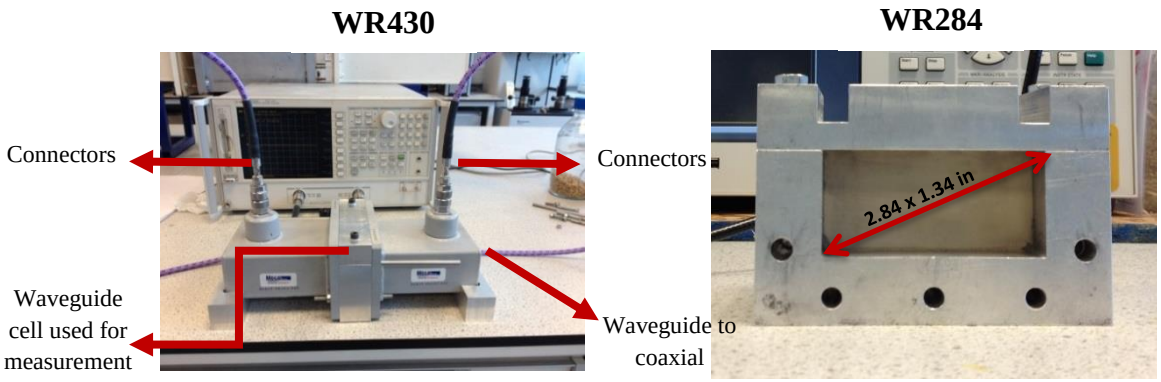

Figure 29. The waveguide cells used to measure the dielectric properties of malt and barley.

In Figure 29, the first image shows the entire measurement kit assembled using the larger waveguide cell and the other image illustrates the dimensions of the waveguide cell and how these relate to the nomenclature WR284.

Table 4. Summarized information about the WR 284 and WR430 waveguide types.

Frequency Band	Waveguide Standard	Frequency Limits (GHz)	Inside Dimensions (inches)	Inside Dimensions (mm)
S band	WR-284	2.60 to 3.95	2.840 x 1.340	72.136 x 34.036
R band	WR-430	1.70 to 2.60	4.300 x 2.150	109.22 x 54.61

2.4 Radio frequency tunnel equipment for pilot scale experiments

The study of the dielectric properties of malted barley grains was the first step to extract useful information about how the properties are influenced by the electromagnetic heating of hydrated malt. The experimental work continued by applying the radio frequency heating energy to hydrated malt and studying the effect of this alternative drying process to the enzyme activity of the malt grains. The equipment used in those experiments is presented in the following figure.

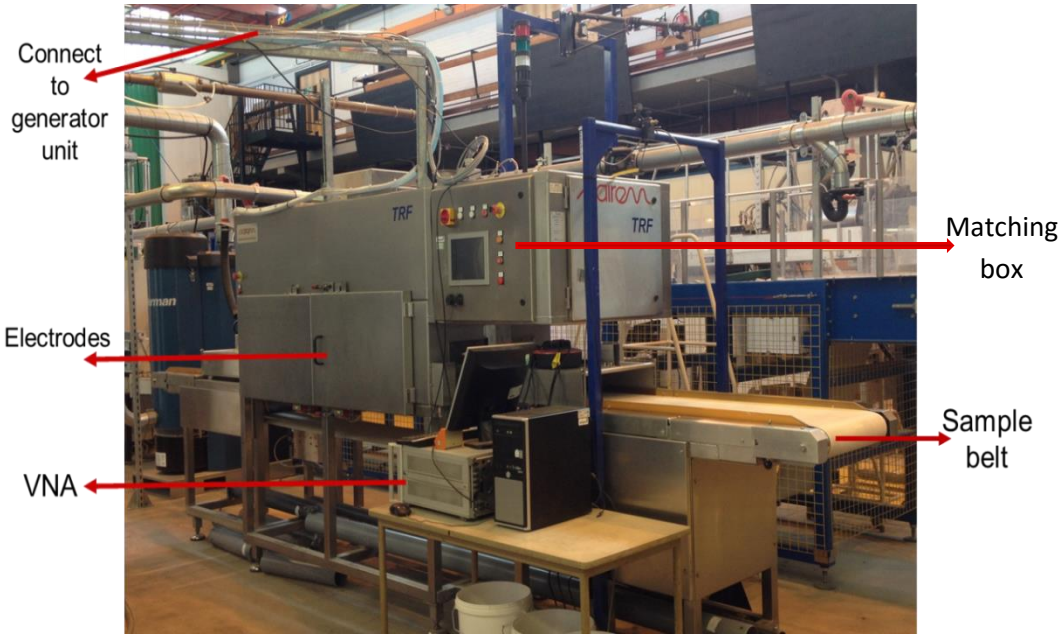

Figure 30. The TRF tunnel used for the electromagnetic heating of malt in the MW lab of the NCIMP group in the Chemical Engineering department.

The TRF tunnel was a SAIREM (12 Porte du Grand Lyon, Neyron, France) model of maximum RF power 20kW and consisted of the generator unit, the control (matching) box, the chiller unit, the main operating unit and a vector network analyser (Figure 30). The dimensions of the tunnel were 5200 x 2570 x 2520 mm with doors and roof panels open. The tunnel was made of stainless steel 304L apart from the parts that allow the transfer of the RF energy which were made of polypropylene. The VNA was a Rohde &Schwarz ZVL portable model with frequency range from 9 kHz to 6 GHz.
The main operating unit included a cavity with a ground (fixed) and a high (mobile) voltage electrode in parallel, a silicone belt conveyor (1200 mm) with variable speed control where the sample is placed during the treatment, an oscillator power source and matching unit circuit set at 27.12 MHz and a thermal imaging camera (H2630 NEC model, 640 x 480 High IR Resolution) to record the change of surface bulk

temperature of each sample before and after the operation as presented in Fig.30. The measuring range of the camera is from -40° to 500°C.

The closed circuit used water supplied from the chiller unit as cooling method. There was also an inlet and outlet choke specifically designed to eliminate RF leakage. The cavity doors included safety interlocks to cut the RF power in case they are opened during operation. The RF energy source was a capacitive applicator which heated the product by converting the electromagnetic energy into heat energy.

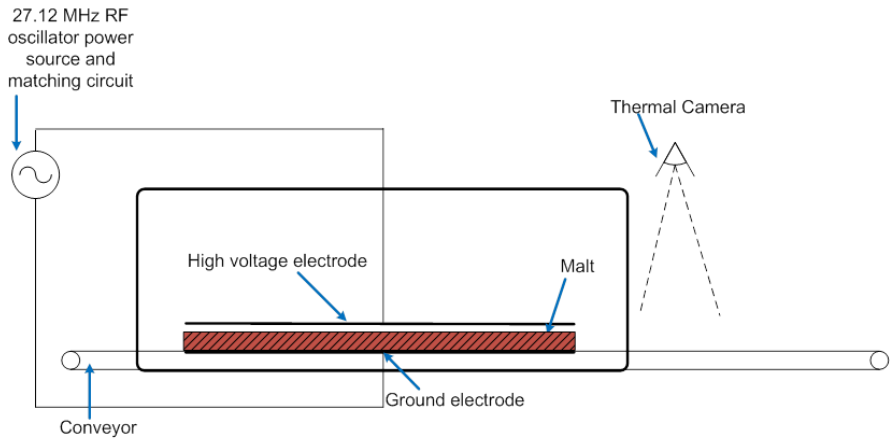

Figure 31. The TRF tunnel operating unit showing the area where the malt sample is placed.

The parameters that could be managed and adjusted were the speed of the belt, the RF power, the RF voltage at the electrodes and the electrode height. The dimensions of the test trays used in the experiments were 290 x 277 x 45 mm. For the optimisation of the experiments an airflow system was added to the main operating unit by applying a constant supply of compressed air to the feed end and extracted with a fume extractor as presented in Fig. 32.

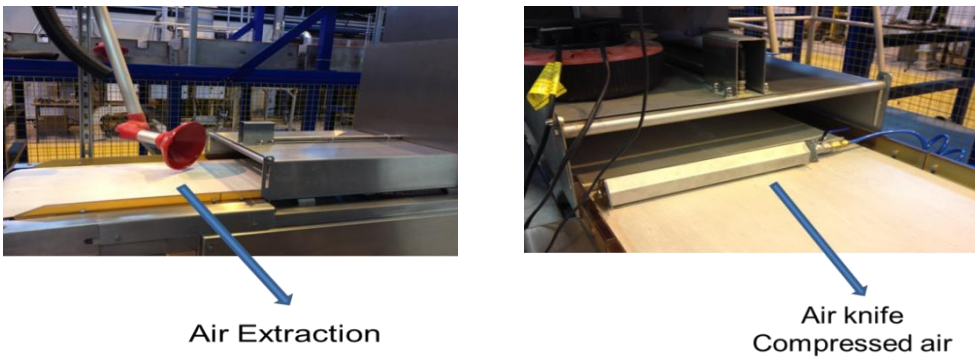

Figure 32. Pictures of the air flow system applied in the RF tunnel to eliminate the condensation of air during processing.

2.5.1 Operational Principles of the TRF tunnel

The RF energy was transmitted to the main unit of the tunnel from the generator by a 50Ω 1" 5/8 coaxial line, a discriminator and a matching box which is a system to adapt impedance. To be more specific, the 50Ω coaxial line allows the transfer of energy at a moderate voltage while the discriminator measures the amplitude and phase of the current/voltage relationship (collects data from coaxial cable). The matching box is used to adjust the impedance to 50Ω while correcting the phase difference between voltage (V) and current (I) in order to achieve the maximum energy transmission. Despite the variations of loading an optimal condition for maximum energy transmission can be possible by maintaining the circuit in optimal condition through a double control loop which is formed by the matching boxes, the control box and the discriminator. The matching box which is integrated within the tunnel consists of two capacitors and two coils in parallel on the applicator. The discriminator is composed by two sensors which collect data from the coaxial cable and is normally placed at a fixed position along the line between the RF generator and the matching box. The LOAD sensor derives from the ratio V_{RF}/I_{RF} and the TUNE sensor is related to the measurement of phase between V_{RF} and I_{RF}.

The impedance of the applicator alters with every change of the load such as the type and the quantity of the product, the humidity and the speed of the conveyor belt. For any variation of the load, a correction of the impedance is required by modifying the variables of the circuit.

Thus, when the output signal of the discriminator is 0 Volt, the impedance is 50Ω and the motor that moves the capacitance LOAD is stopped. If the impedance is not equal to 50Ω the LOAD motor moves and corrects the mismatch. The corrections can be done manually for small changes whilst for industrial processing an automatic system is necessary.

This TRF tunnel model includes also a manual electrode height adjustment system at the loading end with two wheels with exterior movement counter record which permits to optimize the RF energy transmission. The size of the product and its dielectric properties influence the ability of the matching box to eliminate the reflected power during processing. The two wheels should be adjusted together and set at the same position to ensure homogeneity of the RF energy transmission across the treatment area from left to right during processing. However, the higher is the electrode position the greater the risk of arcing and the lower is the sensitivity of the RF field to the product under treatment. For a low dielectric loss item the air gaps between the electrodes need to be eliminated and for that reason there is a metal plate

underneath the belt conveyor that extends the height of the lower electrode and thus reduces the distance from the upper electrode.

Chapter 3 Development of methods for the study of the dielectric properties of hydrated malt at microwave frequencies

3.1 Aims and Objectives

There is a lot of interest about the dielectric properties of cereal grains in food and agriculture science due to their massive use in the worldwide food and beverage industry. Barley malt especially has become very popular because it is an integral component of beer. The accurate knowledge of the dielectric properties of malt and barley grains is important for the rapid, non-destructive estimation of the moisture content (Raghavan, 2005) and could also provide useful information in the study of electromagnetic drying of the grains, the monitoring of moisture content during the milling process and the selective heating of stored grains to control the presence of insects (Nelson, 1981).

Despite the fact that there are many reports indicating the dielectric properties of wheat (Nelson, 1994, Nelson, 1981, Baker-Jarvis et al., 2010) and other cereals at various moisture contents from low to high frequencies, there is a lack of information about the dielectric properties of malted barley in a range of moistures that cover the different hydrating stages during the industrial kilning process. The knowledge of permittivity could be used for the improvement of kilning process by applying electromagnetic heating (AEA, 2011).

The aim of the first experiment described below (Section 3.2) was the investigation of the dielectric properties of malted barley at several moisture contents covering the range typically encountered during the kilning process by using the transmission line technique. Because the malting and the microwave laboratories were at different campuses of the University, it was necessary to store samples for a time period in between removal from the kiln and the analysis of dielectric properties. It was therefore necessary to understand if there was any impact of how the samples were stored on the dielectric measurements due to the different temperature storage conditions. Thus a better understanding of how the material behaves in different moisture contents and temperature conditions could be achieved.

The next step (Section 3.3) included a repeat of the malt dielectric properties test but covering a greater range of moisture contents across the kilning process in order to understand better the behaviour of the material and the study of its dielectric

properties using a different type of waveguide cell. The samples were stored in a refrigerator until the dielectric properties measurement as it proved to be the best option for the time needed between the sampling and the analysis. To achieve getting more samples during the kilning stage a different methodology was developed and applied.

3.2 Study of malt dielectric properties at varying moisture contents; impact of storage temperature between removal from kiln and dielectric measurement

3.2.1 Preparation of malt samples

For the preparation of the malted barley samples, raw barley (variety *Tipple*; 2 kg) was micromalted for 7 days using the CustomLab micromalting equipment following the schedule reported in Section 2.2.3. The programme set was as presented in Table 2.

Only the one channel from the steep/germinator unit (Figure 25a) was used and therefore four cages were filled with the raw material (500 g barley in each vessel) in order to give around 3 kg of green malt in total ready for kilning (considering the fact that each 500g barley sample give approx. 750 g of green malt). A sample of 120 g green malt was taken as a reference at t=0 h. Samples of 120 g each were then taken from the kiln unit after 1h, 2h, 3h, 4h, 6h, 8h and 23h periods after mixing the bulk material manually in the sampling vessel. So the dielectric properties of samples with different moisture contents and at different stages of the drying process were measured.

Because the experiments took place at two different campuses of the University, it was necessary to store samples for a time period in between removal from the kiln and the analysis of dielectric properties. Thus it was important to understand if there was any effect of how the samples were stored on the dielectric measurements.

Each of the eight malt samples according to the different time points of the kilning stage were thus split into three batches, which were stored under different temperature conditions between sampling and analysis (either at room temperature, refrigerated at 5°C or frozen at -20°C).

3.2.2 Moisture content measurements

The moisture content of malt was measured at each time point of sample collection during the kilning process according to the method described in Section 2.3.1. The sample weight used for each measurement was 2 g and the test was performed in triplicate. The results were as presented in Table 5.

Table 5. Moisture contents of malt samples taken at the specified time intervals during the kilning process.

Time (h)	Moisture Content (%)			
	1st test	2nd test	3rd test	Average
0	38.3	38.6	38.0	38.30
1	35.9	35.58	35.6	35.69
2	19.0	19.81	20.22	19.68
3	17.61	17.5	17.32	17.48
4	12.23	11.91	11.77	11.97
6	7.78	7.64	7.86	7.76
8	6.69	6.78	7.18	6.88
23	3.79	4	4.32	4.04

A graphical representation of the time course of moisture content through kilning is presented below (Figure 33); a decrease in the moisture during the drying of malt through the kilning process was observed as expected to a final level of 4%. Zone 1 represents the initial stage of drying where water removes freely from the surface -known as the free drying stage-. Zone 2 is the intermediate (falling rate) phase and zone 3 is the curing phase where the grains are almost dried but colour and flavor are being developed as the final 2-3% moisture content is being removed at high temperature. From the error bars it is apparent that there was acceptable variation in moisture measurement at each time point.

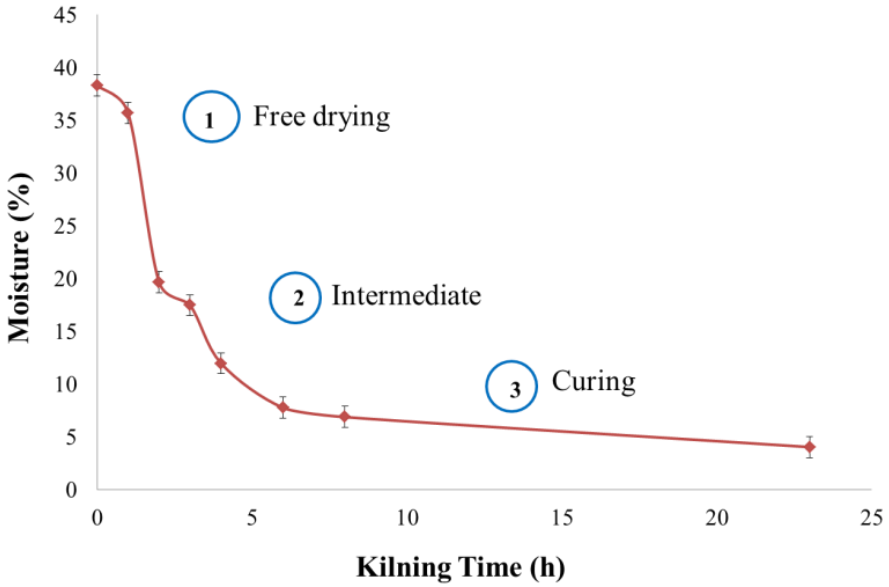

Figure 33. Moisture content over time during the micromalting kilning process.

3.2.3 Dielectric properties measurement: experimental set-up

For the precise measurement of the dielectric properties of materials the criteria that should be taken into consideration are: the physical properties of the material, the frequency range of interest, the required accuracy of the measurement and the sample size restrictions (Baker-Jarvis et al., 2010). For the investigation of the dielectric properties of whole malt grains with microwave energy established at a range of moisture contents occurred during kilning stage, the transmission line waveguide technique was used. This method was selected as more suitable due to its potential to measure the permittivity of low to high loss granular materials with good accuracy (Lee and Tran, 2006). The main advantages of the rectangular waveguide cells that were used in the experiments are the simplicity of sample preparation and the need for relatively small sample quantities (Larsson et al., 2011).

The measurements were performed in three replicates by using the waveguide cell WR284 in a range of frequencies from 2.3 to 2.6 GHz and the methodology was as described in Section 2.4.2. All the measurements were performed at room temperature (~20°C). The S-parameters (S11 and S21), representing the reflected and transmitted signal respectively were measured through the VNA. In order to convert the S-parameters to complex dielectric parameters, an iterative method was implemented via

"Python" software programme and a standard procedure was followed. Therefore, the dielectric constant (ε') and the dielectric loss factor (ε'') were determined.

Because dielectric properties are also dependent on bulk density, the variation of bulk density with moisture content was tested before performing the measurements in the waveguide. The method followed for each sample was to put the material in a 1 L volumetric beaker in a weighing scale and then the exact mass and volume of the sample were measured in order to calculate its bulk density (Mohsenin, 1980). Therefore, the required mass that would fit in the waveguide cell could be determined. The procedure was repeated five times for all samples and the results are presented in Table 6.

Table 6. Bulk densities of malt sampled at variable moisture content during drying and stored at different temperatures between sampling and analysis. The "sample weight" column indicates the weight of each sample that should be used for the dielectrics measurement.

		Room Temperature (20°C)		Refrigerated (5°C)		Frozen (-20°C)	
Time (h)	Moisture content (%)	Bulk density (g/m^3)	Sample Weight (g)	Bulk density (g/m^3)	Sample Weight (g)	Bulk density (g/m^3)	Sample Weight (g)
0	38.3	0.48	11.8	0.46	11.2	0.47	11.4
1	35.7	0.40	9.8	0.40	9.8	0.41	10.0
2	19.7	0.35	8.5	0.34	8.4	0.36	8.8
3	17.5	0.34	8.4	0.33	8.1	0.33	8.2
4	11.9	0.30	7.4	0.31	7.5	0.29	7.0
6	7.8	0.30	7.4	0.28	6.8	0.28	6.9
8	6.8	0.35	8.5	0.28	6.8	0.27	6.5
23	4.0	0.34	8.3	0.29	7.2	0.26	6.4

Because all the samples were collected during the kilning process in the same way and then stored in different conditions, the variation in results for bulk density is more likely explained due to sampling error.

For each different moisture content, the dielectric constant (ε') and the dielectric loss factor (ε'') average and standard deviation of all samples stored in different temperature condition were calculated over a range of microwave frequencies from 2.6 to 3.2 GHz. The test was repeated five times for all the different moisture contents

through the kilning process. The results and their outcomes from the measurements of the dielectric properties of malt were presented below for a selected frequency (2.6 GHz). In the literature it was observed that the increase of microwave frequency leaded to a decrease in the dielectric constant of cereal grains (Nelson, 1981). Thus the presentation of the dielectric properties results for the lowest frequency applied could be useful as an indicator of the trend for even lower microwave frequencies applied.

3.2.4 Malt dielectric properties: results

The results of the dielectric properties at 2.6 GHz are presented in the following figures for samples stored in different temperatures. Every data point in the graphs below is the average of the five replicates that performed in the measurements. The variability was too tight and consequently the error bars are hidden by the data points.

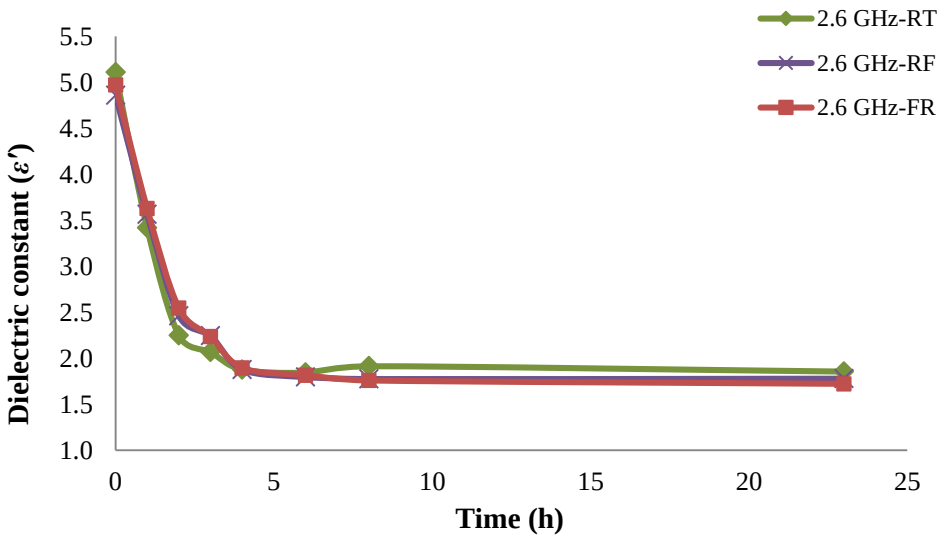

Figure 34. Dielectric constant (ε′) of malt over kilning time at 2.6 GHz stored in different temperatures: RT – Room Temperature, RF – Refrigerated, FR – Frozen

According to the data in Figure 34, the dielectric constant (ε′) of malted barley displayed a downward trend over the early hours of kilning and then remained low and stable over the middle and later hours. As was expected, the decrease of the moisture content during the free drying phase of kilning process caused a reduction in the dielectric constant of malt, as water highly affects the dielectric properties of the material. As the moisture decreased below 10% the dielectric constant became almost constant as the physical properties of malt changed and more energy was needed to remove the remaining moisture from the grain.

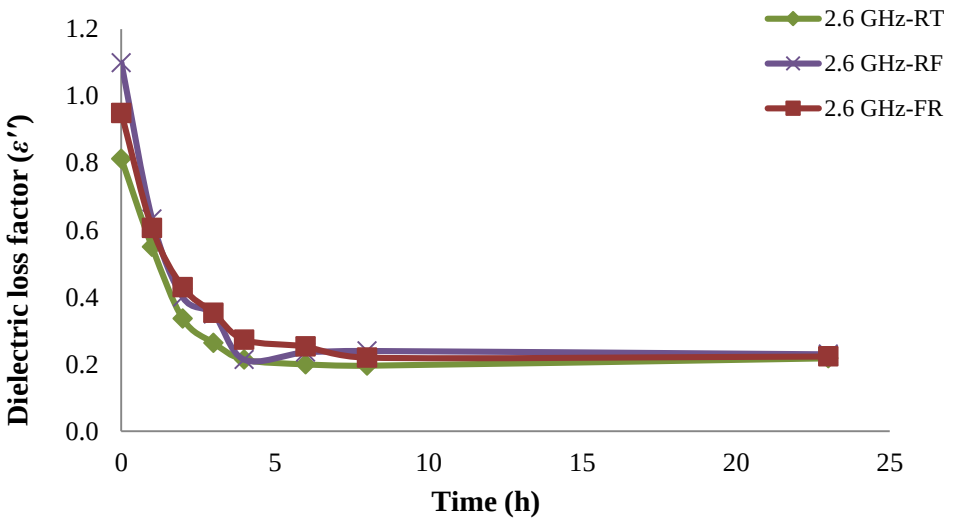

Figure 35. Dielectric loss factor (ε') of malt samples over kilning time measured at 2.6 GHz and stored in different temperatures.

The same trends were observed for the dielectric loss factor (ε'') as it was diminished rapidly during the first four hours of kilning process (free drying) and remained stable at low rates over the intermediate and curing phase. Thus, as the moisture was reduced and the malt was getting drier it was more difficult for malt to absorb the microwave energy and transform it into heat.

Due to the key role of water in determining dielectric properties it is informative to plot the same dielectric data against the sample moisture content (Figures 36 & 37).

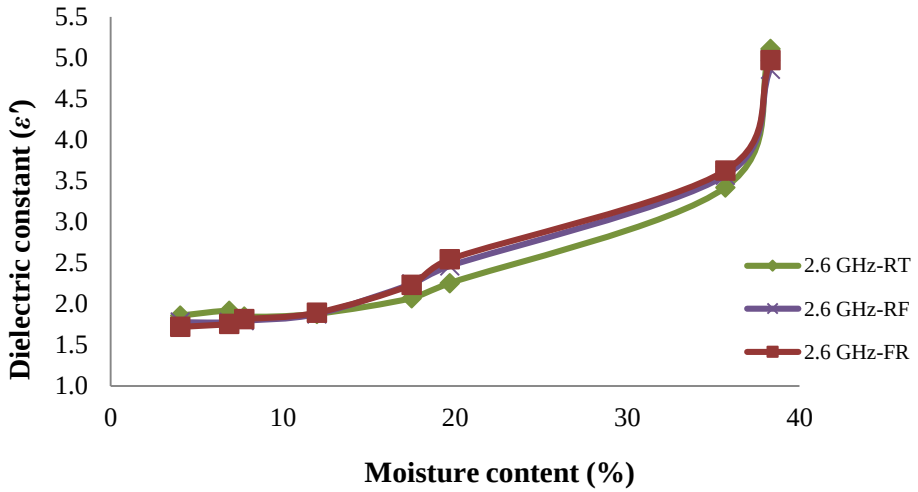

Figure 36. The effect of moisture content (ε') on the dielectric constant of malt during kilning process in different temperature storage conditions.

This graph showed a decrease on the dielectric constant (ε') during the kilning process as moisture content reduced. This trend could be explained by the fact that the moisture content strongly affects the dielectric behaviour of a food product such as malt under different temperature conditions as being observed in other studies (Ryynanen, 1995).

Despite the fact that all samples were tested at room temperature those that had been stored in cool and frozen conditions had higher values of ε' and ε'' at moisture contents above 15% (w/w). This trend could be explained by the fact that some parts of the inside of the grains were still cold when tested because those samples remained at room temperature for about 2 hours before taking the measurements. Consequently, water was present in different forms and ionic conductivity might have occurred due to the different phase of water (Venkatesh and Raghavan, 2004). Similar observations were found for the dielectric loss factor as shown in the following graph.

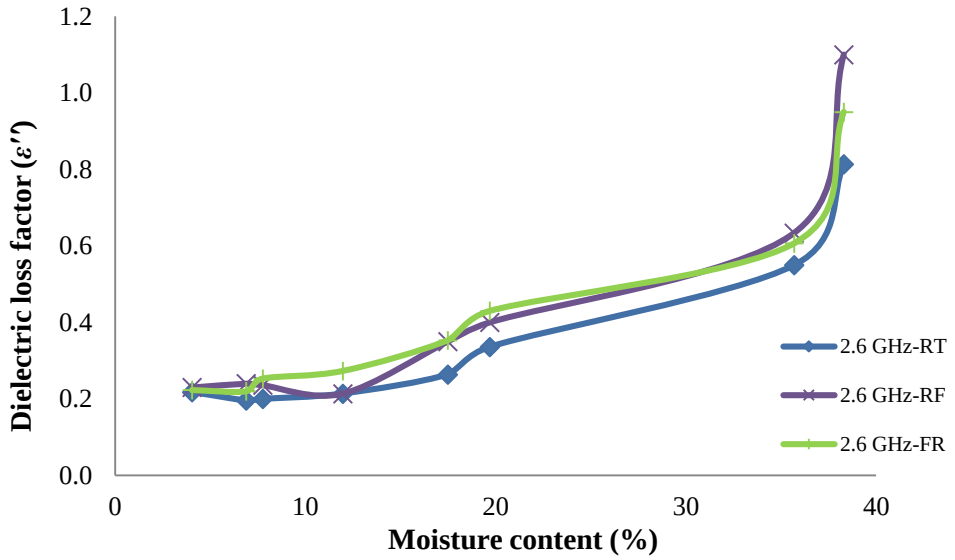

Figure 37. The effect of moisture content on the dielectric loss factor (ε'') of malt during kilning process in different temperature conditions.

It is remarkable to note that the sharpest drop of the dielectric loss factor (ε'') occurred at the highest moisture contents. According to this, the ability of malt to store energy is getting lower while moisture content is diminished and this trend is the same for all different storage conditions in the range of microwave frequencies investigated.

Studying this graph, it is apparent that microwave technology would be preferably applied at low malt moistures where the loss factor follows a more linear and sluggish decrease. In this time period, known as the curing phase (Figure 33), water is no

longer freely available at the surface and moisture should be removed from the interior of the grain by selective heating rather than airflow drying. In contrast, when moisture is higher about 20%, water is readily available at the surface, so the airflow is more appropriate and less costly in order to remove the moisture.

3.2.4 Main Conclusions

Regarding the results from this preliminary experiment, hydrated malt samples stored at room temperature, cool (5°C) and frozen (-20°C) conditions were investigated for changes in their dielectric properties at a range of different moisture contents. The transmission line technique was used for the study of malted barley grains as it is more suitable method for the dielectric properties measurement of medium to high loss materials (Yaw, 2012).

Generally speaking, all sample storage conditions showed the same trend in function to moisture content and time and there was a reasonable consistency between them. The dielectric properties as expected were very much influenced by the water content. The most remarkable changes occurred in the early hours of the kilning process, when the moisture content dropped quite rapidly.

According to the above conclusions, a more extensive study of the dielectric properties of malt is necessary in order to have a clear view of how the material behaves in different moisture contents at a standard bulk density. For that reason, more samples should be taken over kilning time points, especially during the early hours in order to identify where microwave energy would be more effective and thus more efficient to be applied. Malt should be stored in a refrigerator to keep a constant mild cool temperature to eliminate the possibility of continuing the germination until the analysis of the samples. All samples should be tested in a bigger waveguide cell to enable study of the effect of lower microwave frequencies on hydrated malt.

Thus, we expect to get more accurate results and be able to measure a broader range of moisture content malt samples. Moreover, it is important that full malt analysis is conducted to test the impact of microwaves on key malt specifications, for example the differences that might occur in the enzyme activities of malted grains because of the microwave treatment. Thus, the key point is to ensure that the enzymes will be preserved and will not be destroyed.

Furthermore, measurements with other techniques such as cavity perturbation parallel plate and study of various frequencies such as radio frequencies are going to take place. Radio frequencies which range from 300 kHz to 300 MHz, have been applied in

27.12 MHz in a few studies (Eichhorn, 2010, Akaranuchat, 2012) combined with the conventional heating in the drying of barley and rice grains and showed good response as far as it is concerned the time and energy consumption as well as the enzyme preservation of the grains.
Thus, by studying the radio frequencies that will probably have a milder affect in the malted grains we will have a full investigation of the potential use of those alternative heating energy sources in the drying process of malt.

3.3 Measurement of malt dielectric properties using the larger WR430 waveguide cell

3.3.1 Preparation of the malt samples

Malting of barley used the CustomLab Micromaltings equipment as previously described in detail in Chapter 2 (§ 2.2.2).
In this experiment 8 cages were filled with raw material (500 g barley for each cage) in order to give around 6 kg of green malt in total ready for drying in the kiln unit (considering the fact that each 500 g raw barley sample give approx. 750 g of green malt). However, the process of steeping/germination was run in two cycles in order that one channel would finish later and start therefore in different time the kilning process. This alteration in the schedule enabled more samples to be taken during the late hours of kilning stage that would normally take place during the night hours of a programmed experiment.
After the germination process, malt was put in the kiln unit to produce the final malt product following the Standard Ale Malting Programme (details in Chapter 2, § 2.2.3). During the kilning process, air-on temperature, air-off temperature, humidity and airflow were continuously monitored and logged. Two different kilns were operated, programmed with the same time-temperature profiles but with suitably off-set start times in order to cover the 23 hours needed to prepare the final dried product. Samples were taken from the 1st kiln during the initial 8 hours of the cycle, whilst the 2nd kiln was run overnight and lasted 23 hours, providing the later time point samples. Thus samples were taken off at the following times points: t= 0h, 0.5 h, 1h, 2h, 4h, 6h, 8h (1st cycle), 15h, 16h, 17h, 19h, 20h, 21h and 23h (2nd cycle) in order to cover a wider range of malt moisture contents than in Section 3.2.

3.3.2 Moisture content of malt samples

The moisture content of malt samples taken from the kiln was measured in triplicate using the HR83 Halogen Analyser using 2 g of pulverized sample at 105°C. Samples were then vacuum sealed in aluminum bags and stored in refrigerated conditions (4°C) until the performance of the dielectric properties measurement in order to avoid the extreme freeze and thaw of the grains and also prevent any biological changes on their cellular structure because of the presence of the water. The moisture content of the green malt was also tested before running the 2nd kiln cycle.

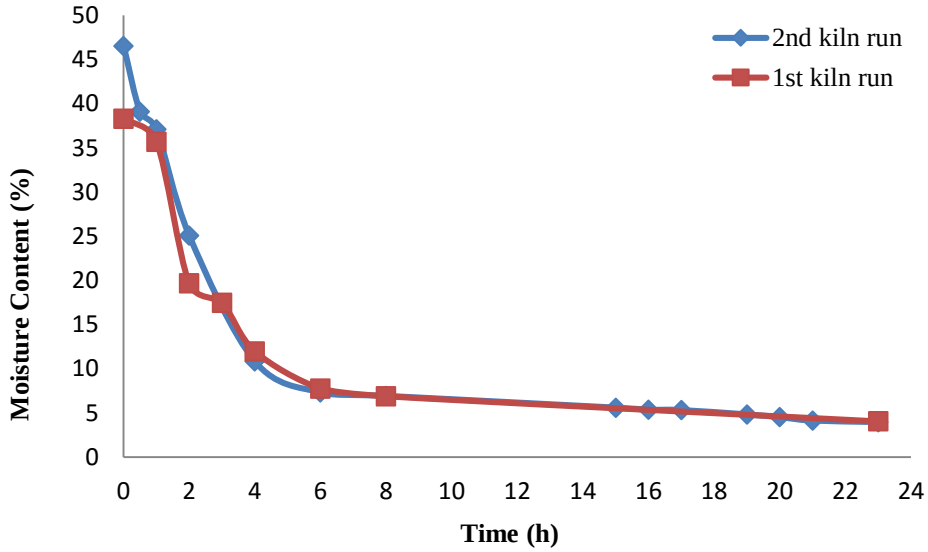

Figure 38. Moisture content of malt versus time during both kiln runs.

The moisture content appeared to have almost the same trend by testing more samples over the 23 hours of kilning process. This trend was expected as the samples were treated in the same way and their moisture was measured using the same method. However, a slight increase appeared in the initial moisture of the green malt sample which could possibly be explained due to sampling error as the initial weight of the green malt was homogenised manually.

3.3.3 Measurements of malt dielectric properties

As previously, the measurements were performed by using the waveguide cell and the principal methodology was as described in detail in Section 2.4.2. In this experiment, the WR430 waveguide calibration kit was used at a frequency range of 2.3-2.6 GHz and waveguide cell dimensions were 20 x 109 x 55 mm according to the equipment

specifications. The sample was fitted tightly into the waveguide cell to reduce faults in measurements causing by air gaps and the dielectric constant (ε') and the dielectric loss factor (ε") were determined. The bulk densities of the samples were tested before starting the measurements in the waveguide using the same method as reported in the previous experiment (Section 3.2). The results are presented in Table 7.

Table 7. The relationship between malt moisture content and bulk density and the sample masses used in WR430 waveguide cell respectively.

Time (h)	Moisture Content (%)	Bulk Density (g/mL)	Test sample Mass (g)
0	46.53 ± 0.67	0.53 ± 0.03	63.4 ± 3.70
0.5	39.09 ± 0.42	0.50 ± 0.03	59.6 ± 3.16
1	37.13 ± 0.6	0.47 ± 0.00	56.0 ± 0.02
2	25.07 ± 0.08	0.34 ± 0.01	41.3 ± 1.15
4	10.83 ± 0.17	0.30 ± 0.00	35.5 ± 0.52
6	7.32 ± 0.34	0.32 ± 0.02	38.7 ± 2.92
8	6.92 ± 0.24	0.33 ± 0.00	39.2 ± 0.02
15	5.59 ± 0.36	0.34 ± 0.02	41.2 ± 2.10
16	5.36 ± 0.33	0.37 ± 0.00	44.4 ± 0.00
17	5.32 ± 0.28	0.36 ± 0.00	42.8 ± 0.05
19	4.82 ± 0.33	0.36 ± 0.00	43.4 ± 0.02
20	4.51 ± 0.36	0.35 ± 0.00	41.8 ± 0.00
21	4.13 ± 0.47	0.32 ± 0.02	38.8 ± 2.05
23	3.96 ± 0.36	0.32 ± 0.00	38.7 ± 0.03

The dielectric constant (ε') and the dielectric loss factor (ε'') average and standard deviation of hydrated malt from various moisture contents ranging from approx. 4% to 45% were measured in triplicate and the results were presented in the following graphs:

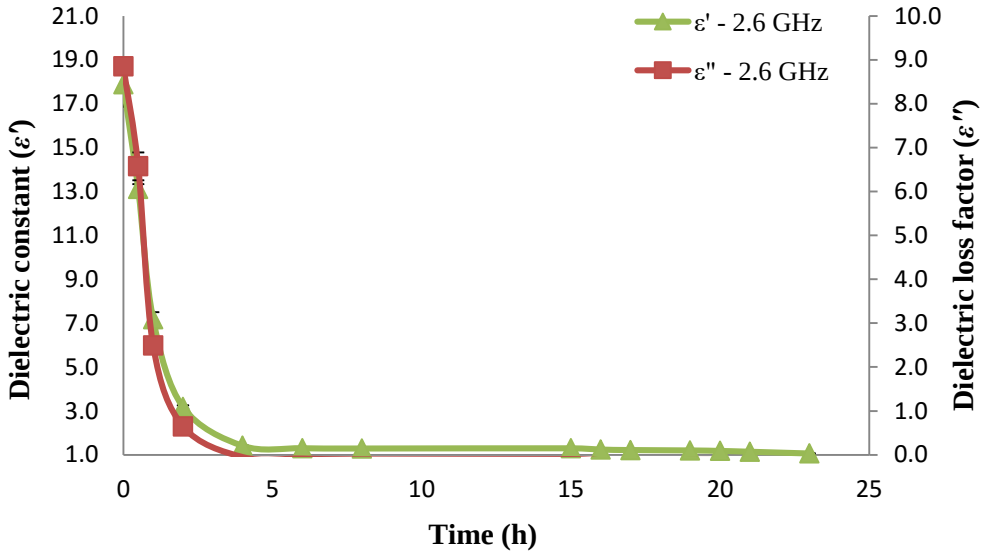

Figure 39. Dielectric properties of malt samples versus kilning time, measured at 2.6 GHz with the WR430 waveguide cell.

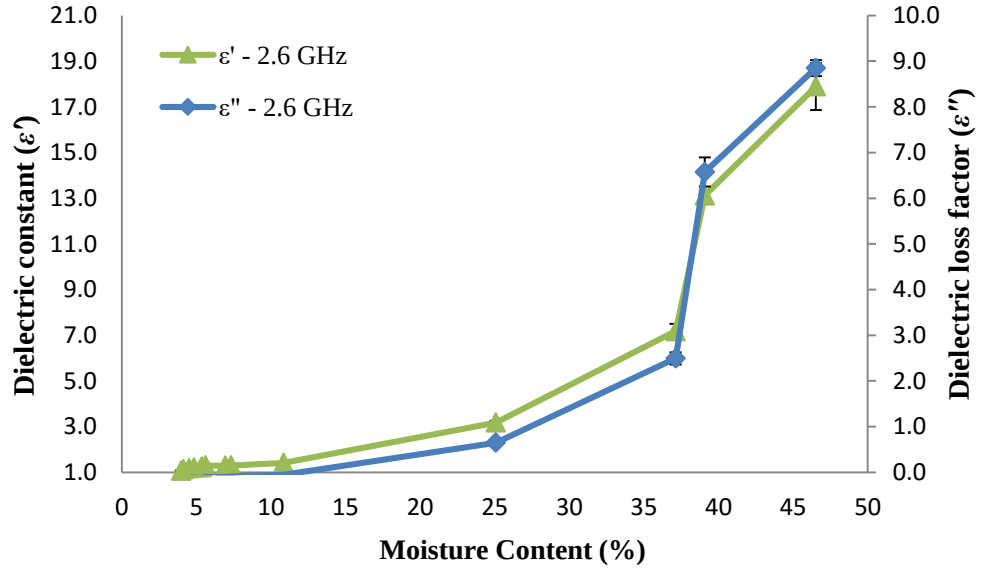

Figure 40. The effect of moisture content on the dielectric properties of malt measured at 2.6 GHz with the WR430 waveguide cell.

The dielectric constant (ε') and the dielectric loss factor (ε'') showed similar trends against moisture content as measured in the waveguide cell WR430 at 2.6 GHz (Figure 40). According to the waveguide measurements, the dielectric properties of malt decreased vigorously when the moisture content reduced from 45% to 25%. It

was also observed that the samples with moisture content from 10% and below, had an almost constant value for the ε' of approx. 1.2 which indicated that the material was quite low loss and consequently it was more difficult for the grains to absorb the microwave energy. At this range of moisture content, water mostly existed in the endosperm of the grain and thus it is harder to transport to the surface and evaporate during the conventional kilning process (Briggs, 1998).

The ε'' values for the hydrated malt under 10% were below zero which meant that there was false measurement. However, the negative values proved that the grains were very low loss and the sensitivity of this waveguide cell was unable to give a reliable result. It is generally accepted that in order to achieve a dielectric heating, loss factor of the material should have a value of $\varepsilon'' > 0.01$ (Piyasena et al., 2003).

Comparing the dielectric properties of malt measured using the two different types of waveguide it was observed that the WR284 waveguide showed more sensitivity and accuracy for the low loss samples (<10% hydrated) whilst the WR430 was more reliable for testing the high and medium loss malt samples as presented in Figure 41.

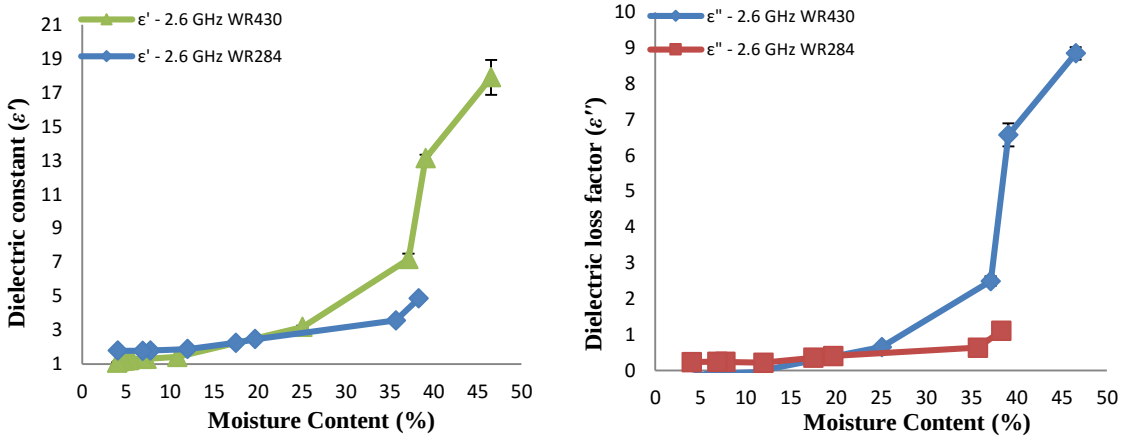

Figure 41. Comparison of the effect of moisture content on the dielectric constant (a) and dielectric loss factor (b) of malt using two different types of waveguide cell as measured at 2.6 GHz.

A comparison of the application of different microwave frequencies to the dielectric properties of malt is also presented in Figure 42 which shows that the decrease of microwave frequency at a range of 0.3 GHz had a slight impact in the dielectric properties. In the most hydrated samples it appeared to a slight decrease in the properties with the application of lower frequencies. However, the ε'' values for the low loss samples were below zero and thus the results were not reliable by using the WR430 waveguide cell.

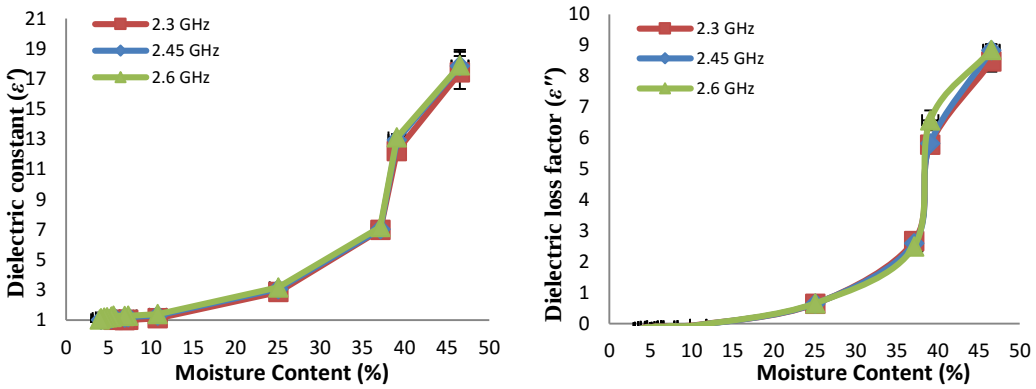

Figure 42. Comparison of the effect of moisture content on the dielectric constant (a) and dielectric loss factor (b) of malt when applying microwave frequencies from 2.3 to 2.6 GHz.

3.4 Comparison of Moisture Content measurement techniques during the kilning of malt

3.4.1 Aims and Objectives

In order to move on to pilot scale experiments the use of large quantities of malt hydrated in different moisture content levels derived from the conventional kilning process was required. For that reason, it is important to ensure the accuracy of the moisture content measurement malt samples using the HR83 halogen analyser as only 2 grams from a bulk sample are used to take a rapid measurement. Thus the comparison of the analyser method to the conventional drying method which uses larger samples and takes longer to give a result would be useful to make sure that the results from the analyser method are accurate and representative of the large bulk material. Furthermore, this study will help to understand how rapid the moisture is decreased in a batch of large amount of green malt. The outcomes will enable the rapid preparation of large quantity of malt samples hydrated at various moisture contents in order to be used for the experimental process of electromagnetic drying in pilot scale.

3.4.2 Preparation of the samples

For the initial preparation of green malt, 6 kg of raw spring barley (variety *Tipple*) was used in the extended capacity micromalting equipment. The process was as follows:

- Use of a steep/germination tank in which a rectangular slotted plate was placed as the bottom support such that the whole chamber could be filled with barley.
- The set programme for the performance of the steeping/germination and the production of green malt was the one used for the preparation of malt for the dielectric properties measurements (Section 2.1). This process took approximately 6 days (5 days and 19 h).
- The total mass of green malt at the end of the germination was estimated to be 9.5 kg.
- On day 7 the green malt was transferred to the kiln unit to continue with the kilning process. The 9.5 kg of green malt was split between two large kiln vessels (4.7 kg each) and the moisture content was tested every 2 hours by using the HR83 halogen analyser and the conventional oven drying method (EBC methods 3.2 and 4.2). The kilning programme cycle was the following:

Table 8. The kilning cycle applied for the drying of malt.

Kiln1: 4h, 55^{o}C
Kiln2: 6h, 60^{o}C
Kiln3: 6h, 65^{o}C
Kiln4: 2h, 70^{o}C
Kiln5: 2h, 85^{o}C
Kiln6: 2h, 95^{o}C
Cooling: 1h, atm.
23 hours

3.4.3 Moisture content test methodology

The comparison of the moisture content results from various time points over kilning process was performed using both the HR83 halogen analyser and the conventional

oven method. The oven used in the experiment was a Genlab OV/50/DIG oven 50 L capacity (Tanhouse Lane, Riverview Industrial Estate, Widnes, Chesire, UK).

For the comparison of the moisture content techniques, 40 g of mixed malt sample collected from each time point during kilning process were used which were split as following:

- 10 g for “barley std.” method in the oven at 130°C for 2 h
- 10 g for “malt std.” method in the oven at 105°C for 3 h
- 10 g for “barley std.” method in the HR83 analyser at 130°C
- 10 g for “malt std.” method in the HR83 analyser at 105°C

The methods applied named as barley standard and malt standard differed in the temperature applied to the samples. The reason why two different temperatures were selected for this test was because the high moisture content samples usually dry at 130°C, the same temperature that green malt is dried to measure its miosture. The low moisture content samples dry at lower temperature usually 105°C as it is suggested in the EBC Methods 3.2 and 4.2.

In all the above trials the mass per test was 5 g and each test was performed in duplicate. Before each measurement the bulk material was mixed manually to achieve homogeneity and a random sample was taken to be tested. To achieve homogeneity a riffle box was used before splitting the malt into the smaller samples and for more accuracy in the results the same type of aluminum dishes as specified in the EBC standard method were used.

3.4.4 Results

Comparing the barley methods for the oven drying standard and the HR83 halogen balance, there was a slight difference in results at the high moistures which could have been due to the different way of drying the sample. More specifically the samples dried in the oven had a long term treatment (2h for barley and 3h for malt samples) and hot circulated air was applied whilst drying in the HR83 halogen analyser used the integral halogen heating module and the samples had a short term drying process (30-40 min). Thus the standard oven drying technique appeared to be more accurate for the high moisture content samples. The “barley std.” and “malt std.” methods came in accordance in both techniques as presented in Figure 43.

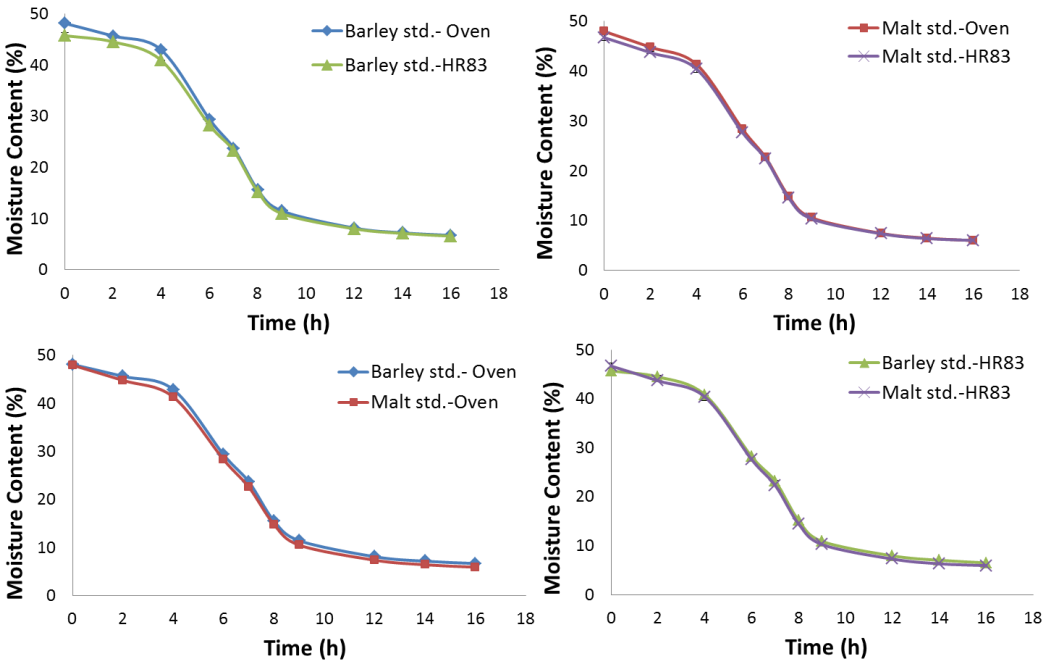

Figure 43. The comparison of barley and malt methods for both HR83 halogen analyser and oven techniques.

3.5 Methods to reproduce hydrated malt from finished dried malt at standard moisture contents

3.5.1 Aims and Objectives

In order to design and perform radio frequency drying experiments with hydrated malt in the TRF tunnel as described in Section 2.4 it was important to reproduce large quantities of hydrated malt in various moisture contents which would be used for sacrificial material in each test. By that we mean material that will not be used for further testing (i.e. amylase analysis) but will cover the rest part of the test zone between the electrodes of the tunnel where the experiments will take place. Those large quantities of sacrificial malt could not be produced using the micromalting equipment as fast as they could be produced by manually add water to already dried product.

Thus, during this study malt samples steeped up manually to different moisture contents and then tested for their reliability in the bulk moisture content by using

different mixing methodologies. The outcomes helped to select the most suitable method to reproduce sacrificial hydrated malt which could be used for the experimental needs of the electromagnetic drying of malt in large scale as mentioned previously.

3.5.2 Experimental procedure

To prepare the samples, three 25 L capacity polypropylene buckets with sealable lids were used. Each bucket filled with 3.6 kg of malt and sprayed with the required amount of water to achieve the different moisture content for each case by using a spray bottle. Three different trials were carried out which aimed to increase the moisture content of finished malt by steeping up to 12%, 16% and 20% moisture content respectively. The moisture was tested in duplicate for each different mixing methodology by manually taking two random samples across the bulk for each bucket.

Mixing methodologies:

1) Turn the closed buckets upside down 3 to 4 times
2) Rotate the closed buckets 3 to 4 times
3) Open the buckets and mix the material very well manually

The required amount of water for each test was calculated as followed:

- To produce 12% hydrated malt approx. 245 mL H_2O were required
- To produce 16% hydrated malt approx. 430 mL H_2O were required
- To produce 20% moisture content 630 mL H_2O were required

In order to calculate the volume of water required to reach the appropriate moisture content level an example is presenting below:

The initial dried malt weight used was 3.6 kg with 6% moisture content. The initial dry matter was 3.384 kg and the water mass at 6% moisture content was 0.216 kg. To achieve 12% moisture content malt 3.384 kg x $\frac{100}{88}$ = 3.82 kg was needed. The water mass at 12% moisture content should be 3.82 kg x 0.12 = 0.458 kg. Thus the water mass required to spray with the dried malt to reach the 12% moisture content was 0.458 – 0.216 = 0.242 kg ≈ 242 mL.

The grains then mixed manually and left in enclosed environment in room temperature condition for 48 hours before testing their moisture. After 48 hours, two random samples from each bucket were taken for each different mixing methodology (18 samples) and their moisture content was measured in duplicate using the “Malt std.” method in the Halogen analyser HR83.

3.5.3 Results of the water uptake of dried malt for the different mixing methodologies

The results for the moisture contents are presented below after 48 hours in enclosed buckets at room temperature and compared to the calculated moisture content which should have been attained in each case:

Table 9. Moisture contents of steeped malt samples compared to their theoretical moisture content using each of the three different mixing methodologies.

	Method 1			**Method 2**			**Method 3**		
Theoretical moisture content (%)	**20%**	**16%**	**12%**	**20%**	**16%**	**12%**	**20%**	**16%**	**12%**
Average (%)	18.39	15.24	11.07	18.43	14.96	10.47	18.24	14.22	11.57

***Methods 1, 2 and 3 are specified in the Section 3.5.2**

The results showed that after 48 hours the samples came in equilibration with the water inside the enclosed buckets and the moisture content was quite close to the expected one. However, small differences were presented by using different mixing methods which could be attributed to sampling errors as random samples were taken manually for testing through the buckets. According to table 8,
the upside down mixing of the buckets was more effective for the 16% moisture content sample, the rotation of the buckets had a better result for the 20% moisture content sample and the manually mixed bucket gave good agreement results for the 12% moisture content.
To summarize with the different mixing techniques did not prove to have a significant effect to the uptake of moisture content of the dried malt. However, the 48 hours appeared to be too small a time period to achieve full mass transfer of the water into the malt and thus for any further experiment a longer time period approx. 5 days was decided to be applied. Moreover, to ensure that the malt samples were homogenised before testing their moisture, the use of a riffle box was specified.

Chapter 4 Comparison of the dielectric properties of whole malt and barley grains and the impact of moisture and frequency on them

4.1 Introduction

The dielectric properties of whole malt and barley grains hydrated in the same range of moisture contents were compared to identify any differences in the behaviour of the grains in the form of raw material and after the conventional malting treatment. Due to the lack of similar studies in the literature, this study could provide important information for the design and the application of the electromagnetic drying process of the grains.

As mentioned previously, there are only few references in the literature on the dielectric properties of barley (Ki-Bok Kim, 2002, Bhargava et al., 2013) and the measurements were performed using different techniques. Moreover, there was no comparison with respective results measured from malt samples. The accurate knowledge of the dielectric properties of malt and barley grains is important for the rapid, non-destructive estimation of the moisture content (Raghavan, 2005) and could also provide useful information in the study of the electromagnetic drying of the grains, the monitoring of moisture content during milling process and the selective heating of stored grains to control the presence of insects (Nelson, 1981).

In this section the methodology followed in order to perform measurements on the dielectric properties of malt and barley samples by applying the transmission line technique is described. The aim of those experiments was to study and compare the impact of electromagnetic energy on the dielectric properties of malt and barley whole grains hydrated in different levels at various stages of the kilning process.

Gathering a full data spectrum of the dielectric properties of both malt and barley and the impact of moisture content on them could contribute therefore to the design of experiments on the impact of electromagnetic heating on different level hydrated malt. Moreover, the benefits of understanding the behaviour of moist malt and barley grains when microwave energy is applied lies on the more accurate design of the drying process of malt in which volumetric heating (MW or RF energy) could be applied to achieve more efficient drying in terms of time and energy consumption and final product quality (AEA, 2011).

4.2 Experimental procedure

4.2.1 Raw Material

Tipple variety barley was used as previously in order to investigate the interactions between the electromagnetic energy (EM) and the range of moisture contents provided during kilning stage.

4.2.2 Preparation of samples

At first, the malting process of barley took place in the micromalting lab using CustomLab Micromaltings equipment. The capacity of the equipment used is described in details in Chapter 2 (§ 2.2.2).

This experiment required 6 kg of green malt, so 8 cages were filled with raw material (500 g barley for each cage) in order to give around 6 kg of green malt in total ready for drying in the kiln unit considering the fact that each 500g barley sample give approx. 750g of green malt.

After the germination process, malt was put in the kiln unit to produce the final malt product following the Standard Ale Malting Programme (details in Chapter 2, § 2.2.3). During the kilning process, air-on temperature, air-off temperature, humidity and airflow were continuously monitored and logged as previously. The malting programme used included a kiln cycle of 23 hours with a start temperature of 55°C and a maximum temperature of 95°C. Samples were then taken of at the following times points: t=0.5 h, 1h, 2h, 4h, 6h, 8h, 15h, and 16h in order to cover the same range of moisture contents with those of barley samples.

For the preparation of barley samples in different hydration levels, 5 samples of 300 g raw barley (variety Tipple) of 12% initial moisture content, wet basis, were immersed in tap water in 5 vessels and were then collected at various time points at t=1h, 2h, 4h, 10h and 48h to achieve the predetermined moisture content levels. Samples were stirred during the addition of water to get homogeneity in the bulk sample moisture (ASAE, 1993). 400 g of raw barley was dried in the Genlab OV/50/DIG oven 50 L capacity for 12 hours from 12% to approx. 5% moisture content at 60°C. For both malt and barley samples the moisture content was measured in triplicate using the HR83 Halogen Analyser following the “Analytica-EBC” 4.2 method using 2 g of pulverized sample collected in random from across the bulk at 105°C for malt and 130°C for barley samples. Samples were then vacuum sealed in aluminium bags and stored in refrigerated conditions (4°C) until the measurements of the dielectric properties were performed.

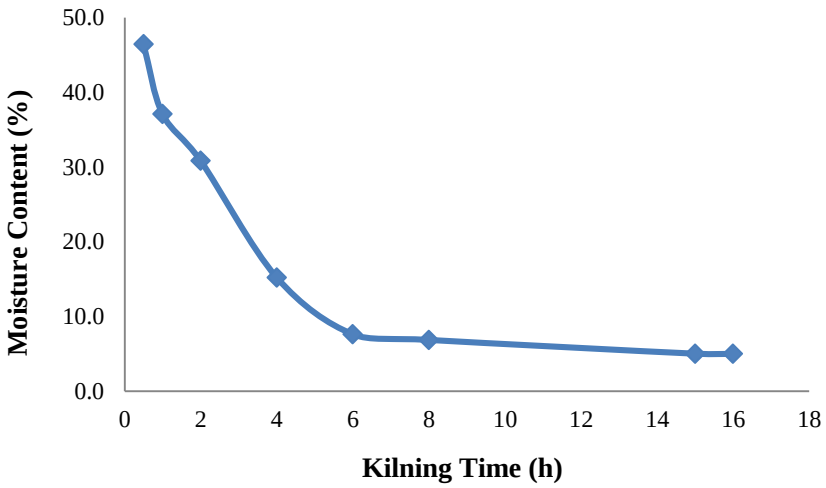

Figure 44. The relationship between the moisture content and time during the kilning process of malt.

4.2.3 Experimental setup for the dielectric properties measurement

The samples were further tested to measure their dielectric properties. The measurements were performed by applying the transmission line technique by applying a waveguide cell. The principle methodology was described in details in Section 2.4.2.

In this experiment, a WR430 waveguide calibration kit was used at a range of 2.3-2.5 GHz and waveguide cell dimensions were 20 x 109 x 55 mm^3 according to the equipment specifications. The VNA used was an Agilent 8753ES model with analysis capacity range 30 kHz – 6 GHz. At first, the connector plane was calibrated before taking any measurements using a type of short and fixed and sliding loads. All the measurements were performed at room temperature (~20°C). The S-parameters (S_{11} and S_{21}) representing the reflected and transmitted signal respectively were measured through the VNA and converted to complex dielectric parameters via "Python" programming language. Therefore, the dielectric constant (ε') and the dielectric loss factor (ε'') were determined.

The sample was fitted tightly into the waveguide cell to reduce faults in measurements causing by air gaps. As previously done the bulk densities of the samples were tested before starting the measurements in the waveguide using the same technique. The results are presented in the following table.

Table 10. Bulk densities and moisture contents of malt samples provided by different time points of the kilning process and the sample weights used in the WR430 waveguide cell. The standard deviation is also shown.

Time (h)	Moisture Content (%)	Bulk Density (g/mL)	Test sample Mass (g)
0.5	46.44 ± 0.05	0.62 ± 0.001	74.3 ± 0.14
1	37.09 ± 0.15	0.62 ± 0.001	74.3 ± 0.07
2	30.85 ± 0.51	0.62 ± 0.004	74.3 ± 0.43
4	15.22 ± 0.40	0.56 ± 0.006	67.1 ± 0.24
6	7.62 ± 0.12	0.52 ± 0.002	62.3 ± 0.12
8	6.86 ± 0.12	0.52 ± 0.001	62.3 ± 0.07
15	5.01 ± 0.24	0.52 ± 0.001	62.3 ± 0.12
16	4.99 ± 0.25	0.51 ± 0.001	62.3 ± 0.18

4.3 Malt dielectric properties results

The average of the dielectric constant (ε') and the dielectric loss factor (ε'') and the standard deviations of hydrated malt from various moisture contents ranging from 5% to 46% were measured in triplicate and the results were presented in the following figure:

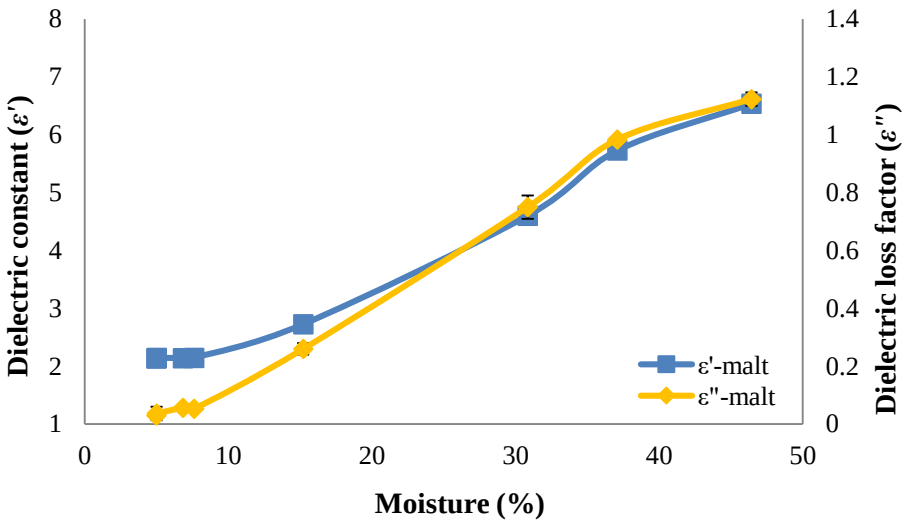

Figure 45. Impact of moisture content on the dielectric properties of malt samples provided by different time points during kilning process at 2.45 GHz, T= 20°C.

The ε' and the ε'' had similar trends compared to the impact of moisture content at 2.45 GHz. According to the waveguide measurements, the dielectric properties of malt increased almost linearly with increasing moisture content due to the effect of polarization of water molecules (Sumnu et al., 2005). It is also known from previous studies on the dielectric properties of wheat grains with bulk densities from 0.76 to 0.83 g/cm^3 at 2.45 GHz that an increase in moisture content leads to an increase in the ε' (ASAE, 1993). For the malt samples with moisture content below 10%, it was observed that ε' had an almost constant value of ~2.14 which indicated that the material was quite low loss and consequently it was more difficult for the grains to absorb the microwave energy. At this range of moisture content, water mostly exists in the endosperm of the grain and thus was more difficult to move in the surface and evaporate during conventional kilning process (Briggs, 1998). The ε'' values for moist malt under 10% were close to 0.03 which also showed that the grains were very low loss. However, the results of the dielectric properties at moisture contents of 10% and lower did not inhibit the possibility of the malt grains to convert the energy absorbed into heat and therefore to remove the existing moisture from the inside. It is generally accepted that in order to achieve a dielectric heating, loss factor of the material should have a value of $\varepsilon'' > 0.01$ (Piyasena et al., 2003).

Nevertheless, from the ε'' results in the low loss malt samples it was observed that the waveguide technique was not that sensitive for measurements in a range of moistures below 7% which came in accordance to similar observations in literature (Baker-Jarvis et al., 2010). Thus in order to provide more accurate results in the low loss samples

another technique such as cavity perturbation (resonant) method could be applied (Yaw, 2012) which gives more reliable results for small and low dielectric loss samples.

4.4 Barley dielectric properties results

4.4.1 Aims and Objectives

The next step was to use steeped barley samples taken at various stages of a manual performed steeping process along with kilned barley samples were investigated for their interaction with EM energy at a range of moisture contents. Before the dielectric properties measurements, the bulk density of the samples was controlled and waveguide technique was used for comparison to the outcomes from the malt dielectric properties measurements.

4.4.2 Preparation of barley samples

At first, raw barley (variety Tipple) was split in five pots of 300 g each and immersed in 1 L of tap water. Then the steeped barley samples were taken at various time points and used for the dielectric properties measurements. Moisture content was then measured in triplicate using the HR83 Halogen moisture analyser in "Barley Std." method. Before testing the moisture content, the surface moisture was removed from the samples by wiping those in blue towel paper to make sure that the analyser measured only the moisture which was absorbed from the grains and get precise results in the test. The method for the preparation of the barley samples was presented in more details in Section 4.2.2.

4.4.3 Dielectric Properties measurements and results

The measurements of the dielectric properties of barley were performed using the waveguide technique WR430 and two different volume cells were compared (V=502.15 cm^3 and V=119.9 cm^3).

For abbreviation in the graphs **502C** represented the cell of V=502 cm^3 and **120C** the cell of V=120 cm^3.

Measurements were highly dependent on bulk density and a variation in bulk density with moisture content studied prior to measurements being performed (Nelson, 1981).

The determination of the bulk density of samples was important to identify the exact mass of the sample that would be tested in the waveguide cell for its dielectric properties. Each sample was tested in five repeats however the relationship between the bulk density and the moisture content as presented in the following graph appeared to be almost constant as they varied from 0.615 to 0.623:

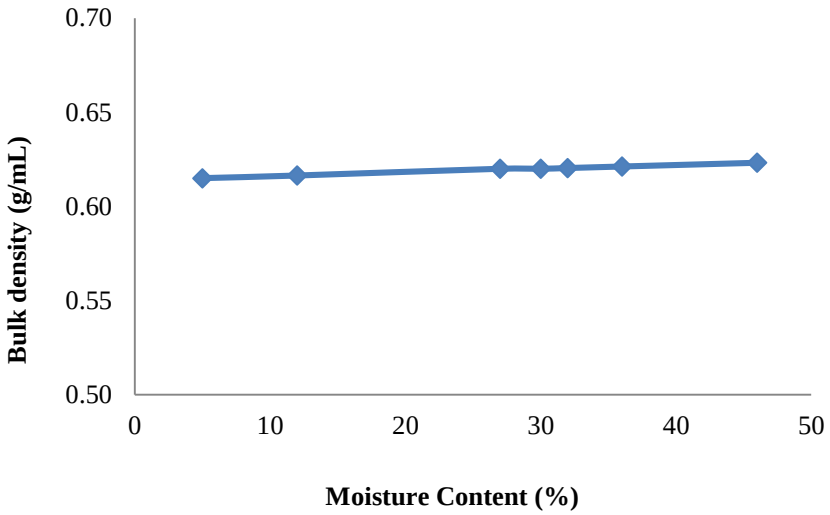

Figure 46. Impact of moisture content on the bulk density of barley samples.

As a consequence, from the above results, a standard bulk density (**0.62 g/mL**) was selected to be used in all samples in both 502C and 120C waveguide cells and the results were compared. In the following graphs, the dielectric properties compared to moisture content in both waveguide cell types at 2.45 GHz are presented.

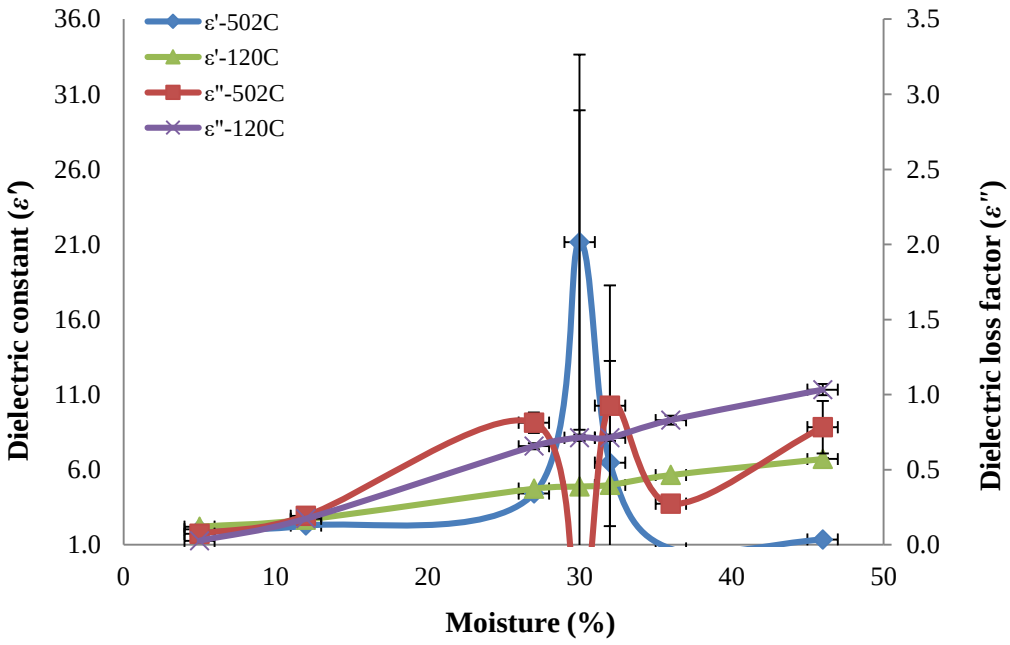

Figure 47. Comparison of the dielectric constant and loss factor to the moisture content of barley samples tested in 502C and 120C waveguide cells at different hydration levels at 2.45 GHz.

From the Figure 47 it could be observed that the samples measured in the waveguide cell 502C did not give reliable results for their dielectric properties something that was verified from the error bars. The above graph could be better presented by showing the dielectric properties of barley samples using the 120C (Fig. 48) and the 502C (Fig. 49) waveguide cell respectively.

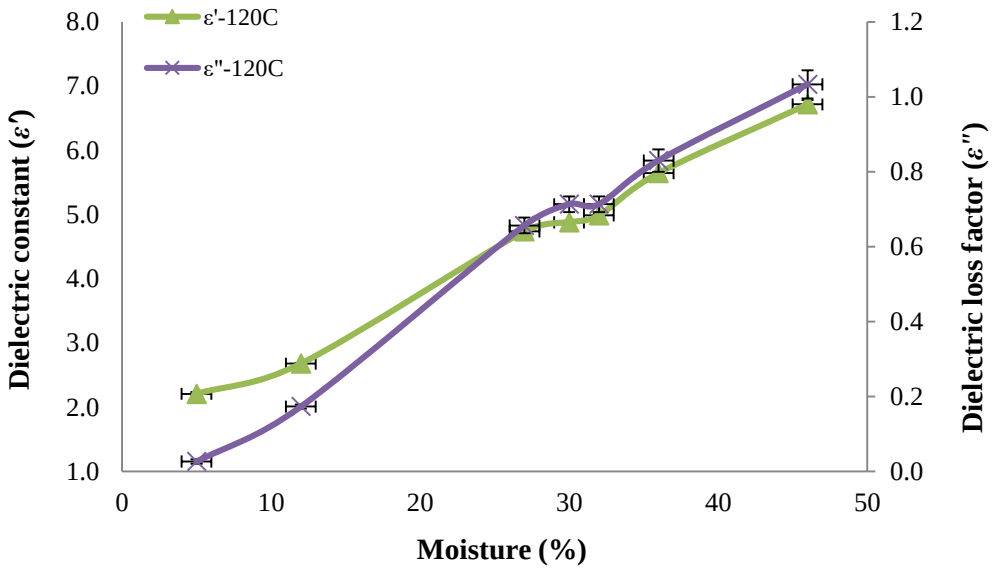

Figure 48. Dielectric properties of different hydration levels barley samples tested using the 120C waveguide cell at 2.45 GHz.

From the graph above (Fig. 48) the dielectric properties results of barley using the 120C waveguide cell had a good agreement with measurements presented in the literature for the 12% moisture content (Bhargava et al., 2013). To be more specific, in Bhargava study the results for the ε' and ε'' of barley were 2.28 and 0.28 respectively whilst in this study the corresponding results were 2.62 and 0.17. The

x axis error bars showed the false variation in the moisture content measurements while the y axis error bars showed the false variation in the dielectric properties measurements.

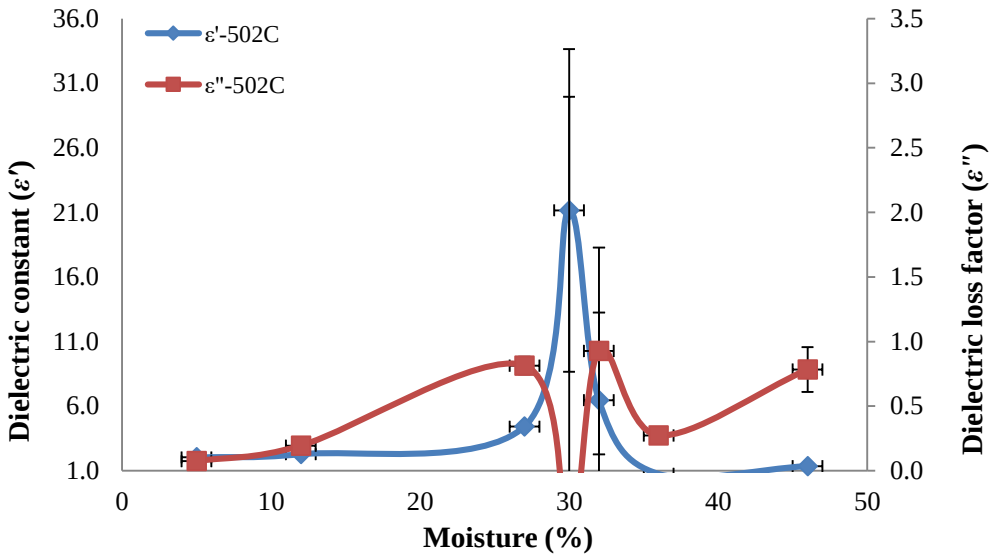

Figure 49. Dielectric properties of different hydration levels barley samples tested using the 502C waveguide cell at 2.45 GHz.

The outcomes from the barley dielectric properties measurements showed that the 502C cell was unsuitable to measure the properties of samples with moisture contents over approx. 25%, while 120C gave reproducible results across full range of moisture contents (coefficient variation <0.5%). As a consequence, 120C waveguide cell will be used for future bulk measurements at MW frequencies. It was observed that the larger the volume the more difficult it was to achieve good performance in the medium to high moisture content samples measurements. This trend could be explained by the fact that the large size cell had different specifications and it was not reliable for measurements of medium to high loss granular materials such as barley grains due to the fact that the waveguide is limited to low accuracy when the length of the sample is the multiple of one-half wavelength in the material (Yaw, 2012).

4.5 Comparison of the dielectric properties of malt and barley samples and the impact of moisture and frequency

A comparison of the dielectric properties results of malt and barley whole grains in the same range of moisture contents at 2.45 GHz was presented in Figure 50 for the measurements performed in the 120C waveguide cell.

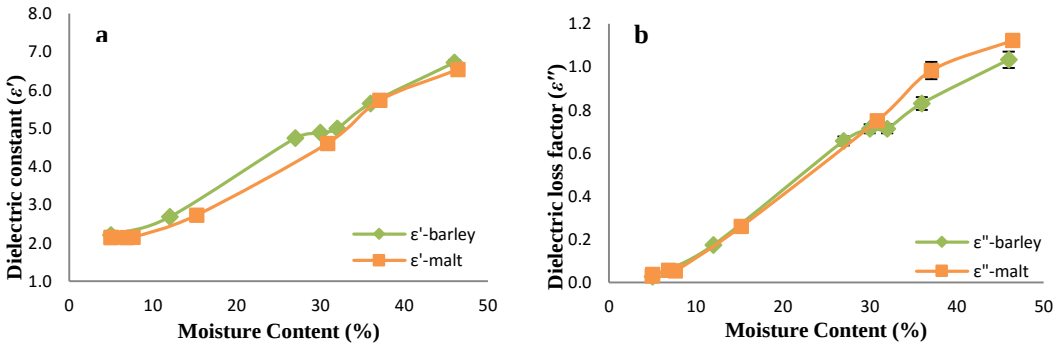

Figure 50. Impact of moisture content on: a) the dielectric constant (ε') and b) the dielectric loss factor (ε'') of barley and malt samples at 2.45 GHz, T= 20°C measured using the 120C waveguide cell.

It was notable that the results for malt and barley samples were quite close for the same range of moistures even though a difference in the bulk density existed as the barley samples were tested using a constant bulk density. The dielectric constant results of barley observed from 10% to 30% moisture content levels were a slightly higher than those of malt (Figure 50a), whilst loss factor results appeared to be identical for the same range of moisture contents. On the other hand, the loss factor results of malt were slightly higher compared to barley results at moistures between 30% and 46% as presented in Figure 50b, whilst dielectric constant results were similar at that range of moistures. An explanation about those small variations could be the difference in chemical composition between malt and barley samples as barley consisted mostly of starch unlike malt where enzymes are formed and released into the endosperm along with starch (Venkatesh and Raghavan, 2004). The almost linear increase of ε'' with moisture content at that range has been also observed in previous studies held on the impact of moisture in dielectric properties of food material (Piyasena et al., 2003) due to the dominant influence of moisture content on the dielectric properties as already has been proved in the literature (Nelson, 1981).

The correlation between the dielectric properties of malt and barley grains in different moisture content levels was summarized in Figure 51 by comparing the loss tangent (tanδ) which is the ratio of the dielectric loss factor to the dielectric constant (tanδ= $\varepsilon''/\varepsilon'$) with the moisture content.

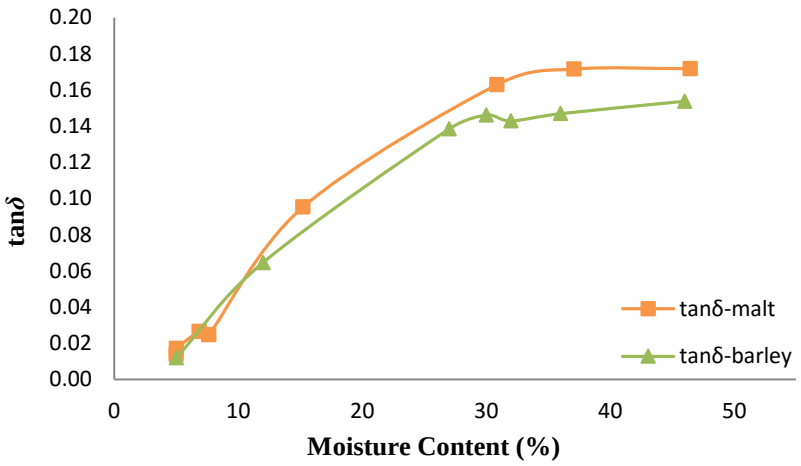

Figure 51. Variation of loss tangent (tanδ) of malt and barley samples with different moisture contents at 2.45 GHz and 20°C.

Because the loss tangent (tanδ) was determined by the dielectric properties of materials it was also observed in Figure 50 that this value increased with increasing moisture content in both malt and barley samples. It is generally accepted that a material with tanδ > 0.01 is characterized as medium to high loss (Piyasena et al., 2003) and thus both barley and malt samples with moisture content above 5% could be considered as good absorbers of microwave energy. Figure 46 verified the significant impact of moisture in the whole malt and barley grains at a constant high frequency (2.45 GHz) and room temperature (20°C) conditions.

The effect of frequency on the dielectric properties of whole malt and barley grains was also studied within the range of 2.3 to 2.5 GHz. The results were presented in Figure 52.

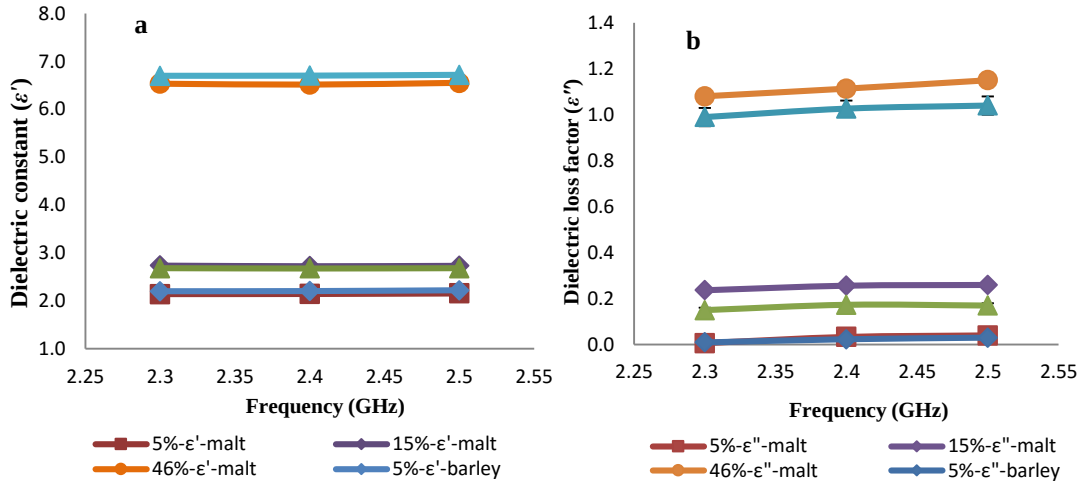

Figure 52. Impact of frequency on: a) the dielectric constant (ε') and b) the dielectric loss factor (ε'') of barley and malt samples at 5%, 15% and 46% moisture content at T=20°C.

From the results for the dielectric constant (ε') of malt and barley whole grains (Figure 51a), an interesting observation was that from 2.3 to 2.5 GHz ε' results were identical for malt and barley samples in each different moisture content. This trend has also been observed in a previous study conducted by Nelson (1981) at lower frequencies (1-50 MHz) in which the ε' was decreased or remained stable with increasing frequency.

The results for the dielectric loss factor (ε'') indicated similar trends about the effect of frequency in both malt and barley hydrated samples. However, the range of testing was only 0.2 GHz and as a result it might be useful to examine a wider range of frequencies to conclude in a safe outcome.

At low loss samples (5% moisture content) malt and barley ε'' had the same values whereas at higher moistures malt samples had slightly higher ε'' than barley samples. According to Nelson's work on the effect of frequency on several cereal grains, a similar trend has been observed for the ε'' results of hard red winter wheat (Nelson, 1981). Moreover, the small increase in the ε'' of malt and barley samples with increasing frequency in the highest moisture content was also observed by Nelson in earlier studies in wheat grains (Nelson, 1981).

4.6 Conclusions

The study of the dielectric properties of malt and barley grains showed that the moisture content was the dominant factor that affected the properties at a constant temperature and frequency. The bulk density of the material did not vary a lot with the moisture content for the bulk samples according to the measurements. The results came in accordance to literature sources related to the impact of moisture to the dielectric properties of barley and other cereal grains (Nelson, 1981) as the moisture content is the dominant factor that influences the dielectric properties of the grains.

From the experiments described in chapters 3 and 4 it could be presumed that the surface water of the grains is evaporated easily by applying the conventional drying process whilst for the evaporation of the endosperm's water the evaluation of electromagnetic energy appeared to be more promising due to the fact that the energy is directly applied to the moisturized part of the grain. Thus energy efficiency in the drying process could be achieved because the need to transfer heat from the low moisture surface into the high moisture interior eliminates (Feng et al., 2012). As a consequence, the electromagnetic heating of the hydrated malt could be more efficient when applied to moisture contents from 15% and below.

Chapter 5 Electromagnetic heating of malt using transmission radio frequency (TRF) tunnel

5.1 Introduction

The traditional drying process of malt in industry known as kilning has been unchanged for many years as it has proven effective and gives a final malt product of good quality (Briggs, 1998). However, the energy costs are high because the energy use can reach the 1000 kWh/t (AEA, 2011).

The major advantage of the application of electromagnetic energy are the selective and efficient delivery of heat to the areas of hydrated food products where the water is found unlike to conventional heating where drying process starts from the surface layers and then is difficult to dry the inside part of the product where the water is insulated (Thostenson and Chou, 1999).

Even though microwaves have been applied for many years in food processing, there are only few studies for the evaluation of electromagnetic heating technology into the malt processing as described below. The main goals of using electromagnetic heating techniques in the drying process of malt are the reduction of kilning time, the lower energy consumption, the preservation of the enzyme activity and the higher homogeneity of the product (Chang et al., 2011) .

The research which has been conducted in this field considers the implementation of microwave only heating, RF only heating or the combination of conventional and volumetric heating into different stages of malting process as referred below. The latter case is a very promising alternative according to recent researches into the kilning process of malted barley. It is important to notice that the knowledge of the safe temperature limits of grains is essential in order to avoid harmful influence of the microwave application such as the deactivation of the enzymes.

In *López-Perea et al. 2008* study, the quality of feed and malting barley was investigated by applying microwave heating in different time points by using a domestic microwave oven. When microwaves applied for more than 10 secs, kernel temperature was increased more than the safe limit for the final product and therefore affected germination. The irradiation of the grains for 4 secs, showed a reduction in kernel hardness as well as an increase in the extract content that leaded to good quality final malt according to the specifications of brewing industry. In contrast, when

microwave heat applied for 8 secs, kernel hardness was higher. The study also showed a significant correlation between the malt extract and the hardness of barley kernel. Moreover, the extract viscosity was measured and had a decrease in all different time treatments of microwave heating. Furthermore, the enzyme activity did not show any influence by microwave irradiation as it appeared by the measurement of diastatic power and α-amylase. The short time treatment of barley grains by using microwave energy allows the softening of the barley kernel endosperm and gives a malt product of acceptable quality in terms of the brewing industry specifications (Lopez-Perea et al., 2008). So this study indicated that the short time treatment of the grains with microwave heat could lead to good quality final product.

A recent study for the use of combined microwave-hot air heat in the roasting of malt was held by *Boivin et al. 2010*, based on the measurement of the energy consumption and the content of harmful compounds. During the experimental procedure, malt was treated with microwave heating in a pilot scale microwave oven at 2.45 GHz for rapid increase of the temperature and then was transferred to a domestic oven. After heating, malt was cooled in ice-water bath to 20°C. The outcomes of the study showed a decrease of the energy cost by 26-40% depending on the type of roasted malt. Moreover, kilning time was reduced and the decrease of surface moisture was more efficient compared to microwave only heating. The final moisture content was in 1-3% which is similar to conventional heating level. The neo-formed contaminants content was reduced from 18-95% depending on the compound (Akkarachaneeyakorn et al., 2010).

In another project, designed by *Eichhorn 2010*, a combination of conventional and radio frequency drying energy was used to optimize the kilning process. For that reason, malt was treated at first with the conventional heating and then with radio frequency heating. The results showed a more rapid and more efficient drying process, as it finished at approximately 15 hours and the grains were more homogeneously dried. Moreover, malt quality was comparable to that of the conventional only heating kilning process (Eichhorn, 2010). Information provided by this study could be used as guidance for the design of experimental work on the radio frequency drying of malt.

The most interesting research was held by *Kruger et al.* in 1981 and refers to the production of malt by using microwave heating. The experimental procedure took place in lab scale by applying switched on and off microwave heating to maintain the temperature constant. The process needed more energy than the conventional drying. The energy consumption for the finished malt was 994 kWh /ton and the moisture content was 5%. However, this drying method was gentler and leaded to better quality

of malt according to malt analysis test that compared both cases (Kruger and Groneick, 1981).

In this series of experiments, the interaction between radio frequency energy and hydrated malt grains was investigated at a range of moistures similar to those which can be found during the conventional kilning process of malt. The aim of this project was to develop and apply a method to reduce time and energy costs and to enhance the product quality in terms of the enzyme activity in the kilning process which is used to dry malted barley by using radio frequency energy.

Through this study a better understanding of the behaviour of the evaporation of moisture while heated by electromagnetic heating could be achieved. Moreover, the impact of several variable factors such as the energy intensity and the absorbed and reflected power was investigated. Knowledge gained will then be useful for further opportunities to apply these technologies to the malting industry. After every experimental treatment by applying the electromagnetic heating the impact to the dried malt enzyme activity was tested. Furthermore, it is important that the treatment must not adversely affect any of the key brewing (or distilling) quality parameters which are currently specified by the malt end-users. Potential benefits from this study include savings in energy and process time whilst increasing product homogeneity and thus the quality of malt.

5.2 Dielectric properties and electromagnetic drying process design

In order to design an electromagnetic drying process, the knowledge of the dielectric properties was essential because they provide useful information about the potential of a material to absorb the electromagnetic energy and convert it into heat. The higher are the dielectric properties values the easier is for the material to be heated when applying the electromagnetic energy. The electromagnetic drying is more advantageous than the surface heating methods due to the selectivity of heating which results in rapid drying of the hydrated interior of a product and consequently to relatively high time and energy efficiency (Feng et al., 2012).

The results for the dielectric constant and the loss factor of malt as presented in the graph in Fig. 40 provided by the transmission line waveguide measurements at 2.45 GHz over a range of moisture contents typically found during the conventional kilning process showed that moisture was the dominant factor that affected the properties. However, for the comparison of the results a rapid series of tests using the cavity

perturbation technique (Section 1.6.5.3) were performed as a more accurate method for the measurement of the dielectric properties of low hydrated (low loss) samples. The results showed that as the frequency decreased the ability of the low loss malt samples to absorb the electromagnetic energy slightly increased. This technique is highly accurate for the low loss malt samples at low moistures. For comparison samples from high and medium loss malt was also tested.

The samples used for each test consisted of 2-3 whole grains inside a 4 mm glass tube which inserted in the centre of the cavity and measurements were taken in 3 repeats for every different moisture content sample test by applying several frequencies. For the determination of the dielectric properties, as described in Chapter 2, the resonant frequency and the quality factor of an empty cavity were firstly measured and then the measurement was repeated by filling the cavity with the test sample. The complex permittivity and permeability were then calculated using the frequency, volume and Q-factor. The sample densities inside the tubes in those tests were 2.5 g/cm^3 for the 45% hydrated malt, 2.16 g/cm^3 for the 35% hydrated malt and 1.45 g/cm^3 for the 10% hydrated malt. The frequencies applied were following the specifications of the cavity.

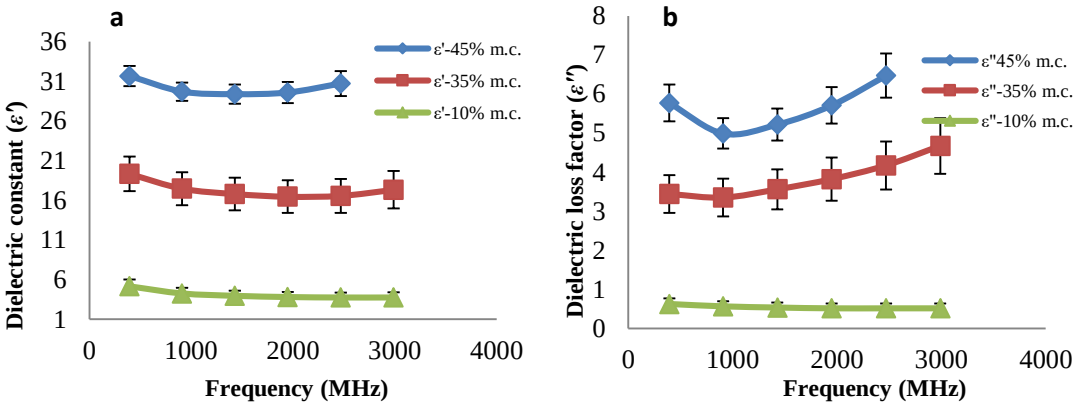

Figure 53. Impact of different resonant frequencies on: a) the dielectric constant (ε') and b) the dielectric loss factor (ε'') of high, medium and low hydrated malt samples with 45%, 35% and 10% moisture content respectively at T=20°C using the cavity perturbation technique.

Table 11. Results from the cavity perturbation measurement using resonant frequencies on the dielectric properties of low moisture content malt samples.

	Dielectric constant (ε')	Dielectric loss factor (ε'')
Frequencies (MHz)	Low loss malt (10 % Moisture Content)	
395	5.15	0.62
910	4.24	0.56
1429	3.94	0.53
1949	3.78	0.51
2470	3.72	0.51
2989	3.73	0.51

The results presented in the Fig. 53 and Table 11, came in agreement with previous experiments on the dielectric properties of low hydrated malt using the waveguide (Chapter 4). The outcomes were a good indicator for the application of low frequencies such as radio frequencies to the low loss malt. It was observed that as the frequency decreased the dielectric properties were slightly higher. This observation was previously mentioned in Nelson's studies (Nelson, 1981).

Moreover, assuming that the grains contain only water the penetration depth of pure water at 2.45 GHz are 2 cm at 40°C and decreases with lower temperature. Thus the penetration depth of a microwave reactor filled with a high loss material will be smaller and consequently unacceptable hot spots will be presented which is the reason why stirring is necessary when microwave heating is applied. However, with decreasing frequency the penetration depth increases and for radio frequencies is close to meters (Stuerga, 2008).

Thus the radio frequencies could be more effective for the evaporation of the water presented in the endosperm as they have greater penetration depth than microwave frequencies and could also be superior for heating large quantities of malt. The high values of the dielectric properties in the medium and high loss samples were not considered to be reliable compared to the results from the waveguide technique for the respective range of moistures.

5.3 Study of the RF energy impact to high moisture malted barley

5.3.1 Aims and Objectives

As a preliminary study, the impact of different energies to high hydrated malt product approx. 45% was investigated. The target energies were based on the theoretical thermodynamic minimum energy required to remove the moisture from the malt.
The experiments of this study had a scoping character in order to understand at first place how rapidly malt is drying by applying different radio frequency energies and in which extend the enzyme activity of the final material is influenced. For this reason, the enzyme activity of the conventional dried malt was compared to RF treated malt through malt analysis tests.

5.3.2 Preparation of malt samples

In this preliminary experiment, 13.4 kg of green malt at around 45% moisture (lager type) was transferred by "MolsonCoors" industry (Burton on Trent, UK) for use in the RF treatment. The material was split in 5 representative samples from which 12 kg were treated through the RF tunnel drying process (3 samples). 1.3 kg of the initial batch was used to dry by the conventional kilning process and take samples at different time points during the day of the experiment (2 samples). The initial moisture content was measured twice using the HR83 Halogen moisture analyser in "Barley Std" method. The procedure followed for the preparation of conventional dried malt samples was the same as previously described in chapter 4 using the large vessels during the kilning process.
The green malt which used in the RF treatment was transferred to the microwave lab to be used in the scoping experiment using the RF tunnel.

5.3.3 Experimental procedure of the RF drying of malt

In every test 4 kg of green malt were used and the samples placed in the belt of the tunnel in four plastic trays (310x400x50mm) which filled the area of the electrodes within the material would be dried as shown in Fig.29. Besides the fact that the capacity of each tray was approx. 2 kg of malt in the study only 1 kg per tray was used

as a preliminary study for the effect of different energies applied to thin layer bulk material. The use of the thin layer bulk material aimed to test the impact of radio frequencies in low volume bulk.

Thus, from the four trays heated in between the electrodes only the one was used as the "test sample" whilst the other three trays used as "sacrificial material" needed to be hydrated in the same moisture content to that of the test sample in each drying process trial. In that way the bulk moisture of the four trays would be kept in the same level so the RF energy could heat uniformly the material. The sacrificial material was necessary to achieve a homogenous distribution of the electromagnetic heat through the electrodes and the test sample was therefore analysed for its enzyme activity.

At the end of each treatment the dried material was placed in large metallic trays (660x740x28mm) and left to cool to ambient temperature for 20 min. All the quantity remained was stored in the freezer in case of reuse as sacrificial material. The samples were packed in vacuum sealed packs and remained in the freezer until testing the moisture content and the malt enzyme activity.

To design the RF processing the theoretical model used to calculate the thermodynamic minimum energy which required to dry 1 kg of green malt from 45% to 5% moisture content was the following (Singh and Heldman, 2014):

- Energy stored: $E=Cp \times dT \times m_{H2O}$
- Energy released: $Q=m_{H2O} \times L$
- Final energy per g H_2O: $E+Q$

Where: - C_p is the specific heat capacity of the wet grains (J / (g x K))

- dT represents the difference between the evaporation temperature of water and the temperature of the sample before the RF treatment which is room temperature (°C)
- m_{H2O} is the weight of water in the initial sample mass per trial where the energy is stored (g)
- L is the latent heat of water (2260 J/g) which represents the energy per weight of water required to evaporate the absorbed moisture of the material

So according to this theoretical model the required energy to dry the malt from 45% to 5% was 1037.8 kJ/ton or 288.3 kWh/ton. Considering the fact that only 12 kg of green malt would be used in this series of tests, three trials were performed at 3 kW forward power as following:

Test 1: 300 kWh/t → t = 1440 s (24 min)
Test 2: 450 kWh/t → t = 2280 s (38 min)
Test 3: 600 kWh/t → t = 3000 s (50 min)

The different energies applied in the RF tunnel for the electromagnetic drying of malt interpreted as different times of processing. Thus for the minimum energy required for the drying of malt, the time of processing adjusted to 24 min and in order to double the energy input almost 50 min of processing required. After the drying process with the RF heating, the surface bulk sample temperature was measured by using an infrared thermal imaging camera which measured the average and the minimum and maximum temperature of a picture.

The initial matching parameters adjusted for all tests were the following:

The electrodes height adjusted to 65 mm, the adjustment selfs (both right and left) to 114.4 mm, the load to 300 and the S11 (impedance) to -35 dB. The initial surface temperature of the bulk sample in the trays was 23.5°C in all trials and was determined by using the IR thermal imaging camera.

In all tests during the RF drying process the variable parameters were autotuned to maintain the power absorbed from the grains in the same rate. After the RF treatment the bulk malt was used to dry to the final 5% moisture content through the conventional kilning process in case that the RF treatment would not succeed to dry the material to this level and study the effect of the combination of the both drying processes to the enzyme activity of malt. The tests for the enzyme activity analysis are described in details in Section 2.3.2.

5.3.4 Results of the RF treatment and malt analysis tests

At the end of the RF drying process the surface bulk temperature was measured by using the NEC thermal imaging camera and the results are presented in the Fig. 54.

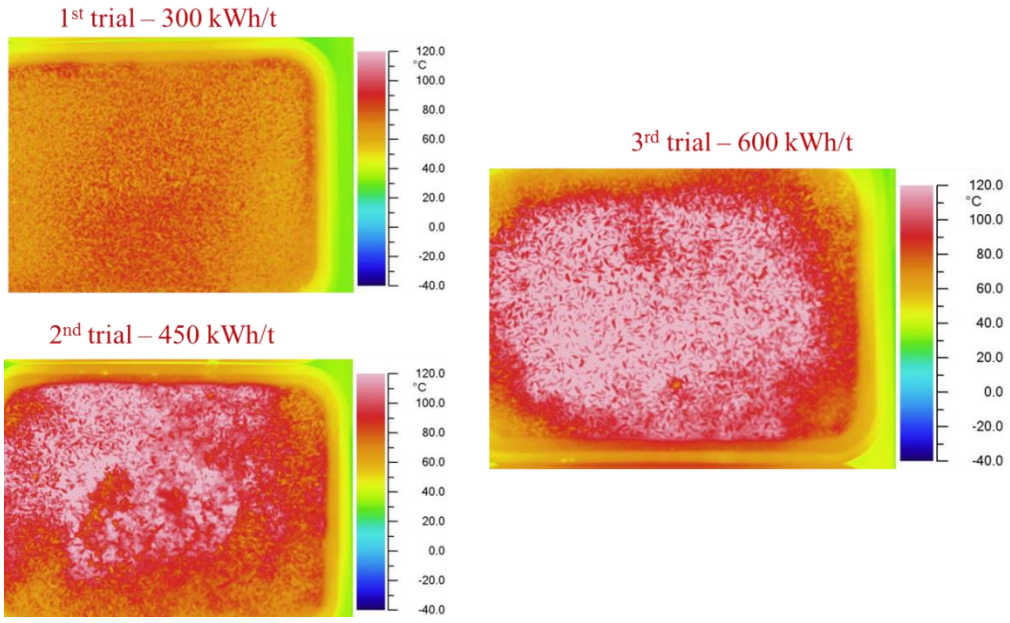

Figure 54. IR images of the surface bulk temperature of malt from the test trays after the different energy input RF drying treatment.

Table 12. Summarized data from the RF drying trials of the 45% hydrated malt.

	Target Energy (kWh/t)	Time of processing (min)	Surface max. temperature after RF drying (°C)	Moisture content at the end of RF drying (%)
1st trial (RF1)	300	24	103	25.3
2nd trial (RF2)	450	38	130	13.2
3rd trial (RF3)	600	50	>130	5.4

From the images above and the moisture content results shown in Table 12 it was clear that the longer was the treatment the more was the water evaporated.

It was observed that during the RF drying process the auto tuning changed the values of the load and electrodes height mostly from 10 to 15 min in all tests and more rapidly from 30 min onwards in 2nd and 3rd trial. The fact that the system auto-changed the variable parameters through processing could be indicative of the changing in the dielectric properties of malt due to the evaporation of water as during the first minutes the water was found in the surface of the bulk material and it was easier to evaporate whilst as the drying process continued the water was mostly found in the endosperm

of malt and thus required more energy input to be selectively heated and evaporate. Thus the system maintained the forward power constant and energy losses did not appear due to reflected power. So the material absorbed the 100% of the energy provided by the system. Consequently, the dielectric properties of the bulk material varied through the electromagnetic drying and as it was proved in the study presented in Chapter 4 the properties decreased with less moisture in the grains.

Malt was burned due to high energy input and low bulk mass in the test trays which indicated by the low depth of bed. It was also observed that most of the burned grains were those in direct contact with the tray which could be explained due to less heat flow away from the sample and the heat was shielded in the interior of the tray because vapour could not escape to the surface.

The material from the "test tray" from each trial was transferred to the brewing lab in order to test the moisture content and then continue the drying process through the conventional kilning process.

The bulk material was mixed with a riffle box in order to achieve homogeneity before continue the drying in the kiln unit. In the meantime, a sample from the initial batch was already put in the kiln unit approx. at the same time as the RF drying experiments started, to be used as a reference and investigate if there was an impact of the time on the germination of the grains and compare its enzyme activity to that of the RF treated samples. Thus two drying processes were running in parallel and the results were compared by testing the final moisture content and the enzyme activity.

The final moisture contents of the combined dried samples were compared to those from the same initial batch which dried only by using the conventional heating process. Moisture content was checked in triplicate using the HR83 Halogen moisture analyser in "Malt Std." method. Then, the samples were vacuum packed in labelled aluminium pouches and stored at the room temperature until the performance of the malt analysis tests.

Table 13. Final moisture contents of samples dried with the RF heating and conventional heating and compared to those dried only through kilning process.

Moisture Contents after Conventional kilning process	
Tests	**Av. M.C. (%)**
Green Malt conventional kilning dried	**3.74**
RF 1 (300 kWh/t)	**4.00**
RF 2 (450 kWh/t)	**3.78**
RF 3 (600 kWh/t)	**3.46**

Following the *β*-Amylase Megazyme kit, the samples were therefore tested for their enzyme activity. The procedures followed for malt extract, FAN and colour were described in details in Chapter 2. The results are presented in the table below:

Table 14. Malt analysis results for Conventional dried and RF dried malt samples.

Malt analysis				
	CD	**RF 1**	**RF 2**	**RF 3**
***α*-Amylase (DU)/g of milled malt**	32.21	6.56	4.99	4.55
***β*-Amylase Betamyl-3 Unit**	5.60	0.47	0.87	0.70
EBC Colour	7.5	17.3	26.2	71.5
FAN (mg/L)	290.7	246.8	231.9	192.9
Malt extract (%)	81.27	77.73	78.91	78.74
Malt extract (%) d.w.b.	85.55	81.82	83.06	82.89

From the results presented in the Table 14, it was observed that the enzymes in the RF treated samples were totally inactivated. However substantial colour was developed probably due to the extra roasting of the grains. The CD malt represented the conventional dried malt from which the test malt was prepared.

As the field intensity for this experiment was too high for the length of exposure, the optimisation of the bed depth by using more quantity of sample to increase the bulk volume as well as the application of longer treatment which means less energy input could help to minimise the fraction of burned grains and keep the energy at low rates.

Additionally, the manual control of the variable parameters could be used to achieve a more extensive study over the absorption of the electromagnetic energy on malt and protect the enzymes to be more resistant during RF processing. In order to avoid the harsh burning of the grains a combination of the drying techniques could be applied by drying first the grains with the conventional kiln to a certain moisture content and the continue the process by applying the RF energy. Moreover, the presence of steam in the test area of the RF tunnel indicated the need to use a system with compressed air and an air extractor to stop the condensation of the evaporated moisture in the system.

5.4 Study of the RF energy impact to medium loss malted barley

5.4.1 Aims and Objectives

In this series of experiment in the TRF tunnel larger bulk volume of initial sample was used in order to increase the depth of bed and achieve better distribution of heat transfer. As the height of the bed increases, more moisture evaporates from the grain due to mass transfer (Srivastava and John, 2002). The effect of the energy applied to barley malt samples with initial moisture content ~30% was tested. The decrease of the initial moisture content was selected because according to the results from the dielectric properties studies the lower was the moisture content of malt the lower was the dielectric loss factor. Thus the power density decreases and consequently less heat could be applied to dry the material and the burning of the grains could be avoided.
The target energies applied to dry the samples to the final 4% moisture content in the RF tunnel by keeping the forward power constant at 3 kW (the lowest power that could be used) were based on the theoretical thermodynamic model. The target final moisture is selected according to the industrial specifications for the final malt product which is 4% - 5% (AEA, 2011).

5.4.2 Preparation of samples for the RF drying test

To run the experiment, 24 kg of malt was used in total in order to run 3 tests in the RF tunnel. The 18 kg was prepared by steeped malt up to 30% moisture content to be used as sacrificial material during the RF drying test. To prepare the material, dried Tipple malt at 6% moisture content was put in a bucket and sprayed with the appropriate

amount of water to reach the 30% moisture content level. More details on the methodology of the steeping process were presented in § 3.5. The material was kept in enclosed buckets in room temperature conditions. At the end of steeping process, the moisture content of the sacrificial malt was tested and found approx. 30.5% hydrated.
The malt used as the "test sample" in the RF tunnel tests, was transferred again by "MolsonCoors" industry. 15 kg of 40% initial moisture content was put in the large kiln vessel to dry using conventional heating to 30% moisture content and use it further in the RF drying experiments. The programme set for the preparation of the "test malt" is presented in details in § 3.6. The moisture content of the "test malt" after the conventional drying process was 31.44%.

5.4.3 Experimental procedure of the RF drying of malt

During the RF drying process the maximum temperature applied should not be more than 90°C to avoid burning the grains and preserve the enzyme activity of the amylases.
The electrodes height was fixed at a specific range of variation (assuming 62-68 mm) before each test according to the observations from the previous series of experiments.
The adjustment selfs and load capacity were adjusted manually to prematch the impedance by looking the dB (i.e. approx. 100 dB means that 99% of the energy applied is absorbed) in the VNA monitor. A value of -30 to -35 dB was acceptable. Then the automatch was applied to adjust the system through the VNA monitor and the reflected power (RP) values were recorded every minute during the drying process from the matching box.
During processing airflow system was applied by using a constant supply of compressed air (140 L/min) applied to the feed end and extracted with the "Nederman" E-PAK 500 fume extractor to avoid the condensation of water inside the tunnel.
All the trials with the RF heating were done at a constant forward power at 3 kW (lowest power). In each test different energies applied as a variable parameter starting with the thermodynamic minimum for 8 kg of malt which is 194.6 kWh /t (31 min). So, the time of processing was different in each test.
Thus in order to dry 8 kg of malt from 30% to 4% at FP=3 kW, 3 tests took place in the RF tunnel:

Test 1: 195 kWh/t → 31 min
Test 2: 300 kWh/t → 48 min
Test 3: 400 kWh/t → 64 min

During processing the system was continuously auto adjusted (changing of the load capacity/electrode height) to keep the forward power constant. However, reflected power was presented as the dielectric properties of malt were changing. At the end of each treatment the dried material was placed in the large metallic trays and left cool back to ambient temperature for 20 min.

In addition, the sacrificial material was thrown away and the surface temperature and moisture content of the "test sample" was measured directly by using the thermal imaging camera and the HR83 halogen analyser respectively. A batch of 1 kg from the "test sample" was kept to measure the moisture content and stored at room temperature until the malt analysis. The last step of the experiment was the investigation of the impact of the RF heating to the enzyme activity of the final kilned malt product by running the α, β-amylases malt analysis tests.

5.4.4 Results of the RF treatment and malt analysis tests

The surface bulk temperature was measured again at the end of the RF drying process by using the NEC thermal imaging camera and the results are presented in the Fig. 55.

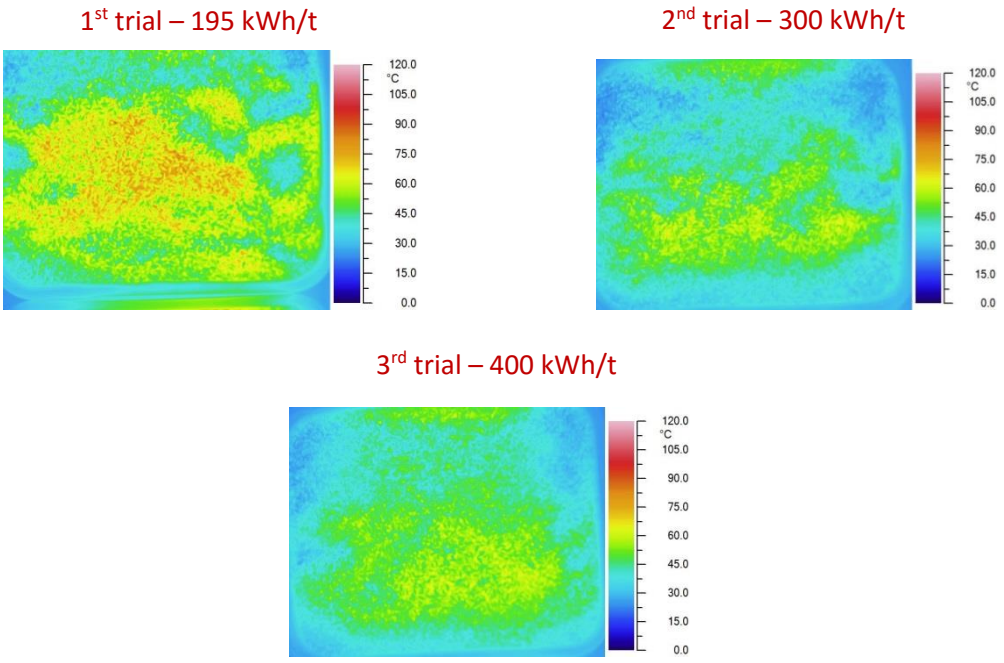

Figure 55. IR images of the surface bulk temperature of malt from the test trays at the end of the RF drying treatment.

From the images above comparing to those in Fig. 54 for the 300 kWh/t and 400 kWh/t, it was shown that by increasing the depth of the sample in the trays gentler drying of the material was achieved. However, the presence of reflected power at the higher energy input tests had as a result the lower final surface temperature. The following table includes the results provided by the 3 tests which took place in the RF tunnel for the 30% hydrated malt by applying different target energies.

Table 15. Summarized data from the RF drying trials of the 30% hydrated malt.

	Target Energy (kWh/t)	Energy Absorbed (kWh/t)	Time of processing (min)	Surface max. temperature after RF drying (°C)	Final moisture content after RF drying (%)
1st trial (RF1)	195	169	31	72.9	16.4
2nd trial (RF2)	300	213.3	48	69.8	13.4
3rd trial (RF3)	400	260.4	64	67.4	8.5

During this series of tests the final surface temperature of the bulk sample was reduced significantly in comparison to the final temperature of the previous experiments due to the larger volume of the test material which leaded to better heat distribution in the test trays and the application of the air flow system. The moisture content of the test sample for each trial was measured in the HR83 halogen analyser using the methodology as described previously. To get sufficient homogeneity of the samples tested a riffle box was used to mix the grains to have representative samples from across the bulk. The final moisture content for the 400 kWh/t trial was quite close (8.5%) to the desirable final moisture content (5%).

After the RF treatment a batch of approx. 1 kg from each "test sample" was transferred to the Brewing Science lab to continue the drying process using the conventional kilning process as being done in the previous experiment to 4% final moisture using the kilning programme described in Section 2.1.3 (table 3). In the meantime, a sample from the initial batch of green malt was already put in the kiln unit at approx. the same time as the RF drying started to investigate the impact of the time gap between the preparation of green malt samples in the Brewing lab and RF drying tests in the Microwave lab on the germination of the grains and the enzyme activity.

At the end of the conventional drying process, the RF treated samples were mixed in the riffle box and their moistures checked again in comparison to conventional dried sample and the amylase activity tests were performed following the β-Amylase Megazyme kit as described in Chapter 2. Tests were only performed in the α and β amylase activity because those are the key enzymes to make the final malt product acceptable for the brewing industry (Briggs, 1998).

Table 16. Moisture content of the RF treated "test malt" after the conventional kilning process compared to conventional dried malt.

	CD malt	**RF1 sample**	**RF2 sample**	**RF3 sample**
Moisture Content after Conventional Drying (%)	3.54	4.37	4.51	4.56

Table 17. Enzyme activity of the RF dried "test malt" compared to conventional dried malt from the same initial batch.

	CD Malt	**RF 1**	**RF 2**	**RF 3**
α-amylase (DU units)	41.50	5.50	12.32	7.89
β-amylase (betamyl-3 units)	13.65	0.14	1.17	0.78

The RF treated samples had higher final moisture contents even after the conventional drying process. The enzyme activity of α and β amylase was again nonexistent compared to the conventional dried initial batch which proved that the amylases of the RF treated grains were destroyed even in lower temperatures.

As an additional test, 350 g from each RF "test sample" which transferred to the Brewing lab was kept and vacuum packed in aluminum pouches and stored in the freezer to study if the different storage conditions could provoke any changes in the enzyme activity of the malt compared to the samples that kept in room temperature until the final drying process in the kiln unit and the malt analysis afterwards.

After 7 days, the RF samples defrost and dried to 4% moisture content along with a sample from the initial green malt batch by using the "Ale1" conventional kilning programme (Table 3) and then the moisture content and the amylase activity was tested.

Table 18. Moisture content of the freezer stored RF treated bulk "test malt" compared to conventional dried malt after the kilning process.

	CD malt	**RF1 sample**	**RF2 sample**	**RF3 sample**
Moisture Content after Conventional Drying (%)	4.63	4.23	4.38	4.41

Table 19. Comparison of the amylase activity of RF treated bulk "test malt" stored in different temperatures and conventional dried malt after the kilning process.

	Samples stored in room temperature				Samples stored in the freezer			
	CD Malt	**RF 1**	**RF 2**	**RF 3**	**CD Malt**	**RF 1**	**RF 2**	**RF 3**
α-amylase (DU units)	41.50	5.50	12.32	7.89	40.82	8.13	7.07	8.50
β-amylase (betamyl-3 units)	13.65	0.14	1.17	0.78	13.30	0.95	0.74	0.99

The malt sample which provided the initial green malt batch was not affected by the freezing and defrosting as it had similar values in both cases. However, it appeared to be a difference in the results of the RF dried samples which could be explained due to sampling errors. The samples used for the malt analysis were selected in random from the initial bulk sample batch after the kilning process without mixing again in the riffle box for each test and consequently they might not be representative of the whole batch.

From the images taken with the thermal camera it was observed that there was variability in the temperature across the bulk sample which could explain the differences found in the enzyme activity of the RF treated malt grains.
The application of lower energy input, the use of bigger bulk mass of the sample and lower initial moisture content appeared to have a positive effect in the decrease of the bulk temperature and the slight increase of the amylase activity. However, the variability of the temperature after the RF treatment across each tray required the need to study more extensively how the temperature and moisture content changes during the RF drying in several smaller samples from across the bulk sample of the tray. Moreover, the middle and lower layers of the trays are insulated and steam could not escape easily, thus the bottom layer appeared more hydrated than the surface layer and that was another reason of the variability in the final moisture in the bulk sample.
Thus the sampling methodology of the "test malt" used for malt analysis should be improved by applying good mixing before doing malt analysis tests in the bulk material or the bulk samples could be studied separately as smaller parts from across the bulk malt in order to understand the changing of the moisture content in the grains.

5.5 Study of the RF energy impact to low loss malted barley and the variability of the bulk samples

5.5.1 Aims and Objectives

The aim of this experiment was to investigate the effect of the RF energy applied to malt samples of low initial moisture contents (20 % and 16%) as the area of interest for the selective heating is the medium to low loss hydrated malt. In addition, to improve the accuracy in the sampling of the "test malt" after the RF treatment, the determination of the variability across the bed of the tray in the "test malt" was tested. The samples were dried in the RF tunnel to reach the 4% moisture content by applying different energies keeping the forward power stable at 3 kW.

5.5.2 Preparation of the test samples

To prepare the sacrificial material for the two different moisture content series of experiments 12 kg of malt was required in total. Dried Tipple malt of 6% moisture content was hydrated in buckets with water up to 20% and 16% moisture by using a

spray bottle. The amount of water required to spray the grains with was calculated as previously (Section 5.3.3).

The quantity of green malt required to get dry in the kiln unit in order to produce the "test malt" was 14 kg in total for the 20% and 16% moisture content. The preparation took place in the micromalting lab and the green malt was put in the kiln unit for approx. 10 hours to decrease the moisture to ~16%.

The green malt used was produced by 4.4 kg of Tipple barley initial batch using the cylinder cages.

It is important to notice that in order to get homogeneity of the material the green malt was split through a riffle box before the kilning process. Afterwards, a random sample of 10 g from across the bulk malt was taken and measured its moisture content by using the HR83 halogen analyser in duplicate to calculate how much time needed to dry the material to 20% and 16% respectively.

During the kilning process a sample of 2 kg at 20% moisture and another sample of 2 kg at 16% moisture were expected to take off which both would be the "test malts" in the RF experiments.

Before checking the moisture of the samples from the kiln unit the riffle box was used again to ensure that the moisture of each sample would be representative of the bulk volume. Both the 20% and 16% hydrated test samples were stored in sealed plastic buckets in refrigerated conditions (cold room) overnight until the performance of the RF trials the following day.

5.5.3 Experimental process of the RF trials

The effect of radio frequency energy applied to 16% and 20% initial moisture content malted barley to the final 4% moisture content was investigated. Once again the decrease of the initial moisture was decided as an effective way to reduce the burning of the final product due to the decrease of the dielectric loss factor and the selective electromagnetic heating of the interior of the grains.

In this test, the lowest target energy was applied calculated by the theoretical thermodynamic minimum for 8 kg of malt to decrease the moisture from 16% to 4% was 86.5 kWh / t (14 min).

For drying the malt from 20% initial moisture content to the final 4% moisture content 200 kWh/ t (32 min) was applied. The trials with the RF heating were performed again at 3 kW forward power. In this series of experiments, the variables were auto controlled again trying to keep the energy losses minor during processing.

In both tests, a riffle box was used before treatment to achieve homogeneity in the bulk malt ("test" and "sacrificial"). Before and after treatment, pictures of the "test tray" with the thermal imaging camera were taken. After each trial, a photo of the test tray divided into smaller areas was taken by using a grid to specify the sampling area of the individual samples from across the bulk which would be used further for malt analysis testing. Thus, 8 to 10 samples per trial of approx. 30 g each would be compared between them for their moisture content and their enzyme activity and to the bulk sample to test the variation in those values. The small samples were packed directly in sealed containers and stored at approx. 5°C till processing.

The rest of the malt was proceeding as a bulk sample after being homogenized by using a riffle box.

If the samples were not dried to final 4% by using the RF tunnel, they would be put in a conventional oven and let dry by setting a programme similar to the "Ale1" in micromaltings with starting temperature 55°C. At the end of the drying process, samples would be vacuum sealed until running the amylase analysis tests.

5.5.4 Results of the RF treatment and malt analysis tests

The surface bulk temperature was measured again at the end of the RF drying process by using the NEC thermal imaging camera and the results are presented in the Fig. 56.

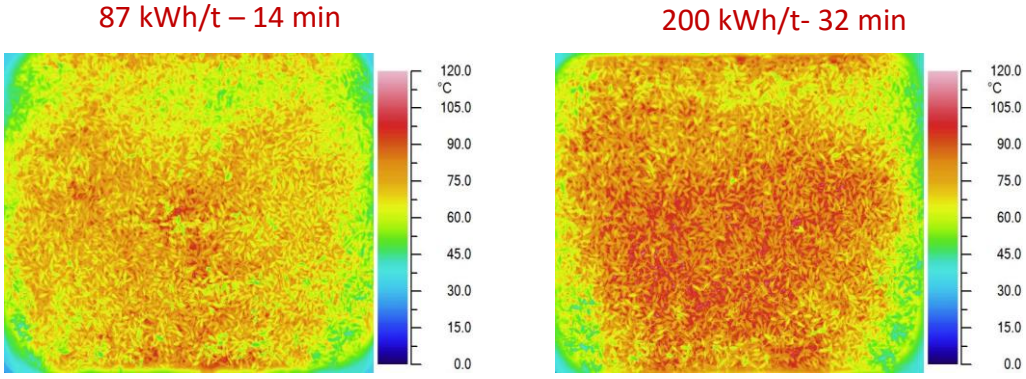

Figure 56. IR images of the surface temperature of the bulk malt from the test trays at the end of the RF drying trials as showed at 120°C high limit.

After taking a photo of the bulk "test samples" at the end of the RF drying process a grid was used to divide the sample into smaller individual samples in random which were therefore tested for their moisture content and enzyme activity. Photos with the

divided samples were taken and the positions of the smaller samples from across the bulk were spotted as presented in Fig. 57.

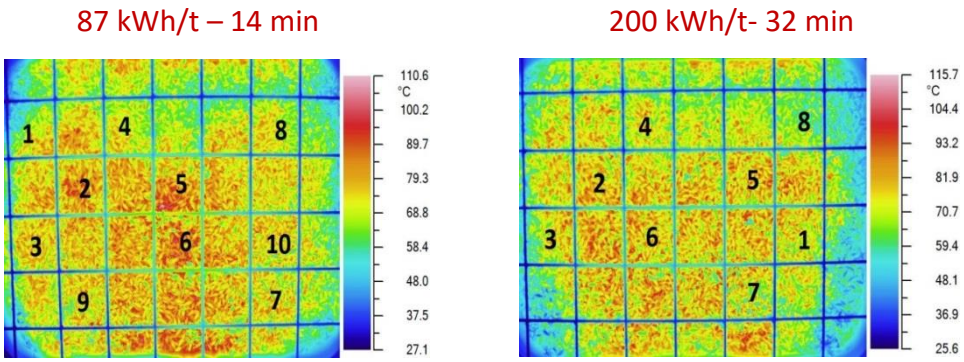

Figure 57. Surface temperature of the bulk malt from the test trays at the end of the RF drying trials and their highest values. The bulk samples were divided with a grid in smaller samples in order to test their moisture content and enzyme activity.

Table 20. Summarized data from the RF drying trials of the 16% and 20% hydrated bulk malt.

	Target Energy (kWh/t)	Energy Absorbed (kWh/t)	Time of processing (min)	Surface max. temperature after RF drying (°C)	Final moisture content after RF drying (%)
1st trial (RF1)	87	82.3	14	110.6	9.6
2nd trial (RF2)	200	192.4	32	115.7	5.6

From the results above for the bulk sample it was clear that the "test malt" achieved to remove the moisture from the inside almost close to the target 4% content which is the industrial specification of the final malt product. Because the average moisture of the bulk samples did not achieve to get dried to 4%, a portion of the bulk sample along with the small samples from across the bulk were therefore dried in the kiln unit for about 5 hours at 55°C. Then their moisture content was tested in the HR83 halogen analyser and the malt analysis was performed using a portion from those samples.

The results of the malt analysis for the small samples from across the bulk and the comparison to the bulk sample are presented in the Fig. 58 below.

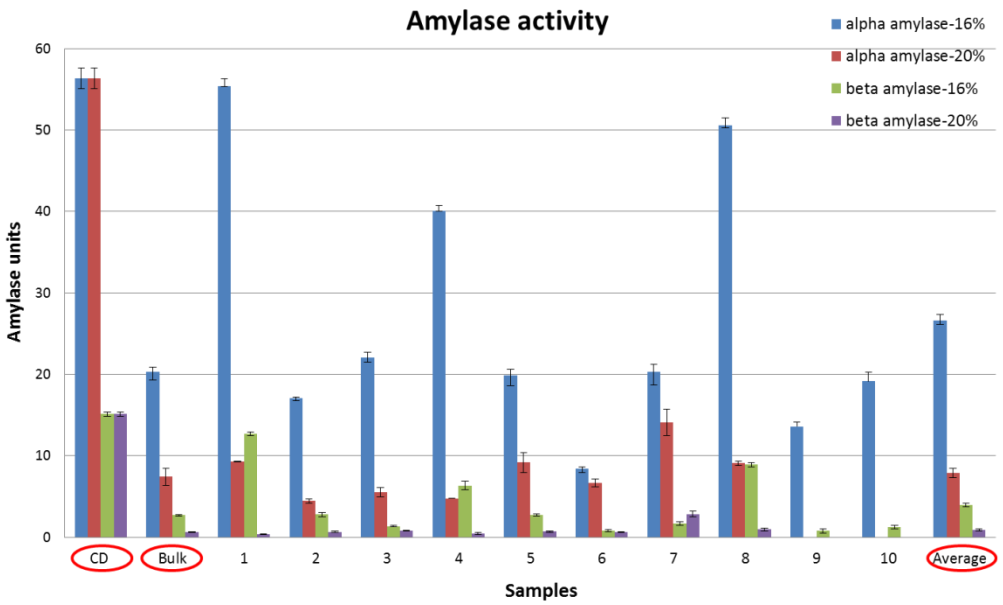

Figure 58. The amylase activity of bulk samples comparing to various small samples from across the bulk volume for the 16% and 20% hydrated samples.

In the graph of Fig. 58 the amylase activity of a reference conventional dried malt sample, the bulk sample of the RF dried "test malt" and the various smaller samples from across the bulk sample was compared. The average bar showed the average amylase activity of the small samples for each test in comparison to the bulk sample activity. The blue and red bars represented the α-amylase activity for the 16% and 20% RF test sample respectively. The green and purple bars represented the β-amylase activity. Despite the high temperatures that presented the key enzymes and especially the α-amylase had better yields of surviving for the 16% hydrated test sample as presented in the graph probably because the treatment was shorter. However the enzyme activity for the small samples from across the bulk for the 16% moisture content sample varied a lot compared to the 20% moisture content sample. Comparing the amylase values for the conventional dried sample with the bulk sample and the average activity of the small samples from across the bulk volume, it was observed that once again the amylase activity was quite low but the results of the bulk volume and the average from the small samples were very similar. Thus the average amylase activity values appeared to be representative of the whole bulk sample. It was also observed that some of the small samples from the bulk volume had a remarkable

activity for the α-amylase. Because β-amylase is more heat sensitive it had lower activity in all cases.

As a complementary test the friability of the bulk RF treated samples was measured as an indicator of the extent to which the endosperm has been modified during germination. The results presented in the following table were very promising in comparison to conventional dried samples as the friability had increased and this was an indication that malt was modified better. The friability of the RF treated samples decreased with increased initial moisture when the lowest energy input was applied for each case.

Table 21. Friability results of the RF drying trials of the 16% and 20% hydrated bulk malt.

	Unmodified part (g)	**Friability (%)**
Conventional dried malt	7.78	84.44
RF Trial 1	5.42	89.16
RF Trial 2	6.64	86.72

5.5.5 Conclusions

The RF drying process of low initial moisture content malt was more promising for the preservation of the enzyme activity especially for the α-amylase. However, the average amylase values from the small samples compared to those from the bulk samples did not have a great difference apart from the α-amylase value for 16% moisture content which appeared to be higher. Friability had greater value for the 16% moisture content sample as presented in the Table 21, something that comes in accordance to the enzyme activity results.

5.6 Study the effect of the impedance matching on low loss malted barley

5.6.1 Aims and Objectives

The impedance matching is necessary to ensure maximum power transfer between the RF energy and its load. Without the impedance matching the phenomenon of reflected power could provoke damage to the RF circuit. As the generator's output impedance is 50Ω the real part of the impedance should be equal to the real part of the load (50) while reactance should be equal and opposite in character (0). Thus in order to achieve matching for a source impedance R+jX the load should be R-jX (Todorow, 2009).

The aim of the following tests was to understand and control the effect of matching to eliminate the energy losses in the system during the RF processing of malt. As the malt dries during processing, dielectric properties change due to water evaporation and reflected power increases. This test was performed to understand how the changes occurred could be controlled so that during drying process the system could be adjusted in such a way to maintain reflected power at low rates.

The impedance matching using material of low moisture contents could provide an operating window to maintain the reflected power during processing at a rate of 1% or lower (approx. -20 dB).

5.6.2 Experimental set up

During those tests the parameters which remained constant were the electrodes height and the moisture content. The variable parameters were the load capacity and the adjustable selfs of the tunnel. Those selfs have wheels adjusted on the exterior, with movement counters to record position in mm to optimize the RF energy transmission.

For the preparation of the samples, five batches of 8 kg of malt each, were used produced in the same way as the sacrificial material in the RF trials. Each batch had different moisture content from 12% to 4% to cover the area of interest for the RF drying experiments of malt. The electrodes height was fixed at 61 mm in all tests as a constant parameter. This value was selected because it represented the lowest distance between the electrodes that could be used without "touching" the material according to the equipment specifications. In that way the voltage of the system remained at low levels.

The attenuation was adjusted at the begging of the experimental process at a range of -20 dB and lower by changing the load capacitance. The selfs were then adjusted to record the data from the "marker" in the VNA to approx. -20 dB from both sides of the curve for every different moisture content test. Thus for every different load value selected, two values for the selfs were recorded. So, by increasing and decreasing the load capacity, the selfs should be adjusted in that way to keep the attenuation constant to -20 dB for each sample.

The frequency range of the triode was 30 Hz which means that from the peak of the curve (settled at 27.12 MHz) a frequency shift of 30 Hz from both sides could be occurred (Fig. 59). The data collected gave information about the range of attenuations in between the system should be adjusted to keep the reflected power at <1% and the range of values in which the parameters should be set to maintain the reflection at 1% or lower.

5.6.3 Results and Conclusions

Starting with the 12% moisture content sample the material covered the area between the electrodes which was normally exposed to the RF energy. Then for every different load value selected to keep the shift of frequency in a certain zone, the attenuation was adjusted to -20 dB or lower by controlling the two adjustable selfs. This value was selected according to the theoretical calculation for the attenuation (dB), as it is known that -20 dB is translated as a decrease of 99% in the reflected power (David Kleinfeld). The results recorded presented in 1MHz screen resolution in the VNA monitor and were extracted as excel files including the range of attenuations as the S_{11} parameters and the frequencies recorded. The data for the 12% hydrated sample from the VNA monitor are presented in the following graph (Fig. 59).

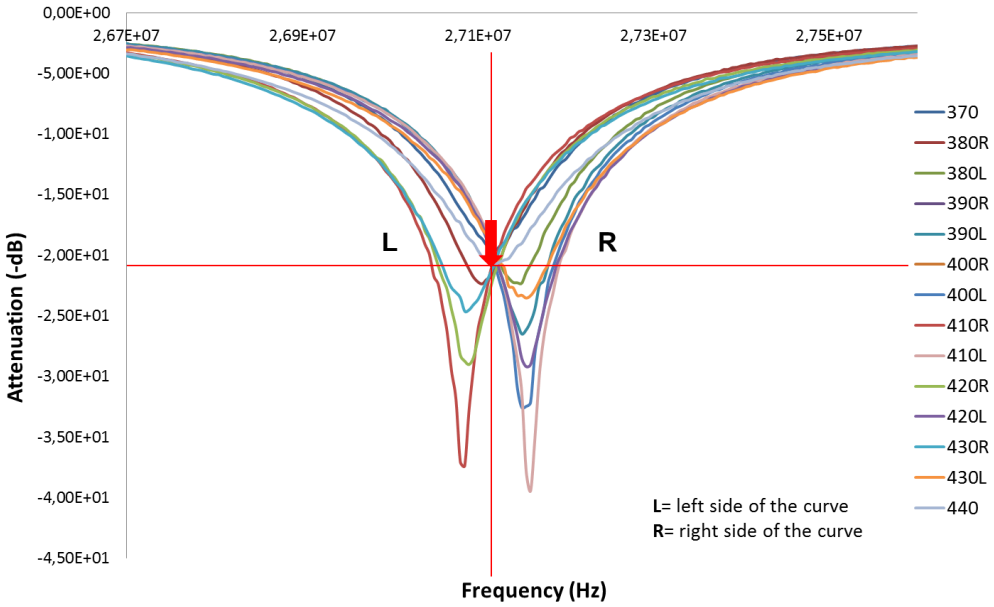

Figure 59. Data collected for several loads with attenuations of -20dB or lower at a screen resolution of 1MHz for the 12% hydrated malt.

In Fig. 59 the arrow represents the "marker" in the VNA monitor and the different coloured lines in the graph represent the different load capacities tested. From the data shown in Fig. 59 the shift of the attenuation according to the different loads applied was not more than 0.02×10^7 Hz. Similar graphs were prepared for the results collected from the 10%, 8%, 6% and 4% moisture content malt samples tested. It was observed that the lower was the moisture content the less was the shift of the frequency from the one to the other side of the curves for the different loads. Thus, fewer changes in the controllable parameters would be required to maintain the reflected power at 1% or lower during processing. This trend was clearer when comparing the bandwidth to the load capacitance for all different moisture content as shown in Fig. 60 below.

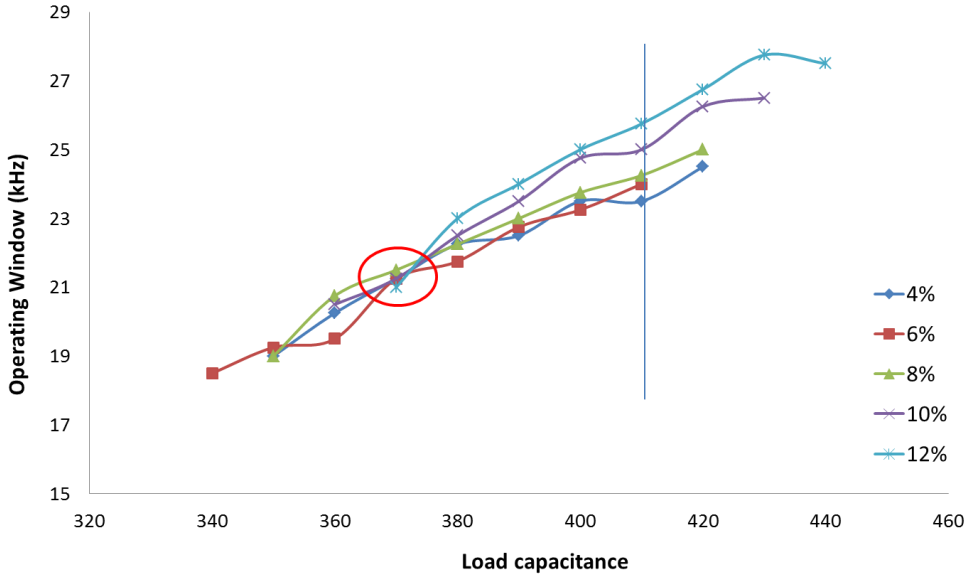

Figure 60. Bandwidths presenting as the operating window for the different load capacitance for all the samples tested.

The bandwidth was calculated by plotting the data collected from the VNA for each different moisture content sample to identify easier the range of frequency shift that could be used as a "secure zone" to achieve -20 dB or lower. From the data presented in the graph it was observed that the wider was the bandwidth the wider was the curve and therefore the shift of frequency could remain in a safe zone between -20 dB and below for all different moistures at a given load capacitance. The vertical blue line shows the ideal load capacitance which could be used for start to provide a safe zone to work on from the range of 12% moisture to 4% final moisture. The load capacitance with value 370 could be the lowest to apply to all the different moisture content samples.

The graph presented in Fig. 61 represented the average values of the data recorded in both sides of the curve at a range of load capacitances for the different moisture content samples. The error bars represented the limited zone in between the attenuation could reach a value of -20 dB or lower as derived from the values recorded from the adjustable selfs from both sides.

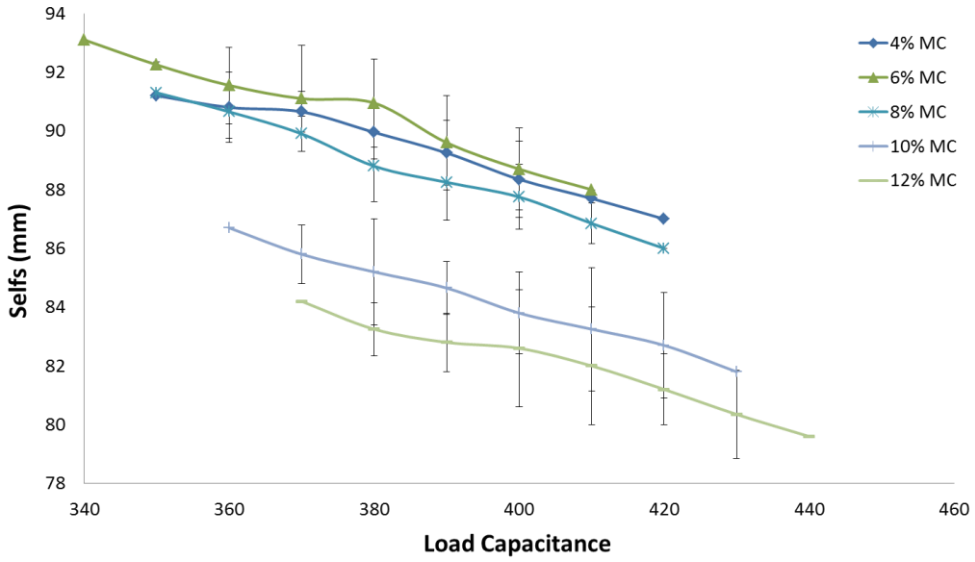

Figure 61. Average values from the adjustable selfs for the different load capacitance and the different moisture content samples.

Thus a matrix with the relationship between selfs and load capacity for all different moisture content was prepared. Using this, the point where keeping the selfs fixed could be identified and the reflected power rate could be controlled to 1% or lower only by changing the load capacitance during processing, according to the different hydration samples used. The last graph (Fig. 62) shows the trend of the changing on the adjustable selfs (both sides) of the RF tunnel for a given load capacitance value when the moisture is increased.

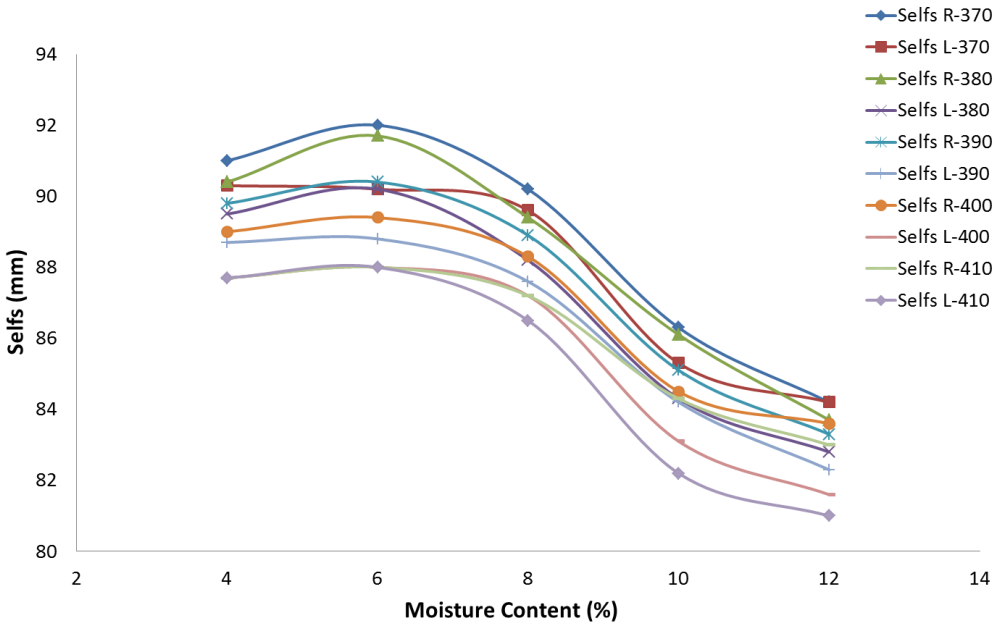

Figure 62. The adjustment trend for the RF tunnel selfs for the various moisture contents samples for a given load capacitance.

It is observed that as the moisture content of malt decreased the selfs values should be increased for a given load capacitance. However, from approx. 7% moisture and below the material appeared to be stabilized. This trend could be explained by the fact that the dielectric properties of the material were changing as the moisture decreases. From 7% moisture and lower malt appeared to be stable due to the fact that dielectric properties did not change, something that came in accordance with the results provided in Chapter 3.

The study of the impedance matching influence to the RF processing of malt was only based on the effect of the different moisture content samples on the adjustable parameters. However, during the RF drying process the effect of temperature was also highly dominant and could have an impact on the energy losses. From the data extracted in this series of tests, starting a drying test with a load value of approx. 410 could provide a promising zone to work with, from 12% moisture to 4% final moisture content with minimum power losses.

Chapter 6 Conclusions and Future Work

The main aim of this project was to study, develop and optimise a methodology for the alternative drying process of malt by applying electromagnetic energy. The expectations from the evaluation of this technology were to reduce the time of processing and the energy costs and also to preserve the quality of the final product according to brewing industry specifications. Bulk grains are complex systems with starchy endosperms formed by organic substances with a protein matrix filled with small and large starch granules (Bamforth, 2009). Thus, the efficient evaporation of water from the inside of the grains is a quite difficult and time intensive procedure.

The investigation of the impact of electromagnetic energy on the dielectric properties of different hydration levels of malt and barley bulk samples at microwave frequencies were first undertaken. The range of frequencies selected for this study was from 2.3 to 2.6 GHz as the wavelength should exceeds the grain kernel dimensions by not less than a factor of 10 (Serdyuk, 2008). Thus, the radiation would interact with the dielectric properties of the malt as the average of its whole bulk volume.

The transmission line technique was selected as more appropriate for the measurement of the dielectric properties of high to low loss samples at a range of MW frequencies from 2.3 to 2.6 GHz. Two different types of waveguide cell were used and the results showed good reproducibility for the WR430 cell for all different hydration level malt samples. According to the results, water content was the leading factor affecting the dielectric properties of both malt and barley grains and there was a strong similarity between malt and barley dielectric properties at a set moisture content. The outcomes of those tests were used as indicators about the behaviour of malt and barley when microwave energy is applied in different levels of hydration.

Following the characterisation of these dielectric properties at microwave frequencies, it was decided that the penetration depth of microwaves in barley grains was unlikely to deliver sufficient throughput for an industrial process to be developed. Hence we opted next to investigate the application of radio frequencies (RF) to the drying of malt. The decrease of frequency leads to an increase of the penetration depth which at frequencies below 100 MHz is close to meters (Stuerga, 2008). However, the microwave energy would be more efficient for the remove of bound water at very low moistures, from 2% and below (Serdyuk, 2008).

In order to design and test the impact of RF energy on malt a lot of steps were required. Firstly, the optimisation of the logistics of sample preparation was important, as the experiments took place across two different campuses and it was important to

ensure that time lapse and storage conditions would not affect the results obtained. Moreover, the development of a methodology to produce large quantities of malt which could then be used in pilot-scale RF drying tests was accomplished under reproducible conditions in a micromalting kiln unit. The accuracy of sampling and measurement of moisture content were important in the design of the RF tunnel drying tests. Hence additional tests were performed in order to study and compare the results provided by both the conventional oven and the HR83 halogen balance method. The outcomes showed that the rapid method of the HR83 halogen balance was as reliable as the more time consuming oven drying method and could be used to test the moisture content of grain in the RF experiments, provided representative sampling methods were used.

The study of the impact of radio frequency drying on hydrated malt in different moisture contents was performed in high, medium and low loss samples. During processing, the forward power was kept constant at the lowest rate that could be applied according to the system specifications (3kW). Different target applied energies were selected for testing based on the basic thermodynamic equations for a certain weight of sample following the assumption that there was only moisture content loss during processing. In each series of experiments optimisations of the system and efforts to reduce the product maximum temperature were attempted by decreasing the applied energy. Unfortunately, α- and β-amylase activities were almost absent for the high and medium loss samples, most likely due to the high final temperatures which destroyed the thermosensitive enzymes.

A better outcome was achieved when RF energy was applied to malt at 16% moisture content instead of 30% or higher, whereby the enzyme activity was somewhat better preserved. The average values were 26.64 DU for α-amylase and 3.93 Betamyl-3 Unit for β-amylase. Moreover, tests for the optimisation of impedance matching were performed on low loss material (from 12% to 4% moisture content) as this was the area of interest to achieve high drying and time efficiency and better enzyme preservation. The application of electromagnetic energy to low loss malt is in theory energy efficient due to the fact that the energy is directly coupled with the moisture and therefore it eliminates the need to transfer heat from the low moisture surface into the high moisture interior as in conventional drying processes. The variation of amylase activity across the bulk treated sample was also tested and the results showed that due to the different temperature profile in the small spots from across the bulk sample the amylase activity showed great variation. However, for the 16% and 20% hydrated malt tested the average amylase activity value (especially α-amylase) as measured from the small random samples from the bulk was quite close to that of the

bulk sample (Fig.58). The combined drying of low loss malt appears to be more promising to achieve a final good quality product.

Suggestions for future work:

Based on the experimental work and the results obtained from this project, the way forward for this area of research could include the following:

- Investigation of the transient temperature profile during the RF drying process. Due to the existing high final bulk temperatures at the end of the drying process of low initial moisture malt samples, monitoring of the temperature during processing by using a fibre optic thermometer is recommended. These could be inserted at several sites across the bulk sample and would be ideal to monitor and help control the temperature profile of the grains. To collect more accurate results, optic fibres should be inserted in between grains found in different layers and regions across the bulk sample, following the observations from the study described in Section 5.5. A comparison with the temperature profile of the bulk malt during the conventional kilning process would be also useful.
- As an additional series of experiments the way that amylase activity is changing for set initial moisture content by the effect of temperature during the RF processing could provide key information with which to optimise the process. This series of tests could help to design the heating profile required to raise and keep the process at a temperature non-destructive for the enzyme activity. Thus, using the outcomes provided by the study of the temperature profile effect and the matching parameters tests, the optimisation of the RF drying process of low hydrated malt could be achieved. Consequently, time and energy efficiency as well as good quality of final product in terms of the preservation of the enzyme activity could be achieved.
- A series of experiments based on the application of lower radio frequencies can be evaluated. To be more specific according to the trend of the dielectric properties as being investigated so far the application of 13.56 MHz frequency might result in even better enzyme survival, shorter processing times and lower energy consumption.
- To verify the above assumption a series of tests to characterize the dielectric properties of barley and malt at radio frequencies over the same range of moistures as those used in the microwave dielectric experiments would be useful.

- Having decided the ideal frequency and energy input for the RF drying of malt the next step could be the application of combined RF drying and conventional drying (kilning) processes as an efficient method to reduce the time of processing and maintain the quality of the final product.

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