Developments in bitumen emulsion mixtures for roads

by

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ABSTRACT

Bitumen emulsion mixtures, or cold mix, offers certain advantages over hot bituminous road mixtures in terms of potential cost savings, environmental factors, energy savings and easing of logistical difficulties inherent with hot mix. For these reasons, they are currently used in a number of regions including the United States of America, Continental Europe, Scandinavia, Southern Africa and Australia. However, cold mix has not been seriously evaluated or utilized in the UK until comparatively recently. This is due to the slow rate at which it builds strength or cures and its susceptibility to rainfall, particularly during this curing period. The overall aim of this research was, therefore, to study the behaviour of cold mix in terms of mechanical properties and the mechanisms involved in emulsion breaking and mixture curing to gain an insight into how performance may be improved.

Mechanical properties were shown to be affected by a number of parameters, including binder grade, void content, curing time and additives such as cement. Fatigue tests showed that without cement, the performance in-situ of cold mix would be very poor. Pavement design calculations revealed that, with cement, emulsion mixtures could be expected to perform similarly to equivalent hot mix. Field trials have indicated that cold mix can be manufactured using conventional hot mix plant and laid using similar techniques. However, problems are still to be overcome in terms of the control of mixture composition and laying in adverse weather conditions.

Fundamental tests have shown that emulsion droplet coalescence (which is an essential part of the curing process) was affected by pressure, bitumen type, emulsifier level, cement and temperature and that cement can cause emulsion charges to become more positive (or less negative) but other parameters had no effect on charge. It was also proved that cement can cause bitumen emulsion to set completely and that the residual binder has an increased viscosity compared with unmodified binder. This was shown to be due to the formation of a composite bitumen cement binder.

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The work described in this thesis was carried out at the University of Nottingham and Akzo Nobel Chemicals' Research Centre at Littleborough between October 1992 and October 1995. This thesis is the result of my own work, except where specific reference has been made to the work of others. No part of this work has been or is currently being submitted for any other qualification.

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CHAPTER 1

INTRODUCTION

1.1 ROADS MIXTURES AND COLD MIX

In recent years mankind has become increasingly dependant on road transport for both commercial and personal use. In the United Kingdom, the majority of Europe and the rest of the developed world, freight haulage has experienced a major shift from water and rail based transport to roads, and the use of the 'family car' has become a way of life. Demand upon road networks in countries all around the world has risen far beyond the levels anticipated 20 years ago and axle loads have increased significantly. Inevitably, due to this increased burden, pavement structures are deteriorating as a result of structural failure and coupled with the need to build new roads to carry yet more traffic, the requirement for road construction is ever increasing.

In the UK, traditional methods of road construction are employed in nearly all of the structural layers of road building projects. These involve the use of hot mixtures of aggregate and bitumen for which manufacturing and laying plant are well established and the technology has a proven record. However, over the last decade, there has been a realization that cold processes, which use bitumen emulsion in place of hot bitumen, have a part to play. A number of countries, such as the United States of America, France and those in Scandinavia, have made use of cold processes for many years as discussed in chapter 3 of this report, but current technology only allows them to be used for particular applications in certain situations. In the United Kingdom, bitumen emulsions are used only in surface overlay processes and bitumen emulsion based mixtures are in their infancy.

1.2 HISTORICAL REVIEW

Throughout history, as civilizations have developed and grown, road construction has been necessary to carry increasing levels of traffic to and from regions of dense population. The earliest examples of roads are known to have radiated from ancient Babylon and highways existed in 5th century B.C. Egypt for the transportation of stone used to build the pyramids and other such monuments (1).

The Romans constructed approximately 80,000 km of road, consisting of 29 highways radiating from Rome and further networks in conquered territories, to link their various cities and encampments. These can still be seen in places today. Archaeological and historical investigations have revealed that the structure was 90 to 120 cm in depth and consisted of a granular subbase and an aggregate basecourse, held together with mortar, and a cobbled wearing course. An important feature was the inclusion of side drainage channels which prevented waterlogging. Roman law stated that anybody could use their roads but that upkeep was the responsibility of the inhabitants of the land through which the road passed. Following the demise of the Roman empire these roads all but disintegrated due to the absence of a central authority.

In the 17th century, the French government built about 24,000 km of roads using a system of enforced labour. Around the same time, the English Parliament instituted a franchise system inviting private companies to construct and maintain roads and allowing them to charge tolls for their use. More than 1,000 turnpike companies maintaining 32,000 km of road were in existence in England in the 1830's but the advent of the railways meant that toll roads became under used resulting in the collapse of the toll system.

In the 19th century, two Scotsmen named Telford and Macadam, improved the methods of highway construction. They realized that the most important factor in a good road is an impermeable layer which evenly distributes the load over the subgrade. This describes the road as an engineering structure rather than one which has merely evolved through the ages. Both engineers devised structures with strong load bearing bases of crushed aggregate or cobbles, capped with fine aggregate. No binder was used in these designs.

Up until the appearance of motor vehicles, these types of roads served society well. However, the extra weight of cars and trucks put unanticipated stress on the pavements which rapidly disintegrated due to rutting and potholing, particularly in wet weather. Conversely, during dry weather, dust was a problem from these fast moving vehicles. A method of sealing and firmly binding the aggregates was required. Initially a whole range of oils were tried but eventually it was discovered that Trinidad Lake Asphalt and then coal tar and finally bitumen were the best solutions. These binders were applied by hot spraying to existing surfaces. The process was fairly successful, at least as a stop gap solution.

1.3 MODERN ROADS

Eventually, full depth restructuring of roads was necessary and methods which are still used today were developed. The general structure on which all roads are based is shown in Figure 1-1 (1). The bottom three layers form the pavement foundation on which the more expensive and structurally more significant layers are built. The subgrade is the naturally occurring soil upon which the road is constructed. This layer is sometimes compacted to improve its bearing capacity. The overlying man-made layers serve to protect the subbase upon which the integrity of the entire structure depends. A capping layer may sometimes be placed on top of the subgrade and this is followed by the sub-base which consists of unbound aggregate. The layers above this form the main structural element of the pavement and are often comprised of graded aggregate and binder based on various recipes. The lower load bearing layer is termed the roadbase and this is followed by the basecourse layer or layers. The final layer is the wearing course, which provides the surface texture and ride quality in terms of noise and spray levels.

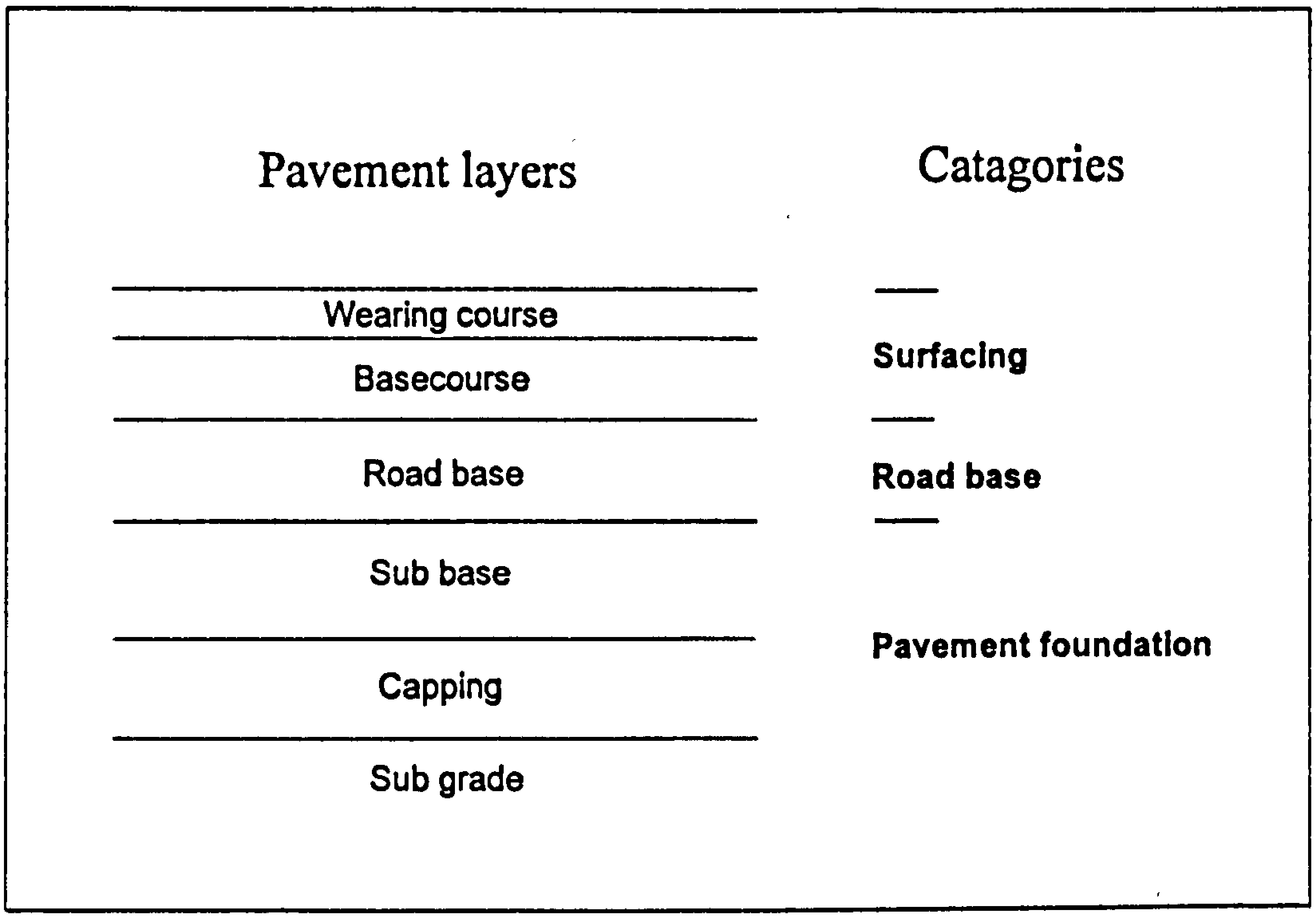


Figure 1-1 : Generalised structure of modern roads

1.3.1 Introduction to pavement mechanics

The purpose of a modern road is to facilitate the flow of traffic in a state of comfort and safety. In recent years the problems of traffic congestion have led to innovations which improve the flow of traffic but pavement reconstruction work itself can have disastrous effects on traffic movement. The objective of modern pavement design, therefore, is to build roads which have predictable lifespans which must be lengthy to minimize the frequency of repair.

Road structures fail through two modes - namely cracking and rutting. The speed with which either of these phenomena occurs is dependent upon the mechanical properties of the asphalt and underlying layers, the thickness of the bituminous layers and traffic volumes. The main purpose of the bituminous layers is to protect the natural underlying surface on which a pavement is built. In order to do this they must have a high stiffness modulus. Stiffness is discussed in detail in chapter 5 and can be likened to Young's modulus - being the ratio of stress over strain. The higher the stiffness of the bituminous layers the greater the distribution of load over the underlying layers. In this context, therefore, mixtures with high stiffness are desirable as they offer more protection to the subgrade. Through analytical pavement design techniques, the engineer can design a pavement structure to provide a specified level of performance based on these factors. This topic is discussed in detail in chapters 5 and 13.

1.4 HOT MIX

The structural layers of modern roads consist of graded aggregate mixtures held together by a binder of some description. In the majority of cases in developed countries, the binder used today in the structural layers is bitumen. Some are constructed with concrete, utilizing cement as the binder, but these are outside the scope of this report. The material most often used to make bitumen bound layers is known as hot mix due to it being made and laid at elevated temperatures. Although bitumens are manufactured to have a range of viscosities, the grades used in the structural layers of roads are almost solid at ambient temperatures. In order to mix with the aggregate mixture, bitumen must first be liquified by heating.

In the manufacturing process, hot bitumen is added to the hot aggregate mixture in a large blender and mixed until full coating of the aggregate is achieved. The bitumen and aggregate temperatures necessary for this are in the region of 110 to 180°C depending on the hardness grade of the bitumen being used. The hot material is then transported to the laying site in insulated lorries and must be laid and compacted while still hot. If the mixture is allowed to cool down too much, the bitumen viscosity becomes too high and the mixture begins to solidify. Below a certain temperature, depending on the grade of bitumen, a mixture will become unworkable, meaning that it can be neither placed nor compacted correctly. After laying, a hot mix pavement can be trafficked as soon as it has cooled to ambient temperature and the bitumen has hardened to regained its original properties.

1.5 BITUMEN EMULSION MIXTURES

An alternative method of liquifying bitumen is to emulsify it in water. In this case no heating of the binder nor aggregate mixture is necessary and mixing and laying are carried out at ambient temperature. This process has been in existence since the early part of the twentieth century and emulsions were actually used as binders for formerly unbound roads.

1.6 CURRENT STATUS OF HOT AND COLD MIX

Bitumen emulsions are used today in a variety of road construction techniques, but mainly only for surface overlays. In most situations, the material used for the structural layers of roads in the UK is hot mix. Several countries use the emulsion based alternative, which has been generically termed "cold mix", in a number of discreet applications but, even in these countries, the majority of paving is hot mix. In the United Kingdom, the road construction industry is only now starting to look to cold mix and lay materials, under pressure from environmentalists and new specifications for reinstatement materials (2).

Cold mix is not utilized in the UK, for example, for a number of reasons. First and foremost, hot mix does an excellent job when manufactured and laid correctly. A great number of hot mix plants exist in developed countries and consequently materials are readily available. Hot mix technology is well developed even though this is based on empirical laboratory and field data rather than fundamental principles. Engineers and contractors have a great deal of experience with hot mix materials, enabling them to have confidence in the performance of a structure comprised of hot mix. Until recently the overall opinion in the industry was therefore one of "If the clock is not broken, don't mend it". Even advocates of cold mix would not claim that it is yet ready to be used in all situations in which hot mix is used and, without the use of modifiers, there is still a shortfall in performance in terms of mechanical properties. Cold mix has very little stiffness during its early life and can, therefore, be damaged if trafficked. Emulsion based mixtures also have an inherent susceptibility to water damage, due to the fact that unset bitumen emulsions are partially water soluble and the binder can, therefore, be washed away by rainfall resulting in a very weak material. There are, however, arguments for the use of cold mix and the debate is gaining momentum. Many of the arguments have an environmental basis.

1.6.1 Benefits of using bitumen emulsion mixtures

As aggregates do not have to be dried for use in emulsion mixtures, dust emissions are eliminated. Hot mix can also lead to gaseous emissions, which are potentially harmful to health (3) and the environment, if proper control measures are not employed. Some efforts are currently being made to contain emissions from both mix plant and paver. An example of the unacceptability of fumes from hot mix plants in the UK was recently reported (4). A major quarrying company planned to install a hot mix facility at one of their quarries in South Wales. Local residents collected scientific evidence on the possible impact and used it to oppose the plans. They succeeded in forcing the quarry company to withdraw their proposal. The quarry company did not accede to the residents' claims in relation to health concerns but withdrew plans on the grounds of the visual impact. It is doubtful that the health concerns voiced by the locals were realistic but public perception proved powerful enough in forcing the issue. This kind of pressure is likely to increase in the future, and could prevent new hot mix plant from being commissioned and perhaps even threaten existing facilities close to populated areas. Production of emulsion based mixtures in place of hot mix could offer a solution to the problem.

As well as very large organic molecules, bitumen contains many low molecular weight hydrocarbons and polycyclic aromatic compounds (PAC's) which are vaporised at high temperatures such as those experienced during hot mix production, transport and laying. Hydrocarbons contribute to atmospheric pollution although this is small scale compared to other sources. Hydrogen sulphide is also released from hot bitumen, with some types being worse than others. In the open, this gas is not present at dangerous levels but in bulk storage tanks it can build up to levels at which it becomes hazardous to health.

However, it is the PAC's which are of more concern as those with 3 to 7 fused rings are known or suspected carcinogens. In particular, benzo (a) pyrene and benz (a) anthracene are considered to be powerful carcinogens. However, the concentrations of these chemicals in bitumen are very low (5). A number of studies have been carried out to assess the potentially harmful effects of these compounds on road workers and the general public (6,7,8). Exposure can be in the form of fumes or contact with the skin; the latter applying only to workers. Skin contact is not eliminated by the use of cold processes (cut-back (see pg 31) or emulsions) and in fact is more likely because a cold material is more likely to be handled than one at over 100 °C. The current consensus is that there is little risk from the carcinogenic compounds in bitumen, but the fact that these materials are present at all is still cause for concern. This seems to be supported by the fact that manufacturers of mix production and laying plant are still attempting to develop systems to prevent the release of fumes. These emissions can be eliminated completely by the use of bitumen emulsion mixtures, as bitumen is only heated during the emulsification process and here it is very easy to contain harmful gases.

Energy and, therefore, cost savings can also be realized through the use of cold mixtures. Hot mix production requires that the binder is heated to temperatures in excess of 140 °C (9). Aggregate mixtures must also be dried and heated to similar temperatures. In the case of cold mix, the emulsification process requires energy to heat the bitumen and drive the emulsion mill, but once made, the emulsion can be used cold and no drying nor heating of the aggregate mixture is necessary. As hot mix plants are more complicated than cold mix versions, they are not normally considered to be portable. This means that transport costs are generally higher for hot mix as both raw materials and finished product have to be carried over longer distances. A number of reports have shown that cold mix uses about half the energy of hot mix on a tonne for tonne basis (10, 11,12). Additionally, investment in cold mix plant is far lower than in the more complicated hot mix plant which, although it is not an important factor in countries with an established network of hot mix facilities, is particularly advantageous in developing countries.

An emerging problem in certain countries is being solved by the use of emulsion mixtures. Old tar containing roads are releasing harmful polycyclic aromatic compounds into the environment due to water leaching (13). Coupled with the fact that these roads are in need of repair, there is a need to carry out reconstruction work using methods which do not allow the pollutants to enter the environment. This subject is dealt with in detail in chapter 3.

Emulsion mixtures also offer potential improvements in performance. Hardening of binder through oxidation and other processes, which can occur during the heating process (14) and can lead to embrittlement and cracking, is avoided. Polymers in latex form can be easily incorporated which is not possible with hot mix, but the effect of these materials on cold mix has not yet been tested.

Finally, cold mix has logistical advantages over hot mix, in that it can be stockpiled or transported over longer distances and it is not necessary to use insulated trucks for shorter journeys. It is, therefore, very useful for small scale reinstatement work or for projects in remote areas.

1.7 OBJECTIVES OF THE RESEARCH

As has already been discussed, cold mix has been utilized in various forms in various regions for a number of years. It is, however, still applicable only for specific environments and types of roads. It is still not possible to use it in all situations due the low structural competence of the material in early life and its lack of resistance to water damage compared with hot mix (15,16). The ultimate objective of the research was, therefore, to develop improved cold mix which could be used in place of hot mix in any situation. The approach taken was to look at the effects of various parameters on mechanical properties of emulsions and mixtures in terms of stiffness modulus, resistance to permanent deformation and resistance to fatigue. Mechanisms for these effects were investigated through studies on the coalescence of emulsion, electrochemical measurements and evaluation of emulsified binders.

Mixture designs were limited to dense aggregate gradings and high binder contents (in accordance with BS4987 (9)) and solventless binders. Only one emulsifier was investigated as it was the best available option for dense aggregate gradings. These restrictions meant that some areas of cold mix, such as those which use open graded aggregate mixtures and different emulsifier types or different binder contents, were not covered, but time did not allow every avenue to be investigated. The work was carried out partly at the University of Nottingham and partly at Akzo Nobel's laboratories near Manchester.

This thesis first describes emulsion technology and the principle of the process of emulsion breaking which returns emulsified bitumen to its original form. The current level of technology and developments to these systems is then discussed.

Several field trials are described which helped to define the current level of performance of the mixture type employed in this research. The experimental work consisted of comprehensive testing on the effects of various parameters on the mechanical properties of mixtures. Fundamental tests were then developed and carried out in which the breaking behaviour of emulsion in contact with aggregate, the electrochemical properties of the emulsion and the properties of the binder residue were studied. The penultimate chapter uses the results obtained to discuss the practical implications of the use of bitumen emulsion mixtures in major road construction in terms of mixture and pavement design. Finally, overall conclusions are drawn regarding the performance of cold mix and possible mechanisms through which various parameters affect performance.

The approach taken in this research is summarised in the flow chart in Figure 1-2.

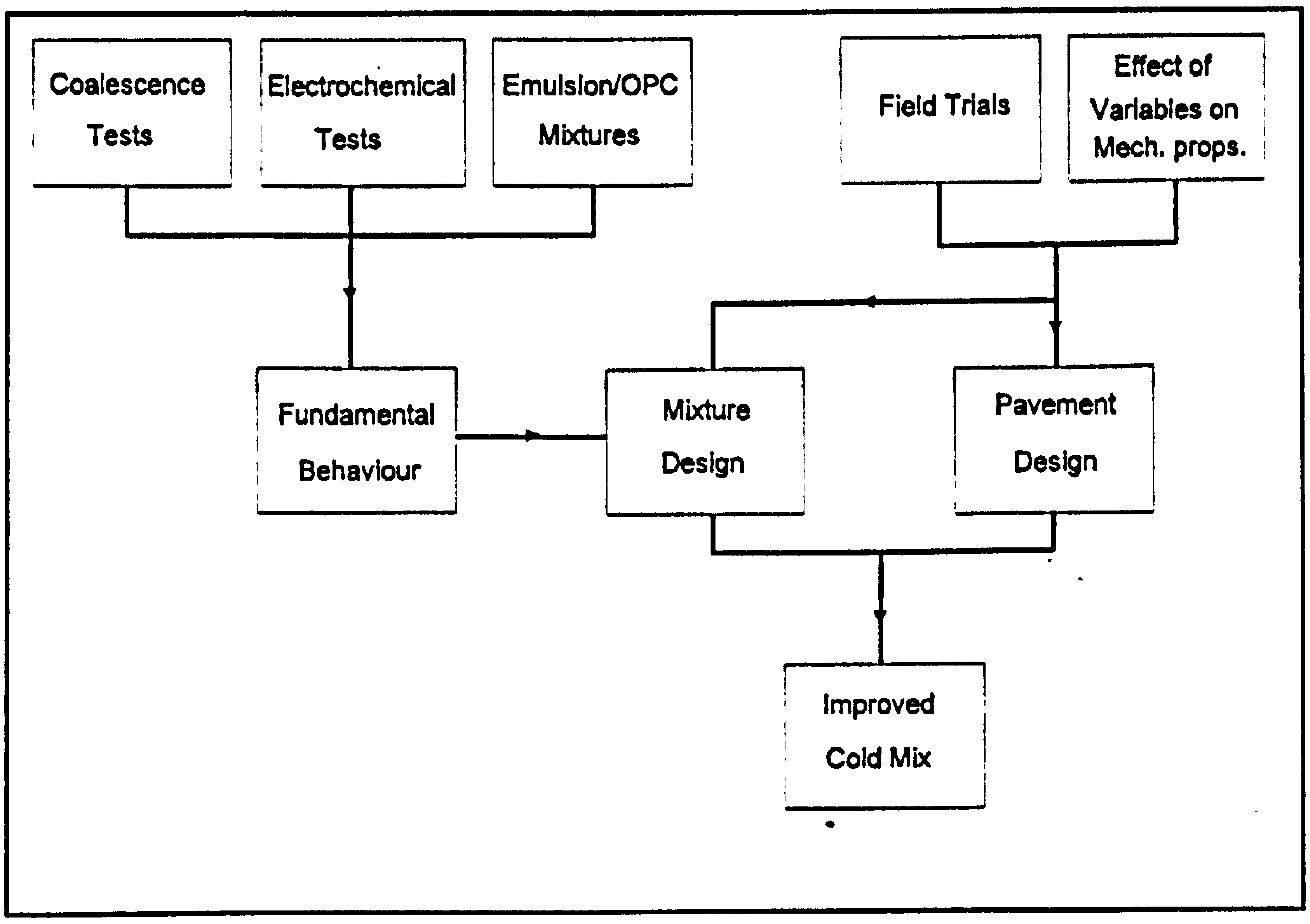


Figure 1-2 : Flow chart to illustrate approach of studies

CHAPTER 2

MAKING, BREAKING AND USING BITUMEN EMULSIONS

2.1 INTRODUCTION

An emulsion is a dispersion of fine droplets of one liquid in another liquid. In contrast to solutions, the two liquids are coexistent rather than mutually mixed. In the case of a bitumen emulsion, these are bitumen, which is a liquid with a very high viscosity, and water. Normally, in good quality bitumen emulsions, the droplets are in the order of 1 to 30 μm in diameter with the majority < 1 μm and the largest volume or mass between 5 and 10 μm. The bitumen content is normally in the region of 60 to 70 % but can be as low as 40 % or as high as 80 %. The globules of bitumen are termed the disperse phase, as they are discreet droplets, and the water is the continuous phase in which the droplets are suspended.

2.2 BITUMEN AND WATER

Under normal circumstances, an oil, such as bitumen, and water, are totally immiscible. This is due to the chemistry of the two materials. Bitumen is composed of two groups of organic molecules called asphaltenes and maltenes (17). The system can be regarded as colloidal, with the asphaltenes being dispersed in the maltenes. Asphaltenes are molecules of very high molecular weight, ranging from 1,000 to 100,000, composed of mainly carbon and hydrogen with some nitrogen, sulphur and oxygen. They are amorphous solids which are fairly polar due to the unsaturate (C=C double bond), nitrogen, sulphur and oxygen content. These are dispersed in the maltenes which can be further broken down into three types of molecules; saturates, aromatics and resins. The saturates are non-polar straight chain and branched aliphatic hydrocarbons which make up between 5 and 20 % of the bitumen. The aromatic portion consists of non-polar aliphatic chains and unsaturated rings with molecular weights of 300 to 2,000. This portion constitutes 40 to 60 % of a bitumen and is the medium in which the peptized asphaltenes reside. Resins have a similar chemical composition to the asphaltenes and act as dispersing agents or peptizers for the asphaltenes. The ratio of resins to asphaltenes dictates the physical nature of a bitumen.

Bitumen is a very complex material which is, overall, non-polar, even though it does contain some polar components. The term "non-polar" means that, generally, the electron distributions in the constituent molecules are evenly spread all over the structures and there are, therefore, no areas of charge concentration or deficiency. Conversely, water is a very polar medium consisting of various ionic species, such as H_3O^+, OH^- and H^+, and the polar water molecule itself. The H_2O molecule is polar as it contains oxygen, which is a very electronegative atom, and hydrogen which is very electropositive. This has the overall effect of concentrating the electrons in the molecule around the oxygen atoms, leaving the hydrogen atoms short of electrons as depicted in Figure 2-1. Thus the molecule gains a small negative charge around the oxygen atom and positive charges around the hydrogen atoms. It is then said to be polar.

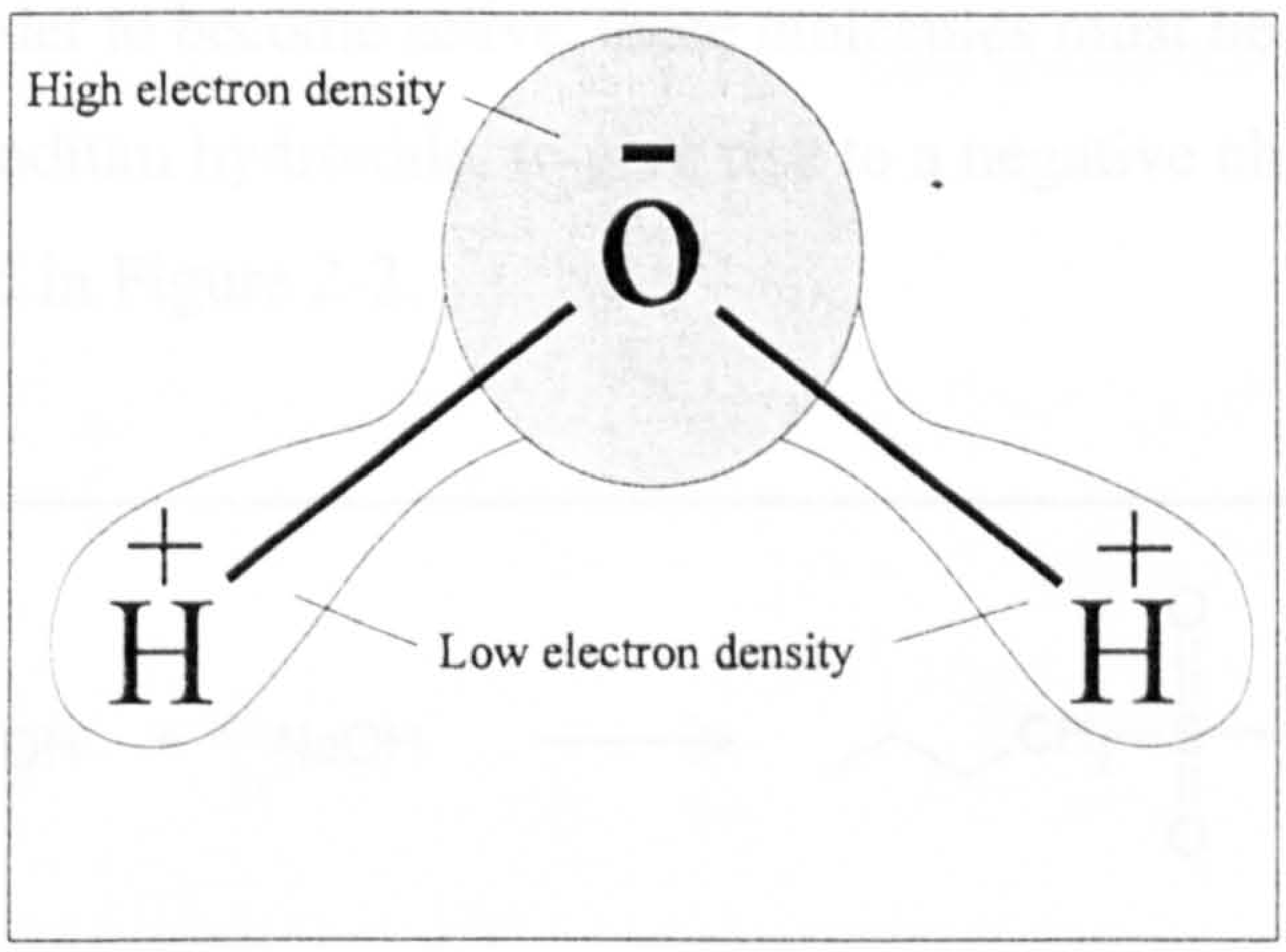


Figure 2-1 : Electron distribution in a water molecule

Under normal circumstances, if an attempt is made to mix polar and non polar compounds, the two substances will separate as the molecules in the polar medium prefer to be in contact with each other in order to cancel out positive and negative regions of charge.

2.3 EMULSIFIERS

In order to produce a stable homogeneous mix of these two immiscible materials a surface active agent or surfactant is required. These molecules are so called because they concentrate and are active at the surface between two immiscible substances in contact (18, 19). This could be a solid and a liquid, a gas and liquid or, as in the case of emulsions, a liquid and another liquid. Surface activity arises due to these molecules consisting of two portions with different properties. One section is composed of a hydrocarbon tail, which is hydrophobic (water fearing) or lipophilic (oil loving), and the other either carries a charge or is polar, making it hydrophilic (water loving) or oleophobic (oil fearing). There are three classes of surfactant - anionic, cationic and nonionic.

2.3.1 Types of emulsifiers

Anionic bitumen emulsifiers are normally fatty acids or alkyl sulphates or sulphonates. These types of molecules were first used as emulsifiers during the late 1920's. In order to become active, these molecules must be deprotonated by a base, such as sodium hydroxide, to give rise to a negative charge on the head group as depicted in Figure 2-2.

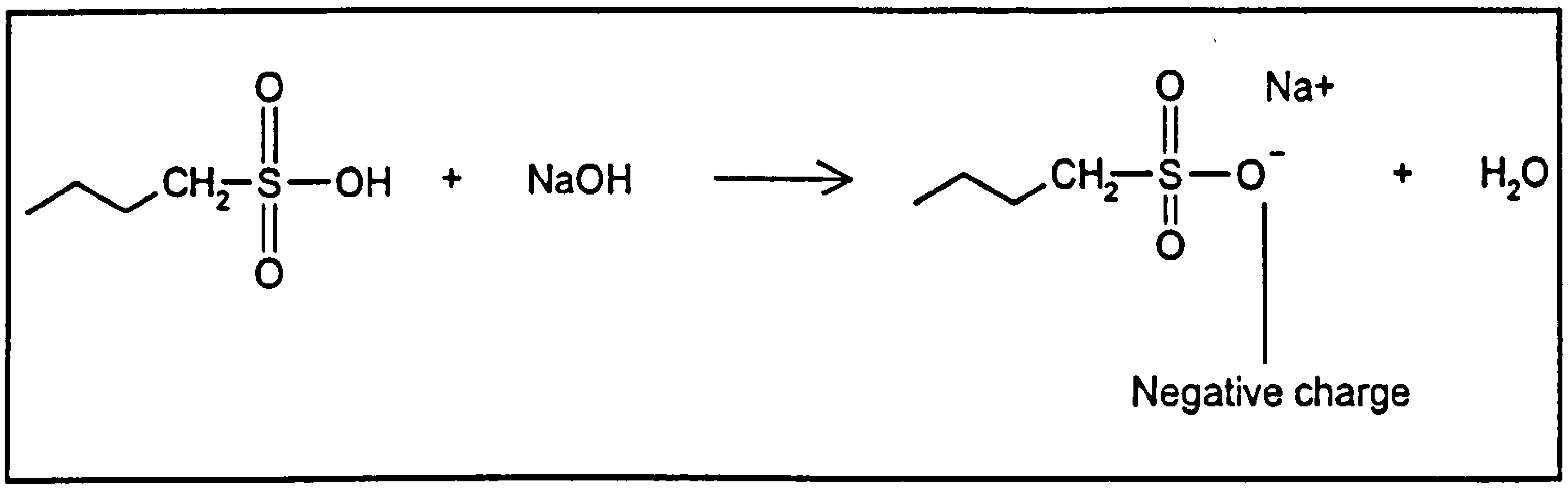
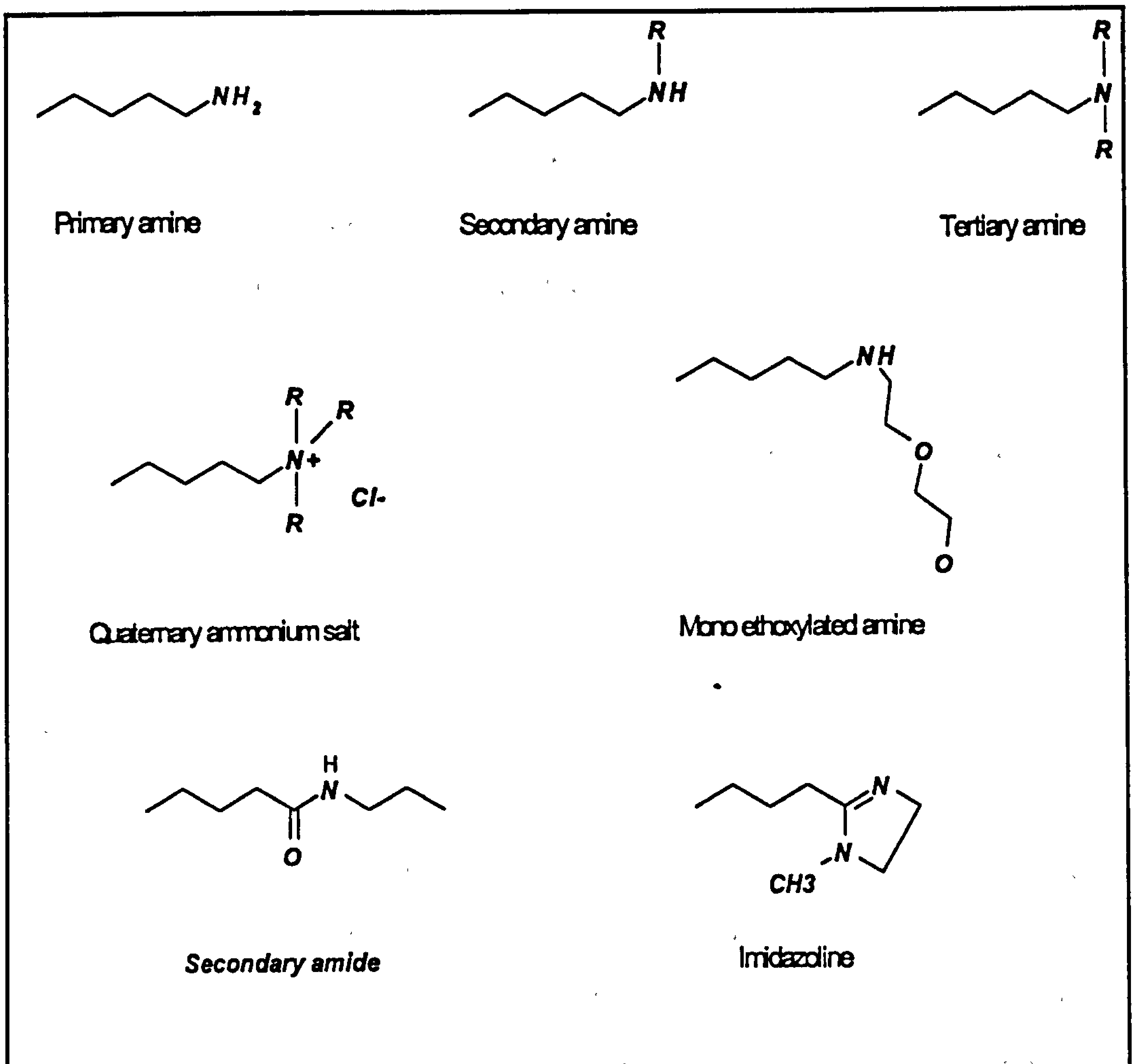


Figure 2-2 : Activation of an alkyl sulphonate emulsifier

The cationic set of emulsifiers is comprised of a range of fatty amines and their derivatives. After first appearing in the 1950's, cationic emulsions have become the favoured type in most road applications. Cationic emulsifiers range from simple primary, secondary or tertiary amines to quaternary amine salts, ethoxylated amines, amides and imidazolines which are shown in Figure 2-3. In order to activate these molecules, the head groups must be protonated by an acid (with the exception of quaternary amines which inherently carry a positive charge) thus gaining a positive charge as shown in Figure 2-4.



R = Methyl group or saturated or unsaturated hydrocarbon chains

Figure 2-3 : General structure of some amines and derivatives used as cationic emulsifiers

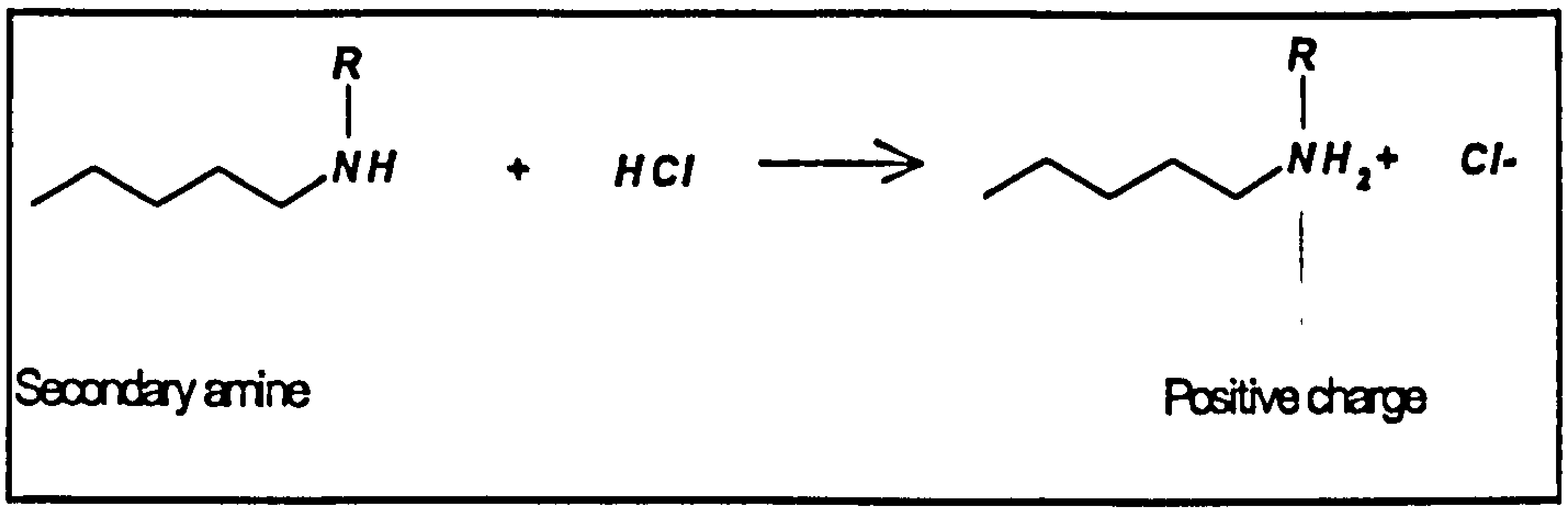


Figure 2-4 : Activation of cationic emulsifier

Nonionic emulsifiers are different from anionic and cationics in that they do not have to be deprotonated or protonated in order to be active. The head groups in these types of molecules are typically but not exclusively chains of ethoxylate groups - C_2H_2O. The situation here is similar to that in H_2O, in that the molecule is polarized as the electrons again become concentrated around the oxygen atoms thus making them into sites of negative charge, leaving the carbon atoms electron deficient and, therefore, positive. An example of a nonionic emulsifier would be ethoxylated nonyl phenol as shown in Figure 2-5.

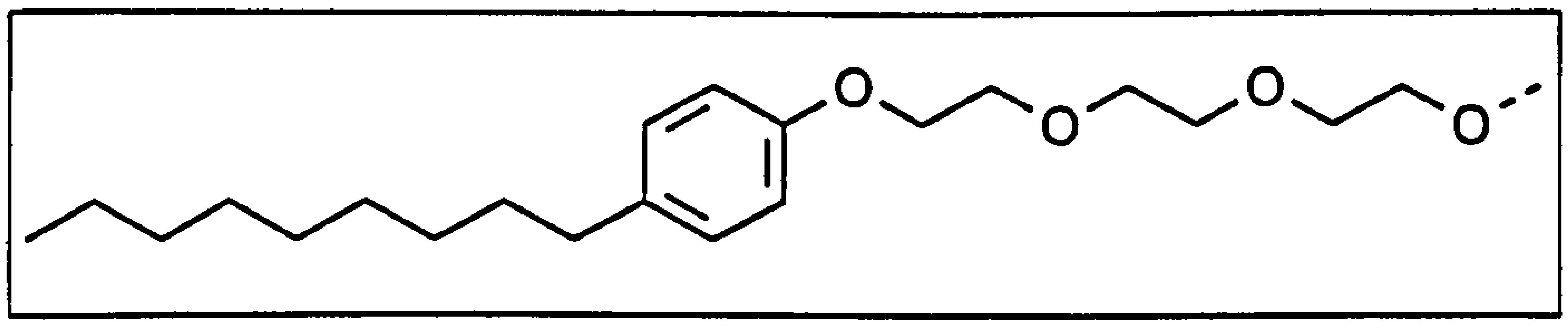


Figure 2-5 : Nonyl phenol ethoxylate

2.4 EMULSIONS

The principle of emulsification is quite simple but the science itself is highly complex. In basic terms, an emulsion consists of globules of one liquid phase dispersed in a second continuous liquid phase. There are two types of aqueous emulsion, namely oil in water (o/w), in which the oil is the disperse phase and water the continuous phase, and water in oil (w/o), where water is the disperse phase and oil is the continuous phase. A bitumen emulsion is an example of an oil in water emulsion, under normal circumstances, but it is postulated that they can invert to water in oil emulsions during the setting process. Setting of bitumen emulsions involves reversion from a dispersion of bitumen in water to pure bitumen and this process is discussed in detail below. The arrangement in a bitumen emulsion, employing a cationic emulsifier, is as shown in Figure 2-6 but obviously the relative size of the emulsifier molecules has been grossly exaggerated for diagrammatic purposes (as they are actually only a few Angstroms in size compared to micron sized bitumen droplets) and they would be much more numerous. The bitumen droplets are suspended in a continuous water phase with the emulsifier residing at the bitumen/water interface, thus stabilizing the system by imparting a charged or polar nature to the droplet surfaces thus making them water miscible. Surfactants also stabilize the system by keeping droplets apart due to charge repulsion or steric effects.

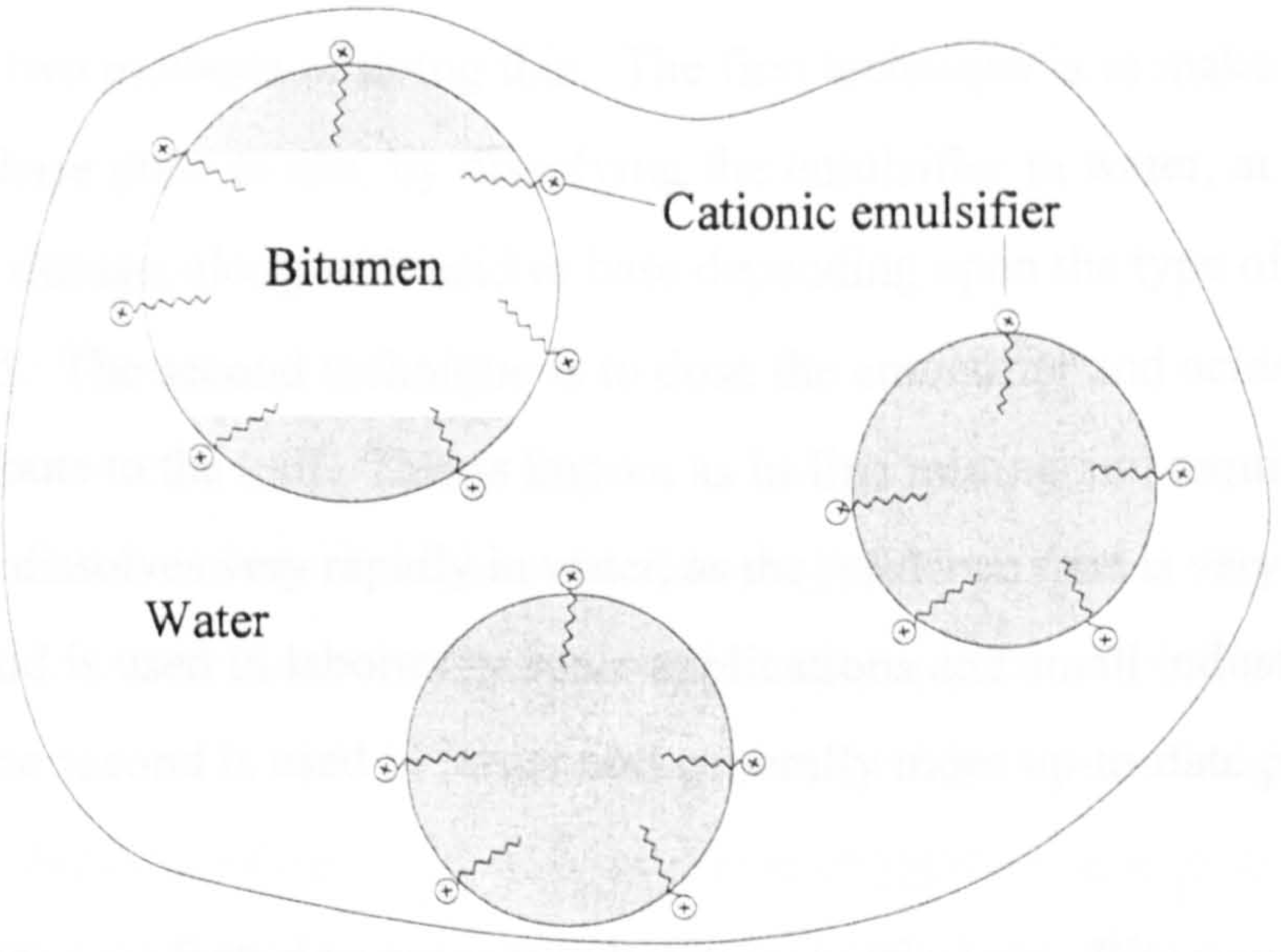


Figure 2-6 : Schematic diagram of a bitumen emulsion

2.5 MANUFACTURE OF BITUMEN EMULSIONS

Bitumen emulsions are manufactured by the intensive mixing together of bitumen and water, dividing up and dispersing the bitumen in the waterphase. The waterphase normally carries the emulsifier in solution in an activated form, ie. acidified or basified. Intensive mixing is achieved by means of a high shear mixer known as a colloid mill which is necessary to finely divide the bitumen. Mill heads come in several forms. The simplest consists of two closely spaced plates, similar to a normal grinding mill, between which the bitumen and waterphase are passed. Another common type consists of two intermeshing sets of circularly placed teeth, one of which rotates with respect to the other. The fixed teeth are termed the stator and the rotating teeth the rotor. A third type of mill, called the SMEP (20), has recently been developed by Esso and does not use a rotating high shear mixing head at all. Instead, the bitumen is dispersed due to intense turbulence created by high pressure injection of bitumen and waterphase into a mixing zone. It is claimed that this method produces an emulsion with an extremely narrow droplet size distribution.

In order for the bitumen and water to mix in the mill, the emulsifier must be added to the system in some manner. It is usually added via the waterphase. There are two methods of doing this. The first technique is to make up batches of waterphase prior to use, by dissolving the emulsifier in water, at the correct calculated dosage, along with acid or base depending upon the type of emulsifier being used. The second technique is to dose the emulsifier and acid/base to the water en-route to the mill. This is known as in-line mixing and requires that the emulsifier dissolves very rapidly in water, as the residence time is very short. The first method is used in laboratory scale applications and small industrial plants, whereas the second is used in larger and generally more up-to-date plants only.

The manufacturing process is batch or continuous. Bitumen must be in a liquid state in order to be pumpable and able to pass through the mill head. This means that it needs to be heated to lower its viscosity sufficiently to allow it to flow; the temperature being dependant upon the penetration grade of the bitumen in question. Penetration is a method of specifying the viscosity of bitumens in simple and easily measurable terms. The test involves measuring the depth to which a standard shaped needle of a specific weight penetrates into a sample of bitumen in a specific period of time (21, 22). The depth of penetration in 1/10 ths mm is the grade of the binder. Obviously, the stiffer the binder the less the needle will penetrate consequently the harder the binder the lower the penetration grade. As an example of the temperature necessary for emulsification, 100 pen bitumen must be heated to approximately 140 to 160 °C. More specifically, the bitumen must be heated to a temperature at which its viscosity is approximately 200 cps. As a rule of thumb, this is about 100 °C above the softening point (temperature at which binder viscosity drops below a certain value) of the binder in question. The waterphase and mill head are also warmed to prevent too much cooling of the bitumen during emulsification which would cause the bitumen to solidify. According to Marchal and Boussad (23), the thermal shock which occurs when hot bitumen meets cooler water, is a cause of the formation of coarse bitumen droplets in an emulsion. Water phase and bitumen are pumped towards the mill head at calculated rates which are controlled to produce an emulsion of the desired bitumen content. Bitumen and waterphase come together just before entering the mill. They then pass through the colloid mill, where the bitumen is finely divided into droplets of predominantly 1 to 5 μm in size, and dispersed in the water. During the mixing process, the surfactant molecules orientate themselves at the bitumen-water interface, thus stabilizing the system as described above. The emulsion then passes out of the mill and is collected. The temperature of the emulsion must be below ~95°C as it leaves the mill in order to prevent boiling off of the water which would push up the bitumen content. If the temperature of the ingredients is too high for this to be possible (eg in the case of polymer modified or oxidised bitumen where the temperature of the binder may have to be in excess of 180 or 200 °C), the mill is pressurised to prevent boiling of the water during emulsification and a heat exchanger is placed on the outlet in order to cool the emulsion before reducing the pressure. Emulsions manufactured on an industrial scale are stored either in tanks (which may be heated in some cases, with agitation provided to prevent settlement of the bitumen droplets) or in transportable vessels such as drums. The diagram in Figure 2-7 is a representation of the emulsification process. The mill head depicted is similar to that found in the Hurrell mill used to manufacture emulsions used in this research. Note that in this mill the rotor is merely a smooth steel wheel and the stator a smooth steel casing around the rotor. The bitumen is dispersed by the turbulence effect created in the waterphase by the rapidly rotating surface of the rotor.

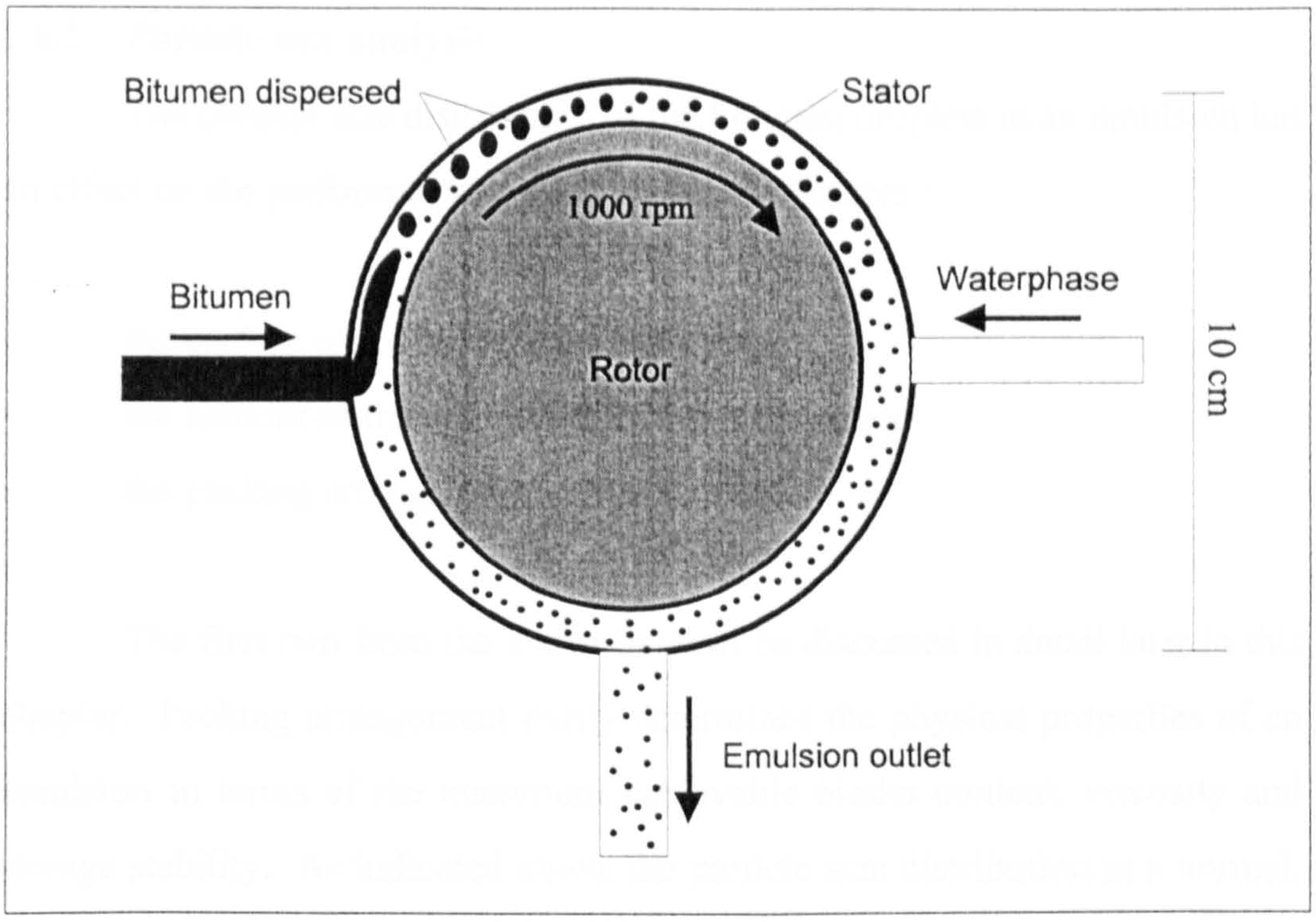


Figure 2-7 : Diagram of emulsification process

2.6 CHARACTERISATION OF BITUMEN EMULSIONS

After manufacture, the quality, and in the case of a novel emulsion, the characteristics, of the emulsion must be checked. A number of standard tests have been developed in order to do this.

2.6.1 Bitumen content analysis

A simple test is normally used to determine the bitumen content of an emulsion (24, 25). The test involves gravimetric measurements to find the percentage weight loss from a sample of emulsion subjected to water evaporation in an oven at ~120°C. Bitumen emulsions are designed to have a range of binder contents necessary to the application for which they are intended and this is covered in detail below. The maximum binder content possible for normal emulsions with spherical or near spherical droplets is ~75 %. Above this level droplet sizes become larger and shape distortion occurs to allow for the high internal phase ratio.

2.6.2 Particle size analysis

The particle size distribution of the bitumen droplets in an emulsion has an effect on the performance of emulsions as it dictates :

- the surface area of the emulsified bitumen
- the amount of free emulsifier in the water phase
- the packing arrangement of the droplets

The first two from the above list will be discussed in detail later in this chapter. Packing arrangement partly determines the physical properties of an emulsion in terms of the maximum achievable binder content, viscosity and storage stability. As indicated above the particle size distribution in a normal, good quality emulsion ranges from sub-micron to a maximum of 30 μm with the majority lying below 1 μm and the largest volume between 5 and 10 μm.

The simplest method of determining particle size is visually by use of a microscope. This method is laborious and probably not as accurate as instrumental methods due to the small number of particles which are sized.

There are two main types of instrument for the measurement of particle size which can be applied to bitumen emulsions. These use the electrozone method, developed by Coulter (25), and laser light scattering. The electrozone method is based on monitoring the flow of current between two electrodes placed in an electrolyte which contains a sample of the particles under assessment. As a particle passes through an orifice positioned between the electrodes, the electrolyte is displaced causing an increase in resistance in the circuit. The size of this increase is proportional to the volume of the particle and thus the dimensions can be calculated. The laser method looks at the diffraction pattern of laser light emerging from a particle dispersion. Through a complicated calculation method, which utilizes the optical properties of the particles under study, the particle size distribution can be found.

2.6.3 Break index

The application of a bitumen emulsion is dependent to a large extent upon its breaking behaviour. Break is the term used to describe the reversion of emulsified bitumen droplets to continuous bitumen. The break index test is used to determine the comparative breaking behaviour of emulsions (26). It involves mixing a 100 g portion of emulsion with a fine aggregate which is normally a quartz sand. Several standard sands or fillers are prescribed in standard test methods. The filler is added to the emulsion in small increments, with stirring for a few seconds after each addition, until the emulsion is seen to break. The mass of aggregate required to break the emulsion is the break index. With rapid setting emulsions, this test is fairly definitive but with slower setting systems the point of emulsion break can be difficult to define accurately. The test does, however, allow emulsions to be ranked as rapid, medium or slow setting quite easily. As a general guide, emulsions with break indices below 80 g are rapid setting and above 120 g are slow setting.

2.6.4 Viscosity

Viscosity or rheology is the flow of a liquid under the action of a force. Viscosity of bitumen emulsions is an important parameter in evaluating bitumen emulsions as it dictates how a material will behave physically in an application. In surface dressing, for example, an emulsion must be viscous enough to remain on the road surface after spraying and not run off. For mixing purposes, emulsions must be of low viscosity to allow them to easily spread over the aggregate during the mixing process.

Measurement of emulsion viscosity is performed on two levels. Most national standards use a flow cup. Flow cups are more or less described by their own name and there are several different versions of the test which differ merely in dimensions and architecture (24). A sample of emulsion is placed in a vessel which has a hole of a specific size in its base. The time required for a certain volume to flow out of the cup is determined and this figure is used to specify viscosity. Several versions of this test exist, each with its own parameters of temperature, hole diameter and collected volume. These tests do not give values with any fundamental basis and merely give comparisons. Rheometers are used to give more information. These instruments will be discussed in detail in Chapter 12.

2.6.5 Sieve residue

The majority of the bitumen droplets in an emulsion are in the order of a few microns in diameter but a small percentage are much coarser. This can arise due to poor dispersion of the bitumen in the mill head or flocculation and coalescence of droplets after manufacture of the emulsion. The sieve residue test is designed to measure the percentage of bitumen above a certain sieve size - normally 150 or 300 μm (24). An emulsion is poured through a sieve and the weight of retained particles determined. The percentage oversize is then calculated. The figures should lie below 0.5 % for a good quality emulsion. High sieve residues can be an indication that an emulsion is unstable. If particle size analysis is available, which can provide results over a wide size range (ie. <1 μm to 600 μm) then the use of a sieve residue test is redundant.

2.6.6 Shake Stability

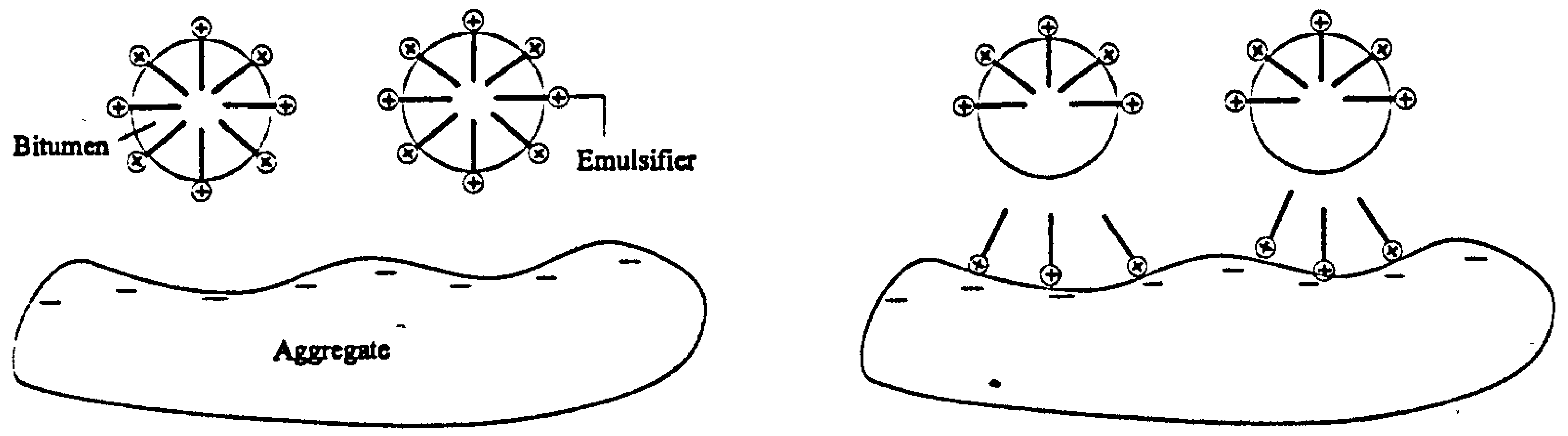
After manufacture, emulsions must be transported to their place of use. Transportation involves a certain amount of agitation of the emulsion and the shake stability test is designed to check a product's transportability. Unstable emulsions can begin to coalesce due to agitation which would have consequences in the field. Tests have been devised in various forms which involve firstly determining the sieve residue of the emulsion. A sample is shaken and then allowed to settle for 2 hours. A sieve residue is then performed. The difference between the sieve residues before and after shaking gives an indication of the emulsion's stability to transport.

2.7 BREAKING OF BITUMEN EMULSIONS

For an emulsion to revert from discreet particles back to continuous bitumen, the droplets must join together or coalesce. The speed of the coalescence process is often termed the breaking or setting rate of an emulsion. The rate at which breaking occurs determines the performance behaviour of an emulsion and, therefore, the application for which it is best suited (27). Applications such as surface dressing, involving the placement of chippings on an emulsion sprayed onto the road surface, require very rapid setting emulsions because the contact area between aggregate and emulsion is very low. Conversely, mixtures of dense graded aggregate and emulsion necessitate the use of slow setting emulsions as the contact area is very large. Applications of bitumen emulsions are discussed in detail below. Mechanisms for the breaking processes of both anionic and cationic emulsions involve an interaction between aggregate and emulsion.

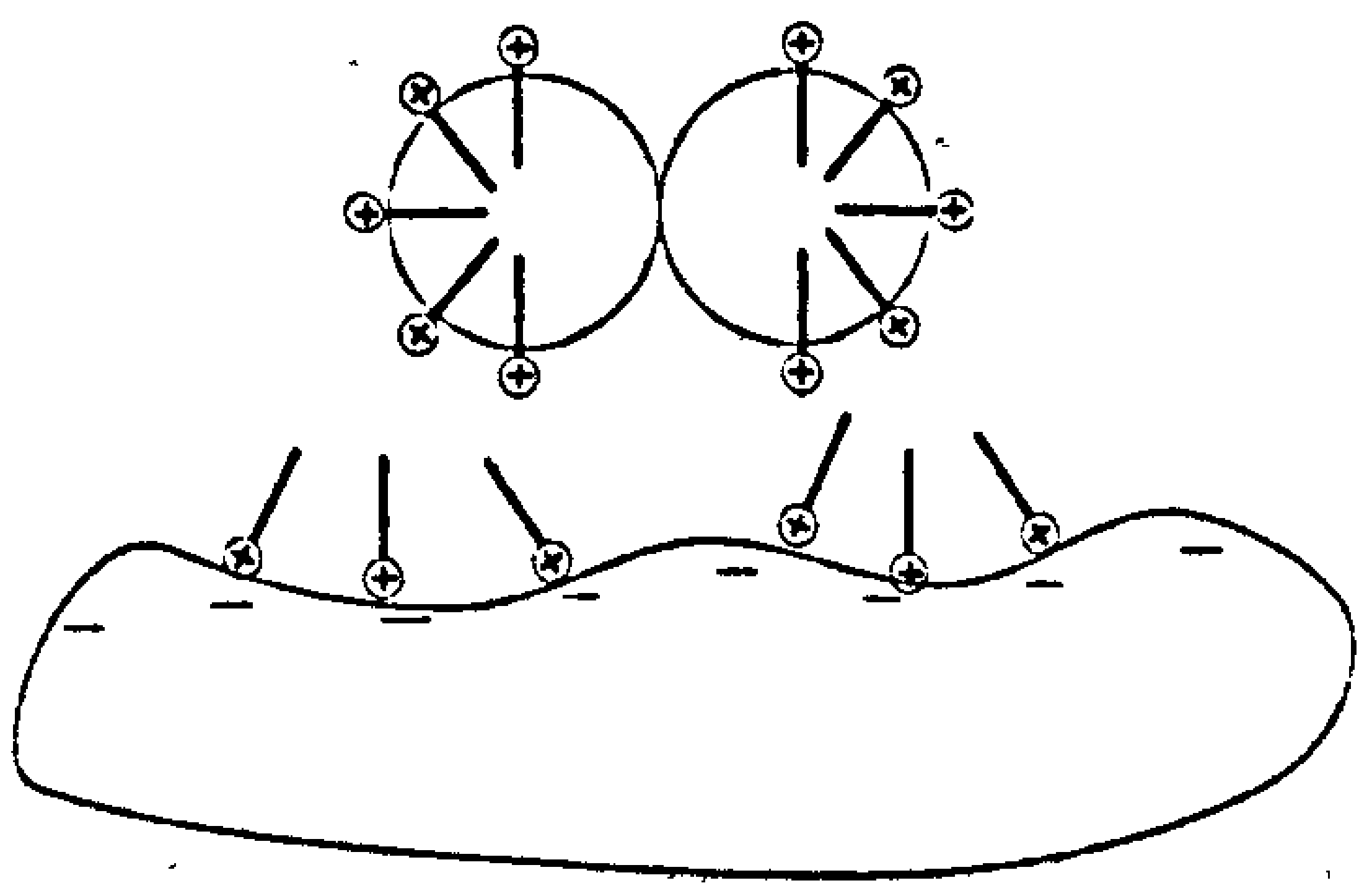
According to Gaestel, there are two breaking mechanisms in the case of anionic emulsions (28). In mixtures with calcareous aggregates, such as limestone which is positively charged, the emulsifier forms a salt with the calcium ions contained in the aggregate thus removing it from the bitumen water interface and causing the emulsion to break. With negative, siliceous aggregates it is the inorganic cation which is adsorbed onto the aggregate surface. Since the emulsifier is deactivated through the loss of the counterion, the emulsion is destabilized and will break. The rate of emulsion break by this mechanism is, therefore, proportional to the concentration of the inorganic cation. The emulsion can be over stabilized by the use of excess base.

According to Wates and James (29), there are three methods by which cationic emulsions are thought to break onto negatively charged, siliceous aggregates. The first is known as emulsifier abstraction. In this case, the emulsifier is withdrawn from the bitumen/water interface by the aggregate surface. The loss of emulsifier causes the emulsion to become unstable leading to coalescence of the droplets (Figure 2-7a). A second process involves deprotonation of the acidified emulsifier, as protons are adsorbed onto basic sites on the aggregate surface, deactivating the emulsifier and thus causing the emulsion to break (Figure 2-7b). The last, and most important process, is termed droplet migration. Here the bitumen droplets are attracted towards the aggregate due to the positively charged emulsifiers on their surfaces. The droplets then spread over the aggregate surface, aided by the surfactant. The final situation is very beneficial as the bitumen is bound to the surface by the surfactant molecules which subsequently act as adhesion or antistripping agents (Figure 2-7c). This is the main reason why cationic emulsifiers have become more widely used than anionics in road construction applications. Probably all of the above processes occur during the breaking of a bitumen emulsion to a greater or lesser extent, depending upon the conditions present in the system.



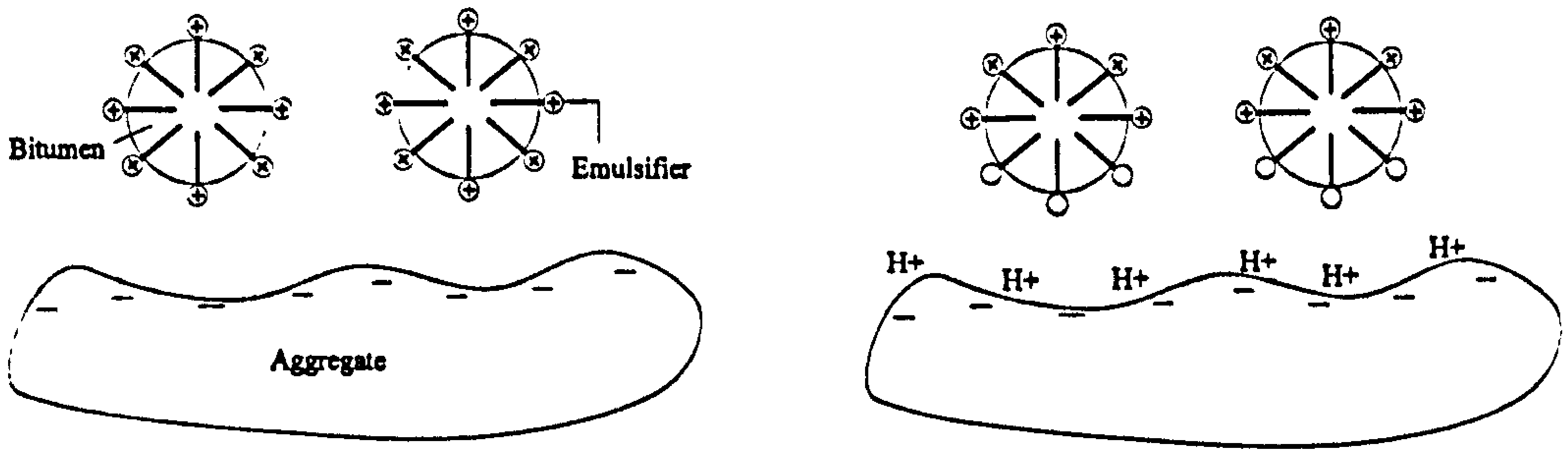
1. Emulsion mixed with aggregate

2. Aggregate abstracts emulsifier from bitumen water interface



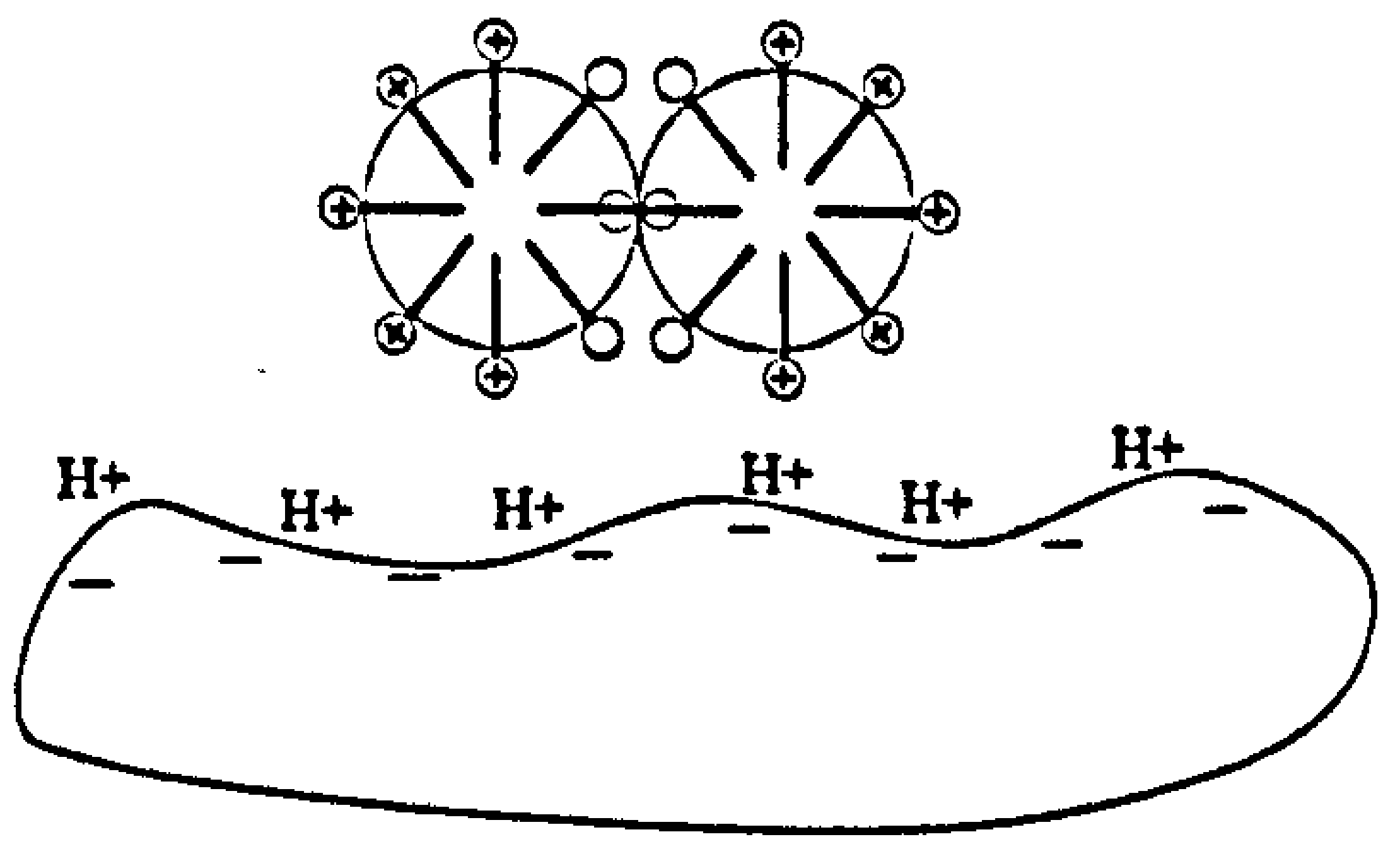
3. Destabilized bitumen droplets begin to coalesce

Figure 2-7a : Breaking mechanisms of bitumen emulsion - Emulsifier abstraction



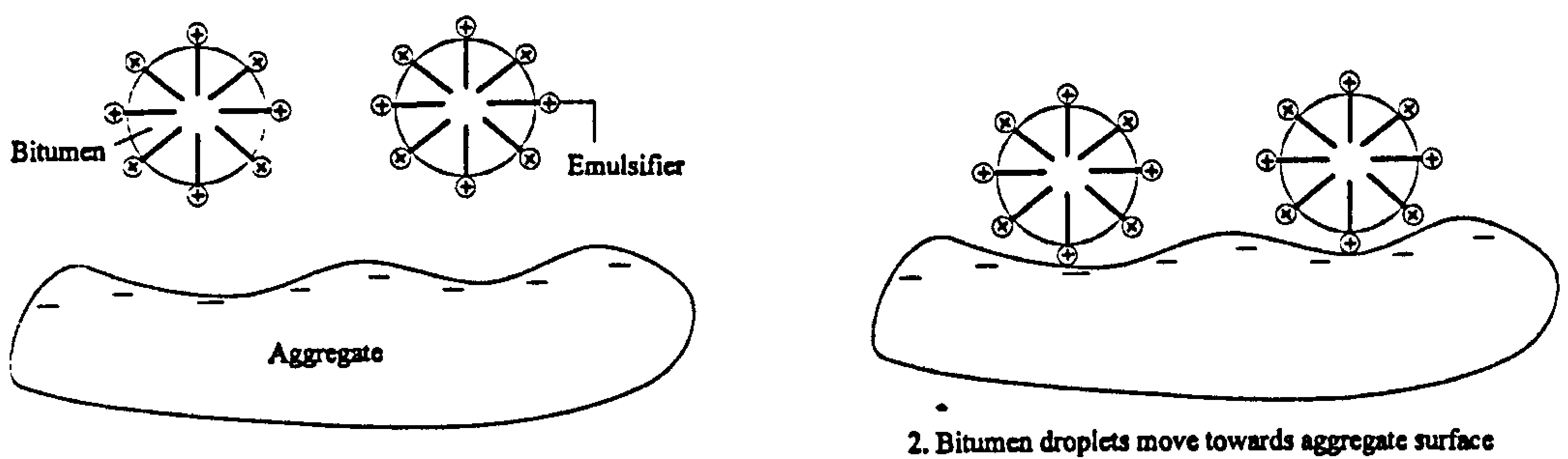
1. Emulsion mixed with aggregate

2. Aggregate abstracts protons from emulsier



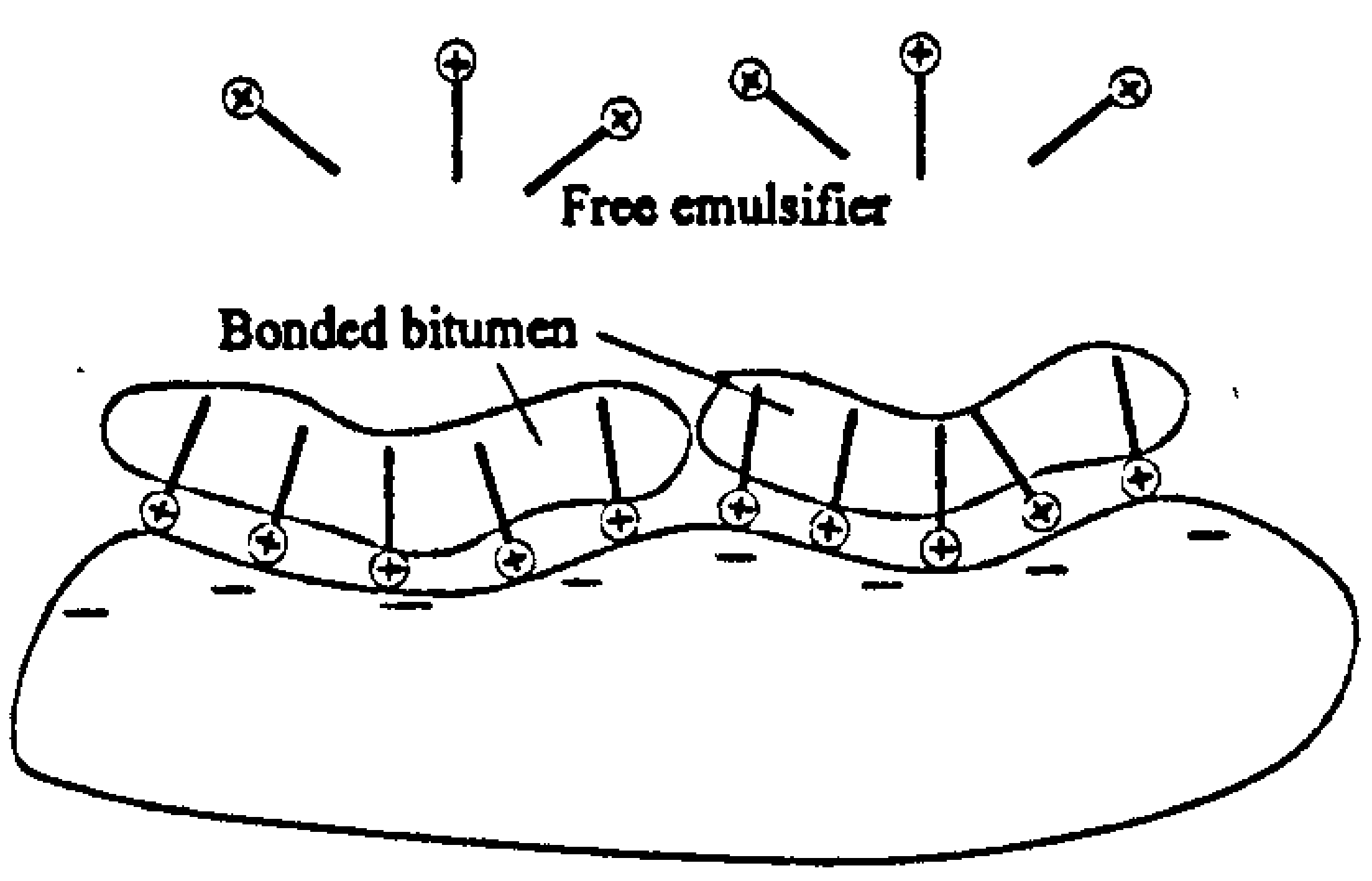
3. Destabilized bitumen droplets begin to coalesce

Figure 2-7b : Bitumen emulsion breaking mechanisms - emulsifier deprotonation



1. Emulsion mixed with aggregate

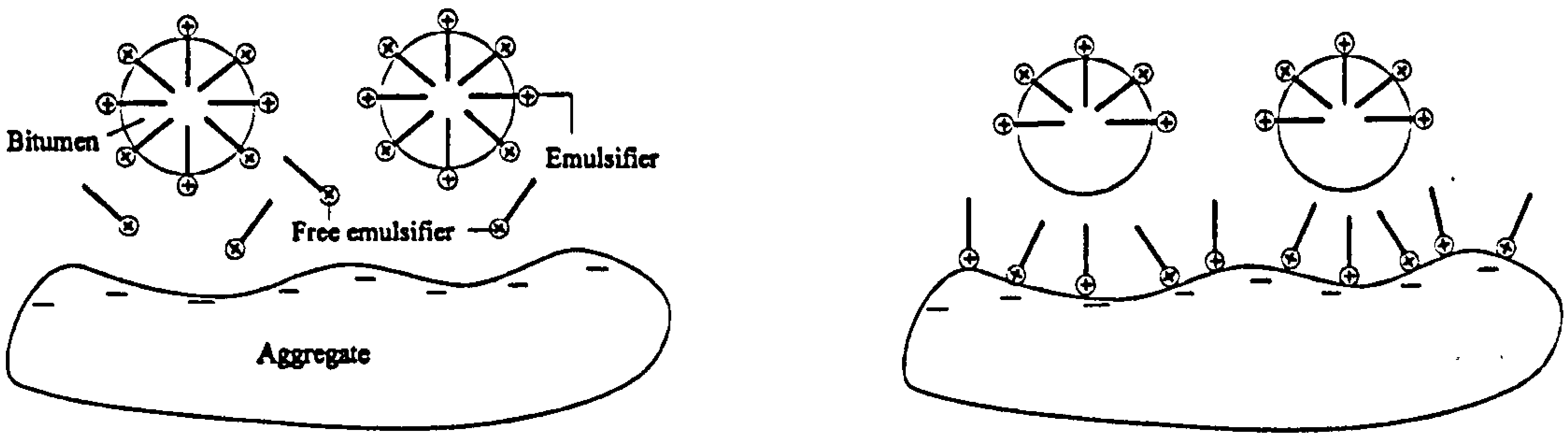
2. Bitumen droplets move towards aggregate surface



3. Bitumen spreads over aggregate aided by emulsifier

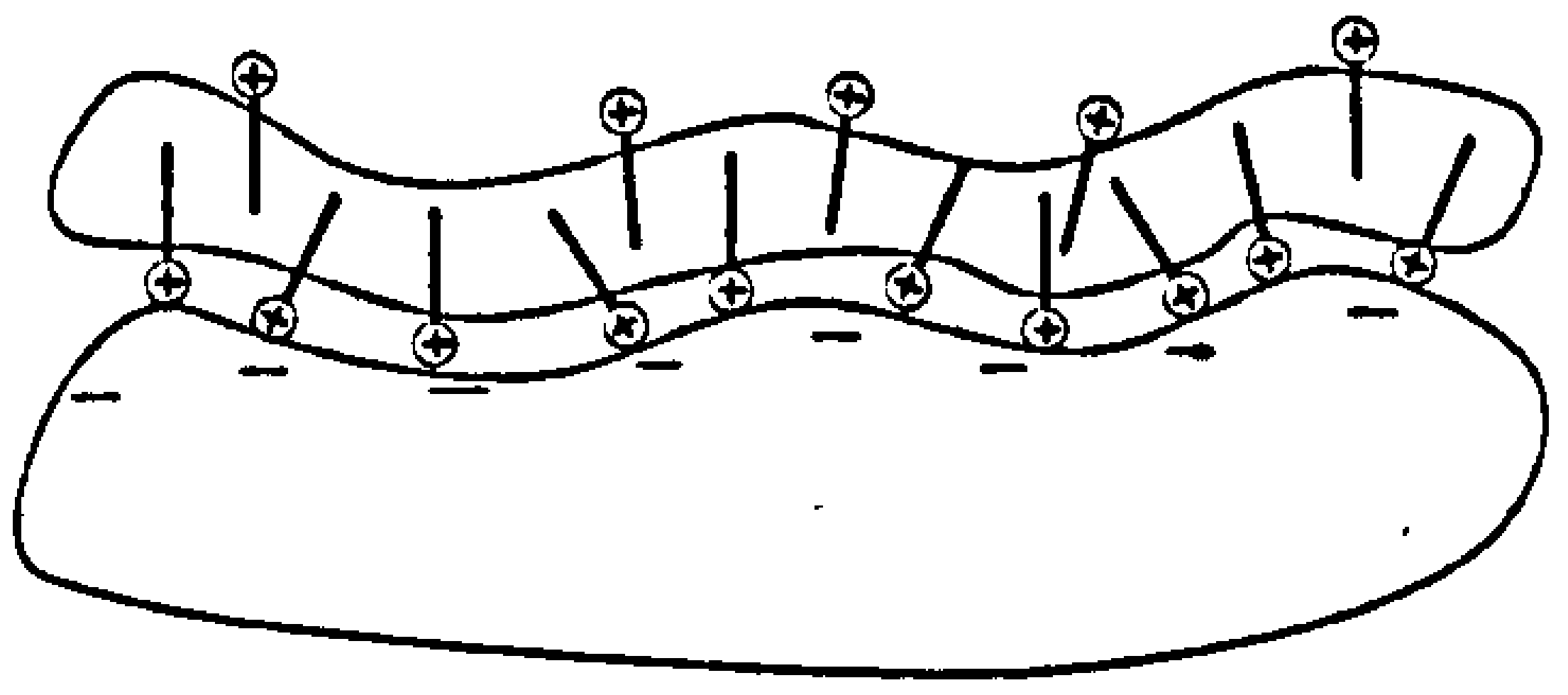
Figure 2-7c : Bitumen emulsion breaking mechanisms - droplet migration

Plotnikova considered the mechanism of breaking to be slightly different (30). It is proposed that firstly the free emulsifier is adsorbed onto the aggregate surface. Next, emulsifier is abstracted from the bitumen droplets and adsorbed onto the aggregate. The bitumen is then attracted to the hydrophobically modified aggregate surface, being hydrophobic itself due to the desorption of emulsifier. This process is illustrated in Figure 2-8. According to this process, the rate of emulsion break would be dependent on the rate of adsorption of the emulsifier onto the aggregate, which is governed by the surface area and chemical nature of the aggregate and emulsified bitumen.



1. Emulsion mixed with aggregate

2. Aggregate adsorbs free emulsifier and abstracts emulsifier from bitumen droplets



3. Bitumen attracted to hydrophobic aggregate surface

Figure 2-8 : Bitumen emulsion breaking mechanisms - emulsifier adsorption

The mechanism by which emulsifier is adsorbed onto the aggregate is thought to be partly chemical and partly physical. Chemi-sorption involves ion exchange between the aggregate and emulsifier. In the case of siliceous aggregates and a cationic emulsion employing a primary amine hydrochloride as the emulsifier, the ion exchange reaction would be :

$$SiOH + RNH_3Cl \text{ ----------> } SiORNH_3 + HCl \qquad (2\text{-}1)$$

Ionic exchange can also occur between cationic emulsions and calcareous aggregates and in fact the ion exchange capacity of these aggregates is ten times larger than that of siliceous materials. The reaction in this case would be :

$$CaCO_3 + 2\,RNH_3Cl \text{ -----------> } (RNH_3)_2CO_3 + CaCl_2 \qquad (2\text{-}2)$$

Plotnikova states that there are insufficient sites available on an aggregate for all of the emulsifier to adsorb chemically and, therefore, physical adsorption must account for the additional adsorption. Physical adsorption arises from the attraction between positively charged cationic emulsifiers and negative sites on the aggregate surface. Studies were conducted on the adsorption rates of anionic and cationic emulsifiers onto wet and dry aggregate surfaces. Anionics were found to adsorb more slowly onto wet aggregates whereas cationics adsorbed more quickly. Wetting of an aggregate surface was therefore seen as a method of controlling the break of anionic mixtures but was not applicable for cationics.

Plotnikova found that doping of the aggregate with quaternary amine prior to the addition of emulsion, slowed down the break of a cationic emulsion. The effect was more pronounced on acidic (negative) aggregates but also occurred on calcareous aggregates which are normally considered to be positively charged. The amine dope can be applied either in solution with pre-wet water or directly during milling of the fine aggregate. The latter was found to be more effective in the laboratory but is not so practical on full scale.

It is more difficult to theorise a mechanism for the breaking of nonionic emulsions as there is no possibility for electrochemical interaction. Breaking of these emulsions, therefore, probably has more to do with the water absorbency of the aggregate or evaporation of water. As discussed above, it is the water medium which keeps the bitumen droplets apart. Thus, if the dispersing phase is removed the bitumen droplets are forced to coalesce. In emulsion mixtures it is, therefore, usually necessary to pre-wet the aggregate before the emulsion is added to prevent rapid absorption of emulsion water which would lead to instant break. Water absorption also plays a part in breaking of ionic emulsions.

2.7.1 Particle size and emulsion breaking

The particle size distribution of an emulsion has an effect on breaking behaviour (31). The finer the emulsion, the larger the surface area at the bitumen water interface. This affects the emulsion behaviour in two ways. Firstly, as with any chemical reaction, an increase in surface area will increase the number of available reaction sites thus speeding up the breaking process. Secondly, the larger the surface area, the greater the capacity for emulsifier adsorption at the bitumen water interface and, conversely, the larger the particle size distribution, the more free emulsifier in the water phase. Logically, if there is more free emulsifier available this will use up the active sites on an aggregate thus slowing down the breaking process according to the mechanism shown above.

These behavioural differences have implications in the adhesion of residual bitumen to aggregate. It can be inferred, and has been proven to some extent (32), that if there is a large amount of free surfactant in an emulsion, this will rapidly attach to the aggregate surface and prevent the binder from breaking onto the aggregate. Instead, the bitumen droplets coalesce away from the aggregate, leading to poor binding of the total mixture. This can result in poor adhesion, resistance to fatigue and ravelling. If, on the other hand, the level of free emulsifier is low, the bitumen droplets will break onto the aggregate thus ensuring that a good binding matrix is formed.

The effect of particle size on emulsion stability should also be taken into account as it works in the opposite direction. In this case the larger the particle size distribution, the less stable the emulsion. Additionally, as suggested by Stoke's equation (2-3), larger particles are more prone to settlement which can lead to a further increase in particle size while the emulsion is in storage.

$$v = \frac{2gr^2(d_1-d_2)}{9\eta} \qquad (2\text{-}3)$$

Where : v = velocity of settlement (ms^{-1})
g = acceleration of gravity (ms^{-2})
r = radius of bitumen droplet (m)
d_1 = specific gravity of the dispersed phase (bitumen)
d_2 = specific gravity of the dispersing phase (water)
η = viscosity of the dispersing phase (water) (mPa.s)

2.8 EMULSION INVERSION

During the breaking process of bitumen emulsions, it is generally thought that at some stage emulsion inversion must occur. The term means that the system inverts from being bitumen dispersed in water to water dispersed in bitumen. As water is lost from an oil in water emulsion, at some point there will be insufficient water to separate the bitumen droplets. Beyond this point, the water will be dispersed in a continuous bitumen phase which constitutes emulsion inversion. It can be appreciated that in this situation it is difficult for water to get out of the mixture as it is sealed in. It seems reasonable to assume that this would have the effect of softening the binder causing a reduction in strength of a mixture containing it.

2.9 CURING OF BITUMEN EMULSIONS

After a bitumen emulsion has coalesced or broken it must fully cure or set for the binder to recover its original physical properties and water resistance. The process seems to be mainly dependent on the evaporation of water but it is not fully understood. Curing is to a large extent dependent upon climatic conditions in the field. Wet and cold conditions have an adverse effect on curing whereas hot, dry conditions have a beneficial effect. Additional rolling can assist in the expulsion of water but only up until a density at which the minimum achievable void content of the mixture has been reached. At this point the excess water merely fills the available air voids and no pressure can be exerted on the water to squeeze it out.

2.10 APPLICATIONS OF BITUMEN EMULSIONS

The range of surfactants available, impart different characteristics to the emulsions they produce. Emulsions with different setting characteristics are necessary for different applications.

2.10.1 Surface dressing

Surface dressing, or chip sealing as it is known in some countries, is a cost effective process used for improving the texture of and sealing small cracks in deteriorating pavement surfaces (33). It also corrects minor irregularities in profile but does not give any structural improvements. A rapid setting cationic emulsion is used for this application (coded K170 in the UK, where K = cationic, 1 = rapid setting, 70 = 70 % bitumen) as the binder must cure quickly to allow early trafficking and sweeping to remove excess chips. The technique involves applying warm (60 °C) bitumen emulsion to the road surface by spraying and then immediately placing single sized aggregate chippings onto the unbroken emulsion. A number of variations on the basic theme have been developed which can involve several layers of emulsion and aggregate. Another process, known as Cape seal due to its place of origin, consists of a surface dressing followed by a slurry seal. A slurry seal is a mixture of fine graded aggregate and emulsion and is discussed in detail below. Whatever the makeup, the dressing is rolled after laying to align the chippings into a good interlocking matrix. As the contact area between emulsion and aggregate is very low in surface dressing procedures in general, the breaking processes described above are minimal. For this reason, the emulsion has to be very easy to break. Thus, very highly charged emulsifiers such as diamines are used as discussed above.

2.10.2 Tack coats

A tack coat is a very light spray of diluted bitumen emulsion used to provide a bond between an old surface and a newly applied layer. It is considered to be good practice to use a tack coat whenever an old surface is overlaid as weathered bitumen can lose its adhesive properties (34). Anionic and cationic slow setting emulsions are used, after dilution with warm water, at an application rate of 0.25 to 0.7 l/m^2. A tack coat emulsion must be able to penetrate any surface cracks or unbound material before breaking, but it must set quickly to allow the new layer to be applied. Tack coats are also used in patching operations. After all loose material has been removed from an excavation, the emulsion is applied to the base and sides. This helps to keep the patch in place and provides a watertight seal between the patch and the surrounding pavement.

2.10.3 Fog seal

A fog seal is similar to a tack coat in composition but it is used as a surface treatment itself rather than an aid to another resurfacing process (34). Fog seals are used on surfaces which have become deficient in binder and are therefore susceptible to loss of chippings or ravelling, or to seal minor cracks. If applied conscientiously and in good time, fog seals can extend the life of a pavement considerably.

2.10.4 Prime coat

A prime coat is an application of bitumen to a granular base in preparation for a bituminous overlay (34). Cut-back bitumen (bitumen diluted with solvent to lower its viscosity) and bitumen emulsion are both used. A prime coat serves several purposes :

- coats and bonds loose particles
- hardens or toughens the surface
- waterproofs the surface
- plugs voids in the aggregate surface
- provides adhesion between the base and overlying course

In order for the prime coat to satisfy these criteria, it must be able to penetrate into the base material. Therefore, slow setting anionic or cationic emulsions are utilized. Until recently, prime coats were considered to be an essential part of good pavement construction, but latterly, they have only been deemed necessary when a granular base is to remain exposed during prolonged periods of adverse weather conditions.

2.10.5 Slurry seal and microsurfacing

Slurry sealing and microsurfacing are two very similar techniques which are used as surface treatments to improve surface texture and correct minor irregularities (34). The only difference between the two is that microsurfacing uses larger aggregate sizes and is used in thicker layers. Unlike surface dressing, these processes can be used on high speed roads as they give a surface with a very even profile with good skid resistance and a secure finish. The level of technology is very highly developed as systems are designed very specifically for certain road conditions, aggregates and climate.

Slurry seal and microsurfacing mixtures comprise finely graded aggregate mixtures, water, cement and bitumen emulsion. There are three different aggregate mixture designs which are used for different applications. The coarsest, type III is used for surface corrections and to impart skid resistance. The second, type II, is slightly finer and is used to correct severe ravelling and loss of binder. The finest mixture, type I, is used for crack filling and pretreatment for hot mix or surface dressing overlays. A slurry seal mixture is produced by first pre-wetting the aggregate mixture with water. This ensures that when the emulsion is added it does not immediately break onto the aggregate due to absorption of the emulsion water into the aggregate. Additive or dope solution may then be added which controls the rate of break of the mixture to a high degree of accuracy when used correctly. This consists of a weak solution of emulsifier which neutralizes the negative sites on the aggregate to some extent thus slowing down the reaction with the emulsion itself. Ordinary portland cement is often added to control the breaking process and improve the setting behaviour and final properties of the seal. The addition levels of dope solution and OPC must be carefully pre-determined and controlled to obtain the desired mixing, breaking and setting rate of the mixture. Finally, the emulsion is added and mixed for a short time. Emulsions used in slurry sealing were traditionally slow to medium setting but in recent years the need for quick set slurries has led to the use of rapid setting emulsions.

Slurry seal can be applied either by hand or a specialized paving machine. The hand method is used on small scale jobs and involves dumping a mixture onto the pavement and spreading it out using squeegees. Slurry seal machines are very sophisticated mix pavers. The mixture components are stored within the machine, mixed together and laid in a continuous process. On today's roads, slurry seals must set within a 1 hour to be acceptable. No rolling is carried out and the seal is merely allowed to cure due to chemical breaking and evaporation of the water.

2.10.6 Bitumen emulsion mixtures - Cold mix

Potentially the largest application area for bitumen emulsions and the subject of the studies reported here, is in place of asphalt mixtures or hot mix. Cold mix technology is discussed in detail in the following chapter.

CHAPTER 3

BITUMEN EMULSION MIXTURES -CURRENT PRACTICE

3.1 INTRODUCTION

Bituminous mixtures consist of graded aggregate bonded together with bitumen which produces a flexible concrete. The material is ideal for pavement structures as it offers a degree of flexibility (which gives excellent riding properties), is cheap and produces tough roads which are resistant to the elements and the action of traffic. Hot mix is currently the principal material used in each of the structural and surfacing layers of roads in the UK and most other countries, from roadbase through basecourse to wearing course.

Many different types of asphalt mixtures are used throughout the world and in 1990 it was reported that there were over 350 recipe based mixture designs currently in use in the UK alone (35). These mixtures have been developed or evolved to accomplish certain requirements in terms of load bearing capacity, durability, surface texture, permeability etc. In recent years, there have been moves to rationalise the approach to mix design on a performance basis. This means that the mechanical properties of the material be fully known and utilized to design a pavement which will withstand predicted traffic levels for the desired period of time. Research has shown that improvements to current practice can enhance mixture performance but implementation of the findings is in its early stages (36, 37).

Bitumen *emulsion* mixtures have been developed in a number of countries with quite varied designs suited to the situations in which they are used. The driving forces behind their integration into the road builders' palette have been covered in the introduction. The emphasis on these factors varies from case to case. For example, in densely populated areas environmental benefits are very important whereas in remote regions logistics would be the main attraction.

The following section aims to introduce the general concept of cold mix and then describe specific types of emulsion mixture technology as they exist and are used today.

3.2 COLD MIX IN GENERAL

Cold mix is currently used in a number of European countries, Scandinavia, the United States of America, Australia and New Zealand, Southern Africa and an increasing number of developing countries. The specific mixtures used in these areas will be covered below but the general principles of manufacture and laying are similar in all cases.

As indicated above, cold mix is manufactured at ambient temperatures, although some processes can use the emulsion warmed to around 60 °C. The aggregate can be used without drying although the water content must be controlled as it has a great effect on the nature of the mixture. The aggregate mixture is fed to a mixing device such as a pug mill, or a rolling drum mixer. Typical layouts for batch and continuous cold mix plants are shown in Figures 3-1 and 3-2 respectively. The main difference between hot and cold mix plants is the absence of heating and drying facilities in the latter, the benefits of which have been discussed above. Pre-wet water is added to surface wet the aggregate to prevent premature break of the emulsion. Bitumen emulsion is then added and mixed until maximum coating is obtained. Care must be taken not to over mix as the input of mechanical energy will cause the emulsion to break further which can lead to a stripped or unworkable mixture.

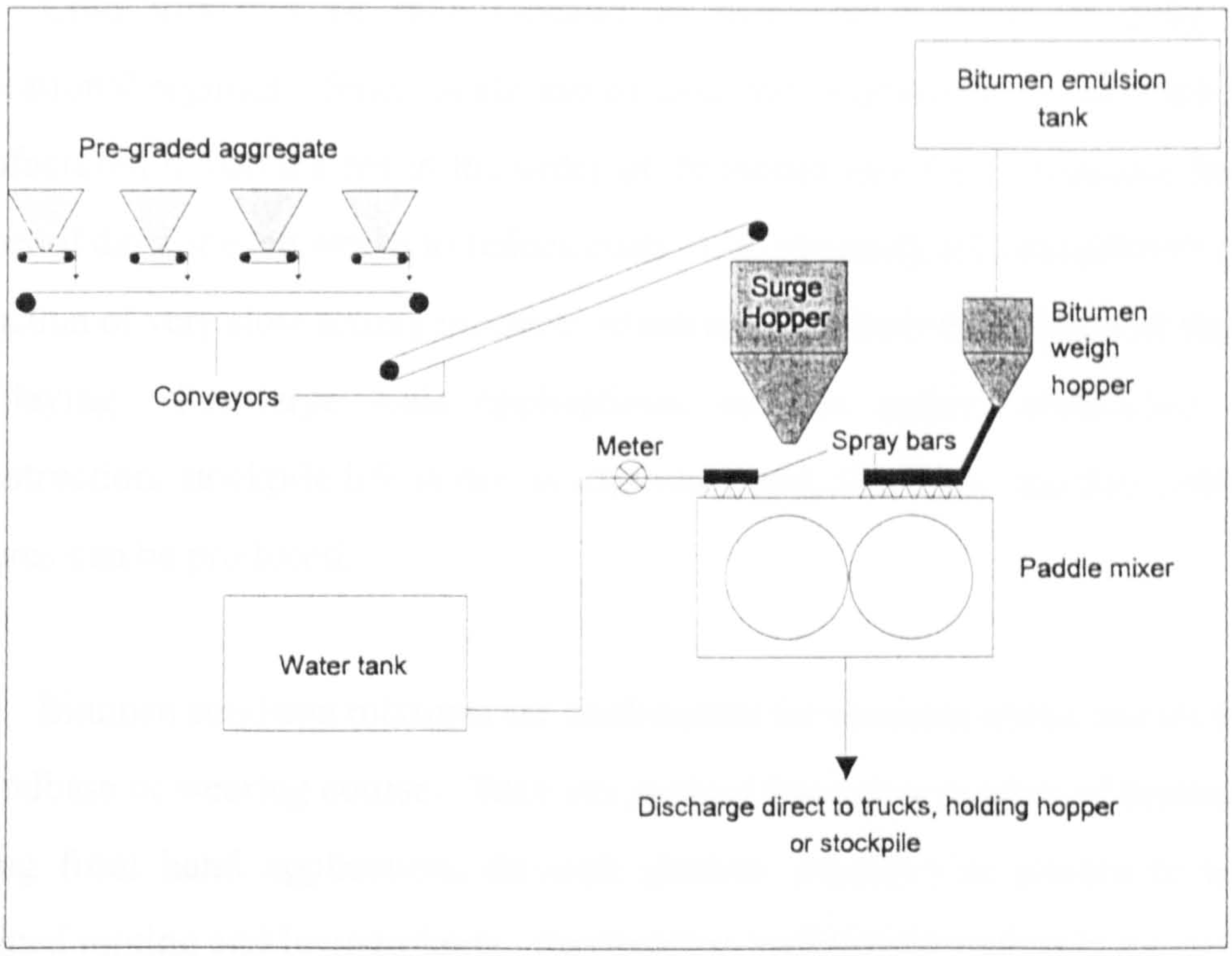


Figure 3-1 : Batch cold mix plant

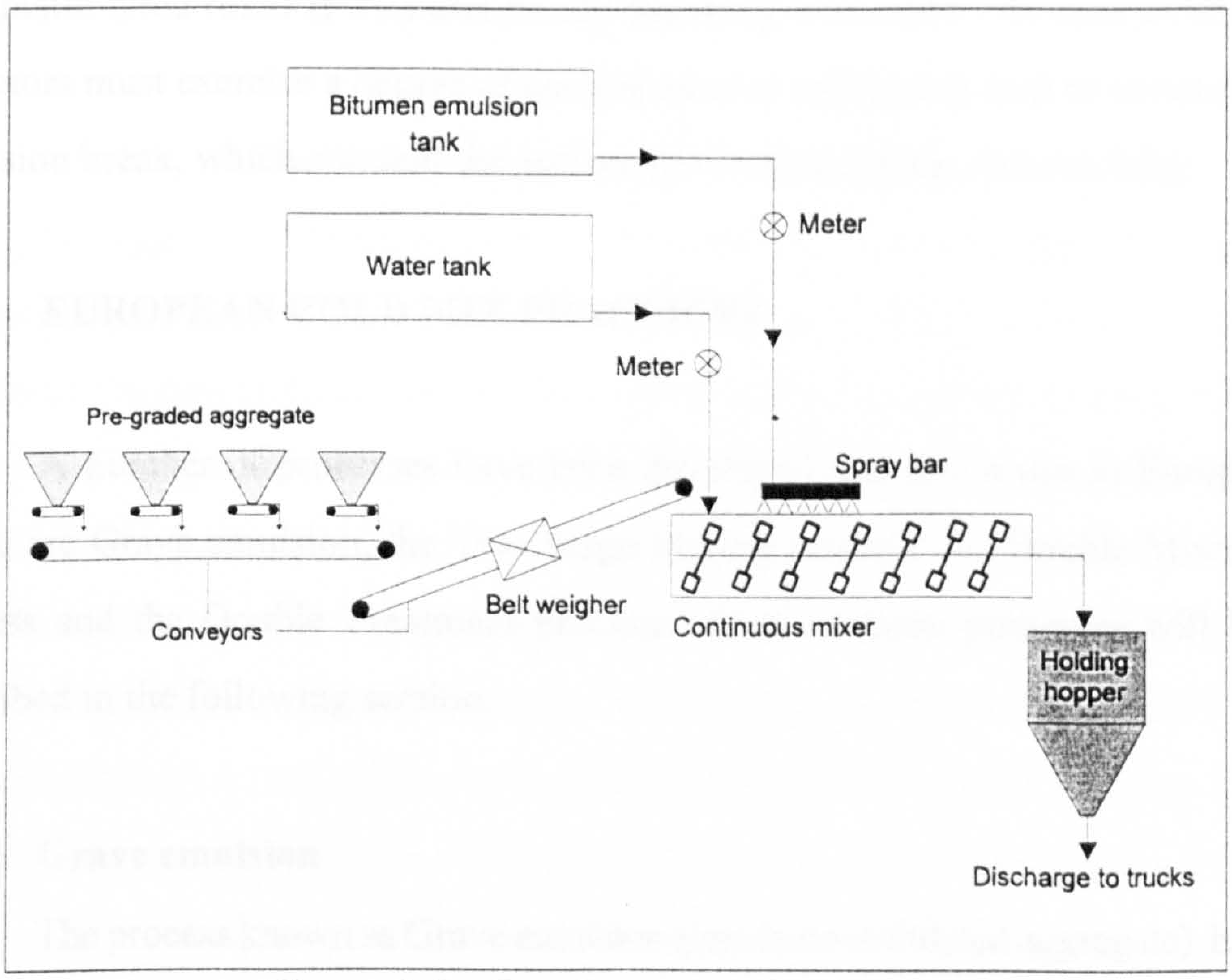


Figure 3-2 : Continuous cold mix plant

Cold mix can be manufactured to cater for a range of different applicational regimes. Small scale use of cold mix requires that a mixture be manufactured in batch sizes in the order of 20 tonnes and then stockpiled for a number of days or even weeks to reduce costs. Unfortunately this necessitates the production of very slow setting mixtures which are very slow to build up strength after laying. For large scale applications, such as major construction or reconstruction, stockpile life is not as important and, therefore, quicker setting mixtures can be produced.

Bitumen emulsion mixtures are used mainly for basecourse and sometimes for roadbase or wearing course. They are applied through a number of methods ranging from hand application, through graders, finishers or pavers to self contained mixing and laying plants. Compaction regimes for cold mix are quite varied at present as different companies advocate and utilize different techniques. However, the preferred method seems to be steel rolling followed by very heavy pneumatic tyred roller (PTR) and finally finishing with steel. As with mixing, operators must exercise a degree of caution as over rolling can lead to excessive emulsion break, which can seal the surface preventing curing, and cracking.

3.3 EUROPEAN COLD MIX PRACTICES

A number of processes have been developed and are in use in Europe. These are Grave emulsion, the Two Stage Mixing process, the Double Mixing process and the Double Treatment process. Each of these processes will be described in the following section.

3.3.1 Grave emulsion

The process known as Grave emulsion (emulsion stabilized aggregate) has been in use in southern France for approximately 40 years (38). It is used extensively as basecourse and for reprofiling, overlaying and strengthening old pavements and construction and reconstruction of lightly trafficked roads. It can also be used for overlaying cement bound basecourse to prevent crack propagation. Being the oldest form of emulsion mixture, at least in Europe, many of the other techniques which will be covered below have some similarities with it, particularly in terms of mixture production and laying methods. Several other countries, notably Spain (39) and Eire (40, 41), have adopted the Grave emulsion technique with little or no alteration.

Grave emulsion was not specified in France until 1974 with the publication of "Instruction for the manufacture of grave-emulsion pavement courses" edited by L.C.P.C. and S.E.T.R.A (42). Grave is not used by all regional authorities in France and it is probably fair to say that it is mainly used in the warmer and drier southern regions due to the water sensitivity of emulsion based mixtures. However, since 1988, quite a large tonnage of Grave emulsion has been laid in the much wetter climate of Eire (40), and such contracts have met with a high degree of success.

Mixture design

The aggregate mixture used in Grave emulsion is densely graded as shown in Figure 3-1. The optimum design has a high sand content to give high internal friction in the aggregate mixture during curing and good surface texture. The latter is often an interim requirement as Grave emulsion layers are nearly always overlaid with a surface dressing to seal the surface. A low filler content is also preferred to minimize the susceptibility to rutting which can occur if the voids are overfilled with a bituminous/filler mastic (43). A maximum void content of 15 % is specified which is very high compared with other densely graded mixtures but is a relic of the presence of water in the mixture initially. Nominal grading sizes are 10mm, 14mm, 20mm and 31.5mm. Most regions are now using the 14mm grade. The aggregate may be either a crushed rock or gravel. Sand is sometimes incorporated to aid compaction. Aggregates must comply with French specifications on durability, angularity and cleanliness.

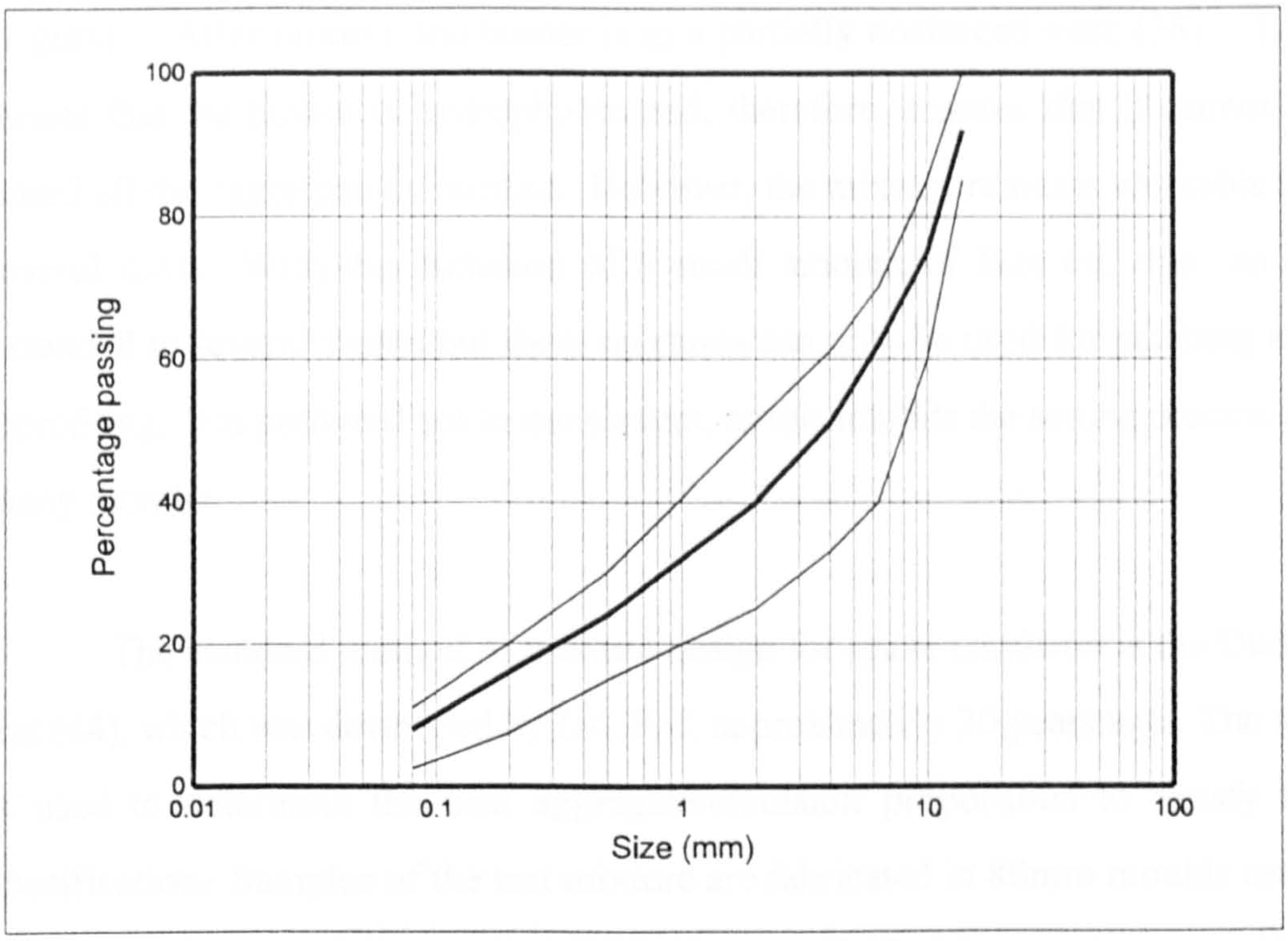


Figure 3-3 : Grave emulsion aggregate mixture design

The most notable feature of Grave emulsion mixtures is the partial coating of the aggregate. The classic binder content is only 3 to 3.5% on the mixture. This gives a Grave mixture a very lean appearance and leaves it potentially susceptible to the ingress of water which is the reason for the normal practice of surface dressing. Low binder contents were used originally in order to promote a high level of aggregate contact to maximise internal friction which resists rutting. In recent years, there has been a move towards higher binder contents of 4% or more, which are not outside the scope of the original French specifications. The specifications have recently been revised to allow even higher residual binder contents to be used.

Emulsions used in Grave mixtures are designed to have a medium setting rate. This only enables the emulsified bitumen to coat the fine aggregate before partial coalescence takes place. The fine coated mastic then acts as a discontinuous cement to bind the larger aggregate matrix together. It is claimed that this property prevents underlying cracks from propagating up through a layer of grave. After mixing, the binder is in a partially coalesced state (38). This means that the binder is hydrophobic and, therefore, ensures that it cannot be rinsed off the aggregate by rainfall. However, the mixture remains workable for several days. With the inclusion of a small amount of flux oil, this can be extended to several weeks but these mixtures can only be used for patching and reprofiling. It is preferred not to use solvent, as this inhibits the setting process for many months.

The standard method of mixture design for grave emulsion is the Duriez test (44), which was developed by L.C.P.C. approximately 30 years ago. The test is used to determine the best aggregate/emulsion proportions to satisfy the specification. Samples of the test mixture are fabricated in 80mm moulds using a compaction load of 12 t applied for 5 minutes. Half of the specimens are then cured at room temperature for 14 days. The other half are cured at room temperature for 7 days and then 7 days under water. The compression strengths of all samples are measured using a hydraulic ram which applies a load at a deformation rate of 1mm/s. The maximum load required to achieve this rate of compression is taken as the compression strength of the mixture. The ratio of wet to dry compression strength is then calculated and taken as the final Duriez test result. To pass the test a material must have a wet to dry strength ratio greater than 0.55 and a minimum compressive strength of 30 bar using 80/120 pen bitumen. A compactivity test is also carried out and a material must have a value above 87 % to be acceptable.

Mixing, laying and compaction

Grave emulsion is mixed in a simple plant similar to that represented in the schematic diagram in Figure 3-2. As Grave is often used in fairly remote regions, the plant is often of the mobile variety which can be located in a quarry near to the road construction site.

Laying of Grave emulsion is normally carried out by use of a grader. Pavers are also used. Both techniques have their merits and drawbacks. Whilst pavers allow very even layers to be laid rapidly, they can lead to segregation of the mixture. Graders, on the other hand, do not cause segregation, but the processes of working and reworking the mixture is slow and can lead to less even profiles.

Compaction of grave emulsion is a very important and well developed factor in the success of the technique. It serves two purposes, namely placing of the material and redistribution of the binder from separate globules to continuous films. Effective densification prevents future settlement or rutting and increases internal friction and cohesion. Compaction can become difficult to carry out effectively due to one or more of the following factors:

- the water content is too low due to mixture design or drying out of the mixture
- coalescence of the emulsion is too far advanced
- the base binder pen is too low
- the ambient temperature is too low
- the bitumen globules are poorly dispersed
- the aggregate is being crushed or the aggregate surface texture is granular

Heavy pneumatic tyred rollers (PTR) are often advised for compaction because their kneading action is very effective in achieving the objectives of densification and redistribution of the bitumen. Vibrating steel wheeled rollers are preferred to dead weight rollers. Care must be taken to achieve an adequate level of compaction without over compacting, as this can lead to surface cracking. As the binder approaches a state of full break, it becomes somewhat dry and crumbly.

This is often referred to as the "cheesy" state. At this point the binder is lacking in cohesion and application of a heavy load can cause the material to break apart rather than stick together.

The final layer in a grave construction is nearly always overlaid with a surface dressing to prevent ravelling by increasing the surface cohesion and thereby resist shearing stresses. As grave mixtures also have a certain amount of porosity, surface treatments are also necessary to prevent the penetration of water.

Grave emulsion can be trafficked immediately after compaction and in fact in some instances, it may even be trafficked during and after grading. This is due to the high internal friction and cohesion which exists in early life of the system. The full strength potential is achieved as a result of water evaporation which is a slow process and is dependent on climactic conditions and partly assisted by the action of traffic. Provided that compaction has been carried out effectively, no deformation should occur during the curing period.

Pavement design

Design of pavement with grave emulsion is carried out in much the same way as with any other material. The design parameters are type and amount of traffic, type and nature of the underlying layer and properties of the new course or courses. Three methods are used:

- experimental, involving deflection or CBR measurements on site
- consult the instruction handbook
- use computerized model

Performance of Grave in-situ

In 1993, Lafon et al. (45) carried out a survey of emulsion based pavement in France which yielded information on the in-situ performance of Grave emulsion. It was found that the aggregate gradings were always dense, having 6 - 10 % < 80 μm, but air void contents were high at a level of 10 - 15 %. The binder contents of mixtures were surprisingly high in the range 4 - 4.5 % and the penetration grade of recovered binders was between 50 and 120. Stiffness moduli ranged from 4,200 to 5,200 MPa tested at 20°C with a load rise time of 120 ms.

3.3.2 Cold mixes

Cold mixes are utilized in a number of European countries (38, 46). There are two classifications based on the storage requirement of the mixtures. Storable mixtures are made with cut-back or fluxed bitumen emulsions. The presence of solvent keeps the binder soft and thus keeps the mix workable. Immediate use or non-storable cold mixes contain non-fluxed emulsions. These mixtures therefore give rise to stiffer mixtures as soon as the water is expelled.

Three types of cold mix, based on aggregate grading are used for repair and maintenance. These are open, semi-dense and dense graded. Open graded mixtures are storable and can be laid with low technology but they are highly porous and easily deformable. Semi dense mixtures have greater cohesion and are less permeable, however when fluxed they are slower to cure. Dense mixtures contain no solvent therefore cure much faster. They have high cohesion and good resistance to deformation due to aggregate interlock. Permeability is also far lower.

Cold mixes are also used for wearing course. They are layable in thicknesses varying from 3 to 22 cm and are designed to have high surface rugosity and allow drainage which can alleviate aquaplaning. If used as a drainage layer, a suitable drainage pipe system must be provided in the basecourse and the road base must be fully sealed. Cold mix wearing course can be used on all but the most highly trafficked roads.

Mixtures can be produced in specialized mixing plant, motor pavers or simple concrete mixers in which case the mixture can be produced on site. For small jobs, laying is carried out by hand and compacted using a small vibrating roller. Reshaping work usually employs a grader but pavers are also used. As the grading used in these mixtures is typically open, layers normally require the application of a sealing coat.

3.3.3 Two-stage mixing

The two-stage mixing process was developed in Sweden and is used on fairly remote roads carrying less than 1500 vehicles per day. It is covered by a specification in the Swedish road manual (47). Both virgin and recycled material can be used with adjustment to the mix design. The basis of the procedure is that coating of the coarse and fine aggregate fractions is carried out in two stages to prevent preferential break of the emulsion onto the fines. The aggregate mixture design for a 16 mm mix is shown in Figure 3-4. The division between coarse and fine aggregate is made at a size of 3 mm.

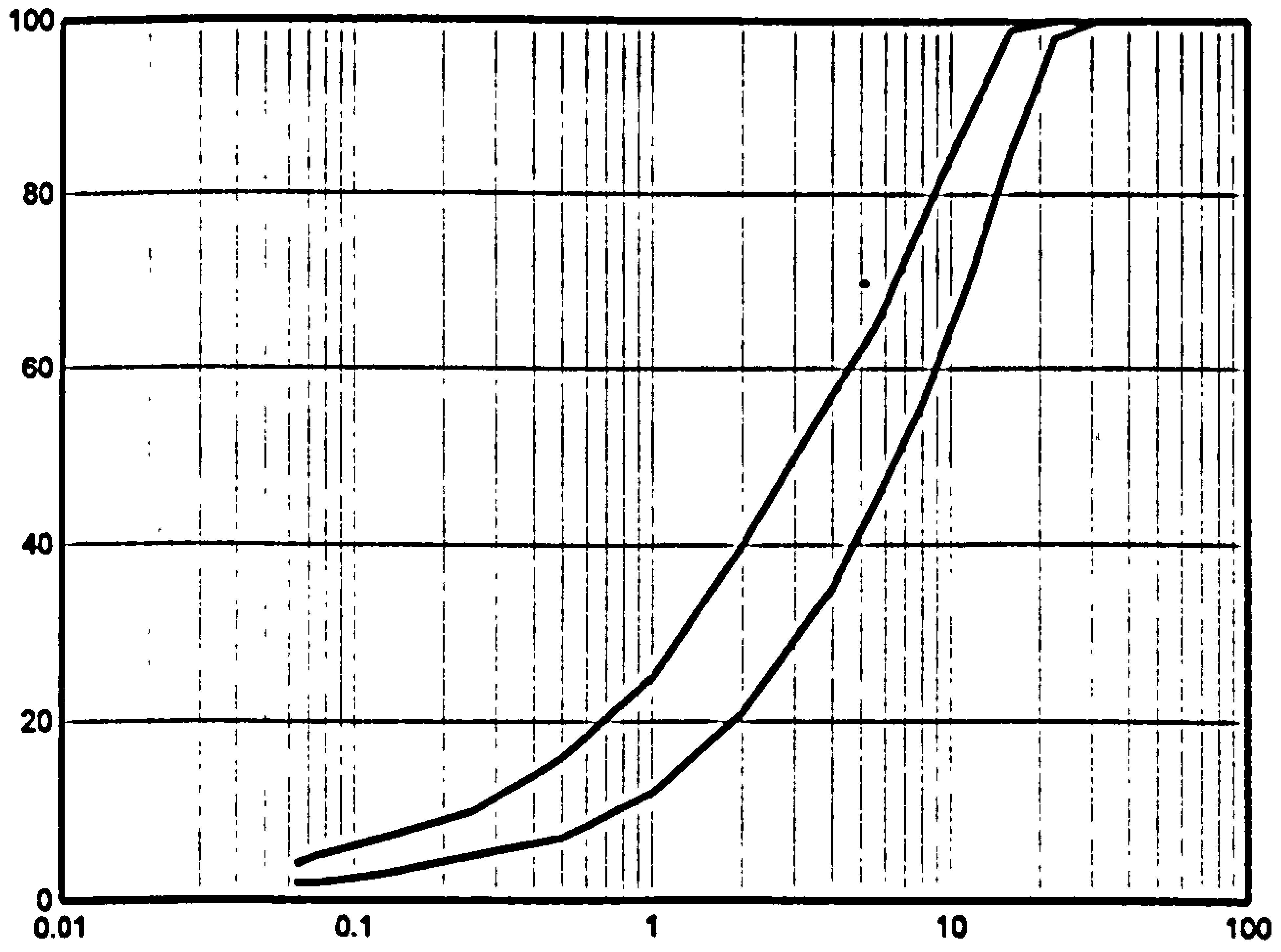


Figure 3-4 : Aggregate grading curve for two-stage mixing

Mixture design

Two stage mixing uses emulsions with very soft binders to avoid low temperature cracking during extremely cold periods, as very heavy logging trucks tend to use these roads. The binder type is expressed in terms of viscosity at 60 °C rather than penetration. Grades range from 2,000 to 10,000 centistokes. For comparison, 100 pen bitumen has a viscosity of 100,000 centistokes at 60 °C which illustrates how soft the binders really are. The soft binders are produced by adding heavy flux oil to normal penetration grade bitumens, and they are often doped with fatty amine based adhesion agents to improve durability. Emulsions are formulated with different levels of emulsifier to give different setting rates. A rapid setting emulsion is used on the coarse fraction and a medium setting version is added with the fines. The reasoning behind this lies in the surface area of the aggregate fractions in that a slower setting emulsion is required to coat the fine aggregate.

Mixing and laying

The mixing procedure itself is continuous and is represented in Figure 3-5. Firstly the 6-12 and 12-16 mm aggregate fractions are charged to the mixer, with pre-wet water being added en-route at a level of <1%. The rapid setting emulsion with the harder bitumen is then added via a spray bar during the mixing process. After ~20 seconds mixing, the pre-wetted fine aggregate fraction is added, shortly followed by the second medium setting emulsion containing the softer binder. Final mixing continues for ~20 seconds after which the completed mixture flows out of the mixer to be collected.

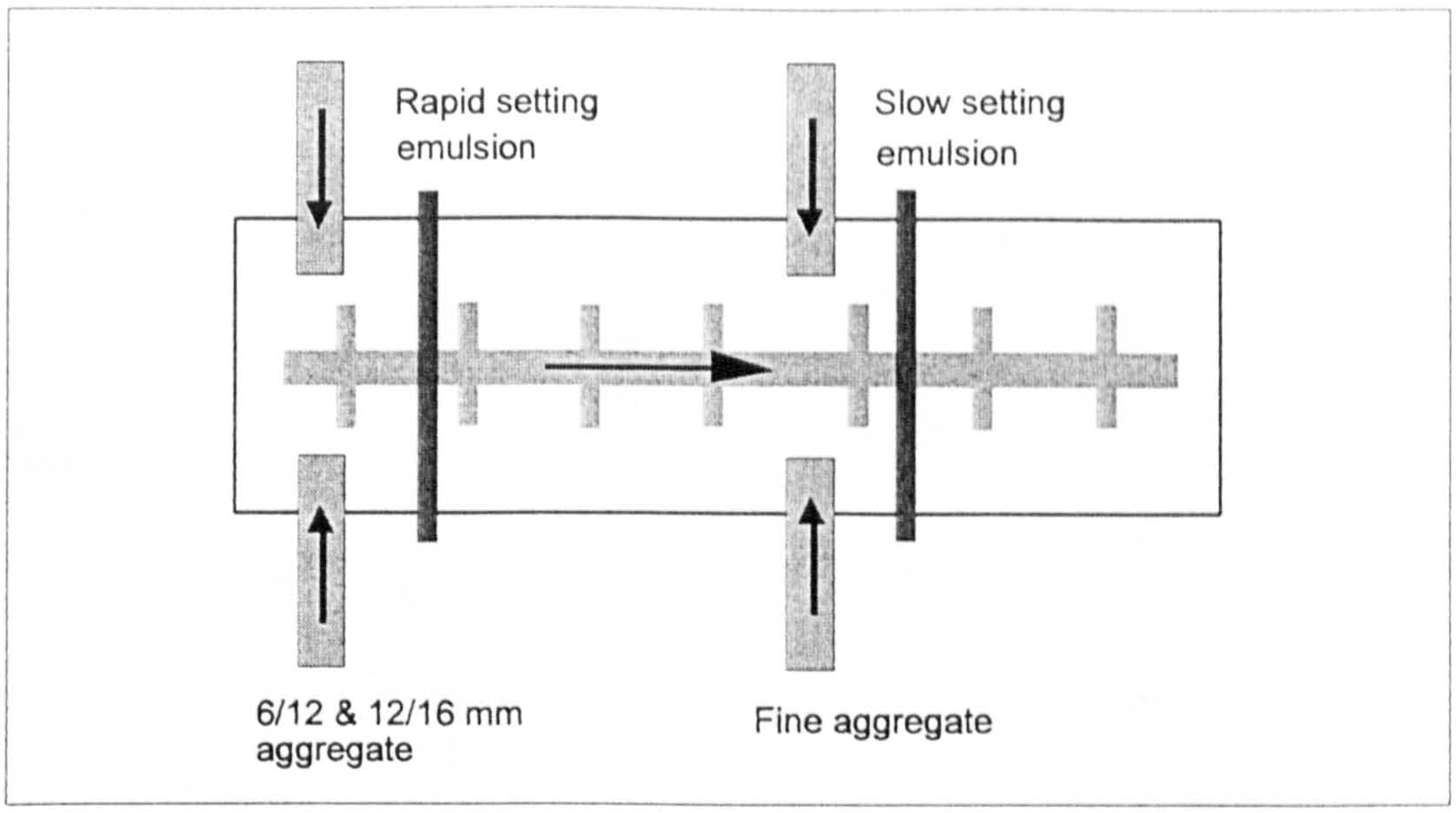


Figure 3-5 : Schematic diagram of two stage mixer

Two stage mixing plants are often mobile and can, therefore, be located near to the area of application. One such example is shown in Figure 3-6. Mixtures are laid using graders or pavers as shown in Figure 3-7.

Figure 3-6 : Two stage mix plant

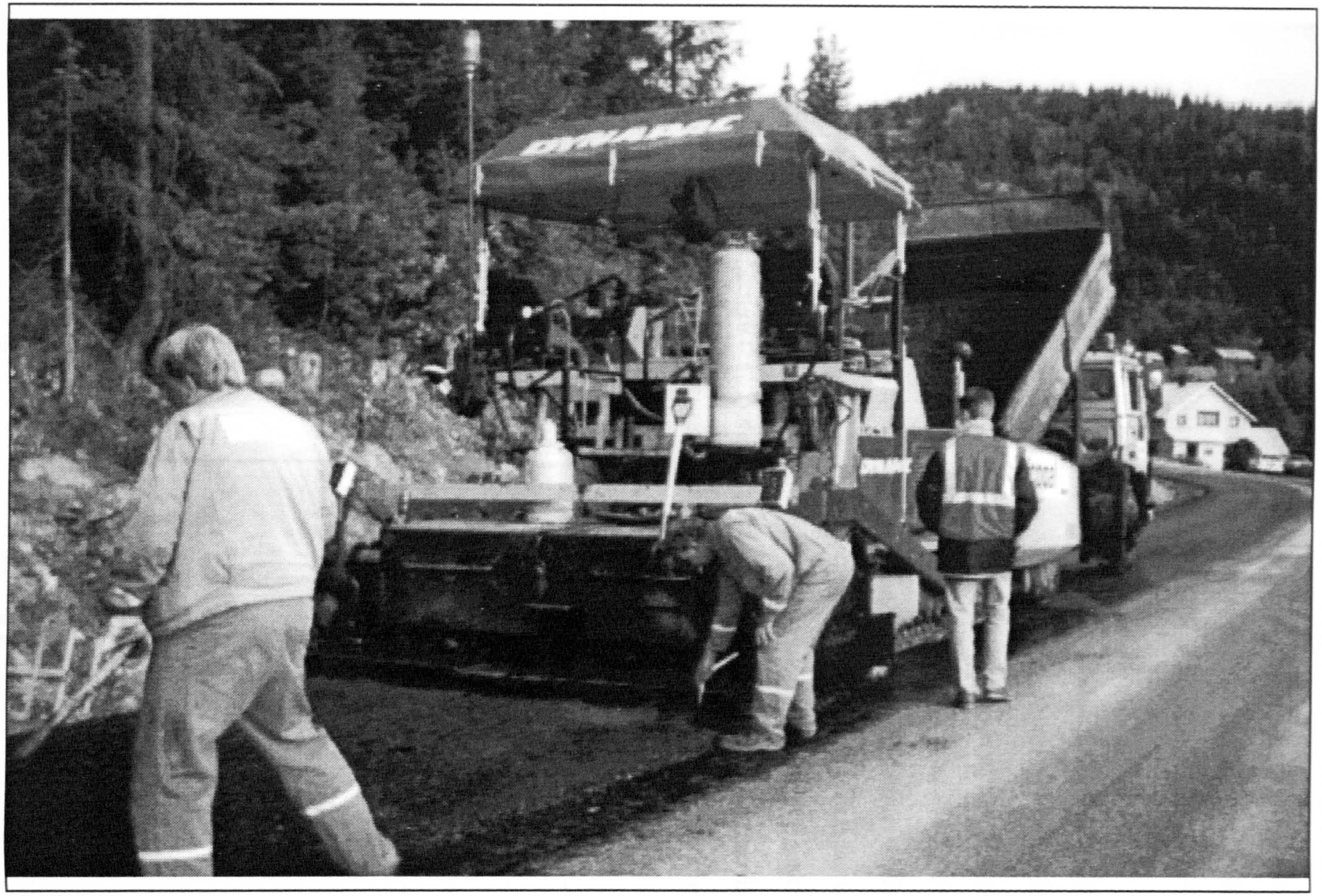


Figure 3-7 : Paving with mixture manufactured using two stage mix process

3.3.4 Double mixing process

The double mixing process was patented by Maheas (48). The method was later adopted and exploited by the French company SCREG and goes under the trade name of COMPOMAC®. It is similar to the two stage mixing process, in that it is designed to avoid preferential coating of the fine aggregate fraction, but it is carried out the opposite way around (49, 50).

A Grave emulsion type aggregate mixture is used with the fine and coarse division being made at the 2 mm size. Figure 3-8 shows the procedure for the double mixing process. Firstly, the fine aggregate fraction is mixed with part of the bitumen emulsion. Alternatively, hot bitumen can also be used at this stage. The mixture is then stockpiled for later use. The second stage involves mixing the stone fraction with the second portion of emulsion and then adding the coated fines. The total residual binder content lies between 5 and 5.8 % which makes this a much richer mixture than normal Grave. It is claimed that the mixture is very uniformly coated which gives rise to benefits in handling and performance characteristics.

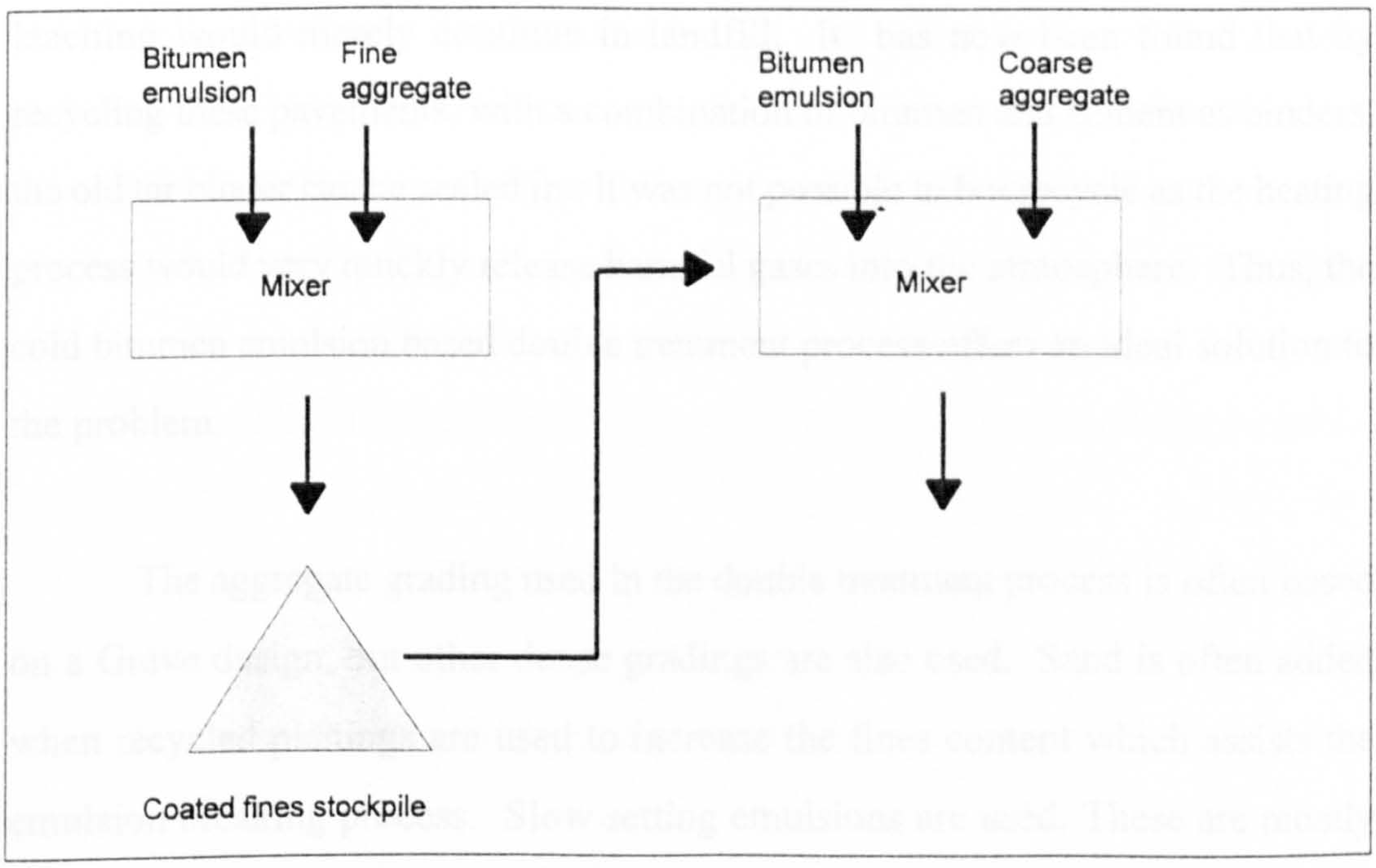


Figure 3-8 : Double mixing process

3.3.5 Double treatment process

The double treatment process of cold mix is utilized in France and Germany and recently in the Benelux countries for basecourse. Over the years it has displaced recycling of bituminous roads using solely cementitious binders. Two French examples of the process are Flexocim® from Beugnet and Stabicol® from Colas. The double treatment process is so called because it uses two binders, namely bitumen and cement (51, 52). The two binders can be added in various orders to both virgin and recycled materials. Due to the nature of the binders, the cured mixture is both hard and flexible. This combination gives rise to great resistance to cracking and also rutting at high temperatures. The increased stiffness means that thinner layers are required in a pavement structure.

Interest from Benelux in this type of mixture has arisen from its ability to seal in old binder when recycled planings from old tar containing roads are used. In the Netherlands and Belgium there are a great number of tar containing roads which are wearing out and will, therefore, have to be repaired. Additionally, it has been found that the harmful polycyclic aromatic components are leaching out, due to rainfall, and entering watercourses. It is not easy to dispose of the planings as leaching would merely continue in landfill. It has now been found that by recycling these pavements, with a combination of bitumen and cement as binders, the old tar binder can be sealed in. It was not possible to hot recycle as the heating process would very quickly release harmful gases into the atmosphere. Thus, the cold bitumen emulsion based double treatment process offers an ideal solution to the problem.

The aggregate grading used in the double treatment process is often based on a Grave design, but other dense gradings are also used. Sand is often added when recycled planings are used to increase the fines content which assists the emulsion breaking process. Slow setting emulsions are used. These are mostly cationic, and bitumen penetration grades of 40/50 or 180/220 are used. The bitumen emulsion is added at a level of 2.2 to 4 % (dependent on whether virgin or recycled material is used) and the hydraulic binder is used at a level between 2 and 2.6 %. The ratio between the two binders affects the nature and performance of the mix in that more bitumen leads to a more flexible pavement which is more susceptible to rutting, whereas more cement gives rise to greater resistance to rutting but also a brittle layer which can be susceptible to cracking and shrinkage. By careful mix design, an optimum mixture can be obtained.

3.3.6 Spain

Emulsion mixtures have been used in Spain since the late 1950's. Gravel and open graded mixtures, which are now widely used, appeared in the 1970's (53). High viscosity, medium setting, cationic or anionic emulsions are used in open graded mixtures which ensure thick bitumen coatings on the aggregate. Cold mix is used on all but the highest volume roads without modified binders but, with modified binders, even these roads can be constructed using cold mix. In addition to the environmental and logistical benefits, open graded emulsion mixtures, with more than 20 % voids, are claimed to have other important properties. These mixtures are resistant to fatigue cracking which can be a problem in thin surfaces like those used in Spain. They also have good skid resistance due to drainage and surface friction. Thick binder films used in open mixtures are resistant to ageing. It has been found that open graded mixtures are more storage stable and yet cure more rapidly once layed. Due to mobilization of the interlocking large aggregate skeleton, they are also said to be more resistant to deformation. Polymer modification of the binder has been employed in these mixtures to further enhance properties (54).

3.3.7 Germany

Rode and Weinert (55) stated in 1993, that

"Cold placing methods have not yet been sufficiently proven in road

construction practice in Germany and it is not yet clear on which grades of roads this mix is to be used"

At that time, more than 90 % of roads were constructed using hot methods but it was predicted that ecological considerations may obstruct the use of hot processes in the future. Due to claims that fumes from bitumen are carcinogenic, hot mix plant was becoming more sophisticated and consequently more expensive. This is a very effective driving force for companies concerned with road construction to look for ways around the problem.

Two methods of cold laying have been identified as mix in plant and mix in place - the latter being particularly useful for recycling. A substantial amount of material has been laid in Germany using milled road material or demolition waste with emulsion and cement as binders. Roads of this nature have been of very high quality. Laboratory tests have been devised to asses the properties which can be expected from cold layed materials and these are based on density specifications, Marshall tests on samples before and after water soaking and splitting tests.

Cold mix placing is known to require different techniques from hot mix. Due to the altered flow properties of the material, the angle of the placing beam in the paver is changed to give the correct layer thickness. Compaction begins immediately after laying using a twin steel drum roller. The use of very heavy equipment is common and pneumatic tyred rollers are recommended. Minimal vibration is necessary apart from a small amount of "deep" vibration. Lower roller speeds compared with hot mix are said to be most effective.

In an environmentally considerate country such as Germany, mix in place cold road recycling is a very appreciated method of road construction. Before any work commences the nature of the road to be recycled must be well known. Old pavement is often milled to a depth of 30 cm. Millings are reduced in size and then combined with a slurry of cement and water and bitumen emulsion. The mixture is then relayed and compacted using the techniques outlined above. Pavement thus constructed has been found to make excellent foundation or sub-base.

It is predicted that due to the excellent performance thus far and the likelihood of improvements in the future, cold laid pavement will become more widely used in Germany in the future.

3.3.8 Italy

In recent years, cold worked bituminous mixtures have come into widespread use in Italy for both the reinforcement of existing roads and the construction of new roads (56). As a consequence, the national research council in Italy has drawn up a set of regulations to control mixtures and construction techniques. The normal cold mix types, design methods and laying techniques are employed and the Italian climate favours the use of emulsion mixtures.

3.4 NORTH AMERICAN PRACTICE

Cold mix has been used in the United States of America since the 1960's. Due to the remoteness of some areas of the country, cold mix is an essential part of the road construction industry. Information on US practice is easily available in manuals compiled by various organizations (57, 58, 59) to advise on all aspects of production and laying of cold mix as well as cold recycling.

Two classifications of cold mix are identified according to mixing method. These are plant-mixed and mix-in-place (road mix). The environmental, economic and versatility benefits are recognized as are the limitations such as sensitivity to wet weather and slow build up of strength.

Two types of bituminous binder are prescribed for cold mix, namely cut-back and emulsified asphalt. Due to environmental concerns, the former is becoming restricted in many areas. Thirteen grades of bitumen emulsion are used with a range of setting rates and binder types. It should be stressed that American emulsions alnost always contain solvent. The anionic grades are labelled MS or SS, for medium or slow setting, followed by a number to indicate the relative viscosity of the emulsion - the higher the number the greater the viscosity - eg. MS-2 and SS-1. Cationic emulsions are denoted by placing a C in front ie. CMS-2. Additionally the letters HF may be placed in front or h at the end of the code. HF, which stands for high float, indicates that certain chemicals have been added to give thicker films of bitumen on aggregate particles. An "h" placed at the end means that a harder base bitumen has been used. Different grades of cut-back are also used. This cold applied binder is produced by diluting bitumen with solvent to liquify the binder at low temperature. Medium curing (MC) cutbacks use kerosene as solvent, which is relatively volatile, whereas slow curing (SC) cutbacks use heavy flux oils or are made by distilling the crude directly to the desired grade. The setting rate is dependent upon the rate of evaporation of the solvent. A table, reproduced here as Table 3-1, assists the engineer in the selection of a suitable emulsion or cutback for a particular type of job. The use of a binder with the highest viscosity possible is advocated as a general rule. It is interesting to note that the use of emulsions for storage stable patching mixtures is not recommended and only fluxed materials can be used. Conversely, fluxed binders cannot be used in open graded mixtures.

Consideration is given to atmospheric conditions in the use of cold mix. As setting is dependent upon the evaporation of water, damp and cold conditions will inhibit curing whereas dry and warm conditions will speed it up. Both extremes have consequences in handling and performance after laying. Cationic emulsions are to be used if possible due to their built in breaking mechanism and adhesion properties. Some emulsions used in the US contain a small amount of solvent to aid mixing and workability. When used, time for evaporation of the solvent, in addition to water, must be given before any overlaying takes place.

Table 3-1 : Guide for uses of asphalt in cold mix

	Emulsified asphalt									Cut-back asphalts						
	Anionic					Cationic				Medium curing				slow curing		
Type of construction	MS2	MS2h	HFMS2S	SS1	SS1h	CMS2	CMS2h	CSS1	CSS1h	70	250	800	3000	250	800	3000
Cold-laid plant mix pavement base and surfaces																
Open-graded aggregate	X	X				X	X									
Well-graded aggregate			X	X	X			X	X		X	X	X	X	X	X
Patching, immediate use				X	X			X	X		X	X			X	
Patching, Stockpile											X	X		X	X	
Mixed In place(Road mix) Pavement base and surfaces																
Open-graded aggregate	X	X				X	X					X	X		X	X
Well graded aggregate			X	X	X			X	X		X	X		X	X	
Sand			X	X	X			X	X	X	X	X				
Sandy soil			X	X	X			X	X		X	X				
Patching, immediate use			X	X	X			X	X		X	X			X	
Cold mix recycling																
All mixtures	X	X	X	X	X	X	X	X	X							

A whole range of aggregate types from soil-aggregate combinations to silty sands and well graded aggregate mixtures are successfully cold mixed. High quality aggregates will give rise to optimum mixtures but in areas where these are scarce, inferior substitutes are accommodated. For this reason, no strict gradings are given and mix designs are left to the engineer.

There is no universally accepted bitumen emulsion mixture design method. However, either the Marshall (60) or Hveem (61) methods, or modified versions, are nearly always used. Examples are the Illinois method which is based on the Marshall procedure, and the Asphalt Institute's method which contains elements of the Hveem method plus resilient modulus tests. Resilient modulus or, as it is termed in the UK, stiffness modulus is discussed in detail in chapter 8. If laboratory tests are not possible, empirical formulae for addition levels of emulsified or cut-back asphalt to densely graded aggregate mixtures are provided as a substitute. The formula for the former binder is as shown below.

$$P = (0.05A + 0.1B + 0.5C) \times (0.7) \qquad (3\text{-}1)$$

Where : P = Percent by weight of asphalt emulsion, based on weight of graded mineral aggregate
A = Percent of mineral aggregate > 2.36 mm
B = Percent of mineral aggregate < 2.36 mm and > 75 µm
C = Percent of mineral aggregate < 75 µm

Two types of mixing plant are used, namely in-place and central plant mixing equipment. Mixers for in-place production comprise of rotary mixers which are essentially mobile mixing chambers used for adding binder to formerly unbound roads, motor graders which mix on the roadbed and travel plants which are used for placing mixtures of new material. The last two alternatives can also be fed with planings in a cold recycling process. Spreading equipment includes motor graders and pavers. Compaction is carried out using a combination of pneumatic tyred rollers, for initial compaction, and steel rollers with and without vibration for finishing. Central plant mixers are essentially the same as hot mix plants, except that there are no drying and heating facilities, and can be either batch type or continuous.

In addition to cold mix with new materials, a great deal of cold mix recycling has been carried out in North America since the 1970's (62, 63, 64, 65). This option is growing in popularity due to potential cost savings and logistical benefits as availability of good quality aggregates becomes limited in some regions due to depletion of deposits. The asphalt contained in the old layers is also being seen as a valuable resource which should not be squandered. Additionally, dumping restrictions make it preferable to reuse material rather than to attempt to dispose of it. In the American Asphalt Recycling Manual (written in 1983) it was stated that there are more than a million miles of roads in the United States, which are either already in need of repair or soon will be, that are recyclable (66).

Before a recycling project can be carried out, an assessment of the existing pavement structure, including the subgrade, and the materials used must be carried out (67, 68). Binder content of the mixture is determined and then a sieve analysis is performed on the aggregate mixture. The aggregate should also have either an acceptable plasticity index if it is a soil type material (69) or sand equivalent value (70) to ensure that it does not contain excessive amounts of clay or other fine aggregate. The aggregate's resistance to binder stripping is also sometimes evaluated. If new aggregate is required, this should also be of an acceptable quality. Medium or slow setting emulsions are used and mix tests are carried out to determine whether a cationic or anionic emulsion is best suited to the recycled material. MS emulsions are utilized if the gradation is coarse and SS for finer gradings.

Mix design is based upon laboratory tests, empirical formulae or past experience as no universally accepted method exists. Firstly, the aggregate mixture design is formulated. This will be based on the recovered aggregate grading plus any new material that is required. Both open and dense graded mixtures are used. The binder requirement of the mixture is determined and then the amount of new asphalt required is found from the difference between the residual binder content of the coated material and the total bitumen requirement of the uncoated aggregate. In the case of open graded mixtures, the objective is to use as much bitumen as possible without excessive run-off. Adjustments to the pre-determined design are made on site.

Pavement design is treated in exactly the same way as cold mix with virgin materials with the three factors of traffic volume, subgrade modulus and mixture properties being utilized along with design charts (71, 72). Pavement design is described in more detail in chapter 13.

The favoured and most economical method of pavement recycling involves all steps being carried out in-situ. This is known as in-place recycling. Firstly, drainage must be established as failure to provide a competent system will lead to failure of an emulsified asphalt pavement. The old pavement is then ripped up and size reduction of the material carried out. Alternatively, a process of cold milling is used which directly grinds up the old road on the road bed. Motor graders, rotary mixers or travel plant, which have been discussed above, are used to manufacture the mixture. The final product is often placed on the road surface in a windrow and may be allowed to dry out to some extent to optimize the water content for compaction. This is then spread using a grader. Compaction is carried out with a combination of pneumatic tyred and steel rollers.

Central plant recycling is also used for projects that require high rates of production or close control of mixture design. The old road can either be ripped up and then transported away to be crushed and screened or size reduced in situ for later transport to the mixing plant. Stationary mixing plant consists merely of a batch drum or continuous type mixer and auxiliary equipment and can therefore be located near to site.

3.5 MISCELLANEOUS

3.5.1 Southern Africa

Bitumen emulsion mixtures have been employed in Southern Africa for several years. They are known as granular emulsion mixtures or "GEMS" (73, 74). In addition, large quantities of cement treated base are rehabilitated using emulsion and this material is termed emulsion treated base or ETB (75). The residual binder content of these types of mixtures is only 1 % on average. A significant amount of evaluation work has been carried out and the long term performance of emulsion based mixtures was of particular concern. Use was made of a heavy vehicle simulator (HVS) which was considered to be an ideal tool for accelerated testing. Results from HVS tests suggested that ETB was a reasonable material for pavement construction and that its performance, in terms of strength, durability and resistance to cracking, could be somewhat improved by the addition of cement or lime. On the whole, cement modified emulsion mixtures were found to be more resistant to cracking than concrete but less so than normal hot laid asphalt.

Mixture design methods in Southern Africa are much the same as those used in the United States in that the optimum water and emulsion contents are determined by means of Marshall testing. Stiffness modulus testing has also been employed along with soaking of samples in order to measure resistance to water damage. The mechanical properties determined in the laboratory have been consistently lower than those observed in the field but predictions of field performance have been found to be accurate.

3.5.2 Australia and New Zealand

A consideration of the suitability of the French Grave emulsion mixture for use in Australia was recently reported by Bullen (76). The economic, environmental, and logistical benefits of the mixture were highlighted. It was felt that, due to the warmer and dryer climate of Australia compared with Europe, Grave emulsion should cure more rapidly. However, this increased temperature would also result in a reduced stiffness of the cured mixture. Layer thickness equivalencies were determined and found to be 1:1 with respect to a DBM type mixture and 2:1 compared with crushed rock. Overall, Grave emulsion was considered to be a viable alternative to crushed rock and asphaltic concrete on a unit cost basis.

A more novel version of cold mix has been developed by the Australian company Emoleum (77). Hot mix aggregate gradings are used and these can be either open or dense graded. The technology utilizes foamed bitumen (see later) or very high binder content emulsions. Binder contents in the order of 80 % have been evaluated and these emulsions have been found to develop cohesion much more rapidly than normal 60 % emulsions. Standard optimization of water and binder content is carried out and a process similar to double mixing (detailed above) has been used to improve aggregate coating in some instances.

Laboratory tests have shown that mixtures containing the higher binder content emulsions cured more rapidly than the conventional mixtures and that the resistance to moisture was improved. The effect of fillers was also investigated and it was found that they made a significant improvement to mechanical properties.

Cold recycling techniques have also been adopted in Australia. Remtulla and Escobar reported on studies relating development of a highly specialized recycling system (78). The phenomenal growth of recycling was highlighted and said to be due to energy and resource conservation, costs, shortage of landfill sites, increasing availability and the development of new equipment and materials.

The recycling technology put forward here did not merely involve remixing old pavement with bitumen emulsion but had the added benefit of rejuvenating the old binder through the addition of chemicals. As discussed in the previous chapter, bitumen consists of five groups of components - Asphaltenes, polar compounds and unsaturated, slightly unsaturated and saturated hydrocarbons. The last four belong to the maltenes group and the ratio of these components gives rise to the durability of bitumen as a cementing agent. A parameter termed the maltene distribution ratio was given as

$$MDR=\frac{PC+A1}{A2+S} \qquad (3\text{-}2)$$

Where : PC = polar compounds
A1 = unsaturated resinous hydrocarbons
A2 = sl. unsaturated hydrocarbons
S = saturated hydrocarbons

Generally this is the ratio of the most reactive to the least reactive fractions. Rejuvenation of the binder can be achieved by chemically determining the MDR and adding a rejuvenating agent to correct any maltene deficiencies. A bitumen with a MDR between 0.2 and 1.2 and a PC/S ratio > 0.5 is acceptable and so this is the target for any corrective action. The basic requirements are workability, durability and good appearance.

A 3 step design procedure has been developed. Firstly the composition and properties of the old pavement must be determined in terms of bitumen content and consistency and aggregate grading and bitumen demand. The bitumen demand of the uncoated aggregate can be found using the Centrifuge Kerosene Equivalent method (CKE) (79) or the following equation.

$$P = \frac{(4R+7S+12F)C}{100} \qquad (3\text{-}3)$$

Where :

P = Weight percent of bitumen in mix

R = Weight percent aggregate > 2.38 mm

S = Weight percent aggregate < 2.38 mm and > 75 µm

F = Weight percent aggregate < 75 µm

C = circumstantial factor (1.0 for new aggregate).

The level of recycling agent required then equals the bitumen demand of the aggregate minus the residual bitumen content of the old mix. The recycling agent requirement is then determined to achieve the correct workability and durability of the mix by improving the MDR and mix consistency. Values from the first two steps are then used to design the mix which is optimized by use of Marshall, Hveem or MATTA (another version of the NAT which is discussed in chapter 8) techniques.

It is claimed that field trials using the above methods have resulted in the production of recycled pavement with performance equivalent to new asphalt pavement.

3.5.3 Emulsion mixtures for low volume roads and developing countries

Bitumen emulsions are used in the United States for soil stabilization and for minor or temporary roads such as those required by the foresting industry and elsewhere (80, 81, 82). In these cases, low quality aggregate or soil mixtures are utilized as cost saving is the primary objective and requirements for pavement quality are rudimentary. Bitumen emulsions offer a highly flexible and cheap way of constructing these types of roads.

Cold mix processes are under evaluation in a number of developing countries as many of the benefits listed in chapter 1 are very attractive to countries with limited funds and infrastructure (83). A method of road construction offering economic and logistical benefits is naturally very alluring. Quite basic processes are currently being adopted using marginal aggregates but, considering the reduced traffic burden and standards, such roads can be quite acceptable. A significant amount of in place recycling of unbound roads is underway and this is an efficient and simple way of improving existing routes (84, 85). With the use of deep milling, recycling allows the bearing capacity of unbound or formerly cement bound roads to be significantly increased and the incorporation of bituminous binder also improves durability.

3.6 FOAMED BITUMEN

An alternative cold process utilizes a binder added as a foamed bitumen. Foamed bitumen is produced by mixing hot liquid bitumen with water plus a surfactant. Rapid boiling of the water causes the bitumen to expand to fifteen times its volume and form a foam which is stabilized by the surfactant. This cool binder is then combined with an aggregate mixture and, due to the increased volume of the binder, is said to give a high degree of coating. The foamed bitumen process is not an emulsion system and, therefore, it was outside the scope of this report.

3.7 DISCUSSION

Despite the established use of cold mix in certain countries, for certain applications, it still has several limitations which restrict its use as indicated in chapter 1. Unlike hot mix, emulsion mixtures do not reach their full potential level of mechanical properties or durability until several weeks or months after laying. This may be acceptable in some situations, but in the case of heavily trafficked roads, a newly laid cold mix pavement would probably disintegrate before it had the opportunity to cure. The studies described in this report were aimed at addressing exactly this problem.

CHAPTER 4

RECENT DEVELOPMENTS IN BITUMEN EMULSION MIXTURES

4.1 INTRODUCTION

In chapter 3, the current methods of bitumen emulsion mixture production and laying were discussed. The most widely used methods, namely Grave emulsion and the methods used in the United States of America, have been in use for over 30 years and yield serviceable roads. More recently evolved methods, such as the double mixing process, two stage mixing and the double treatment process, have involved making adjustments in mixture manufacturing methods or using secondary binders in an attempt to improve performance to enable emulsion mixtures to be more widely used.

Yet more recently, other innovations have appeared which build further on the well established technology. These methods will be discussed in this chapter along with developments which are still confined to the laboratory or small scale trials. The last section in this chapter gives a specific account of the use of ordinary portland cement in emulsion mixtures.

4.2 GAP GRADED COLD ASPHALT CONCRETE

An innovative type of cold mix has been developed by the company Jean Lefebvre in France (86). As it is based on a gap graded aggregate mixture combined with bitumen emulsion it has been named Gap Graded Cold Asphalt Concrete. Figure 4-1 shows the grading curves for 6 and 10 mm mixtures.

This process also utilizes two additives which give the mixture enhanced properties. These are polyacrylonitrile fibres and a polymer called EVA (ethyl vinyl acetate). The fibres are nominally 4 mm in length and are added at a level

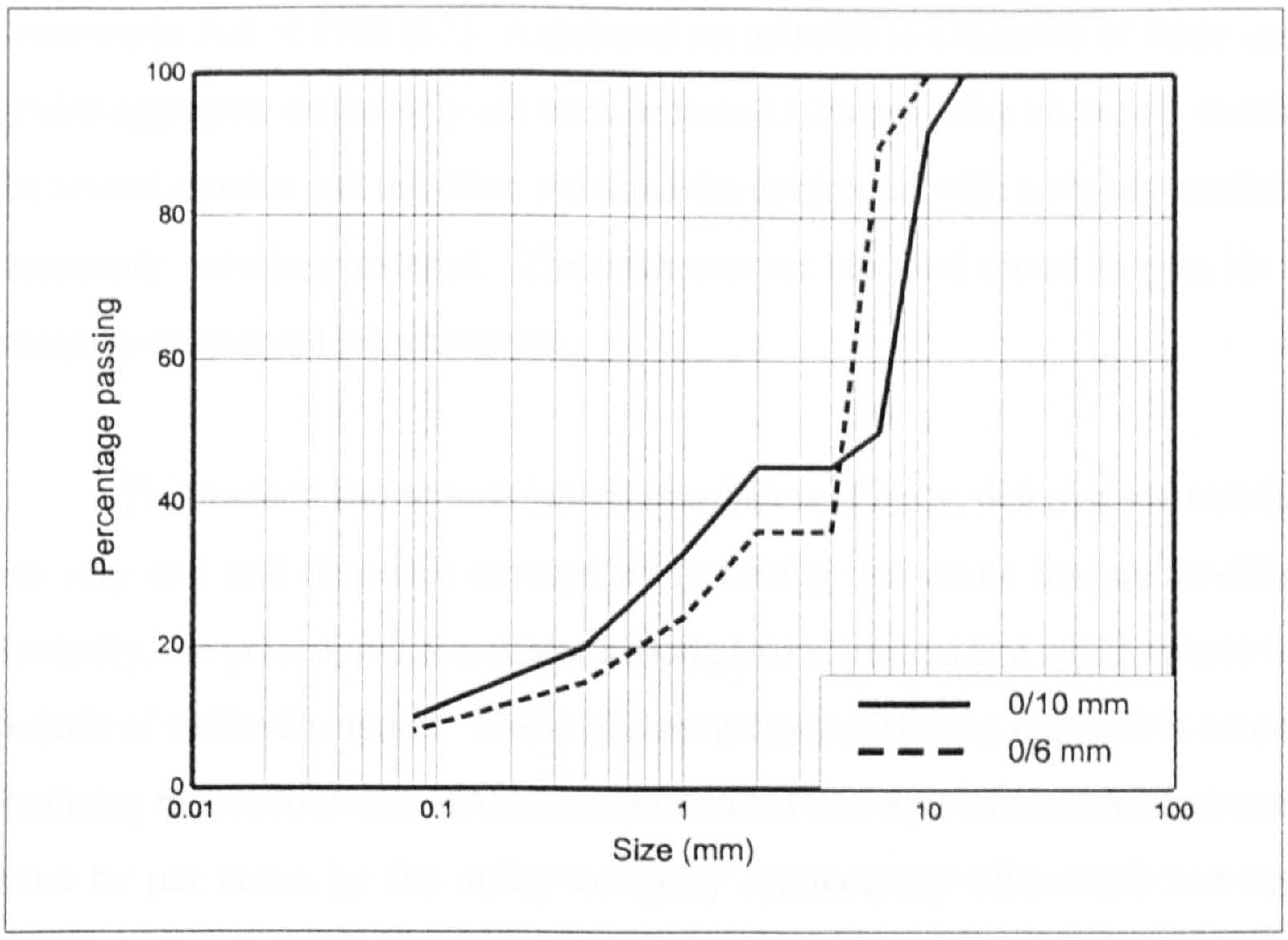


Figure 4-1 : Gap graded aggregate mixture design

of 0.1 to 0.2 % on the dry aggregate. They are said to reduce segregation of the mixture, reduce chip loss and increase shear strength after cure. EVA is sometimes added to bitumen used in hot mix to increase resistance to rutting. In this process, it is added to the binder at a level of ~5 % prior to emulsification in order to improve cohesion, temperature susceptibility, rheological behaviour and adhesion. The mixture is claimed to have improved skid resistance, durability and wearing resistance compared with normal cold mix materials.

4.3 NEW UK SPECIFICATIONS

In 1986, a group known as the Highways and Utilities Committee (HAUC) was set up in the UK, to investigate ways in which the reinstatement of highways after excavations had been carried out could be improved. The recommendations became law in 1991 when the new roads and streetworks act (2) came into force. Prior to this, normal practice was to use a layer of deferred set material as the surfacing material over a backfilled trench, and later to replace this with a permanent reinstatement as laid down in the Public Utilities

Streetworks Act of 1950 (87). A deferred set material is a mixture of fairly open graded aggregate and heavily cut-back or fluxed bitumen. The mixture is storable for several months and therefore provided the contractor with a readily available temporary patching material. The onus was on the road owner to provide an adequate permanent reinstatement.

This method had several inherent problems. Firstly, deferred set materials are very soft and therefore susceptible to rutting and stone loss or ravelling. Secondly, the procedure necessitates making two visits to a site which causes two periods of traffic disruption. Lastly, the temporary surfacing material is wasted. Realizing these problems, HAUC recommended that a permanent reinstatement must be put down by the utility company immediately after work had been completed, and that the material and workmanship must comply with a set of specifications. If a material fails within the guarantee period, the contractor is responsible for carrying out remedial action and must bear the cost. The prescribed values basically precluded the use of deferred set material and, therefore, hot mix or an alternative material with similar properties would have to be used.

Initially, attention focused on the use of hot boxes to allow small quantities of hot mix to be used for small jobs. However, what utilities really needed was a material that could be stockpiled and would always be available day and night. Consequently, focus then centred on permanent cold lay surfacing materials or PCSM's. In 1990 a series of field trials were carried out in Nottingham followed by laboratory tests at Nottingham University to evaluate the currently available PCSM materials. Overall, the studies showed that cold lay materials could not achieve performance values comparable with equivalent hot mix (88). Tests carried out with the Nottingham Asphalt Tester (a piece of apparatus which enables the mechanical properties of bituminous materials to be measured - covered in detail below) were used as a guide for a set of specifications for PCSM's. These specifications became appendix A10 of the

New Roads and Streetworks Act 1991 which is reproduced in Table 4-1.

In order to gain approval, a new PCSM must undergo a formal trial and comply with the current specifications as shown in the table below, within a two year period or three years for deep openings. Unfortunately, for parties attempting to gain approval, the specifications are continuously being altered as arguments push and pull the debate in various directions (89). Recently, the use of the NAT has been questioned and other pieces of test equipment such as wheel trackers have been suggested. Most utility companies have begun formal approval trials to some degree, and a number of Highway Authorities now use better examples of these newly improved cold laid materials. The first fully approved materials were expected in 1994 but to date none have actually gained full approval. It should be stated that this is due to late starts rather than failure of materials. However, some materials in the later stages of assessment look promising.

Table 4-1 : Minimum permanent cold-lay surfacing material requirements

Permanent cold lay surfacing material	Minimum Property Requirement at 20°C for equivalence to :		
	50 pen hot-laid Elastic stiffness (MPa)	100 pen hot-laid Elastic stiffness (MPa)	200 pen hot-laid Elastic stiffness (MPa)
20 mm nom. size basecourse	4600	2400	900
10 mm nom. size wearing course	3800	1900	800
6 mm nom. size wearing course	2800	1400	600
All materials	**Uniaxial creep (resistance to permanent deformation)** **NAT test** : Results shall be plotted as microstrain vs. time (seconds as horizontal axis). The characteristic shall exhibit a distinct stabilization during the first 2000 seconds, a near horizontal final phase and shall not exceed 10,000 microstrain at any time during the test. **Note 1** : A wheel-tracking test is permitted as an alternative to the NAT test **Note 2** : The tabulated values are provisional and may be subject to review		

(Appendix A10 New Roads and Streetworks Act 1991)

4.4 STONE FILLED EMULSION ASPHALT

A different approach to the use of emulsions in mixtures has been investigated by Khweir et al (90, 91), partly driven by the HAUC specifications described above. The material is known as stone filled emulsion asphalt and consists of a large stone matrix (ie 20 mm single size) which is infilled with an emulsion based grout. Laying is carried out by first placing a single size aggregate into the prepared excavation which is then compacted. The grout slurry is poured onto the stone matrix and should penetrate right down to the base. The benefit of this method is that the grout can be produced on site thus removing the need for a storage stable mix. The grout is formulated to be quite rapid setting so it is claimed that the mixtures themselves cure quickly in the pavement. A K2/70 emulsion is used which denotes a medium setting cationic emulsion containing 70 % bitumen.

The grout consists of bitumen emulsion, sand, cement and water. The ratio of these components dictates the flow properties of the grout and the setting rate and mechanical properties of the final material. A plasticizer is also incorporated to improve the properties of the slurry. The fluid phase must always be of a viscosity low enough to allow it to flow into the pores in the large stone matrix. A typical mixture recipe for the grout is shown in Table 4-2.

Table 4-2 : Typical grout recipe

Ingredient	**Percentage by weight**
Cement	30
Sand	30
Emulsion	20
Water	19.82
Plasticizer	0.18

With cationic emulsifiers, grouts remain fluid for about an hour and set after six. These figures have been determined through flow measurements and standard cone penetration measurements respectively. Marshall stability measurements on the mortar have shown that quite high stabilities, in the order of 5 to 15 kN, can be achieved after 7 days. This is very dependent on the proportions of the mixture ingredients. A field trial indicated that the material also performs well in practice. The grout was to found to have fully penetrated the stone matrix, no rutting or ravelling of the section was observed and stiffness of 3500 MPa were recorded for material cored from the trial using a Nottingham Asphalt Tester (NAT - see Chapter 5) which is comparable with hot mix. However, this core was from the top of the layer and material from the base had a stiffness of only 800 MPa. This suggests that the mixture is not as rapid curing as first claimed and relies to a large extent on evaporation of water as do other emulsion based mixtures.

Stone filled emulsion asphalt is potentially quite useful for trench reinstatement; the only drawback being that it requires some on site mixing. However, it could not be used on a large scale due to the nature of the process.

4.5 COLD RECYCLING IN THE UK

Although cold recycling has been in use in many countries for a number of years, as described in chapter 3, the technique has only recently gained respectability in the UK (92, 93). Walsh states that the 'Retread' process has been in circulation since the 1930's but this low tech, low performance method was not acceptable on any large scale. With the advent of more powerful machinery, capable of reducing recycled material to acceptable gradings, better coated mixtures have been possible which have been closer to UK expectations.

In 1986 and 87, several recycling projects were carried out in Kent in the South of England. Site investigations were undertaken, involving CBR measurements on the subgrade and assessment of the old material in the road. Pavement design adopted the United States' rule of thumb in that cold mixed material is about 80 % as effective as comparable hot mix. The thickness design was therefore increased by a factor of 1.25 (see chapter 13 for more information on pavement design). The projects mainly involved the use of foamed bitumen but emulsions were also employed. The former is claimed to be less affected by adverse weather conditions. In addition, cement stabilized mixtures were produced but these were said to be less satisfactory on a number of counts. The pavements resulting from the cold bituminous mixtures were trafficked soon after laying and were perfectly stable and did not deteriorate due to the action of traffic. In situ testing and testing of cores taken from the roads, indicated that the mechanical properties of the mixtures were similar to those of hot mix. In addition to the environmental and obvious cost saving advantages of cold mix, some of the practical benefits were also realized in that kerbs, ironwork and pipework did not normally need to be altered.

4.6 THE ADDITION OF ORDINARY PORTLAND CEMENT

Some fo the cold mix techniques described in chapter 3 and earlier in this chapter utilize ordinary portland cement (OPC) to improve mechanical properties. In this section, other descrete studies are reviewed which have also centred on this topic.

Since the 1970's it has been known that the incorporation of small amounts of ordinary Portland cement (OPC) in bitumen emulsion mixtures can have a beneficial effect on the mechanical properties of these mixtures. Terrell and Wang (16) carried out what can be considered as pioneering work on the subject. Having identified the possibility of using marginal aggregates for basecourse in combination with bitumen emulsions, they focused on the use of OPC and lime in these mixtures as a means of enhancing performance. In addition to increasing the curing rate under warm conditions, particularly in the early stages, it was found that OPC enabled a mixture to cure under cold and damp circumstances. Tests on mixtures with cement only and mixtures with emulsion with different levels of cement, revealed that the combination of bitumen and around 1 % OPC yielded the optimum performance. The emulsions used in Terrell and Wang's studies were anionic and it was postulated that the Ca^{2+} ions from the cement neutralize the emulsifier, thus allowing the bitumen droplets to coalesce and then adhere to the aggregate.

Schmidt, Santucci and Coyne (15) built on these studies also utilizing a slow setting anionic emulsion. It was found that after one day of curing, the presence of 1.3 % OPC in a mixture increased the stiffness modulus 5 times and after 60 days, a doubling in stiffness was achieved. Schmidt et al found that the more cement they used, the greater the stiffness developed in the mixture. A benefit in resistance to water damage was also observed. On soaking, without OPC, stiffness dropped to 18 % of the unsoaked value but with 1.3 and 3 % OPC, 70 and 85 % of the initial stiffness remained in the mixtures respectively. The studies also involved a small number of fatigue tests. It was found that hot mix had better resistance to fatigue cracking than equivalent cold mix and that the addition of OPC only made the situation worse due to embrittlement. However, due to the increased stiffness of cement treated mixtures, strain at the base of a pavement layer would be reduced implying that thinner layers of emulsion mixtures could be layed if cement were included. The reasoning behind this is explained in chapter 13.

Shortly after the two sets of studies described above, Head (94) carried out further work based on the previous findings but this time a slow setting cationic emulsion was employed. Using the Marshall rather than resilient modulus test, Head arrived at much the same conclusions as Terrell and Wang and Schmidt et al.

The latter two of the above investigations involved field studies to verify laboratory findings. Schmidt et al showed that the beneficial effects of cement found in the laboratory, in terms of stiffness and resistance to water damage, could be reproduced in the field. Head describes a successful laying operation but no measurements of mechanical properties are provided. The logistical and cost saving benefits of emulsion mixtures were advocated in both reports.

Recently, other workers in the field have reiterated the beneficial effect of OPC. Dardak (95) used OPC in sand emulsion mixtures as its inclusion in these types of mixtures achieved a doubling and even trebling of mixture stability. This enabled unbound layers, which were formerly 22 cm in thickness, to be replaced by emulsion/OPC layers which were only 12 cm thick.

Arora and Arabiat (96) utilized OPC in mixtures involving materials available in the Gulf region, to enable dense sands and Marl aggregates to be utilized.

Stone filled mastics, developed at Heriot Watt university by Khweir et al (90), use OPC to accelerate the emulsion break and stiffen the final cured mixture. This method involves the addition of a slurry, composed of sand water and cement, to a layer of single sized aggregate placed in the hole.

Poncino (97), found that the addition of 2 % OPC or hydrated lime increased the resilient modulus of dense graded granitic mixtures by 125 and 66 % respectively, whilst developing cold mix for use in Argentina.

Sainton (51) advocated the use of bitumen/cement composites as the combination of the two binders offers the best of both worlds. Bitumen serves to reduce cracking which can occur in cementitious pavements whilst cement reduces rutting to which bituminous pavements can be susceptible. Sainton's mixtures were based on a grave type mixture in terms of the aggregate grading and low residual binder content of 2 - 3 %. Cement was used at a level of 2 - 3 % also. Mixtures based on this composition are claimed to have stiffness of 13,000 MPa after 90 days curing at 20 °C.

OPC has been utilized for several years in in Japan but without being fully understood according to Uemura and Nakamari (98). They describe a study in which emulsion mixtures were carefully designed and subjected to laboratory evaluation by measuring unconfined compressive strengths on soaked and unsoaked samples. Additionally, field surveys have been carried out on 51 cement modified bituminous emulsion roads in Japan in order to establish pavement service indices (PSI). The conclusion was that these types of mixtures provided acceptable performance on a serviceable life basis as well as being environmentally more acceptable.

CHAPTER 5

MECHANICAL PROPERTIES OF BITUMINOUS MIXTURES AND METHODS USED TO MEASURE THEM

5.1 INTRODUCTION

In order to be able to use a material in an engineering structure, its mechanical properties must be known to enable the engineer to design the structure so that the material can withstand the stresses and strains placed upon it. As pavement technology moves towards performance based design, this is as true for roads as it is for any other structure. Over the years, many different tests have been employed in an attempt to characterize materials, but these have mostly only given comparative values without a fundamental basis. Figures obtained from such tests enable the engineer to design mixtures and pavement structures based on recipes learnt through experience. The Marshall test, for example (60), has been employed in many countries for many years and has provided useable figures, but pavement designs based on these figures are empirical rather than theoretical. The same can be said for the Duriez test (44) described in Chapter 3 but it would be difficult to argue that the French have not obtained good service from roads designed using this method. Over the years, a number of test protocols have been proposed for the design of cold mix employing a range of mixture and specimen manufacturing methods and tests for assessing mechanical properties (99, 100, 101, 102, 103, 104).

The methods used to measure the mechanical properties of mixtures in these studies, were all based on the Nottingham Asphalt Tester (105). This apparatus uses engineering principles and, therefore, the figures it produces are related to the fundamental properties of a mixture.

5.2 MANUFACTURE OF SPECIMENS USED IN LABORATORY TESTS

The instrument used for mechanical properties tests, namely the Nottingham Asphalt Tester (105) which is described later in this chapter, requires the use of cylindrical specimens of compacted mixture. This is an advantage as cores can easily be taken from roads and this is already common practice in order to perform the frequently used Marshall test. For research purposes, cylindrical specimens can also be manufactured from mixtures prepared in the laboratory or plant by a number of methods. In order for laboratory generated results to relate to those in real life, any method of preparation in the laboratory should result in samples with similar properties to materials in a real pavement. As data on cold mix is sparse, particularly for the types of mixtures covered in these studies, it was difficult to make comparisons. In the absence of firm guidelines, therefore, manufactured specimens were evaluated in their own right, and relation to real pavement situations applied accordingly. The aim of these studies was to evaluate the effect of certain parameters on the mechanical properties of mixtures under certain conditions.

5.2.1 Manufacture of mixtures

Most plant mixers are either pug mills or continuous drum mixers. Neither of these are commonly found in the laboratory and instead, simpler agitators are usually used. Whilst they provide a different type of stirring action, these mixers produce material which is not unlike the plant scale mixtures. Most of the experience has been gained with hot mix, but plant scale mix trials carried out during the work described here suggested that the same can be said for cold mix.

Two different mixers were employed during the course of these studies, the first being a Hobart and the second a more sophisticated set up designed and built by the University of Nottingham. The Hobart mixer uses a whisk as a stirrer, whereas the Nottingham mixer has a flat paddle. The stirrers in both pieces of equipment describe a sun and planet type of action which is very effective in obtaining rapid and even distribution of materials.

In the laboratory preparation of bitumen emulsion mixtures, firstly the dry aggregate is placed in the mixing bowl. If required, cement or lime can be added. Water is added next to pre-wet the aggregate before the addition of emulsion and it is, therefore, termed "pre-wet water". The pre-wet water addition level is carefully adjusted depending upon the type and grading of the aggregate and emulsion being used. The aim is to surface saturate the aggregate so that the water from the emulsion is not immediately absorbed which would have the effect of breaking the emulsion. The level of pre-wet water also has an effect on the physical nature of a mixture in that too much will result in a sloppy uncompactable mixture, whereas too little will give rise to a dry, friable mixture lacking in cohesion. Once the pre-wet water has been thoroughly mixed, the bitumen emulsion can then be added. It is important to use the correct mixing time for the emulsion as several factors are at work. Firstly, the binder must be evenly distributed throughout the mixture. Secondly, the emulsion should be partially broken in order that it can not easily be washed off or run off the aggregate. The mechanical energy provided by mixing causes the emulsion to break. Lastly, the emulsion must not be overworked. Emulsion in an advanced state of coalescence will result in an unworkable mixture which will not be cohesive after compaction.

5.2.2 Compaction of mixtures

Several methods of compaction were used in these studies to produce cylindrical specimens.

The Marshall hammer method

Although the Marshall test itself (60) was avoided as a means of assessing the mechanical properties of bituminous materials in these studies, the manufacture of briquettes by this technique was used extensively. The Marshall hammer action consists of a 4.5 kg weight dropped vertically through 457 mm which acts on a 100 mm plate which sits on top of the material under compaction. A 3600 g batch of mixture was manufactured which was divided into three 1200 g portions. These were then placed in three separate steel moulds of 101 mm in diameter. The 3 portions were compacted using fifty blows of the Marshall hammer, inverted, and given another fifty blows to the other side. The compacted specimens were left in their moulds overnight and then extruded the following day.

The Percentage Refusal Density apparatus

During much of the development work on the NAT at Nottingham University, a vibrating hammer was used for the manufacture of moulded specimens (106, 107). This equipment was originally designed to carry out the percentage refusal density (PRD) test to evaluate maximum compaction densities of hot mixtures in the field (108). Due to its availability and flexibility, it was thought to be an ideal tool for specimen fabrication. The equipment consists of split moulds of 150 mm in diameter and a vibrating hammer which can be fitted with two different feet of 100 and 150 mm in diameter. The smaller foot is used first to provide a kneading action by moving the foot in a N, S, E, W, NE, SW, NW, SE pattern on top of the mixture in the mould. Compaction continues for various periods of time (1 to 2 minutes) depending on the desired voids content. The top surface is finally flattened off with the larger foot.

Though this method was found to be useful for hot mix, its use in the early stages of these studies revealed that it was not so applicable for dense cold mix. The resulting specimens were found to be lacking in cohesion and were, therefore, very friable. This was thought to be due to vibrations causing rapid break of the emulsion which prevented proper compaction of the mixture. Additionally, it was suggested that vibration after break of the emulsion had occurred caused the binder to strip off aggregate. Thus, the PRD apparatus was not used after the early part of this research.

The Static Load Press

In order to prepare some of the specimens used in these studies, very intense compaction was required. This was facilitated by the use of a static load press capable of exerting loads of up to 40 t on a 5" diameter RAM which is roughly equivalent to 32 MPa. Static load compaction is used in the French Duriez test, as discussed above (44, 109), but the specified regime was not used in these studies. Mixtures were compacted in Marshall moulds in 1200 g batches, in triplicate, and handled in the same way as Marshall compacted specimens. A 20 t (16 kPa) load was usually used in these tests.

The Roller Compactor

The roller compactor used in these studies was unique to Nottingham as it was designed and built by the University. Small scale rolling compaction is the most effective method of emulating field compaction in the laboratory, as the modes of vertical and horizontal stresses are the similar to those provided by a full scale roller.

The equipment consists of an arc of roller drum which is loaded by a pneumatic actuator. Mixtures are placed in rectangular moulds of 280 by 400 mm and 120 mm in depth. As ~25 kg batches of mixture are normally compacted in the roller compactor, segregation could be a problem. This is minimized by manufacturing the mixture in five batches of ~5 kg each in weight and adding them to the mould via a five way divider. The mould is placed under the roller and moves back and forth as the roller pushes down. The apparatus is shown diagrammatically in Figure 5-1.

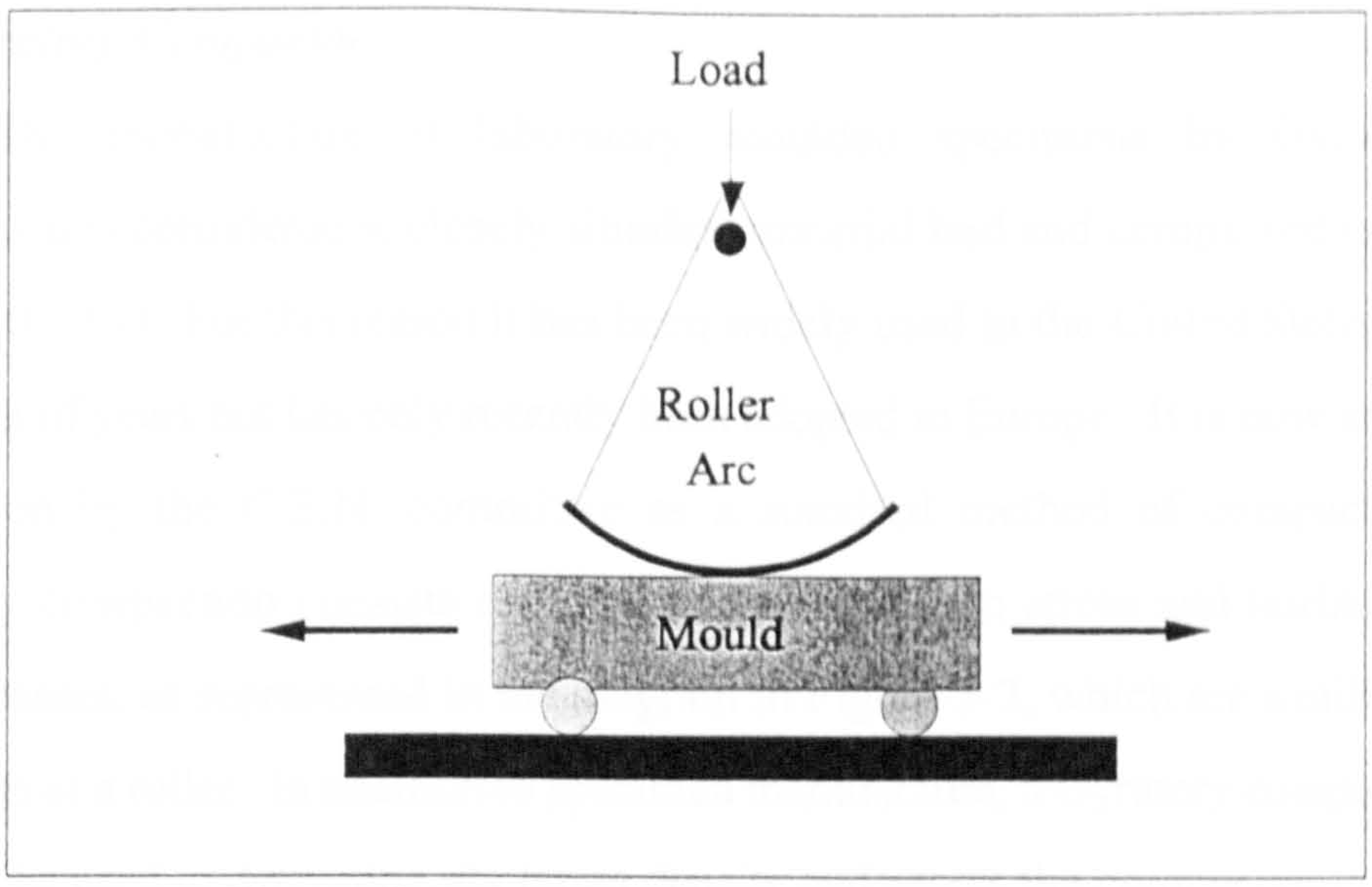


Figure 5-1 : Nottingham roller compactor

The slab of compacted mixture obtained must then be cored. In the case of hot mix this may be carried out either vertically, or, in order to obtain a greater number of useable cores, horizontally. The ends of the rough cores are then trimmed with a circular saw to give even surfaces at 90 ° to the sides. With cold mix it was found that the slab was too delicate to allow horizontal coring. Even the vertical coring and trimming processes yielded cores which were partially damaged. In addition to the mechanical stress involved, the main problem for bitumen emulsion mixtures is that large amounts of water must be sprayed over the cutting tools as coolant and lubricant. If the emulsion is still in a partially unbroken state, this will lead to removal of the water rinsable binder and break down of the material. The roller compaction method could, therefore, only be used on slabs which had been cured for long periods. This was both time consuming and not practical as early life stiffness was to be studied in addition to long term mechanical properties. For these reasons the roller compactor was not widely used in these studies. In some parts of the United States, this problem is circumvented by dry coring. In this case, the coolant is liquid Oxygen which is blown down the shaft of the corer where it vaporizes and, therefore, cools the drill and specimen without damage to the tender cold mix material. This method was not known at the time of these studies and so was not utilized. However, it should be evaluated in any future studies for both laboratory and field coring.

The Gyratory Compactor

The manufacture of laboratory moulded specimens by Gyratory compaction is considered to closely simulate material laid and compacted in the road (110, 111). For this reason it has been widely used in the United States for a number of years but has only recently been adopted in Europe. It is now under evaluation by the C.E.N. committee as a standard method of compaction. Gyratory compaction consists of a vertical compression stress and horizontal shear stresses, as represented in the diagram in Figure 5-2, which are similar to the action of a roller. In addition to specimen manufacture, a Gyratory compactor can also be used to determine site target density and assess the compactability of a mixture.

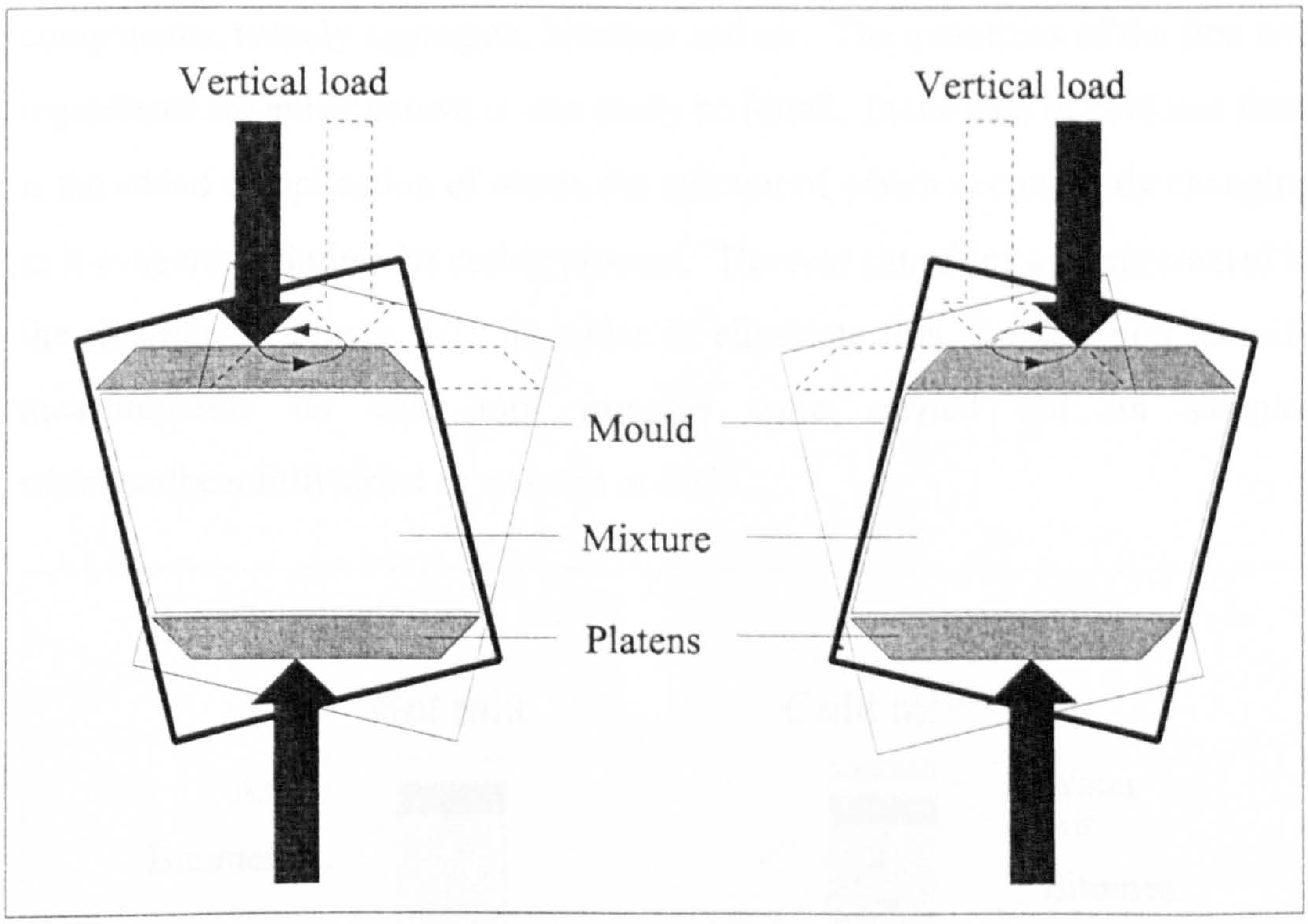


Figure 5-2 : Gyratory compaction mechanism

The specimen is compacted in a cylindrical mould which is clamped so that it cannot rotate. The axis of the mould is inclined at an angle of 1 to 4 ° to the vertical. A static axial stress is applied to the mixture in the mould. The axis of the mould is gyrated and this action generates horizontal shear stresses in the mixture which enables aggregate particles to orient themselves during compaction. Other types of compaction do not achieve this action and this is why gyration is considered to be beneficial. The main elements of the Gyrocomp equipment used in these studies is shown in Figure 5-2a.

5.3 VOLUMETRIC PROPORTIONS

For the mechanical properties measurements described below to be meaningful, the physical nature of the mixtures under assessment must be known. This requires measurement of the densities of cores or moulded specimens and more specifically calculation of the air void content.

Bituminous mixtures of the hot mix variety are composed of three components, namely aggregate, bitumen and air. The quantities of the first two ingredients are either known or can easily be found. In the case of cold mix there is the added complication of water, the amount of which is constantly changing as it evaporates during the curing process. The two situations are represented in the diagram in Figure 5-3. In order to eliminate this complication, density measurements on cold mix samples were carried out on samples which had been fully dried in an oven at 60 °C.

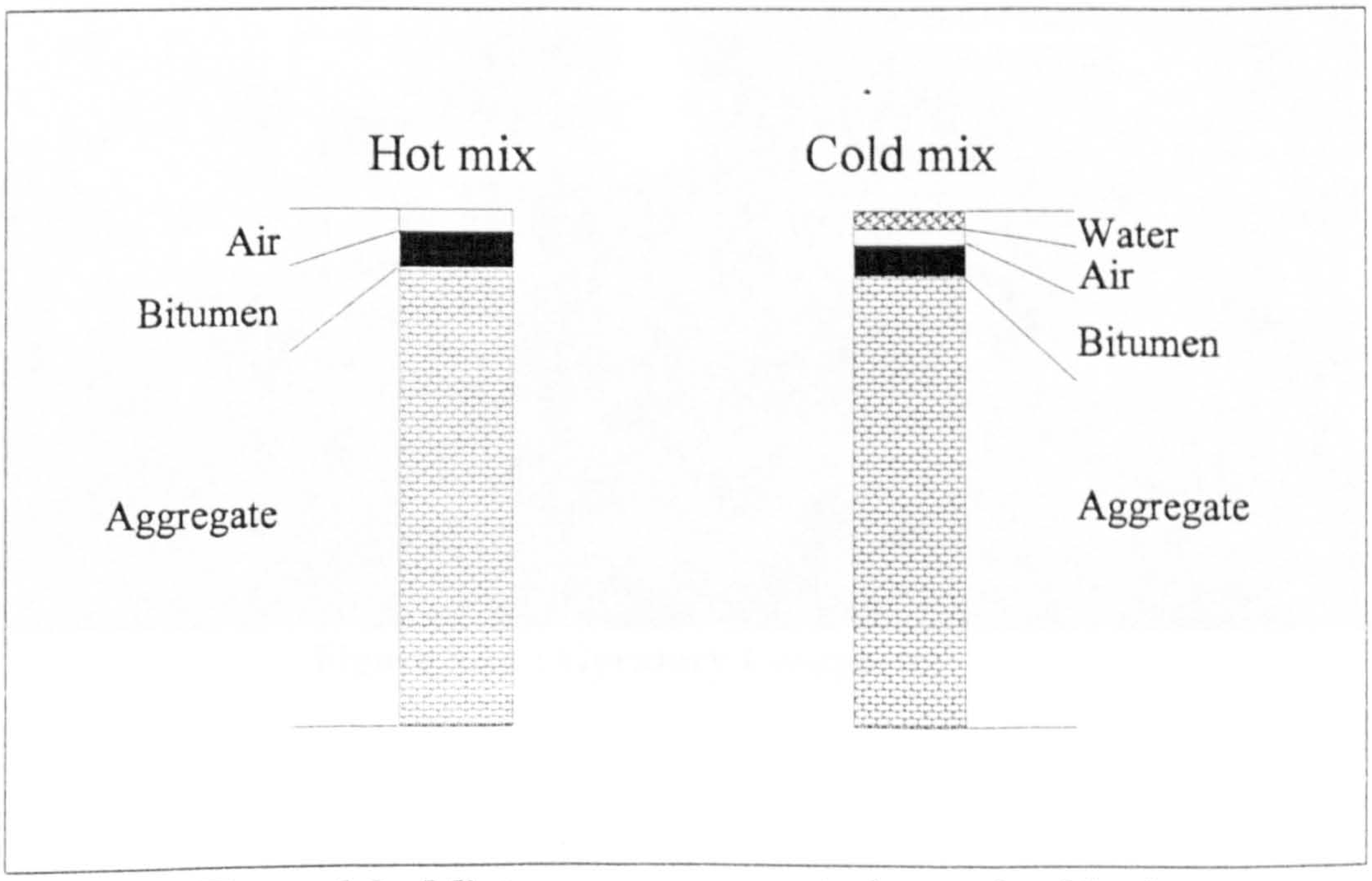


Figure 5-3 : Mixture components in hot and cold mix

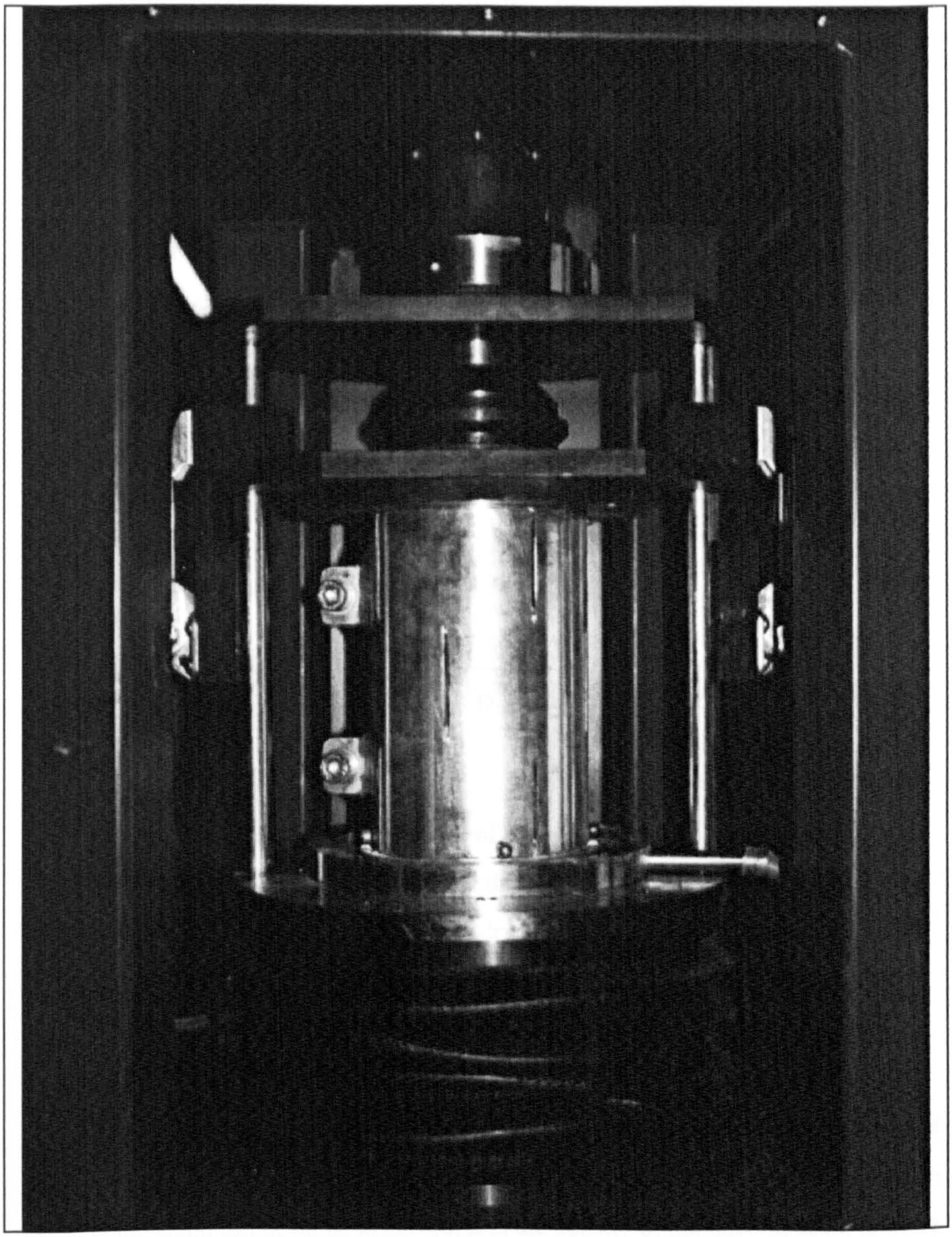


Figure 5.2a : Gyratory Compactor

5.3.1 Measurement of volumetric proportions

The standard method of determining the density of a core or moulded specimen is as follows (112). Firstly the specimen is weighed in air and then coated with a water proof substance which completely seals the sample allowing an accurate measurement of voids to be made. The water proof substance is traditionally molten wax, but recent work at Nottingham, where specimens have nearly always been cores, has involved using adhesive tape as it is more easily removed allowing the same samples to also be used for measurement of mechanical properties. However, during the course of these studies, as most of the samples tested were moulded specimens having more uneven surfaces than cored specimens, it was found to be much simpler to use wax. After sealing, the specimen is reweighed in air and then in water. The loss of weight in water being equal to the volume of the sample. The density is then calculated using equation 5-1 shown below :

$$\rho_{core}=\frac{A}{D-E-\frac{D-A}{F}} \qquad (5\text{-}1)$$

Where : ρ_{core} = Bulk density of core (kgm^{-3})
A = Weight of the dry specimen in air (kg)
D = weight of the dry specimen plus wax coating in air(kg)
E = weight of the specimen plus wax coating in water (kg)
F = bulk specific gravity of the wax (0.9)

In order to find the air void content, the maximum density of the compacted material without any voids must also be determined. As 100 % compaction can never be achieved, it is the density of the uncompacted mixture that is actually measured which is equivalent to the maximum mixture density. Outside the USA this was normally done by calculations based on the mixture design, but a better method is to find the density by measurement using a piece of equipment known as the Rice pot (113). This is basically a large pycnometer which is a piece of apparatus that enables the mass of a material plus a reproducible amount of water to be accurately measured, allowing the volume of preweighed sample of a material to be found. This is accomplished by firstly filling the Rice pot with water and weighing it. A preweighed sample of loose mixture is then placed in the pot which is partially filled with water. The vessel is then evacuated to remove any trapped air from the mixture. Finally, the pot containing the mixture is fully filled with water and reweighed. The density of the uncompacted mixture is then calculated using equation 5-2 shown below :

$$\rho_{mm}=\frac{A}{A+B-C}\cdot 0.9971 \tag{5-2}$$

Where : ρ_{mm} = maximum density of mixture (kgm-3)

A = Mass of dry sample in air (kg)

B = Mass of pycnometer filled with water at 25 °C (kg)

C = Mass of pycnometer filled with water and sample at 25 °C (kg)

The air void content can then be calculated as follows :

$$V_v=\frac{\rho_{mix}-\rho_{core}}{\rho_{mix}}\cdot 100 \tag{5-3}$$

Where : V_V = Percentage air voids in the compacted core

ρ_{mix} = Density of the uncompacted mixture (maximum mixture density) (kgm^{-3})

ρ_{core} = Density of the compacted core (kgm^{-3})

The density, or more specifically the air void content of a material, is important for a number of reasons (114). If a mixture's density is too low, due to poor compaction or low workability, the aggregate structure will be ineffective as the stone to stone contact is low. This will have the effect of placing any load stress on the binder which will be unable to cope resulting in deformation of the mixture. Conversely, if the air void content is too low, again the mixture can be unstable. This situation can only arise through two mechanisms. The first being that the mixture has been over compacted, resulting in crushing of the aggregate and the second that the original mixture design was wrong, in that too much binder was added, resulting in overfilling of the voids in the mineral aggregate.

5.4 MECHANICAL PROPERTIES

5.4.1 Stiffness modulus

The stiffness modulus of a mixture indicates the ability of a layer to spread load and thereby reduce stress concentration at the base of the bituminous layers and on the underlying layer. It also determines the degree of strain at the base of a pavement layer. Stiffness modulus is, therefore, a determining factor in the lifetime of a road in terms of failure through subbase deterioration and fatigue cracking (114). This stated, cold layed pavement with low stiffness can perform well in the field (64), so stiffness is not the only factor which defines a serviceable cold mix. However, as dictated in the HAUC specifications, in the UK, stiffness is a prerequisite for bitumen emulsion mixtures even for patching.

Measurement of the stiffness modulus of a mixture provides the engineer with definitive values related to fundamental properties of the material in question. Stiffness modulus can be measured in the laboratory by subjecting a cylindrical specimen to a load across its diameter and monitoring the resultant horizontal displacement at right angles. This is analogous to Young's modulus for elastic materials. However, bitumen is a visco elastic substance which means that when deformed, there is a small permanent strain resulting from viscous flow. The behaviour of a bituminous mixture under an applied load is represented in Figure 5-4.

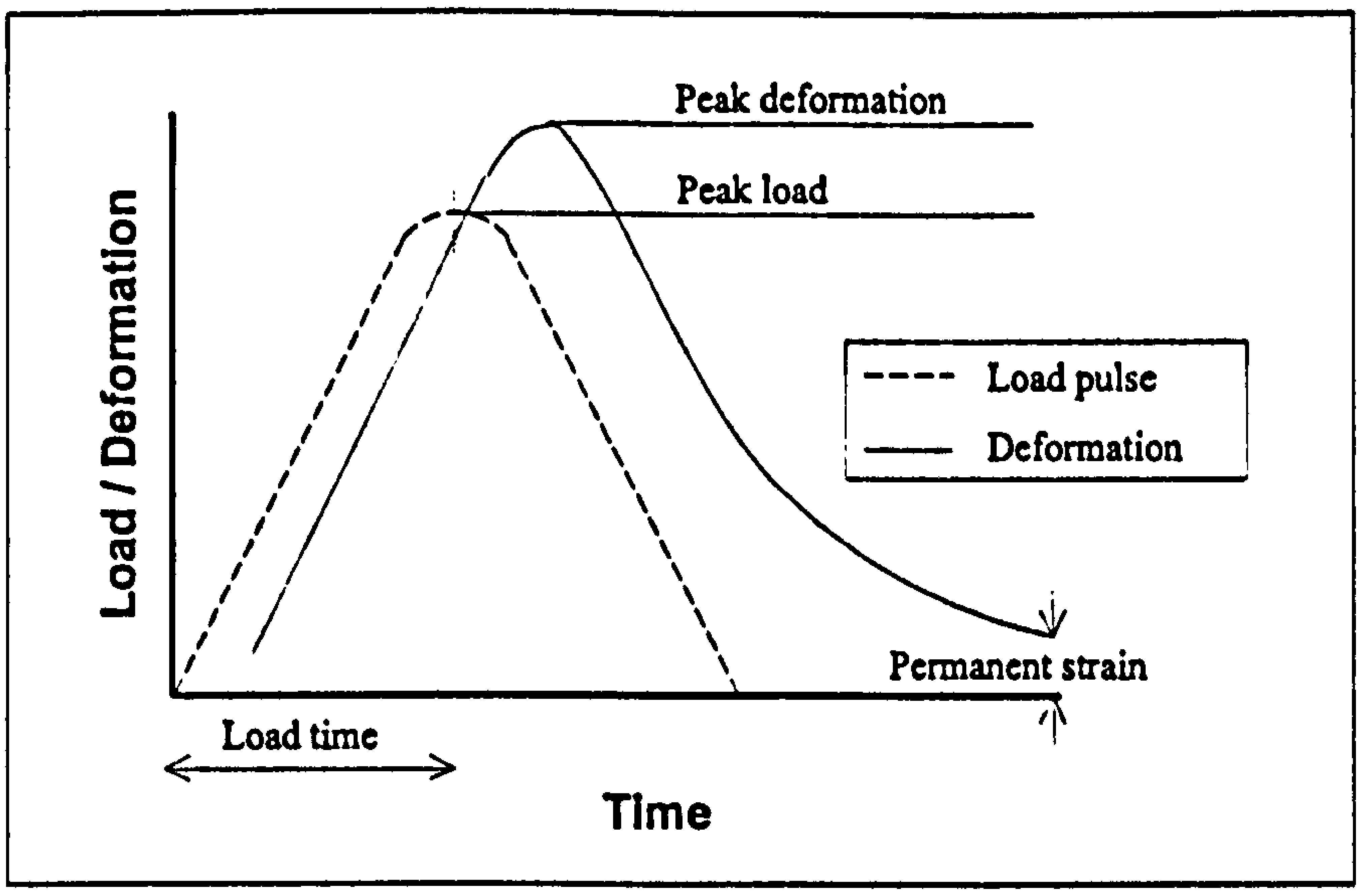


Figure 5-4 : Load and deformation in stiffness modulus test

The stiffness modulus of a mixture is dependent on the applied load, the resultant deformation, dimensions of the sample and Poisson's ratio, which is material and temperature dependent. Indirect tensile stiffness modulus is calculated in units of MPa using equation 5-4 shown below (114). Stiffness of bituminous materials is also dependent on speed of loading due to the viscoelastic nature of the binder (115).

$$S_m = \frac{L}{D*t}*(\nu+0.27) \tag{5-4}$$

Where :
L = peak level of applied vertical load (N)
D = peak horizontal diametral deformation resulting from applied load (mm)
t = mean thickness of test specimen
ν = Poisson's ratio for the material at the test temperature (0.35 for bituminous materials at 20 °C)

The Indirect Tensile Stiffness Modulus test

The resilient modulus test (116) has been in existence for approximately 30 years, but recent development work at the University of Nottingham has resulted in a highly sophisticated yet easy to use version of the test (115, 117).

The equipment used for the test has been termed the Nottingham Asphalt Tester or NAT, and the test itself the Indirect Tensile Stiffness Modulus (ITSM) test. The NAT has been used throughout these studies to assess the mechanical properties of cold mix and in particular to monitor the changing properties of specimens during the curing period. This was fundamental to the work as the effect of various parameters on curing rate, or the absolute stiffness values at a given time after laying, were to be quantified. The ITSM test is perfectly suited for this requirement , as it is virtually non-destructive when used on the kinds of materials it was developed for.

Cylindrical specimens of road material are tested on the NAT with diameters of 10 or 15 cm and depths of between 4 and 8 cm. These may be cores taken from a road or laboratory fabricated specimens. The core or moulded specimen sits in a yoke which ensures that it is correctly positioned. The apparatus uses compressed air to drive an actuator which applies a load to the specimen across its diameter through two loading jaws at the top and bottom of the sample. The applied load, measured by means of a load cell situated in line, is approximately sinusoidal and the rise time can be specified by the operator. For historical reasons, the rise time is normally 120 ms which is generally slower than that associated with moving traffic (a rise time of 50 to 60 ms would be more appropriate but this would be difficult for the pneumatic system to achieve). The resultant horizontal deformation at right angles to the load is measured by two linear variable displacement transducers or LVDT's. The applied load is controlled by a computer as is logging of deformation and subsequent calculation of stiffness modulus. The NAT setup is shown schematically in Figure 5-5 and the photographs in Figure 5-6 show how the actual testing part of the equipment looks in reality, configured for testing of stiffness modulus, resistance to fatigue and resistance to permanent deformation (the latter two mechanical properties are explained below).

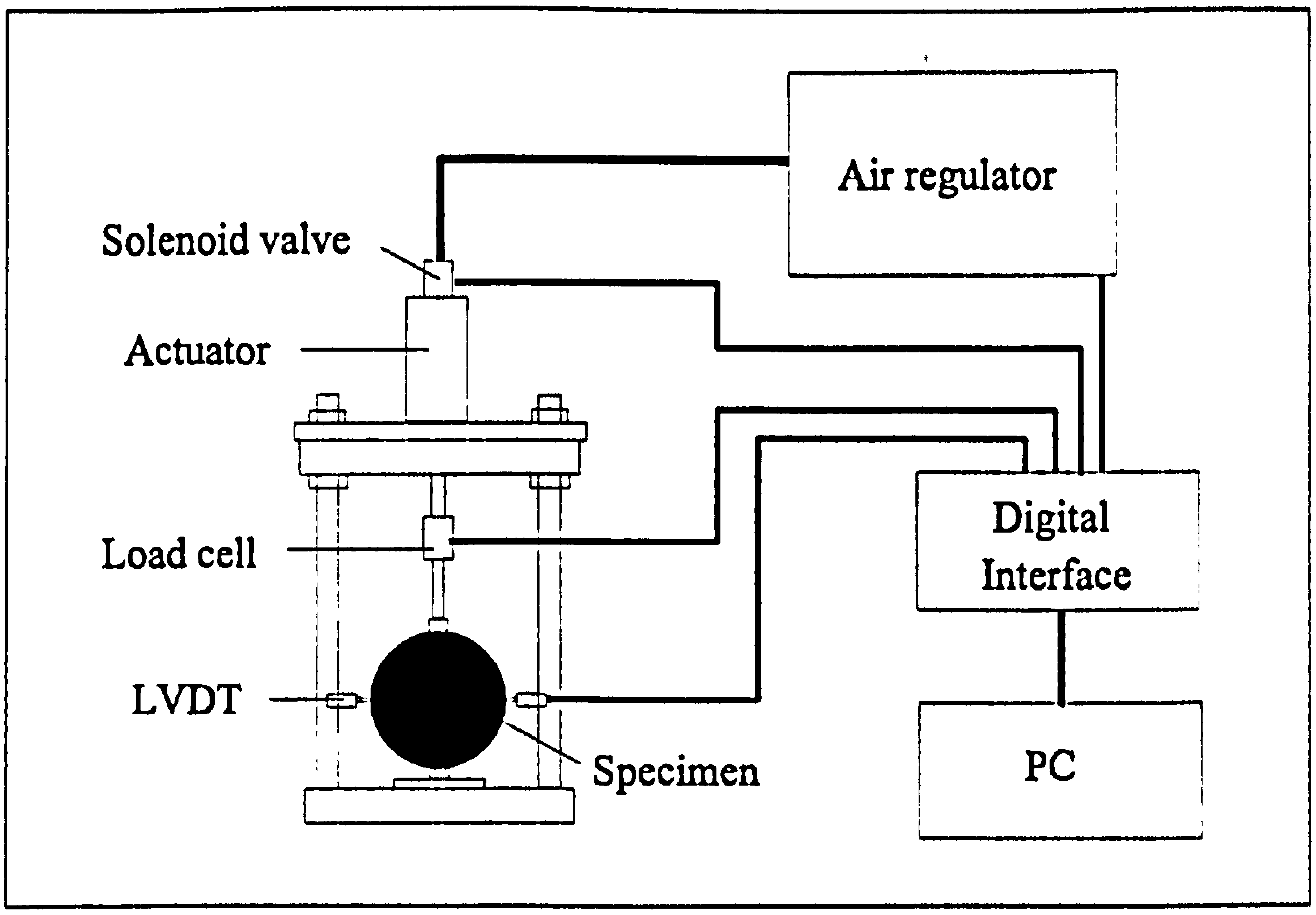


Figure 5-5 : Schematic diagram of NAT in ITSM test mode

The ITSM test can be carried out in two ways. The first allows the operator to request the NAT to apply a loading stress of a magnitude necessary to induce a desired degree of horizontal deformation in the specimen. This can range from 2 or 3 μm to ~20 μm. Obviously, the lower the deformation the less the damage to the specimen, but very small deformations can compromise the accuracy of the test as small errors are magnified. Alternatively, the operator can specify the test stress, which is useful for fatigue testing as described below. In either case, the NAT uses five conditioning pulses to self adjust and bed in the specimen. A further five measuring pulses are then applied, and the average stiffness modulus calculated.

Detailed coverage of how the ITSM test was applied in these studies will be given in the following chapter, but generally, measurements were carried out periodically on specimens with a range of mixture design parameters to assess the curing behaviour in terms of stiffness modulus. During the early part of these studies it was discovered that a problem existed in using the NAT on soft materials such as cold mix in early life. This was in the form of the load pulses

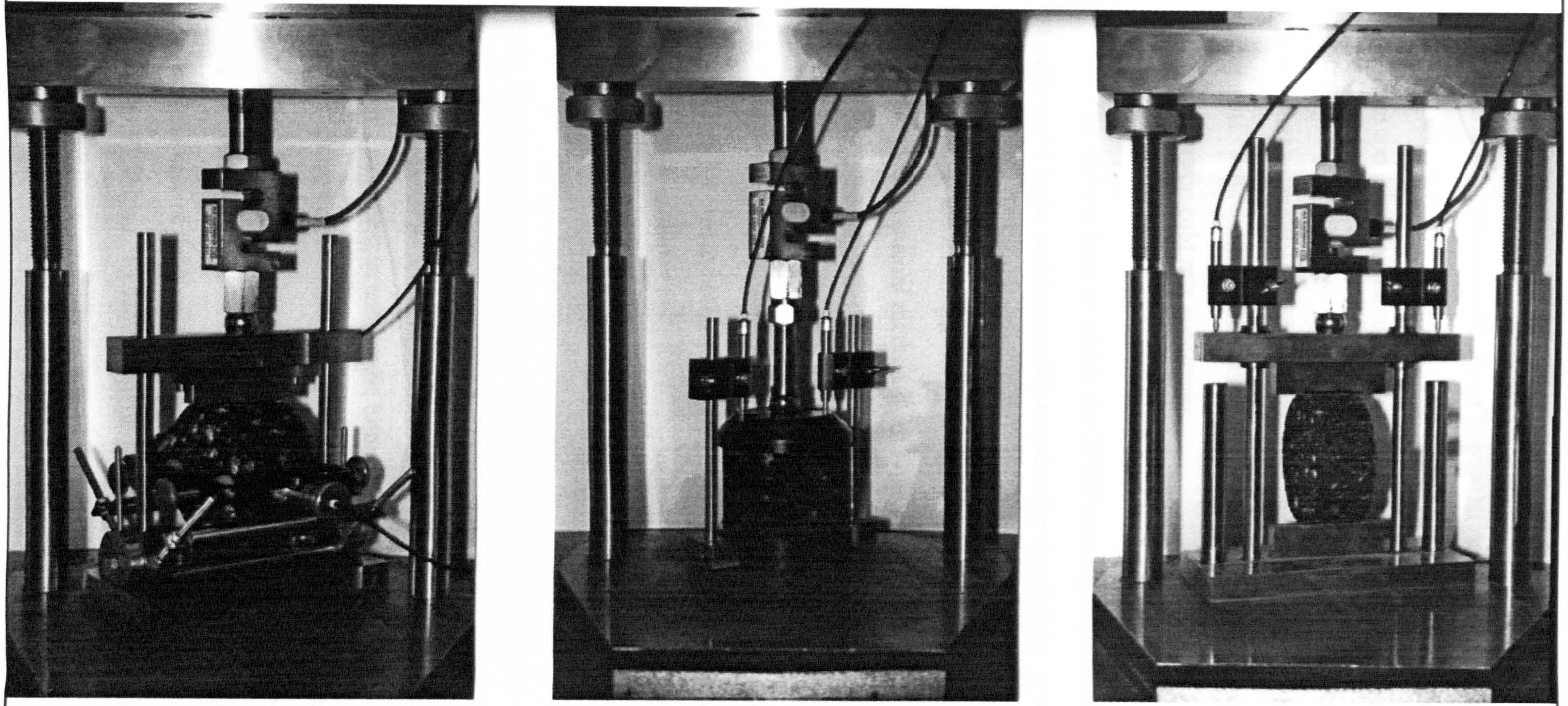


Figure 5-6 : a) ITSM test b) RLA test c)ITF test delivered during the conditioning period. In order to self adjust to give a load pulse of the correct magnitude to give the desired amount of deformation, the NAT first has to make an estimate as to how large the first pulse should be. As the NAT was originally designed for testing of hot mix material, this initial pulse was often too large for the materials being tested in these studies, resulting in various degrees of damage to the samples. It was, therefore, necessary to adjust the software to apply a very small first load and then increase the load in small increments. With these adjustments the NAT was found to be very effective for measurements on cold mix specimens.

The earliest time at which the ITSM test could be used on cold mix samples cured under ambient conditions was after 4 or 5 days. It was found that testing any earlier caused damage to the specimens and due to the lack of any elastic response, the technique was not really applicable. In order to test very young materials, a triaxial method would have been more appropriate. In this method, the material is supported as it would be in a pavement and a more accurate assessment of mechanical properties is possible. Unfortunately, the procedure is very time consuming and, therefore, it could not be employed in these studies for which mechanical property measurements were only a part. Triaxial resilient modulus tests were emploed by Terrel and Wang (16) in order to allow measurents in early life starting from immediately after compaction and these studies have been described in chapter 4.

5.4.2 Fatigue

The application of a load to any structure imposes stresses and strains. In the case of a pavement structure, the bituminous layers can be considered to be beams, which experience localized loading due to the passage of vehicle wheels. The resulting tensile stresses imposed on the layer at various points gives rise to tensile strains as depicted in Figure 5-8. It can be seen that tensile strains are present at the base of the layer directly beneath the wheel load, and at the surface either side of the load. Repeated tensile strains such as these, which stretch the binder matrix, have the potential to cause cracking.

Regions of tensile strain are liable to cracking if the load exceeds the tensile strength of the material in question. Additionally, even if the strain does not reach this level, repeated loading can lead to cracking through a process known as fatigue. Fatigue is traditionally defined as (107)

> *"The phenomenon of fracture under repeated or fluctuating stress having a maximum value generally less than the tensile strength of the material".*

The notion has been put forward in the UK, that cracking, can also be caused by strains resulting from thermal effects. Therefore, a broader definition has recently been put forward (107) but loading is generally still considered to be the predominant factor :

> *"Fatigue in bituminous pavements is the phenomenon of cracking. It consists of two main phases, crack initiation and crack propagation, and is caused by tensile strains generated in the pavement by not only trafficking but also temperature variations and construction practice."*

There are two stages to cracking (107). The first is crack initiation. The number of load cycles taken to initiate a crack is dependent on the intensity of the strain which is in turn dependent upon the stiffness of the mixture. An additional complication can be the presence of crack raisers which are tiny fissures in the surface that promote crack initiation. The process which follows crack initiation and leads to failure of the pavement is known as crack propagation. This stage is also dependent on the tensile strength of the binder in the mixture as well as strain levels.

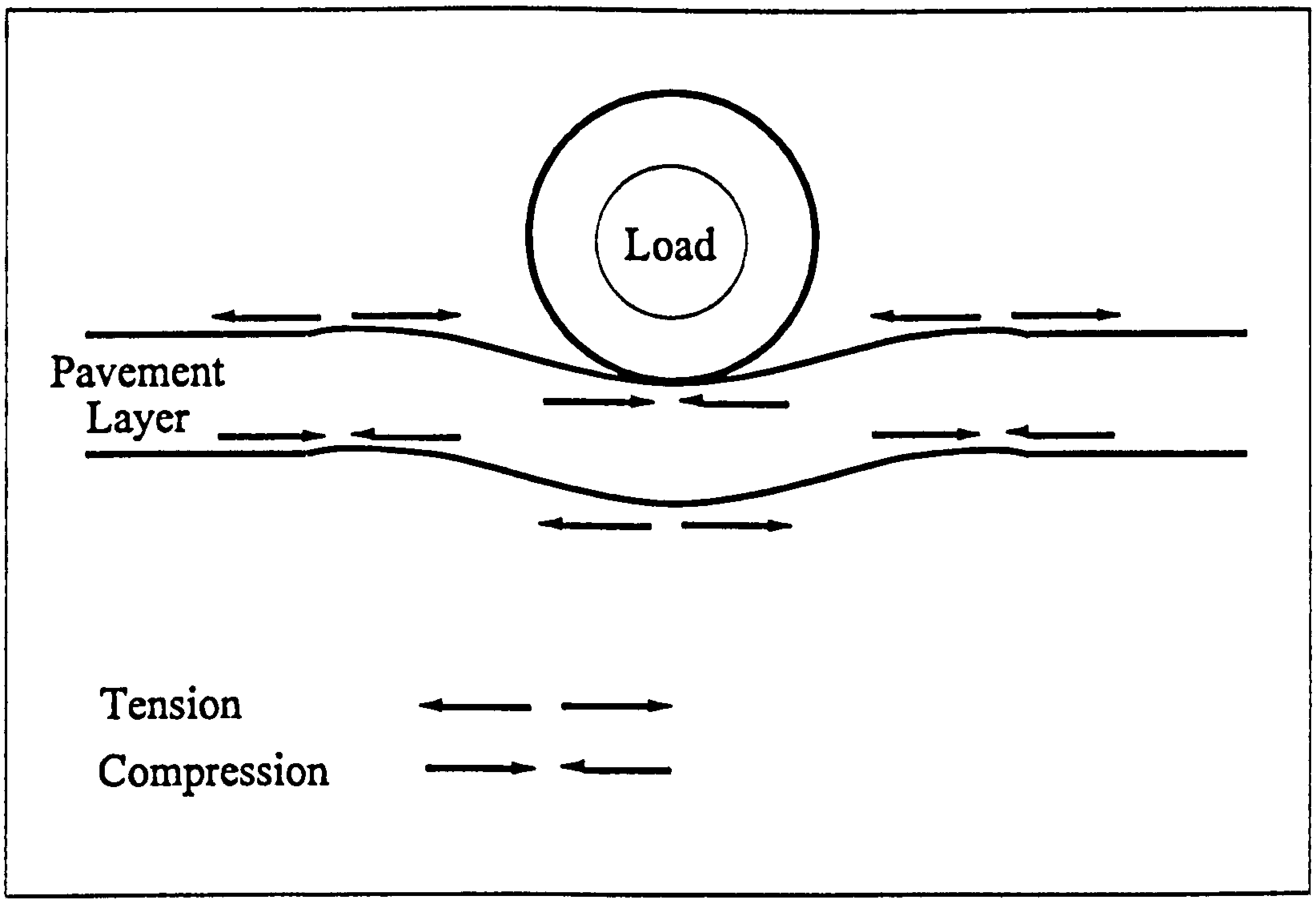


Figure 5-8 : Strains in a loaded pavement layer

Previous studies (118, 119) have shown that strain is the principal fatigue criterion for crack initiation rather than stress. As stated above, stiffness is partly dependent on temperature and speed of loading. By plotting results of fatigue tests in terms of number of cycles to failure against strain, it was found that results for different stiffness coincide. This indicates that strain is the criterion of failure and that the effects of temperature and speed of loading can be accounted for by their effects on stiffness. This is true only up to the point of crack initiation but in some mixtures (ie containing polymer modified binders) crack propagation can be a significant factor.

The magnitude of the tensile strain is dependent upon the stiffness of the layer, the stiffness of the underlying layer (ie the amount of support) and the load. The greater the stiffness of the layer and sub-layer and the smaller the load, the greater the resistance to fatigue cracking.

Fatigue testing

Fatigue testing is traditionally carried out using either the bending beam or the trapezoidal test (bending of a cantilever) which both give equivalent and reproducible results (107). These tests do, however, have the disadvantage that samples are difficult to manufacture. Kennedy was the first to use an indirect tensile method of fatigue testing, which used cylindrical specimens (120). Research has recently been carried out by Read et al. to refine the test using the NAT (113). The Indirect Tensile Fatigue Test (ITFT) has been developed as a result of this work. The method is now the subject of a draft version of a British Standard (121).

The Indirect Tensile Fatigue Test

As discussed above, the important parameter in fatigue failure is strain. For this reason, it is this factor which is utilized in the ITF test. It has been shown that the maximum tensile strain at the centre of a cylindrical specimen loaded across its diameter can been calculated using the following equation (107):

$$\epsilon_{xmax} = \frac{\sigma_{xmax} \cdot (1+3\upsilon)}{S_m} \cdot 1000 \qquad \text{(5-5)}$$

where :

ϵ_{xmax} = maximum tensile horizontal strain at the centre of the specimen (μstrain)

σ_{xmax} = Maximum tensile stress at the centre of the specimen (kPa)

υ = Poisson's ratio

S_m = Indirect Tensile Stiffness Modulus at σ_{xmax} (MPa)

The maximum tensile stress at the centre of the specimen can be calculated using the following equation :

$$\sigma_{xmax} = \frac{2 \cdot P}{\pi \cdot d \cdot t} \qquad (5\text{-}6)$$

where :
P = Vertically applied line loading (kN)
d = Diameter of the test specimen (mm)
t = Thickness of the test specimen (mm)

The values for the sample dimensions and the applied load are found by simple measurement with a ruler or micrometer. The indirect tensile stiffness modulus for the sample to be tested, at the test stress, must be measured using the ITSM test. In earlier versions of the NAT, it was necessary to measure the stiffness modulus of each core or moulded specimen at a range of target strains and construct a graph of stiffness modulus against stress for each sample. The theoretical stiffness modulus of the sample at the fatigue test stress could then be read off the graph.

Though serviceable, several problems existed with this method, and one in particular was found to be problematic for testing of materials in the scope of these studies. Firstly, the process was very time consuming. The main disadvantage, however, was that at least eight ITSM measurements had to be carried out on a specimen before it could be fatigue tested, in order to obtain an accurate graph. Even though the ITSM test is considered to be non destructive, at high stress, with soft materials, it was found that during the course of the test the stiffness would decrease dramatically indicating that the sample was being damaged. Further measurements on the same sample revealed that this damage was permanent. It was, therefore, impossible to define the stiffness modulus of a specimen at a selected stress with any degree of accuracy. Fortunately, a new version of the ITSM software became available during the course of this work, which enabled controlled stress ITSM measurements to be carried out. This meant that the stiffness of a specimen could be measured once at the stress level to be used in the fatigue test, imposing minimum damage on the sample. If necessary, the ten load pulses delivered during the ITSM test could be included in the number of cycles to failure figure obtained from the fatigue test.

To quantify the resistance to fatigue of a material using the ITFT, several samples are subjected to repeated loading at a range of initial strains. The ITFT actually operates under controlled stress. This means that the operator defines the stress to be applied in the test and this level is maintained throughout. The actual resulting initial strain is calculated using equations 5-5 and 5-6 above. As the stiffness of the specimen decreases, the strain on the specimen increases as the stress remains constant. Consequently, the test mainly measures the life to crack initiation and the period of propagation is very short. Alternative protocols use controlled strain. In these cases, as the specimen weakens, the applied stress must decrease accordingly and thus a large proportion of the test measures crack propagation.

In contrast to the traditional methods, the ITF test allows cylindrical specimens of 10 cm diameter to be tested which can be easily cored from the road or fabricated in the laboratory. In the test, the vertically placed specimen is repeatedly loaded across its diameter with a prescribed stress, whilst the vertical deformation is monitored. The load pulse consists of a sinusoidal waveform, normally with a rise time of 120 ms and a space of 1.5 s between stress peaks. With materials of high stiffness requiring high loading levels, it was found that this had to be increased to 2 s to allow time for the pneumatic system to recover between pulses. A sample is considered to have failed when the deformation exceeds 8 mm and the number of load cycles applied is taken as the cycles to failure. The test layout is illustrated in Figure 5-9.

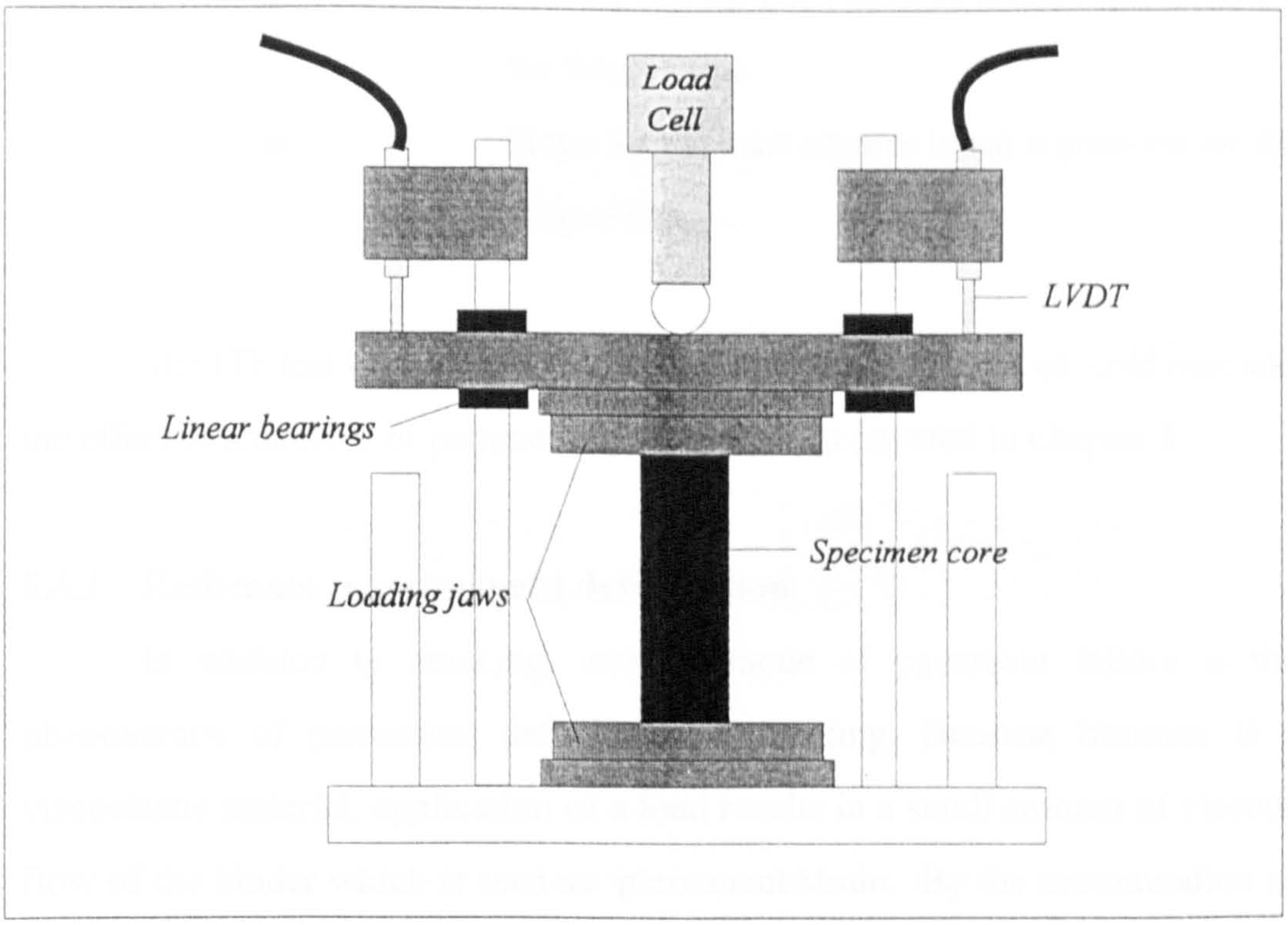


Figure 5-9 : ITF test layout

The fatigue line

A fatigue line is constructed from the results of a number of samples tested at a range of applied stresses. Tensile strains are calculated using equations 5-5 and 5-6 and plotted on the y axis against number of cycles to failure on logarithmic axes. A straight line is plotted through the points using least squares linear regression, and the equation for the line is found in the form shown in equation 5-7. Figures of slope and intercept are quoted along with the fatigue line as the magnitude of these figures can be used to make comparisons between mixtures.

$$\log N = (m \cdot \log \epsilon_{xmax}) + c \qquad (5\text{-}7)$$

where : N = Number of cycles to failure
log = Logarithm to base 10
ϵ_{xmax} = Maximum tensile horizontal strain at the centre of the specimen (μstrain)

c = Intercept of the least squares linear regression for the fatigue line

m = Slope for the least squares linear regression for the fatigue line

The ITF test was used to study the resistance to fatigue of cold mix and the effect of a number of parameters. This work is covered in chapter 8.

5.4.3 Resistance to permanent deformation

In addition to cracking, another mode of pavement failure is the phenomenon of permanent deformation or rutting. Because bitumen is a viscoelastic material, application of a load results in a small amount of viscous flow of the binder which is seen as permanent strain. By the accumulation of many small strains under the action of traffic, significant amounts of permanent deformation can occur. This is commonly known as rutting and is seen as depressions in the vehicle wheel tracks relative to the surrounding pavement (1, 114).

There are two ways in which permanent deformation can occur. The first is secondary compaction which results in densification of a road mixture after initial compaction. Provided that a material has been properly compacted during construction, this should not be able to occur. The second method is via a shearing action. In this case, resistance is dependent on the binder properties and internal friction of the aggregate mixture. These two modes, vertical displacement or "rutting", and horizontal displacement or "shoving" are illustrated in Figure 5-10.

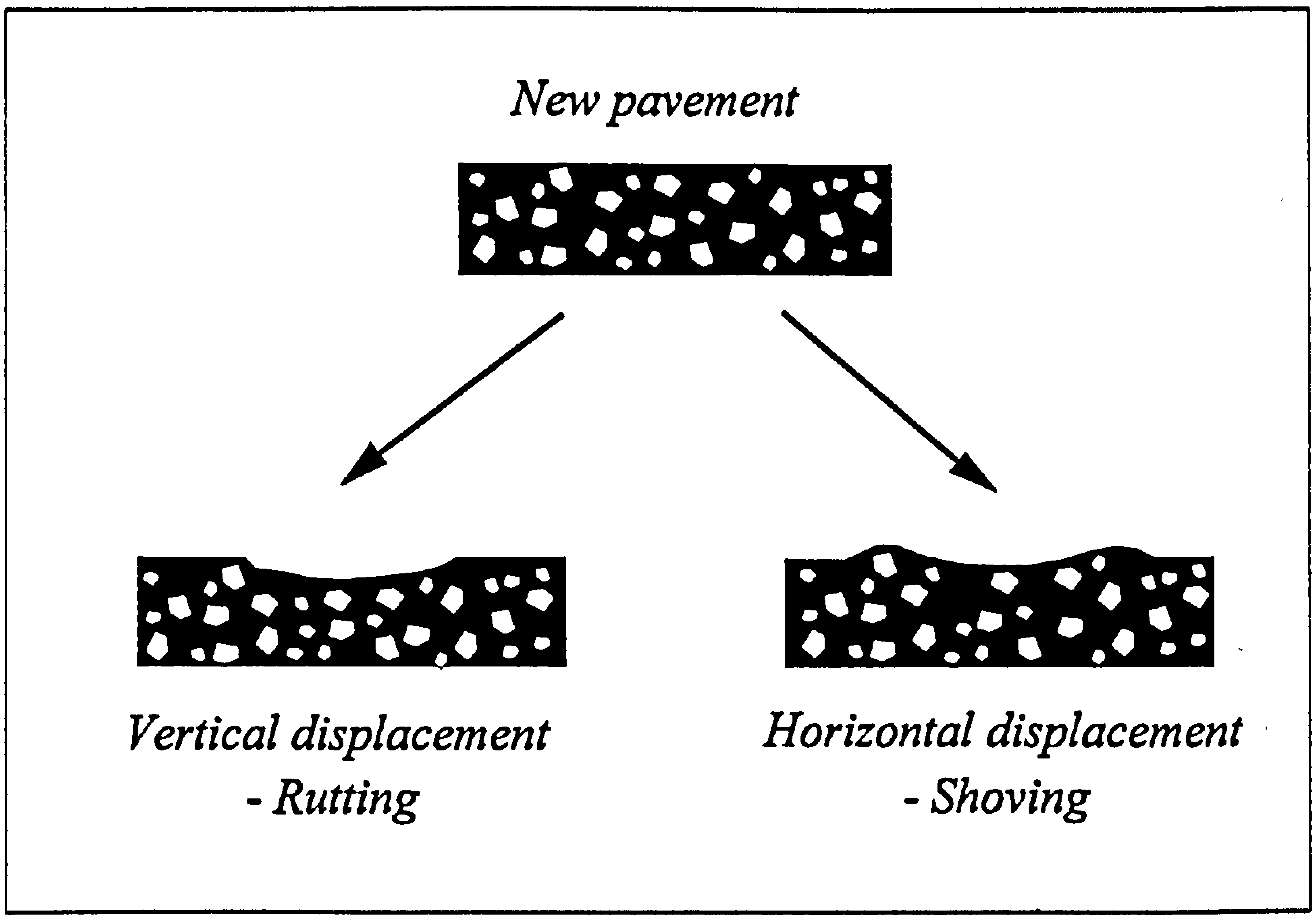


Figure 5-10 : Diagram to illustrate modes of permanent deformation

On the road, permanent deformation is seen as unevenness in road profile and if deformations become substantial, a decrease in ride quality and even danger to road users can result. As the region of maximum stress in a road is along the wheel tracks, this is where most of the deformation occurs. Whilst permanent deformation does not represent structural failure, unless accompanied by cracking, in the UK a 10 mm rut is classed as a critical situation requiring remedial action and a 20 mm rut corresponds to "failure" (114).

Pavement design may also be a factor in permanent deformation. A sub-base which has been inadequately compacted or becomes water-logged may deform, resulting in rutting of the overlying surface due to a lack of support. However, this is outside the scope of these studies.

The Repeated Load Axial test

In addition to ITSM and resistance to fatigue testing, the Nottingham Asphalt Tester can also be used to carry out resistance to permanent deformation tests (107, 122). The protocol which has been developed is known as the

Repeated Load Axial (RLA) test.

As with all NAT based tests, cylindrical samples are used and these can be 100 or 150 mm in diameter with depths of preferably 60 to 100 mm. In the test, the specimen is located vertically between upper and lower loading platens, which are slightly wider than the sample and are made of hardened steel. Various test temperatures can be used but most commonly 30 or 40 °C are used. An elevated temperature is used to lower the viscosity of the binder thereby accelerating the test. A repeated load, of 100 kPa in magnitude is applied axially, whilst the vertical deformation of the sample is measured. The load pulse consists of a square wave form with a frequency of 0.5 Hz (ie 1 s on, 1 s off). This simulates the action of slow moving traffic which is the situation which leads to the most deformation in a real road. The full test consists of 3600 load pulses, but if the sample deforms more than 8 mm during the test the sample is considered to have failed and the test is stopped. The test layout is illustrated in Figure 5-11.

Until recently, the RLA test has not used confinement on the test briquette. In this situation there is a large reliance on stiffness and tensile strength of the binder. As material in an actual road situation would be supported in all directions (except from above) this is a rather artificial and severe situation. Confinement of a mixture mobilizes the internal aggregate interlock, arising from friction between particles, by keeping aggregate grains in contact under the action of a shearing force (Figure 5-12). If confinement is not present, there is no mechanism to provide this action. A system has been developed at Nottingham to maintain confinement in the form a rubber sheath which is placed over the specimen (107). External pressure is simulated by applying a partial vacuum to the system via a porous lower platen which sucks in the rubber sheath. At the time of these studies this equipment was not available and so the work carried out did not use sample confinement.

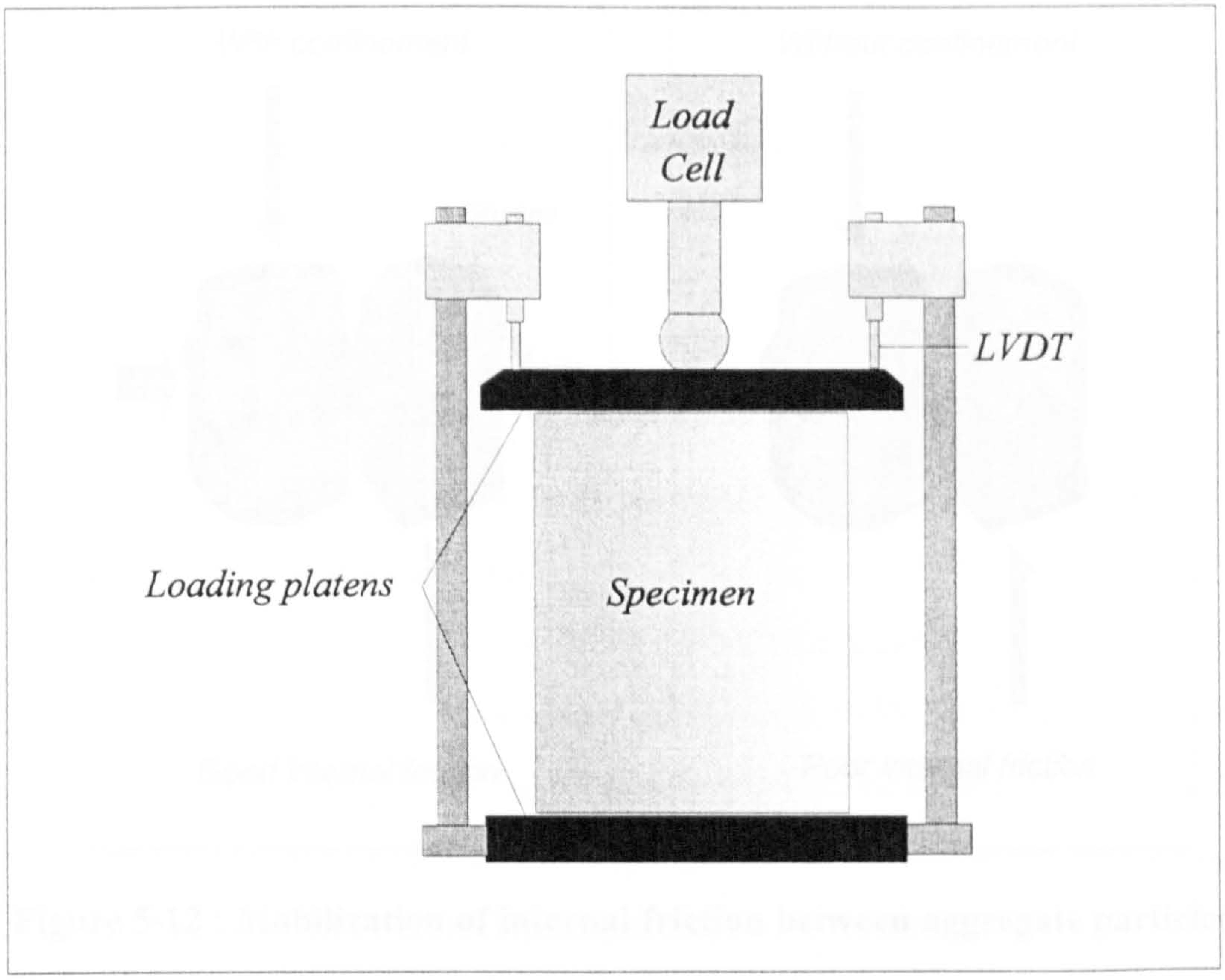


Figure 5-11 : Layout of the RLA test

The output from an RLA test consists of vertical deformation of a sample plotted against number of load pulses. There are various ways to look at this data, the simplest of which is merely to plot vertical deformation on the y-axis against number of load pulses on the x-axis.

An alternative method of data presentation is to plot the deformation per load cycle, or strain rate, against number of load cycles. Some workers have also suggested calculation of the average strain rate over the entire test or the minimum strain rate which is indicative of performance of a mixture over the majority of its life (123). A problem with the average strain rate is that it is weighted towards the early part of the test as the majority of the deformation occurs at this stage and in fact says little about overall performance.

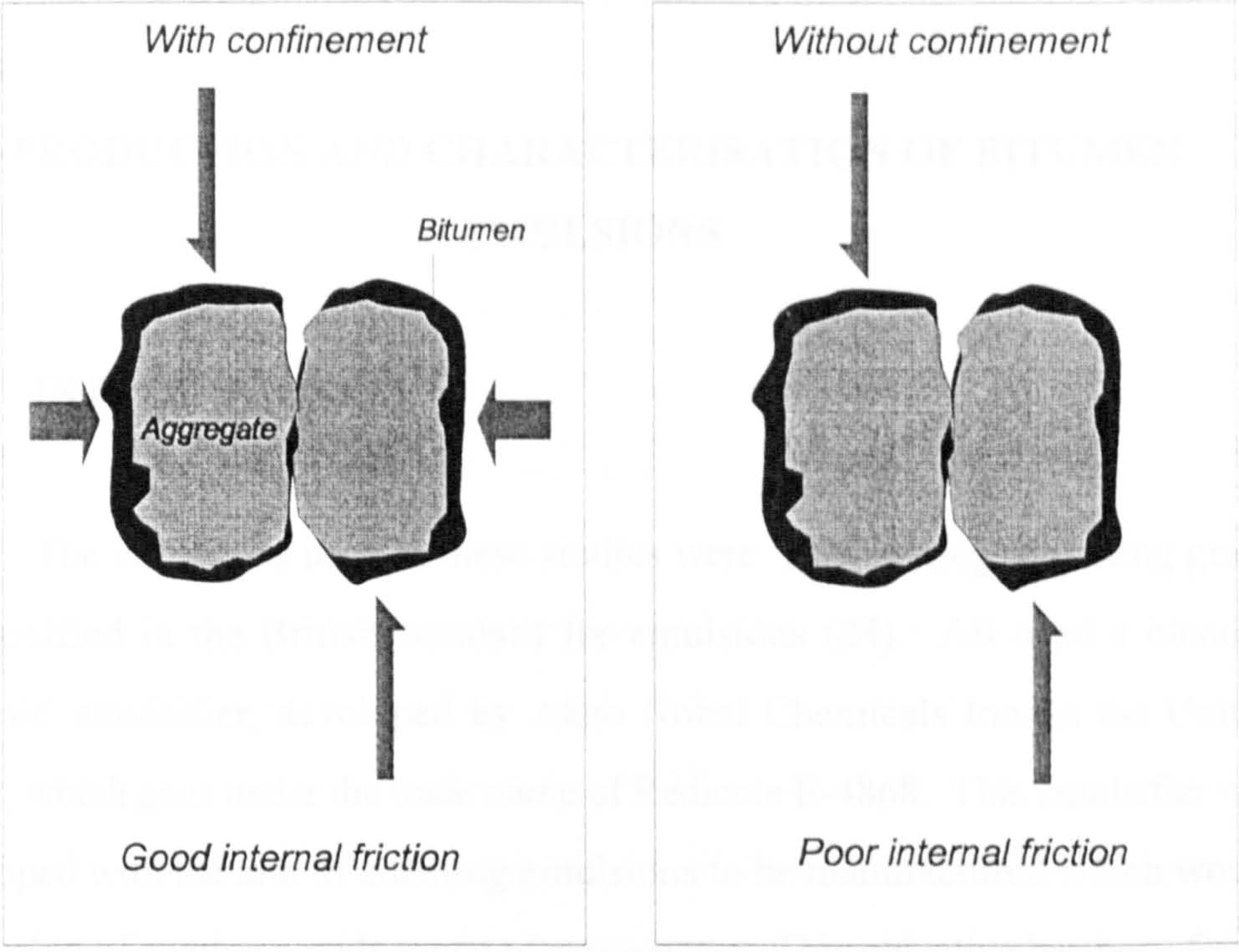


Figure 5-12 : Mobilization of internal friction between aggregate particles

5.5 SUMMARY

The test methods described above provided several means for the manufacture of specimens for testing. The NAT based tests enabled comprehensive determination of the mechanical properties of mixtures to be carried out using one piece of apparatus. The manufacturing and test methods described above were used to fabricate and measure the mechanical properties of specimens as described in the following chapters. It should be noted that these methods were developed for hot mix originally but were found equally as suitable for emulsion mixtures during the course of these studies. The sole limitation was that they could only be used after a certain amount of curing which meant that very early life mechanical properties could not be measured. This would have required the use of confined testing such as triaxial but this type of equipment was not employed in these studies.

CHAPTER 6

PRODUCTION AND CHARACTERISATION OF BITUMEN EMULSIONS

6.1 INTRODUCTION

The emulsions used in these studies were slow setting or mixing grade as classified in the British standard for emulsions (24). All used a blended 'cationic' emulsifier, developed by Akzo Nobel Chemicals Inc. in the United States, which goes under the trade name of Redicote E-4868. This emulsifier was developed with the aim of enabling emulsions to be manufactured which would be capable of coating a wide range of aggregates. This objective has been found to be fulfilled through laboratory testing and use of the product in the field, as the product had been successfully used for a number of years. However, very little data exists on the mechanical properties of mixtures based on this system.

Emulsions for use in these studies were manufactured with a range of bitumen grades, bitumen contents, emulsifier levels and polymer additives.

6.2 EMULSION MANUFACTURE

The general method of emulsion manufacture has been described in chapter 2. This chapter describes the production of emulsions specifically for use in this research.

6.2.1 Waterphase production

Before production of an emulsion was undertaken, the weights of the ingredients required to produce the desired emulsion formulation were calculated. Emulsions are prepared from a waterphase, which contains the emulsifier plus acid, and bitumen. The weight of emulsifier, which must be dissolved in the waterphase to give the correct concentration in the final emulsion, was calculated as shown in the following example for an emulsion containing 1.2 % emulsifier:

Target binder content in emulsion : 62 %
(Target water content in emulsion : 38 %)
Emulsifier level : 1.2 %
Final weight of emulsion required : 20 kg

Weight waterphase required = 38 / 100 * 20000 = 7600 g
Weight emulsifier required = 1.2 / 100 * 20000 = 240 g

The waterphases were prepared by dissolving the desired weight of Redicote E-4868 emulsifier in the correct amount of water It was necessary to warm the dispersions slightly to assist dissolving of the emulsifiers on addition of acid. The pH was adjusted to 2.5, as determined by a pH probe, using concentrated (33%) hydrochloric acid. The waterphases were then either used immediately or covered, to prevent evaporation of water, and stored.

6.2.2 Emulsion mills

The majority of emulsions used in these studies were prepared on a Hurrell colloid mill. The original design is capable of producing several hundred litres of emulsion per hour but the version used here had been geared down to reduce the rate of output to make it more suitable for laboratory purposes. The layout of the mill in shown schematically in Figure 6-1.

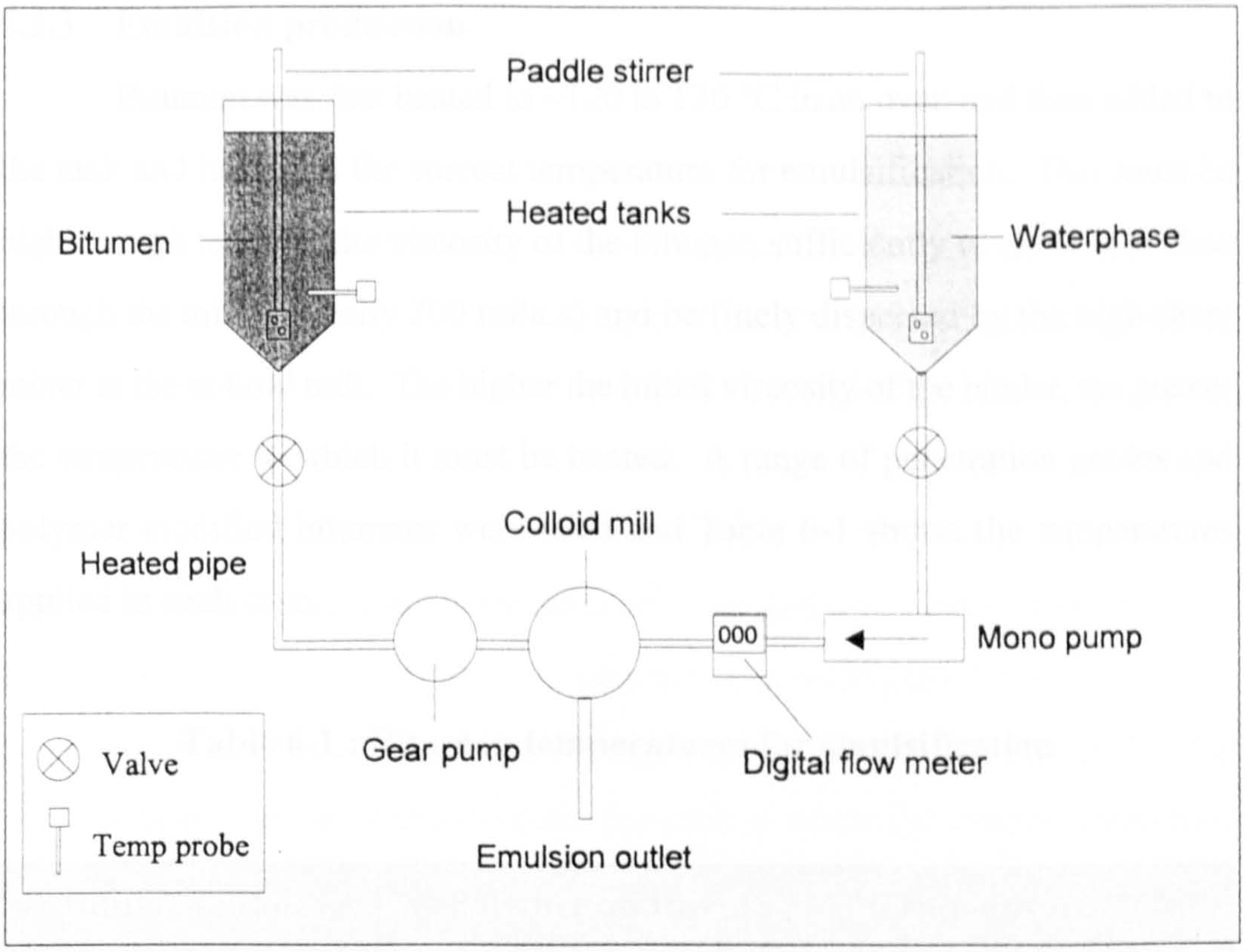


Figure 6-1 : Schematic diagram of the Hurrell colloid mill

A second mill was used later on in this work as it became available and was capable of manufacturing emulsions of smaller particle size due to the design of the high shear mixing head and the speed at which it rotates. This mill was manufactured by the Swedish company, Scanroads, which is now part of Akzo Nobel, and is called the Scanroad Emulsion Plant (SEP). This mill is more sophisticated than the older Hurrell in a number of ways. Firstly, the waterphase and bitumen temperatures and the bitumen flow rates are all monitored electronically, in addition to the waterphase flow rate. This allows better control of production. Secondly, the speed of rotation of the high shear mixing head is adjustable which facilitates manipulation of particle size distribution in the emulsion. Lastly, the mill incorporates a heat exchanger on the outlet and it is possible to exert back pressure to the mill head. Both of these features mean that emulsions can be prepared at higher temperatures which may be necessary when emulsifying very hard or polymer modified bitumens. Although this mill is very different in outward appearance, it is, in principle, very similar to the Hurrell mill.

6.2.3 Emulsion production

Bitumen was first heated to ~120 to 130 °C in an oven and then added to the tank and heated to the correct temperature for emulsification. This must be high enough to lower the viscosity of the bitumen sufficiently to allow it to flow through the mill (typically 200 mPa.s) and be finely dispersed by the high shear mixer in the colloid mill. The higher the initial viscosity of the binder, the greater the temperature to which it must be heated. A range of penetration grades and polymer modified bitumens were used and Table 6-1 shows the temperatures applied in each case.

Table 6-1 : Bitumen temperatures for emulsification

Bitumen grade (Venezuelan)	Polymer additive	Temperature (°C)
50 pen	-	140 to 150
100 pen	-	135
200 pen	-	130
300 pen	-	120
100 pen	3 % SBS	160
100 pen	3 % EVA	160
100 pen	5 % EVA	160

The prepared waterphases were added to the other tank and also heated to a specific temperature. This figure is critical, as with the Hurrell mill there is no facility to cool the emulsion after production. If the temperature of the emulsion were to exceed ~95 °C, the water would boil and, therefore, evaporate, resulting in an emulsion low in water content. Thus, the temperature must be kept low enough to ensure that the combined bitumen and waterphase temperature is less than ~95 °C, and is, therefore, dictated by the bitumen temperature. It must also be borne in mind that the combined temperature must not be too low, as this would result in stiffening of the bitumen in the mixing head making it difficult to emulsify.

Prior to emulsion production, the bitumen pipework and the colloid mill itself are heated to prevent rapid cooling of the bitumen on contact. Excessive cooling would result in the bitumen becoming too viscous to pass through the mill head

In order to produce an emulsion with the desired bitumen content on the Hurrell mill, the flow rate of the waterphase is controlled. The bitumen is pumped at a constant rate by means of a gear pump ahead of the the mill. The mill has been calibrated allowing the operator to select the correct waterphase flow rate for the desired bitumen content. Using the SEP, both the bitumen and waterphase flow rates can be adjusted and controlled. In practice, the bitumen flow rate is kept constant and the waterphase flow rate adjusted to a calculated value.

When the correct temperatures had been reached emulsion production commenced. The emulsions were allowed to cool before undergoing a series of characterisation tests as described in chapter 2.

6.3 EMULSION FORMULATIONS AND RESULTS OF CHARACTERISATION TESTS

The formulations of emulsions prepared for these studies are shown in Table 6-2 along with the results of characterisation tests.

For the purposes of these studies the emulsion containing 1.2% Redicote E-4868 and 62 % 100 pen Venezuelan bitumen was designated the standard emulsion formulation.

The particle size distribution analyses were carried out on a CILAS laser diffraction particle sizer sited at Akzo Nobel's laboratories in Stockholm, Sweden.

6.4 DISCUSSION

All of the emulsions manufactured either had bitumen contents of the required values after production or were diluted to the correct levels with waterphase if found to be high on analysis. Addition of water phase is not an ideal solution as it places free emulsifier in the waterphase instead of at the bitumen water interface. However, it is the only way in which the correct overall levels of bitumen and emulsifier can be achieved in the emulsion. The extra controllability of the SEP mill enables the correct solids to be obtained without adjustment being necessary.

The results of the particle size analysis tests showed that the majority of the emulsions contained median droplet sizes around 2 to 4 μm and maximum sizes of 20 to 25 μm. This distribution was typical of these types of bituminous emulsions. The emulsion containing SBS modified bitumen was much coarser, having a median of ~15 μm and a maximum of 42 μm which is a very significant difference. This was probably due to the difficulty in dispersing the harder binder during emulsification. No attempt was made to produce a finer emulsion. The findings should be borne in mind when assessing performance in applicational tests.

Table 6-2 : Emulsion formulations and results of characterisation tests

(- standard emulsion)*

4868 level (%)	Binder pen	Additive level (%)	Binder level (%)	Particle size (µm)			Break index (g)	Viscosity (mPa.s)
				Median	10 % <	90 % <		
1.2 *	100	-	62	2.65	0.63	23.88	148.86	87.3
"	50	-	"	3.43	0.77	20.79	135.84	76.9
"	200	-	"	2.61	0.66	6.37	128.76	95.8
"	300	-	"	3.91	0.7	20.13	112.67	95.2
0.8	100	-	"	4.88	0.99	17.27	130.9	59.4
1.6	"	-	"	3.13	0.68	6.23	159.74	77.2
2.5	"	-	"	3.07	0.66	25.72	142.8	54.1
5	"	-	"	-	-	-	151.65	77.1
1.2	"	-	70	3.03	0.7	12.66	126.8	322.4
"	"	SBS - 3	62	15.46	1.66	41.99	68.7	48.2
"	"	EVA - 3	"	2.93	0.84	19.24	135.11	49.0
"	"	EVA - 5	"	4.01	0.91	22.25	129.25	49.1
"	"	Latex - 3	"	4.2	0.85	25.51	106.86	41.7

The break index test results revealed a number of important features regarding the emulsion compositions :

- There was a general decrease in break index with increasing binder pen.
- The break indices did not increase with increasing Redicote E-4868 emulsifier level.
- Polymer modification of the binder led to a decrease in break index. This was particularly pronounced in the Latex and SBS modified emulsions but in the latter case this may be partly attributed to the increased particle size in this emulsion.

Several points are worthy of note from the viscosity results :

- Viscosity increased with increasing penetration grade of the binder.
- No trend was evident with regard to the emulsifier level.
- The emulsion with 70 % bitumen was several times more viscous than those with 62 % bitumen.
- The polymer modified emulsions were less viscous than the standard.

CHAPTER 7

FIELD TRIALS

7.1 INTRODUCTION

Laboratory tests for road mixtures are increasingly more able to predict performance in a road situation. However, there are still factors which arise in the field which can be neither foreseen nor duplicated in the laboratory. It is essential, therefore, to carry out field trials to compare laboratory performance with that obtainable in reality. The results obtained could give valuable information regarding performance or highlight practical problems which may never be encountered in a laboratory situation. During the course of this project, three opportunities to participate in trials arose. The first being a chance to carry out small plant scale mix trials and the other two to manufacture and lay significant quantities of cold mix.

7.2 PATCHING MIXTURES

At the time when these studies began, Akzo Nobel was cooperating with a small storage stable mixture producer and slurry seal contractor in an attempt to develop a cold mix for patching applications. This company had previously been producing bagged mixtures containing cut back emulsion but, due to the HAUC specifications (2) described in chapter 4, it was envisaged that these mixtures would soon become obsolete. A storable, solventless emulsion mix was, therefore, required which would comply with HAUC in terms of stiffness modulus and resistance to permanent deformation. The intention was to design mixtures in the laboratory and then attempt to manufacture them on a plant scale.

7.2.1 Laboratory tests on mixture design and performance

Aggregate samples were obtained as 10, 6, 5 mm and dust fractions. A sieve analysis was carried out and aggregate mixture designs calculated to comply with target gradings. The gradings were as shown in Figure 7-1 along with the specification for a 10 mm close graded wearing course macadam (CGWM) from BS 4987 Part I.

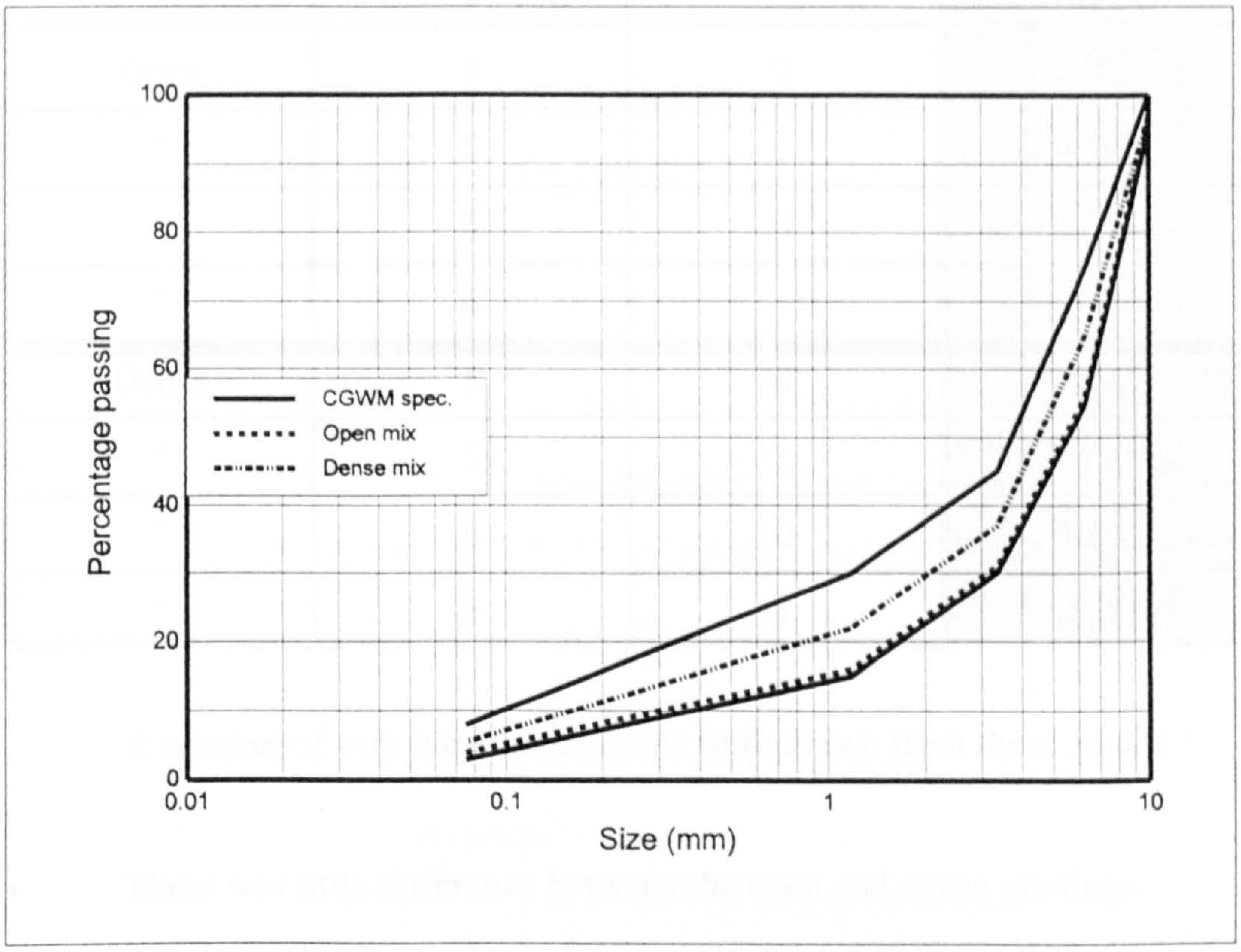


Figure 7-1 : Aggregate gradings used in patching mixtures

Mixtures were manufactured using the Hobart mixer as described in the previous chapter with formulations as shown in Table 7-1. The standard emulsion described in Table 6-2 in chapter 6 was used. No pre-wet water was used in mixtures without OPC and 1 % was used in mixtures with OPC. These levels had been determined by small scale mix tests. Samples were compacted in triplicate in Marshall moulds using a static load of 12 MPa applied for 90 s. The samples were cured in an oven at 60 °C for 5 days. The stiffness moduli of the moulded specimens were measured at 20 °C using the NAT. The results of these tests are shown in Table 7-1.

Table 7-1 : Formulations and stiffness moduli of patching mixtures

Aggregate grade	Residual binder content (% on mix)	OPC level (% on agg.)	Stiffness Modulus (MPa)
Open	5	0	2583
"	5	1	4217
"	6	0	1800
"	6	1	2433
Dense	5	0	2392
"	5	1	4125
"	6	0	1525
"	6	1	2300

A number of very clear conclusions were drawn from these results :

- There was little difference between the open and dense gradings
- The lower binder content of 5 % gave greater stiffness than the higher level of 6 %
- The addition of 1 % OPC had a beneficial effect on stiffness and this was irrespective of aggregate grading and binder content.
- Mixtures which complied with the HAUC specifications could be produced using these bitumen emulsion mixtures

It will be noted that due to these samples being cured in the oven, the results are an indication of the ultimate achievable stiffness of these mixtures only. In order to assess the performance under ambient curing conditions, further specimens were manufactured. This time the emulsion manufactured for the plant mix trials, as described below, was used in order to also check the emulsion quality. The mixture designs were based on the open aggregate grading, 5 %

residual binder and 0 and 1 % OPC. The stiffness of these specimens was measured after storage at room temperature for 24 hrs and found to be between 100 and 200 MPa; the higher levels corresponding to mixtures with OPC.

7.2.2 Plant mix trials

Following the laboratory trials, some of the mixture designs tested were scaled up to plant mix scale. The mixer used was a large sun and planet type, capable of producing ~200 kg batches, and was, therefore, very similar in principle to the Hobart mixer used in the laboratory. A 200 kg drum of emulsion was supplied by Nynas UK AB which should have been manufactured according to the standard formulation. Unfortunately, a misunderstanding led to the incorporation of 2 % Redicote E-4868 emulsifier instead of 1.2 %. As it was not possible to manufacture a second batch of emulsion quickly, the emulsion had to be used as it was.

Four mixtures were manufactured during the course of the trial with the following formulations and results :

1-A. *Dense aggregate grading, 5 % residual binder, 1 % OPC*

No prewet water was added as the aggregate was considered to be wet enough. There were no facilities available to measure the moisture content and so experience was relied upon for evaluation. The emulsion failed to fully coat the aggregate due to there being insufficient fluids in the mixture.

1-B. *Dense aggregate grading, 6 % residual binder, 1 % OPC*

Again the binder failed to spread over the aggregate despite the additional emulsion added.

1-C. *Open aggregate grading, 5 % residual binder, 1 % OPC*

Approximately 3 % water was added to this mixture prior to the emulsion.

The aggregate was over wet and the mixture became very sloppy. Mixing was continued for 10 mins in an attempt to force out some of the water which was partially successful. The final product was, however, still too moist.

1-D. *Open aggregate grading, 5 % residual binder, 1 % OPC*
Only 1 % pre-wet water was added to the aggregate in this mixture. The resulting product was similar to lab produced mixtures in appearance and was considered to be "acceptable" by all parties present

These trials highlighted at an early stage in these studies, the difficulties in transferring laboratory designed emulsion mixtures to a plant scale. The main source of error seemed to arise from the variation and indeterminability of the water content in the aggregate. Samples of mixtures 1-C and 1-D were taken back to the laboratory and compacted in Marshall moulds using a static load of 12 MPa applied for 90s.

Due to the partial success of these trials a second attempt was made. It was felt by some of the parties concerned that for these types of mixtures, the aggregate grading should be opened up to allow water to be squeezed out during compaction and evaporate after laying. Thus, different aggregate gradings were used which were based on the curve shown Figure 7-2 along with specifications for 10mm close graded (CGWM) and open graded (OGWM) wearing course mixtures from BS 4987 Part I for comparison. Two aggregate mixtures were tested; one of which adhered closely to the curve shown in the graph whilst in the other some of the stone fraction was replaced with dust to give a slightly less open graded mixture. This was done during the trials as the very open mix appeared to be unable to absorb the fluids. Hydrated lime was used in these mixtures in place of OPC as it was felt to be closer to normal practice in the road industry. The mixtures produced and the results were as described below :

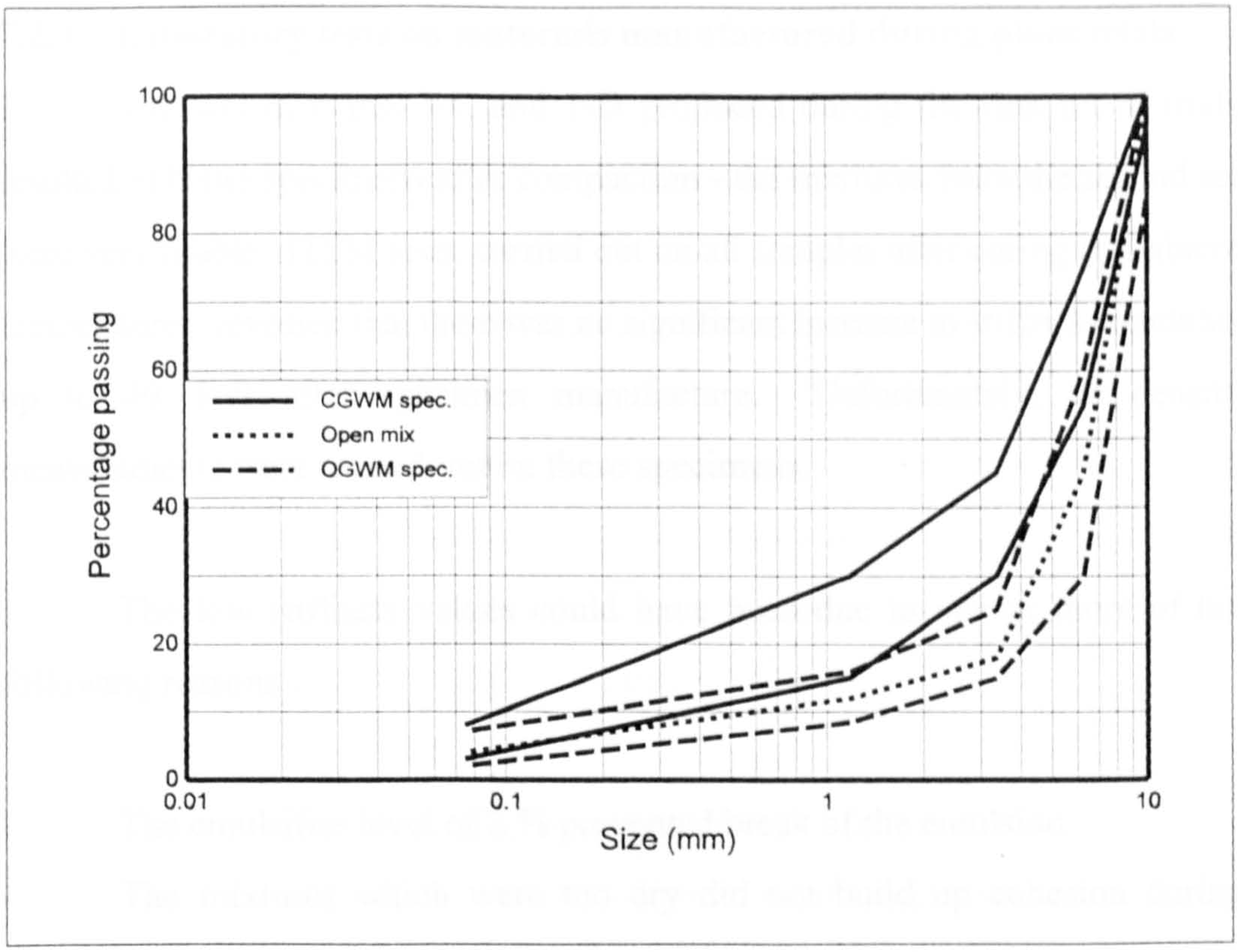


Figure 7-2 : Open graded aggregate curve used in patching mixtures

2-A. *Very open aggregate grading, 0.5 % lime, 5 % residual binder, 1.2 % water*

Mixture was over wet and emulsion was seen to be running off.

2-B. *Less open aggregate grading, 0.5 % lime, 5 % residual binder, 1.2 % water*

Emulsion appeared to be over broken and mixture was, therefore, too dry and lacked cohesion.

2-C. *Very open aggregate grading, 1 % lime, 5 % residual binder, 1.2 % water*

Lime caused the emulsion to break to a greater degree compared with mix 1 and the mix was considered to be too dry.

Samples of the mixtures manufactured in these trials were taken back to the laboratory and compacted in Marshall moulds using a static load of 12 MPa applied for 90 s.

7.2.3 Laboratory tests on materials manufactured during plant trials

The wet mixtures 1-C and 1-D produced during the first set of trials resulted in better specimens after compaction - the mixtures from the second set were very friable. ITSM tests, carried out on all samples after curing at ambient temperatures, revealed that there was no significant increase in stiffness modulus up to 40 days after specimen manufacture. Unfortunately, no density measurements were carried out on these specimens.

The low stiffness values could have been due to one or more of the following reasons :

- The emulsifier level of 2 % prevented break of the emulsion
- The mixtures which were too dry did not build up cohesion during compaction
- The void content of the dry mixtures may have been high due to low workability of the mixtures
- The mixtures which were too wet were not properly compacted due to hydraulic resistance
- The open graded aggregate mixtures were lacking in structural interlock

7.2.4 Conclusions

These studies were not successful in developing a usable cold mix for patching purposes on a plant scale, despite the encouraging results found in the laboratory tests carried out prior to the plant trial. However, they were useful in highlighting the need for a more pragmatic and scientific approach to the problem and tighter control of variables on a plant scale. Additionally, the use of OPC was found to be very effective in the laboratory and it was unfortunate that it was not tested in the field.

7.3 BASECOURSE TRIALS

Shortly after the plant mix trials described above there was opportunity to manufacture and lay several tonnes of cold mixed basecourse. The TRL (Transport Research Laboratory), in cooperation with BACMI (British Aggregate Construction Materials Industries), the RBA (Refined Bitumen Association) and the DoT (Department of Transport), were planning a trial for bitumen emulsion mixtures. The aim was to evaluate products currently available or under development in the UK. It was decided that a mix should be designed and included in the trial as part of these studies.

Before any work began, partners were found for manufacture of the emulsion and the mixture. Nynas UK AB and CAMAS Aggregates at Croft were approached to make the emulsion and mixture respectively. Akzo's role in the partnership was to provide the emulsifier and carry out all of the mixture design and testing, as part of the studies being undertaken by the author.

7.3.1 Mixture design

It was decided from the beginning of the work that the emulsion should be based on 100 pen bitumen to the formulation described as the standard in Table 6-2 and that the aggregate grading should be based on the 20 mm DBM envelope from BS 4987 Part II (9).

Aggregate gradation :

The aggregate from CAMAS Croft's quarry is granitic. Aggregate samples of the nominally 20, 14, 10, 6 mm and dust fractions were obtained and two mixture designs formulated as shown in Figure 7-3. The intention was to look at one mixture in the middle of the specification and another on the open graded edge : the idea being that more water could be squeezed out during compaction allowing better densities to be achieved and also that mix water would evaporate more easily after compaction causing the mixture to cure more rapidly. All aggregate fractions were fully dried in an oven at 120 °C before use, to enable the water content in the mixtures to be tightly controlled.

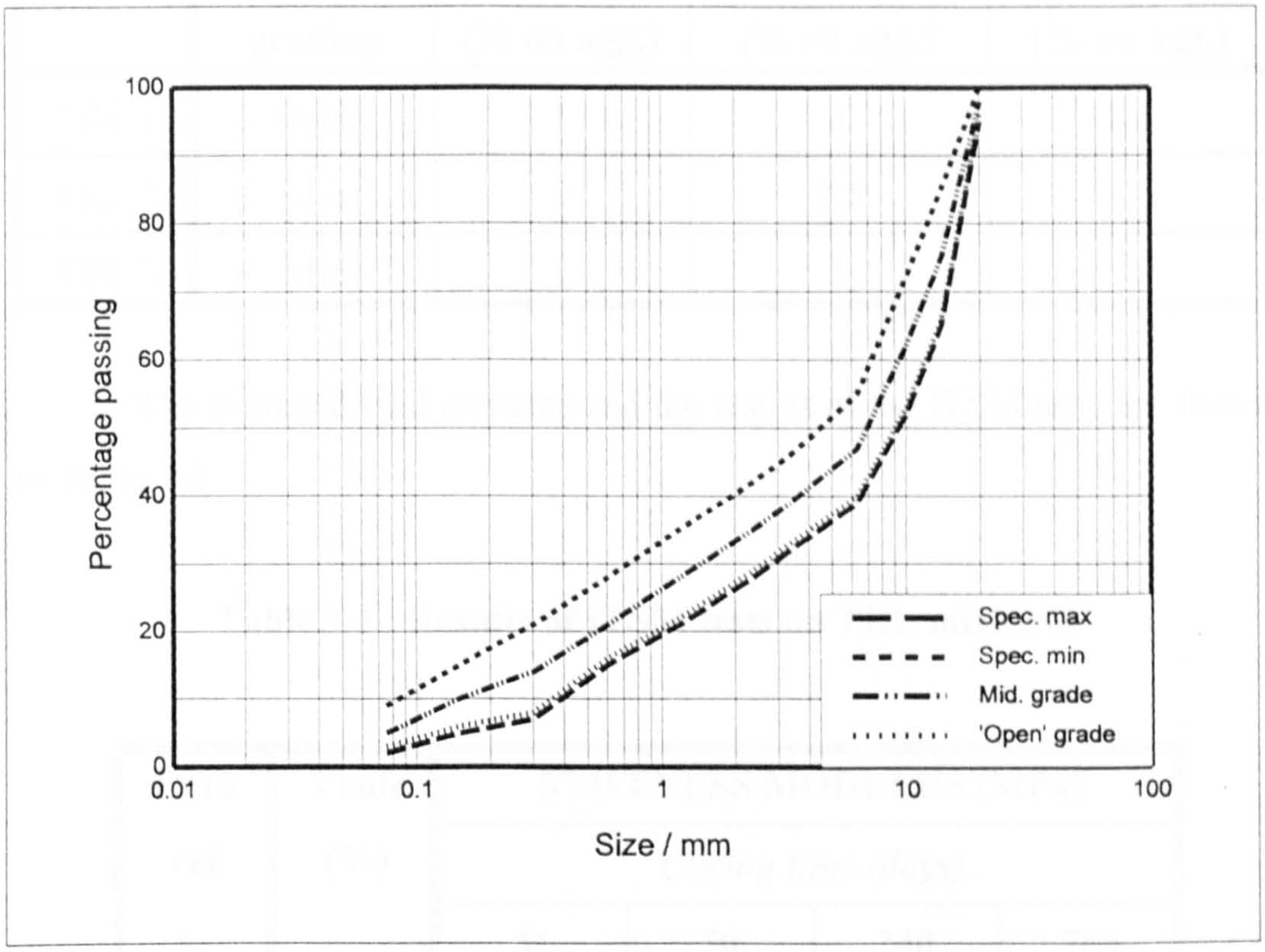


Figure 7-3 : 20 mm DBM aggregate grading envelope

Three mixtures were manufactured with the formulations shown in Table 7-2. The pre-wet water level had been optimized in advance by carrying out small scale mix tests. The emulsion level of 8.1 % corresponded to a residual binder content of 4.7 % on the total mixture which is the level specified in BS 4987 (9) for a 20 mm DBM hot mix. Five 5 kg batches of each mixture were manufactured using a Hobart mixer and compacted on the University's roller compactor as described in the previous chapter. Slabs were stored at ambient temperature for 1 month before coring was carried out. The six cores from each slab were then also stored at ambient but uncontrolled temperature and tested periodically on the NAT using the ITSM test. The ambient temperature was around 20 °C during the daytime falling to 10 to 15 °C at night

Table 7-2 : Mixture designs

Mix ref	Aggregate grading	Cement (% on agg.)	Pre-wet water (% on agg.)	Emulsion (% on agg.)
TRL 1	Open	1	3	8.1
TRL 2	Mid	0	2.5	"
TRL 3	Mid	1	3	"

The averaged void contents and the results of the ITSM tests are shown in Table 7-3.

Table 7-3 : Results of ITSM tests on TRL mixtures

Core ref	Voids (%)	STIFFNESS MODULUS (MPa)			
		Curing time (days)			
		31	50	240	700
TRL 1	14.88	600	800	800	1300
TRL 2	11.18	250	500	850	1700
TRL 3	12.3	900	1500	2100	3000

It was evident from these tests, that on the basis of stiffness modulus alone, the best mix design for the trial was that with the mid spec aggregate grading and 1 % OPC. The voids content of the specimens were considered to be very high compared with those typical of equivalent hot mixed materials.

Binder content :

Once the optimum aggregate grading had been identified, attention turned to the binder content. Mixtures were prepared in batches of 3600 g with the mid BS 4987 20 mm DBM aggregate grading, 1 % OPC, 3 % prewet water and emulsion contents to give residual binder contents in the range 4 to 6 %. 1200g portions of these mixtures were compacted in triplicate using 50 blows of the Marshall hammer to each side of the specimen. Compacted specimens were cured at 20 to 25 °C for 15 days and then tested on the NAT in the ITSM mode.

The averaged stiffness moduli of the moulded specimens were as shown in the graph in Figure 7-4.

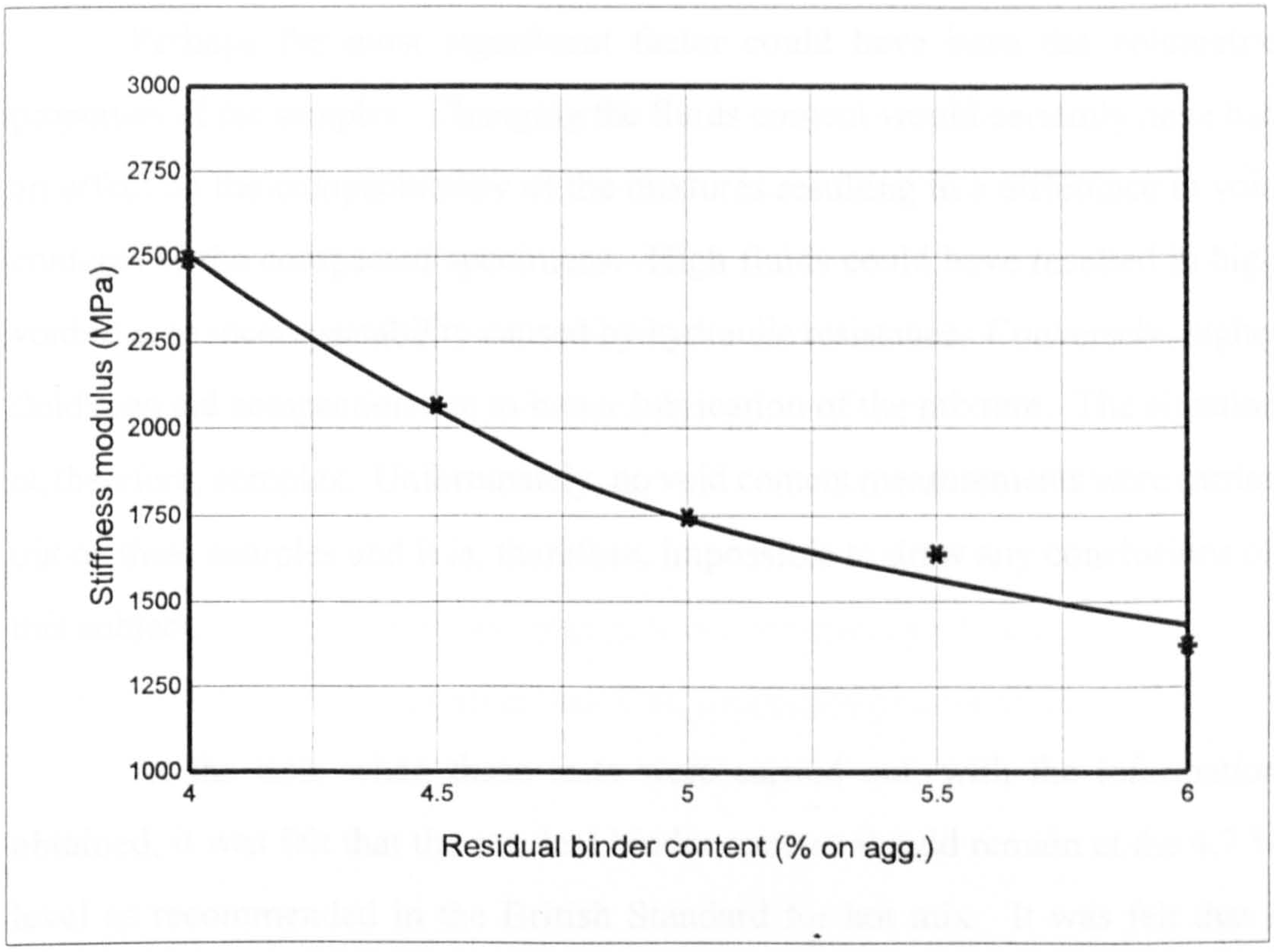


Figure 7-4 : Effect of binder content on stiffness modulus

It can be seen from the graph that the higher the binder content, the lower the stiffness modulus. This was a surprising result as in the case of hot mix, there is normally an optimum value in the 4.5 to 5.5 % range (106). The difference found here could be due to several factors. Firstly, the stiffness moduli of these specimens were measured after only a short curing period. According to results from tests described later in this report, at this stage, the binder would be in a partially cured state. It seems reasonable to assume that the greater the amount of semi-cured binder in the mixture, the lower the stiffness is going to be.

Secondly, and on reflection quite wrongly, the same pre-wet water level was used in all of the mixtures despite an increase in the level of emulsion. The level used was 3 % which had been found to be the optimum for the 4.7 % residual binder content. As the total fluids content increased in the mixtures it should have been possible to reduce the pre-wet water level. It is suggested that a high level of water would have resulted in higher void contents and slower break of the emulsion and cure of the mixture.

Perhaps the most significant factor could have been the volumetric properties of the samples. Changing the fluids content would certainly have had an effect on the compactability of the mixtures resulting in a difference in void contents of the compacted specimens. High fluids could have resulted in high voids due to incompactability caused by hydraulic resistance. Conversely, higher fluids can aid compaction due to better lubrication of the mixture. The situation is, therefore, complex. Unfortunately, no void content measurements were carried out on these samples and it is, therefore, impossible to draw any conclusions on this subject.

At the time when these tests were carried out, with the information obtained, it was felt that the residual binder content should remain at the 4.7 % level as recommended in the British Standard for hot mix. It was felt that a reduction in binder may have ramifications in terms of resistance to fatigue and durability but no measurements were carried out to verify this due to time constraints. There was no apparent justification for increasing the binder content.

7.3.2 Transportability tests

The mixture produced for the trial had to be transported ~150 miles, which was a 4 hour journey by truck, due to the locations of the mix plant and the TRL trial site. It was, therefore, thought that tests should be carried on the mixture to confirm that it could be transported without deteriorating. The input of mechanical energy due to transport could have had the effect of breaking the emulsion, causing the mixture to set before it could be layed. The main distress modes the mixture could experience were predicted to be compaction by weight in the stockpile, vibration and run-off of emulsion. The following tests were carried out to study the likely effects of these parameters.

Compaction in stockpile

The maximum load under a 20 tonne stockpile of about 1.5 m in height was calculated and found to be roughly equivalent to a 25 kg load on a Marshall sized specimen. A 1200 g portion of the mixture with the recipe to be used in the trials was manufactured, placed in a Marshall mould and loaded with 25 kg for 6 hrs. This time period was used as it was likely that the mixture would be stockpiled for some time before and after transportation. After this time the material was broken up by hand and then compacted using 50 blows of the Marshall hammer to each side of the specimen.

Shaking

A portion of mixture was placed in a container and shaken for 4 hours at a frequency of 5 Hz. The material was then compacted in a Marshall mould using 50 blows of the Marshall hammer to each side of the specimen.

Run-off

A portion of mixture was placed on a 600 μm sieve and vibrated for 4 hours at a frequency of 50 Hz. The sieve and contents were weighed before and after shaking and no weight loss occurred. The mixture was compacted in a Marshall mould using 50 blows of the Marshall hammer to each side of the specimen

Control

In addition to the samples subjected to the conditioning regimes described above, a further specimen was manufactured in the same manner from mixture which had been left to stand for four hours without distress as a control.

All of the moulded specimens were cured at ambient temperature for 2 weeks. Stiffness moduli were measured in the NAT at 20 °C.

Results and discussion

The results from the transport stability tests are shown in Table 7-4.

Table 7-4 : Results from transport stability tests

Test	Comments	Stiffness modulus (MPa)
Control	-	1521
Load	Slight cohesion after loading but easily broken up	1069
Shake	Crust of fines on surface. Some stripping in bulk	1253
Run-off	No run-off of emulsion observed	1180

All of the distress regimes to which the samples were subjected, resulted in a decrease in stiffness moduli of the moulded specimens compared to the control sample. Simulated stockpiling had the biggest effect followed by vibration on a sieve and then shaking in a tin. It is postulated that the decrease was due to partial break of the emulsion as a result of distress which resulted in a noticeable drop in workability of the mixtures. This may have resulted in higher void contents and less cohesion in the less workable mixtures. However, in conclusion, as all mixtures remained workable and decreases in stiffness were not large, the mixture was considered to be transportable.

7.3.3 Plant scale mixture production

Prior to the date of manufacture for the TRL trial, a dummy run was undertaken to ensure that the mixture could be produced correctly on a plant scale. Several 2 tonne batches of mixture based on a mid BS 4987 20 mm DBM mixture were manufactured on CAMAS's Barber Green pug mill mixer. The aggregate fractions were first added to the mixer in the proportions necessary to produce the correct aggregate grading. As the aggregate came from the hoppers it was assumed to be more or less dry. 1% OPC was added manually to the aggregate before addition of 3 % pre-wet water and 8.06 % emulsion. A mix time of 45 s was found to be the optimum to produce a fully coated but workable mixture. Good looking mixtures were manufactured.

On the morning of the trial itself, 20 tonnes of mixture was produced using exactly the same method. The first five 2 tonne batches turned out to be very wet. It was not felt necessary nor prudent at the time to adjust the mixture formulation but later it was concluded that the aggregate feed must have contained a significant amount of water. The final five batches to be manufactured were as intended, suggesting that a dry quantity of aggregate was being fed from the hoppers. The material was loaded onto a truck in two halves - one wet and the other in spec. Due to the time constraints, it was not possible to manufacture further product and, therefore, the load was transported to TRL for use in the trial.

7.3.4 Laying at the TRL site

The material was layed on a specially prepared unbound road base at a site within the TRL grounds, along with three other products (124). The intention was to lay two lifts each with a depth of ~100 mm. The wet material was laid first and proved to be impossible to roll fully. The material which matched the target specification could not be placed on top as intended but was layed further along the test site as a single lift instead. All of the other mixtures in the trial were layed in two lifts. Compaction consisted of a combination of steel wheel rolling, with and without vibration and a pneumatic tyred roller was also employed. A thin surfacing layer of Hot Rolled Asphalt (HRA) was finally layed half way over all of the trial sections to assess the curing behaviour of the materials in covered and uncovered situations.

7.3.5 Tests carried out by TRL

TRL carried out ITSM tests on various specimens of all materials layed in the trial (124). Only those relating to the mixture provided as a result of these studies will be reported here. One set of specimens was produced by PRD compaction of samples of mixtures taken from the loads arriving on site. These were tested after storage at 20 - 25 °C for 1 week and later tests revealed that little increase in stiffness occurred after this time. The figures obtained were, therefore, taken as the ultimate achievable stiffness of the mixtures. Two sets of three cores were also taken from the trial sections, one soon after laying but only from the area not covered by wearing course and the second 17 weeks after laying from both the covered and exposed sections. The results obtained from the ITSM measurements for both the laboratory and field samples are shown in Table 7-5. The voids contents were found to be 6.7 % in the covered area and 7.5 % where exposed.

Table 7-5 : Results of TRL ITSM tests

Specimen type		**STIFFNESS MODULUS (MPa)**			
		Curing time (weeks)			
		1	**6**	**14**	**17**
Lab compacted		1360	-	1480	-
Early core		-	1350	-	-
Late core	Covered	-	-	-	1100
	Exposed	-	-	-	1190

These results revealed that the PRD compacted samples were lower in stiffness modulus than equivalent samples reported above. It can be seen that after 6 weeks in situ curing, the mixture had almost reached what TRL considered to be its "ultimate potential value" defined as the stiffness obtained from the lab cured samples. Later measurements on samples cored after 17 weeks were lower in stiffness than those cored after 6 weeks which is at first difficult to comprehend. It is thought that the mixture must have been damaged by the elements to some extent - most likely the action of frost. The presence of small amounts of water in the semi-cured material would have caused frost heave if freezing did occur, and this would have weakened the mixture. The fact that the stiffness values for cores taken from both the covered and exposed areas after 17 weeks were virtually equal suggests that the mixture was able to cure even without the evaporation of water. This was attributed to the action of the OPC in the mixture, as the other mixtures in the trial, which did not contain OPC, did not behave in the same manner.

7.4 HAUNCHING BASECOURSE TRIAL

In the second year of the studies described in this report (summer 1994), it was still necessary to evaluate the performance of mixtures under evaluation in the laboratory under field conditions. Nynas UK AB and CAMAS Aggregates agreed to participate again and Northamptonshire County Council offered a site to lay some material.

The site consisted of a road widening project involving full depth haunching construction. The total project was approximately 1 mile in length and we were designated a 100 yard stretch. The remainder of the project was to use conventional hot mix. Basecourse was to be layed in two 150 mm lifts on an unbound graded roadbase, and surfaced with either cold or hot mixed 10 mm close graded wearing course, depending on the success of the cold mix basecourse. This required the production of some 150 tonnes of cold mix. The trial site before work commenced is illustrated in the photograph shown in Figure 7-5.

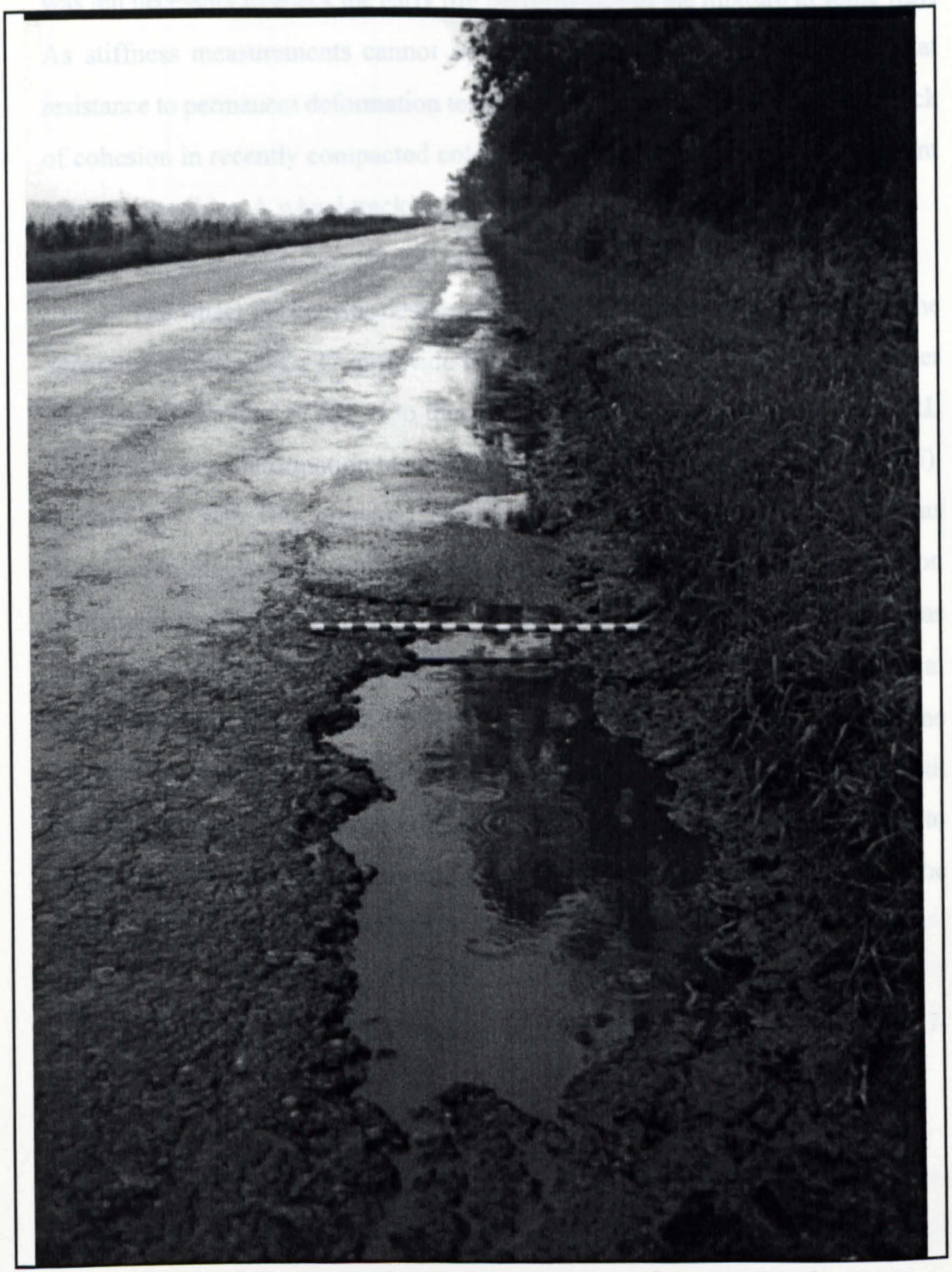


Figure 7-5 : Road edges before repair work commenced

Test of early life strength

As this trial was to be carried out on a public road and not a test track, it was felt necessary to check the early life performance of the mixture in some way. As stiffness measurements cannot be made immediately it was decided that resistance to permanent deformation tests should be done instead. Due to the lack of cohesion in recently compacted cold mix, the RLA test without confinement was not possible. A wheel tracking test was, therefore, carried out instead.

The wheel tracker was designed and built by Nottingham University. The apparatus consisted of a 47 mm wide hard rubber wheel of 204 mm in diameter which applied a load of 520 N to the slab. This regime was developed by TRL during extensive deformation testing and has virtually become a standard (125). A cold mix slab was prepared using the University's roller compactor as described in chapter 5. The mixture was prepared to exactly the same formulation as that prepared for the TRL trial. For comparison, a hot mix equivalent slab was also prepared with 4.7 % residual binder. The slabs were cured for 24 hours at ambient temperature (~15 to 20 °C) and then subjected to 5,000 passes of the wheel at a temperature of 23 °C. The wheel tracking test is normally carried out at elevated temperature to accelerate the deformation rate. It was not feasible to warm the cold mix slab as this would have resulted in accelerated curing of the mixture which was to be avoided.

The results of the wheel tracking tests are shown in the graph in Figure 7-6.

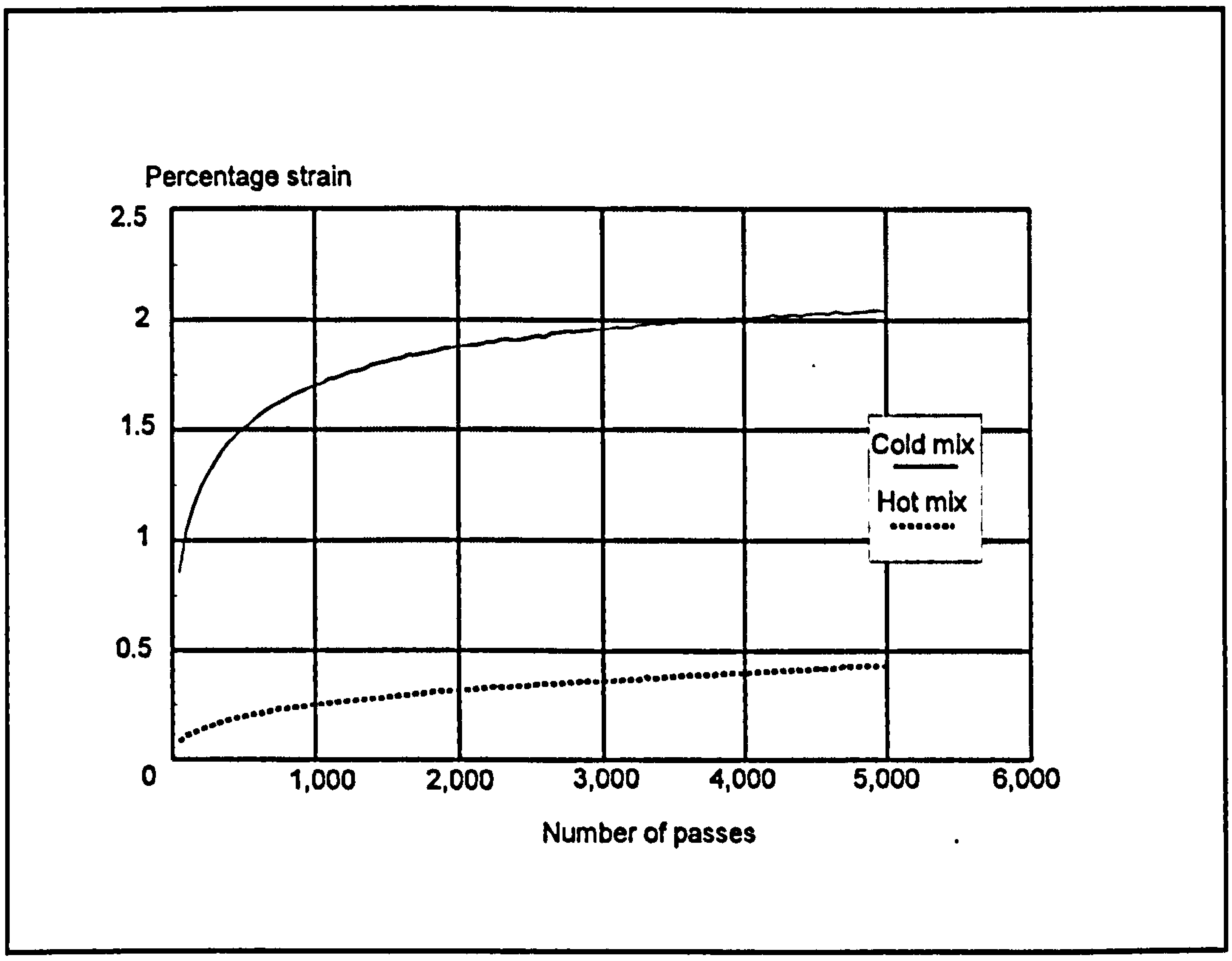


Figure 7-6 : Wheel tracking of hot and cold mix slabs

It can be seen from the graph in Figure 7-6 that the cold mix slab deformed about four times more than the equivalent hot mix. Despite this, the result for cold mix was not disastrous. Unlike hot mix which acquires its full strength immediately after cooling, the strength of cold mix increases over the first weeks and months of its life. The wheel tracking test, however, applies a lifetime's 'traffic' in a period of a few hours. This is, therefore, rather an unfair test for cold mix and in real life, cold mix could be expected to perform rather better.

Mixture production

Exactly the same material as that manufactured for the TRL trial was to be supplied for the basecourse layers but the intention was to control the water content thus ensuring that it was made to the correct specification. A 10 mm close graded wearing course cold mix was designed which was also based on BS 4987 Part II. In order to control the water content in the mixtures, CAMAS ensured that their hoppers were filled with dry aggregate direct from the crushing and sizing plant rather from the outside stockpiles. On the day before full scale production, mix trials were carried out, using the same method as that used for the TRL trial, to ensure that the mix could be produced correctly. These trials were successful.

Laying

During the night before laying, very heavy rain fell resulting in the excavated trench becoming saturated (Figure 7-7). On the first day of laying, 60 tonnes of cold mix was manufactured by CAMAS and supplied to site. A 150 mm lift was layed by rake and shovel (Figure 7-8) and compacted using a two tonne, twin steel drum roller (Figure 7-9). The rain persisted through much of the period during which the first lift of cold mix was layed. It became obvious during laying that the cold mix material was not coping with the wet conditions. Water was soaking up from the unbound road base and preventing the emulsion from breaking and adhering to the aggregate (Figure 7-10). During the afternoon, half of the second lift was layed on top of the first to see if any improvement could be achieved (Figure 7-11). Load bearing tests were carried out by Northamptonshire Engineering Services Laboratory which confirmed that the material was not gaining any strength (Figure 7-12). Nuclear density meter readings showed that the layers were waterlogged. The decision was, therefore, taken to remove the second layer and replace it with hot mix. None of the wearing course material was manufactured nor laid.

7.5 CONCLUSIONS

Despite being rather unsuccessful in terms of fulfilling the original objectives, the trials described above yielded useful information and a great deal of practical experience was gained. It was found, overall, that mixtures which looked perfectly good in the laboratory were difficult to reproduce on a larger scale. The main revelation was that it is very difficult to tightly control the variables during large scale production; particularly with materials which are

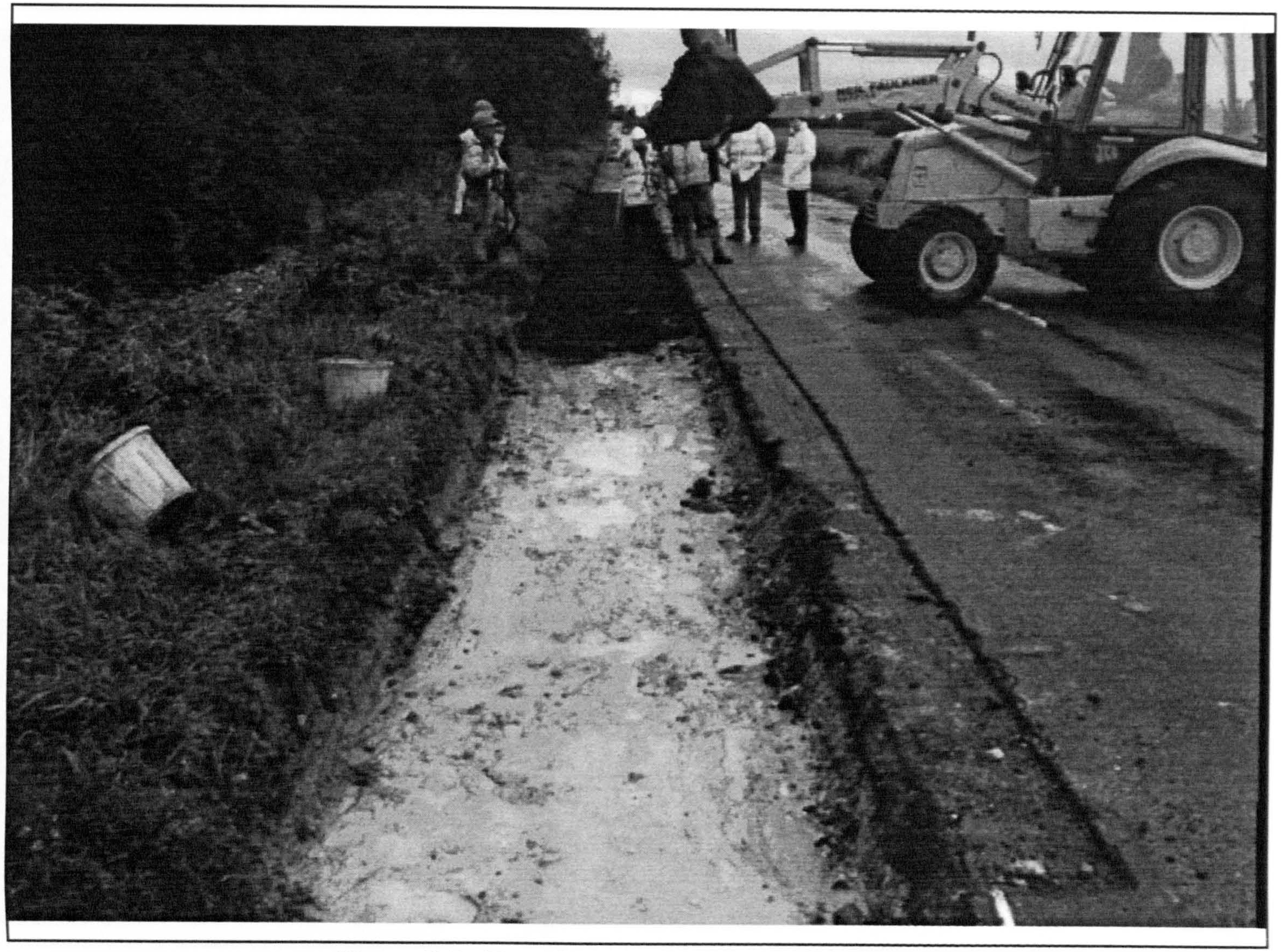


Figure 7-7 : Trench before laying of emulsion mix

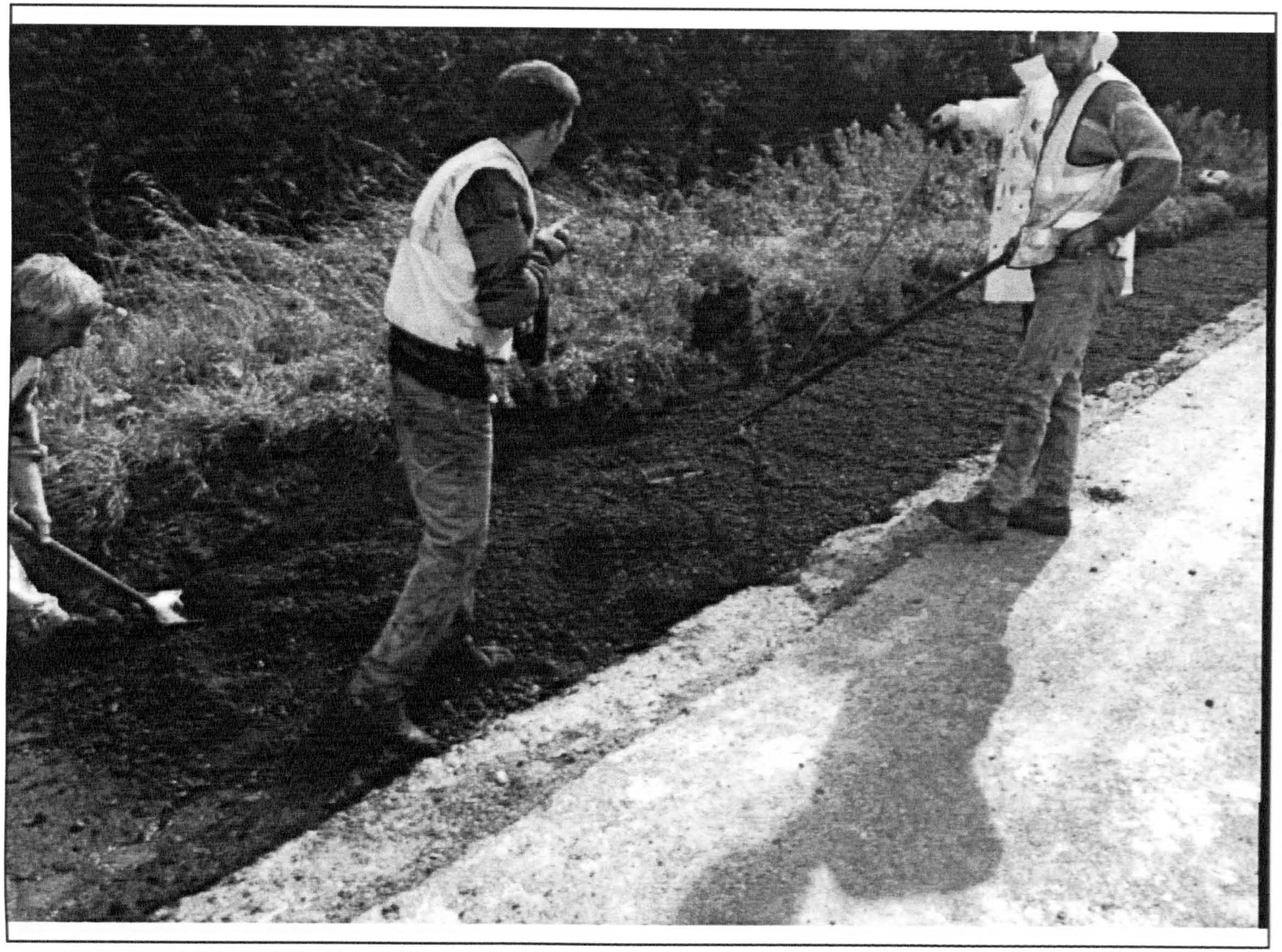


Figure 7-8 : Laying by rake and shovel

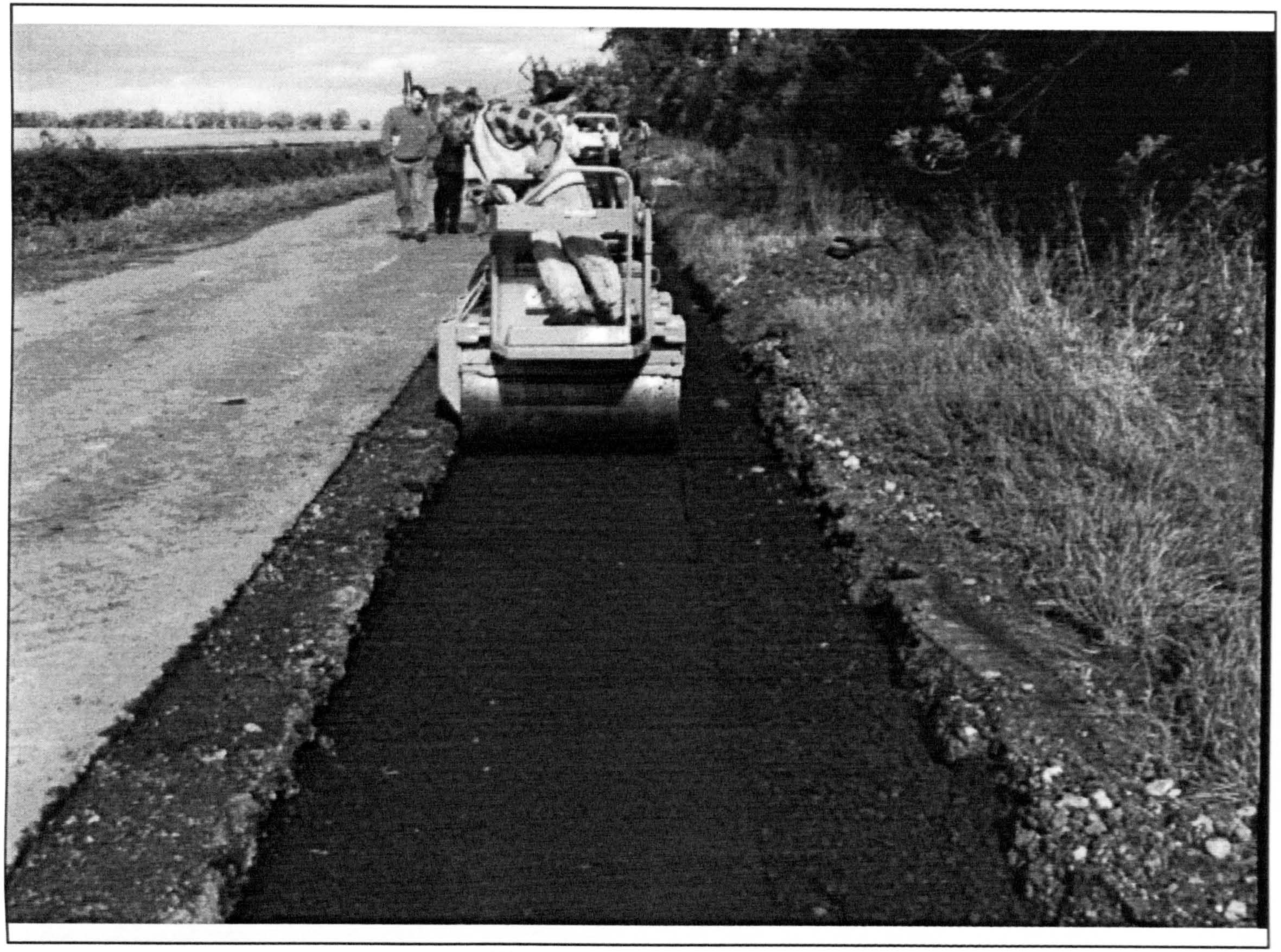


Figure 7-9 : Compaction

Figure 7-10 : Waterlogging of first course

Figure 7-11 : Surface of second layer

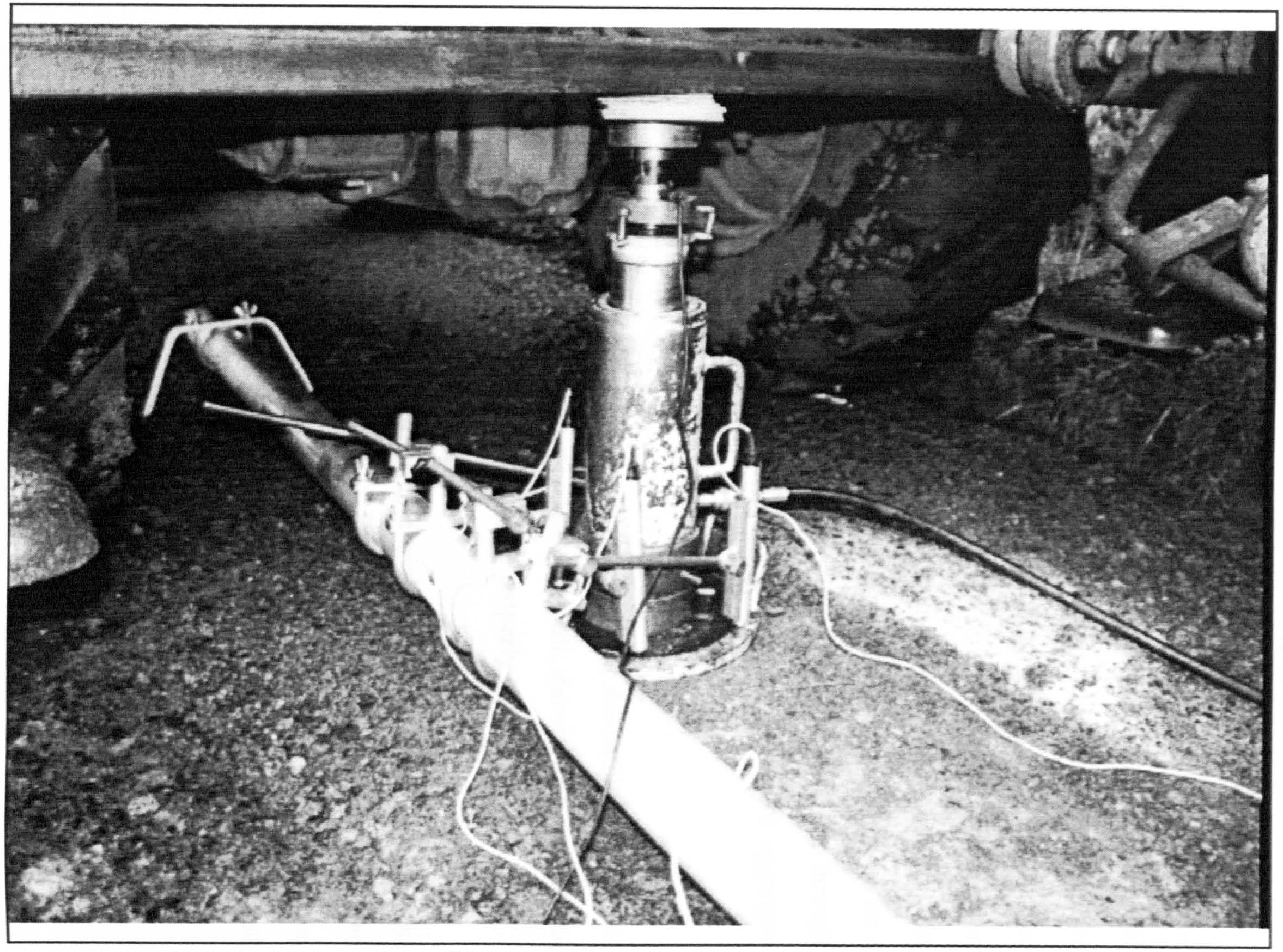


Figure 7-12 : Load bearing tests

unfamiliar to plant operators. It was also found that the mixtures manufactured for these trials are not suitable for laying in wet conditions. In some countries it is feasible to wait for favourable conditions but in the UK, at least, it would be over optimistic to place a similar reliance on the state of the weather.

With hindsight, some if not all of the problems encountered during these trials could be avoided. The importance of tighter control on the mix plant should be emphasized and better equipment made available to ensure adherence to the required recipe. Before commencing the haunching trials, an agreement was made on the dry weather conditions required for cold mix of the type used in these studies. In reality, this was not adhered to and the contractor claimed that laying only in favourable conditions would be impossible to comply with on a daily basis. Resistance to adverse weather conditions could be improved through the use of more cationic emulsions but, in these trials with dense aggregate gradings, this was not an option.

The TRL trial results were encouraging in that the batches of material manufactured and layed correctly performed quite well, showing that the bitumen emulsion mixture could be successful layed if properly manufactured.

The overall conclusion from these trials was that further development work was required to produce cold mix materials which can be used and relied upon in the same way as hot mix. The studies described in the following two chapters attempted to address this problem by looking at the effect of OPC and other variables on the mechanical properties of bitumen emulsion mixtures.

CHAPTER 8

THE EFFECT OF ORDINARY PORTLAND CEMENT ON MECHANICAL PROPERTIES OF BITUMEN EMULSION MIXTURES

8.1 INTRODUCTION

As described in chapters 3 and 4, ordinary portland cement has been used in a number of instances over the years to improve the mechanical properties of a range of cold mix materials. Additionally, laboratory work carried out for the field trials described in chapter 7, has shown that OPC can have a beneficial effect on the mixtures it was used in, but these tests were not comprehensive. In the work described here, the aim was to more firmly establish and quantify the effect of OPC with reference to a traditional British, high binder content, solventless, dense bituminous macadam mixture.

8.2 CEMENT

A cement is any material that hardens and becomes strongly adhesive after application in plastic form (126). In engineering, the term refers to a finely powdered substance consisting of a range of mineral compounds which harden and adhere after being mixed with water. There are a wide range of cements which differ in setting rate, chemical resistance, volume change on setting and heat of hydration. The most commonly used products are known as Portland cements. They are so called because they are similar in appearance to Portland limestone after setting. Within this family there are several grades specified by the criteria listed above. The basic grade on which the others are based is known as ordinary portland cement and this has a medium setting rate with respect to the group.

Portland cements are manufactured by intimately grinding together lime-bearing materials, usually limestone, with clay, shales or blast furnace slag, containing aluminium and silicon oxides. The mixture is then heated to temperatures of ~1600 °C whence it melts and fuses together to form a clinker. This is then re-ground to give the final cement product.

Typical portland cements are composed of many different compounds but only the four shown in Table 8-1 are considered to be important (127). Cement technologists use abbreviated forms of the true chemical formulae of the compounds to simplify things and these are also shown in Table 8-1. In addition to these compounds, gypsum (calcium sulphate) is often added to either increase the setting rate (by rapidly absorbing water) or slow down the hardening process.

Table 8-1 : Mineral compounds in Portland cement

Name	Chemical composition	Abbreviation
Tricalcium silicate	$3CaO.SiO_2$	C_3S
Dicalcium silicate	$2CaO.SiO_2$	C_2S
Tricalcium aluminate	$3CaO.Al_2O_3$	C_3A
Tetracalcium aluminoferrite	$4CaO.Al_2O_3.Fe_2O_3$	C_4AF

These active compounds are very reactive with water and on doing so, they rearrange their chemical structure to form different, insoluble compounds. The initial setting of cement (within the first 14 days) is caused by the hydration of C_3S, which forms jelly like hydrated silica and calcium hydroxide. Over a period of time, these substances crystallise and act as a strong binder. C_3S is the compound which gives rise to 'rapid hardening' cements. The fineness of grinding also contributes to setting rate by increasing the surface area of the mineral available to undergo hydration. C_3A acts in the same way as C_3S initially but does not contribute to the final strength of the cement. Its reaction products are very susceptible to sulphate attack. Both the C_3S and C_3A reactions are quite exothermic and so generate a considerable amount of heat. C_2S hydrates much more slowly and does not contribute any strength in the first 7 days. It continues to harden over a couple of years. C_2S reaction products are resistant to sulphate attack and shrinkage. C_4AF reacts very rapidly with water and setting occurs in minutes. However, this compound does not contribute at all to the strength of the cement.

8.3 STIFFNESS MODULUS

8.3.1 Materials

A granite aggregate from CAMAS Aggregate's (formerly ECC) quarry at Croft was used. Nominally 20, 14, 10, 6 mm and dust fractions were supplied. Grading analyses for all fractions were obtained from CAMAS and these were used to prepare reconstituted mixtures with a range of gradings.

The bitumen emulsions described in Table 6-2 were used.

8.3.2 Mixture design

The aggregate grading used in these tests was in the middle of the 20 mm DBM specification (9) as shown in Figure 8-1. OPC, pre-wet water and emulsion addition levels were as shown in Table 8-2. The standard emulsion as described in Table 6-2 was used. It was necessary to adjust the pre-wet water addition level in accordance with the OPC to accommodate extra water absorption by the "filler".

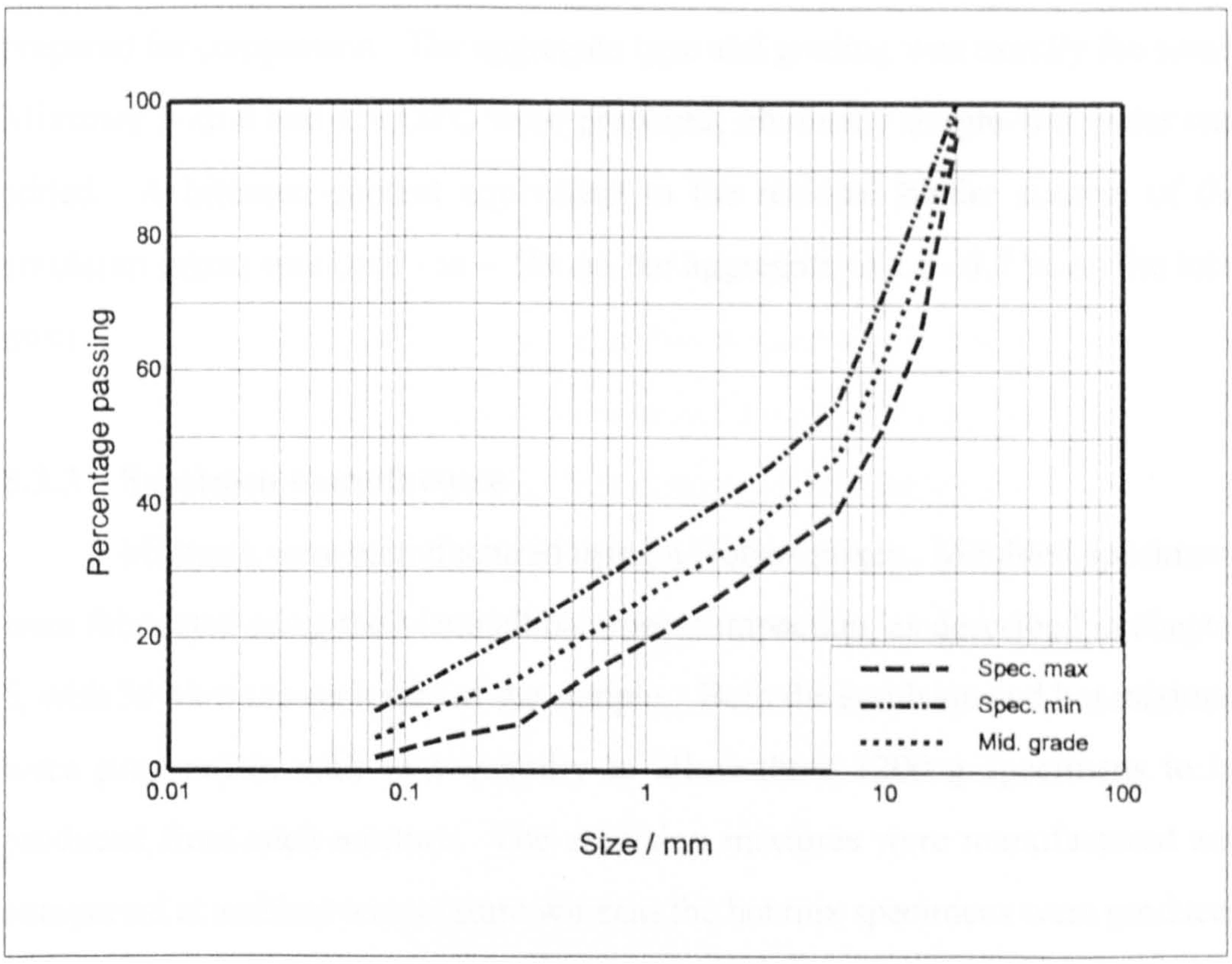


Figure 8-1 : Mid BS 4987 20 mm DBM aggregate grading

Table 8-2 : Mixture compositions used to study effect of OPC

Mix component	Percentage on aggregate mix				
Granite aggregate 20 mm	26				
14 mm	15				
10 mm	9				
6 mm	15				
Dust	35				
Ordinary Portland Cement	0	1	2	3	4
Pre-wet water	2.5	2.5	3.0	3.5	4.0
Bitumen emulsion	8.06				

In addition to the emulsion mixtures, conventional hot mixes were prepared for comparison. The aggregate type and grading was exactly the same. Mixtures with 0 and 1% OPC were prepared; obviously no pre-wet water was added. A bitumen content equivalent to the residual binder content of the emulsion mixes was used - ie ~ 5% on the aggregate mix (~ 4.7 % on the total mix).

8.3.3 Specimen manufacture

Mixtures were manufactured using a Hobart mixer. Moulded specimens were fabricated using the Marshall hammer compaction, as described in chapter 5, with 50 blows to each side of the sample. Both the emulsion and hot mixtures were prepared in sufficient quantity to allow three 1200 g specimens to be produced from each mixture. The emulsion mixtures were manufactured and compacted at ambient temperature whereas the hot mix specimens were produced at a temperature of 135 to 140 °C. Cores were extracted from their moulds after ~16 hours and then stored in a constant environment room at a temperature of 20 (+/- 1)°C and a relative humidity of 50 (+/- 5) % during the testing period.

8.3.4 ITSM and water loss tests

ITSM tests on the emulsion mix specimens were first carried out after 5 days, and then periodically as the specimens cured, on two out of the three cores produced. Specimens were tested at 20°C, with a load target rise time of 120 ms and a target horizontal displacement of 5 μm. The results of these tests are shown in Table 8-3 and illustrated in the graph in Figure 8-2. The third cores were used for water loss measurements which were simply taken as the loss in weight. The results of these tests are shown in Figure 8-3. When the ambient curing tests had been completed the weight loss cores were fully dried in an oven at 60°C, to constant weight. This served two purposes. Firstly, it allowed the weight of the aggregate, OPC and bitumen mix, in the absence of water, to be measured. This figure would allow back calculation of the water loss over the curing period. Secondly, these cores were used for ITSM tests to obtain stiffness figures for "fully cured" cores (quoted as 365 days cure).

The hot mix cores were ITSM tested after 5 days and again after approximately 2 months.

Unfortunately no void content measurements were made on specimens used in these tests but the dimensions recorded as part of the stiffness parameters showed an increase in depth of the briquettes with increasing OPC level. This suggested that the voids increased slightly with increasing OPC level. Later in this research the same mixtures have been prepared and compacted in the manner used here. In these specimens the void contents of the cold mix cores were found to be in the region of 10 to 15 % whereas equivalent hot mix normally has voids of around 5 to 8 %.

Table 8-3 : Stiffness moduli of cold mix cores with different OPC levels

Curing time (days)	**STIFFNESS MODULUS / MPa**					
	OPC addition level / % on aggregate mix					
	Cold mix				Hot mix	
	0	1	2	4	0	1
7	506	1,346	2,334	4,482	2,074	2,074
14	706	1,499	3,127	5,928		
21	754	1,954	4,024	7,074		
26				8,400		
28	929	2,113	4,492			
33	798	2,347	4,665			
49				7,644		
58	1,088	2,386	5,515			
120	1,168	2,208	5,243	9,249		
365	3,478	5,665	7,493	12,267		

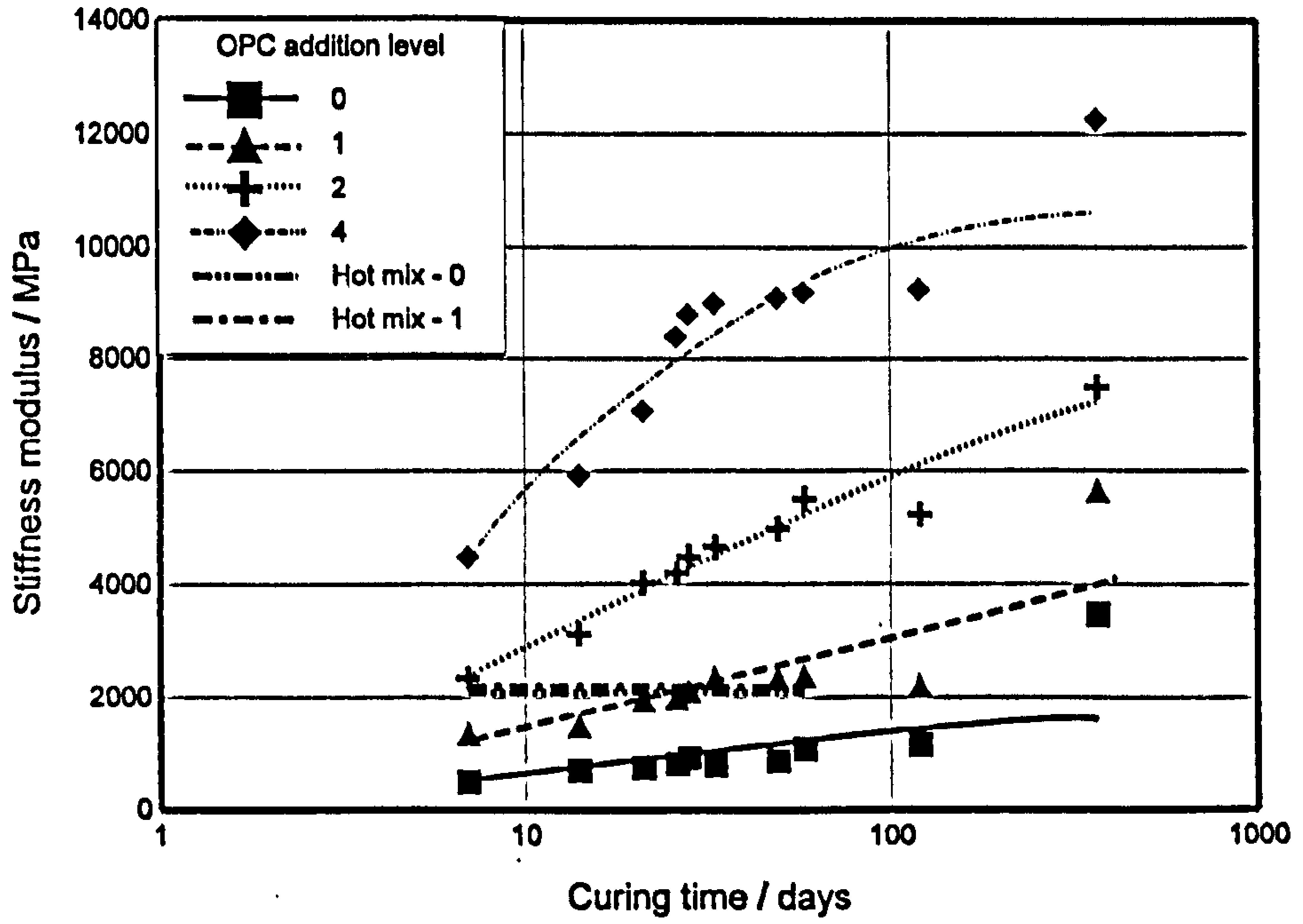


Figure 8-2 : Stiffness moduli of cold and hot mix specimens with different levels of OPC

From the ITSM results the following points are worthy of note :

- Stiffness moduli of the emulsion mixture cores increased steadily over several weeks, in contrast with the hot mix which showed no discernable change.
- The rate of stiffness increase of the emulsion mixtures increased with OPC addition level whereas the hot mix was unaffected by cement.
- The final stiffness of the emulsion mixtures increased with OPC addition level.
- The stiffness recorded for oven cured specimens seemed disproportionately high compared with those after ambient cure. This raises the question - does 60 °C oven curing affect the binder viscosity through chemical alterations such as oxidation or loss of volatiles?

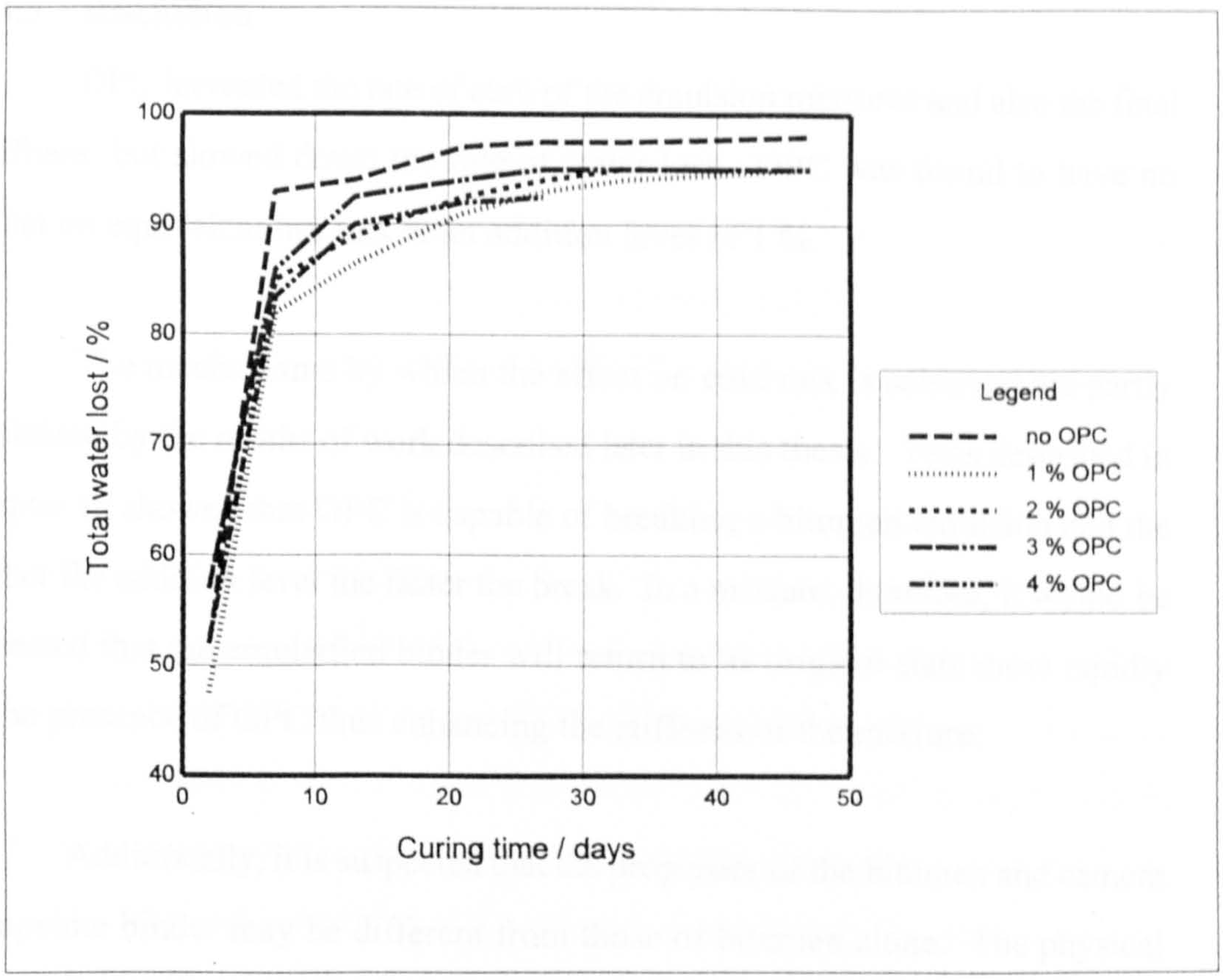


Figure 8-3 : Water loss from cores with various levels of OPC

From the water loss results the following points are worthy of note :

- The majority of the water loss occurred in the first week after specimen manufacture
- Generally, the addition of OPC slowed down the rate of water loss but the results did not follow a trend
- The remainder of the water continued to evaporate over several weeks and it seems likely that it may never disappear completely.

These data are in good agreement with the findings of Puzinauskis and Jester (128). They also found that the majority of water loss occurred in the first few days after compaction and that this was regardless of the emulsion or water content.

8.3.5 Discussion

OPC increased the rate of cure of the emulsion mixtures and also the final stiffness, but slowed down the rate of water loss. OPC was found to have no effect on equivalent hot mix at an addition level of 1 %.

The mechanisms by which the effect on cold mix is achieved are partly explained by the results of work described later in this thesis. Tests described in chapter 12 showed that OPC is capable of breaking a bitumen emulsion and the higher the addition level the faster the break. In a mixture, therefore, it would be expected that the emulsified binder will return to its original state more rapidly in the presence of OPC thus enhancing the stiffness of the mixture.

Additionally, it is suspected that the properties of the bitumen and cement composite binder may be different from those of bitumen alone. The physical nature of the binder plus OPC was studied directly in these studies and this work is covered in a later chapter.

8.3.6 Comparison of OPC with hydrated lime and $CaCl_2$

Other tests carried out in this research and previous studies (15, 16, 94) have indicated that hydrated lime can have a similar effect to cement on breaking of bitumen emulsions and curing of mixtures. Zeta potential measurements described in chapter 11 also showed that lime and $CaCl_2$ render the Redicote E-4868 emulsion more cationic or less anionic under certain conditions. As discussed in chapter 2, this can have an effect on the interaction between emulsion droplets and negatively charged aggregate surfaces. As it is suspected that Ca^{2+} may be partly responsible for the mechanism, it was felt that a mixture containing a small amount of $CaCl_2$ may also exhibit accelerated curing. This compound would provide the mixture with a source of Ca^{2+} ions but should not offer any secondary binding effect. Thus, a series of tests were carried out in order to compare performance of lime and $CaCl_2$ with OPC.

Mixture design

Moulded specimens were manufactured in exactly the same way as those used in the measurements described above. OPC and hydrated lime were used at a level of 1 % on the aggregate mixture whereas the $CaCl_2$ was added to the emulsion at a level of 1 %. The samples were stored at 20 °C and 50 % relative humidity and periodically tested in the ITSM test on the NAT. The results are shown in Figure 8-4.

Discussion

From the results plotted in Figure 8-4 it can be seen that hydrated lime and $CaCl_2$ do not have the same beneficial effect as OPC on stiffness modulus of bitumen emulsion mixtures of the type tested here. As lime has been seen to cause bitumen emulsion to break in other tests in this thesis, these results suggest that OPC enhances the stiffness of a mixture by stiffening the binder but lime does not. Rheological tests described in a later chapter attempted to measure this directly.

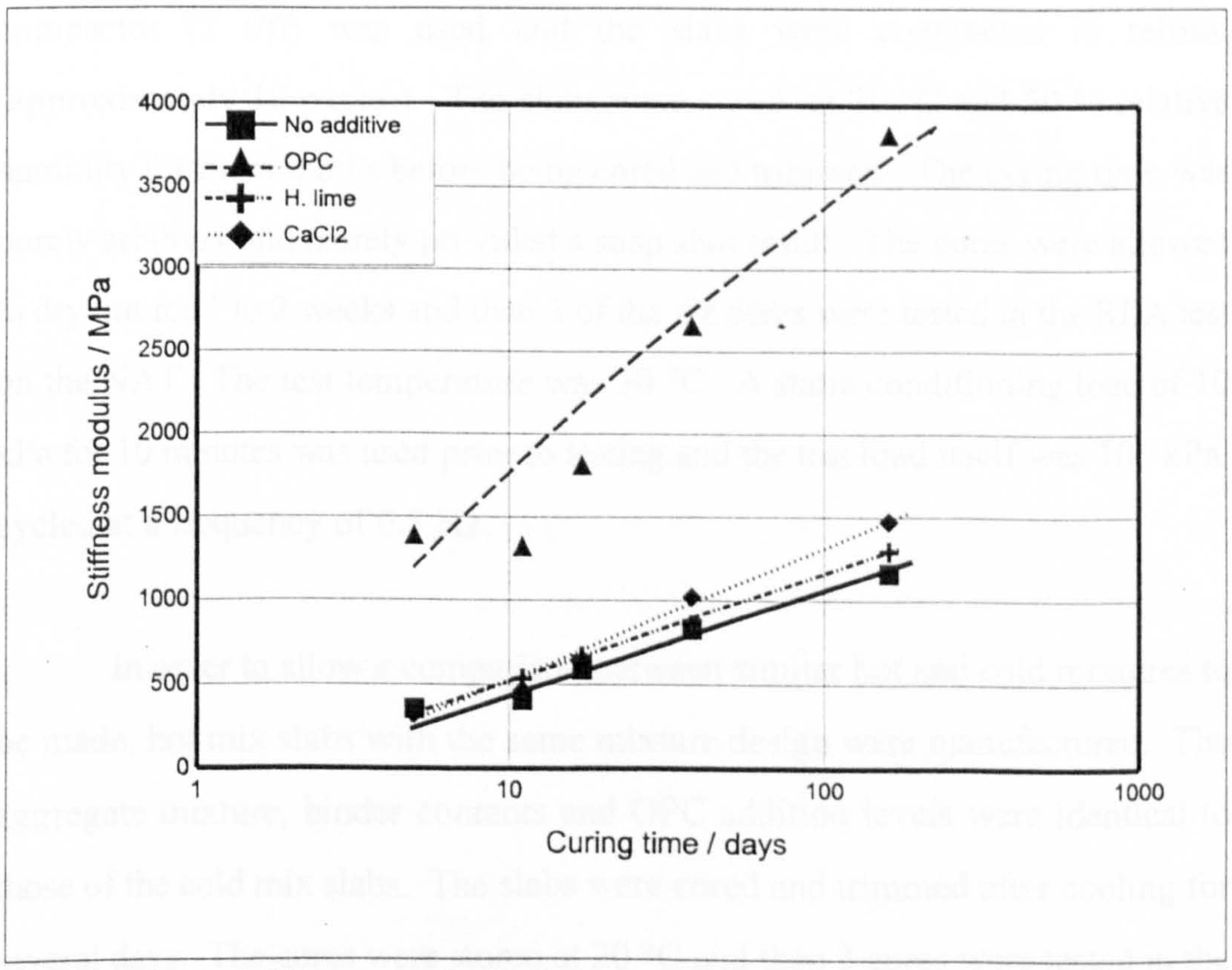


Figure 8-4 : Stiffness moduli of cold mix specimens with OPC, lime and $CaCl_2$

8.4 RESISTANCE TO PERMANENT DEFORMATION

Having established that OPC had the expected effect on stiffness, focus then moved on to look at the effect on resistance to permanent deformation.

8.4.1 Specimen manufacture

The resistance to permanent deformation of mixtures with the same mix design as those used in the stiffness modulus tests described above were determined using the RLA test. However, in these tests, the specimens were manufactured using a different method of compaction. The roller compactor described in chapter 5 was used in the hope that more uniform specimens, in terms of segregation and evenness of specimen surfaces, could be obtained. The latter comes about due to the fact that the specimens are cored from a large slab and the edges trimmed. Flat ends are necessary for RLA tests as surface irregularities can result in erroneous results due to alterations at the surface rather than in the bulk of a specimen. The maximum load available from the roller compactor (2 t/ft) was used and the slabs were compacted to refusal (approximately 10 passes). The slabs were cured at 20 °C and 50 % relative humidity for four months before being cored and trimmed. The curing time was purely arbitrary and merely provided a snap shot result. The cores were allowed to dry out for 1 to 2 weeks and then 3 of the six cores were tested in the RLA test on the NAT. The test temperature was 30 °C. A static conditioning load of 10 kPa for 10 minutes was used prior to testing and the test load itself was 100 kPa, cycled at a frequency of 0.5 Hz.

In order to allow a comparison between similar hot and cold mixtures to be made, hot mix slabs with the same mixture design were manufactured. The aggregate mixture, binder contents and OPC addition levels were identical to those of the cold mix slabs. The slabs were cored and trimmed after cooling for several days. The cores were stored at 20 °C and then 3 cores were tested in the RLA test under the conditions described above.

8.4.2 Results from cold mix cores

The graph shown in Figure 8-5 shows the averaged results obtained from RLA tests on cores in triplicate with different levels of OPC. Data are plotted on log-log axes here to facilitate plotting all of the lines on one graph as there was a wide range of axial strains. Data for an equivalent hot mix obtained from a report from Brown et al (107) are indicated on the graph. This is to allow a comparison with a typical hot mix from an independent source to be made.

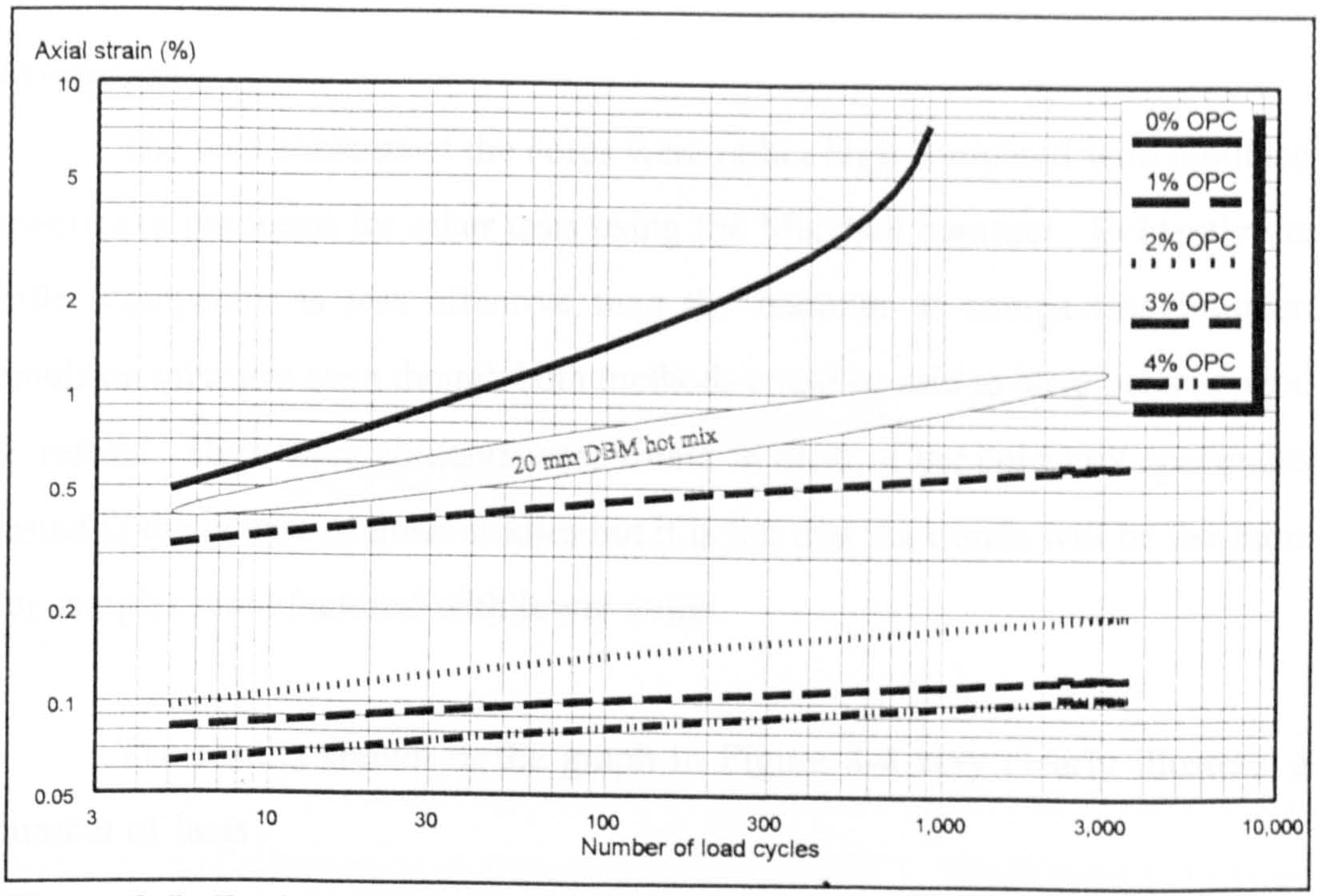


Figure 8-5 : Resistance to permanent deformation of cold mix with range of OPC levels

The averaged void contents of the 3 cores of each mixture were determined, using the method described in chapter 5, and these results are shown in Table 8-4.

Table 8-4 : Void contents of cold mix cores used in RLA tests

OPC addition level	Void content (%)
0	16.4
1	20.3
2	18.2
3	16.6
4	14.7

Discussion

The void contents of the cores were rather high compared with moulded specimens produced for other tests using the Marshall hammer. Evidently the roller compactor is less effective than the hammer at compacting bitumen emulsion mixtures even though both methods could be said to have been applied to refusal. High were contents were found in most of the cold mix specimens tested in the course of these studies but it is felt that the trends will be the same for samples manufactured with lower voids.

The results shown in the graph in Figure 8-4 very clearly illustrate a number of facts :

- Without OPC, cold mix did not last the full 3600 load cycles used in the RLA test and failed after < 1,000. This mixture exceeded 1 % strain after only 40 load cycles. Both of these facts suggest that unmodified cold mix of the type used in this test has very poor resistance to permanent deformation.
- The resistance to permanent deformation was greatly improved by the addition of OPC. The larger the addition level the greater the improvement obtained.

This result is not surprising in some respects and yet evidence from field trials in the past, has suggested that the performance of cold mix in terms of resistance to permanent deformation, even without cement, is not a problem. Even though the binder in cold mix is quite weak, particularly in the early stages of curing, the aggregate interlock would be considered to be similar to that in hot mix. This is thought to be an important factor in resistance to permanent deformation. However, when the void contents of the cold mix cores are taken into account, a shortfall in performance could be easily understood. The void contents of the emulsion based cores used in these tests are at least 2 or 3 times higher than those which would be expected for equivalent hot mix. Data from Nottingham (107), represented in Figure 8-5, reveals that a 20 mm DBM mixture with 5 % Venezuelan 100 pen performs similarly to the cold mix with 1 % OPC. In these mixtures the void contents were quoted as 6 %. The fact that cold mix with far higher voids than equivalent hot mix performed similarly indicated that the emulsion mixtures with cement had a much greater resistance to permanent deformation than would be expected.

Strain rate

In addition to graphical data plots, mean strain rate, minimum strain rate and final strain figures may be used to characterise resistance to permanent deformation of mixtures as discussed in chapter 5 (123). Strain rates have been found to be more meaningful as a result of work carried out at Nottingham. These studies involved making deformation measurements with LVDT's mounted on the sample and off the sample which is the method used in the normal RLA test. Results showed that the strains measured with on sample measurement were always lower, suggesting that local effects - ie at the ends of cores - play a significant part in the deformation of samples tested using the standard RLA test format. Quotation of strain rate figures, rather than absolute strains are, therefore, advocated. Table 8-5 shows these figures for the data recorded for the cold mix cores in these tests.

Table 8-5 : Strain figures for cold mix with range of OPC levels

OPC level (%)	Final strain (%)	Mean strain rate (μstrain/cycle)	Minimum strain rate (μstrain/cycle)
0	Failed	102.55	44.57
1	0.589	3.292	0.090
2	0.195	1.146	0.023
3	0.122	0.494	0.010
4	0.106	0.495	0.007

The figures for the mixture without OPC are very high due to failure of the mixture (~ 8 mm deformation) after only 900 load cycles. The mixtures with OPC performed a lot better having very low mean and minimum strain rates.

8.4.3 Hot mix

The graph in Figure 8-6 and data in Table 8-6 show the results of the RLA tests on the equivalent hot mix cores. Data have again been plotted on log-log axes to allow an easy comparison with the cold mix specimens to be made. The voids contents of the specimens were as shown in Table 8-7.

Table 8-6 : Strain figures for hot mix with range of OPC levels

OPC level (%)	Final strain (%)	Mean strain rate (μstrain/cycle)	Minimum strain rate (μstrain/cycle)
0	1.137	8.635	0.597
1	1.389	9.512	0.740
2	2.031	12.807	1.150
3	1.560	11.445	0.713
4	1.956	14.146	0.857

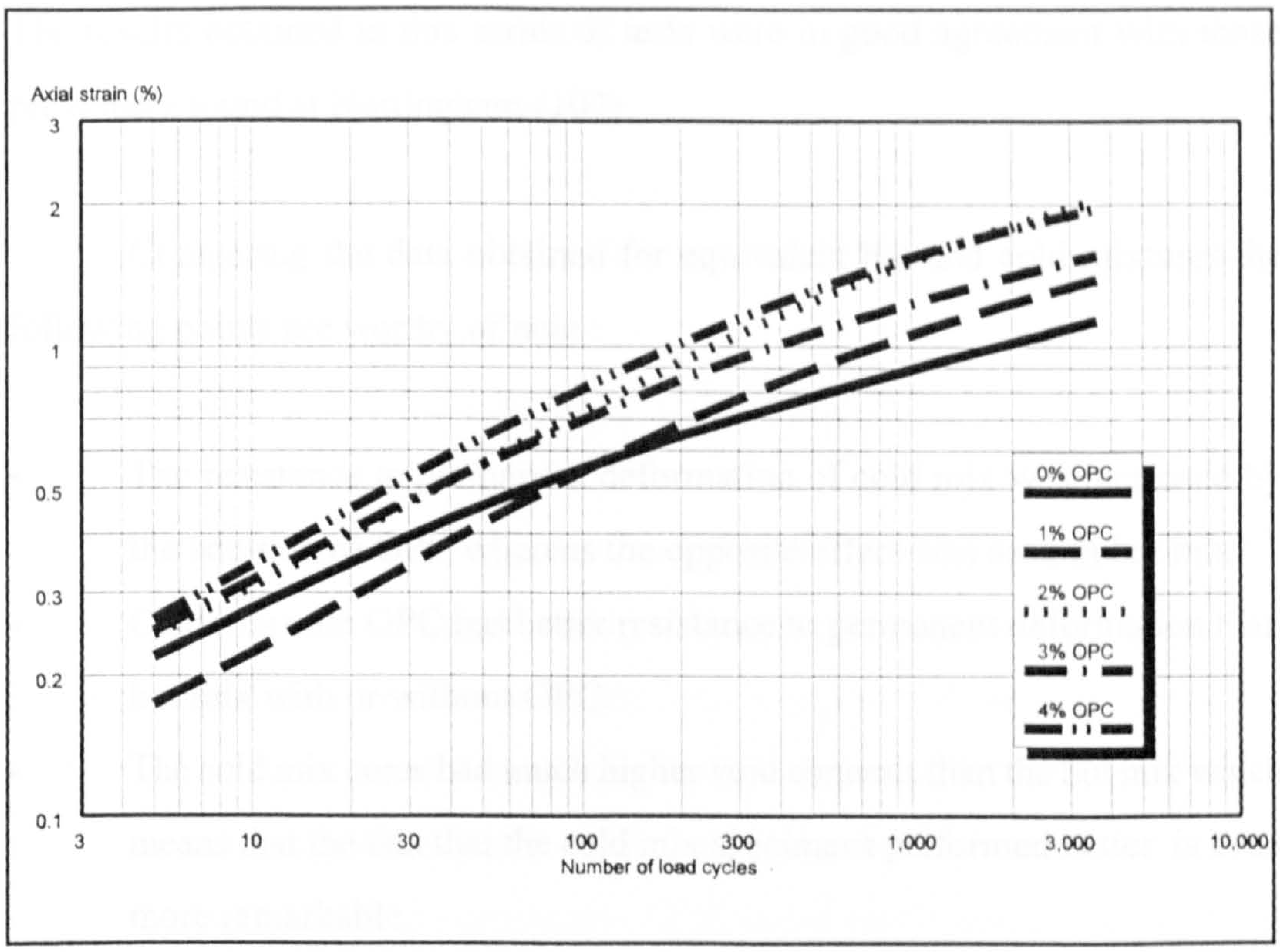


Figure 8-6 : Resistance to permanent deformation of hot mix with range of OPC levels

Table 8-7 : Void contents of hot mix cores with range of OPC levels

OPC addition level (%)	Voids (%)
0	4.99
1	4.24
2	11.73
3	4.51
4	4.70

Discussion

The results shown above indicate that, generally, the resistance to permanent deformation of the hot DBM mixtures decreased with increasing OPC level. The mixture with 2 % OPC did not fit into the pattern but this seems to have been due to the very high void content. The cause for this is not known.

The results obtained in this series of tests were in good agreement with those previously found at Nottingham (107).

Comparing the data obtained for equivalent hot and cold mixtures the following points are worthy of note :

- The resistance to permanent deformation of cold mix was increased by the addition of OPC whereas the opposite effect was seen in hot mix.
- Cold mix with OPC has better resistance to permanent deformation than hot mix with or without OPC.
- The cold mix cores had much higher void contents than the hot mix which means that the fact that the cold mix specimens performed better is even more remarkable.

These results clearly suggest that OPC acts as a secondary binder in cold mix. Naturally, hydration is necessary to activate the cement and this can only occur in the cold mix material in which water is available.

Cabrera and Nikolaides carried out comprehensive testing on the creep performance of unmodified emulsion mixtures and compared performance with hot mix (129). Overall, they concluded that equivalent hot and cold mix materials exhibited comparable resistance to permanent deformation. The effect of a number of variables was also investigated and resistance to permanent deformation of DBM cold mix was found to be unaffected by binder content, improved with curing and improved with good coating of the aggregate.

8.4.4 Confinement

The tests described above used cylindrical specimens without any form of confinement. In a road situation, the material is supported externally. The RLA tests is, therefore, a severe test in comparison. It places a large dependency on the cohesion of the binder as well as the packing arrangement of the aggregate.

Recent work has been carried out at Nottingham into the use of confinement in the RLA test as described in chapter 5 (107). It was not possible to use the apparatus in the course of these studies but it would be of interest to repeat the tests under such circumstances in the future.

8.5 RESISTANCE TO FATIGUE CRACKING

The resistance to fatigue cracking of mixtures with a range of OPC addition levels was determined using the ITF test described in chapter 5. Specimens were manufactured using mix designs similar to those employed in the ITSM and RLA tests described above but in this instance, OPC levels of 0, 1 and 3 % were used. Mixtures were compacted using 50 blows of the Marshall hammer to each side of the specimen. 12 moulded specimens of each mixture were thus prepared and cured for 2 weeks at ambient temperature and then in an oven at 60 °C for 3 days.

This curing method was used for a number of reasons. Firstly, in a practical sense, it was not possible to use partially cured specimens. Due to the length of the tests themselves, it would have been difficult, if not impossible to test all specimens at the same stage of curing. This would have resulted in erroneous results. Secondly, it was felt that cold mix in a fully cured state would be more susceptible to cracking than partially cured mix. In reality, the situation for cold mix would be very complicated as loading of the pavement would take place as the mixture was curing and its properties changing over a long period of time. This was not possible in the laboratory and, in fact, each specimen was subjected to a "lifetime's" loading at a particular instance in its life - this being at the point of full cure.

8.5.1 Results

The results from the ITF tests carried out on cold mix specimens with a a range of OPC levels are shown in the graph in Figure 8-7.

The R^2 values, slopes and intercepts for the data shown in Figure 8-7 were as shown in Table 8-8.

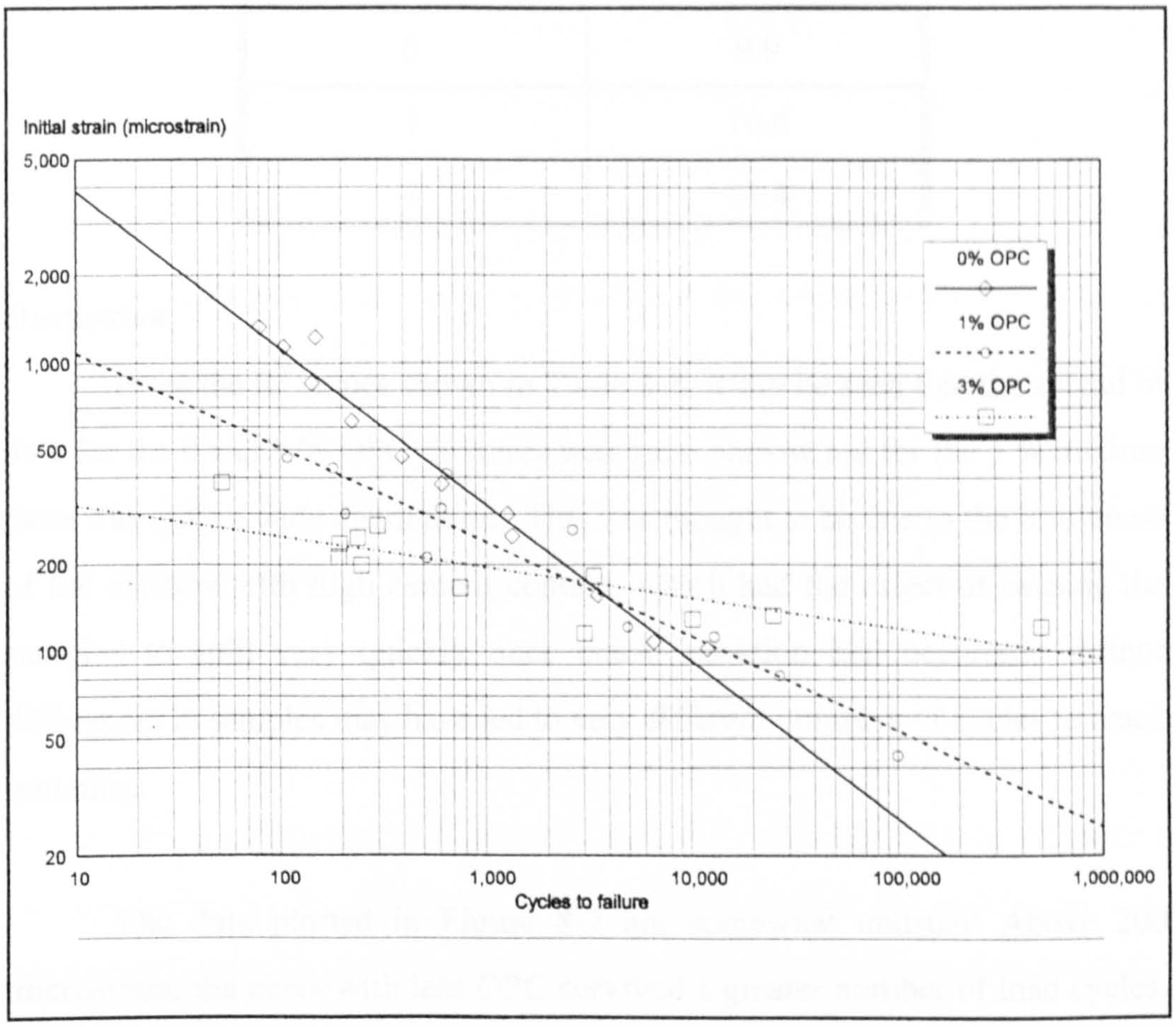


Figure 8-7 : Cycles to failure v initial strain for mixtures with range of OPC addition levels

Table 10-8 : Linear regression values for mixture with OPC

OPC level (%)	R^2	Slope	Intercept
0	0.98	-3.47	13460
1	0.90	-2.12	2293
3	0.61	-1.28	408

The void contents for the specimens were as shown in Table 8-9.

Table 8-9 : Void contents of specimens with OPC used for ITF tests

OPC level (%)	Voids (%)
0	9.9
1	10.6
3	13.4

Discussion

From the R^2 values shown in Table 8-8, it can be seen that the spread of data for the 0 and 1 % OPC mixtures was quite narrow but for the 3 % mixture there was quite a wide distribution. This was thought to be due to the brittleness of the mixture with high cement content, which had the effect of causing the samples to split very quickly once crack initiation had occurred. Minor differences in samples may have led to very different numbers of cycles to crack initiation.

The data plotted in Figure 8-7 are somewhat unusual. Above 200 microstrain, the cores with less OPC survived a greater number of load cycles. Below 200 microstrain the reverse was true. This seems to raise the question - is there something significant about the 200 microstrain level? It is likely that it is due to embrittlement caused by the inclusion of the cementitious binder. This imposes a threshold value on the level of strain which can be tolerated by the mixture. Below the threshold, a mixture would survive a very great number of load cycles but above, it would crack and fail very quickly. This behaviour is typical of any cement treated mixture.

Theoretical analysis and field studies have indicated that strains in the order of 30 to 200 microstrain are experienced under a standard axle load at the bottom of the main structural element of a pavement structure (114). This is dependent on variables such as mixture and subgrade stiffness, load and layer thickness. It can, therefore, be argued, that only the portion of the graph in Figure

8-7 below 200 μstrain should be considered. In this case, the addition of OPC clearly extends the fatigue life of the basecourse mixtures used in these tests.

The high void content of the specimens with 3 % OPC, which was caused by the dry nature and subsequent lack of workability of the mixture, could have had an effect on fatigue performance. Intuitively, more open mixtures might be expected to be less resistant to fatigue cracking, as crack propagation could be aided by spaces in the binder aggregate matrix. Observations made during testing indicated that the high cement content mixture failed very rapidly once crack initiation had occurred which could have been a due to a combination of embrittlement and high voids content. Work is currently under way at Nottingham to study the effects of various parameters on crack propagation.

8 5.2 Effect of polymers on resistance to fatigue

The results presented above suggested that OPC may have caused some embrittlement of the mixture due to the decrease in resistance to fatigue cracking at high initial strain. SBS polymers are often used in hot mix to increase resistance to cracking and EVA, although normally used to improve resistance to permanent deformation of hot mix (5, 130), was found to increase stiffness of emulsion mixtures as discussed in chapter 9. These polymers were, therefore, incorporated in emulsion mixtures to see if they could improve the resistance to fatigue cracking of OPC containing cold mix.

Specimens were manufactured with exactly the same mixture design as those used in the fatigue tests described above but using emulsions with EVA and SBS polymer modified binders as described in Table 6-2. The addition levels of the polymers were 3 % on the bitumen. The mixtures were also cured and tested in the ITF test in the same manner as those above.

Results

The results of the ITF tests on polymer modified mixtures with OPC were as shown in Figure 8-8 and Table 8-10.

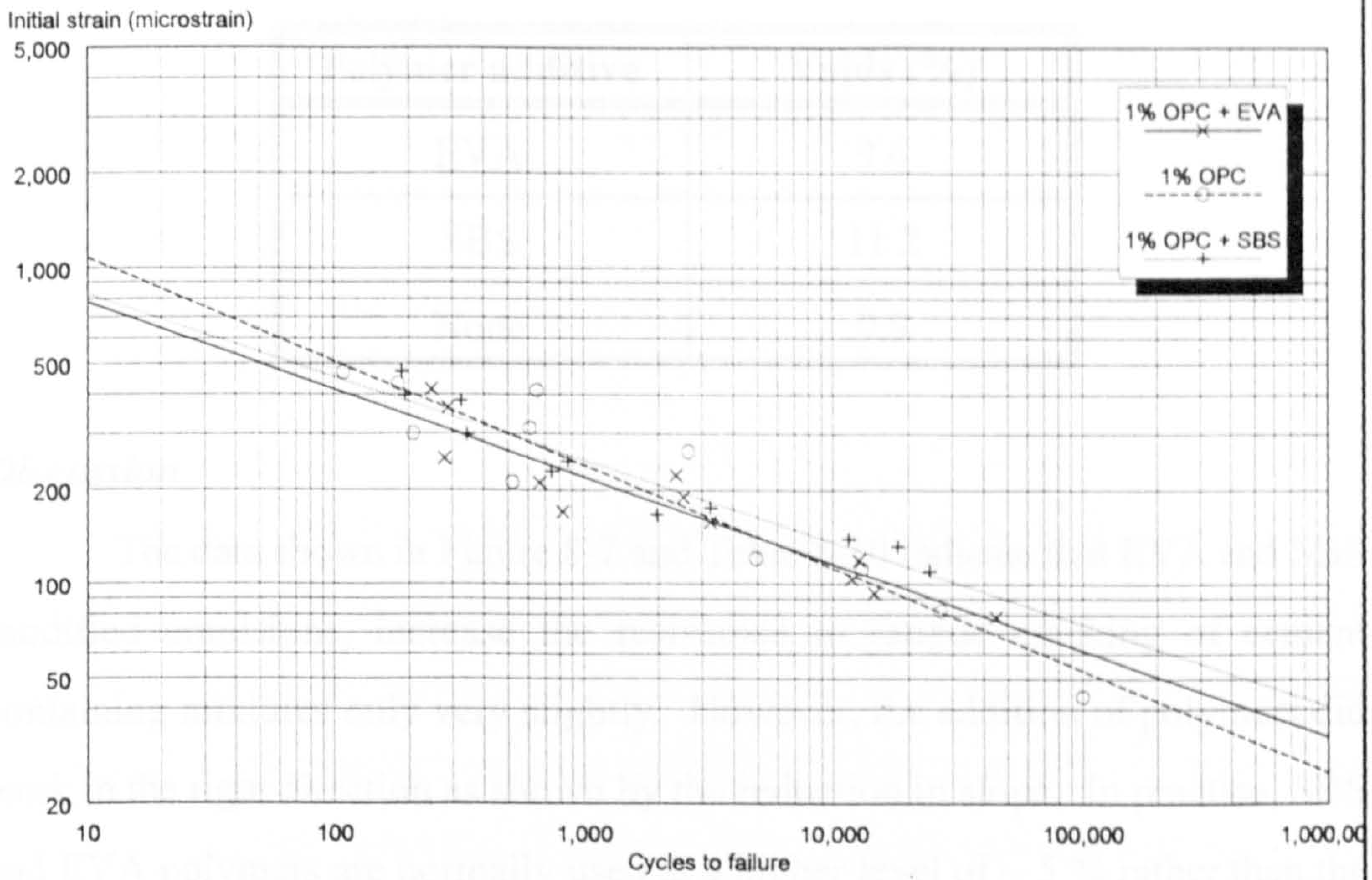


Figure 8-8 : Cycles to failure v initial strain for mixtures with polymer modified emulsions and OPC

Table 8-10 : Linear regression values for mixtures with polymer modified emulsions and OPC

Polymer additive	R^2	Slope	Intercept
EVA	0.88	-1.91	1474
SBS	0.93	-1.82	1514
None	0.90	-2.14	2292

The void contents of the specimens with polymer modified emulsions and OPC used for the ITF tests were as shown in Table 8-11.

Table 8-11 : Void contents of of specimens with polymer modified emulsions and OPC used in ITF tests

Polymer additive	Voids (%)
EVA	9.6
SBS	11.2
None	9.9

Discussion

The data shown in Figure 8-7 and Table 8-10 indicate that EVA and SBS modified emulsions, increase the resistance to fatigue cracking of cement containing mixtures only very slightly. However, the addition of polymers did work in the right direction as shown by the reduction in slope. In practice, SBS and EVA polymers are normally used at a higher level of ~ 5 % rather than the 3 % level used in these studies. This level is necessary to facilitate the formation of a continuous polymer matrix which results in the best improvement in binder properties (130). At this level SBS, in its swollen state, contributes the largest volume to the binder and the elastic properties are subsequently significantly altered. It was not possible to perform further tests with higher polymer levels and this would be an interesting topic for future work. The cost factor, however, may preclude the use of polymers as emulsified binders are comparatively expensive even without modification.

8.5.3 Effect of stiffness on resistance to fatigue

In addition to the effect of any additive on the resistance to cracking of mixtures at a particular initial strain, the effect upon stiffness of the mixture must also be considered. In a pavement situation, the passage of vehicles would subject the structure to a specific level of stress at the surface. The level of strain experienced by the bottom of a layer would be dependant upon the stiffness of the pavement element; the greater the stiffness, the lower the level of strain. It can, therefore, be assumed, that for mixtures of the same thickness, the mixture with the higher stiffness would have greater resistance to fatigue cracking, as the strain at the base of the layer would be less. Following this argument, as OPC and EVA (chapter 9) have both been found to increase the stiffness of emulsion mixtures these additives would also have an effect on resistance to fatigue. Pavement design implications of the findings from these studies are further discussed in chapter 13.

8.6 DURABILITY

The effect of OPC on the resistance to water damage of mixtures has been reported by a number of authors including Ishai et al and Lottman (131, 132). Its effect on durability of mixtures used in these studies was appraised by measuring stiffness of mixtures before and after soaking. In addition to OPC, hydrated lime and $CaCl_2$ were also assessed. Hydrated lime has been used extensively in hot mix to improve adhesion and has also been found to offer similar benefits in emulsion mixtures (15, 16). It has also been used in OGEM in parts of America (64) and various cold recycling projects with good success. $CaCl_2$ was tested due to the suspicion that Ca^{2+} ions play a part in the adhesion mechanism. This additive was incorporated in the emulsion at a level of 1 %. The OPC and lime were added to the dry aggregate mixture at a level of 1 %. Mixtures with the same design as those used in the tests described above were assessed using the NAT before and after soaking. The mixtures were compacted using 50 blows of the Marshall hammer to each side of the specimen: Moulded specimens were subjected to the following soaking and testing protocol :

- Cured for 11 days at 20 °C
- 5 days under water at room temperature
- ITSM test 1
- Soaked under 28" Hg vacuum for 20 mins
- 12 days under water at room temperature
- ITSM test 2

The results of the ITSM tests were as shown in the graph in Figure 8-9.

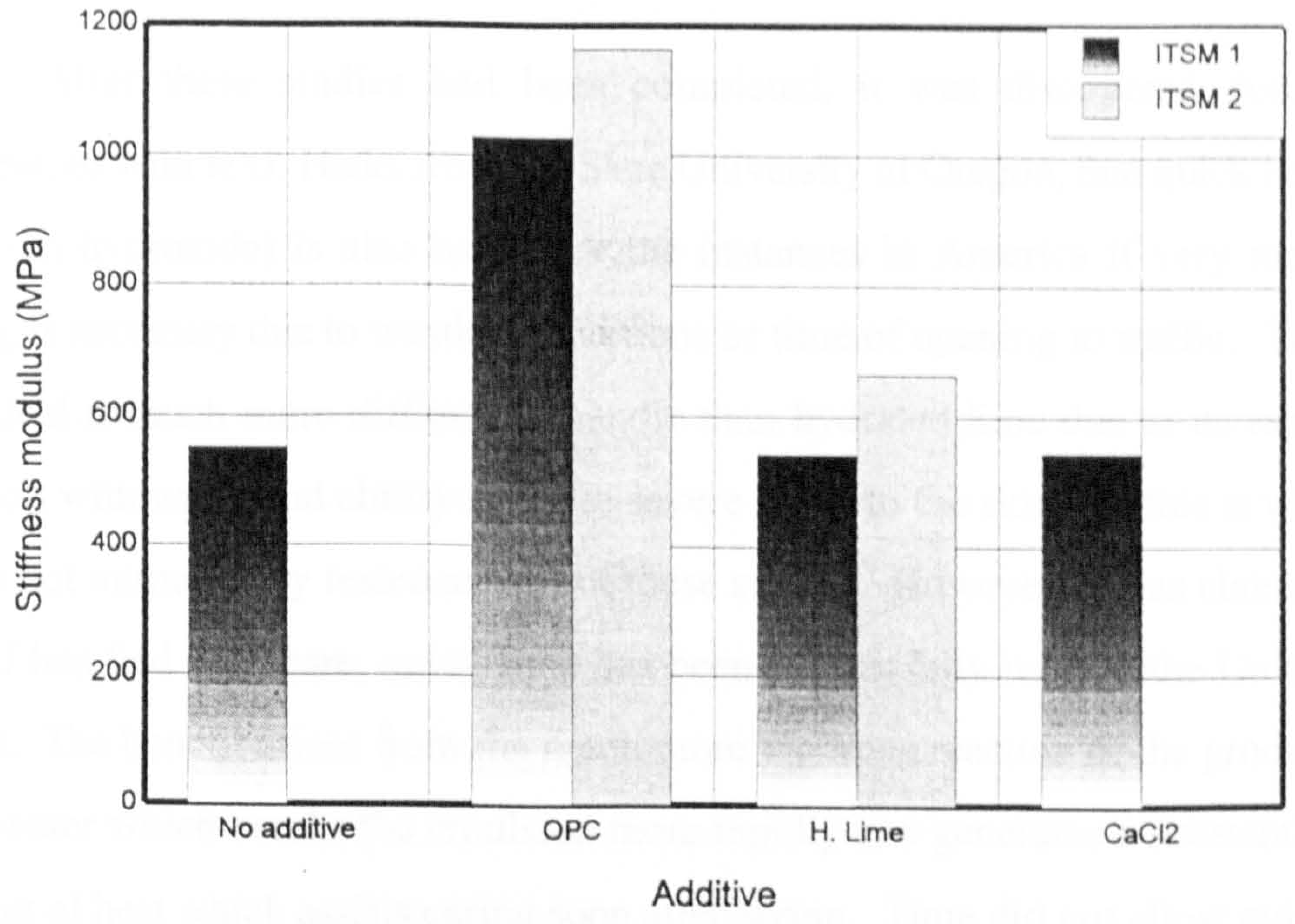


Figure 8-9 : Stiffness moduli of mixtures with additives subjected to soaking in water

Discussion

The results shown in Figure 8-9 reveal a number of interesting facts :

- The mixture with OPC had a higher stiffness than the other mixtures after the first soaking period.
- Only the mixtures with OPC and hydrated lime survived the second soak
- The mixtures with OPC and lime increased in stiffness very slightly during the second soak but this was much less than has been observed if samples were stored dry

These results seem to conclude that OPC and hydrated lime are effective adhesion agents for emulsion mixtures. Without these additives, the mixtures studied in these tests had poor resistance to water damage. As the addition of

$CaCl_2$ to the emulsion did not offer any improvement, this material is ineffective and indicates that the addition of Ca^{2+} ions alone is not the mechanism by which OPC and hydrated lime work.

After these studies had been completed, it was discovered during discussions with R.G. Hicks from the State University of Oregon, that quick lime (calcium hydroxide) is also used in some instances in America if very rapid curing is necessary due to weather conditions or time of opening to traffic. This chemical is much more difficult to handle than hydrated lime due to its rapid reaction with water and ability to cause severe burns to the skin and this is why it was not immediately tested as part of these studies. However, it was claimed that if handled with care, quick lime has been successfully used in the United States. The benefit arises from the much more vigorous reaction of the product with water which breaks the emulsion more rapidly and generates a substantial amount of heat which assists curing soon after laying. Time did not allow quick lime to be evaluated in these studies but it is suspected that it may be quite effective and should, therefore, be tested in the future in the types of mixtures used herein.

In order to directly measure the adhesion effect of OPC, a simple boiling adhesion test was carried out according to a standard ISSA test (133) normally used to test wet stripping resistance of cured slurry seal mix. Uncompacted samples of mixtures with and without cement were fully cured in an oven at 60 °C for 3 days. 10 g samples were boiled in water in a beaker for 3 minutes and allowed to cool. When ebullition had ceased, cold water was run into the beaker until the overflow became clear. The mixtures were removed from the beakers and placed on a filter paper. A visual appraisal of the percentage coverage of the mixtures was then made. The mixture without OPC had only 50 % coverage whilst the mixture with 1% OPC still had 95 to 100 % binder on its surface. This illustrates the improved adhesion of the binder in the presence of OPC.

8.7 OVERALL CONCLUSIONS

The tests described above allow the following conclusions regarding the effect of OPC (and hydrated lime) on dense bitumen emulsion macadam mixtures to be drawn :

- OPC increases rate of increase in stiffness modulus
- OPC increases final stiffness
- OPC increases resistance to permanent deformation
- OPC increases resistance to fatigue cracking at initial strains below 200 μstrain
- OPC reduces resistance to fatigue cracking at initial strains above 200 μstrain
- OPC and hydrated lime improve resistance to water damage

CHAPTER 9

EFFECT OF OTHER VARIABLES ON THE INDIRECT TENSILE STIFFNESS MODULUS OF BITUMEN EMULSION MIXTURES

9.1 INTRODUCTION

Chapter 5 explained how the stiffness modulus of a road mixture relates to its performance in the pavement. It is well known that unlike hot mixed materials, bitumen emulsion mixtures take time to build up stiffness (15, 16). The studies reported in chapter 8 showed that OPC is effective in enhcancing stiffenss of cold mix but this option adds cost and could present some handling problems on a large scale. In 1981, Mamlouk wrote about the effect of a number of variables on emulsion mixtures as determined using Marshall and resilient modulus tests (134). He concluded that emulsion mixtures take time to cure and he theorized that this was partly dependent upon voids, in that voids enabled water to evaporate. However, low densities were also found to have an adverse effect on stiffness. In addition, it was noted that an increase in binder content caused a reduction in resilient modulus which is in good aggreement with the results of tests reported in chapter 7 of this thesis.

The performance of emulsion mixtures based on traditional British designs used for hot mix have not been quantified and the processes which are at work during the curing process are not fully understood. The aim of the work described in this chapter was, therefore, to establish how an emulsion mixture based on traditional British hot mix recipes would perform over time and in what way various parameters other than the use of OPC could be used to improve performance in terms of the rate of stiffness build up and ultimate stiffness.

The variables studied were :

- The effect of aggregate grading
- The effect of base binder penetration grade
- The effect of emulsifier level
- The effect of compaction density
- The effect of polymer modified binders

The parameters tested would undoubtedly have an affect on other mechanical properties and possible durability, but time did not allow these to be studied.

9.2 EXPERIMENTAL

9.2.1 Materials

The same granite aggregate as that used in the work described in the previous chapter from CAMAS Croft, was used throughout the tests described here. Similarly, the grading was again based on the 20 mm DBM mixture from BS 4987 Part I as shown in Figure 8-1 and in most cases, the gradings were in the middle of the specification. The bitumen emulsions described in Table 6-2 were used.

9.2.2 The effect of aggregate grading

Previous work carried out at the University of Nottingham on design of hot mix, has shown that by using slightly different grading curves than those specified for DBM mixtures in BS 4987 Part I or the Fuller curve (146) (9-1), improvements in resistance to permanent deformation could be achieved (106). These grading curves are calculated using equation 9-2 shown below.

$$P=\left(\frac{d}{D}\right)^{0.45} \qquad (9\text{-}1)$$

Where : P = percentage of material passing sieve size d mm

D = Maximum particle size

Exp. 0.45 = maximum density gradation

$$P = \frac{(100-F)(d^{n}-0.075^{n})}{(D^{n}-0.075^{N})} + F \qquad (9\text{-}2)$$

Where :

P = percentage of material passing a sieve of size d mm

D = maximum particle size, mm

F = filler content, %

n = an exponent between 0 and 1

This formula differs from the traditional Fuller equation (9-1) in that it allows the engineer to set the filler content thus opening-up the mixture. The exponent, n, controls the balance between the coarse and the fine aggregate. Opening up the grading increases the dominance of the coarse aggregate skeleton. This has been found to increase resistance to permanent deformation, not at the expense of stiffness, for mixtures with equal binder and void contents.

The Nottingham equation (9-2) allows the construction of a continuous grading between an upper value (the maximum particle size) and a lower bound value (the filler content). The exponent value dictates the shape of the curve with n = 0.45 corresponding to the maximum density gradation. As gradings based on this equation offer the possibility of 'opening up' the grading, it was felt that they may be beneficial for emulsion mixtures for several reasons. Emulsion mixtures inherently have a higher total fluids content during mixing and compaction due to incorporation of water through both the emulsion and prewet water. The presence of this water is problematic in two senses. Firstly, during compaction of a mixture, the fluids tend to fill or partially fill the voids. If water is unable to flow out, as seems to be the case in dense mixtures noted in the trials described in chapter 7, it prevents full compaction of the mixture due to hydraulic pressure resistance. After evaporation of the water has taken place, the mixture will be left with a high void content which has been found to weaken the mixture in the case of hot mix (106). Failure to reach adequate levels of compaction will also lead to a deformable layer under the action of traffic. The second problem is that water must be lost, either during compaction or by evaporation after compaction, in order for the emulsified bitumen to revert to its original form. This is more difficult in dense mixtures having fewer openings to allow water to escape.

Mixture design

Grading curves were calculated for exponents n = 0.5, 0.6, 0.7 using equation 9-2 above, with a filler content of 5.17 % and a maximum aggregate size of 20 mm. The 5.17 % filler content was selected as it was in the middle of the BS 4987 specification and had been used previously. A mid 20 mm DBM mixture was also to be made as a standard. These curves are shown in Figure 9-1 along with the 20 mm DBM envelope from BS 4987 for comparison. It can be seen that the gradings become more open with increasing exponent and that the curves are more even compared with the 20 mm DBM envelope.

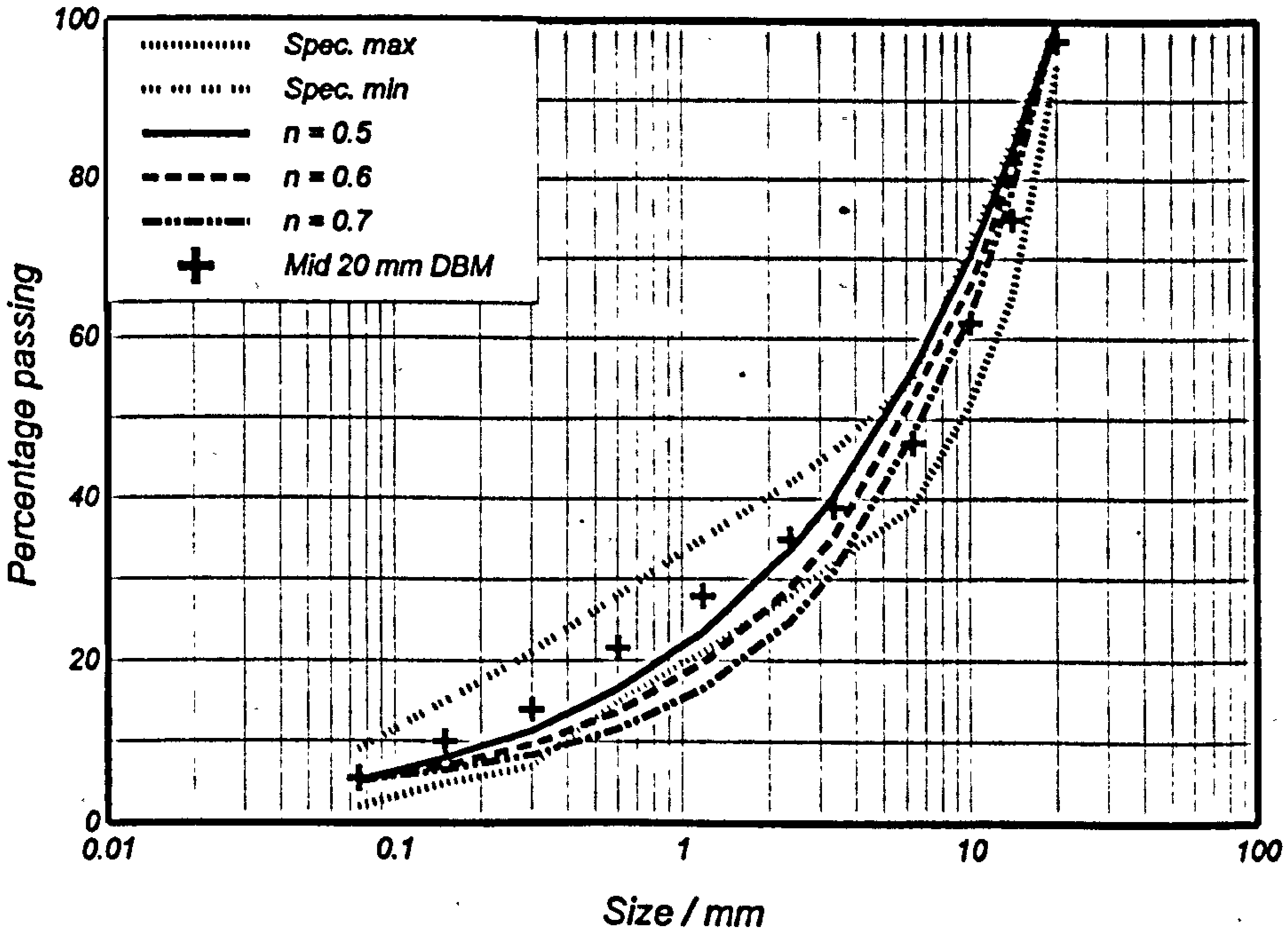


Figure 9-1 : Mid BS 4987 20 mm DBM aggregate grading

In practice, using the analyses of the 20, 14, 10, 6 mm and dust fractions supplied by CAMAS Croft, aggregate gradings were constructed which adhered very closely to the target gradations.

Pre-wet water addition levels were determined in small scale mix tests. It was found that slightly less was required in the more open mixtures which is a benefit in itself. The amounts used in the full scale mixtures were as shown below :

Mid 20 mm DBM	:	2.5 %
n = 0.5	:	2.5 %
n = 0.6	:	2.2 %
n = 0.7	:	2.2 %

The standard emulsion as described in Table 6-2 was used at a level of 8.06 % in all mixtures, which corresponds to a residual binder content of 4.7 % on the mixture.

Manufacture and testing of specimens

Mixtures were prepared using a Hobart mixer using a mix time of ~ 45s at a temperature of 20 °C. The mixtures were compacted using 200 gyrations on a Gyratory compactor, with a stress of 0.6 MPa and an angle of gyration of 1°, to make 3 specimens of each mixture. The specimens were stored at 20 °C and stiffness moduli determined at 20 °C periodically using the NAT. After the measurement made at 77 days, the samples were placed in an oven at 60 °C for 3 days to simulate full curing. Stiffness moduli were again measured and these figures were taken as the ultimate potential stiffness. These figures are plotted on the graph at 365 days but this is an arbitrary time as it is not known how long the samples would have taken to fully cure at ambient temperatures.

The maximum density of the uncompacted mixture and the densities of the cores after full cure were determined using the rice pot and weighing in air and water methods respectively. The voids contents were calculated from the results of these tests.

Results

The results obtained from the ITSM tests were as shown in Figure 9-2. The voids contents for the specimens were as shown in Table 9-1.

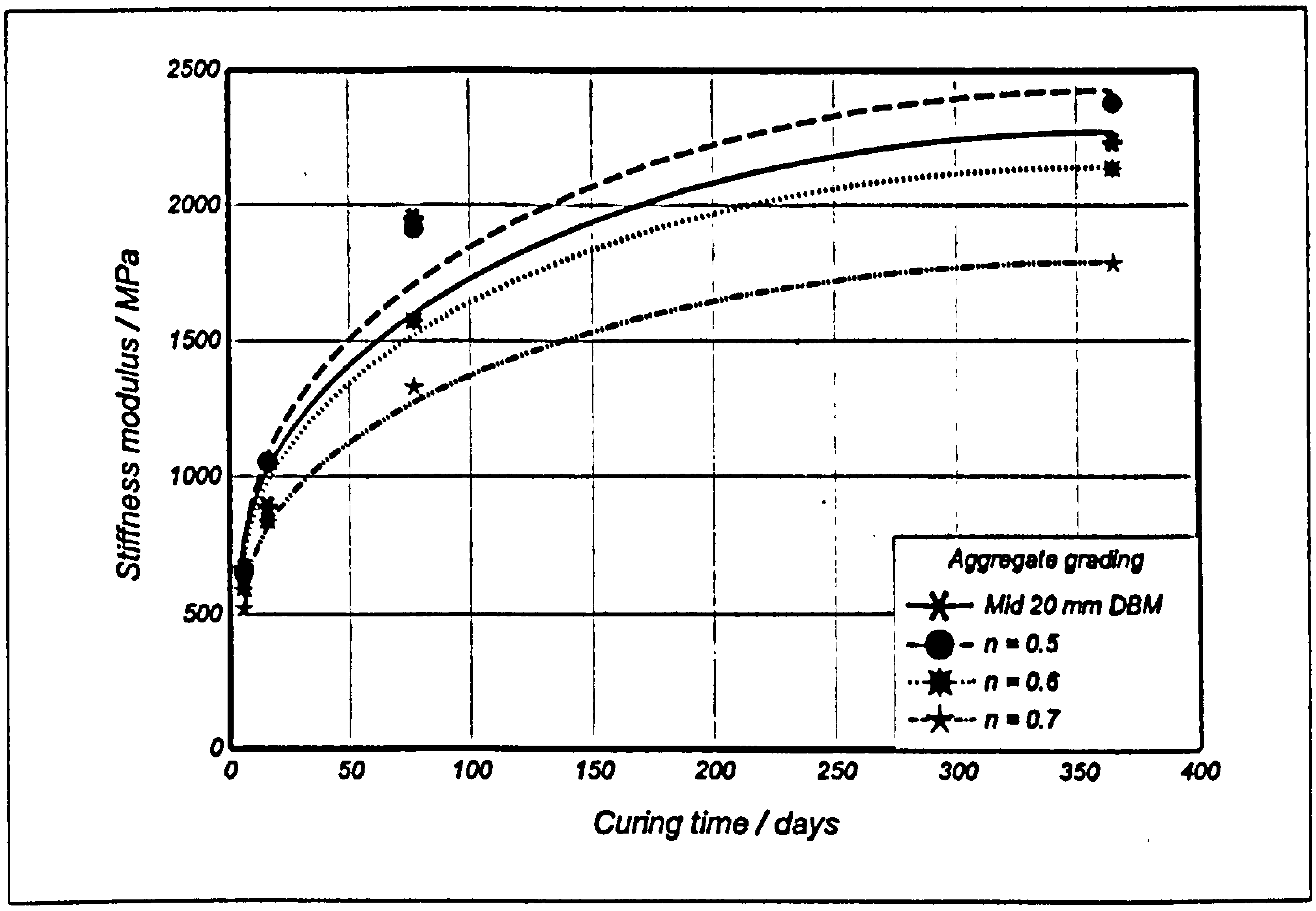


Figure 9-2 : Stiffness moduli of mixtures with different aggregate gradings

Table 9-1 : Void contents of specimens with different aggregate gradings

Aggregate grading	Voids (%)
Mid 20 mm DBM	7.3
n = 0.5	7.3
n = 0.6	7.7
n = 0.7	8.5

It is interesting to note that the void contents obtained in these specimens using the Gyratory compactor were significantly lower than those achieved in other parts of this research where the Marshall hammer, PRD apparatus or roller compactor were employed. This is a consequense of the shearing action which arises in gyratory compaction. Despite the increased densities, the stiffness results were not substantially different from those obtained for mixtures compacted using other techniques.

Discussion

The results obtained from these tests show very clearly that no discernable benefit in terms of stiffness modulus was gained by the use of aggregate gradings based on equation 9-2. The n = 0.5 mixture performance was virtually equal to that of the mid 20 mm DBM mixture but those based on exponents of 0.6 and 0.7 (ie more open) were inferior.

This is similar to previous experience encountered during the design of mixtures for the TRL trial described in chapter 7. Opening up of the aggregate grading always results in a decrease in stiffness modulus of the mixture. As the the void contents of the specimens were found to increase with increasing exponent with the n = 0.5 mixture being equal to the standard, this is probably the reason behind the small differences in stiffness results. Irrespective of the compaction and curing factors described above, final mixture density would appear to be the overriding factor.

It should be stressed that the mixtures investigated here all had the same binder content and were given the same amount of compaction. In the original thesis from Nottingham (112), for each aggregate grading, three levels of binder and compaction were tested resulting in a total of 27 combinations. The simplification of the test carried out here may have resulted in a combination of binder content, compaction density and aggregate grading based on the exponent curves, which would have given improved performance, being missed.

9.2.3 Effect of base binder penetration grade

Mixtures based on emulsions containing Venezuelan bitumens with a range of penetration grades were tested.

Mixture design

The mid 20 mm DBM aggregate grading was used. Mixtures were prepared with and without 1 % OPC. 2.5 % prewet water was added to the mixtures without OPC and 3 % for those with OPC. An emulsion level of 8.06 % was used in all mixtures. The emulsion formulations are shown in Table 6-2. All contained 1.2 % emulsifier and 62 % bitumen. The bitumens ranged from 50 to 300 pen.

Specimen manufacture

Mixtures were prepared on a Hobart mixer. It was noted during manufacture that the emulsion containing the softest binder tended to strip off the aggregate to some extent. It was felt that this could have been due to the presence of lighter oil fractions in the bitumen which are known to have a detrimental effect on adhesion, as a result of research carried out by other workers at Akzo Nobel's laboratories on related projects. The mixtures were compacted using a Marshall hammer and then stored at 20 °C during the ITSM test period. After 15 days ambient curing the specimens were placed in an oven at 60 °C for 3 days to simulate full curing. The ultimate potential stiffness was measured on the fully cured samples. Void contents were determined as described in chapter 5.

Results

The results of the ITSM tests and void contents are shown in Figure 9-3 and Table 9-2 respectively.

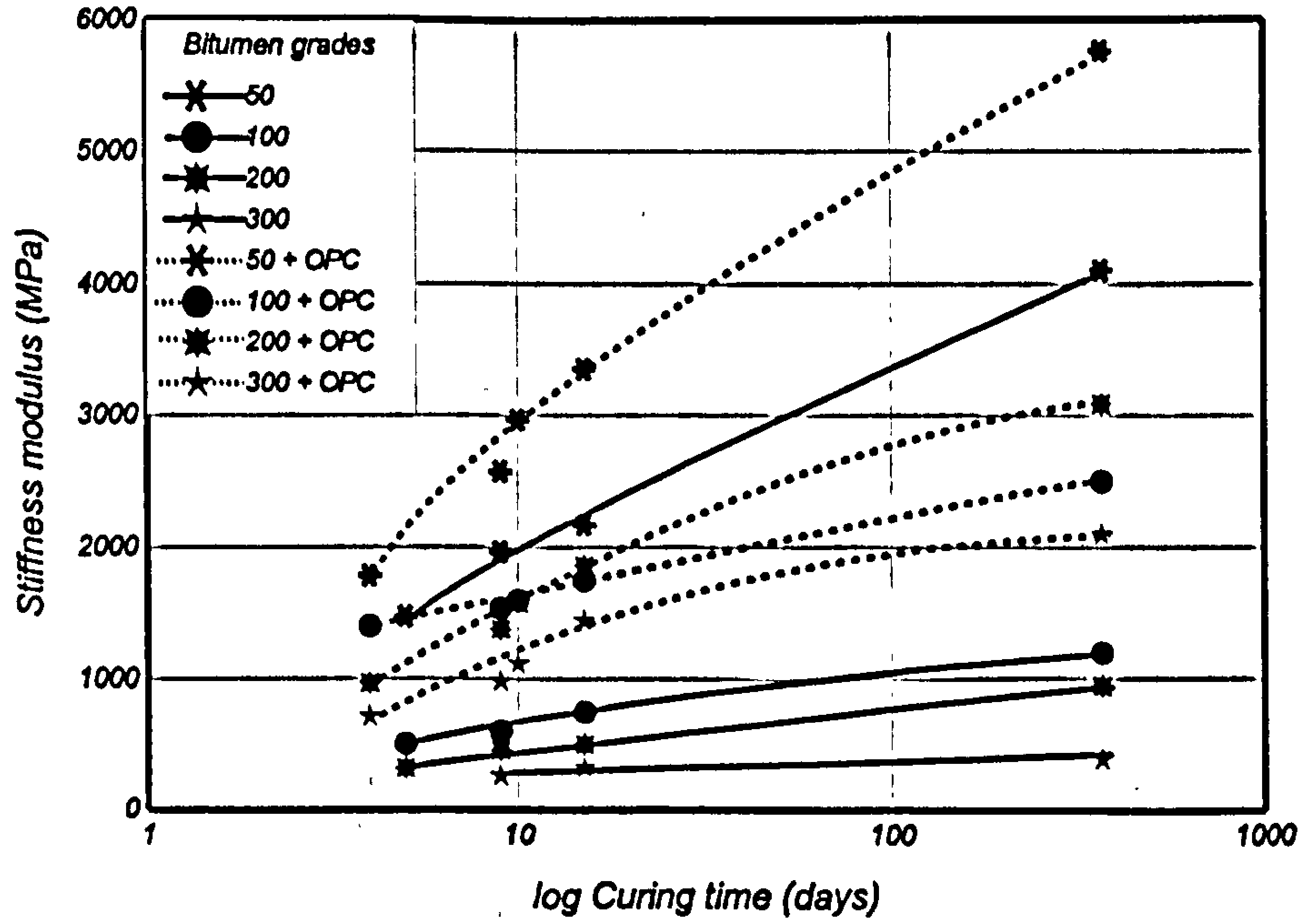


Figure 9-3 : Stiffness moduli of mixtures with range of bitumen grades

Table 9-2 : Void contents of specimens with different bitumen grades

Bitumen grade	Percentage voids in mixture
50 pen	13.7
100 pen	9.9
200 pen	10.7
300 pen	9.1
50 pen + OPC	10.9
100 pen + OPC	10.7
200 pen + OPC	11.0
300 pen + OPC	11.0

Discussion

The results shown in Figure 9-3 show that the harder the grade of bitumen used in the mixture the higher the stiffness modulus obtained. The addition of 1 % OPC raised the stiffness of mixtures with all grades of binder to the same degree. It is interesting to note that in the mixtures without OPC, the void contents of the compacted specimens decreased with increasing pen grade. This suggests that softer bitumen gave rise to more workability or compactability. The results from the coalescence tests described in chapter 10 showed that emulsion break under compaction increased with increasing binder penetration grade. It is, therefore, surprising that an increase in compactability was observed in these tests. It is possible that even though emulsions with softer binders break more quickly, the binders themselves still allow more compaction due to their reduced viscosities.

9.2.4 The effect of emulsifier level

As discussed in chapter 2, emulsifier level and distribution between waterphase and binder / water interface affects the rate of coalescence and break of an emulsion. The tests described here were carried out in order to determine whether or not dosage has an effect on stiffness modulus.

Mixture design

The mid 20 mm DBM aggregate grading was used. Mixtures were prepared with and without 1 % OPC. 2.5 % prewet water was added to the mixtures without OPC and 3 % to those with OPC. An emulsion level of 8.06 % was used in all mixtures. The emulsion formulations were as shown in Table 6-2. All contained 62 % 100 pen bitumen. Emulsifier levels of 0.8, 1.2 and 1.6 % were used.

Specimen manufacture

Mixtures were prepared using the Hobart mixer. There was a noticeable increase in workability with increasing emulsifier level. The mixtures were compacted using the Marshall hammer with 50 blows to each side. Moulded specimens were stored at 20 °C for 15 days and then fully cured in an oven at 60 °C for three days. Stiffness moduli were measured periodically in the NAT. Void contents were determined using the methods described in chapter 5.

Results

The results of the ITSM tests and void contents are shown in Figure 9-4 and Table 9-3 respectively.

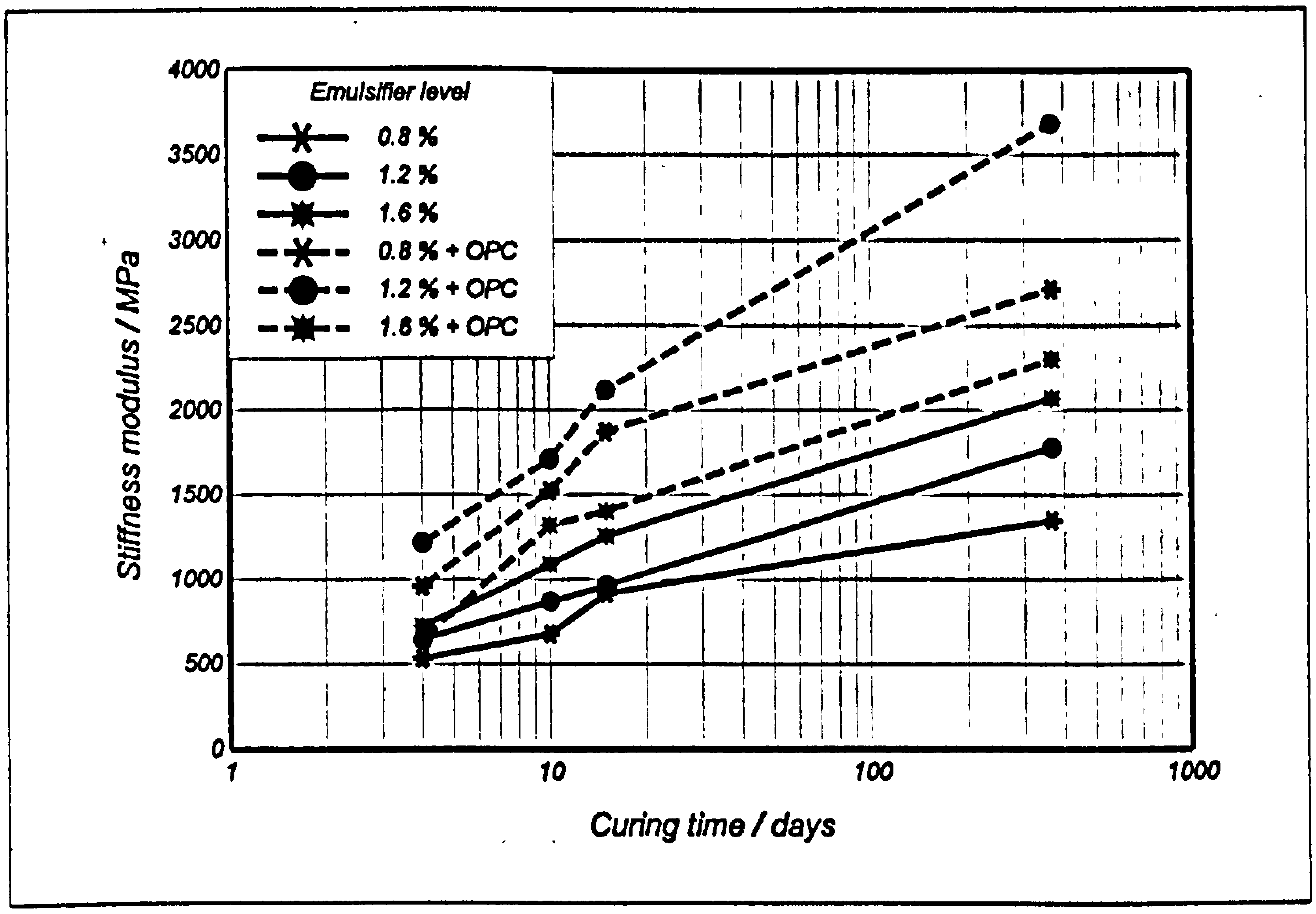


Figure 9-4 : Stiffness moduli of mixtures with range of emulsifier levels

Table 9-3 : Void contents of cores with different levels of emulsifier

Emulsifier level (%) / OPC	Voids (%)
0.8	12.2
1.2	12.5
1.6	10.7
0.8 + OPC	12.4
1.2 + OPC	12.0
1.6 + OPC	12.0

Discussion

As expected, the specimens containing cement were stiffer than those without cement. As the voids contents for all of the samples were similar, the differences in stiffness can be attributed to the state of the binder rather than the physical nature of the mixture. Bearing this in mind, the results are confusing when compared with those from the coalescence tests described in chapter 10. As a result of the coalescence tests, it was suggested that the higher the level of emulsifier, the lower the amount of emulsion break. The data here, particularly for the mixtures without OPC, suggest that this does not manifest itself in untreated mixture stiffness. In the mixtures without OPC, stiffness increased with increasing emulsifier level which seems to be in direct contradiction. In the case of mixtures with OPC, the data do not follow a trend in either direction. However, the timescales in the two tests were different. The coalescence tests were concerned with the time immediately after compaction and were, therefore, dependent on emulsion break, whereas stiffness measurements did not begin until 5 days after manufacture so curing was more prevalent in this case.

9.2.5 Effect of compaction density

In the case of hot mix, it is well known that variation in void content over a range of only 3 to 7 % can have a dramatic effect on stiffness modulus (114). The void contents of cold mix specimens produced in these studies and elsewhere are often in the order of 10 to 12 %. This is due to the use of compaction methods which are unable to force much of the water out of the mixture. In mixtures containing ~8 % of a 62 % bitumen emulsion and 2.5 to 3 % pre wet water, it can be easily calculated that the total water content is ~ 5.5 to 6 % by weight. This corresponds to ~ 15 % by volume due to the densities of water and the main mixture ingredient - aggregate - being 1 and ~ 2.6 respectively. If the majority of this water remains in the mixture after compaction, after full evaporation has occurred, the mixture will be left with a corresponding level of air voids.

If water could be forced out of a mixture during compaction, a material with a much lower void content would result. This should result in an increase in stiffness according to results obtained from hot mix studies. The tests described here were carried out to assess the effect of void content on the stiffness modulus of cold mix.

Mixture design

The mid 20 mm DBM aggregate grading was used. Mixtures were prepared with 1 % OPC and 3 % prewet water. The standard emulsion as described in Table 6-2 was used at a level of 8.06 % on the aggregate.

Specimen manufacture

Mixtures were prepared in the Hobart mixer and compacted to different target densities on the Gyratory compactor. Target densities were calculated in order to obtain void contents of 5, 7, 9 and 11 % in the final dry specimens. The same compaction load of 0.6 kPa and 1 ° angle of gyration (provisionally recommeded for a CEN standard) were used for all compaction sequences and higher densities were achieved through larger numbers of gyrations. The specimens were stored at 20 °C and periodically tested in the NAT.

Results

The results of stiffness modulus tests are shown in Figure 9-5.

The average number of gyrations required for compaction and the actual void contents obtained were as shown in Table 9-4.

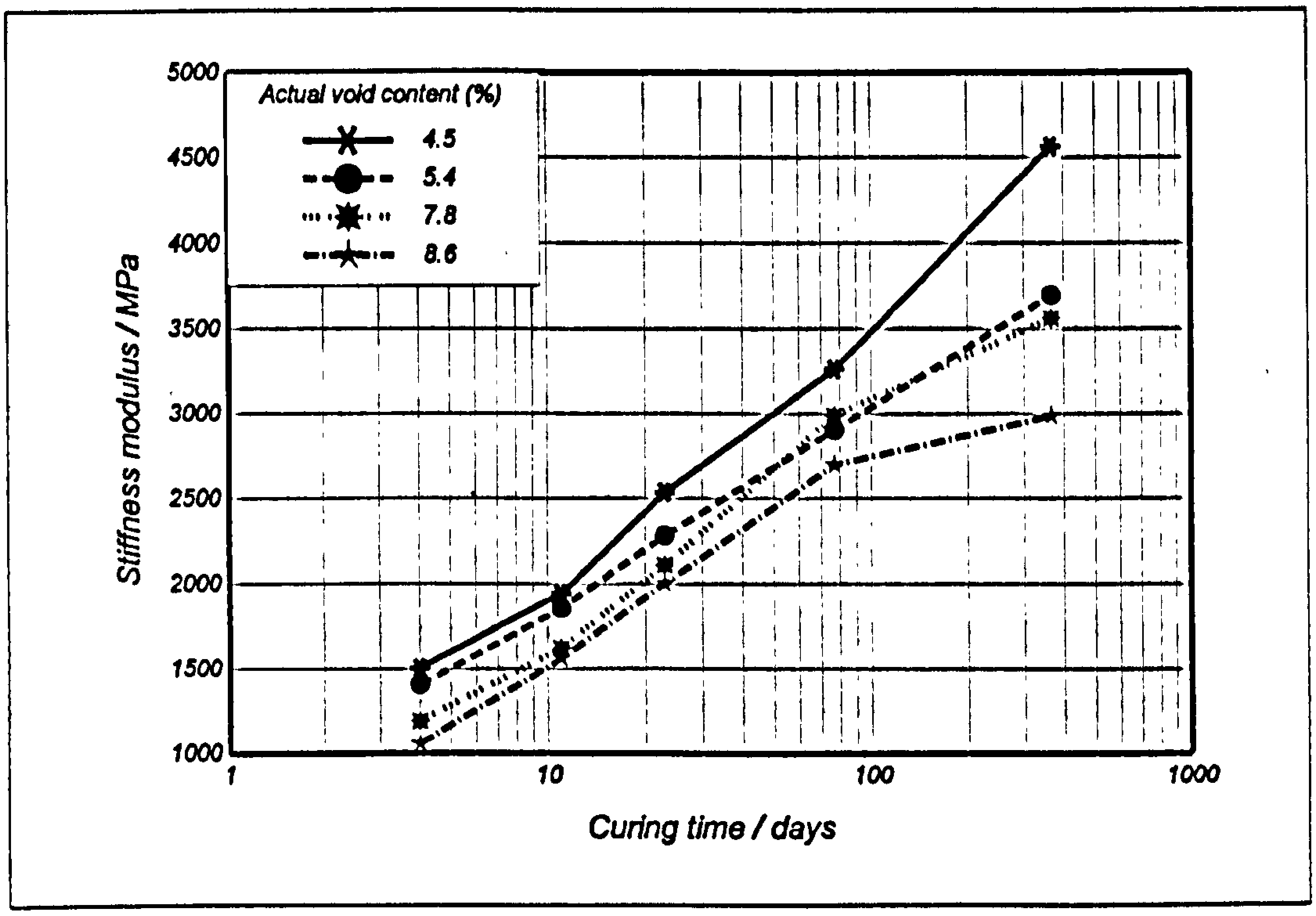


Figure 9-5 : Stiffness moduli of mixtures with range void contents

Table 9-4 : Number of gyrations and void contents

Target void content (%)	Average number of gyrations	Actual void content (%)
11	45	8.6
9	63	7.8
7	156	5.4
5	280	4.5

Discussion

The actual void contents determined for the cores manufactured for these tests were not very close to the intended values. However, a range of void contents was obtained even though it was narrower than the desired one. The reason for the error is not known but it was most probably due to insensitivity of the density measurement facility of the gyrocomp equipment.

During gyratory compaction, fluids were seen to flow out of the mixtures;

the amount dependent upon the level of compaction. The fluids did not consist of solely clear water and it was evident that a certain proportion of the binder was also lost. In order to find the binder contents of the specimens it would have been advantageous to collect the run-off 'water' and determine the binder content. Alternatively, the residual binder contents of the samples could have been measured directly by extraction. Reduction in binder content would undoubtedly have had an effect upon the stiffness moduli of the specimens.

Stiffness moduli were found to increase with decreasing void content in line with the behaviour of hot mix, even over the limited range available in these tests. If the range were increased further up to ~11 %, as was intended, a much greater spread of stiffness would probably have been obtained, as has been found previously in other tests described above, in which less effective methods of compaction were employed.

As can be seen in Table 9-4, significantly more gyrations were necessary to produce cores with lower void contents. This extra mechanical energy input may have induced more emulsion break during compaction which could have been an additional factor in stiffness build up.

9.2.6 The effect of polymer modified binders

Polymer additives have been incorporated in bitumen for over 20 years in order to enhance performance. Polymer modified binders have been used in surface dressing, slurry seal and tack coat emulsions and in hot mix. Porous asphalt or pervious macadam (an extremely open graded mixture which reduces surface spray by enabling water to drain away) requires the use of polymer modified binders due to the high stress placed on the binder in the absence of fines. Polymer modified binders have been included in bitumen emulsion mixtures relatively recently.

Polymer types

Unfortunately, only a handful of the vast array of polymers which are available today are miscible with bitumen (5). When mixed in the correct manner, they form homogeneous and stable blends which can be used as normal binders or emulsified. The compatible polymers which are commonly used are:

EVA (Ethylene vinyl acetate) - These polymers are plastomers (polymers which exhibit visco plastic properties) composed of ethylene and vinyl acetate. Different grades are available based on the relative amounts of the two components and molecular weight. When added to bitumen they have a stiffening effect.

SBS (Styrene butadiene styrene) - This family of polymers are classified by their styrene content, molecular weight and structure (ie linear or branched). They are thermal plastic elastomers and therefore increase the elasticity of bitumen. Similar polymers composed of styrene and isoprene (SIS) are also used.

These polymers are added to hot bitumen. SBS requires intensive high shear mixing to ensure satisfactory distribution but EVA can merely be stirred in as it melts.

Prior to the availability of these synthetic polymers, natural rubber was sometimes added to bitumen to increase its resistance to cracking. Similarly, latices (emulsions of rubber) were also utilized but these are only applicable in bitumen emulsions. In addition to natural rubber latex, there are many synthetic latices with different chemical compositions and structures available, as either anionic or cationic emulsions. Obviously, a latex must have the same charge as the bitumen emulsion it is added to or the two emulsions will break on mixing. Latices are added either via the water phase or to the emulsion after manufacture. The latex and bitumen droplets exist separately in the emulsion and so these systems are termed "biphase" emulsions. The latex and bitumen only mix after the emulsion has broken.

Measurements described above showed that without the addition of cement, the stiffness moduli of emulsion mixtures are generally lower than those of equivalent hot mixes. It has been previously observed that the use of polymer modified binders can have a beneficial effect in emulsion mixtures (86). The following tests were, therefore, carried out in order to investigate whether any performance enhancement could be achieved in the DBM mixtures studied in this project.

Mixture design

The mid 20 mm DBM aggregate mixture was used. Emulsions as described in Table 6-2 were used at a level of 8.06 % on the aggregate mixture. Mixtures were prepared with polymer modified emulsions only and also in combination with OPC at a level of 1 % on the dry aggregate mixture.

Specimen manufacture and testing

Mixtures were prepared using a Hobart mixer. Mixtures were compacted using 50 blows of the Marshall hammer to each side of the sample. The specimens were stored at 20 °C and 50 % relative humidity and tested in the ITSM test periodically. After the final ambient ITSM measurement, specimens were fully cured in an oven at 60 °C for 3 days. The stiffness of the fully cured specimens were determined and these results are plotted as 365 days curing. Void contents were determined by measurement of maximum mixture and specimen densities as described in chapter 5.

Results

The results of the ITSM tests on mixtures with polymer modified emulsions without OPC and the void contents of these specimens were as shown in Figure 9-6 and Table 9-5 respectively.

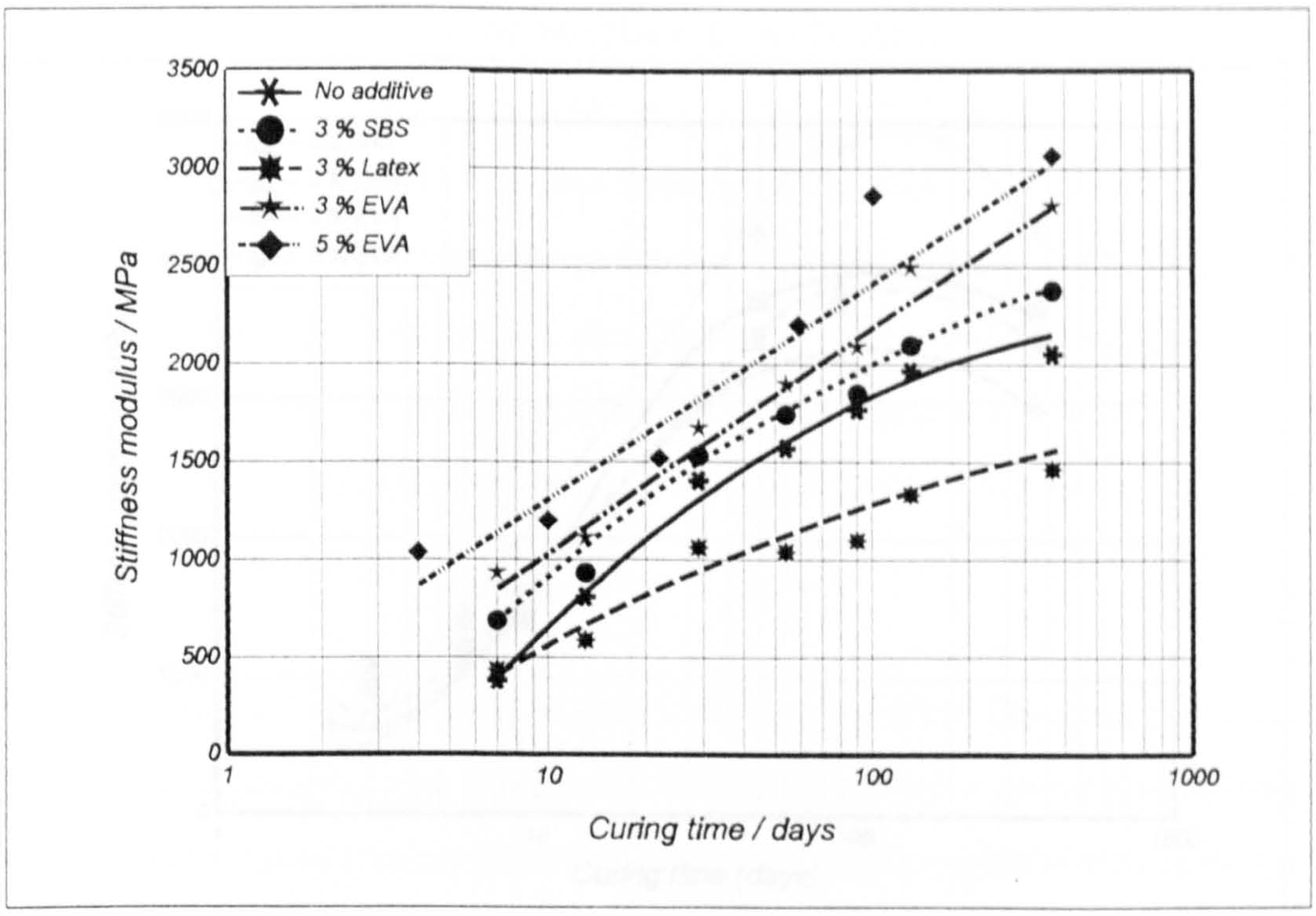


Figure 9-6 : Stiffness moduli of mixtures with polymer modified emulsions

Table 9-5 : Void contents of specimens with polymer modified emulsions

Polymer additive	**Void content (%)**
None	10.0
3 % SBS	10.8
3 % Latex	12.1
3 % EVA	10.0
5 % EVA	10.0

The results of the ITSM tests on mixtures with polymer modified emulsions in combination with 1 % OPC and the void contents of these specimens were as shown in Figure 9-7 and Table 9-6 respectively.

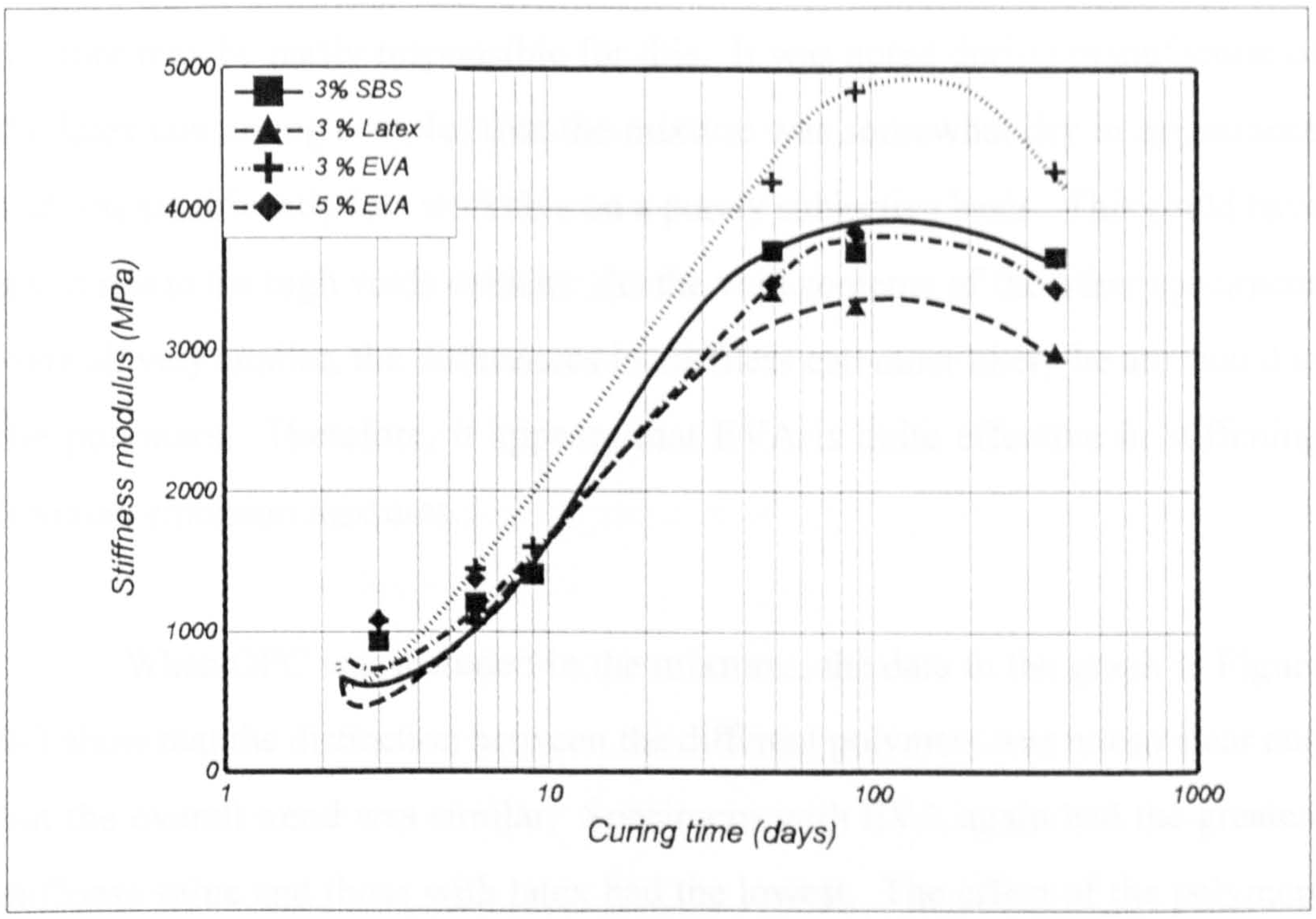


Figure 9-7 : Stiffness moduli of specimens with polymer modified emulsions and 1 % OPC

Table 9-6 : Void contents of specimens with polymer modified emulsions and OPC

Polymer additive	Voids (%)
3 % SBS	11.0
3 % Latex	12.2
3 % EVA	10.2
5 % EVA	9.8

Discussion

The results shown in the graph in Figure 9-6 very clearly show the effect of polymer modification of emulsions used in cold mixtures. The addition of EVA made the greatest improvement in stiffness and dosage was also significant. SBS caused an increase in stiffness as well but this was not as large as the effect of EVA. The inclusion of latex actually produced mixtures with lower stiffness than those without any modification. The high void content of the latex modified mixture may be partly responsible for this. It was noted during manufacture of the latex containing samples that the mixture was somewhat dry in appearance and was significantly less workable on a purely subjective basis. This could have given rise to the high voids content. As the void contents of the other specimens were all very similar, the differences in stiffness can most likely be attributed to the polymers. Therefore, it appears that EVA is quite effective in stiffening bitumen emulsion mixtures.

When OPC was included in the mixtures, the data in the graph in Figure 9-7 show that the distinction between the different polymers was not so clear cut, but the overall trend was similar. Specimens with EVA again had the greatest stiffness value and those with latex had the lowest. The effect of the polymers was probably masked to some extent by the very significant effect of the OPC. Comparing the data in this graph with the line in Figure 8-2 for a similar mixture with 1 % OPC and no polymer, it can be seen that the polymers led to an overall increase in stiffness

An unusual feature of the data in Figure 9-7 was the fall off in stiffness after oven curing in all of the specimens. The reason for this is not known but it may have been due to the following :

- degradation of the polymer
- alteration of OPC/bitumen/polymer mastic (ie change in the way in which the components are associated)

Other possible reasons for the drop could have been incorrect ITSM test temperature or damage of the sample in the ITSM test prior to oven curing. As these problems were not evident in any other ITSM tests these are less likely.

9.3 OVERALL CONCLUSIONS

The results of the tests described above can be summarised as follows :

- Alteration of the grading from the middle of the BS 4987 20 mm DBM specification did not offer any benefits in stiffness
- Harder base bitumen led to higher stiffness
- Stiffness was increased in some mixtures by using emulsions with a higher emulsifier content
- Lower void contents gave rise to higher stiffness
- Mixtures with emulsions containing bitumens modified with EVA and SBS polymers had higher stiffness values than those with unmodified binders

CHAPTER 10

STUDIES ON BREAKING OF BITUMEN EMULSIONS IN MIXTURES

10.1 INTRODUCTION

The biggest problem which has plagued cold mix since its inception, is its slow curing rate (15, 16). Hot mix should approach its full design strength as soon as the bitumen has cooled to ambient temperature and thus reverted almost to its original properties, apart from a certain amount of hardening as a result of heating. The process is much slower and more complicated for emulsion mixtures. Immediately after compaction it is clear, from studies of mechanical properties described earlier in this report, that cold mix has a limited amount of strength arising from interlock and friction of the aggregate structure, cohesion arising from the capillary action of water in the mixture and possibly a small amount of cohesion from the partially coalesced bitumen. The latter is dependent in magnitude on the amount of coalescence of bitumen which has occurred during compaction and its adhesion to the aggregate. Coalescence is the term used to describe the transition from emulsified to continuous bitumen, ie the joining together of bitumen droplets as discussed in Chapter 2. It is this process which produces an effective bituminous binder from the bitumen emulsion. Binder cohesion is necessary to give the mixture its stiffness, tensile strength and durability. Coalescence is not only important in mechanical terms but also in the event of precipitation, as unbroken emulsion is dispersible in water and can, therefore, simply be washed away by water. In the days and weeks following compaction, a mixture will continue to cure by the processes of chemical action and the evaporation of water. Both are very slow processes and are reliant to a large extent on the weather conditions, which are often unfavourable in many countries. It is, therefore, essential that the majority of the emulsion coalescence or break occurs during compaction.

Testing of the mechanical properties of bitumen emulsion mixtures such as that described earlier in this thesis does not directly measure coalescence. In order to gain an insight into the curing process, a test method was developed to measure the quantity of emulsified bitumen which coalesces during compaction and in the early life of a compacted mixture to give a continuous bituminous phase which is resistant to water rinsing. The effects of the following range of parameters were investigated :

- bitumen penetration grade
- emulsifier level
- compaction regime
- cements
- polymer modification of binder
- storage time
- temperature
- breaking agents

10.2 COALESCENCE OF BITUMEN EMULSION ONTO AGGREGATE IN MIXTURES

10.2.1 Test procedure

In summary, the test involved mixing an aggregate sample with emulsion, followed by compaction, rinsing off of the free emulsion and finally measurement of the retained bitumen content by solvent extraction. This being taken as the percentage coalescence of the emulsion during compaction.

Aggregate preparation

In order to facilitate the final rinsing stage of the test, a "fines free" aggregate sample was required. A fraction of Bardon Hill aggregate (Porphyrite) with a particle size of <1.18mm was sieved out of the dust fraction. This was wet sieved on a 300 μm sieve for about 1/2 hour to remove as much of the <300 μm material as possible. The remainder was removed by repeatedly washing the aggregate in a large vessel and pouring off the supernatant containing the fines, until the washings were completely clear. The 300 µm to 1.18 mm fraction thus prepared was fully dried in an oven at 120 °C. This aggregate fraction was used because it was not possible to use a realistic, graded aggregate mixture as the fines content and large stone fractions could not be accommodated due to the nature of the test.

Mixture preparation and compaction

A 200 g portion of the aggregate was used in the mixtures. Pre-wet water, additives and bitumen emulsion (20 g) were added and mixed as dictated by the test in question. The bitumen emulsions used were those described in chapter 6. The mixture was immediately placed in a Marshall mould, in most cases, and compacted as required by the particular test. The specimen was quickly removed from the mould, broken up, and placed in a glass jar. A 300 g measure of a 2% solution of quaternary amine was added and the jar sealed, placed on a set of rollers and rolled for 20 minutes. The purpose of this procedure was to redisperse and remove all of the bitumen which may have been flocculated but not fully adhered to the aggregate. After rolling, the contents of the jar were repeatedly rinsed with cold water. The supernatant was decanted off until all of the free emulsion had been removed. The sample, consisting of bitumen coated aggregate, was then fully dried in a fan oven at 60 °C and allowed to cool to room temperature.

Bitumen content analysis

The bitumen content of the coated sample was determined by xylene extraction. A sample was accurately weighed into a pre-weighed paper thimble, 100 mm deep by 25 mm in diameter. The bitumen was then extracted using soxhlet apparatus with xylene as solvent. This apparatus allows the sample to be repeatedly rinsed with hot solvent which extracts all of the bitumen from the aggregate. The stripped aggregate was fully dried in an oven at 150°C and then reweighed after cooling. The bitumen content was found by subtracting the weight of the stripped aggregate from the coated.

10.2.2 Description of test parameters and results

Base bitumen pen

Emulsions with four different nominal penetration grade base bitumens were tested as described above. The emulsion formulations were as shown in Table 10.1.

Table 10.1 : Emulsion formulations with bitumens of various pen grades

Component	Proportion
Venezuelan 50 pen bitumen	62 %
100 pen	
200 pen	
300 pen	
Emulsifier (Redicote E-4868)	1.2 %
conc. HCl	to pH 2.5 on waterphase
Water	to 100.%

Samples were compacted using two levels of static load - 8 and 16 MPa - applied for 3 periods of 30 s each. A compaction level of 16 MPa (roughly equivalent to 20 tons on 5 " diameter RAM) was suggested by Dybalski (136). It appears to be very intensive, however, it was said to be necessary to produce specimens in the laboratory with void contents similar to those achieved in the field. As these loads had been used previously in the production of specimens for mechanical properties tests, they were used in these in this instance also in order to be consistent. Preliminary tests indicated that loads of this order would be effective in causing measurable amounts of coalescence in the mixtures.

Table 10.2 and Figure 10.1 show the effect which the penetration grade of the base bitumen in the emulsion had on coalescence during compaction.

Table 10.2 : Effect of base bitumen pen on coalescence

Bitumen pen	50		100		200		300	
Compaction load (MPa)	8	16	8	16	8	16	8	16
Coalescence (%)	12.2	30.7	27.6	51.1	42.0	55.6	68.0	70.9

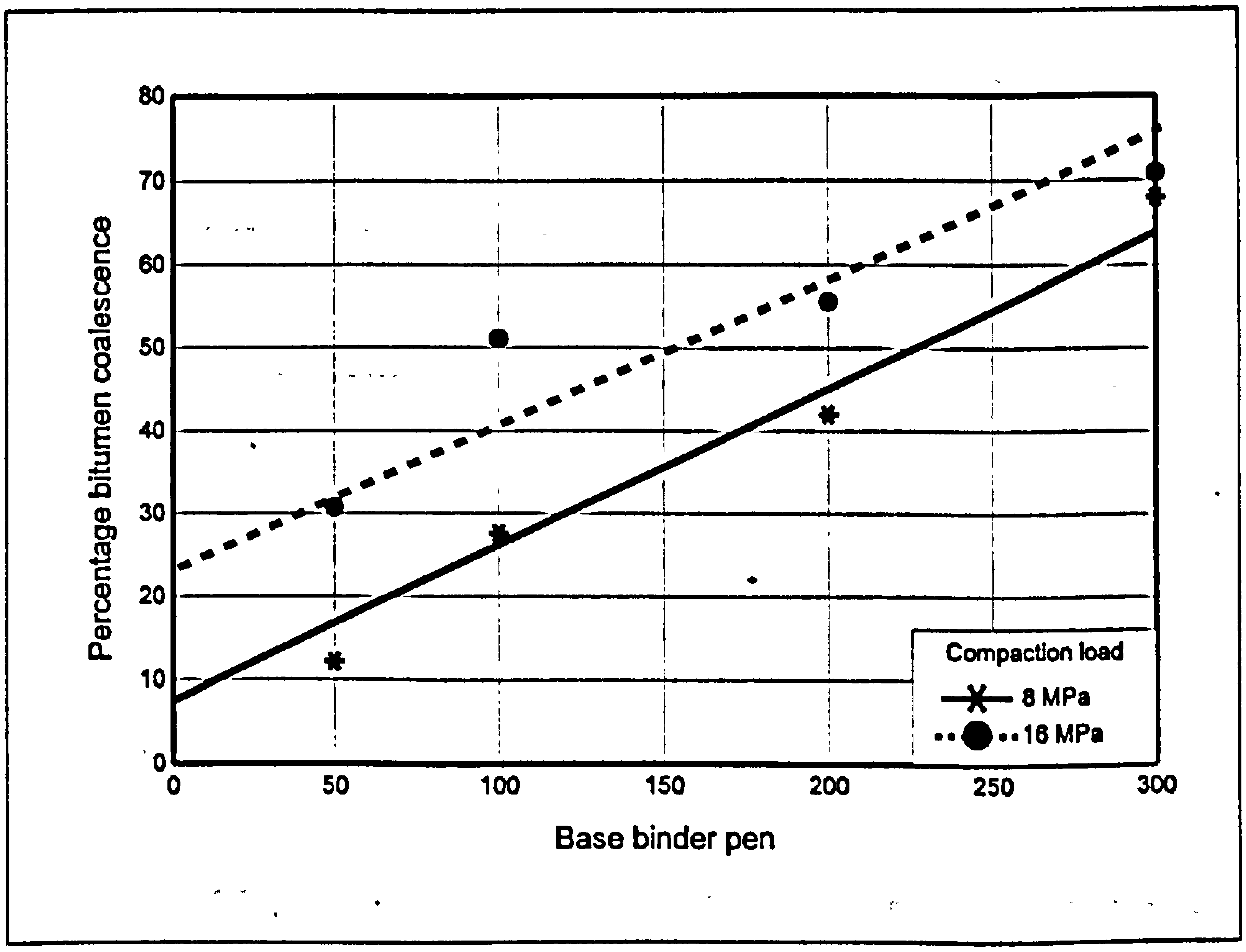


Figure 10.1 : Effect of base bitumen pen on coalescence

Coalescence increased with increasing binder penetration. It is postulated that this is due to softer bitumen droplets deforming more easily under compaction. Droplet deformation may have the effect of bringing globules into intimate contact thus leading to coalescence.

Emulsifier level

Emulsions with five different levels of emulsifier were tested as described above. The emulsion formulations were as shown in Table 10.3.

Table 10.3 : Emulsion formulations with various emulsifier levels

Component	Proportion
Venezuelan 100 pen bitumen	62 %
Emulsifier (Redicote E-4868)	0.8 %
	1.2 %
	1.6 %
	2.5 %
	5 %
conc. HCl	to pH 2.5 on waterphase
Water	to 100 %

Two sets of samples were prepared. One set was not compacted whilst the other was compacted using a 16 MPa static load applied for 3 periods of 30 s. The results are shown in Table 10-4 and Figure 10-2.

Table 10.4 : Effect of emulsifier level on coalescence

Emulsifier level (%)	no compaction	0.8	1.2	1.6		
Coalescence (%)		12.8	10.9	10.3		
Emulsifier level (%)	16 MPa compaction	0.8	1.2	1.6	2.5	5.0
Coalescence (%)		45.1	41.7	29	20.6	13.9

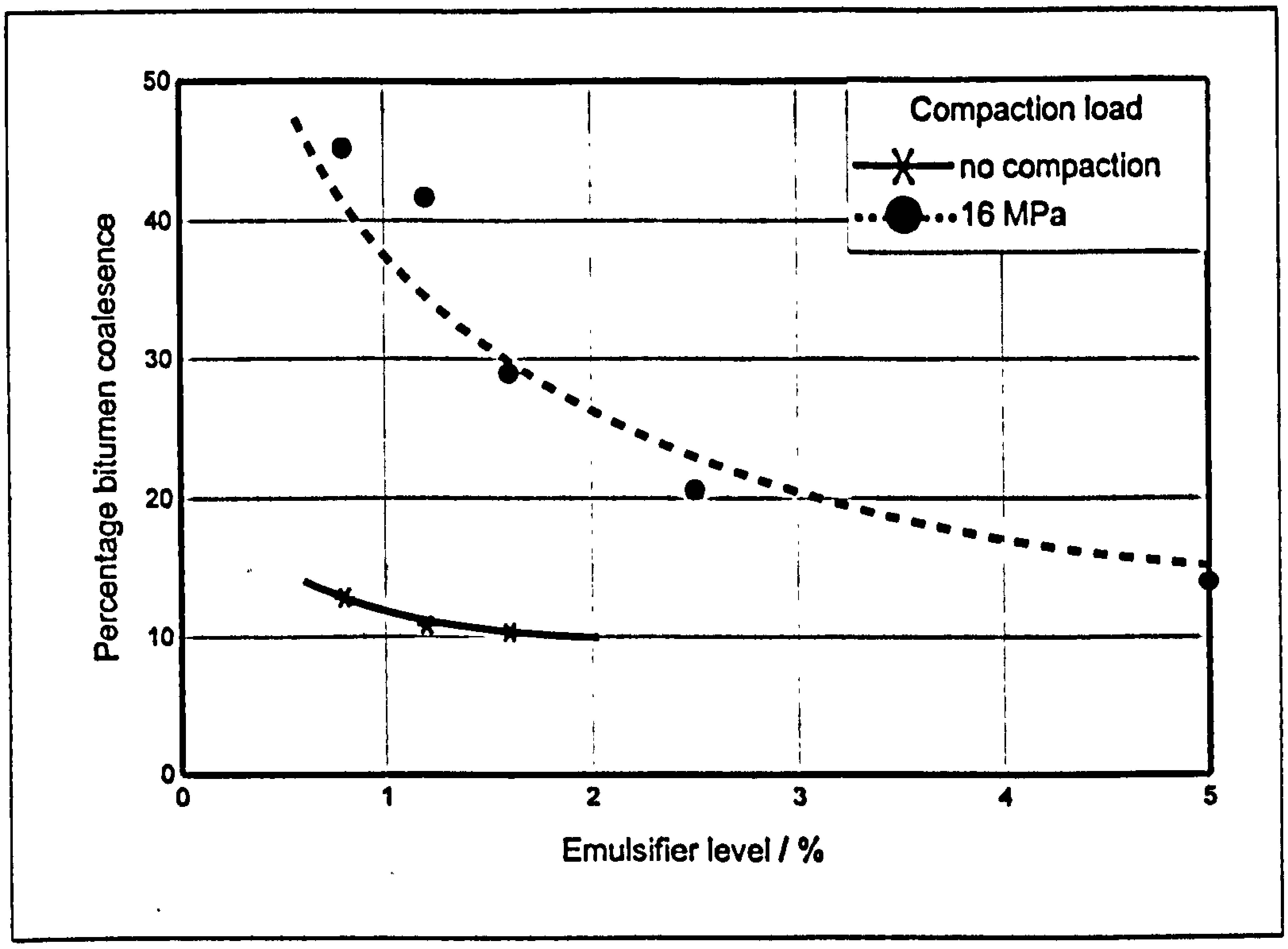


Figure 10-2 : Effect of emulsifier level on coalescence

Coalescence decreased with increasing emulsifier level under compaction, but was virtually unaffected if no compaction took place. There are two possible causes for this. In reality, probably both processes are at work.

The first possibility is related to the repulsive force between bitumen droplets in an emulsion. The concentration of emulsifier on the surface of bitumen droplets is related to the concentration of emulsifier in the total emulsion. Coalescence is a two stage process. Firstly the droplets come together and flocculate. At this point there is still a thin film of continuous phase between the droplets. This film then ruptures to allow droplets to flow together or coalesce (137). The larger the surface charge on the droplets or the physical barrier provided by the emulsifier molecules, the greater the resistance to flocculation and film rupture.

The second explanation is based on doping of the aggregate surface.

Although a large amount of emulsifier in an emulsion resides at the bitumen water interface, a certain amount is dispersed in the water phase. As emulsifier molecules are partially hydrophobic, they are most likely to exist as colloids. Colloids are aggregates of molecules which form in order to minimize the contact area between hydrophobic parts of surfactant molecules and water. They are often spherical in shape with the hydrophobic tails hidden on the inside surrounded by a surface of hydrophilic head groups. Other configurations are possible, depending upon the surfactant concentration, but all serve the same purpose. When an emulsion comes into contact with a surface, such as aggregate, this dispersed or free emulsifier will be attracted to charged sites. As discussed in chapter 2, emulsions can be broken through the attraction of surfactant molecules at the bitumen water interface to charged sites on an aggregate surface. If these sites are used up by free emulsifier, the emulsion will not break as quickly. Therefore, the higher the level of emulsifier, or more exactly free emulsifier, the slower the break of the emulsion.

Compaction load

Mixtures were prepared with the standard emulsion formulation (Table 6-2) and compacted using static loads of 4, 8, 16, 24 and 32 MPa applied for 3 x 30 s. The results are shown in Table 10.5 and Figure 10.3.

Table 10.5 : Effect of compaction load on coalescence

Compaction load / MPa	4	8	16	24	32
Coalescence / %	13.99	21.43	38.11	63.06	78.81

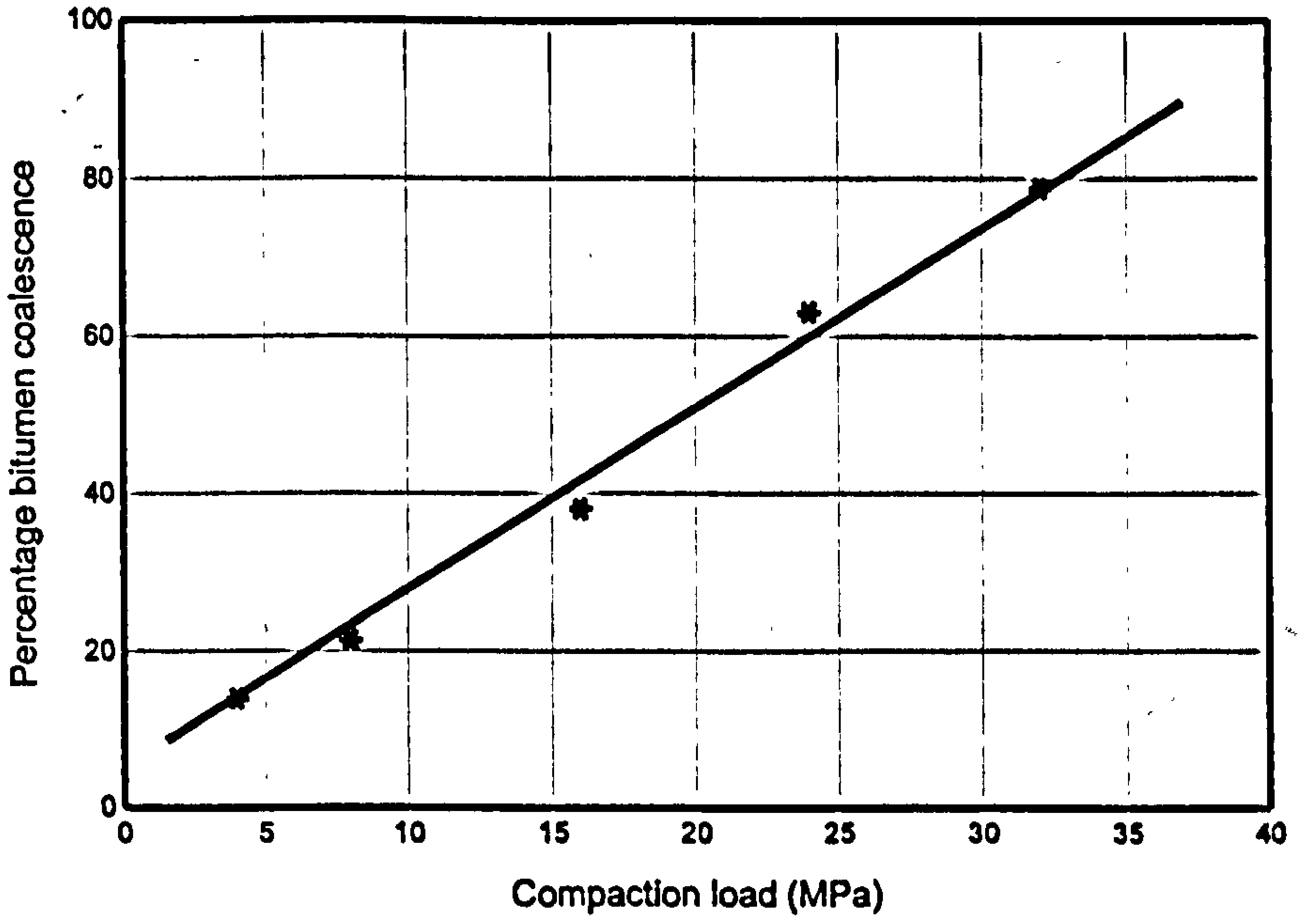


Figure 10-3 : Effect of compaction load on coalescence

Coalescence increased with compaction load. Before these tests were carried out it was not known whether or not emulsion break was sensitive to pressure. It seems likely that there is an electrostatic or steric boundary between droplets which must be overcome in order for the droplets to come together and join. The higher the compaction pressure the larger the energy input to force globules together.

Number of blows of Marshall hammer

Mixtures were prepared with the standard emulsion formulation (Table 6-2). One series was prepared without ordinary portland cement (OPC) and another with 1% OPC added to the dry aggregate mixture prior to water and emulsion. OPC has, on numerous occasions, been found to improve the setting rate of bitumen emulsion mixtures and this topic has been discussed in detail in chapter 8. Both sets of mixtures were compacted with a range of numbers of blows from the Marshall hammer. The results are shown in Table 10-6 and Figure 10-4.

Table 10.6 : Effect of number of blows of Marshall Hammer

Number of blows		0	20	50	100	200	400
Coalescence / %	no OPC	3.7	5.6	5.8	7.2	10.9	25.1
Coalescence / %	1% OPC	5.0	6.83	8.4	11.	15.9	29.7

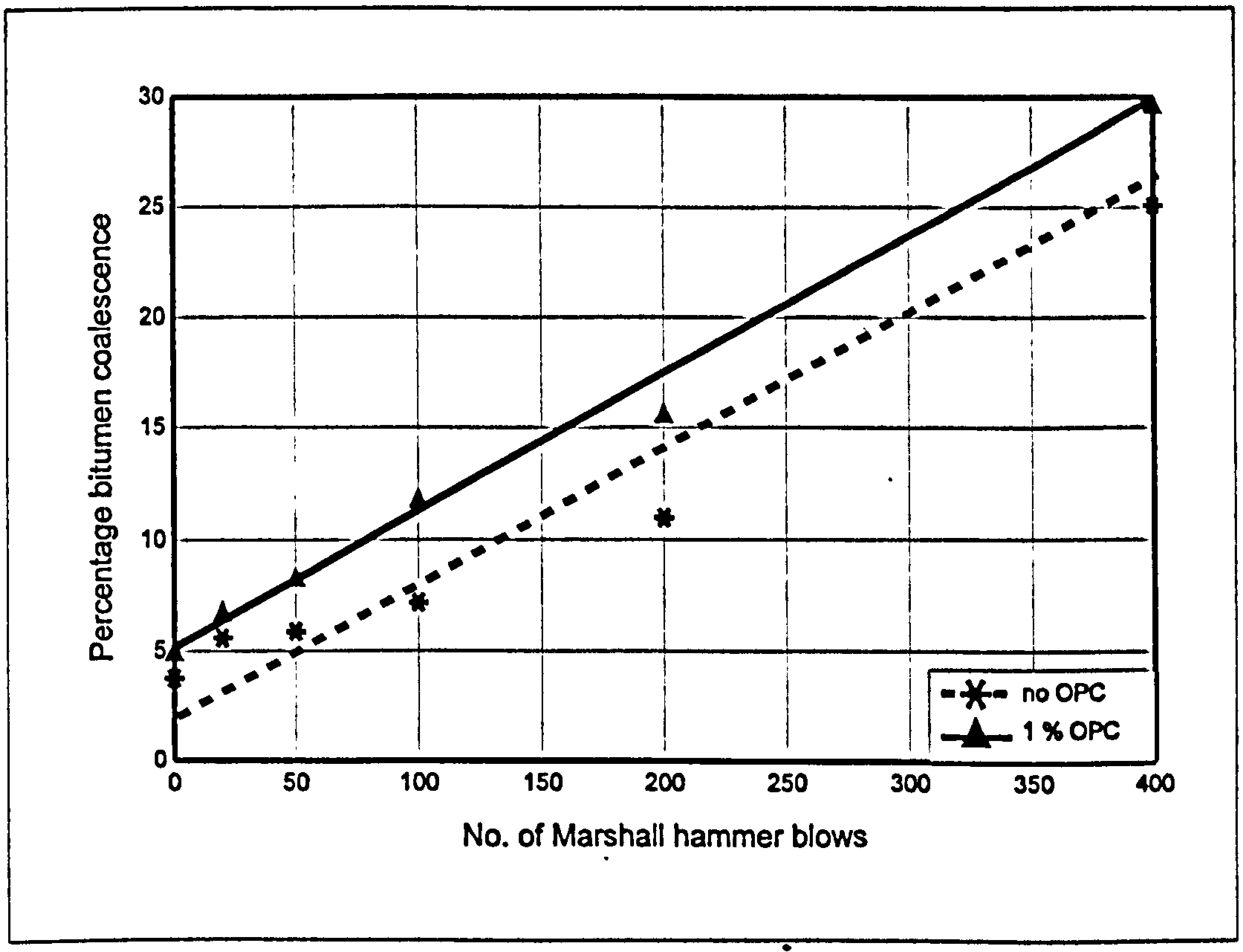


Figure 10-4 : Effect of Marshall hammer blows on coalescence

Coalescence increased with number of Marshall hammer blows. This is again a function of the amount of compactive effort. Though the load does not change, as a core becomes more compact, the internal pressure on the emulsion must increase.

Compaction load - time sequence

In addition to the magnitude of load which can be applied to compact a paving material, the compaction frequency is variable over a certain range. The following tests were undertaken in order to investigate the possibility of inducing higher levels of emulsion break as a result of the frequency of vibration of compaction load.

Mixtures were prepared with the standard emulsion formulation (Table 6-2). Compaction was carried out using a Mand servo hydraulic press, capable of holding a constant load or vibrating at any desired frequency with a square or sinusoidal waveform. In this set of tests, two magnitudes of load were applied; 4 and 8 tonnes. The total load x time was kept constant and so the 4 tonne load was held for a total time period of 180 s and the 8 tonne load for only 90 s. The loads were then applied in vibration mode using a square waveform which resulted in the load being either on at its maximum or zero. This was a rather artificial scenario compared to compaction regimes experienced in real life but of interest for the purposes of the test. The vibration frequencies were increased up to a maximum of 30 Hz with the total application time of the load always the same as those described above. This resulted in samples being subjected to loads for the same total periods of time in smaller and smaller increments.

Table 10-7 and Figure 10-5 show the effect which the load and duration sequence have on coalescence when applied as a square waveform.

Table 10-7 : Effect of compaction load - time sequence

Compaction sequence	1 x 180 s		180 x 1 s		720 x 0.25 s		3600 x 0.05 s	
Load / tonnes	4	8	4	8	4	8	4	8
Coalescence / %	11.3	16.1	18.9	18.3	18.3	23.8	20.7	29.6

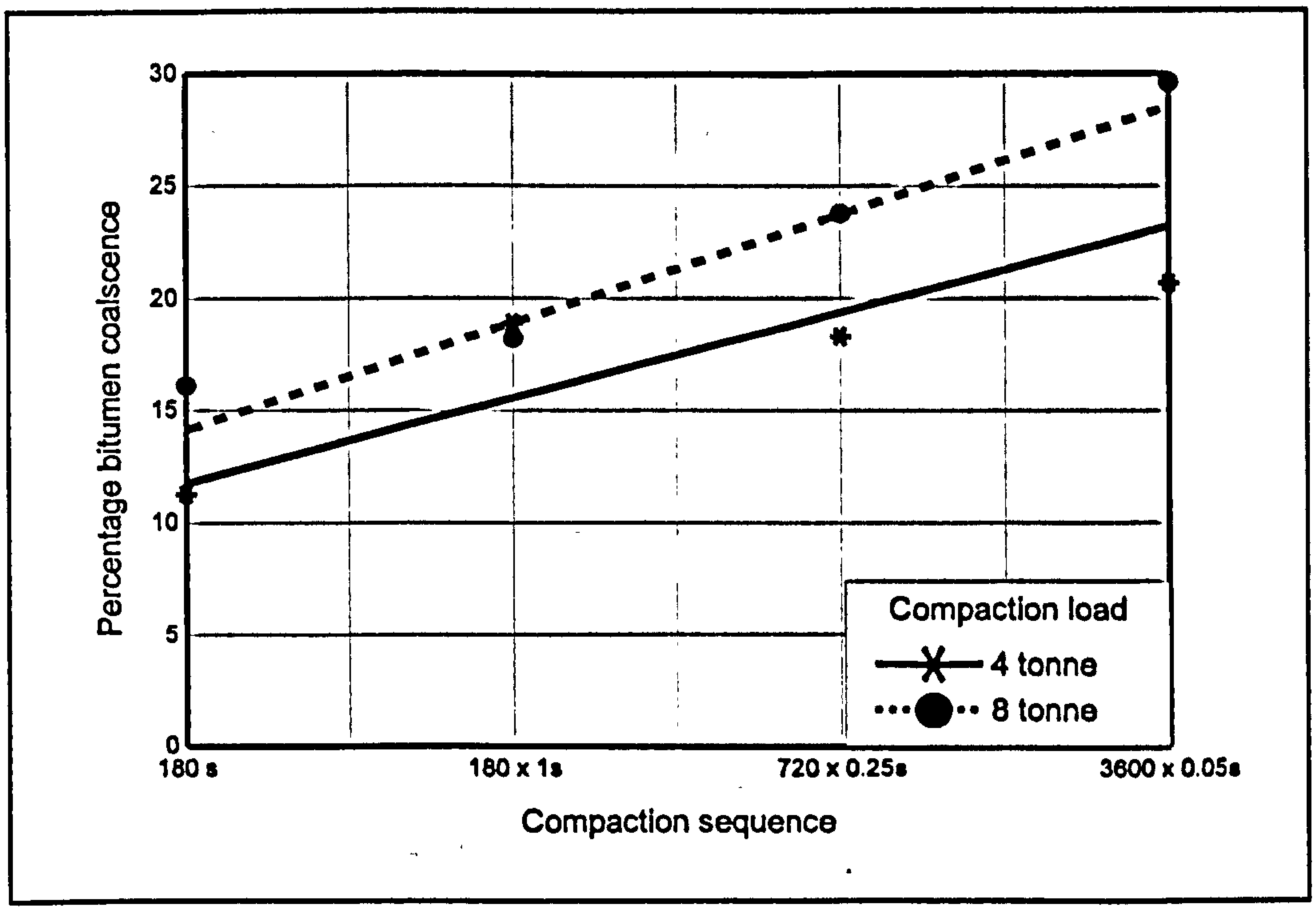


Figure 10-5 : Effect of compaction load - time sequence

From these results it can be said that loads applied in many small time increments give rise to more coalescence than fewer applications for longer periods. This can be stated more succinctly as coalescence increased with compaction frequency. Again, an increase in coalescence due to increased load can also be seen.

Compaction frequency

Mixtures were prepared with the standard emulsion formulation (Table 6-2). Compaction was similar to that described above except that this time a sinusoidal waveform was used which oscillated between 6 and 8 tonnes. The intention was to simulate a vibratory compactor which would exhibit compaction modes similar to this. The similarities were in the rounded wave form and the dead weight load of 6 tonnes at minimum loading. The load was again applied at a range of frequencies.

Table 10-8 and Figure 10-6 show the effect of compaction frequency on coalescence.

Table 10-8 : Effect of compaction frequency

Compaction frequency / Hz	5	10	20	30
Coalescence / %	14.4	16.1	16.7	17.4

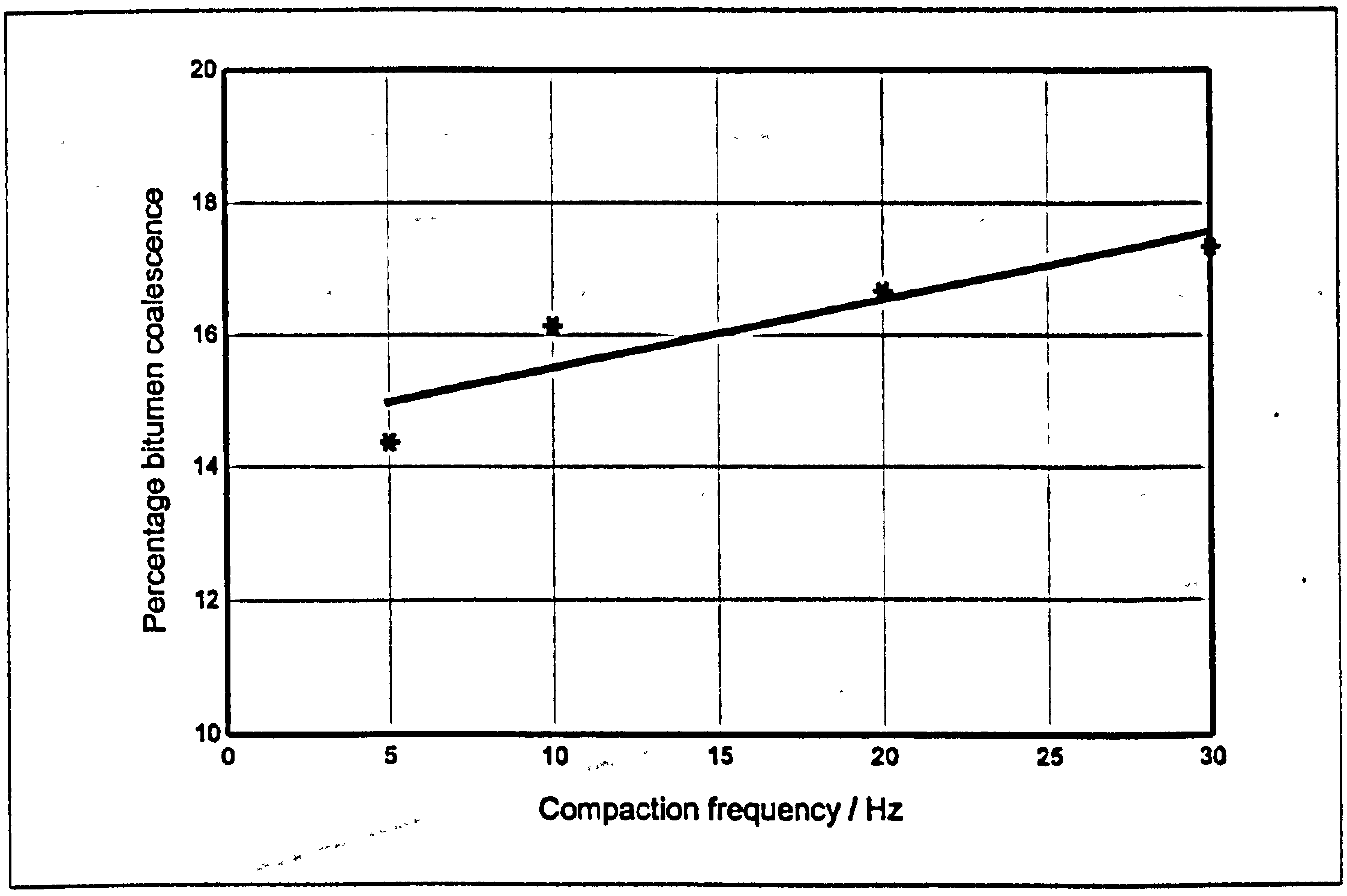


Figure 10-6 : Effect of compaction frequency on coalescence

Using a load oscillating between 6 and 8 tonnes with a sinusoidal waveform, the amount of coalescence did not increase significantly with increasing frequency. This is in contradiction with the square waveform compaction. One possible explanation could be that the former gives rise to a greater internal pressure, due to the load oscillating between 0 and 4 or 8 tonnes which allows for greater movement of particles, and therefore more densification of the mix. Alternatively, the frequency range covered in the test may not have been large enough to have a significant effect. It was not possible to use higher frequencies at these load magnitudes as the Mand press could not achieve them.

Ordinary Portland Cement

Mixtures were prepared with different levels of OPC added to the aggregate, prior to pre-wet water and standard bitumen emulsion (Table 6-2), and compacted using a static load of 16 MPa applied for 3 x 30 s. The results are shown in Table 10.9 and Figure 10.7.

Table 10.9 : Effect of cement addition level

Cement level (%)	0	1	2	3	4
Coalescence (%)	36.4	59.2	82.9	71.6	80.4

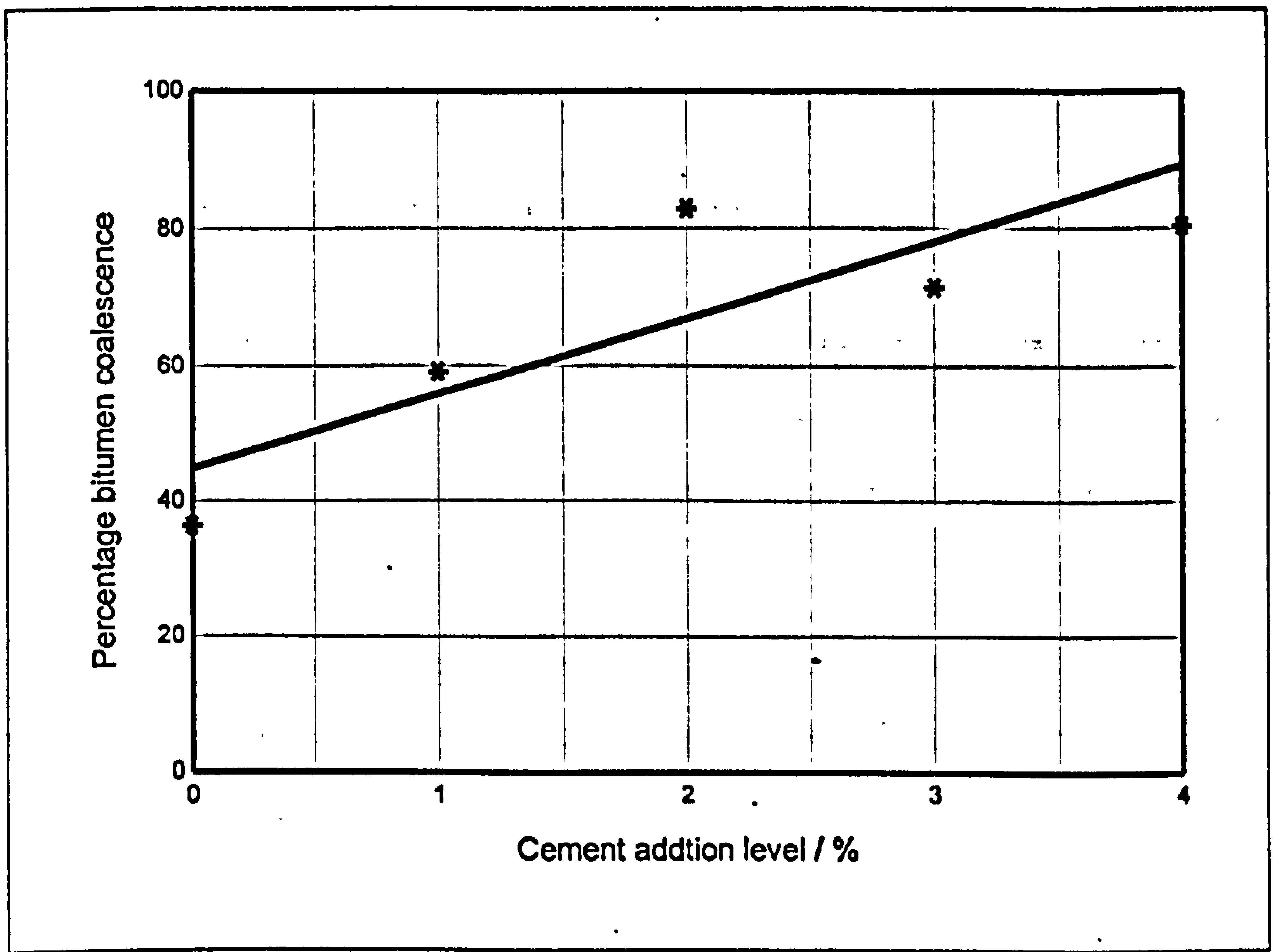


Figure 10-7 : Effect of OPC addition level on coalescence

The addition of ordinary portland cement to the mixture appeared to increase the amount of coalescence (even though the results showed a high degree of scatter which was probably due to loss of OPC during the rinsing or extraction processes). There are several possible mechanisms behind this effect and tests described elsewhere in this report went some way towards explaining them.

Rapid setting cements

Cements are available in a wide range of grades according to setting rate, heat of hydration, capacity for air entrainment and other parameters. Two rapid setting cements, namely Aquablend and Astrablend, were obtained from the company Pozzament. Aquablend was claimed to be the least rapid setting of the two, and capable of absorbing 1.7 times its own weight in water. Astrablend was more rapid setting and able to absorb 2.4 times its own weight in water. The cements were added to the dry aggregate mixture, followed by pre-wet water and standard bitumen emulsion (Table 6-2) and thoroughly mixed. Mixtures were compacted using a static load of 16 MPa applied for 3 x 30 s.

Table 10-10 and Figure 10-8 show the effect which rapid setting cements had on coalescence compared to ordinary portland cement.

Table 6-10 : Effect of rapid setting cements on coalescence

Cement type (1% on agg)	None	OPC	Aquablend	Astrablend
Coalescence (%)	34.2	32.7	21.3	74.1

Supposedly rapid setting cements did not automatically lead to more coalescence than ordinary portland cement. Although Astrablend did increase coalescence, Aquablend did not.

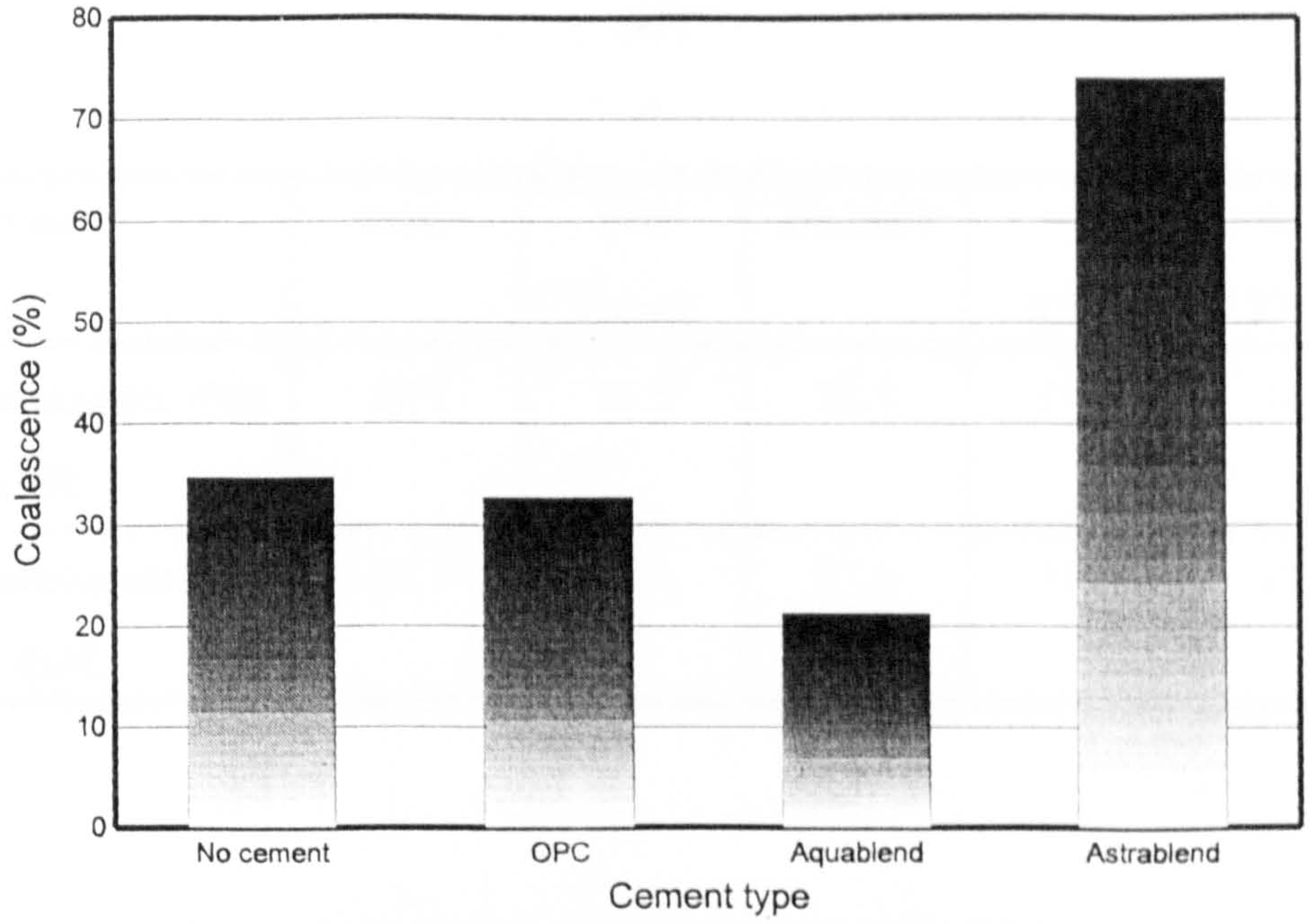


Figure 10-8 : Effect of rapid setting cements on coalescence

Polymer modification of emulsion

It is well established that polymer modification of bitumen, whether it is used hot or as an emulsion, has an effect upon the properties of the binder (5, 138). Polymer types have been discussed in detail in chapter 9. This should manifest itself in the mechanical properties of the cured mixture, but it was also postulated that polymers may affect the curing rate by accelerating or slowing down the coalescence process. A selection of polymer modified emulsions described above (Table 6-2) was screened in the coalescence test with and without OPC. OPC was added to the dry aggregate, if required, followed by pre-wet water and bitumen emulsion and thoroughly mixed. Mixtures were compacted using a static load of 16 MPa applied for 3 x 30 s.

Table 10-11 and Figure 10-9 show the effect which polymer additives have upon coalescence both in the presence and absence of OPC.

Table 10-11 : Effect of polymer additives on coalescence with and without OPC

Polymer	none	3% Latex	3% SBS	3% EVA	5% EVA
Coalescence (%) no OPC	34.7	31.8	36.3	17.2	14.7
Coalescence (%) 1% OPC	32.7	61.5	57.9	17.8	17.8

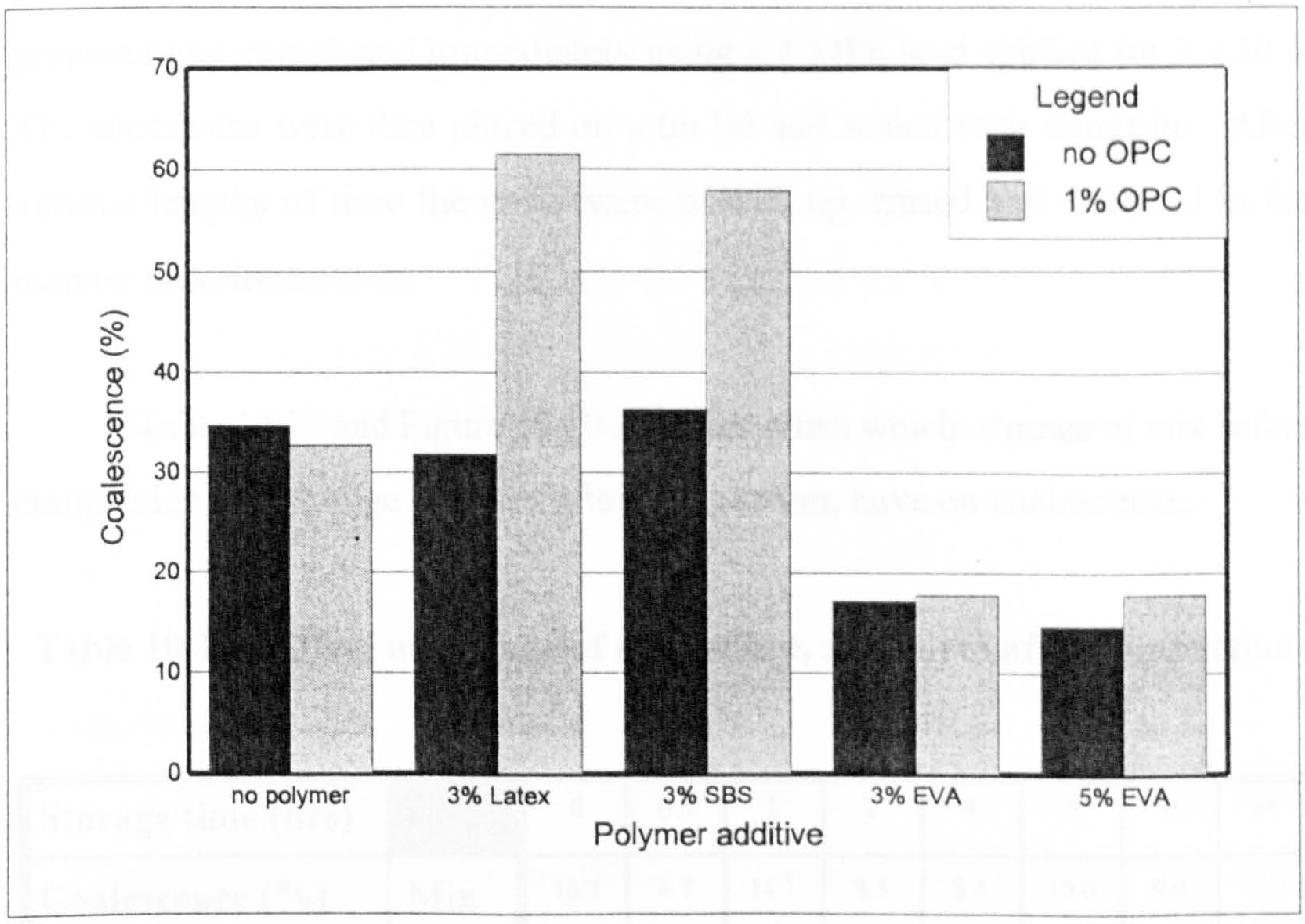


Figure 10-9 : Effect of polymer additives with and without OPC on coalescence

Polymer additives had a significant effect on coalescence. Latex and SBS led to a large increase in coalescence but only when OPC was included in the mixture, whereas EVA caused a decrease, irrespective of the use of OPC. It seems likely that chemical as well as physical processes are at work in these systems but is not possible to say what these might be as a result of these studies.

Storage time of mixture and curing time

In some potential areas for the application of emulsion mixtures, there is a requirement for storage stability for at least one day and preferably for 5 days or more after manufacture. During this time the mix must remain workable which means that no large amount of emulsion break should occur. Standard emulsions mixtures were prepared and stored in sealed containers for differing lengths of time, before compaction which comprised of an 8 MPa static load applied for 3 x 30 s.

Although the primary objective of these tests was to examine the degree of coalescence during compaction, some indication of curing in the 24 hour period after compaction was also of interest. Standard emulsion mixtures were prepared and compacted immediately using a 8 MPa load applied for 3 x 30 s. The specimens were then placed on a tin lid and sealed with clingfilm. After various lengths of time the cores were broken up, rinsed and analysed in the manner described above.

Table 10.12 and Figure 10.10 show the effect which storage of mix before compaction and storage of cores after compaction, have on coalescence.

Table 10-12 - Effect of storage of mix before, and cores after compaction

Storage time (hrs)		0	0.5	1	2	4	6	16	24
Coalescence (%)	Mix	10.4	8.7	11.1	8.5	8.4	10.0	9.0	10.1
Coalescence (%)	Cores	11.0	15.8	10.0	16.1	15.6	17.4	17.0	23.1

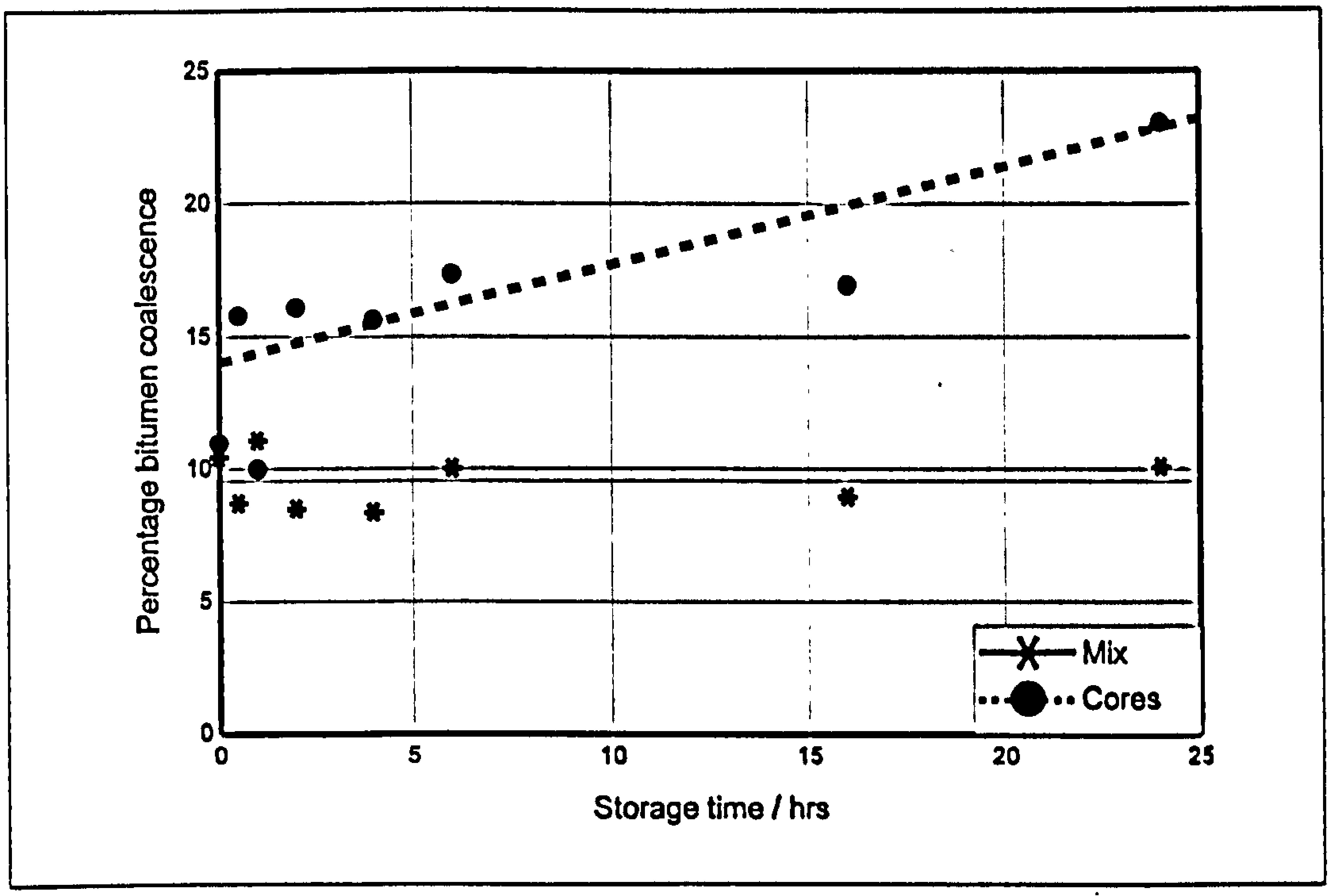


Figure 10-10 : Effect of storage time of mix before, and specimens after compaction on coalescence

No significant amount of coalescence appeared to arise in the uncompacted mixture over a 24 hour period. After compaction, the emulsion steadily coalesces over a 24 hour period. This suggests that compaction was necessary to initiate coalescence, which then proceeds with age. The absence of full coalescence without compaction is desirable as it gives rise to workability and storage stability of a mix.

Temperature

It is well known from the experiences in the field in these studies, some of which have been described in chapter 7, that cold mix cures less well under cold and wet conditions. These parameters will obviously affect the rate of water loss from a mixture, but they may also play a part in the breaking process.

Stewart and Fitzgerald investigated the effects of cold temperature mixing of emulsions (139). They considered the 'normal' lab methods involving mixing and compaction at ambient temperatures and Marshall testing at 60°C to be unrealistic. In certain areas of North America mixing and laying in the field is carried out under much lower temperature conditions. Their work attempted to use a lab test protocol similar to field conditions. Mix preparation, compaction and testing were all carried out at 8.5 °C as was storage and aeration of the mix prior to compaction. The physical properties and Marshall stability were determined finally, also at 8.5 °C.

Marshall stability was found to increase proportionally to the square root of curing time and also with aeration time of mix prior to compaction, with the optimum being 4 hrs. However, the voids content increased with increasing aeration time indicating a reduction in workability due to partial setting of the mix. The optimum bitumen content was 4-5%.

Low temperature curing had a significant reducing effect on the stability increase over time. As testing was carried out at 8.5 °C it is not possible to directly compare these results with those obtained at 60 °C. Aeration of the mix prior to compaction is very beneficial with cold mix, particularly for use at low temperatures. Further work was suggested and included testing at 0-5 °C, with a wider range of binder contents and efforts to reduce the rather high void contents obtained under low temperature compaction.

Therefore, in order to further investigate the effect of temperature purely on emulsion break, mixtures were manufactured at a range of temperatures from 3 to 45 °C and compacted using a static load of 16 MPa. The results of the tests are shown in Table 10-13 and Figure 10-11.

Table 10-13 : Effect of temperature on coalescence

Temperature (°C)	3	10	20	30	40	45
Coalescence (%)	37.09	43.37	41.38	46.64	50.12	52.69

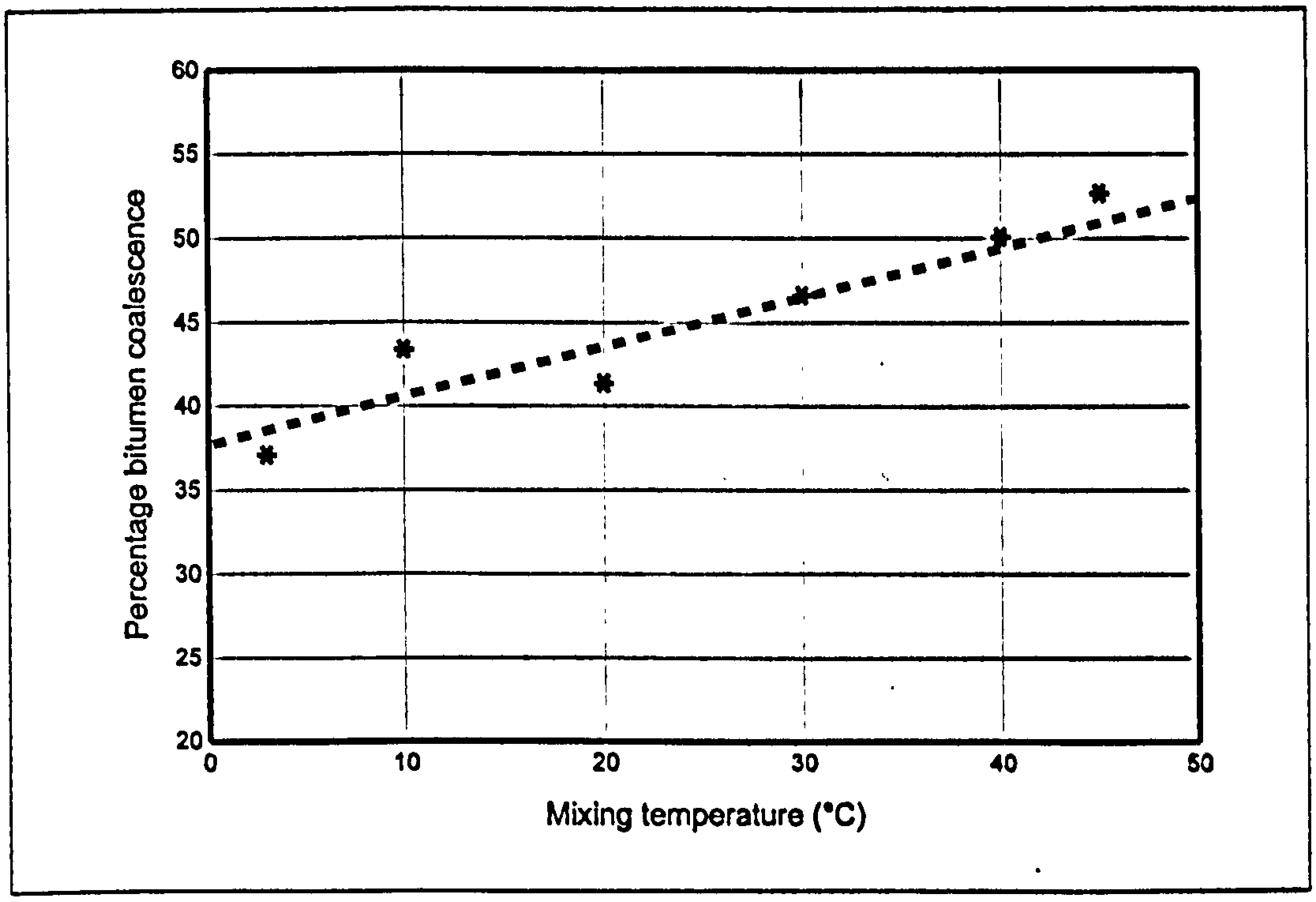


Figure 10-11 : Effect of temperature on coalescence

The results shown in Figure 10-11 indicate that coalescence under compaction increased with temperature. The increase was not enormous over the range covered in these tests and so temperature does not seem to be as significant as may have been suspected in the emulsion breaking process. The mechanism for this effect could be due to a number of factors :

- Softening of the binder leading to easier deformation and coalescence of droplets
- Increase in rate of chemical reaction between emulsifier and aggregate

Breaking agents

A breaking agent system, developed and patented by the Swedish company Nynas, is claimed to promote the breaking of emulsions soon after addition (140). It can be used in a range of applications - mainly surface dressing and cold mix. The agent is based on a double emulsion system which consists of an aqueous solution of a weak base emulsified in light mineral oil, which is itself emulsified in water (Figure 10-12). The idea is that the base is initially kept away from the bitumen emulsifier by the mineral oil. Shortly after mixing, the base migrates through the oil and begins to neutralise the emulsifier, thus breaking the bitumen emulsion.

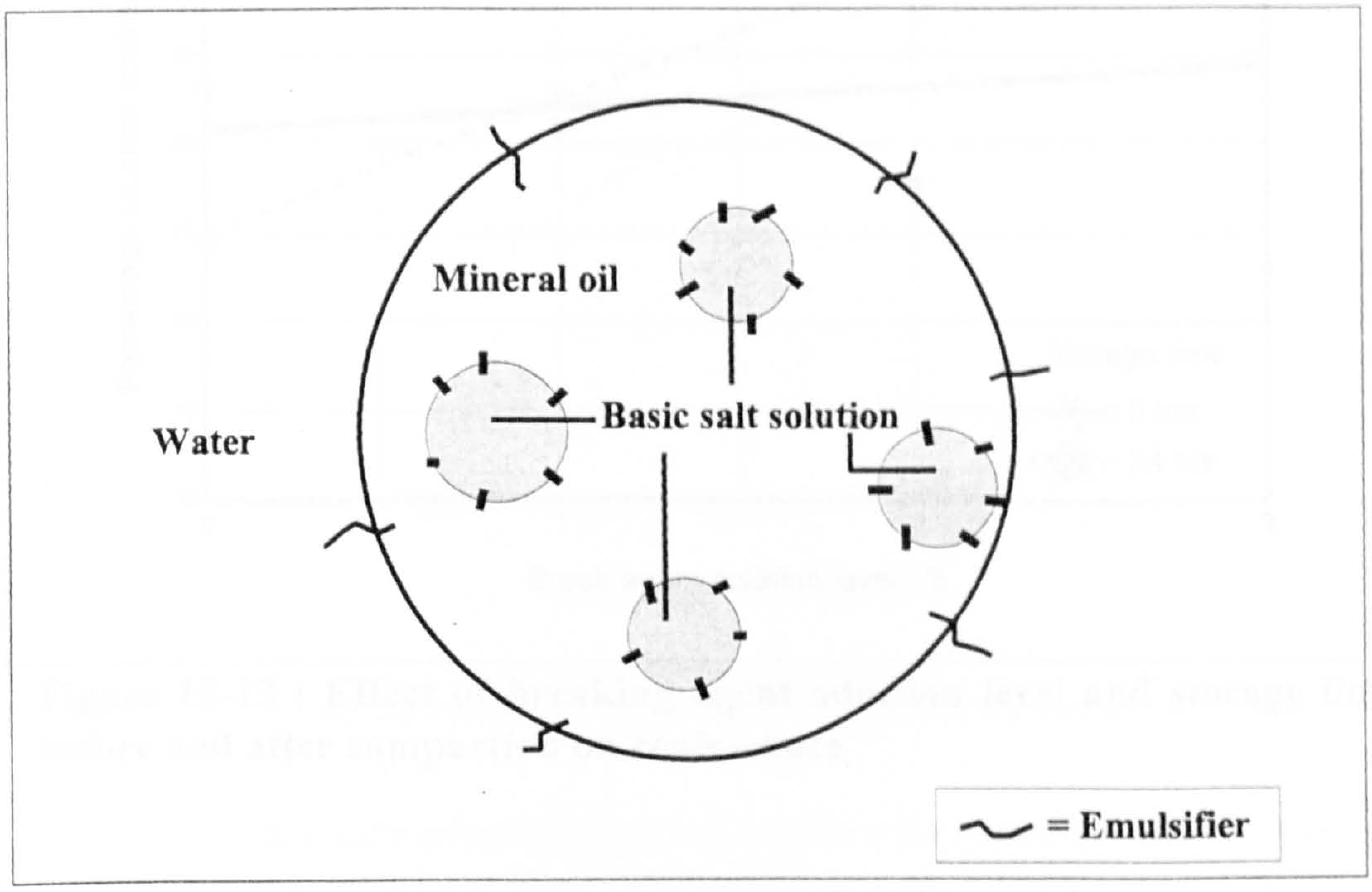


Figure 10-12 : Breaking agent double emulsion

Standard emulsion mixtures were prepared but with breaking agent added to the emulsion immediately before use. Tests were performed on mixtures rinsed immediately after compaction, with a 3 x 30 s, 16 MPa static load, and on others stored before rinsing. A third set of tests was carried out on mixtures compacted at a range of loads from 0 to 32 MPa.

Table 10-14 and Figure 10-13 show the effect which the addition of breaking agent to the emulsion, at a range of levels, has on coalescence.

Table 10.14 : Effect of breaking agent - addition level and storage time after compaction

Break agent level / %	Storage	0	1	2	3
Coalescence / %	0 hrs	33.1	32.8	27.7	39.1
Coalescence / %	24 hrs	25.5	30.7	43.2	46.5

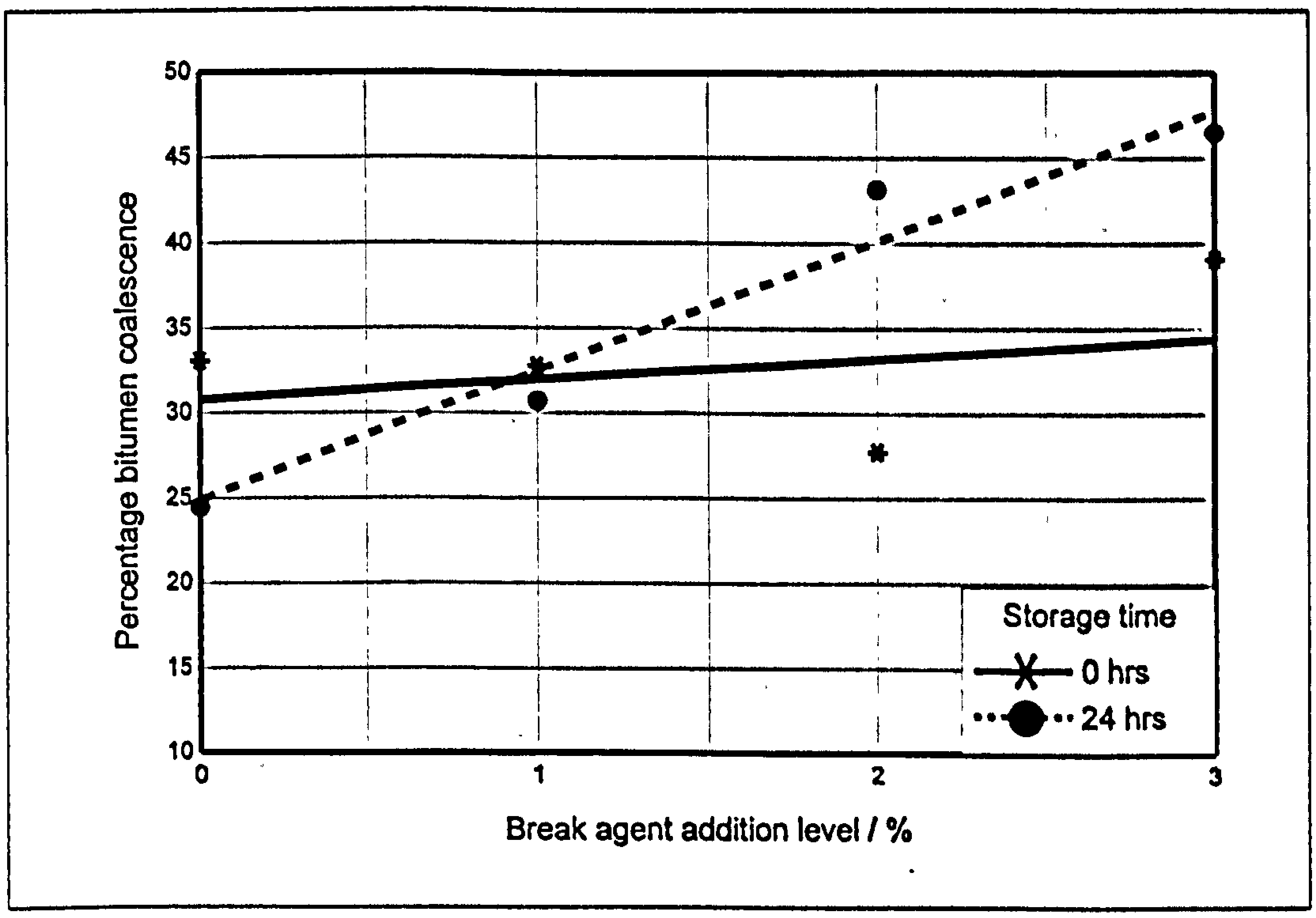


Figure 10-13 : Effect of breaking agent addition level and storage time before and after compaction on coalescence

Immediately after compaction, the breaking agent did not lead to an increase in coalescence. However, in mixtures stored for 24 hours before analysis, an increase in coalescence was observed with increasing breaking agent. This suggests that the time lag effect of the double emulsion system caused a longer delay in the breaking process than was intended.

Table 10-15 and Figure 10-14 show the effect of compaction load on the coalescence of emulsion with and without breaking agent.

Table 10-15 : Effect of breaking agent - compaction load

Compaction load (MPa)	0	4	8	16	24	32
Coalescence (%) no brk. agent		14	21	38	63	79
Coalescence (%) 1% brk. agent	17	25	28	28	54	65

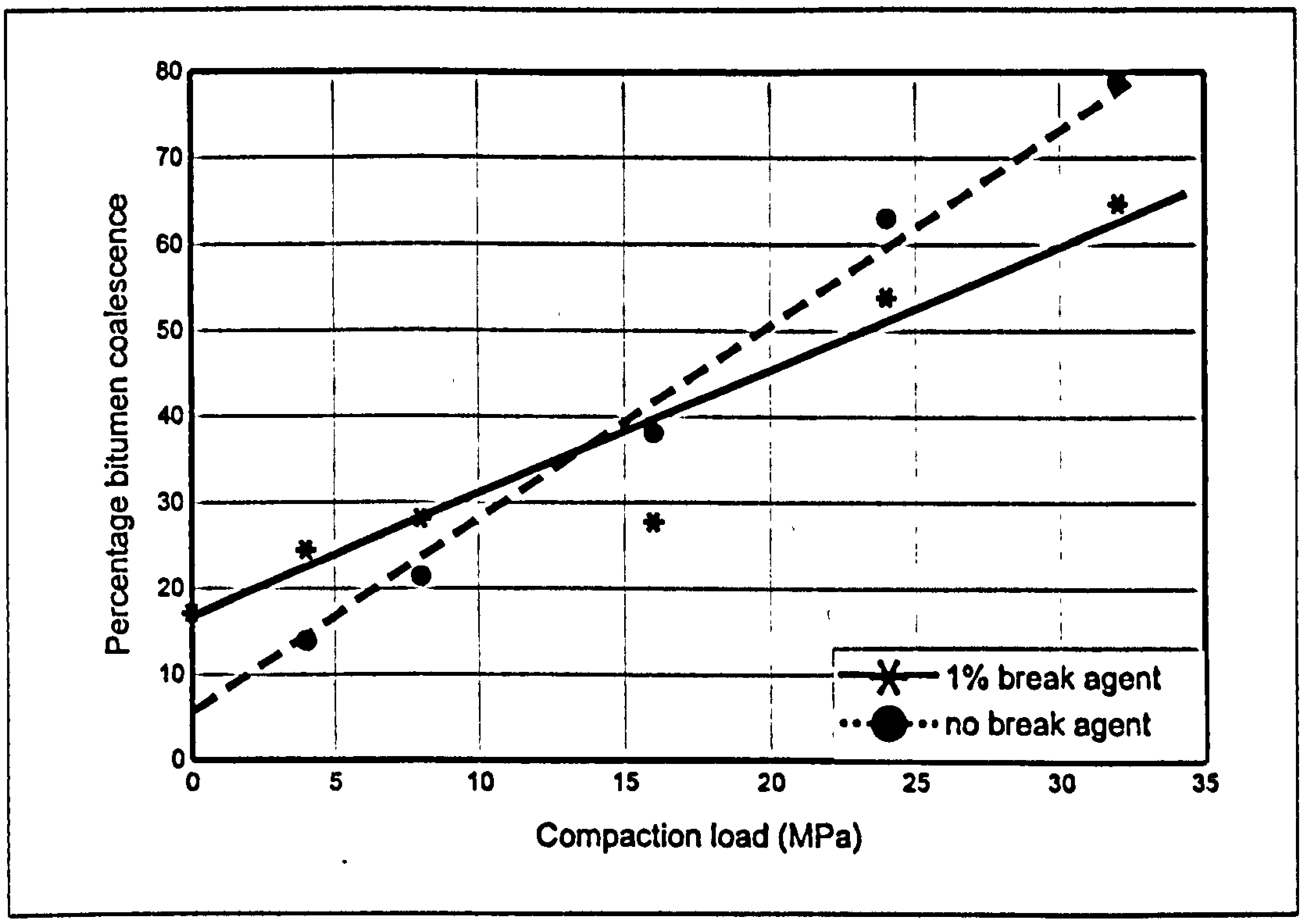


Figure 10-14 : Effect of breaking agent and compaction load on coalescence

The results shown in this graph indicate that the breaking agent system is not pressure sensitive. Increasing the load did not induce more emulsion break if breaking agent wa included.

10.3 COALESCENCE OF BITUMEN EMULSION IN SUSPENSION

The tests described above were designed to determine the amount of coalescence of bitumen emulsion occurring on the aggregate surface in a mixture. However, it is also possible for bitumen droplets to coalesce in suspension and not in contact with the aggregate at all. If this occurred in the tests reported above, the process would not be detected by the method employed. Freely suspended droplets, no matter how large, would be washed away during the mixture rinsing stage and not appear in the binder content analysis.

In order to measure the amount of coalescence occurring in suspension, a slightly modified version of the method described above was used. Instead of analysing the binder content of the aggregate, a particle size analysis was carried out on the rinsings. Any bitumen coalescence would appear as an increase in particle size compared with that of the original emulsion used.

10.3.1 Tests and results

Mixtures were prepared as described above with the standard emulsion (Table 6-2) and compacted using static loads of 8, 16 and 24 MPa. Compacted specimens were broken up and placed in a vessel with 2 % emulsifier solution and rolled for 20 minutes. Samples were then immediately taken from the supernatent before any settlement of bitumen droplets could occur. The particle size distribution was then analysed using an Elzone particle sizer.

Particle size distribution analyses were also carried out on the supernatants from the following two tests as controls :

1) A sample of the sieved and washed aggregate and emulsion added to a portion of 2 % emulsifier solution without being mixed together.
2) A mixture of emulsion and aggregate added to a portion of the 2 % emulsifier solution without compaction.

Particle size analysis revealed that there was no discernable difference between any of the samples tested. The median particle sizes were approximately 5 μm in all cases.

10.4 DISCUSSION

The results acquired in the above tests were not very reproducible. For example, using 3 x 30 s, 16 MPa static load compaction and an emulsion with 1.2% emulsifier and 100 pen bitumen, results of 51.15, 41.69, 38.11,36.41 and 33.09 percent bitumen coalescence were obtained in various tests. This is a wide spread of results but, considering the crude nature of the test method, particularly in terms of the rinsing stage, it is probably to be expected. However, it was felt that the results were sufficiently accurate, at least within a set of tests, to allow trends to be seen and conclusions to be drawn.

10.5 SUMMARY

The results of each test in this chapter have already been discussed above but the paragraph and table below draw this information together.

Increasing bitumen penetration grade increased coalescence and this was thought to be due to droplets of softer binders flowing and joining together more easily. Increasing emulsifier level decreased coalescence as might be expected as more emulsifier should stabilize the emulsion and dope the charge sites on the aggregate surface. Compactive effort was shown to be a factor in that an increase led to an increase in coalescence. Compaction frequency was also found to influence coalescence but this was complicated by a dependency on compaction regime. The addition of OPC had a marked effect and the greater the addition level the greater the amount of coalescence. A rapid setting cement was found to have a greater effect on coalescence. SBS, EVA and Latex polymers all had some influence - SBS and Latex led to and increase in coalescence whereas EVA had the opposite effect. Coalescence was found to increase with time after compaction but time had little influence before compaction took place. It would, therefore, appear that compaction was needed to initiate the breaking process. An increase in temperature caused an increase in coalescence and this could have been due to an effect on the rate of reaction or softening of the binder. A patented breaking agent was shown to have little effect immediately after compaction but led to an increase in coalescence if the mixture was stored for 24 hours before analysis. The effect of the breaking agent was unaffected by compaction load.

The conclusions which can be drawn from the results obtained in the tests reported above are further condensed in Table 10-16.

Table 10-16 : Summary of coalescence test results

Parameter	Details	Effect on coalescence (+, -, =)
Increase bitumen penetration grade	50 to 300 pen	++
Increase emulsifier level	0.8 to 1.6 % Redicote E-4868	-
Increase compaction load	4 to 32 MPa	++
Increase number of Marshall hammer blows	0 to 400	+
Use greater number of shorter compaction load pulses	1 x 180 s to 3600 X 0.05 s	+
Increase compaction vibration frequency	5 to 30 Hz	=
OPC	0 to 4 %	++
Polymer additives in emulsion	3 % SBS or Latex with OPC 3 or 5 % EVA with or without OPC	++ -
Time	0 to 24 hrs before compaction 0 to 24 hrs after compaction	= +
Temperature	3 to 45 °C	+
Breaking agent	Immediately after compaction After 24 hrs storage	= +
Breaking agent and compaction load	Increase compaction load	=

CHAPTER 11

ZETA POTENTIAL MEASUREMENTS

11.1 INTRODUCTION

The breaking and curing behaviour of cold mix is dependent upon a number of factors. Temperature and humidity conditions affect the rate of water evaporation and also the rate of chemical reaction. Porosity of the aggregate has an effect on the rate of emulsion water loss through absorption. A further significant parameter is the relative surface charges of aggregate particles and bitumen emulsion droplets. Dybalski showed that the rate of setting of bitumen emulsions in aggregate mixtures is related to the charge difference so that highly (cationically) charged emulsion droplets lead to faster setting emulsions (141).

Most aggregates, including those used in emulsion aggregate mixtures, are negatively charged under aqueous conditions (142, 143). In an application such as surface dressing, therefore, highly charged cationic emulsions are used which ensure that there is a large attractive force between aggregate and emulsion which causes rapid break to occur. In the case of cold mix, however, a very low degree of interaction is required to allow mixing without premature break of the emulsion. Nevertheless, it is beneficial for there to be some attraction between bitumen droplets and aggregate in order to promote adhesion during mixing process and during and after curing. In emulsion mixtures, the aggregate will always be water wet and, therefore, a physio chemical barrier must be overcome in order to allow the hydrophobic binder to adhere. The emulsifier may help in this situation by coating the aggregate surface and thus providing a hydrophobic surface to which the bitumen can adhere (144).

The emulsifier used in these studies, namely Redicote E-4868 exhibits the desirable characteristics of mixability and good coating of the aggregate.

However, in terms of curing rate and water susceptibility, the performance of Redicote E-4868 emulsions is not particularly good unless OPC is added to the mixture as discussed in chapter 8.

A range of emulsions were prepared for the investigations described in this report with different emulsifier levels, bitumen penetration grades and polymer additives as detailed in chapter 6 of this report. The surface charges of these emulsions were determined through zeta potential measurements (see below) in order to further characterise them in terms of their electrochemical properties.

The addition of ordinary portland cement is known to improve the performance of some types of cold mix as has been discussed in detail in chapter 8. Measurements were made on Redicote E-4868 emulsions in the presence of OPC and other additives, in an attempt to understand the effect of these additive.

11.2 ZETA POTENTIAL

Charged species, such as an emulsified bitumen droplet or a grain of aggregate in a polar or ionic medium, attract layers of polar molecules or ions onto their surfaces (145). These particles may be positively or negatively charged which dictates the sign of the ions attracted. The first layer is strongly attached and the surface of this mantle is known as the Stern plane. Further diffuse layers are attracted until the charge is neutralised and there is no further effective charge from the central particle. The inner and outer layers are known as the electrical double layer. If the charged particle is placed between two electrodes, it is caused to move towards the plate of opposite electrical sign. This process is known as electrophoresis. As the particle moves through the ionic medium, a shear plane arises between those ions which are held by the charged particle and ions in the bulk solution. The electrical potential across this region is known as the zeta potential. It is closely related to the charge on the central charged species but it should be stressed that it is not the actual potential at the surface of the particle itself. Figure 11.1 illustrates the above details diagrammatically for a particle with a positive surface charge. The graph included in the diagram shows how the potential changes with distance from the particle surface. Equation 11-1 can be used to determine the electrostatic potential, Ψ, as a function of the distance from the particle surface, x, with a potential at the surface, Ψ_0. κ is the parameter which measures the decay of the potential with distance :

$$\Psi = \Psi_0 . e^{-\kappa x} \qquad (11\text{-}1)$$

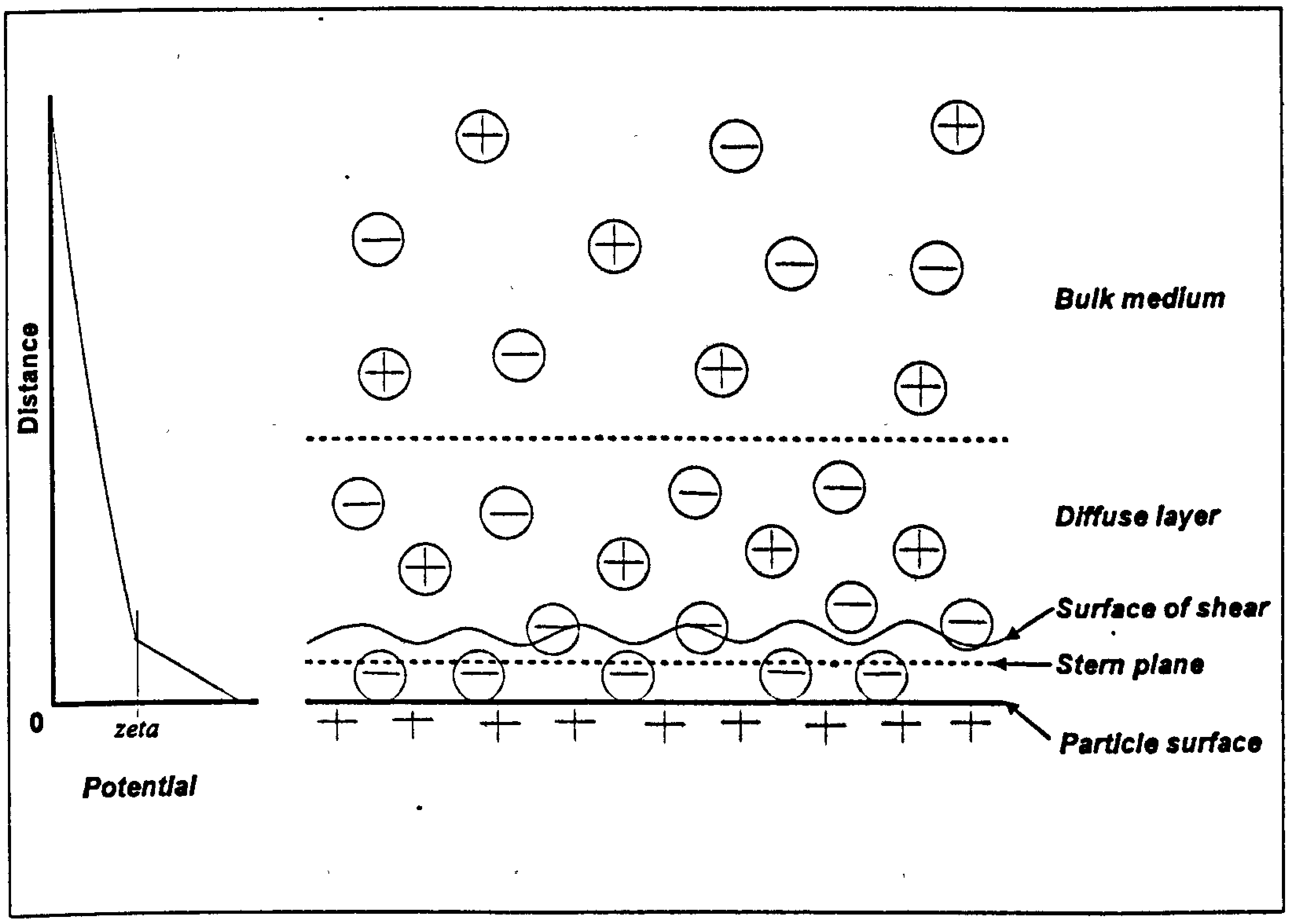


Figure 11-1 : Charged species in an ionic medium

11.2.1 Zeta potential measurement

Various methods are available to measure zeta potential. In this study, apparatus which determines electrophoretic mobility was used. When a positively or negatively charged particle, as described above, is placed in an electric field (E, units Vcm^{-1}), ie. between two electrodes, it moves towards the electrode of opposite sign. The velocity of the particle (V_s, units μs^{-1}) as it travels through the suspending medium is related to the electric field strength by a factor μ_e (units $\mu.s^{-1}.V^{-1}.cm$) known as the electrophoretic mobility. ie.

$$V_s = \mu_e . E \qquad (11\text{-}2)$$

Zeta potential apparatus use various techniques to measure the velocity of particles moving in an electric field. The relationship between zeta potential and electrophoretic mobility is given by either of the two equations below (11-3,11-4). These are based on different models after Smoluchowski and Hückel (146). They apply at opposite limits of the magnitude of dimensionless unit, κa, where a is the radius of the kinetic unit or the particle plus any accompanying species from the suspending medium. The two equations are :

Hückel Limit : $\mu_e = (2\varepsilon\varsigma/3\eta)$ for $\kappa a << 1$ (11-3)

Smoluchowski Limit : $\mu_e = \varepsilon\varsigma/\eta$ for $\kappa a >> 1$ (11-4)

Where : ε = Permittivity of the dispersing medium
ς = Zeta potential (mV)
η = Viscosity of suspending medium (mPa.s)

For the particle sizes involved in emulsion and aggregate samples, it is the Smoluchowski equation which applies.

Rearranging equations 11-3 or 11-4 for zeta potential and equation 11-2 for mobility it can be seen that zeta potential can be found from velocity, electric field strength and viscosity and permittivity of the dispersing medium. Permittivity is a measure of the ability of a medium to allow interaction between charged species.

In the zeta potential measuring technique, velocity is determined via measurement of the Doppler shift of a laser beam scattered by particles moving at right angles to the incident beam. This method is employed in the 'Brookhaven Zetaplus' apparatus used for the measurements carried out in these studies (147). The diagram in Figure 11-2 shows the basic layout and workings of the Zetaplus apparatus.

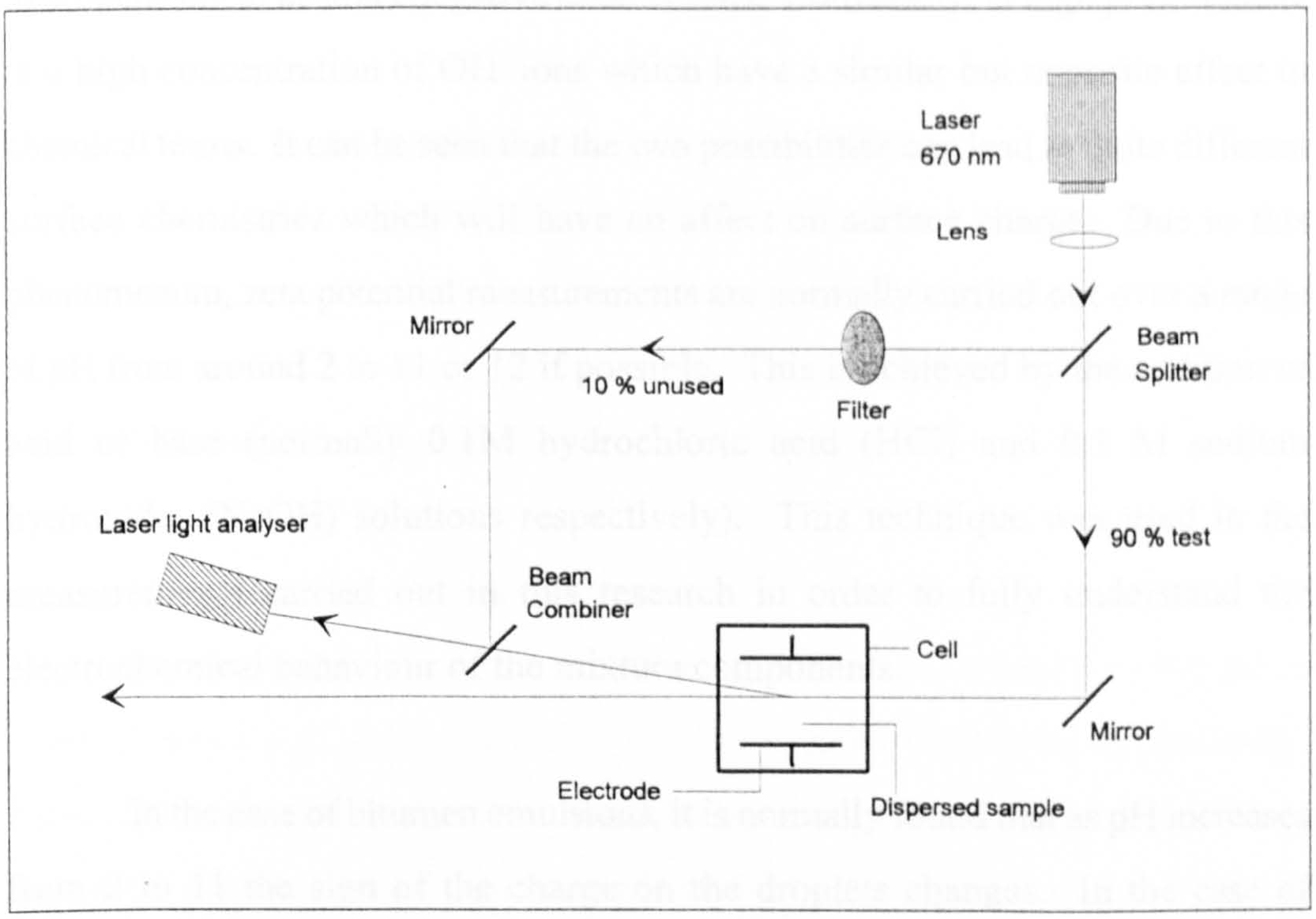


Figure 11-2 : Basic layout of the Brookhaven Zetaplus

An incident laser beam of 670 nm wavelength is split into two. One part is directed through the cell containing the material under test whilst the other is unused. As the beam moves through the sample cell it is scattered by particles moving in the electric field which causes a Doppler shift of the beam's frequency to occur. The frequency shift is only a few tens of Hertz compared with the frequency of the laser light of 10^{14} Hz. The unused incident beam and the shifted test beam are then recombined and the difference in frequency analysed. This difference is used to calculate the velocity of the particles moving in the cell. Software provided by the Brookhaven Zetaplus converts the measured velocities to zeta potentials using the Smoluchowski equation (11-4).

11.2.2 Variation of zeta potential with pH

The surface charge on a particle can be affected by the pH (acidity or basicity) in the suspending medium. This is due to changes in the chemistry of the surface. At low pH (<7) there is a high concentration of H^+ ions in the suspending medium, which can either be bonded onto the surface of the particles in suspension, or abstract negative counter ions. Conversely, at high pH (>7)there is a high concentration of OH^- ions which have a similar but opposite effect in chemical terms. It can be seen that the two possibilities can lead to quite different surface chemistries which will have an affect on surface charge. Due to this phenomenum, zeta potential measurements are normally carried out over a range of pH from around 2 to 11 or 12 if possible. This is achieved by the addition of acid or base (normally 0.1M hydrochloric acid (HCl) and 0.1 M sodium hydroxide (NaOH) solutions respectively). This technique was used in the measurements carried out in this research in order to fully understand the electrochemical behaviour of the mixture components.

In the case of bitumen emulsions, it is normally found that as pH increases from 2 to 11 the sign of the charge on the droplets changes. In the case of cationic emulsions, the change is from positive to negative as pH increases. The point at which the droplets have neutral charge (ie change sign) is known as the zero point of charge (ZPC). This figure is dependent upon the cationicity of the emulsion, being at higher pH for strongly cationic and lower pH for weakly cationic systems, as is the magnitude of zeta potential itself.

11.3 EXPERIMENTAL

Zeta potential measurements were carried out on emulsions and on aggregates used in the course of these studies in order to determine the effect of various parameters on their electrochemical properties.

Measurements were also carried out on the standard bitumen emulsion with and without three additives. The additives in question were Ordinary Portland Cement, hydrated lime and $CaCl_2$. OPC is known to have a marked effect on curing rate of emulsion mixtures (16), as will be discussed later in this report, and has been shown to increase the coalescence of emulsion under compaction. Hydrated lime and $CaCl_2$ were to be investigated due to the suspicion that Calcium ions in cement may give rise to these effects as these materials are also a source of calcium ions.

11.3.1 Preparation of emulsion samples with additives

The emulsion used for all tests comprised of 62 % Venezuelan 100 pen bitumen with 1.2 % Redicote E-4868 as the emulsifier as described in Table 6-2. OPC, lime or $CaCl_2$ were added to the emulsion and stirred in 2 hours before measurements were made to allow the system to equilibrate.

11.3.2 Zeta potential measurements

Several drops of each emulsion in question were added to ~400 ml of distilled water and gently stirred. The dispersions were used immediately. Measurements were made on a Brookhaven Zetaplus. Zeta potentials were measured over a range of pH. The pH was adjusted up or down using 0.1M NaOH or 0.1M HCl solutions as necessary, measured by means of a Corning pH meter to two decimal places.

11.3.3 Limitations of the technique

In order for the laser beam to pass through the sample in the apparatus shown in Figure 11-2, the sample must be very dilute. According to standard practice, only a few drops of emulsion or a few grams of aggregate are dispersed in ~ 400 ml of distilled water. This is a very artificial situation for the particles as the concentrations are very low and the dispersing medium is free of ions other than OH^-, H_3O^+ and H^+ or surfactant. Dilution has the effect of greatly increasing the inter particle distances and ionic concentration is known to have an effect on the double layer (145). However, it is generally accepted that the measurement of zeta potential in this way is representative of the real situation.

11.4 RESULTS

The graph in Figure 11-3 shows the results from the zeta potential measurements on Redicote E-4868 emulsions with 100 pen binder and a range of emulsifier levels.

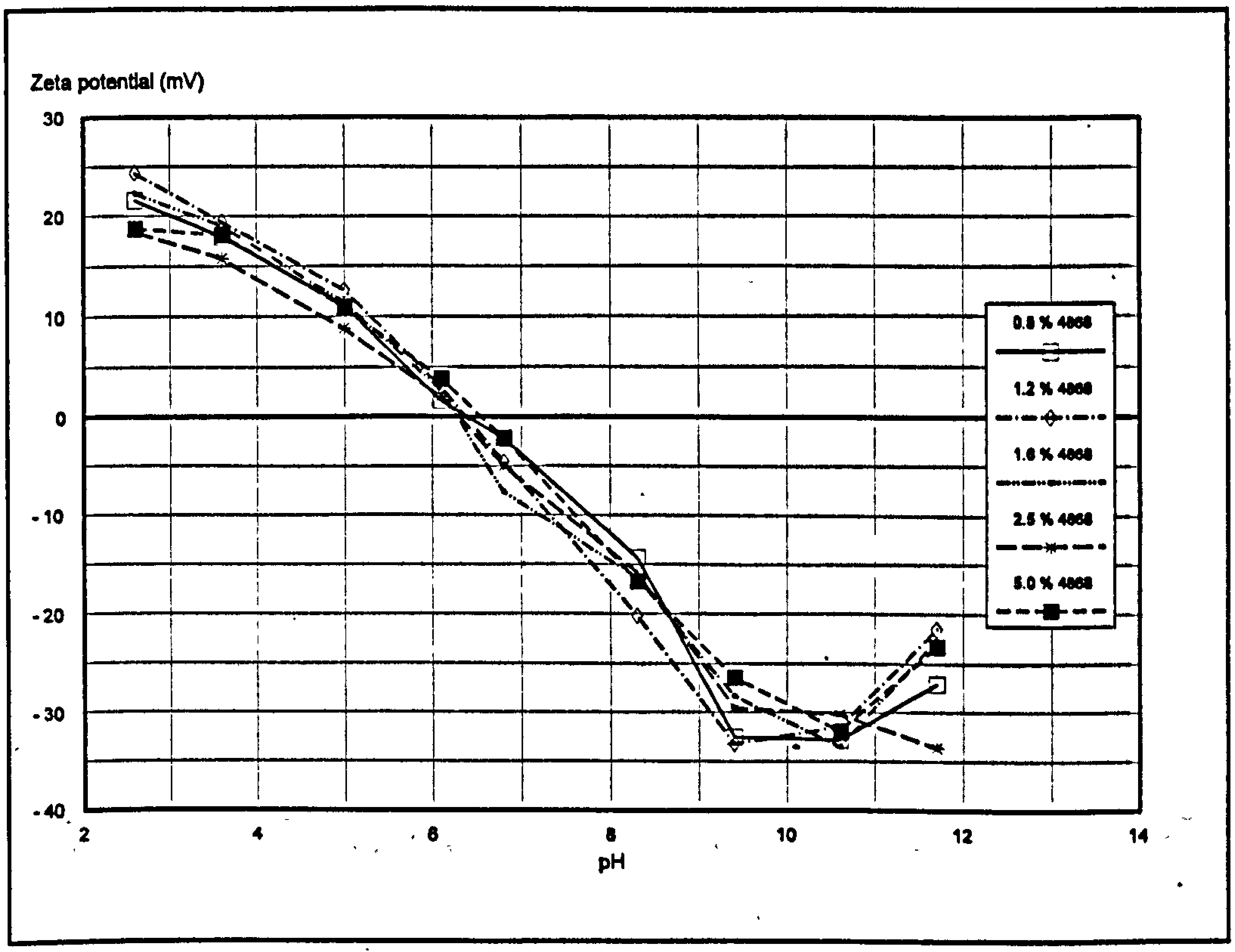


Figure 11-3 : Zeta potentials of emulsions with range of emulsifier levels

The graph in Figure 11-4 shows the results from the zeta potential measurements on 1.2 % Redicote E-4868 emulsions with a range of bitumen penetration grades. The profile for Croft granite, the aggregate used in the majority of these studies, is included for comparison.

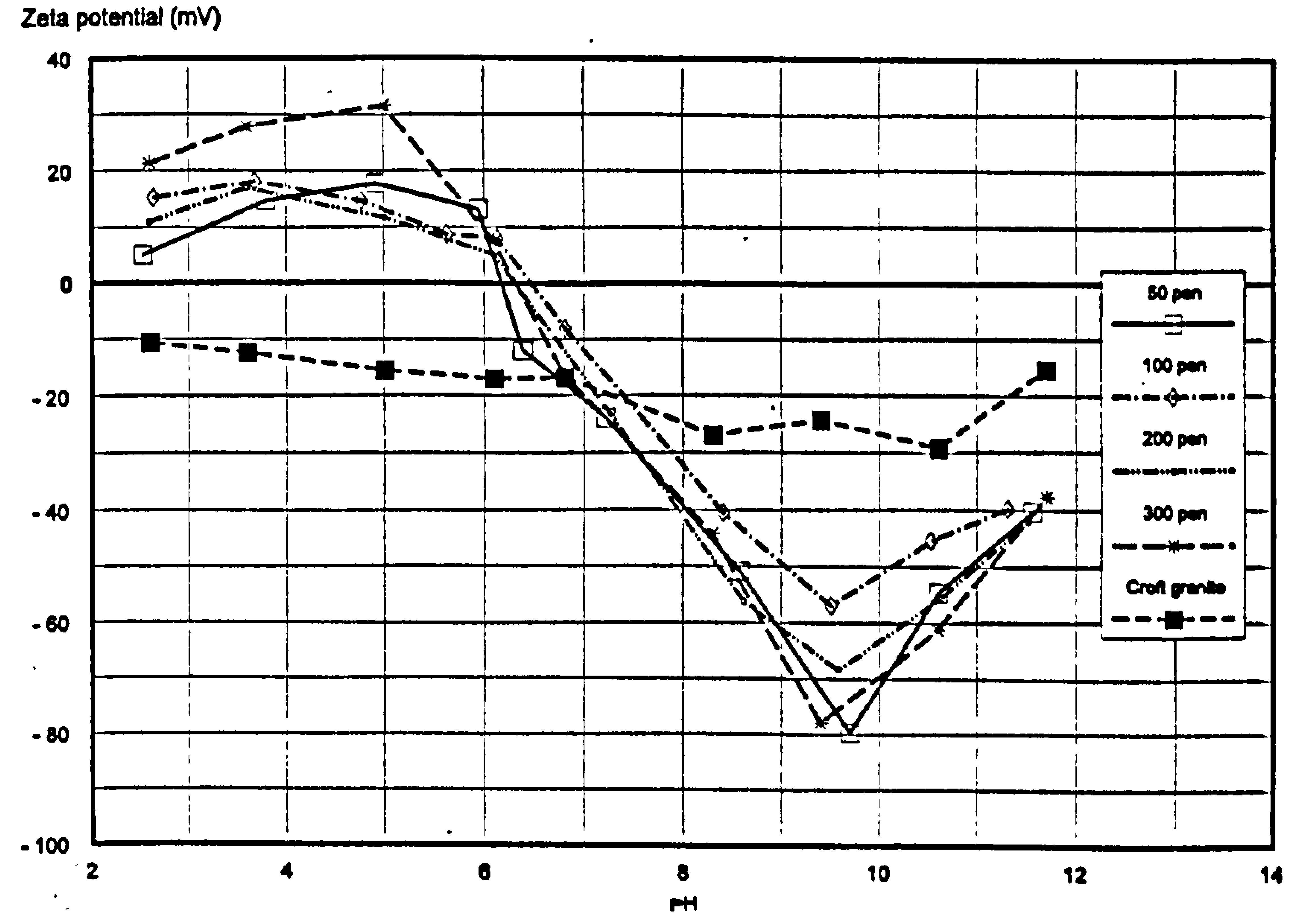


Figure 11-4 : Zeta potentials of emulsions with range of bitumen grades

Figure 11-5 shows the results of zeta potential measurements on emulsions containing polymer modified binders.

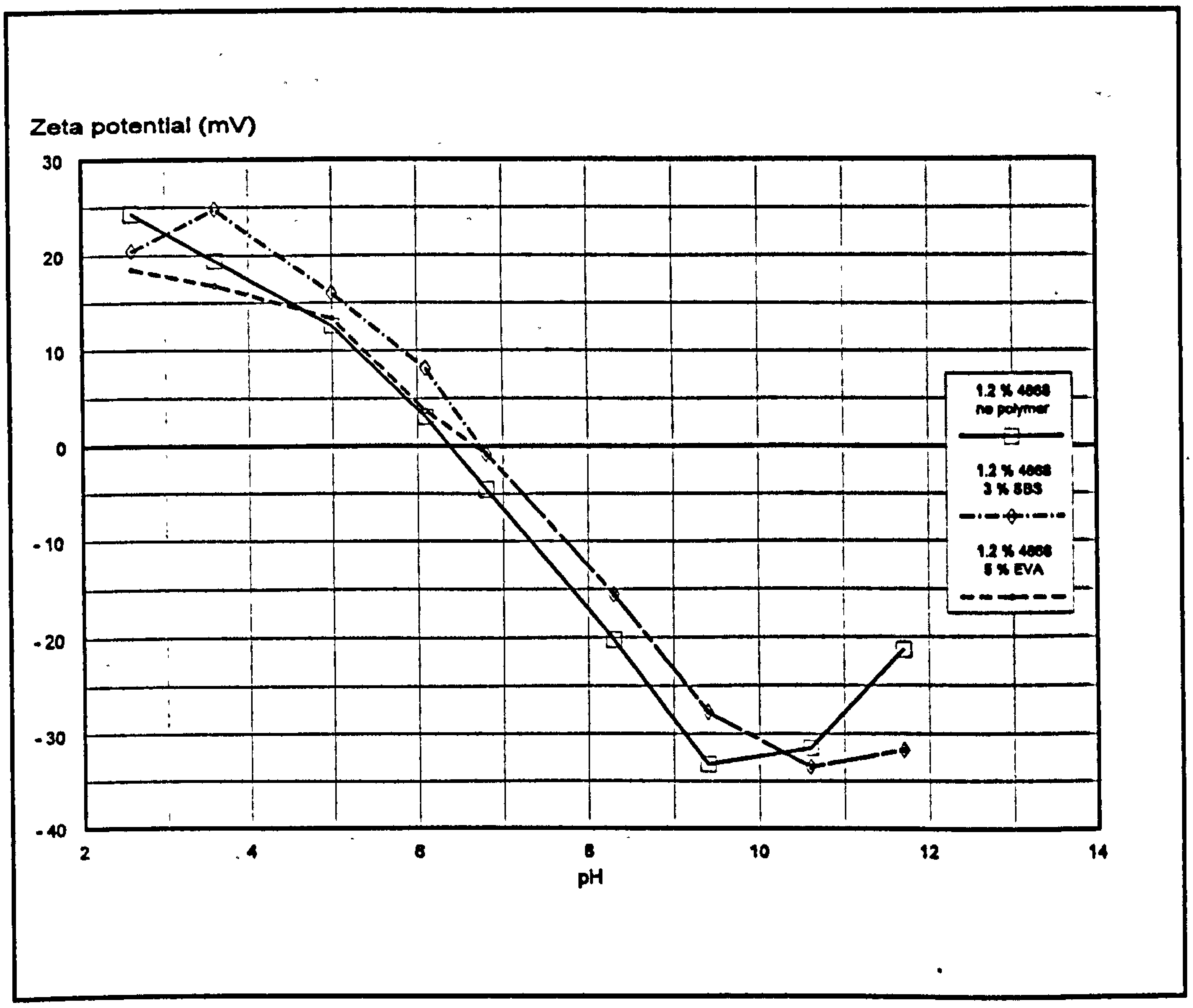


Figure 11-5 : Zeta potentials of emulsions with polymer modified binders

Figure 11-6 shows the results of zeta potential measurements on the standard emulsion with OPC, hydrated lime and $CaCl_2$.

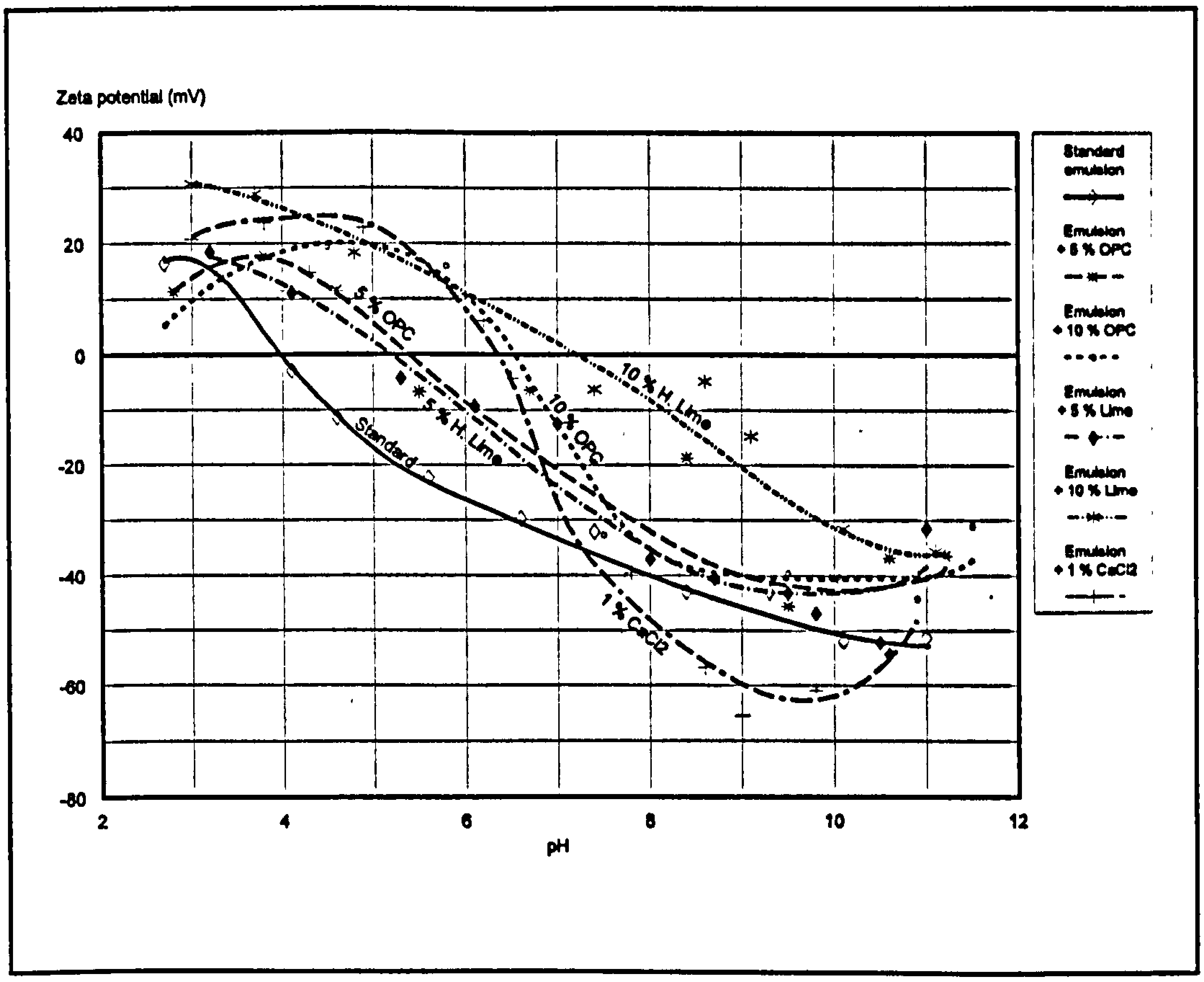


Figure 11-6 : Zeta potentials of emulsions with OPC, lime and $CaCl_2$

11.5 DISCUSSION

Comparing the data obtained for the standard emulsion in each set of tests, it can be seen that there is a slight variation in the zeta potential profiles. The samples of emulsions used for these measurements were often taken from different batches of emulsion or the same batch of emulsion at different lengths of time after manufacture. It appears that the electrochemical properties of the emulsions changed over time and that different batches of nominally the same emulsion are also slightly different. However, as the same emulsion was used within each set of tests, the results in each graph can be compared.

In all cases the emulsions were found to be amphoteric. This means that they changed sign - being cationic at low pH and anionic at high pH. The point of zero charge was around 6 to 7 in most cases, except for the emulsion used in the OPC and lime additive tests where it was only 4.

From the graph in Figure 11-3, it can be seen that increasing the dosage of the Redicote E-4868 emulsifier had no effect on the zeta potential profile of the emulsion, even up to a dosage level of 5 %. This suggests that the bitumen water interface is already saturated at the lower emulsifier dosages and, therefore, further addition of emulsifier merely raises the concentration of free emulsifier in the waterphase. Thus the droplet charge is unaffected. Similarly, the data in Figure 11-4 showed that the penetration grade of the bitumen also had no effect on zeta potential. According to the results shown in Figure 11-5, polymer modification of the binder also had no effect on zeta potential. The data for the granite aggregate (Figure 11-4) showed that it had a negative zeta potential over the entire pH range which is typical of a siliceous aggregate such as this (143).

The data in Figure 11-6 are somewhat scattered compared to that in the other graphs. This was thought to have been a consequence of deviations in pH which occurred during the tests. pH deviated due to solvation of cement and lime additives during the measurement period which would have led to an increase in pH. It is therefore difficult to say categorically in what way the addition of OPC, hydrated lime and $CaCl_2$ affected zeta potential. However, if appraisal of the data is limited to the pH region 4 to 7, it would appear that a definite increase in zeta potentials or shift to higher pH of the ZPCs occurred. The pH of a normal mixture with aggregate is around 7 and it was thought, therefore, that the electrochemical change affected by the additives could have a significant influence on the breaking behaviour of these emulsions in contact with negatively charged aggregate.

As the common feature between OPC, hydrated lime and $CaCl_2$ is

Calcium ions, it would seem likely that it is they which give rise to the increase in cationicity; particularly as they are themselves strong cations. At this stage it is not known by what mechanism these ions are adsorbed. Due to the polymeric molecular nature of some of the components of the Redicote E-4868 emulsifier, however, it is possible that Calcium ion complexes are formed. A complex is a formation composed of metal ions surrounded by electrically attracted organic molecules. If Calcium ions were locked onto the surface of bitumen droplets in this way the droplets would become significantly cationic.

11.6 ZETA POTENTIAL MEASUREMENTS IN RUN OFF WATER

Subsequent consideration of the system with OPC initiated further tests. It was thought that if OPC were used in a mixture, the pH of the system would probably be raised due to the basicity of this material. Therefore, mixtures were prepared with no OPC and 1 % OPC. These were compacted using a static load press with a load of 16 MPa. A substantial amount of run-off water was squeezed out and this was collected. The pH's of the break water samples were measured using a pH probe. The specimens were centrifuged and then the supernatants were filtered through very fine graded filter paper to remove all solids. The pH's of the filtered samples were remeasured to ensure that they had not altered. The samples were quite different in colour with the water from the mixture containing OPC being a much darker shade of yellow. Both of the samples had the odour of the emulsifier and/or bitumen to some degree.

Samples of standard emulsion and granite aggregate in run-off water were prepared and used for zeta potential measurements. As a comparison, samples of emulsion and aggregate in distilled water, adjusted to pH's equivalent to those of the run-off samples, were prepared.

11.6.1 Results

The results of the pH and zeta potential measurements described above are shown in Table 11-1.

Table 11-1 : Zeta potentials of emulsions in run-off water and distilled water

Dispersing solution	pH	Zeta potential (mV)	
		Aggregate	Bitumen emulsion
Distilled water	7.4	-30.5	-12
Distilled water	11.7	-12.5	-19.5
Run-off water (ex mixture with no OPC)	7.4	0	-2.5
Run-off water (ex mixture with 1% OPC)	11.7	+4	-2

Discussion

The data in Table 11-1 show that the zeta potentials of aggregate and emulsion in run-off water were very different from those in distilled water at the same pH. In distilled water, both the aggregate and emulsion had quite negative zeta potentials but in run-off water, they were both virtually neutral, with or without OPC. The reason for this is thought to be that the ionic concentrations in the samples of run-off water are so high that the emulsion or aggregate particles are doped with an assortment of ions from solution which masks the charges arising from surface properties of the species under normal conditions. There is a slight positive charge on the aggregate in the high pH run-off water coupled with a slight negative charge on the bitumen droplets. This may give rise to a small attraction in a mixture but it is doubted that it would be significant.

The effect of high ionic concentration is to compress the double layer as discussed above. The diagram in Figure 11-7 shows how the zeta potential against distance from the particle is affected. It can be seen that at any given distance the zeta potential on the particle will be lower when the ionic strength is high. This was probably the main cause of the results shown in Table 11-1.

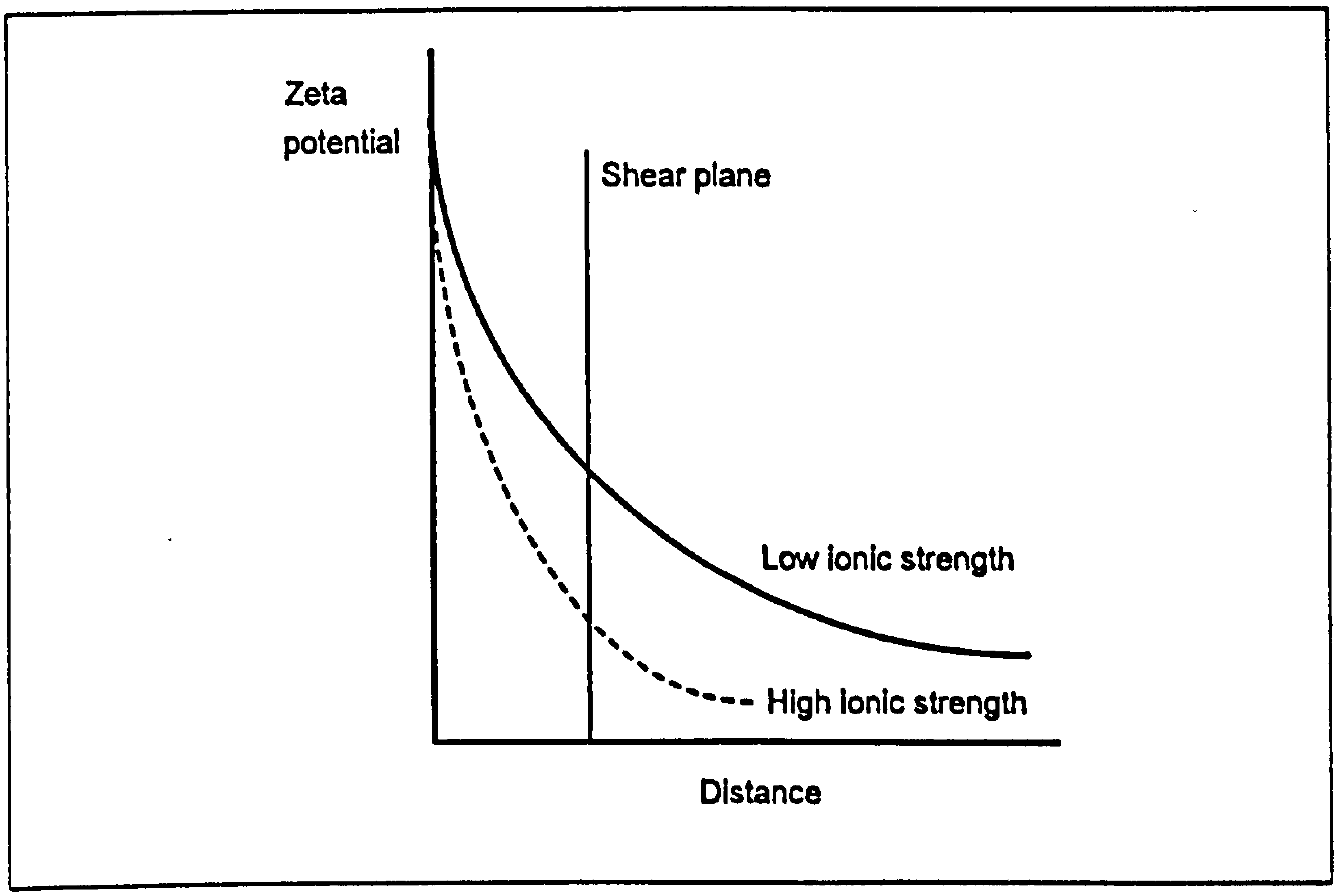


Figure 11-7 : Effect of ionic strength on zeta potential

11.7 CONCLUSIONS

The results obtained from the above tests suggest that variation of emulsifier level, binder grade and polymer modification of the binder have no effect on the electrochemical properties of the standard emulsion. In a mixture it would appear that both the emulsion and aggregate bear a negative charge. This would account for the fact that the emulsion can be mixed with a dense graded aggregate mixture without premature break due to the fact that there is no electrical attraction between granite aggregate and emulsion. However, it does not help to explain the differences in coalescence rates due to variation of certain parameters as described in chapter 10.

In distilled water, OPC, lime and $CaCl_2$ increase the zeta potential of the standard emulsion over a limited pH range but in run off water, which is more representative of the situation in a real mixture, it was found that both the emulsion and aggregate were virtually neutral. This lack of charge difference in realistic conditions suggests that the increased coalescence rate of cement containing mixtures does not appear to be due to electrochemical effects.

CHAPTER 12

PROPERTIES OF BITUMEN EMULSION RESIDUES

12.1 INTRODUCTION

In chapters 8 and 9 of this report, the results of various tests were reported which showed the ways in which the mechanical properties of bitumen emulsion mixtures develop over time. The effects of various additives were also addressed and were found to affect both the curing process and ultimate stiffness. The main variable in these tests was the binder, as the aggregate mixture was the same in most cases. The tests described in this chapter aimed to study the behaviour and properties of the binder in combination with various additives in the absence of aggregate.

12.2 BITUMEN EMULSION AND ORDINARY PORTLAND CEMENT MIXTURES

ITSM tests showed that the addition of OPC to bitumen emulsion mixtures increased the rate of stiffness build up and also final stiffness. Coalescence tests showed that OPC increased the amount of break of an emulsion in an aggregate mixture under compaction. Thus, mixtures of emulsion and cement were studied in order to determine the emulsion breaking process and also the properties of the composite binder.

12.2.1 Breaking behaviour of emulsion and OPC mixtures

Mixtures of bitumen emulsion and cement were produced. 200g samples of the emulsion were used, to which a certain amount of water had been added based on that which would be required in a real mixture with aggregate. Cement was added at levels of 1 to 10 %, by weight on a hypothetical aggregate mixture, and stirred for about 30 seconds. In most of the mixtures made throughout the course of these studies, emulsion was normally added at a level of 8.1% on the aggregate mixture and so, in relation to the emulsion, these addition levels corresponded to ratios of 1 to 10 : 8.1 (cement : emulsion). The mixtures were covered and inspected periodically. An assessment of the percentage break of the emulsion was made by means of a comparative estimate. This method was very subjective but it was felt that the decisions recorded were fairly accurate, at least within this set of tests. After 48 hours, all of the mixtures had set completely and were found to consist of a solid mass of bitumen - cement mastic with slightly yellow coloured, clear water on top. The setting rates of mixtures with various OPC addition levels were as shown in Table 12-1.

Table 12-1 : Breaking behaviour of bitumen emulsion with cement

Cement	Percentage coalescence of mix				
*	1 hr	4 hr	6.5 hr	24 hrs	48 hrs
1	0	0	5	10	100
2	0	0	10	100	-
3	0	10	50	100	-
4	0	10	100	-	-
5	0	50	100	-	-
6	10	100	-	-	-
7	15	100	-	-	-
8	15	100	-	-	-
9	15	100	-	-	-
10	15	100	-	-	-

* - expressed as percentage weight on a hypothetical aggregate mix

These results show that the addition of as little as 1 % OPC to an aggregate mixture (corresponding to 12 % OPC on the emulsion), caused the emulsion to fully break within 48 hours. Increasing the level of OPC resulted in shorter and shorter break times. The speed with which the emulsion broke was a surprising discovery when compared with the findings from ITSM tests on full mixtures containing cement in chapter 8. In the ITSM tests, stiffness was found to build steadily over a number of weeks. According to the tests described here, the binder should be fully cured after only 48 hours resulting in the ultimate stiffness being reached. This raises questions as to what is different about the emulsion and OPC mixture in the presence and absence of aggregate.

12.2.2 Penetration of cured binder and OPC mastics

The mixtures obtained from the breaking tests described above were used for the tests which follow. Water was poured off the solidified binder residues, weighed and a measurement of pH taken. These results are shown in Table 12-2. After a further 48 hour drying period at ambient temperature, the mixtures were all brought to a temperature of 25°C in a water bath. The penetrations of the cured mastics were then measured, using a standard penetrometer (22) with a total needle weight of 100 g, and load times of 5 and 60 s. The 60 s load time was used as the 5 s period gave very low values due to the very hard nature of some of the mastics. The results of these tests are shown in Table 12-3.

Table 12-2 : Weight and pH of water evolved from emulsion and OPC mixtures

Cement *	1	2	3	4	5	6	7	8	9	10
Weight / g	26.1	33.4	35.4	36.8	33.5	37.5	36.7	35.7	30.9	29.7
pH	13.34	13.47	13.56	13.65	13.71	13.78	13.79	13.82	13.87	13.91

* - expressed as percentage weight on a hypothetical aggregate mix

Table 12-3 : Penetration of cured mastics

Cement *	1	2	3	4	5	6	7	8	9	10
Pen / 5 s (dmm)	112	82	27	10	13	10	4	3	5	5
Pen / 60 s (dmm)	189	157	100	48	36	30	20	18	18	15

* - expressed as percentage weight on a hypothetical aggregate mix

From the results of the pH measurements on the water evolved from emulsion breaking, it can be seen that with only 1 % OPC added to the emulsion, the pH increased from about 6 to over 13. Increasing the level of OPC only caused a slight further increase in pH. OPC is a very alkaline substance and, therefore, the results were not surprising. The mechanism of emulsion break through the addition of OPC may, therefore, be partly due to the pH shift. However, the fact that 1 % OPC has almost the same effect on pH as 10 % OPC indicates that the increase in breaking rate of mixtures with higher levels of OPC was not due to a pH effect. It should be noted that the pH of run-off water from a full mixture was found to be 11.7 as discussed in chapter 10. This is slightly lower than the pH's reported here but still very alkaline.

The results shown in Table 12-3 revealed that the presence of OPC had a significant effect on penetration. The bitumen in the emulsion used for these tests was nominally 100 pen. The addition of 2 % or more OPC resulted in a reduction in penetration of the residual binder. It is uncertain why the mastic with 1 % OPC was softer than the virgin bitumen but it was probably caused by the entrainment of a small amount of water in the mixture. Stiffening of the binder in this way would have a major effect on the mixture stiffness. Mixture stiffness can be predicted using equation 12-1 below (114) which uses only the binder stiffness and void content of the mixture :

$$S_{me} = S_b\left[1 + \frac{257.5 - 2.5VMA}{n(VMA - 3)}\right]^n \qquad 12\text{-}1$$

Where : S_{me} = Stiffness modulus of the mixture (MPa)

VMA = voids in the aggregate mixture (%)

$$n = 0.83\log\left[\frac{4\cdot10^4}{S_b}\right] \qquad 12\text{-}2$$

Sb = Stiffness of the bitumen (MPa)

The stiffness of the bitumen can be determined from equation 12-3 which was derived by Ullidtz (148) based on Van der Poel's nomograph (149). Strictly speaking, this equation applies only to pure bitumen but it serves to illustrate the part that binder stiffness or penetration plays in overall mixture stiffness :

$$S_b = 1.157\cdot10^{-7}t^{-0.368}2.718^{-PI_r}(SP_r - T)^5 \qquad 12\text{-}3$$

Where : t = Loading time (s)

PI_r = Penetration index

SP = Softening point (temperature at which the binder has a penetration of 800) (°C) (ASTM D 36-66 T - appendix 1)

T = Temperature (°C)

This equation is only applicable over a limited range where :

t is between 0.01 and 0.1 s

PI is between -1.0 and + 1.0

Temperature difference, (SP-T), is between 20 and 60 °C

Penetration index can be determined using equation 12-4 (114) :

$$PI = \frac{20(1-25A)}{1+50A} \qquad 12\text{-}4$$

Where :

$$A = \frac{\log(800) - \log P}{SP-25} \qquad 12\text{-}5$$

Where : P = Penetration (1/10 mm)
SP = Softening point (°C)

Due to the findings of the penetration tests, a more rigorous rheological evaluation of the properties of the cured binder was undertaken. In addition to OPC, the effects of hydrated lime and also limestone filler were evaluated.

12.2.3 Rheological studies on mixtures of binder with OPC, hydrated lime and filler

Mixtures of bitumen emulsion with OPC and hydrated lime and 100 pen Venezuelan bitumen with limestone filler were prepared and used for rheological measurements on a dynamic shear rheometer described later in this section.

Basic rheological concepts

When a fluid is subjected to stress (greater than a certain yield value) it flows (150, 151). Figure 12-1 depicts an element of material subjected to a shear stress deforming at a certain rate of shear.

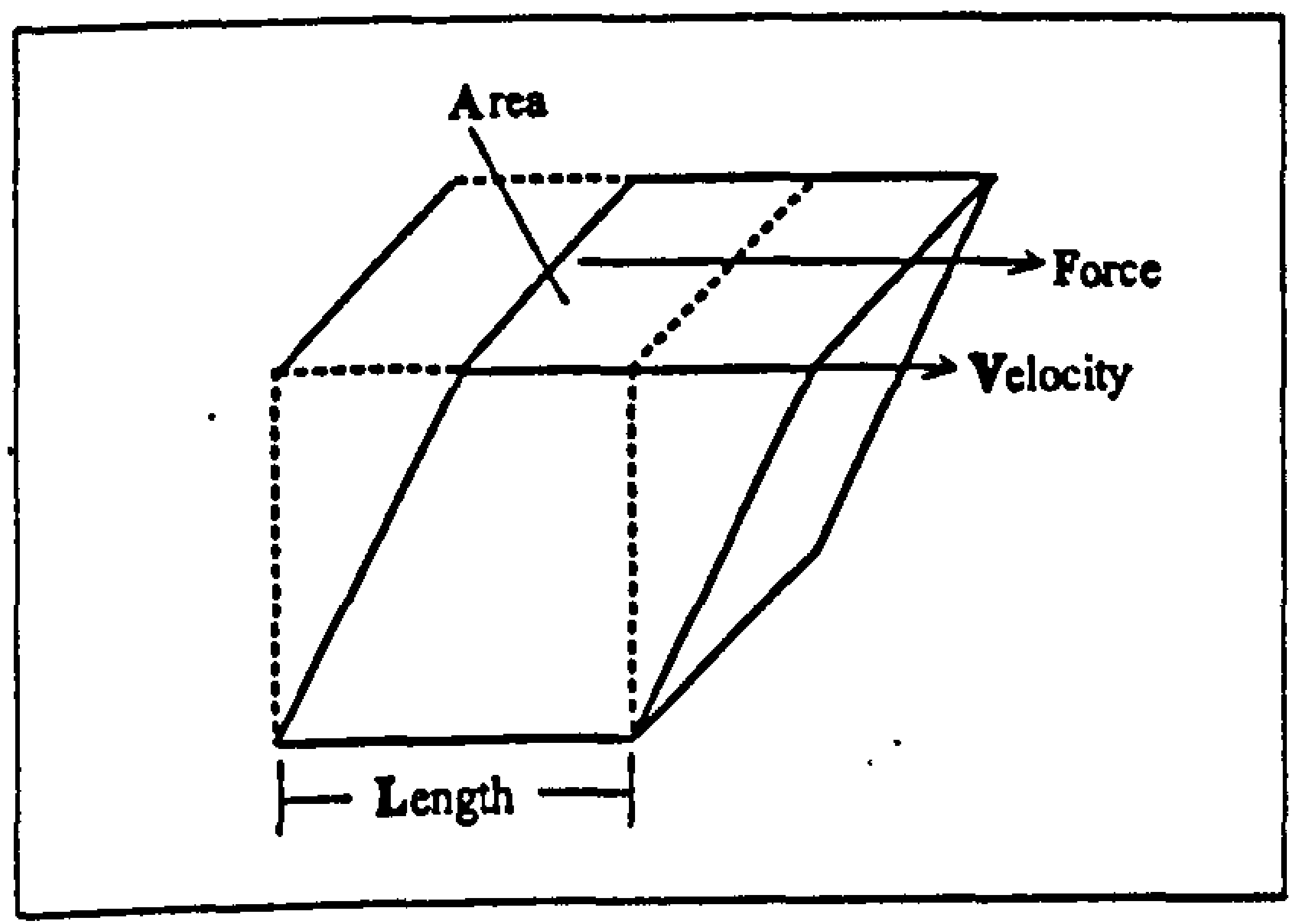


Figure 12-1 : Shear stress and shear rate on a fluid element

Shear stress, $\tau = F / A$ (Nm^{-2})
Shear rate, $d\gamma/dt = V / L$ (s^{-1})

Viscosity, $\eta = \tau / (d\gamma/dt)$ ($Nm^{-2}.s$ or Pa.s)

Fluids which show a proportional increase in shear rate with shear stress are said to be Newtonian. Fluids which become more viscous with increasing shear rate are dilatant whilst those which become less viscous are shear thinning or plastic. Bitumen is a viscoelastic material which means that it exhibits some viscous or plastic and some elastic behaviour. Under long loading times and high temperature, bitumen will behave in a viscous manner, deforming permanently, whereas, under short loading times and low temperature it will behave elastically.

Through dynamic rheological measurements, information regarding the elastic and viscous or damping response of viscoelastic materials can be obtained. Dynamic measurements involve subjecting a sample of material to oscillating sinusoidal stress at a range of frequencies and measuring the response. For paving materials, this is analogous to the passage of a vehicle load over a pavement structure. If a perfectly viscous material is subjected to a stress, a phase lag of 90° occurs between the stress and the resulting strain. Conversely, perfectly elastic materials show a phase lag of 0°. Viscoelastic materials exhibit a phase lag somewhere between 0 and 90° dependent on their nature and the test parameters. The angular difference between stress and strain is termed the phase angle. Complex stress and strain of viscoelastic materials is given by equations 12-6 and 12-7 respectively (152) :

$$\tau^{*}=\tau_{0}e^{i(\omega t+\delta)} \qquad \textbf{12-6}$$

$$\gamma^{*}=\gamma_{0}e^{i\omega t} \qquad \textbf{12-7}$$

Where : i = square root of -1

ω = angular frequency (ω = 2πf)

δ = phase angle (radians)

Complex stress and complex strain are vectors in complex planes. They can be resolved into real (in phase) and imaginary (90 ° out of phase) components such that :

$$\tau^{*}=\tau'+i\tau'' \qquad 12\text{-}7$$

$$\gamma^{*}=\gamma'+i\gamma'' \qquad 12\text{-}8$$

The shear modulus can also be represented by a complex variable known as the complex dynamic shear modulus, G*, which is the ratio of the complex stress and strain :

$$G^{*}=\frac{\tau^{*}}{\gamma^{*}} \qquad 12\text{-}9$$

The dynamic modulus can be resolved into two components :

$$G^{*}=G'+iG'' \qquad 12\text{-}10$$

Where :

$$|G^{*}|=\sqrt{(G')^{2}+(G'')^{2}} \qquad 12\text{-}11$$

and :

$$G'=|G^{*}|\cos\delta \qquad 12\text{-}12$$

$$G''=|G^{*}|\sin\delta \qquad 12\text{-}13$$

G' is the storage modulus. It is in phase with the real components of τ^* and γ^* and is associated with the energy stored in elastic deformation. G" is the loss modulus. It arises from the out of phase components of τ^* and γ^* and is associated with the viscous energy dissipation or damping.

The variables described above can be determined by means of an instrument known as a dynamic shear rheometer.

The Dynamic shear rheometer

The dynamic shear rheometer (DSR) is an example of a parallel plate rheometer. As illustrated in Figure 12.2, specimens are placed between two parallel metal discs, with a known gap spacing and subjected to torsional stress by rotation of one with respect to the other. Measurement of speed and torque allows shear rate and shear stress to be determined using equations 12-4 and 12-5 below (151) :

$$\gamma_R = \frac{R\Omega}{H} \qquad 12\text{-}4$$

Where : γ_R = Shear rate at the disc rim
R = Disc radius
Ω = Rotational velocity
H = Distance between discs

$$\tau_R = \tau_{RN}\left[\frac{3}{4} + \frac{1}{4}\frac{d\ln\tau_{RN}}{d\ln\gamma_R}\right] \qquad 12\text{-}5$$

Where : τ_R = shear stress
τ_{RN} = shear stress at disc rim and is given by
γ_R = shear strain

$$\tau_{RN} = \frac{T_0}{1/2\pi R^3} \qquad 12\text{-}6$$

Where : T_0 = Torque on rotating disc

In these studies, specimens were tested under an oscillation mode, at a range of frequencies from 0.01 to 10 Hz. The top plate moves sinusoidally with respect to the fixed lower plate. This action is representative of the passage of a vehicle in a road situation - the rate of oscillation simulates different vehicle velocities. The low rate of oscillation is analogous to slow moving or parked vehicles and the higher rate corresponds to fast moving vehicles. During the test, the bitumen sample is held at a constant temperature by immersion in a temperature controlled water bath. The DSR apparatus is shown diagrammatically in Figure 12-2.

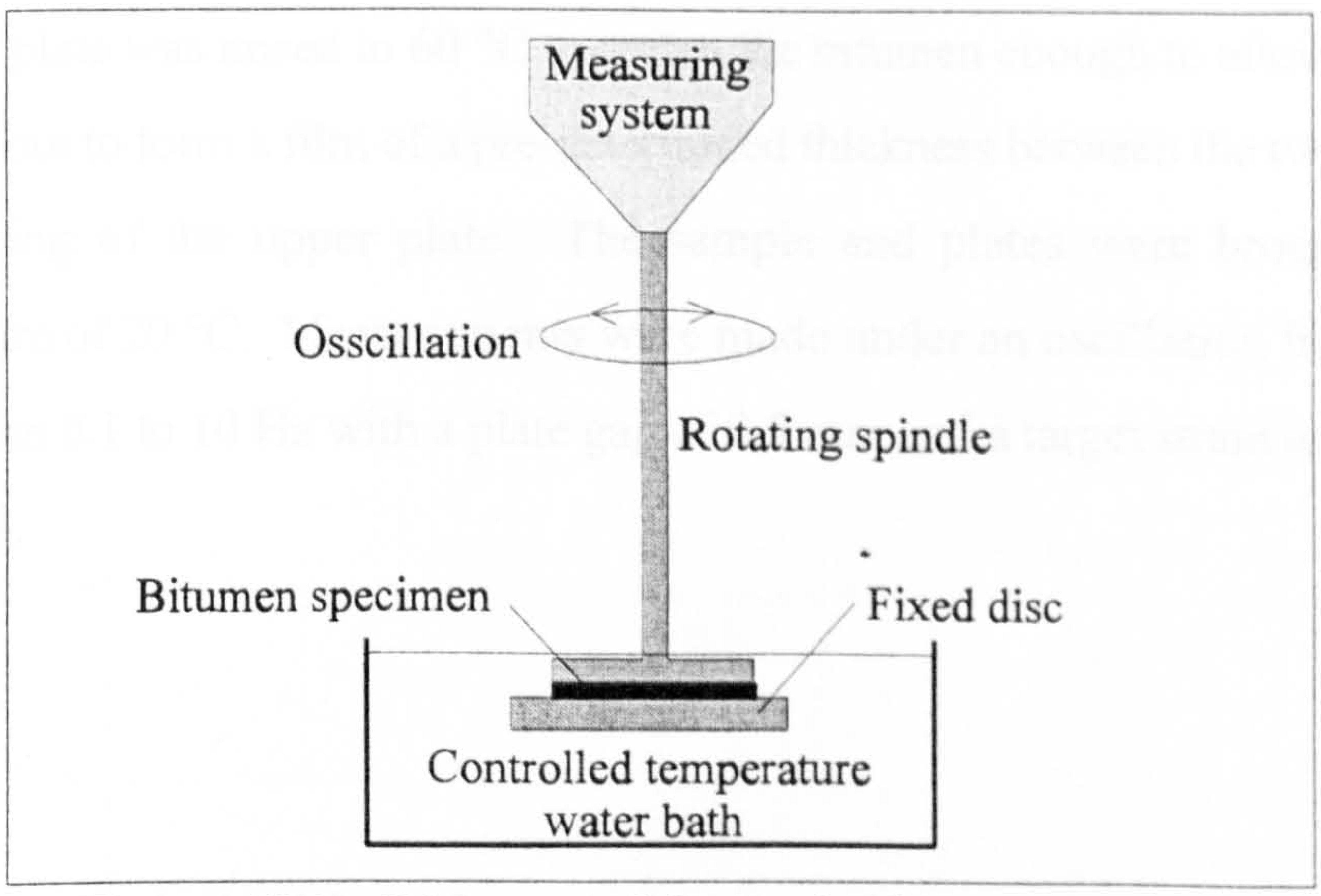


Figure 12-2 : Diagram of dynamic shear rheometer

Production of mixtures

Mixtures similar to those used for penetration measurements described above were prepared, by adding OPC and hydrated lime, at a range of addition levels, to samples of the standard emulsion and rolling for several days until full emulsion break had occurred. Any excess water was poured off and the mastics left to dry at room temperature for several days. In addition, mixtures of hot binder and limestone filler were produced. Mixtures of emulsion residue and limestone could not be produced as this material would not have fully broken the emulsion. Filler was added at a range of addition levels to 100 pen Venezuelan bitumen at ~ 130 °C and stirred. The purpose of these samples was to provide a comparison with a binder containing inert filler rather than active materials such as OPC and lime. The initial particle sizes of all three additives were similar (ie < 75 μm) but on addition to emulsions, some dissolution of the OPC and lime additives would have resulted in alteration of particle sizes in these additives. All of the samples produced were then used for rheological measurements on a Dynamic shear rheometer.

Rheological measurements

Samples of bitumens and residues recovered from emulsions were placed on the lower plate at a temperature of 20 °C using a spatula. The temperature of the lower plate was raised to 60 °C to soften the bitumen enough to allow it to be squeezed out to form a film of a pre-determined thickness between the two plates, on lowering of the upper plate. The sample and plates were brought to a temperature of 20 °C. Measurements were made under an oscillation frequency sweep from 0.1 to 10 Hz with a plate gap of 0.5 mm and a target strain amplitude of 0.01 %.

Results

Results obtained from the tests described above are shown in the graphs in Figures 12-3 to 12-11 below. Figures 12-3 to 12-5 show the effect of OPC, hydrated lime and limestone filler on viscosity. Figures 12-6 to 12-8 and Figures 12-9 to 12-11 show the effect on the elastic (G') and viscous (G") properties respectively. It is important to note that the test temperature was 20 °C in all tests.

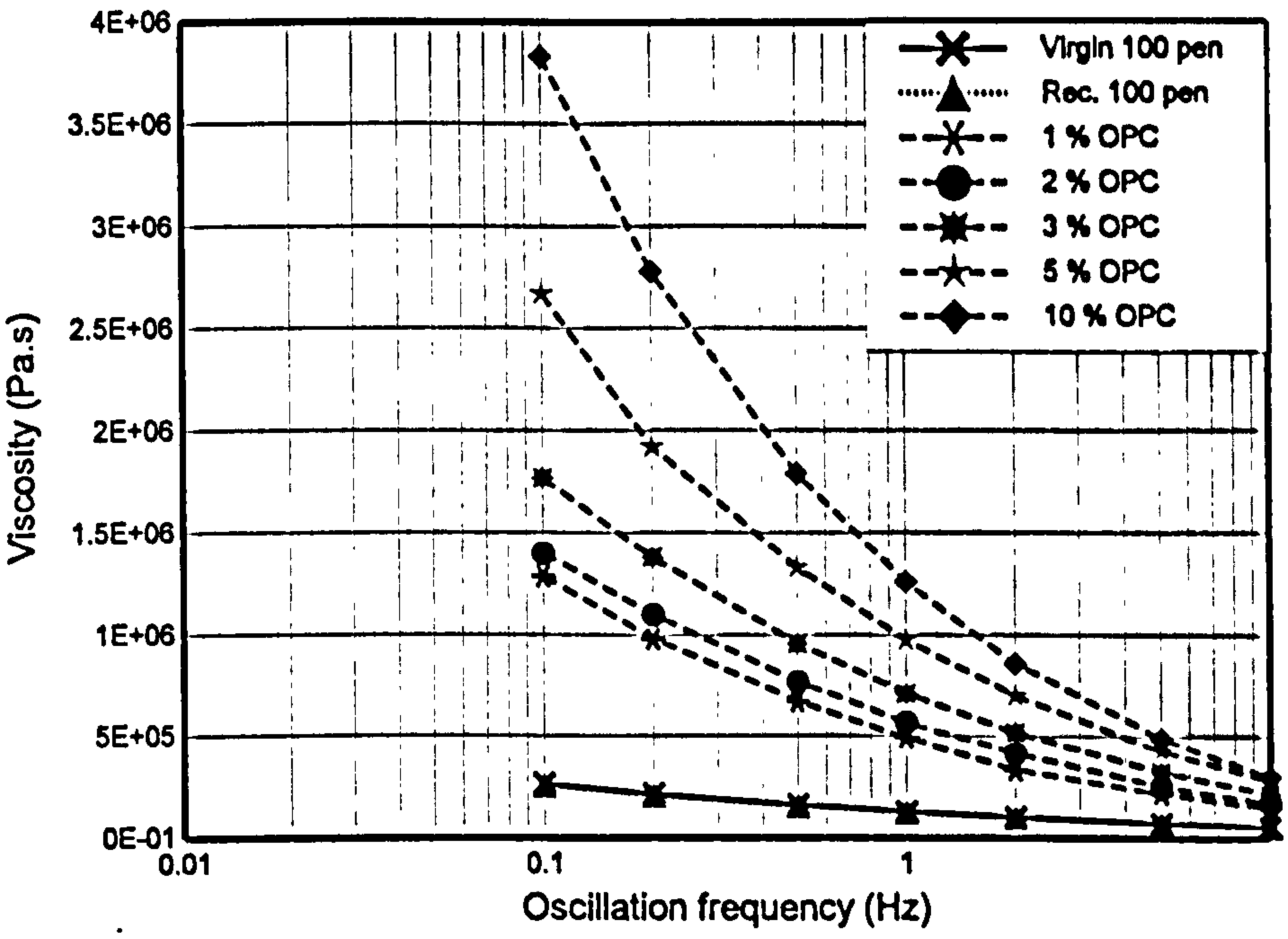


Figure 12-3 : Viscosity of bitumen and emulsion residues with different levels of OPC

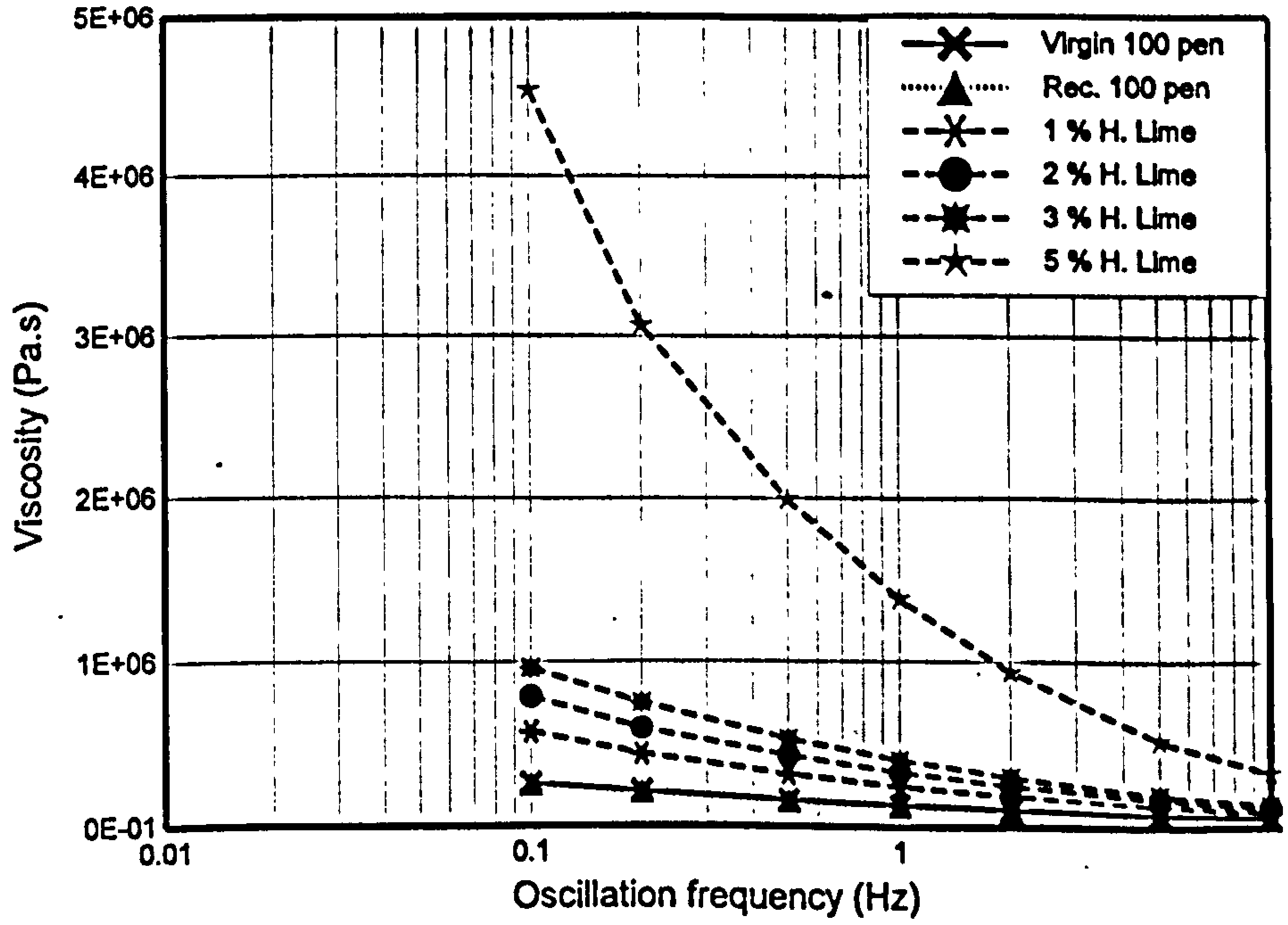


Figure 12-4 : Viscosity of bitumen and emulsion residues with different levels of hydrated lime

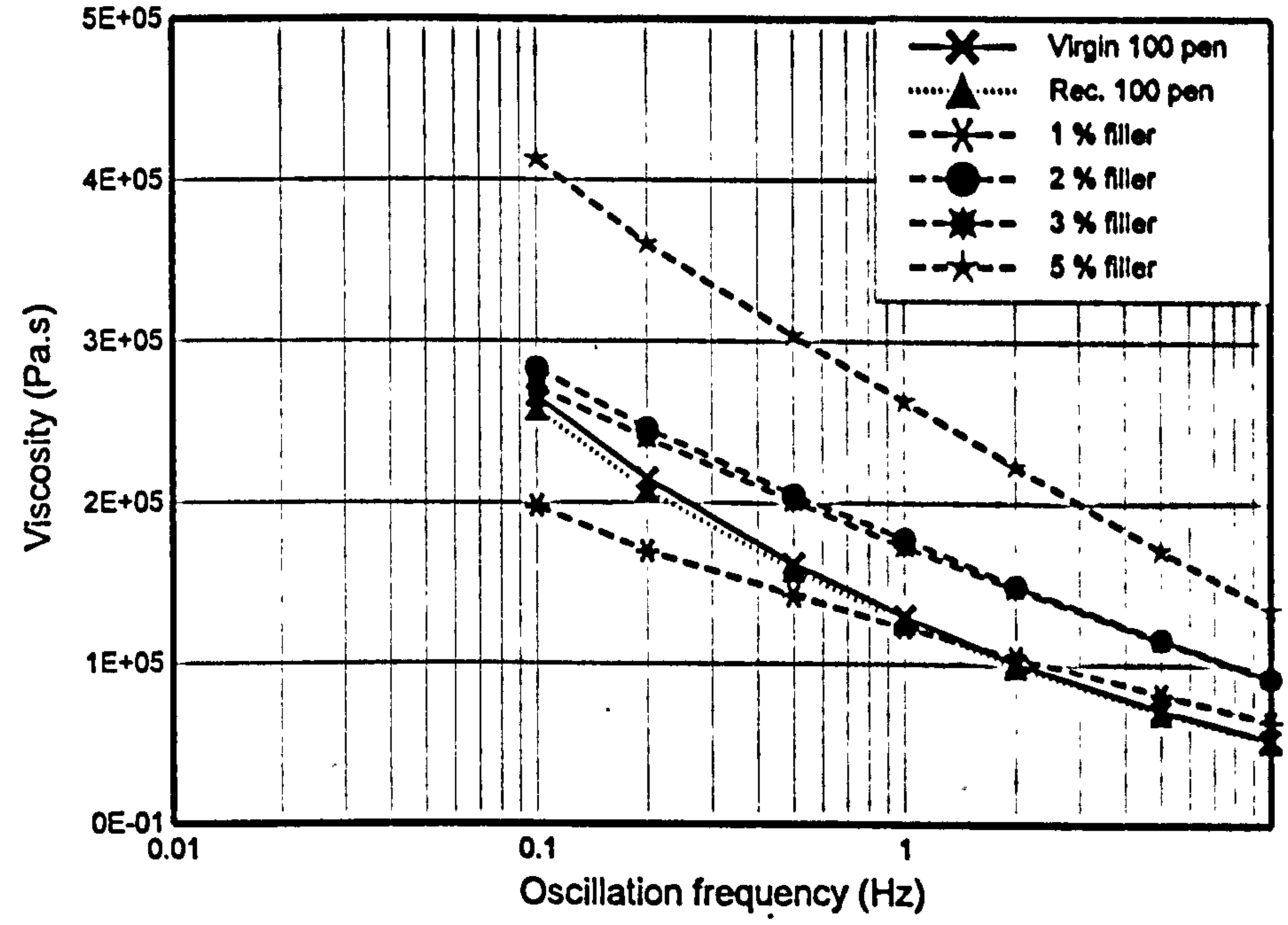


Figure 12-5 : Viscosity of bitumen and emulsion residues with different levels of limestone filler

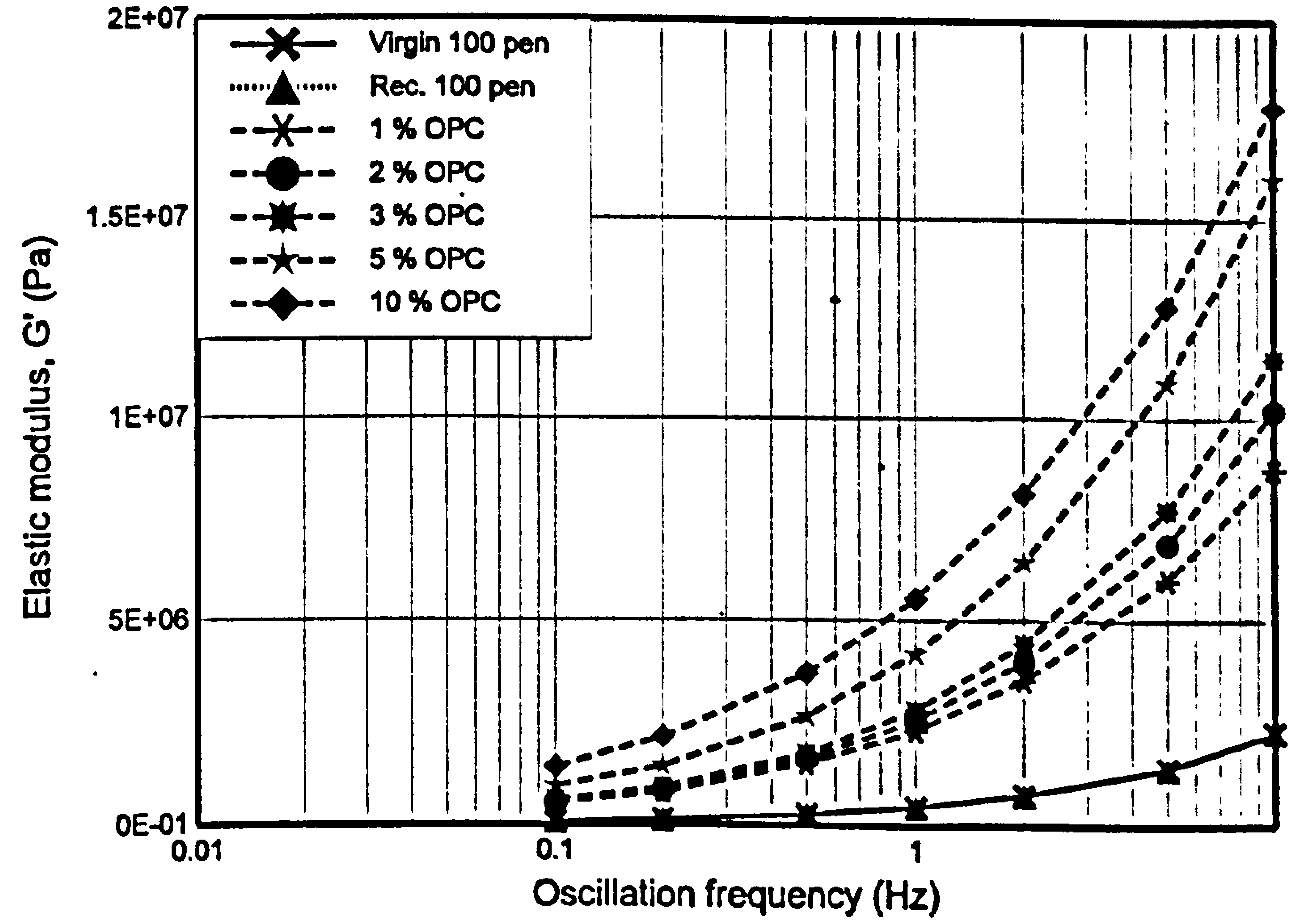


Figure 12-6 : Elastic modulus (G') of emulsion residues with different levels of OPC

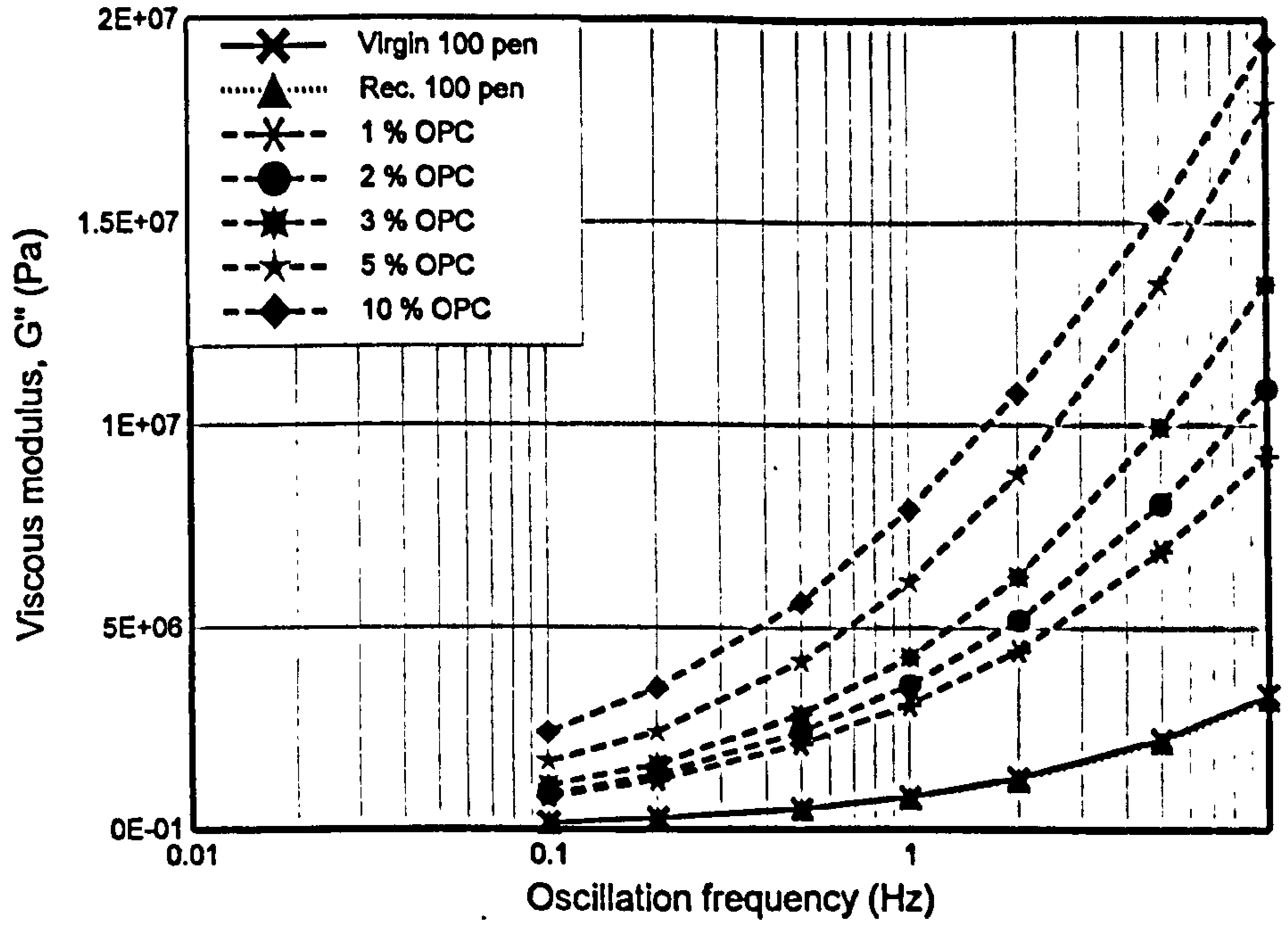


Figure 12-7 : Viscous modulus (G") of emulsion residues with different levels of OPC

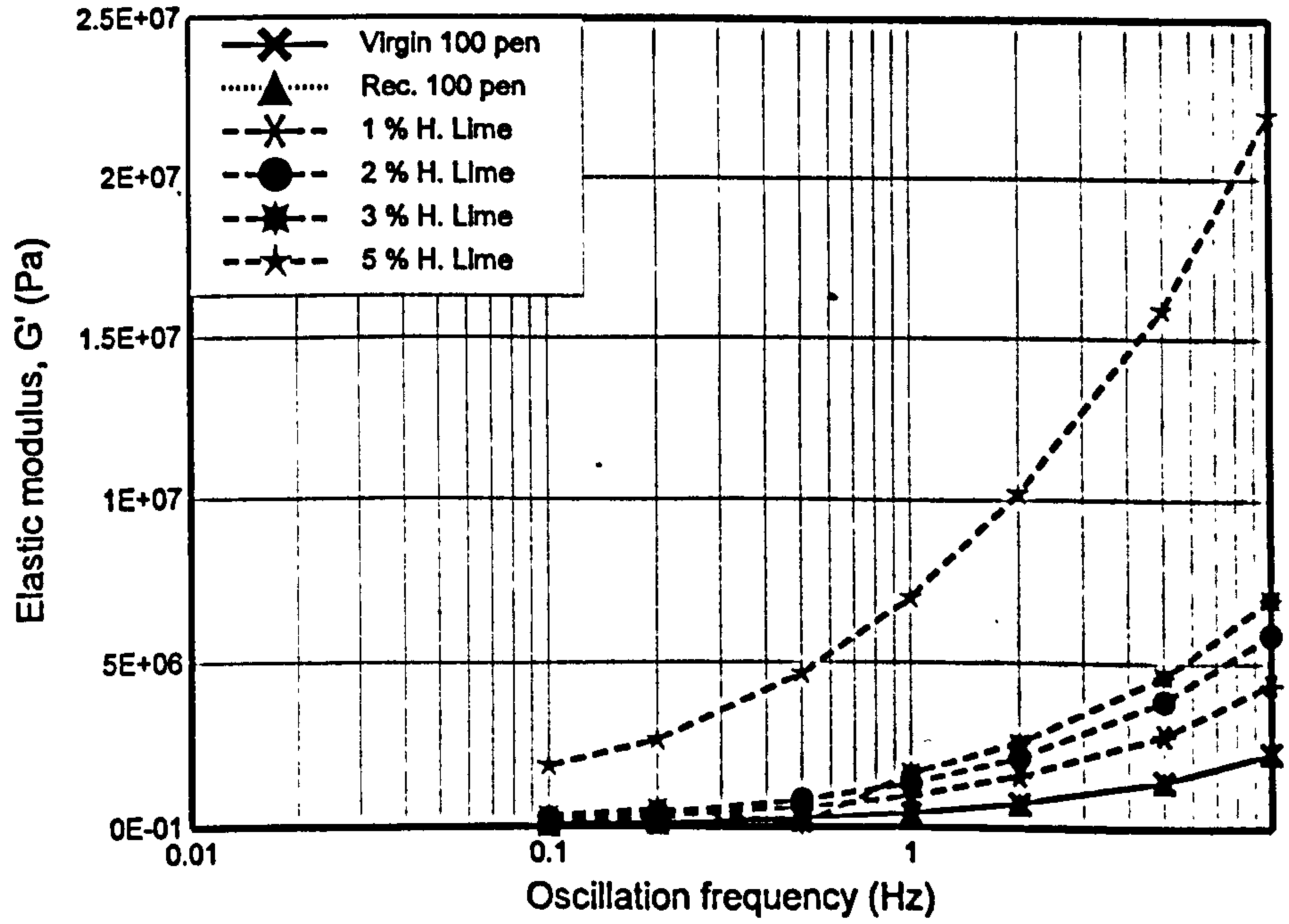


Figure 12-8 : Elastic modulus (G') of emulsion residues with different levels of hydrated lime

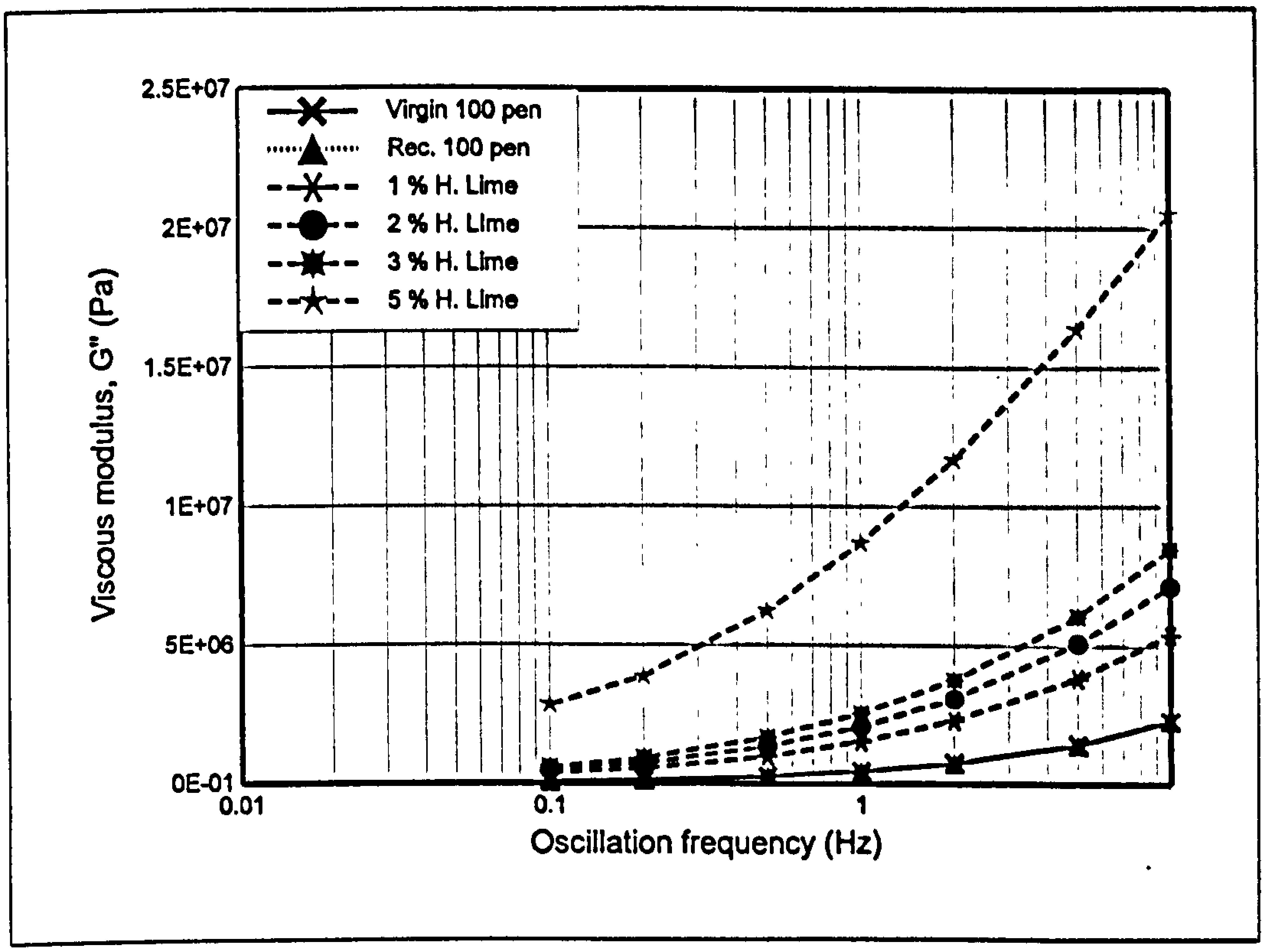


Figure 12-9 : Viscous modulus (G") of emulsion residues with different levels of hydrated lime

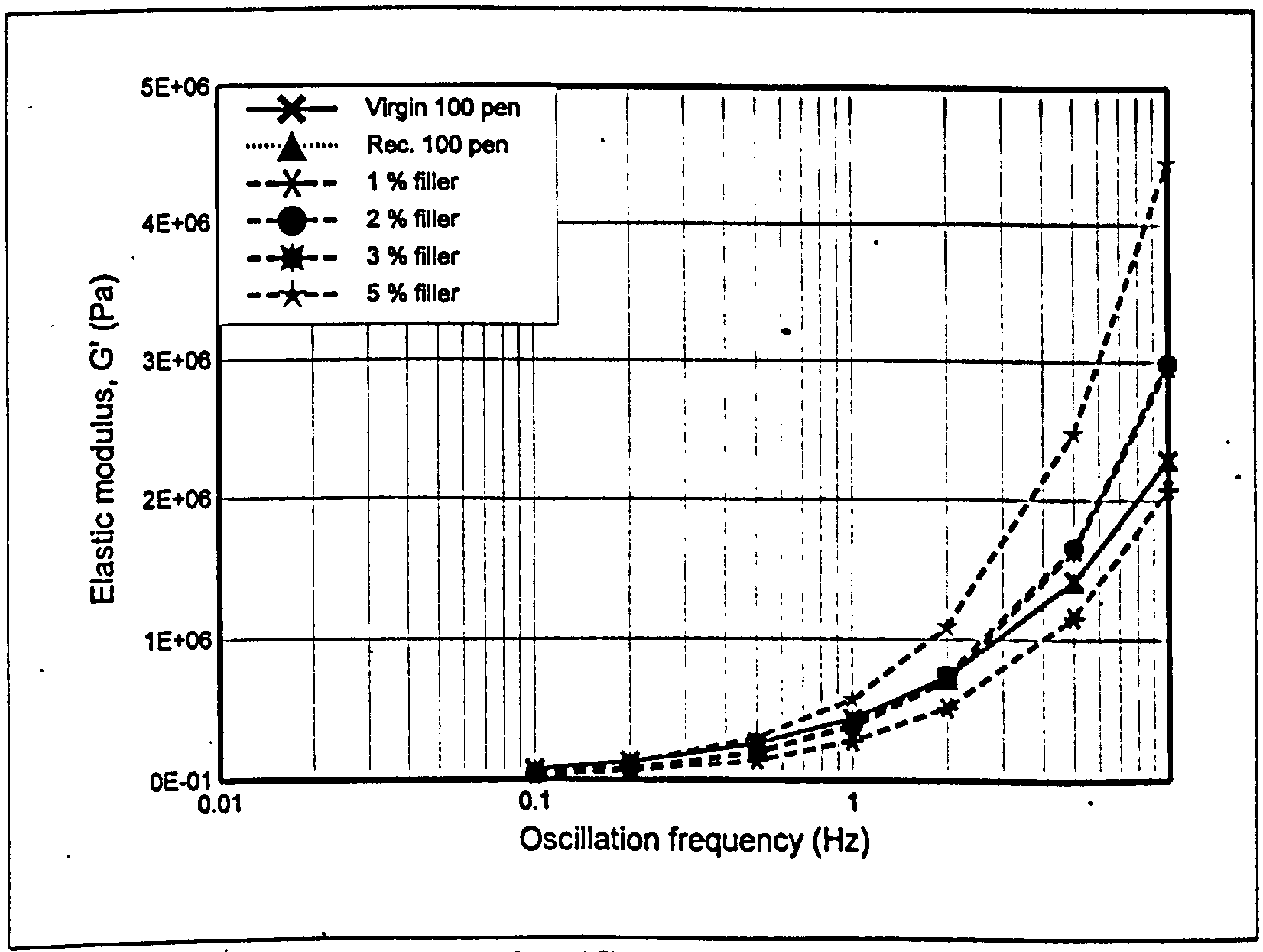


Figure 12-10 : Elastic modulus (G') of emulsion residues with different levels of limestone filler

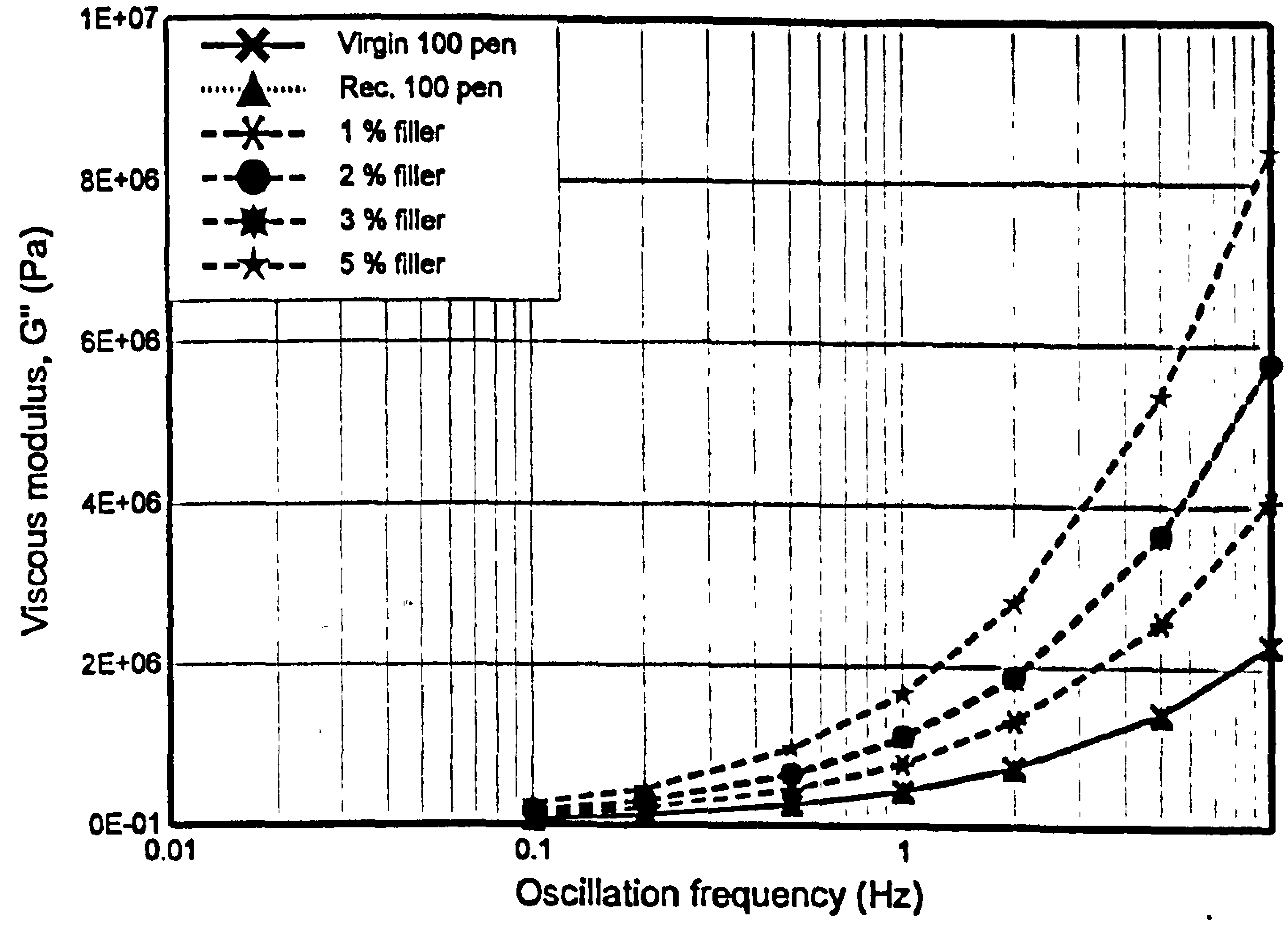


Figure 12-11 : Viscous modulus (G") of emulsion residues with different levels of limestone filler

In order to illustrate more clearly the effect of OPC, hydrated lime and limestone filler on viscosity, elastic modulus and viscous modulus, the data are shown in a different format with respect to additive levels in Figures 12-12 to 12-14. In these graphs, the values measured for the 1 Hz oscillation frequency only are plotted.

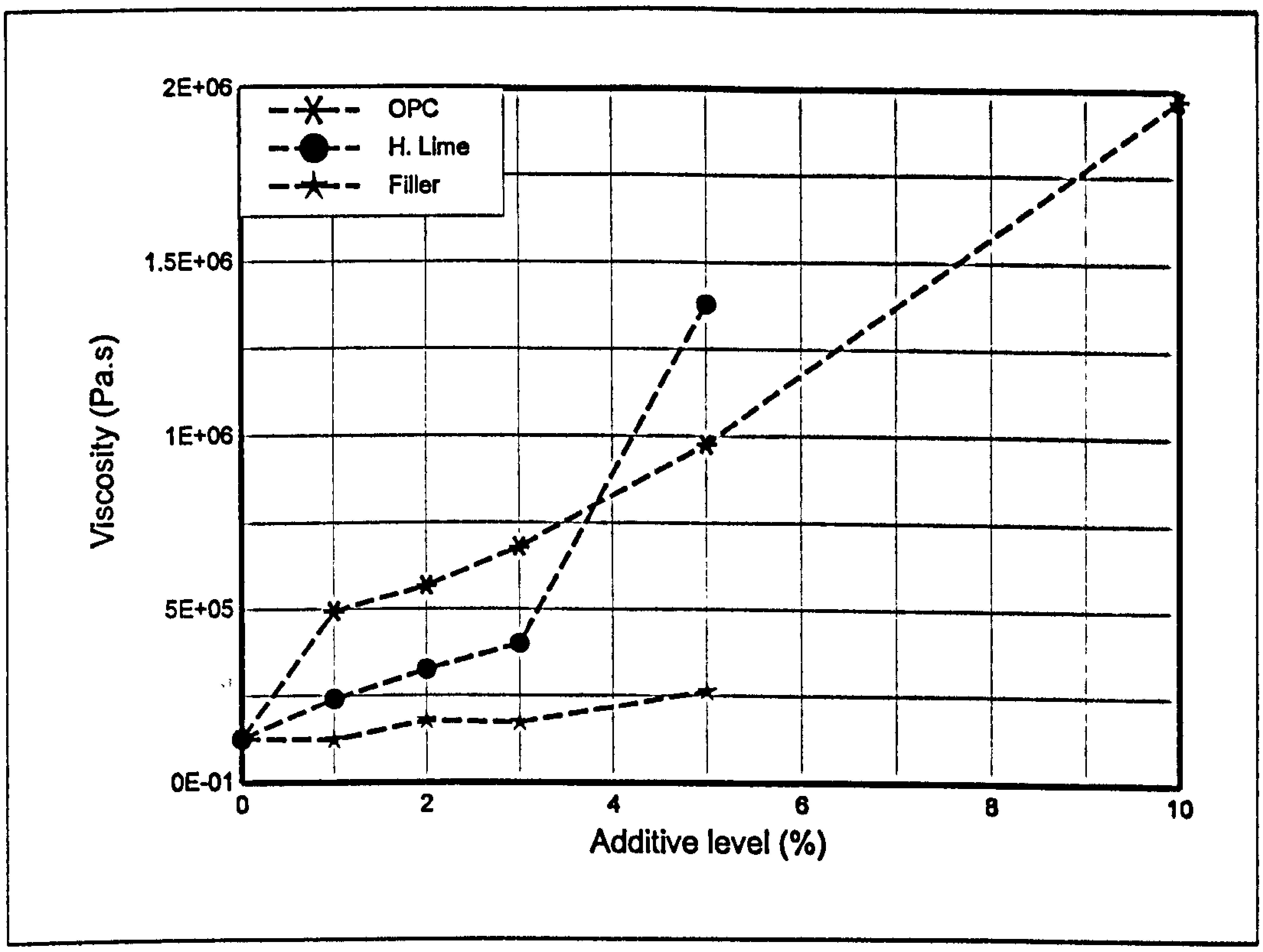


Figure 12-12 : Viscosity of emulsion residues v additive level for 1 Hz oscillation frequency

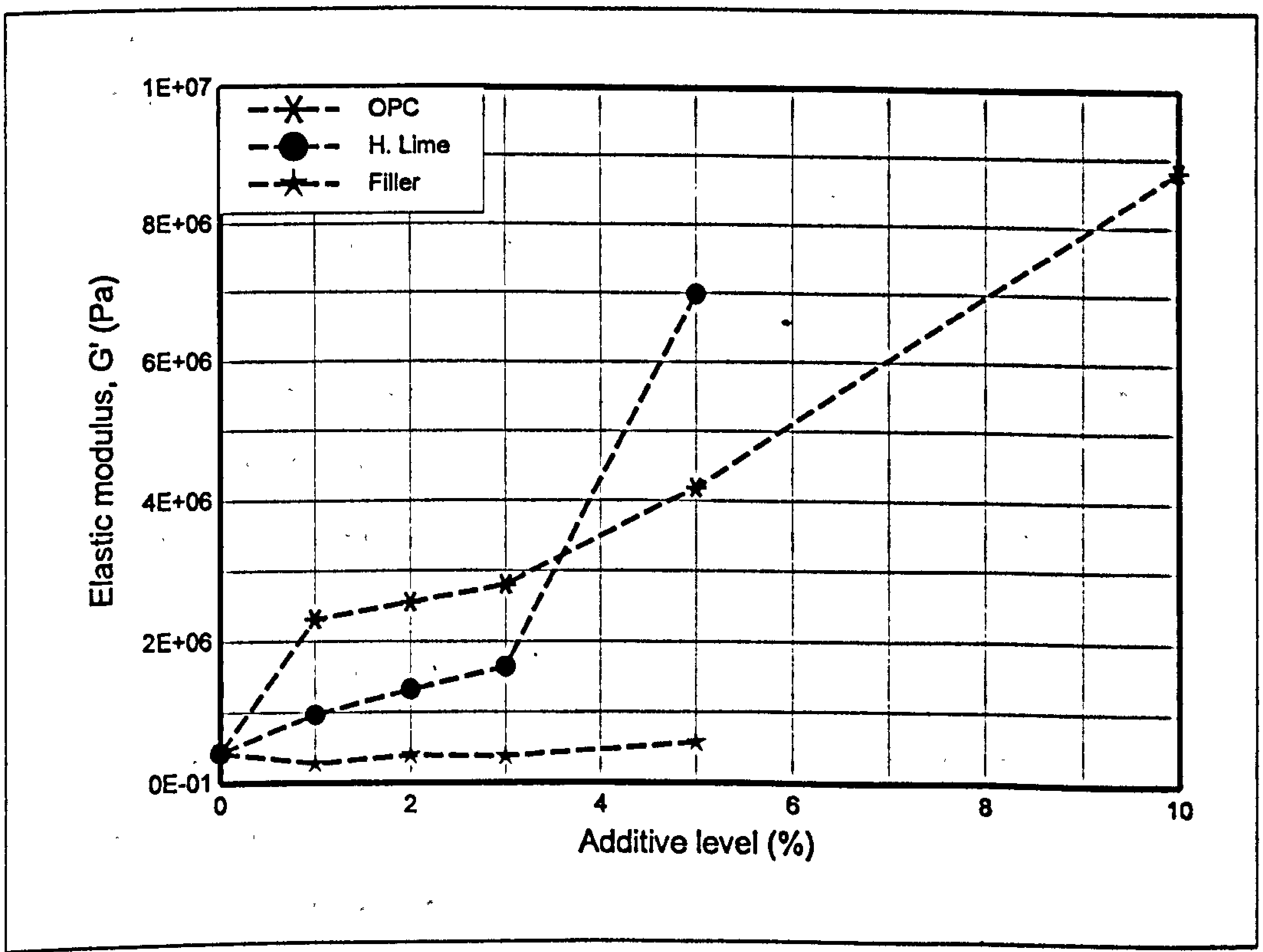


Figure 12-13 : Elastic modulus (G') of emulsion residues v additive level for 1 Hz oscillation frequency

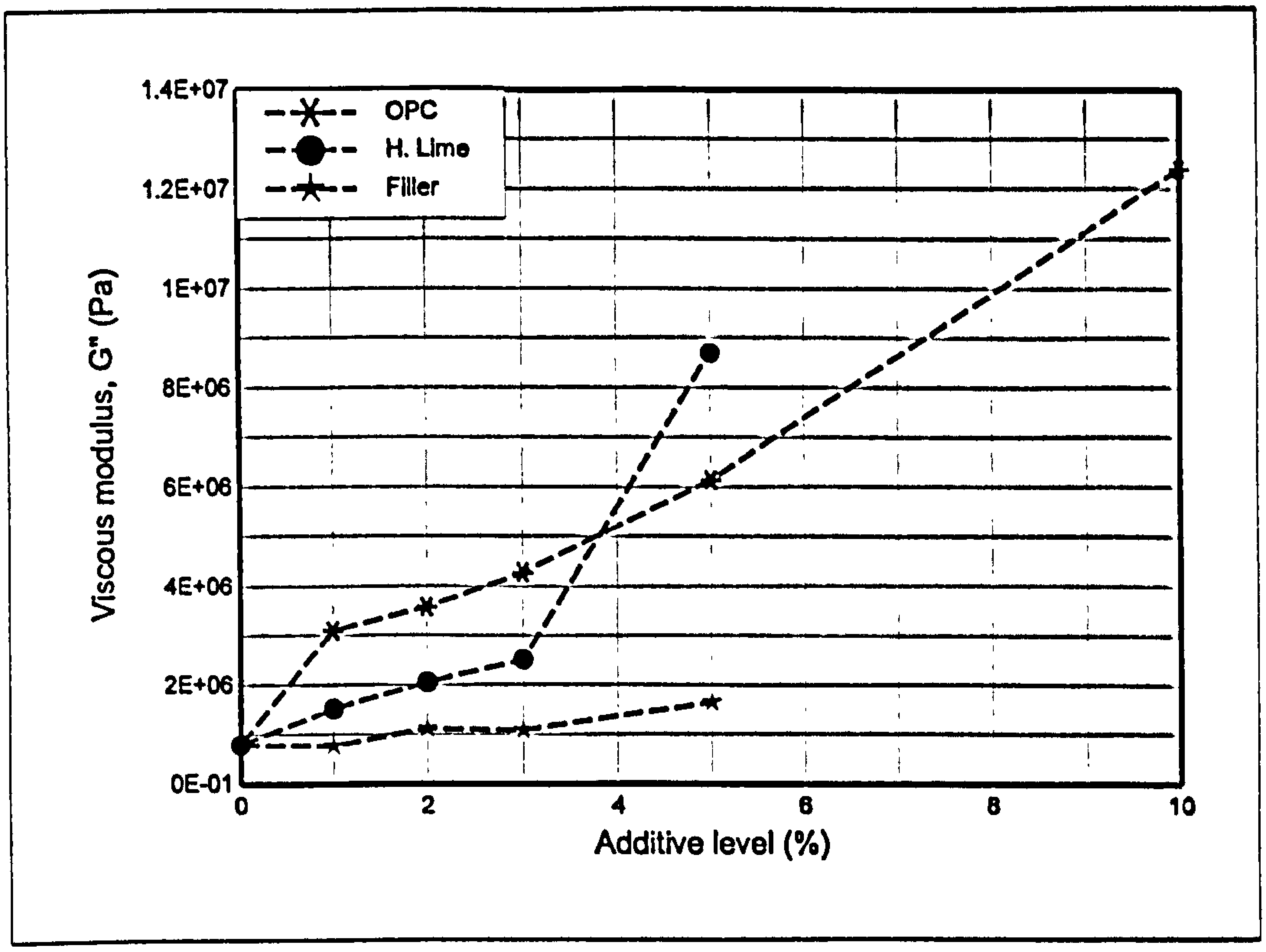


Figure 12-14 : Viscous modulus (G") of emulsion residues v additive level for 1 Hz oscillation frequency

Discussion

The results plotted in the above graphs reveal a number of interesting features regarding the rheological effects of OPC, hydrated lime and limestone filler on bitumen and bitumen emulsion residues.

- Results of measurements on virgin 100 pen bitumen and the same binder recovered from an emulsion without modification showed that the emulsification process itself had no effect on the rheological properties of the binder.
- All of the graphs in Figures 12-5 to 12-11 show that binder viscosities decreased with increasing oscillation frequency whereas elastic and viscous moduli increased. This behaviour is typical of visco elastic materials such as bitumen.
- OPC and hydrated lime both increased the viscosity of the recovered binders with OPC having the largest effect up to the 3 % addition level.

Above 3 %, lime had a greater effect than OPC. It is unclear exactly why the switch occurred but the following theory is suggested. Hydrated lime has a much larger volume than OPC having a lower bulk density. It may be that a point was reached at which the lime contributed such a large volume to the binder that it became the predominant component in the binder / lime mixture thereby having a dramatic effect on viscosity. This situation could be envisaged as a paste in which the lime solids were merely partially coated with bitumen rather than being fully dispersed. Or, it may have been that proper mixing was not achieved in the blend with 5 % lime.

- Limestone filler had a very limited effect on viscosity compared with OPC and lime. Mixtures with filler were produced to provide a comparison between the active ingredients OPC and lime and an inert compound. If the effects of OPC and lime were merely due to a filler effect then filler itself should result in the same effects. This was not found to be the case and, therefore, it would appear that OPC and lime do something which filler alone cannot.
- From Figures 12-12, 12-13 and 12-14 it can be seen that the effects of OPC, lime and filler on viscosity, elastic modulus and viscous modulus all followed the same trend. This indicates that all of these additives affected viscous and elastic properties to the same degree.

12.2.4 Temperature rise in emulsion and OPC mixtures

It is well known that the hydration reaction of OPC can give rise to a noticeable temperature rise in concrete mixtures (126). Different grades of cement are available which give rise to more or less evolution of heat as thermal effects can be either useful or detrimental to concrete mixtures. The effect of heat of cement hydration on bitumen emulsion mixtures may also be significant. A rise in temperature would increase the rate of reaction between aggregate and emulsion and, if large, could increase the rate of water evaporation from a mixture. Both of these effects could increase the breaking rate of the emulsion and the curing rate of the mixture. Due to the acidity of some emulsions, such as the one used as the standard in these studies, the cement hydration reaction may be even more rapid and exothermic.

A test was carried out to measure the temperature rise which occurs during the reaction between cement and the acidic bitumen emulsion in the absence of aggregate. A mixture of standard Redicote E-4868 emulsion and 10 % OPC was produced and placed in an insulated beaker. The change in temperature was monitored over a period of time by means of a sensitive mercury thermometer. pH was also monitored in an attempt to follow the progress of the reaction. The results of these measurements are shown in Table 12-4.

It can be seen that the change in temperature over the 1 hour measurement period was very small rising from 22.2 °C initially to a maximum of only 25 °C after 30 minutes. The pH was seen to jump from 3.47 before the addition of OPC to 12.25 immediately after and then rose very slightly to 12.62 and stabilized at this level. These results suggests that the reaction was over after 30 minutes and that it did not result in a significant temperature rise, large enough to affect the curing rate of an emulsion mixture.

Table 12-4 : Change in temperature and pH of emulsion/cement mixture over time

Time	Temperature (°C)	pH
Before cement addition	22.2	3.47
0 s	22.4	12.25
30 s	22.4	12.4
60 s	22.5	12.5
2 min	22.7	12.58
5 min	22.9	12.61
10 min	23.2	12.62
20 min	24.1	12.62
30 min	25	12.62
60 min	24	12.62

12.2.5 Cement and water mixtures

Emulsion mixtures basically consist of aggregate, water and bitumen emulsion, and sometimes additives such as cement. As the emulsion, added at a level of ~8 %, contains around 38% water, and pre-wet water is added at a level of 2 - 3 %, the total amount of water in a mix is about 5 to 6% by weight on the aggregate. When cement is added to the mixture, at a level of 1 or 2% by weight on the aggregate, the ratio of cement to water is very high at 1 : 6 to 2 : 6. According to the literature (126), cement is able to absorb only 20 to 25% of its own weight in water. There is, therefore, a large excess of water in an emulsion mix, with respect to the cement. The following tests were carried out to verify the 25% limit, and to see what kind of product is obtained at higher ratios of cement to water.

Ordinary Portland Cement was added to 100 g portions of water in increasing amounts. The mixtures were stirred for 30 s and then sealed. They were inspected after intervals of 2 hours, 24 hours and 1 week. After 1 week, the cement and water phases had separated in all cases, resulting in either fully set cement or a cementitious precipitate. The water was poured off, weighed and its pH measured. The results of these tests are shown in Table 12-5.

Table 12-5 : Behaviour of cement and water mixtures

Cement	Water	Mixture appearance		
		Initial	After 2 hrs	After 24 hrs
1	6	Thin dispersion	Ppt. in water	Soft ppt., xs water
1	5	"	"	"
1	4	"	"	"
1	3	"	"	"
1	2	"	"	"
1	1	"	Thin paste, xs water	Solid, xs water
2	1	Slurry	"	"
3	1	Paste	Gel, no xs water	Solid, no xs water
4	1	v. thick paste	Solid	-
5	1	Unmixable	-	-
6	1	-	-	-

The results in Table 12-5 show that OPC was able to fully absorb a maximum of 33 % its own weight in water. At a ratio of 1:1 OPC to water, the cement was still able to set and excess water separated out. Above this ratio the cement was unable to set properly and merely formed a soft precipitate. As discussed above, in a real mixture the ratio of cement to water is 1:6 if 1 % OPC is employed. In this situation, the cement would be unable to set initially and would only do so after a substantial amount of the excess water had evaporated. This is probably the cause for the steady increase in stiffness of mixtures containing OPC, albeit more rapid than mixtures without. In advanced stages of curing when the water content was low, OPC would have been able to set properly and act as a competent binder.

12.2.6 Electron microscopy on mixtures of binder residues and OPC

It has been well established, through the results from tests described above, that Ordinary Portland cement has a beneficial effect on an emulsion mix in a number of ways. The coalescence tests have shown that OPC increases the breaking rate of the bitumen emulsion, which will without doubt increase the curing rate of the mixture. Mechanical properties measurements have shown that OPC increases the curing rate and final stiffness of a mixture. Not only this, cold mix with 1% or more of cement can reach a higher stiffness modulus than an equivalent hot mix. It has been shown that this is not due to a filler effect as hot mix with 1% OPC is little different from that without. Rheological measurements showed that OPC increases the viscosity of the cured mastic. The evidence seems to suggest that the incorporation of cement causes a composite binder to be formed with the curing or cured bituminous binder which has very much improved properties compared with bitumen alone. However, the mechanism is unknown.

The purpose of the electron microscope observations which are described in the following section, was to attempt to resolve the question of how the cement achieves these effects, by looking directly at the cement/bitumen mastic itself. As cement particles are in the region of 5 to 75 μm in size and recrystallized cementitious crystals are in the order of a few microns, and emulsified bitumen droplets average 5 to 10 μm, it was felt that there should be a good chance of observing any features which may evolve following the cure of a cementitious cold mix.

A selection of cores were prepared from emulsion mixtures, with and without OPC, and observed through an electron microscope after a prolonged period of curing to ensure full cement cure was achieved.

Core manufacture and curing

Mixtures were prepared with the standard emulsion and granite aggregate with a mid 20 mm DBM grading. Ordinary Portland Cement was added to the dry aggregate mixture at levels of 0, and 2 %. The cores thus produced were stored at 20°C and ~50 (+/- 5) % relative humidity for several months to allow sufficient time for any composite structures to form. Mixtures of granite aggregate and the cement were also prepared to enable the appearance of normal concrete type mixtures to be seen.

In addition to the full mixtures, blends of emulsion and cement were prepared in the absence of water, as it was suspected that it may be difficult to see any features in the mixtures including aggregate. The proportions of emulsion and cement were such that the ratio of ingredients was the same as that which would be used in a full mixture.

Specimen preparation

After curing, the sample cores and emulsion / cement mastics were crudely broken up and small pieces obtained by breaking segments off by hand. The intention was to produce surfaces showing exposed binder which had not been subjected to any treatment which would alter their appearances. It was felt that processes such as sawing would only serve to smear the bitumen phase thus obscuring many features of interest.

The small specimens were fixed on 1" metal discs by means of an adhesive patch. The mounted samples were coated with a very thin layer of gold in a special piece of apparatus. The gold coating is necessary to provide a conductive surface on the specimen which is essential to attract the incident electron beam in the electron microscope which produces the image. In the coating apparatus, the specimen is placed in the bottom of a vacuum chamber which is purged with Argon which is an inert gas. A gold plate is situated a few centimetres above the sample and is heated which causes gold atoms to evaporate and fall onto the specimen below. The process is known as sputtering. After sputtering a thin coating of gold could be clearly seen on the specimens.

Electron microscopy

The gold coated and mounted specimens were introduced to the vacuum chamber of the electron microscope and observed. The samples were photographed at a range of magnifications from x20 to x1000 or even x3000. At medium magnification, features of interest were picked out and further magnified if this proved beneficial. Thus, a series of micrographs of the mixtures without OPC and with 2 % OPC, the granite and OPC alone, and the bitumen and cement mastics were obtained at a number of magnifications.

In addition to producing magnified images, the electron microscope is able to analyse the elemental composition of selected areas of a sample. This is achieved by the process of X-ray analysis. When the incident electron beam strikes the sample, some of the electrons in low energy orbitals in the atoms are knocked out. The electron structure of the atom is then unstable, and so an electron from a high energy orbital drops down to fill the gap. To do this it must lose energy and this is emitted as X-rays (Figure 12-6). For every type of atom there is a known pattern for the electron readjustment and its subsequent emitted X-ray wavelength. Within the electron microscope there is an X-ray analyzer which can detect X-rays of particular wavelengths and thus identify elements within the sample. Small regions can be picked out and analysed, which enables discreet features to be elementally analysed. It is, therefore, possible to say what a selected feature is composed of from a definitive analysis rather than a visual appraisal. Elemental analysis was carried out during microscopy on the full mixtures to determine which features were aggregate and which were cementitious. It was not done on the emulsion / cement mixtures as all crystalline structures could only be cementitious and the crystal morphologies alone were sufficient to allow identification.

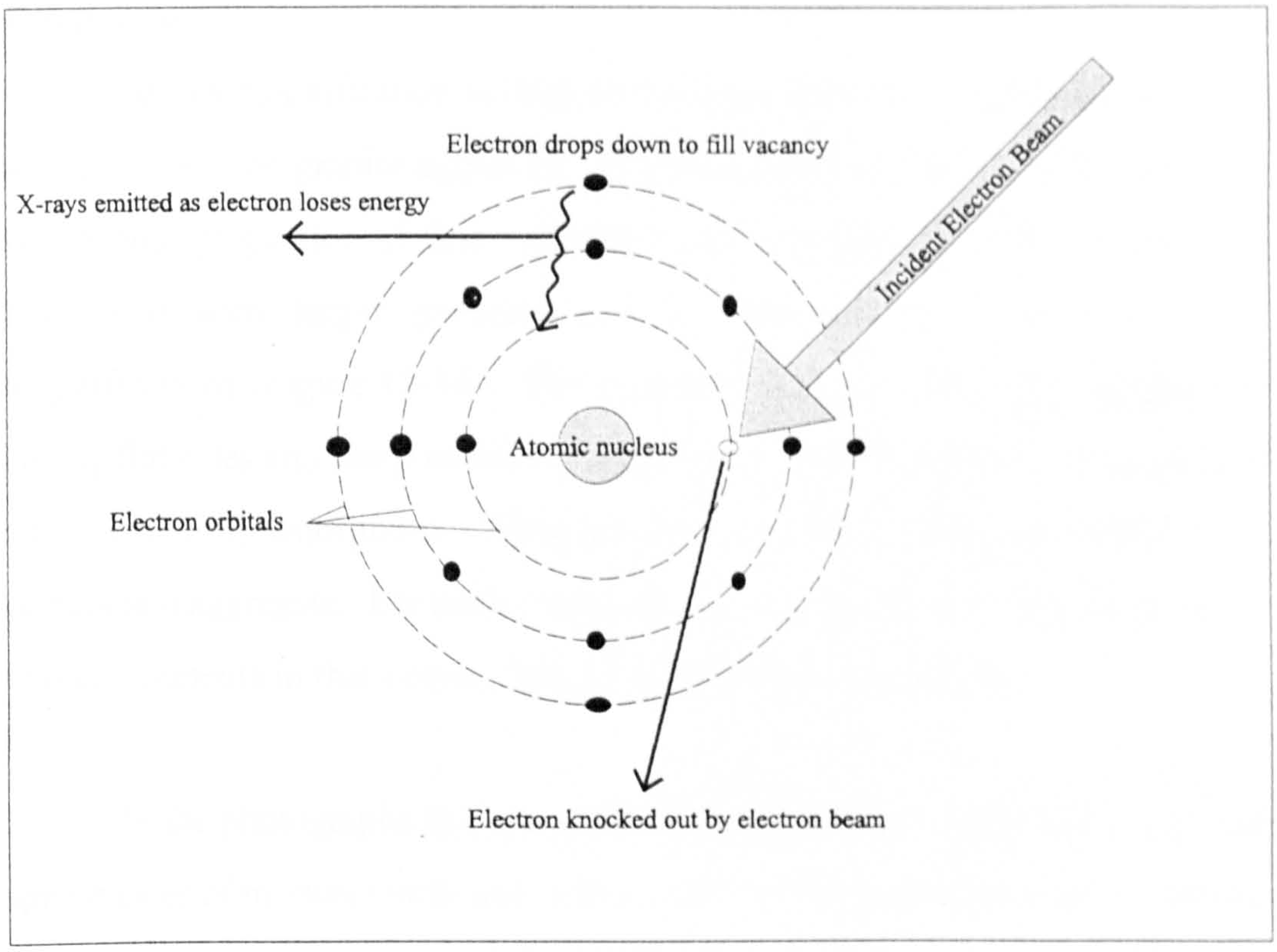


Figure 12-15 : Origin of X-rays arising from electron rearrangement

Results

The electron microscope photographs can be seen in Figures 12-16 to 12-12-24. The first 6 photographs are concerned with the full mixtures, OPC and granite aggregate whereas the latter 3 show the bitumen and cement mastics.

Elemental analysis showed up a clear difference between aggregate and cement in full mixtures with and without cement. The difference was in the calcium content, with cement showing much higher levels than other components. Consequently, the particles shown in pictures in Figures 12-19 and 12-21 were identified as cementitious due to their high calcium content. Additionally, some of the aggregate particles showed high levels of iron and magnesium but this is of no value for the purposes of these studies.

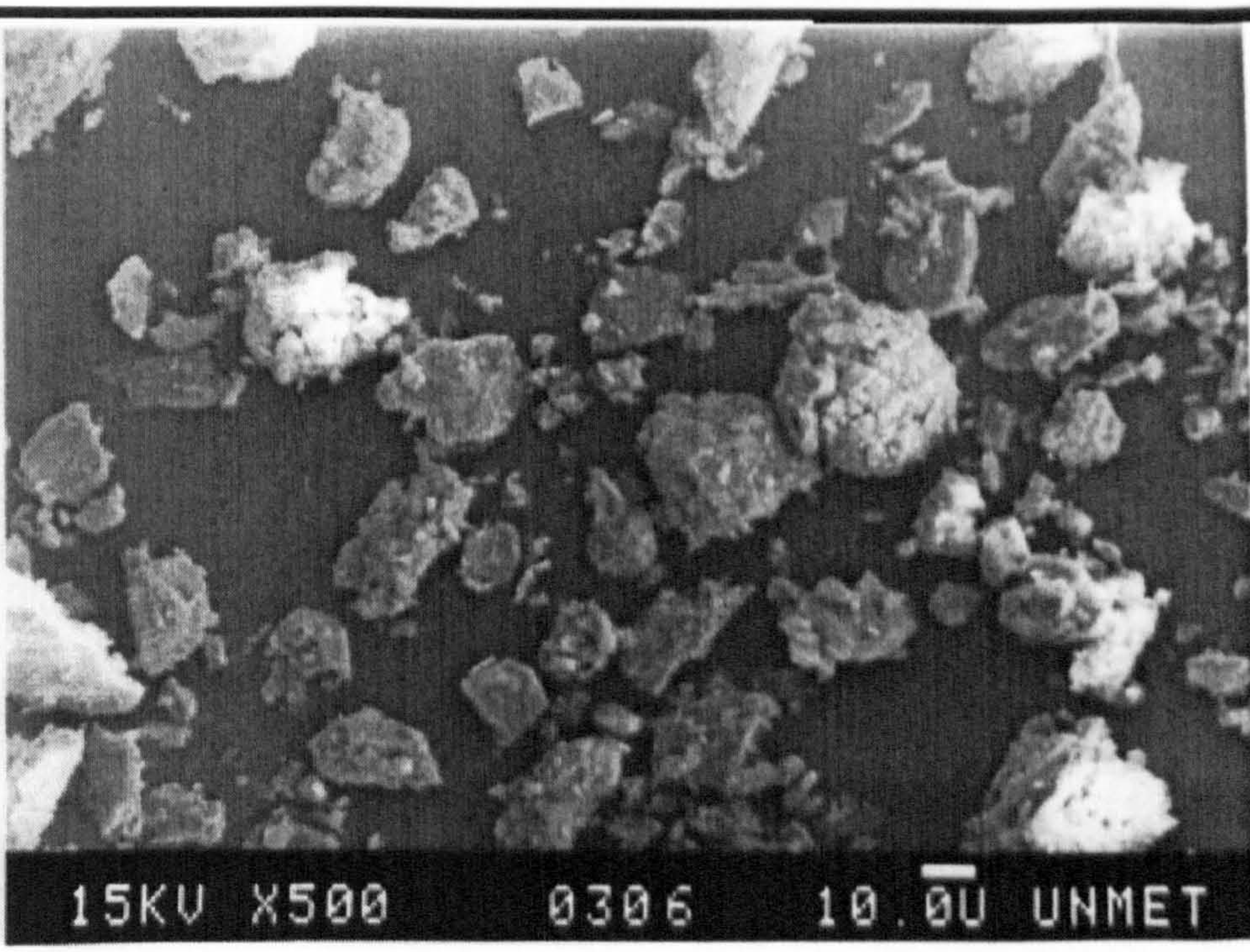
Discussion

At low magnification (x100) the Ordinary Portland Cement appears to be a lot finer than the granite aggregate. Higher magnifications revealed that there was a high proportion of fine material in the aggregate as well, but that it is associated with larger particles and is therefore not obvious at lower magnifications (Figure 12-16). The cement is more crystalline in appearance, having flat sides and sharp corners (Figure 12-17), compared with the aggregate which is mainly amorphous. These are the only visual differences between the cement and aggregate. Elemental analysis showed up definite differences in the two components in that cement has a far higher calcium content.

In the photographs in Figures 12-18 and 12-19, and 12-20 and 12-21, the appearances of mixtures with and without OPC at two magnifications are shown. Discreet cementitious or aggregate particles are not immediately obvious in any of the images. There are, however, definite differences between the two specimens. It is very clear from the x100 pictures that the mixture with 2% cement is much rougher in texture, having a pock marked surface compared with the smooth surface of the mixture with no cement. It is tempting to conclude that this is evidence of gas bubbles which could have been released during the hydration reaction of the cement. At higher magnifications (Figure 12-21), the cement containing mixture has many crystalline features which are not evident in the other specimen, and some definite bubble type artifacts could be seen in images at x1000 magnifications. The elemental analysis results confirm that these are cementitious regions as the calcium contents are very high.

The mix without cement looks as if the entire surface of the inorganic mix constituents are completely covered by a featureless layer of bitumen (Figure 12-20). In contrast, the cementitious mix appears to show a composite binder incorporating bitumen and cement in mutual contact.

A lot more features can be clearly seen in the photographs of the mixtures



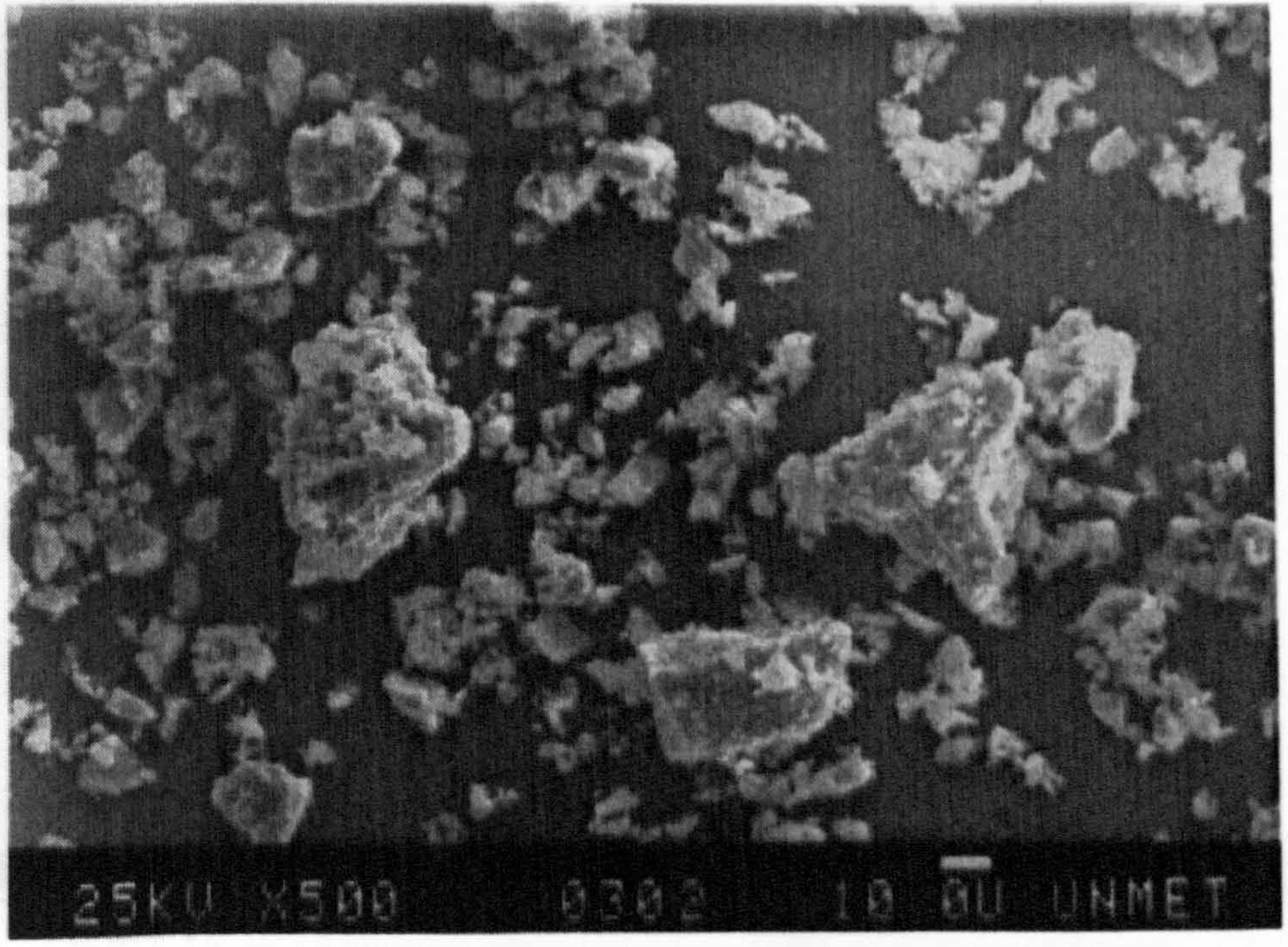
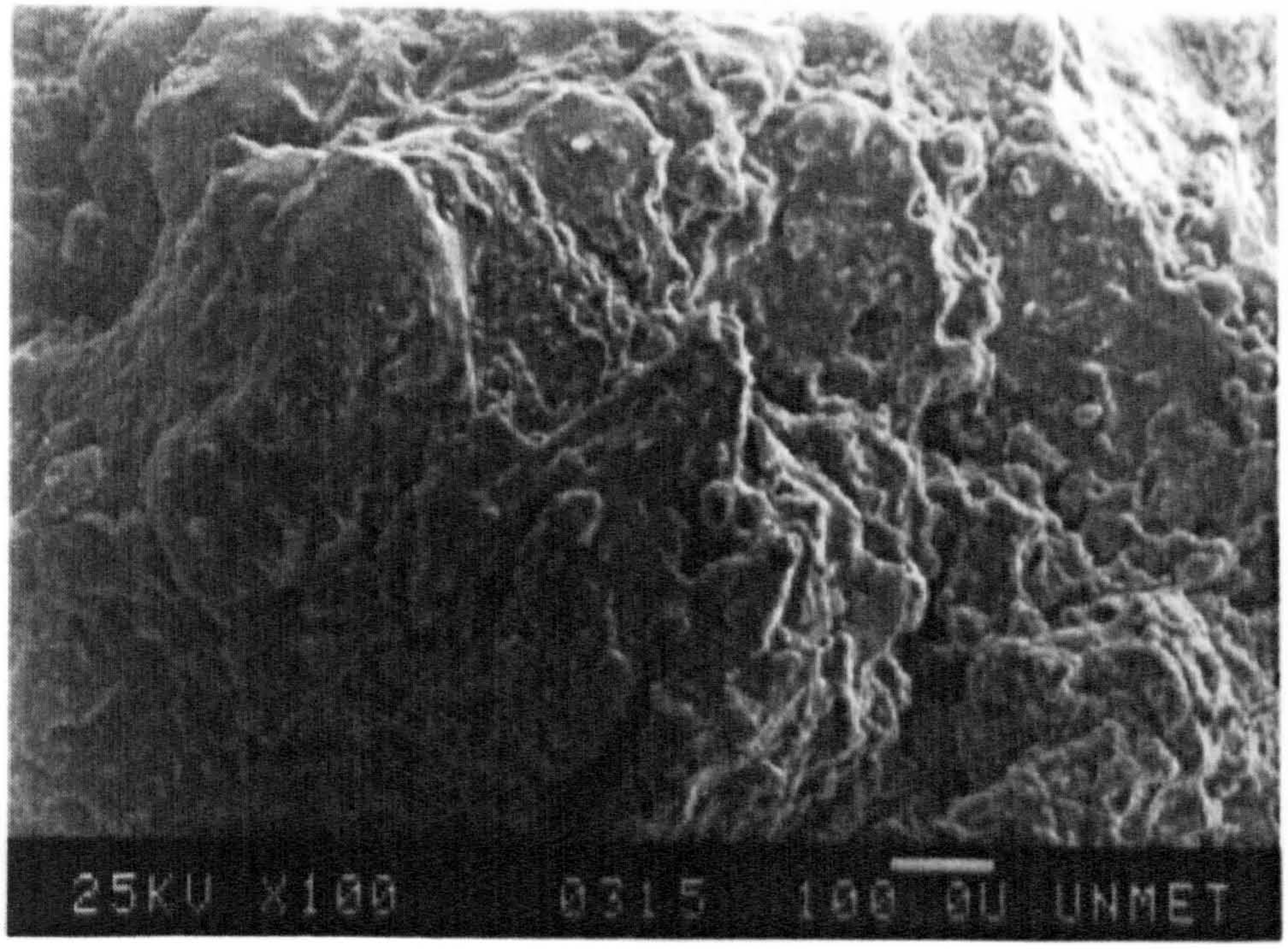


Figure 12-16 : Aggregate grains (top)
Figure 12-17 : Cement grains (bottom)



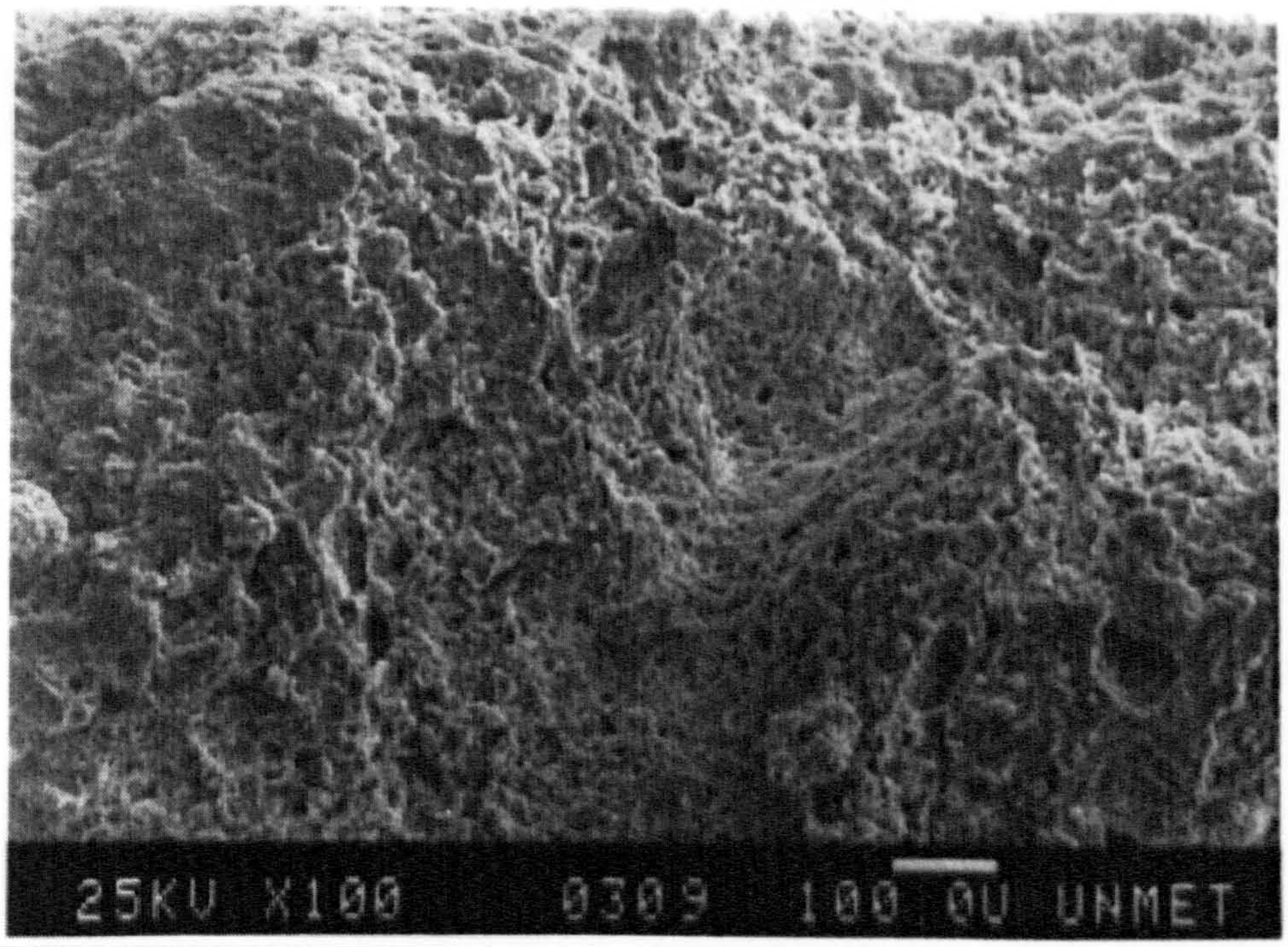
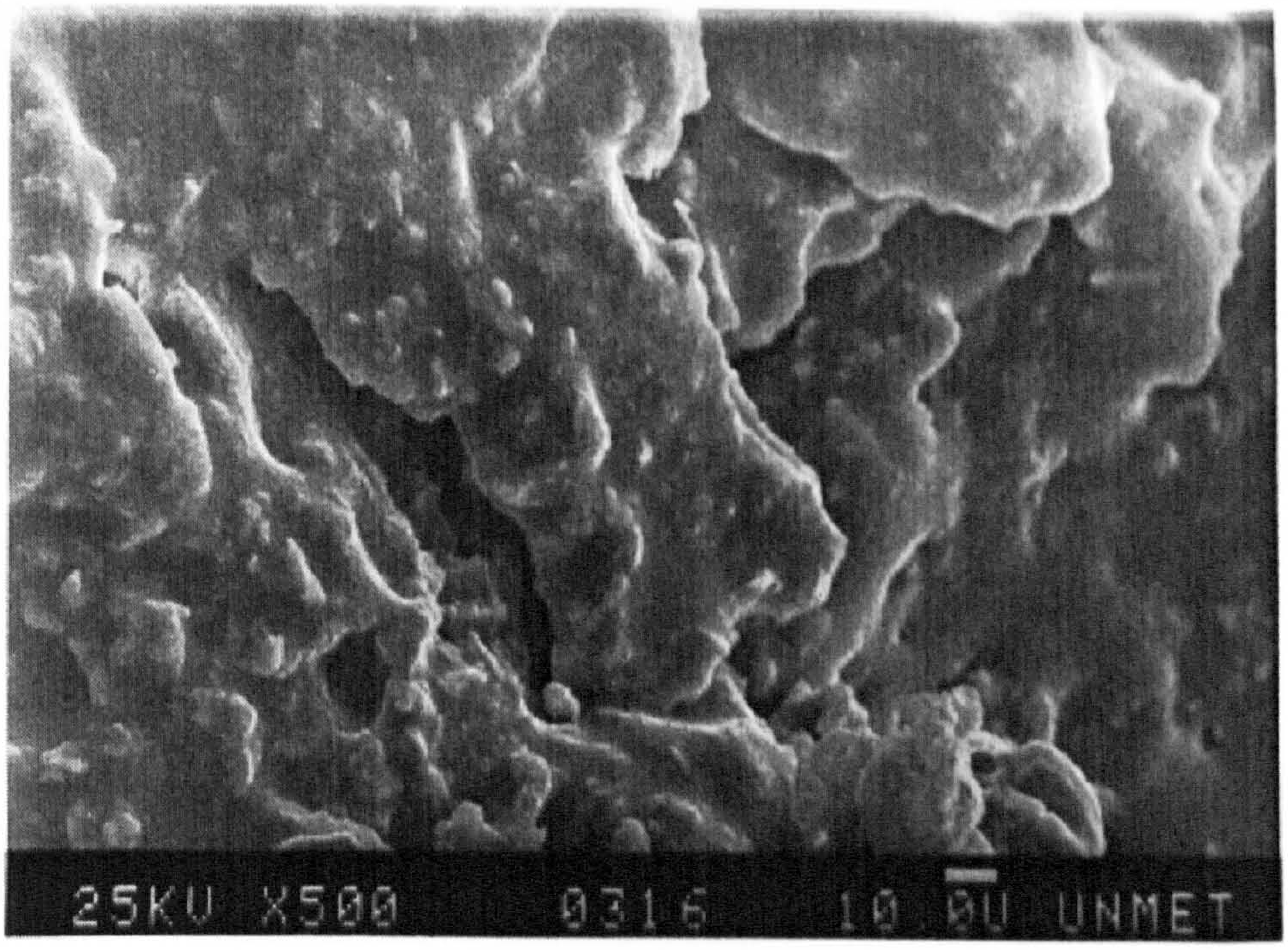


Figure 12-18 : General photo of mix with no OPC (top)
Figure 12-19 : General photo of mix with 2 % OPC (bottom)



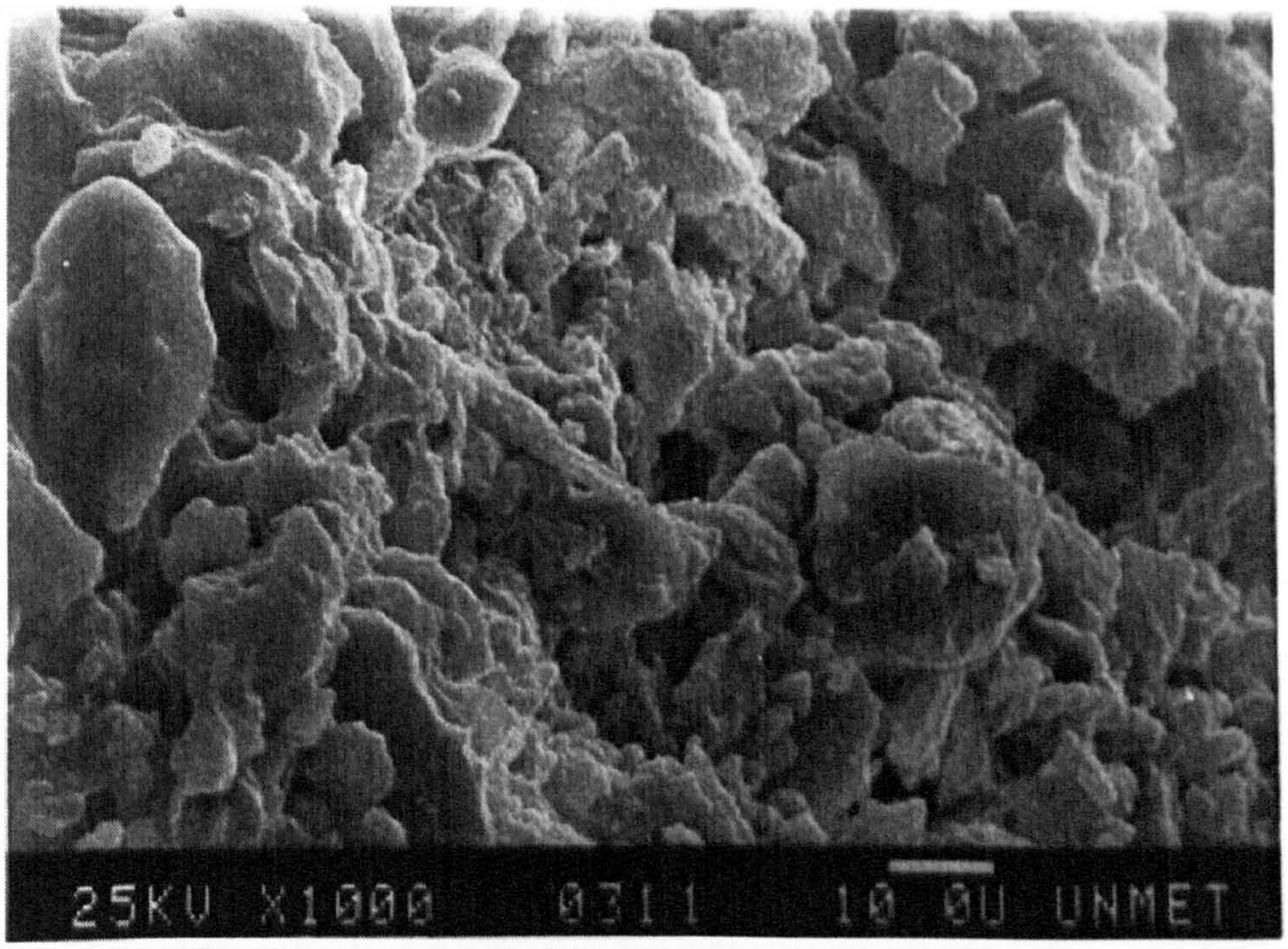
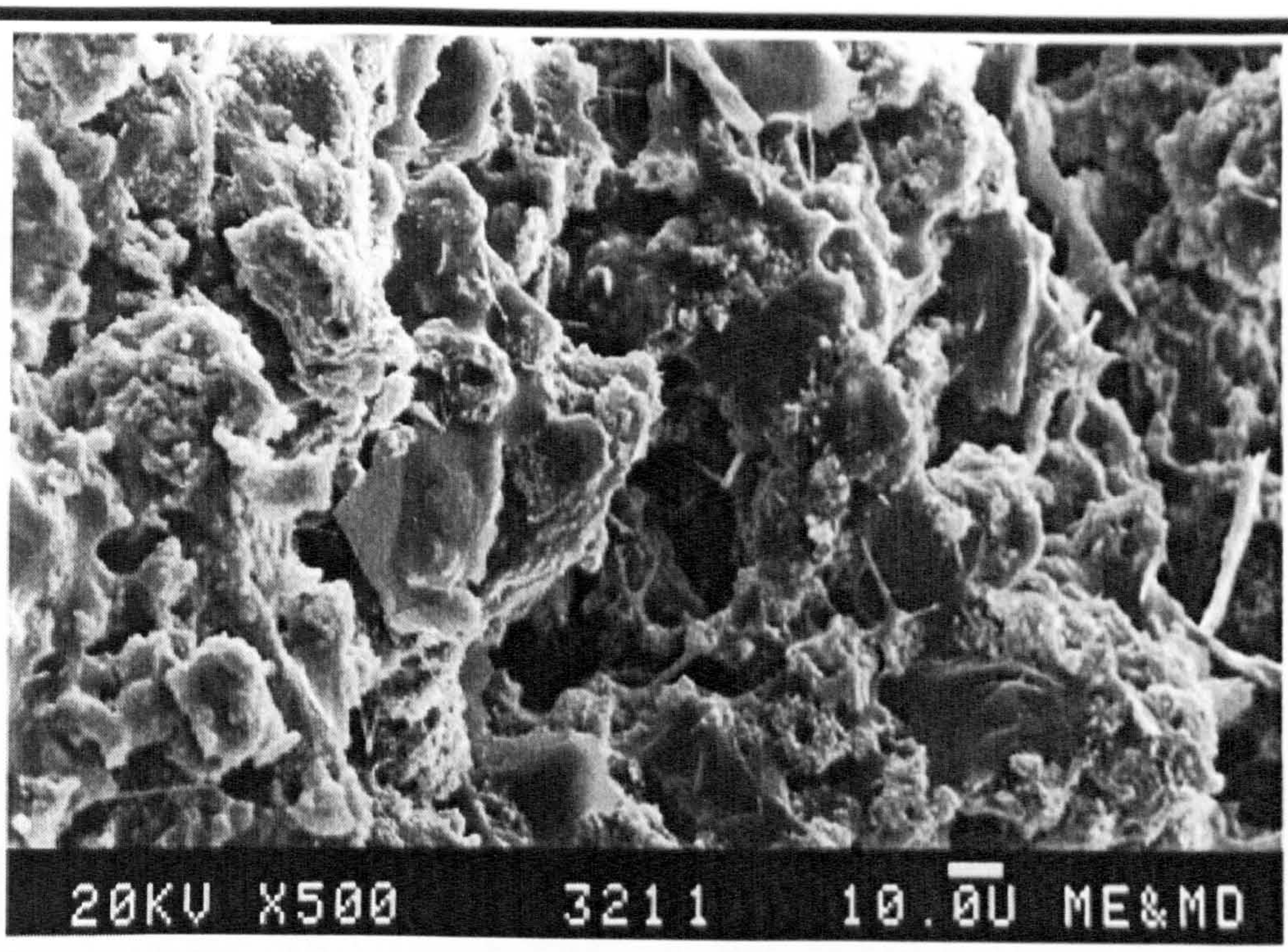


Figure 12-20 : Typical mix with no OPC (top)
Figure 12-21 : Cementitious region (bottom)



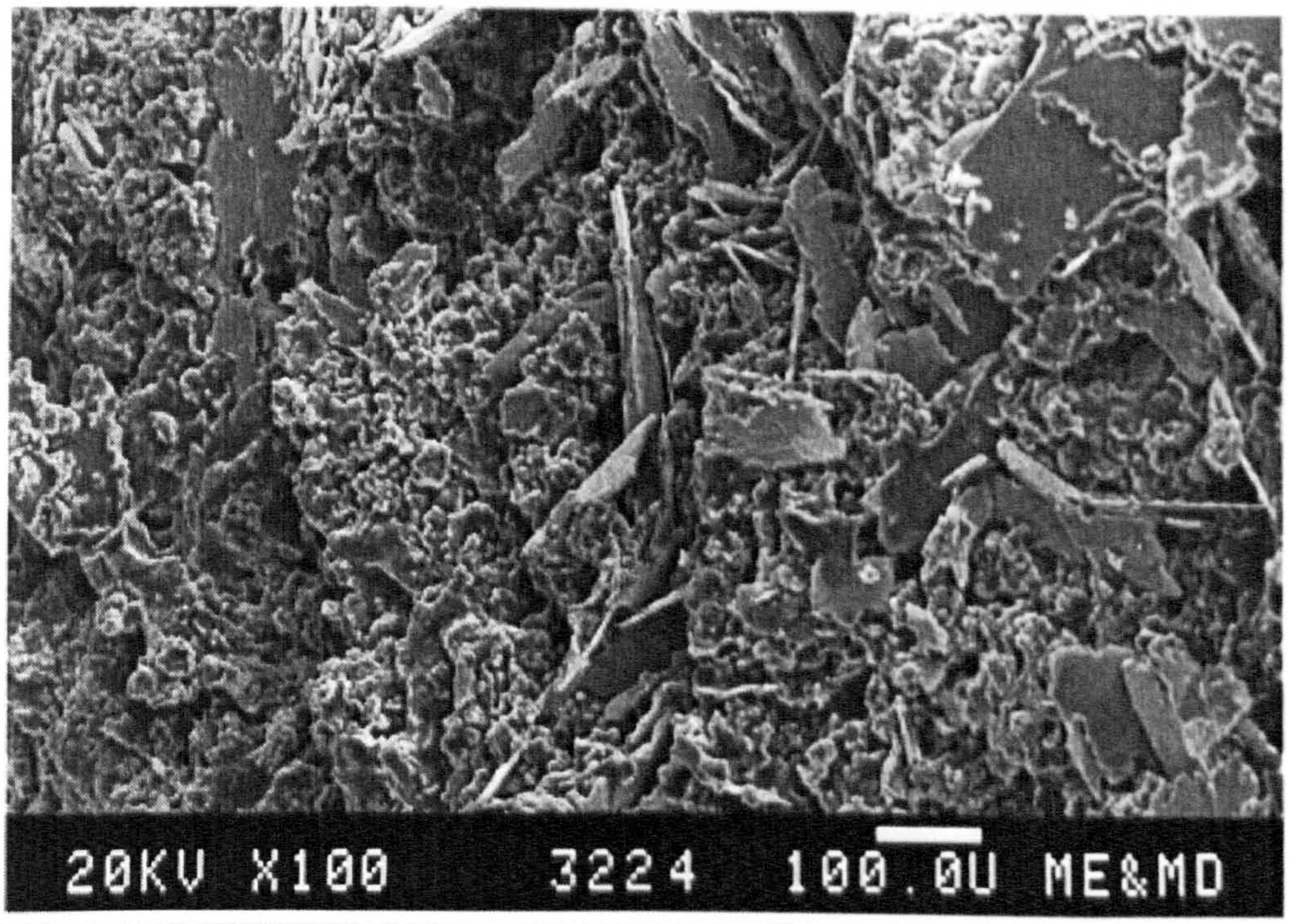


Figure 12-22 : Ettringite crystals (top)
Figure 12-23 : CH crystals (bottom)

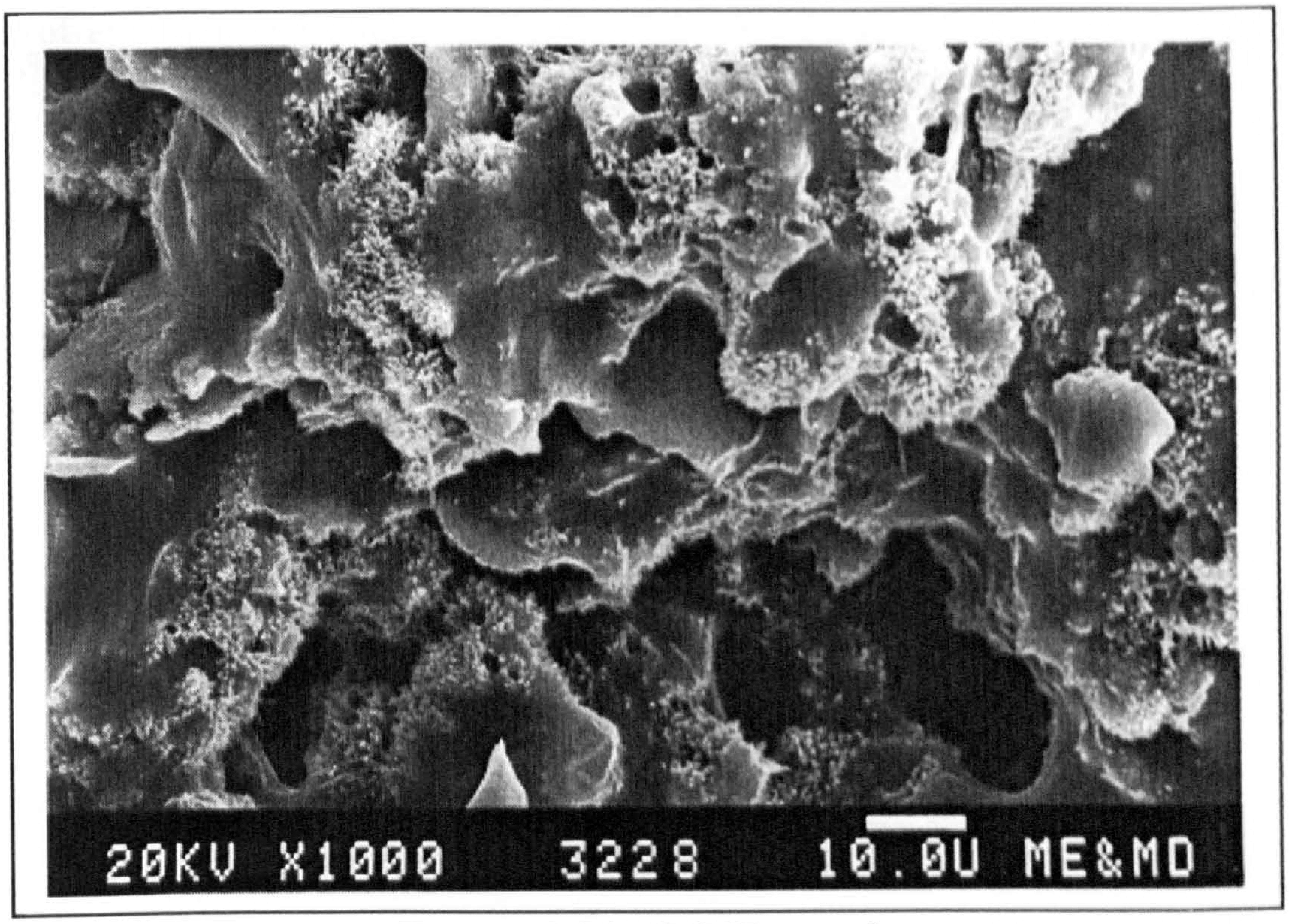


Figure 12-24 : C-S-H crystals

of emulsion and cement only. In these photographs, the smooth areas are certainly bitumen and the different phases of recrystallized cement can also be seen. The photographs were shown to Dr. I.G. Richardson , a concrete specialist from the University of Leeds, for an expert interpretation. Three distinct crystal morphologies are present - rods, needles and plates. The rods are typical of ettringite species (Figure 12-22). The crystal structure is based on columns of composition $Ca_6[Al(OH)_6]_2.H_2O$ with additional water molecules and sulphate ions residing in channels, between which may be substituted an assortment of other ions. No formula is strictly adhered to and ettringite is labelled as AF. The spiky, fibrous elements are hydrated calcium silicate - C-S-H (Figure 12-24). The large plates are calcium hydroxide - C-H (Figure 12-23).

All of these species are to be found in normal hardened concrete, though often the separate crystal morphologies are intergrown obscuring most of the detail. In addition to the crystal groundmass, as it is termed, concrete also normally contains pores which arise mostly from gas bubbles or evaporation of water droplets. Some pores were evident in the mixtures observed here. Most of the evidence of these observations, therefore, seems to suggest that the cement cured in much the same manner as it would in a normal concrete mixture. Thus, it would be expected that the material acts as a binder to some extent. Some of the images also suggest that the cementitious phase is dispersed within the bituminous binder. This could have the additional effect of stiffening the organic binder.

12.3 RHEOLOGICAL MEASUREMENTS ON POLYMER MODIFIED BINDER RESIDUES

ITSM tests reported in chapter 9 of this report showed that EVA had an effect on the stiffness of bitumen emulsion mixtures. The rheological properties of polymer modified binder residues were, therefore, investigated.

12.3.1 Sample preparation

100 g portions of the polymer modified emulsions described in Table 6-2 in chapter 6 were placed in filter funnels. The samples were left for a period of several days, during which time some of the clear emulsion water filtered through. The binder residues were then broken up, placed on fresh filter papers and allowed to dry out fully over several weeks.

12.3.2 Rheological measurements

Rheological measurements were again carried out on a parallel plate, dynamic shear rheometer. An 8 mm plate, a 0.5 mm gap and a target strain of 0.01 % were used. The samples were loaded onto the apparatus in the same manner as that described above for the cement containing mixtures. In addition to the polymer modified emulsion residues, measurements were also made on virgin polymer modified binders as a comparison.

Results

The results of rheological tests on polymer modified emulsion residues and polymer modified bitumens were as shown in Figures 12-25 to 12-27. The first set of graphs show the effect on viscosity and the second set deals with the elastic and viscous moduli.

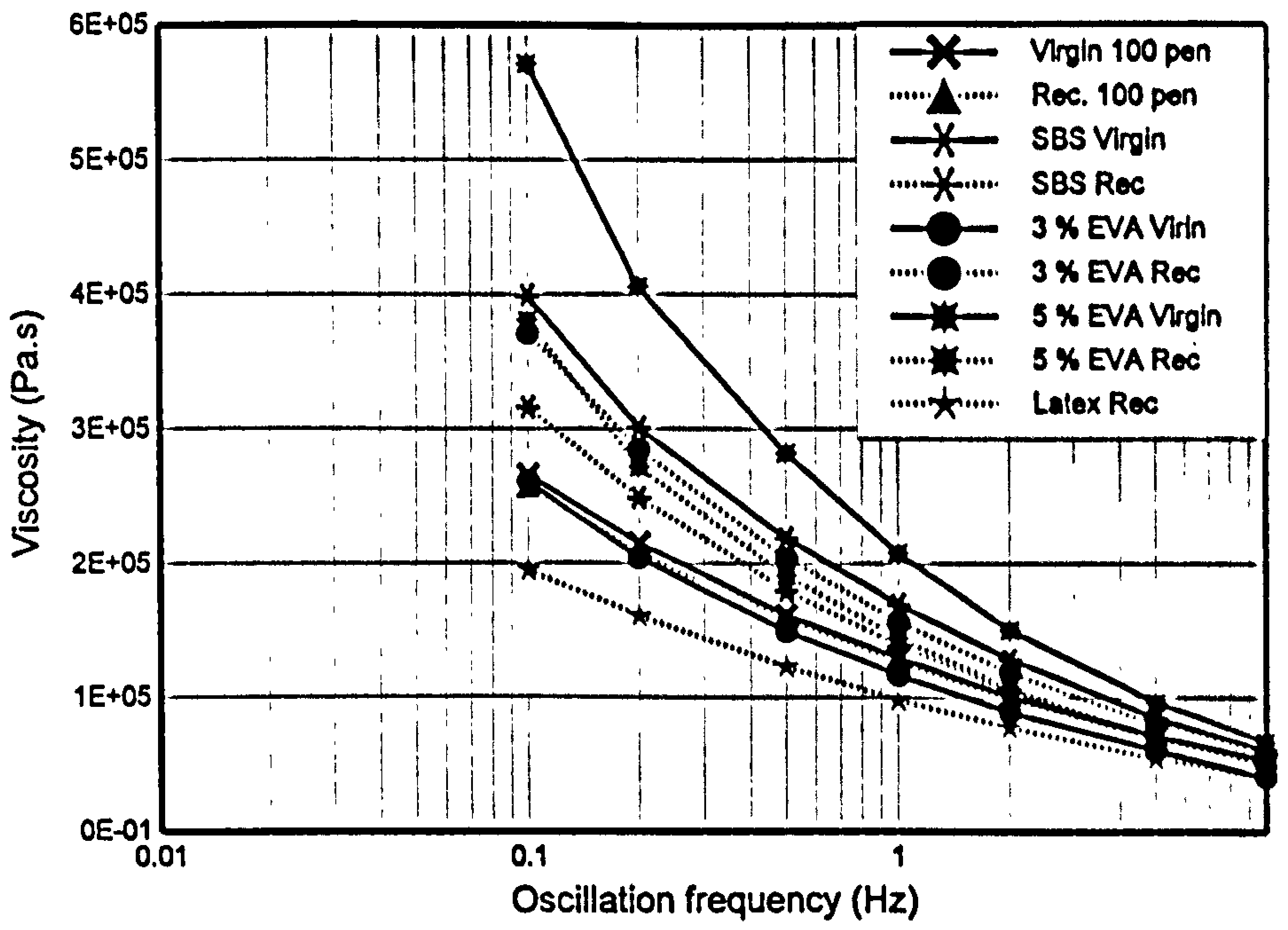


Figure 12-25 : Viscosity of bitumen and emulsion residues with different polymer additives

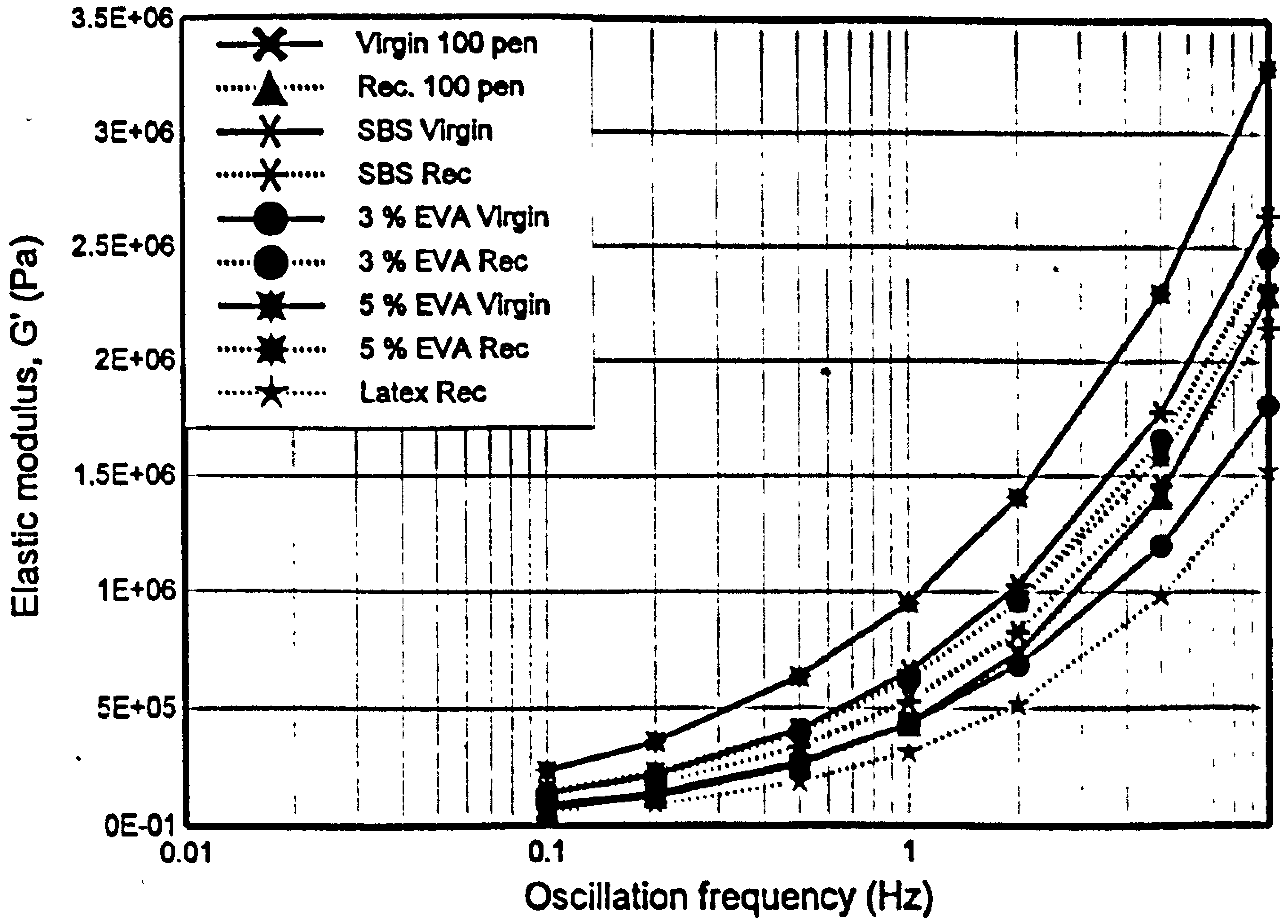


Figure 12-26 : Elastic modulus (G') of emulsion residues with different polymer additives

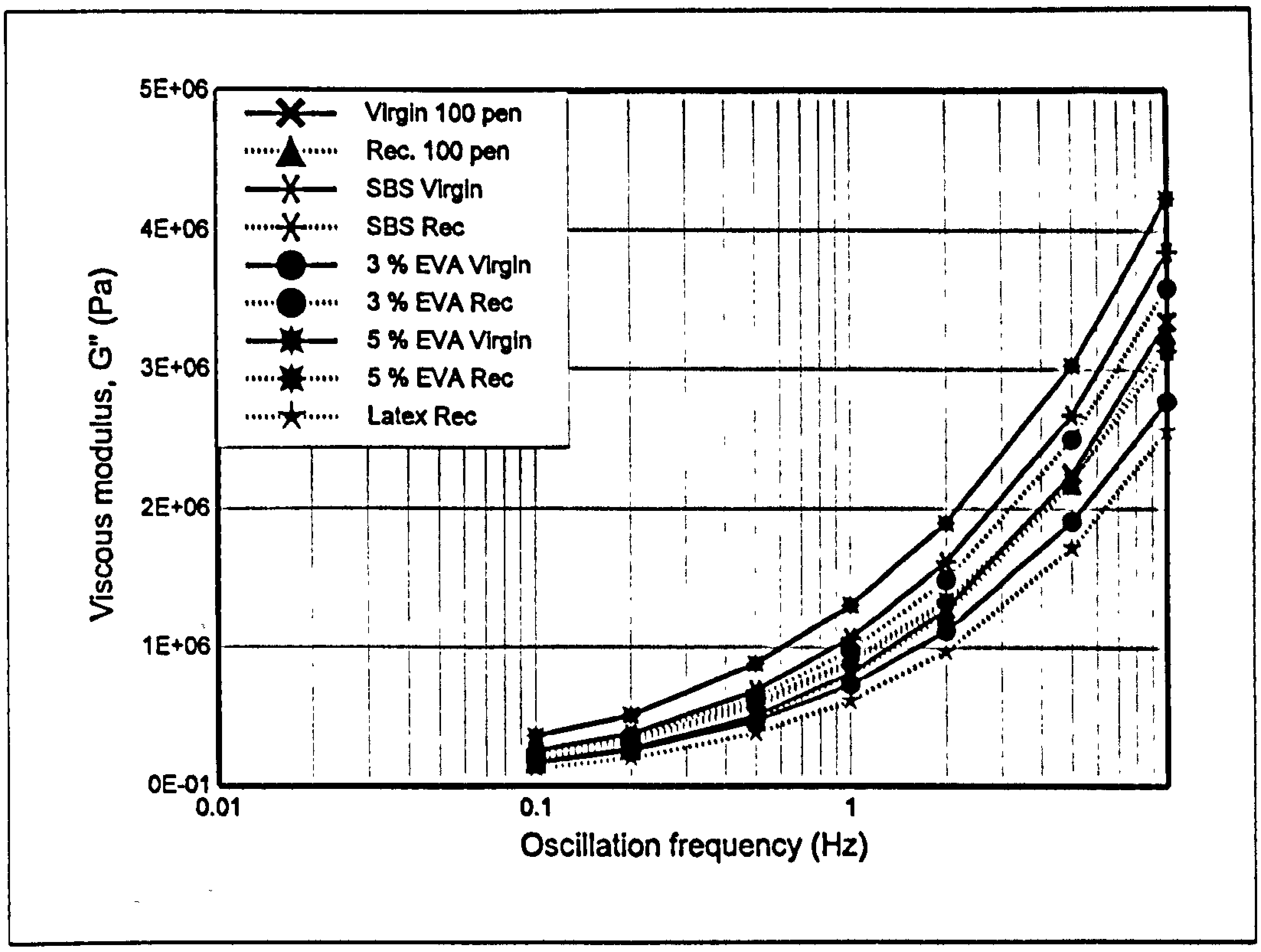


Figure 12-27 : Viscous modulus (G") of emulsion residues with different levels of OPC

Discussion

None of the polymer modified binders tested here had significantly altered rheological properties compared with unmodified 100 pen Venezuelan bitumen. In contrast with the unmodified bitumen and emulsion residue, the polymer modified equivalents showed a slight difference between the virgin and recovered binders. The reason for this is unknown but it is suspected that it was due to incomplete drying of the emulsion residues.

From Figure 12-25 a number of features are apparent. Latex caused a slight decrease in viscosity and EVA at the 3 % level had little effect at all. SBS and EVA (at a level of 5 %) increased viscosity to a small degree. The data in Figures 12-26 and 12-27 show that the polymers had the same effect on elastic and viscous moduli respectively.

These findings are quite surprising in some respects as EVA and SBS polymers were expected to have had a more significant effect on the binders. In the case of SBS, a more noticeable increase may have been observed if this polymer had been used at a level of 5 %. It is generally considered that this level of polymer is necessary to produce a continuous SBS phase in the mixture. Another reason for the smaller than expected alteration of viscosity may have been temperature. Polymers are mainly used to improve the properties of binders at elevated temperatures in excess of 30 or 40°C and also at temperatures below freezing. Under these conditions, SBS and EVA tend to have more significant effects. If rheological measurements had been made over a range of temperatures, a more pronounced change would almost certainly have been observed but time constraints did not allow these measurements to be made in these studies.

12.4 OVERALL CONCLUSIONS

The results obtained from the tests described above, allow a number of overall conclusions to be drawn about the behaviour of emulsions and emulsion and cement mixtures and properties of the cured binder.

- The addition of OPC to a bitumen emulsion of the type used in these studies, causes the emulsion to break. The higher the addition level, the faster the break. It is possible to hypothesize as to the mechanisms behind this phenomenum. One possibilty is that the Ca^{2+} ions from the cement form complexes with the emulsifier. A complex is an aggregate of polar or ionic organic molecules around a central metal ion. Exploitation of the emulsifier molecules in this way would destabilize the emulsion by removing the stabilizer from the bitumen water interface. This explanation is reasonable except that mixtures with $CaCl_2$ revealed that this additive, which is a source of Ca^{2+} ions, does not lead to break of the emulsion. Another possible mechanism is that OPC withdraws water from the emulsion thus forcing the bitumen droplets to come into contact. However, the emulsion broke in the mixtures described earlier which had only 10 % OPC on the emulsion, and in this situation, it is doubtful that the OPC could have withdrawn enough water to cause this to happen.

- The composite binder residue which results from bitumen emulsion broken with OPC, has reduced penetration or increased viscosity compared with the base bitumen.
- OPC is able to set in the presence of excess water but at levels above 2:1 water to OPC, it is unable to set properly. This level is exceeded in cold mix with OPC levels of 1 to 4 % and, therefore, cement setting can only take place after a substantial amount of water has evaporated. This is probably the cause for the steady, albeit accelerated, curing process of cement modified mixtures.
- Evidence from electron microscope images suggested that OPC cures in the same manner in bitumen emulsion mixtures as it does in normal concrete. Therefore, it probably acts as an additional binder in emulsion based mixtures.
- Polymer modification, of the types used in these studies, did not result in significantly enhanced rheological properties of the binders at 20 °C. This may have been due to dosage levels and temperature. The reason for the effect for EVA on mixture stiffness at 20 °C is, therefore, unknown.

CHAPTER 13

PRACTICAL IMPLICATIONS OF RESEARCH

13.1 INTRODUCTION

Cold emulsion mix is used in various forms throughout the world as discussed in chapter 3. In many situations it has been a perfectly functional material and quite a number of roads throughout the World bear testimony to this. However, in most cases, emulsion mixtures are currently only used for low volume roads, predominantly, but not exclusively, in fairly warm regions. This chapter presents a consideration of the usefulness of the mixtures covered in this report for major roads in a temperate climate such as that in the U.K. The main emphasis is on the use of OPC as this additive was found to offer the best improvement in properties.

13.2 MECHANICAL PROPERTIES

13.2.1 Stiffness modulus

Stiffness modulus was found to be very slow to increase in the standard unmodified mixture and the ultimate stiffness was also very low. The addition of as little as 1 % OPC had a marked effect on curing rate and final strength which brought cold mix performance up close to that of equivalent hot mix. Higher levels of OPC enhanced the stiffness even more. In a pavement, a mixture with very low stiffness would not be a very effective material to use. As discussed in chapter 5, the load spreading ability of a layer is dependent upon stiffness; a low value means that a high level of tensile strain is induced in the base of the asphalt layers and a high vertical strain on the subgrade (114). These can lead to cracking and rutting respectively. From the results obtained in this research it would, therefore, appear that OPC is essential as it serves to raise the stiffness of bitumen emulsion mixtures.

A further factor affecting stiffness, however , is void content. The mixtures tested in chapters 8 and 9 were found to have high void contents compared with the 'equivalent' hot mix. It could, therefore, be argued that the hot and cold mixtures were not equal as the emulsion mixtures were only partially compacted. However, as all samples received comparable amounts of compaction it could also be argued that in a real situation, cold mix would end up with a higher void content similar to that produced in the laboratory and, therefore, the samples were representative. However, the actual void content achieved in the field with these types of mixtures was measured as part of the field trial with the TRL as discussed in chapter 7. In this instance the void content of the 20 mm DBM cold mix was found to be around 7 % which would be a fairly typical value for hot mix also. The stiffness of the laboratory prepared samples could, therefore, be said to be unfairly low compared with that which could be achieved in the field. An ongoing project at Akzo Nobel has shown that the stiffness of cold mix is indeed very dependent on void content in the same way as hot mix. If values of 5 to 7 % voids are achieved, significantly higher stiffness can be reached. This should be taken into account in the pavement design calculations which follow.

13.2.2 Resistance to permanent deformation

RLA tests on mixtures with and without OPC revealed that the additive is also important in terms of resistance to rutting. If OPC was not included in the mixture, it was very susceptible to permanent deformation but this may have been partly due to high voids contents. Coupled with the likelihood of permanent strain in the subgrade without OPC as discussed above, an unmodified cold mix pavement would appear to be very susceptible to rutting. With the inclusion of 1 % OPC, this can be alleviated, not only by increasing the resistance to permanent deformation of the mixture but at the same time reducing the vertical strain on the subgrade due to stiffening of the bituminous layers. It should be stressed that the principles involved in subgrade strains were derived for hot mix and, therefore, may not be completely applicable to cold mix.

13.2.3 Resistance to fatigue cracking

Indirect tensile fatigue tests have shown that OPC also has an effect on resistance to fatigue cracking. However, the situation was found to be more complicated in that it was dependent on the initial strain. At strains below 200 microstrain, the addition of OPC increased resistance to fatigue cracking whereas above 200 microstrain it appeared to have the opposite effect.

13.2.4 Durability

Tests showed that water soaking of unmodified emulsion mixtures caused moulded specimens to disintegrate. Through the addition of 1 % OPC, specimens withstood the soaking procedure and retained their initial level of stiffness. These mixtures were only cured for a period of two weeks and, therefore, had not reached their full potential. However, in temperate regions where rainfall is a common occurrence, mixtures which cannot withstand it would be unusable in a practical sense due the risk of damage during the early days in the life of a pavement. Thus the inclusion of OPC would again appear to be a necessity in these types of mixtures.

13.3 IMPLICATIONS OF RESEARCH IN MIXTURE AND PAVEMENT DESIGN

13.3.1 Mixture design

It has already been explained that the studies reported in this thesis concentrated on mixtures with dense aggregate gradings, slow setting emulsions, and high binder contents. Other types of mixtures are in use in various parts of the World which have quite different designs and these have been covered in chapters 3 and 4 of this thesis. These mixtures are, therefore, proven and can presumably be utilized provided any restrictions are adhered to. Bearing the current technologies and the findings from these studies in mind, tentative recommendations on mixture design can be made.

The aggregate type and grading used in a cold mix, not only has an effect on the mechanical properties of a mixture as it would on hot mix, but also influences the rate of emulsion break and cure which ultimately also affects mechanical properties. As discussed in chapter 2, the chemical interaction between emulsion and aggregate can cause the emulsion to break. Therefore, it is likely that the finer the aggregate grading, the faster the rate of emulsion break. In practice emulsions are selected to work with certain gradings, in that slow setting varieties are used with dense gradings and medium setting with more open gradings. In contrast with emulsion break, when it comes to emulsion curing which is reliant upon loss of water from the mixture, the more open the aggregate grading the faster the rate of cure. Thus, selection of aggregate grading and emulsion type is fundamental to mixture behaviour.

These studies and others already mentioned in chapter 4 have clearly shown the beneficial effects of the incorporation of small amounts of ordinary portland cement in emulsion mixtures. The additive increases the rate of stiffness increase, ultimate stiffness, durability and resistance to permanent deformation and fatigue cracking at strains below 200 μstrain. Consequently, it is recommended that 1 or 2 % OPC be used, particularly in cold mix of the type used in these studies, as performance without this additive would appear to be fairly poor.

The total fluids content (water and bitumen) in emulsion mixtures is crucial in designing a good cold mix as it affects all aspects of behaviour. The approach taken in these studies, which also seems correct on a larger scale, was to first set the desired residual binder content which was suitable for the aggregate grading used. This should be the same whether the mixture is manufactured using a hot process or cold with emulsion, as the final fully cured mixtures should be the same. This is in fact open to debate as compactability of cold mix is inferior to hot mix meaning that the volumetric proportions of supposedly equivalent final mixtures will not actually be the same.

Once the binder content has been set, the amount of pre-wet water required to produce a good mixture has to be determined. This is done on the basis of the degree of coating of the aggregate, aiming to have maximum coating, and workability and compactability of the mixture. Degree of coating can be determined by means of small scale mix tests made with different levels of water. Increasing the water content from 1 to 2 % may improve coating as the emulsion is better able to spread over the aggregate without breaking but at higher levels the mixtures may become over wet, preventing the binder from adhering to the aggregate. Workability can also be assessed in small scale mixtures on a purely subjective level. More rigorous testing of workability and compactability can be carried out using the gyrocomp equipment described in chapter 5. It is important to assess these two parameters on both newly made mixture and mixture which has been stockpiled for a number of hours or days because one of the main attractions of cold mix is its storability compared with hot mix.

13.3.2 The analytical approach to pavement design

The function of a pavement is to enable the movement of traffic in a satisfactory state of comfort and safety. This requires that the structure has an even profile and is capable of withstanding the loads placed upon it. In order to satisfy the latter criterion, a pavement must be designed and constructed correctly. Up until the mid 1980s, an empirically based code of practice entitled Road Note 29 (153) was used in the U.K. This relied upon experience and the measurement of mechanical properties of the subgrade (154). The method endured due to its widely accepted ability to produce perfectly functional roads and the preconceived complexity of a theoretically based approach. As a result of research, this was replaced in mid 1980s by LR 1132 (155) which employed an analytical approach to pavement design.

The philosophy behind the analytical approach is that the pavement structure be treated in the same manner as any other engineering structure, in that the design parameters and methods are based on scientific principles. The procedure can be summarized as follows (36, 114) :

1. Specify the loading
2. Estimate the size of the components in the structure
3. Consider the materials available and their mechanical properties
4. Carry out a structural analysis using theoretical engineering principles
5. Compare critical stresses, strains or deflections with allowable values
6. Make adjustments to materials or geometry until a satisfactory design is achieved
7. Consider the economic feasibility of the result

Failure modes

There are two modes of failure, namely cracking and rutting (Figure 13-1). These two phenomena have been covered already in chapter 5 but their significance in pavement deterioration will be reemphasised here. Fatigue cracking begins at the base of an asphalt layer as a result of repeated tensile strains (ε_t) which are below the tensile strength of the material but large enough to induce cracking under repeated loading. The magnitude of strain is dependent on the pavement design in terms of the stiffness of the asphalt layers, the thickness of the asphalt layer and the stiffness of the supporting layers. The other failure mode, namely rutting, arises from the accumulation of permanent strain. It is dependent on deformations in the bituminous layers and vertical strain (ε_z) in the subgrade. Analytical design must ensure that this parameter is kept below a certain level to prevent excessive rutting. Rutting can be minimized by good compaction of the asphalt layers, the sub base and subgrade.

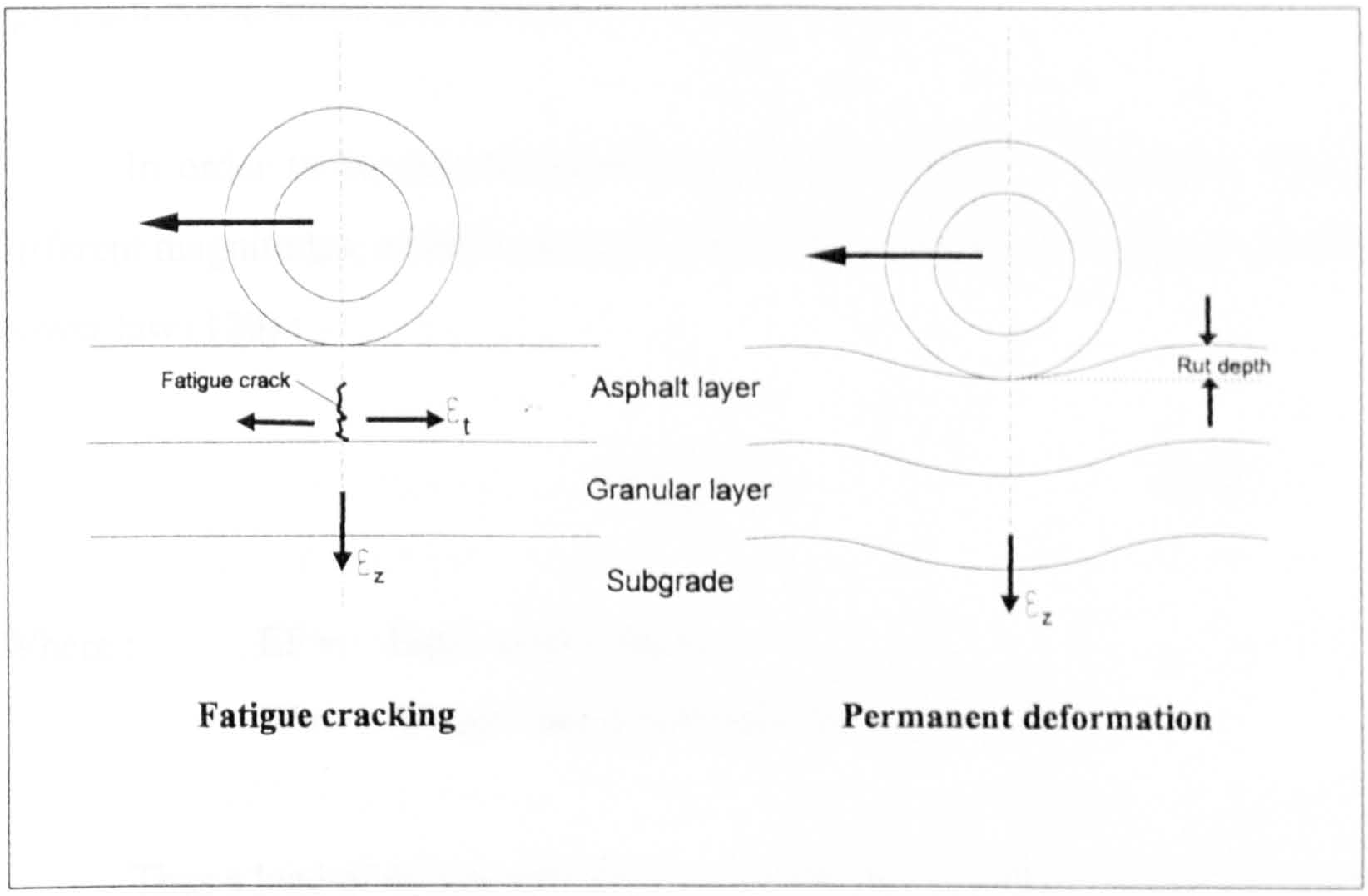


Figure 13-1 : Critical strains and pavement failure modes

The objective in pavement design is to devise a structure which will last for a specified period of time in terms of load applications, ie traffic. There are two phases which mark the end of the design life of a pavement - namely the critical stage and the failure stage. Pavements do not fail suddenly, except in very extreme circumstances, but degrade gradually over time. The critical stage is the point at which the pavement is showing signs of deterioration and marks the time when repair work must be carried out to save the residual pavement in order to extend its serviceable life. If repair or strengthening is not carried out at this point, the pavement will move quickly to total failure at which point it becomes unserviceable and requires major reconstruction.

Pavement loading

Pavement loading is complicated, consisting of a range of load magnitudes and rates of application. In order to simplify pavement loading in the design process, standard units of traffic loading have been adopted. A standard axle is defined as an 80 kN load comprising two equal wheel loads of 40 kN. This standard originated in the USA (156) and has been adopted in the UK and many other countries. In the pavement design process, the actual loading spectrum is converted into numbers of standard axles.

In order to be able to assess the damage done to a road by loads of different magnitudes, an equivalency formula was introduced based on a fourth power law (120) :

$$EF = [\frac{W}{40}]^4 \qquad (13\text{-}1)$$

Where : EF = Equivalency factor

W = Wheel load in kN (half the axle load)

Thus a load of 40 kN will have an EF of unity, a load of 20 kN 0.06 and a load of 80 kN 16. This illustrates the exponentially high level of damage done to a pavement by trucks compared to motor vehicles.

Conversion from load applications in laboratory tests to standard axles

In order to be able to compare laboratory generated data with performance in the field, it is necessary to be able to convert laboratory load applications to standard axles. Brunton proposed a multiplication factor of 440 for hot mix (157). It is not known how applicable this figure is to cold mix but in the absence of any other figure it will be employed in the calculations which follow. This was built up from figures of 20 to allow for crack propagation, 1.1 to allow for lateral wheel load distribution and 20 to allow for rest periods (20 x 1.1 x 20 = 440).

13.3.3 Pavement design with hot mix

The procedure for pavement design is based on the use of charts or computer models. The steps can be summarized as follows :

1. Determine the mixture stiffness in situ (dependent on temperature and time of loading (ie average vehicle speed))
2. Specify the design life in standard axles

3. Determine the subgrade stiffness (MPa)
4. Calculate the maximum allowable strains, ε_t and ε_z, to reach specified design life, from suitable charts
5. Calculate the minimum design thickness from charts of asphalt strain v thickness and subgrade stiffness

13.3.4 Pavement design with cold mix

The above method as used in the UK, has only been applied to hot mix. Some other countries have developed pavement design methods for use with emulsion mixtures. The Southern African Bitumen and Tar Association (SABITA) published a manual on "the design and use of granular emulsion mixes" in 1993, to aid in the design of emulsion mixtures and structural procedures (73). Two approaches are described. For the stabilization approach, in addition to the parameters required for hot mix, cold mix design requires that the stiffness of the emulsion mixture is known at various intervals during the curing period. In the modification approach, only one stiffness value is used. Once the stiffness values have been established, these can be used in dedicated computer programs to calculate maximum permissable stresses and strains. Fatigue and vertical compressive strain charts are provided for the two approaches (ie stabilization and modification) to facilitate the calculation of lifetimes in terms of standard axles in relation to initial strains. These charts relate to void and binder contents of 5 and 5 to 8 % respectively but a formula is provided to convert to different values (13-2). In addition to a chart for normal emulsion mixtures, there is also one for mixtures modified with 2 % by mass of OPC.

$$Nc = Nf \cdot 10^{m} \qquad \textbf{13-2}$$

Where : Nc = Corrected number of repetitions to failure

Nf = Number of repetitions to failure at a given horizontal strain level and resilient modulus

m = 4.84 x [Vb / (Vv + Vb) - 0.69]

where : Vb = Volume of bitumen
Vv = Volume of voids

The stabilization method was first put forward by Santucci (158) but has been simplified in the Southern African method. Santucci's method required input of the changing stiffness of a mixture at monthly intervals whereas the SABITA method uses intervals of 3 months. Stiffness in situ is a function of ambient temperature and curing rate of the mixture. It is claimed that in southern regions of North America, cold mix cures in 6 months whereas in the North it takes 2 years. The design procedure based on tensile strain is as follows :

1. Assume pavement thickness
2. Conduct early cure analysis by examining monthly variations in mixture stiffness.
    a. Determine the appropriate stiffness month by month (from chart)
    b. Determine horizontal tensile strain, ε_t (from chart)
    c. Determine lifetime to failure, N_f, from fatigue line
    d. Calculate $1/N_c$ for each month and sum at end of analysis period
    e. Determine predicted damage for early cure period, D_E, by multiplying $(\sum 1/N_c)_E$ by the average monthly traffic.
3. Repeat step 2 for fully cured mixture and calculate damage factor, D_F, by multiplying $(\sum 1/N_c)_F$ by number of years in service.
4. Sum D_E and D_F to obtain total damage factor, DF
5. Repeat steps 1 to 4 for different assumed thickness.
6. Plot total damage factor, DF, versus thickness to determine thickness required for a damage factor of unity.

A similar procedure can be carried out to determine pavement thickness on a vertical strain basis and then the larger is taken as the design thickness.

Although the procedure is straightforward, it is laborious and so computer programs have been written to perform the calculations.

In the American Asphalt Institute manual MS-1, "Thickness Design - Asphalt Pavements for Highways and Streets" (156), the method for thickness determination for emulsified asphalt base pavements does not adopt Santucci's method. Instead, design charts are based on expected stiffness values after 6 months of curing which is a marked simplification. Different charts are provided for different mixture types and ambient temperatures which enable minimum thickness to be found knowing merely the subgrade modulus and the desired lifetime of the road in standard axles. Procedures are based on an application of elastic layer theory which uses the results of acceptable research. Using these charts, the required thickness of a type II emulsified asphalt mixture and a full depth asphalt concrete (hot mix) would be 450 mm and 330 mm respectively, for a design life of 10^7 standard axles on a subgrade with a stiffness modulus of 30 MPa at an ambient temperature of 15.5 °C. It can be seen from this that the emulsified asphalt pavement has to be somewhat thicker than the asphalt concrete version, in order to obtain the same level of performance.

13.4 DESIGN CALCULATIONS BASED ON DATA OBTAINED IN THIS RESEARCH

In order to assess the pavement design implications of the data obtained in these studies, calculations were carried out using analytical techniques. The American method (MS-1) could not be utilized as this is only applicable for set mixture types with predictable stiffness. Santucci's method was thought to be overcomplicated and in any case sufficient data were not available from these studies to carry out the procedure in full. Similarly, the SABITA method was not completely applicable and the thickness calculation part of the method is computer based and was not available. However, use was made of the fatigue charts to some extent. Most of the calculations which follow made use of the

British design procedure even though it was originally devised for use with hot mix. Calculations based on failure through cracking only were made, as data for failure through rutting were not available.

13.4.1 Calculation of pavement thickness

Cold mix

The calculations below use data obtained from tests described in chapter 8.

1. Assume a pavement design life to failure of 10^7 standard axles $= 10^7/440 \sim 23000$ Indirect Tensile Fatigue Test load applications
2. From Figure 8-7 the maximum allowable strains are as shown in Table 13-1 for different OPC addition levels
3. From the graph in Figure 13-2 (114) the required layer thicknesses are as shown in Table 13-1, based on the stiffness values for each mixture at the fully cured staged as estimated from Figure 8-2. The Figure for 3 % OPC (9,000 MPa) is interpolated as a mixture with this level of OPC was not tested. Note that the graph in Figure 13-2 is based on a subgrade stiffness of 30 MPa.

From these calculations it can be seen that the addition of OPC allows much thinner layers of asphalt to be laid. This is a consequence of both the increase in stiffness which reduces the tensile strain on the base of the asphalt layer and the increase in resistance to fatigue cracking at a given level of strain.

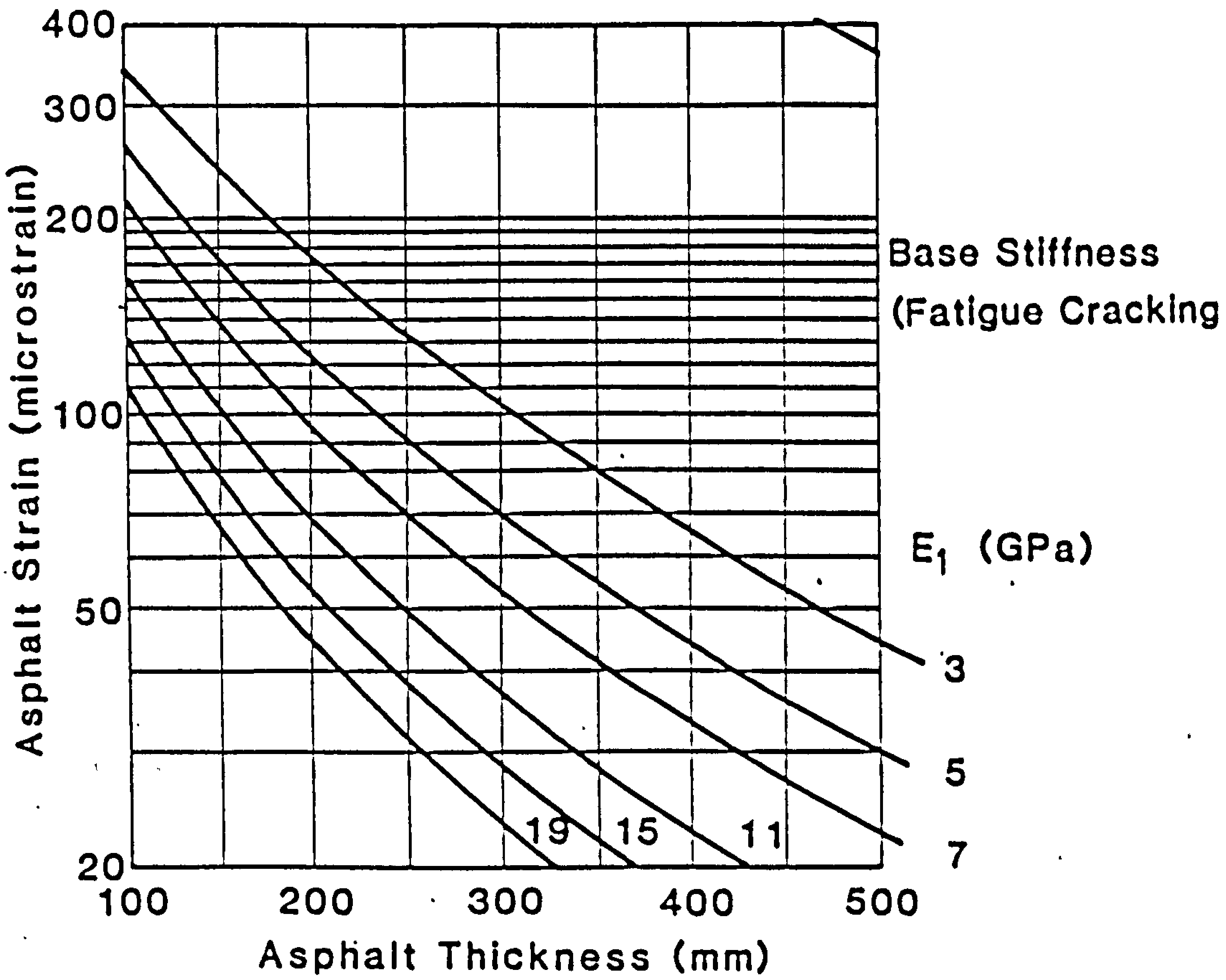


Figure 13-2 : Critical strains as functions of stiffness and thickness of asphalt (subgrade elastic stiffness = 30 MPa, sub-base thickness 200 mm)

Table 13-1 : Layer thicknesses for cold mix pavement with different levels of OPC

OPC level (%)	Max allowable strain (μstrain)	Stiffness of cured mixture (MPa)	Layer thickness (mm)
0	60	1500	500
1	85	2500	360
3	140	9000	140

If these calculations are repeated using the fatigue lines provided in the SABITA manual for granular emulsion mixtures without OPC and with 2 % OPC (reproduced in Figures 13-3 and 13-4 respectively) (73), the results shown in Table 13-2 are obtained.

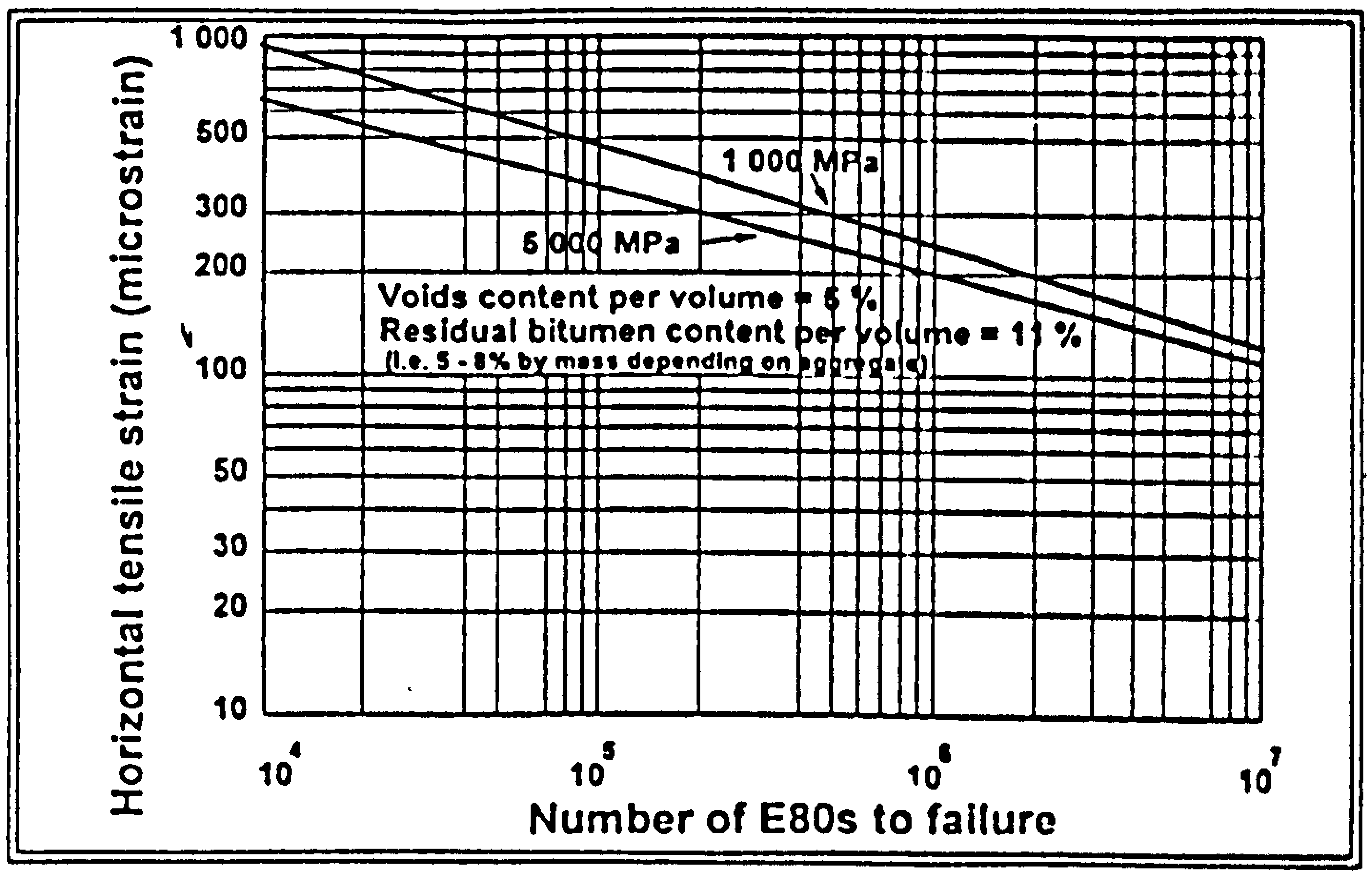


Figure 13-3 : Fatigue criteria for granular emulsion mixtures (SABITA)

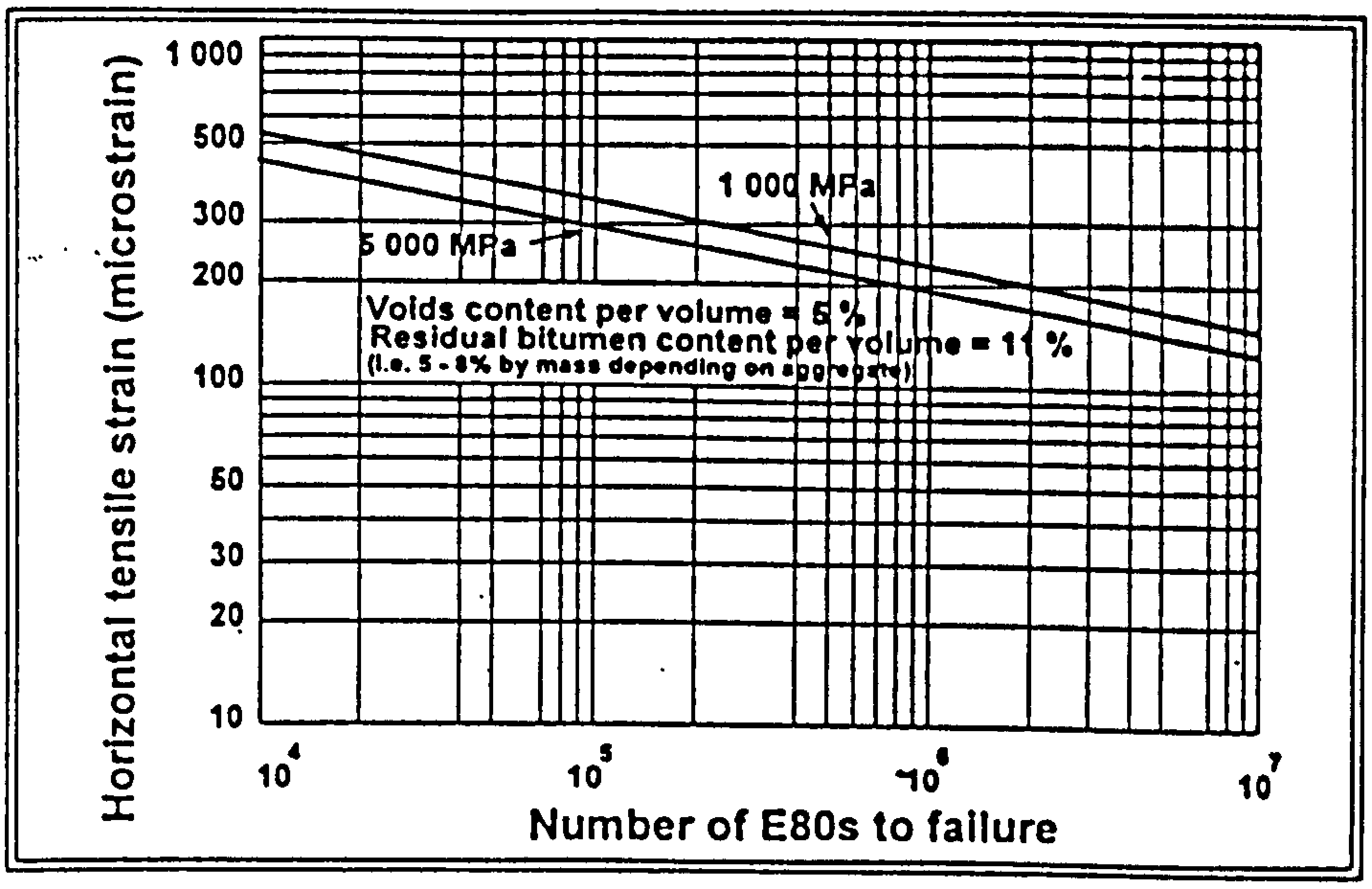


Figure 13-4 : Fatigue criteria for cement modified granular emulsion mixtures

Table 13-2 : Layer thicknesses based on data from SABITA manual

OPC level (%)	Max allowable strain (μstrain)	Stiffness of cured mixture (MPa)	Layer thickness (mm)
0	140	1000	330
2	120	5000	210

Comparing the figures obtained using the data from this research and that from the SABITA manual (derived from Santucci (158)), it can be seen that the latter specifies lower layer thicknesses. This arises from the difference in the fatigue lines with the SABITA lines having greater maximum permissable strains for a given number of load cycles. Discrepancies could also have arisen from errors in the approximation method used in converting laboratory load applications to standard axles. The high void content of the laboratory prepared specimens used to generate the data in Table 13-1 should also be taken into account.

Hot mix

A similar calculation can be carried out on an equivalent hot mix material. In the following calculations a mixture stiffness of ~ 2,500 MPa is assumed. Firstly the recovered Softening Point of the bitumen must be found :

Recovered penetration, $P_r = 0.65 \times P_i$ (initial penetration) **(13-3)**

$\therefore$ For 100 pen bitumen, $P_r = 65$

Softening Point of the recovered binder, $SP_r = 98.4 - 26.35 \log P_r$ **(13-4)**

$\therefore$ For bitumen with recovered penetration of 65, **$SP_r = 50.63$**

The volume of the binder is also required and can be found from :

$$V_B = \frac{(100-V_V)(M_B/G_b)}{(M_B/G_b)+(M_A/G_a)} \qquad \textbf{(13-5)}$$

Where : $V_v =$ Volume of voids in the mixture, ~5 %

$M_B =$ Percentage of bitumen in the mixture by mass, 4.49 %

$M_A =$ Percentage aggregate in the mixture by mass, 95.5 %

G_b = Density of the bitumen, 1.1

G_a = Density of the aggregate, 2.6

Thus : **$V_B \sim 9.5$ %**

From the nomograph after Brunton (157) (reproduced in Figure 13-5), the maximum allowable tensile strain for 10^7 standard axles is : **140 microstrain**

From the graph in Figure 13-2, the required layer thickness for a 20 mm DBM hot mix with a stiffness of 2,500 MPa is : **260 mm.**

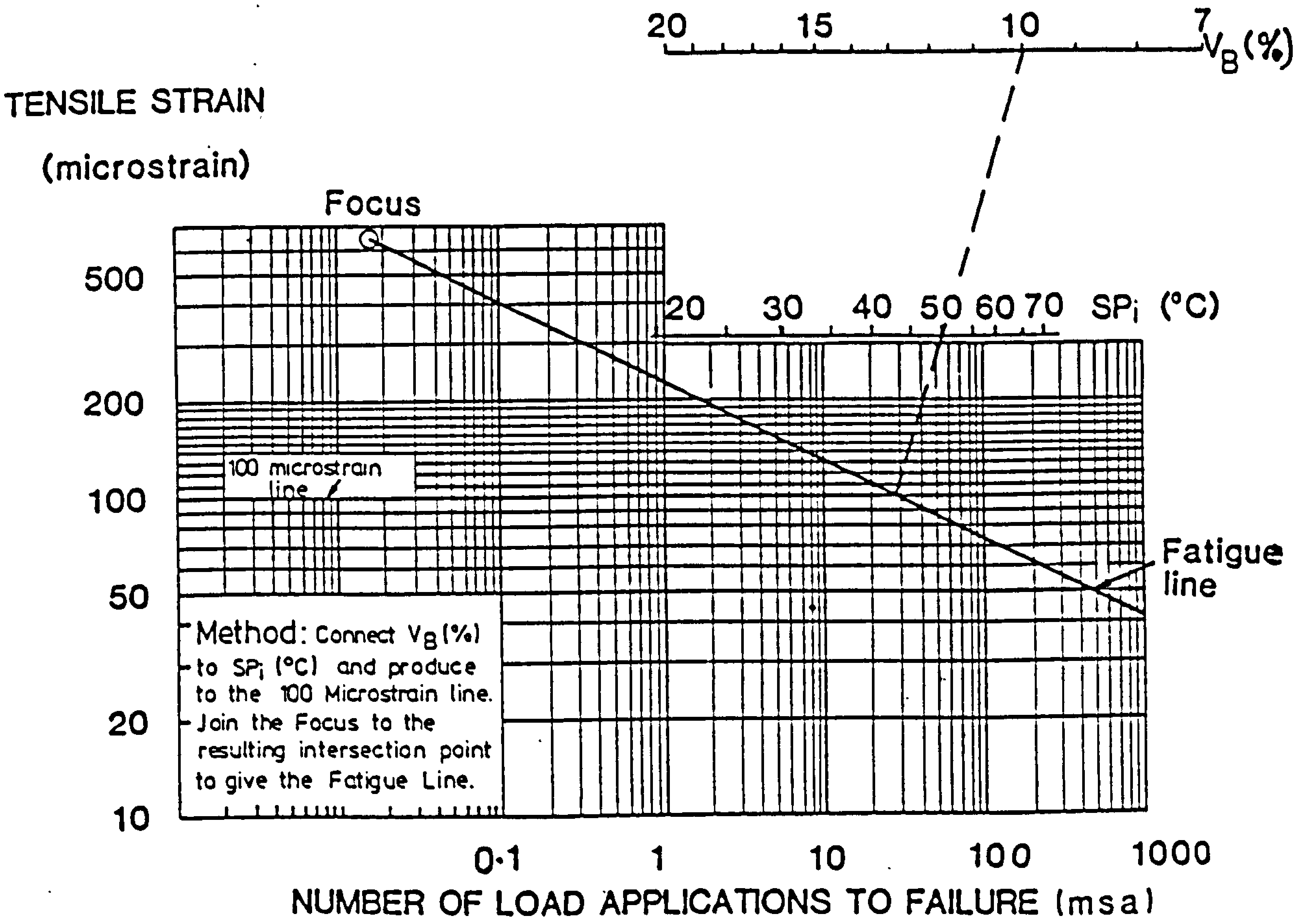


Figure 13-5 : Nomograph for the determination of fatigue life to failure

Comparing hot and cold mix

Figure 13-6 shows a graph of layer thickness requirements for hot mix and cold mix, based on results from this research and SABITA figures, at a range of

OPC levels. From this graph it can be seen that without OPC, thicker layers of cold mix are required. According to the graph, if 1.2 % and 1.9 % OPC are added to cold mixtures, using figures obtained from the SABITA manual and this research respectively, thicknesses equal to those of 20 mm DBM hot mix are possible. These figures are in good agreement with those proposed by Schmidt et. al. (15). From calculations similar to those above, they found that cold mix with 1.3 % OPC could be laid at the same thickness as equivalent hot mix.

As discussed above, the figures derived from cold mix tested in this research were obtained from mixtures with significantly higher void contents than those which are typical of equivalent hot mix. This will have reduced the stiffness of the emulsion mixture which affects the performance and, therefore the layer thickness. If lower void contents could be achieved in an emulsion mixture, then the difference in performance may not be so large.

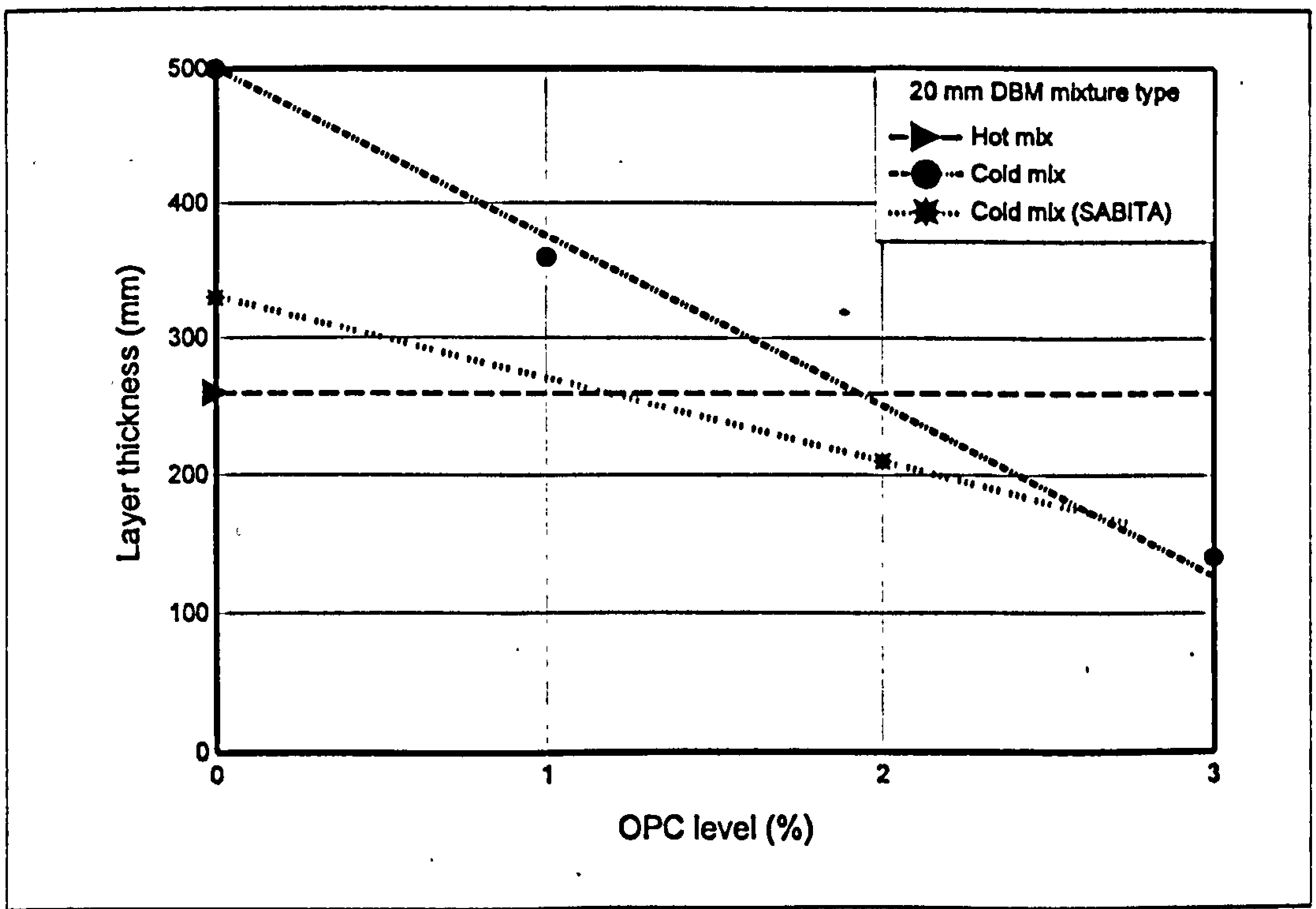


Figure 13-6 : Layer thickness for different mixture types

13.5 EARLY LIFE CONSIDERATIONS

The calculations above were based on cold mixtures in advanced stages of curing. As shown in the literature and in this research, emulsion mixtures develop their full strength over a period of time. This can be several weeks or several months depending on the mixture formulation and ambient conditions. During the curing period, a mixture has a substantially lower stiffness which is changing all the time. Subsequently, the tensile and vertical strains will be higher than those predicted using stiffness values of fully cured mixtures. As discussed above, Santucci's (158) and the SABITA (73) methods take this into consideration and the resulting layer thicknesses would inevitably be higher.

The rate of degradation of cold mix pavement would also be dependent on time of opening to traffic and traffic volume. In the case of low volume roads, such as those currently built with cold mix, it would be comparatively easy to keep traffic off for a few weeks after laying and, even after opening to traffic, the low traffic levels would not place excessive demands on the pavement. If cold mix were to be used in major roads, however, this tender period would create a major problem as the road would have to be opened to traffic soon after laying and traffic volumes would be high. From the evidence gathered in this research it would, therefore, appear that emulsion mixtures of the type covered herein are not suitable for major road construction.

13.6 CONCLUSION

From the calculations and discussion above, it can be concluded that cold mix is potentially a viable material for use in all kinds of pavements in the UK due to the fact that it can be designed to have mechanical properties equivalent to hot mix. However, the slow curing nature of emulsion mixtures and their sensitivity to rainfall during this curing period means that in reality they may not as versatile in practice as theory might suggest.

CHAPTER 14

OVERALL CONCLUSIONS AND FUTURE WORK

14.1 OVERALL CONCLUSIONS

The research reported in this thesis has combined bitumen emulsion technology and pavement engineering in order to better understand the mechanisms involved in cold mix. This has been achieved through a study of the mechanical properties of bitumen emulsion mixtures and the effects of certain variables. These mixtures have then been broken down and examined by means of other tests. As a result of this research, the following overall conclusions seem to be justified. The statements below relate to the emulsion, aggregate and mixture types employed in this work. It is likely that they apply to other formulations but it is not claimed that they will apply to all.

- Bitumen emulsion mixtures can be manufactured using conventional hot mix plant (without heating) provided correct mixture recipes are closely adhered to. Mixtures thus produced can be laid using conventional plant or by hand. Cold mix material of the type manufactured in the course of these studies was found to be susceptible to rainfall.

- Bitumen emulsion mixtures require several weeks or months to reach their ultimate potential stiffness. Without additives, the mixtures tested in this research never achieved the stiffness level of equivalent hot mix. The slow curing process was due to the slow total evaporation of water which must take place in order for the bitumen to revert to its original form and thus regain its original properties. It would appear that without modification, the binder never fully returned to its original form and this seems to have been due to the presence of a small amount of water in the mixture which never escaped. However, the overriding factor seems to be that emulsion mixtures never reach the stiffness of equivalent hot mix because they are not equivalent in terms of void content. Due to the presence of water in cold mix during compaction and the inferior lubricating effect of emulsion compared with hot bitumen, the void contents of emulsion mixtures are always higher using the same compaction regime. If compacted to the same level of voids, the stiffness of hot and cold mix are comparable.

- Ordinary portland cement increases the curing rate (in terms of stiffness modulus) the ultimate stiffness modulus and resistance to permanent deformation and fatigue cracking (at strains below 200 microstrain) of cold mix. Several mechanisms were found to be responsible for these phenomena. OPC increases the rate of coalescence of bitumen emulsion under compaction. The addition of OPC leads to an increase in cationicity of the emulsion under certain conditions which could increase the rate of break of the emulsion in contact with negative silicious aggregates. OPC forms a composite binder with bitumen which has a lower penetration and a higher viscosity than the bitumen alone. OPC was seen to set as it would in a normal concrete mixtures and, therefore, it is likely that it acts as a secondary binder.

- EVA polymer modification of the bitumen increases the stiffness modulus of cold mix. This is partly due to stiffening of the binder which is achieved with this polymer. However, rheological measurements revealed that the increase in viscosity of the binder was not large. EVA and SBS do not significantly affect the resistance to fatigue of OPC modified cold mix at a level of 3 % on the bitumen.

- The durability of cold mix is poor but it can be much improved by the addition of OPC or hydrated lime to the mixture.

- Stiffness modulus of cold mix can be increased by using lower penetration grade bitumen, compacting to lower void content or adding EVA to the binder.

- A number of parameters were found to have an effect on the rate of coalescence of bitumen emulsions under compaction in mixtures with aggregate. The most notable, which caused an increase, were increasing binder penetration grade, increasing compactive effort, ordinary portland cement (OPC), some polymer additives, time and increasing temperature.

- Emulsifier level, binder penetration grade and polymer modification of the bitumen have no effect upon the electrochemical properties of a bitumen emulsion. The addition of OPC shifts the charge of an emulsion towards a less negative or more positive electrical potential in conditions of low ionic concentration but in run-off water, charges on aggregate and emulsion are all virtually neutral.

- OPC greatly increases the viscosity of the bitumen residue, more so than hydrated lime or inert fillers. EVA and SBS also increase viscosity but only marginally.

- Microscope observations showed that OPC cures as it would in a normal concrete suggesting that it will act as a binder in emulsion mixtures.

- Without OPC, cold mix pavement would need to be laid twice as thick as an equivalent hot mix to obtain the same level of performance. Through the addition of between 1 and 2 % OPC, the required thickness would be similar. However, limited field experience suggested that early life trafficking and adverse weather conditions (wet and / or cold) would cause cold mix pavement to deteriorate or prevent it from curing.

14.2 FUTURE WORK

14.2.1 Field trials

One of the most important objectives of this research which, for one reason or another, was not fulfilled, was to obtain hard data from the field on bitumen emulsion mixtures. As previously stated, this is required in order to be able to compare laboratory prepared specimens with what is actually achievable in the field in order to be able to predict mixture performance in the future. Trials in this research showed that emulsion mixtures could be manufactured and laid, in favourable conditions, but no data on voids and stiffness values was obtained apart from that supplied by the TRL on a mixture of variable quality. A major objective of any future research would, therefore, be to rectify this lack of data. Large scale trials could be performed in the controllable environment of the pavement test facility at the University of Nottingham to obtain accurate results on performance but this is no substitute for real field conditions in which the loading and weather conditions are constantly changing and often much more demanding.

14.2.2 Development of laboratory tests

In order to develop mixtures which satisfy the UK requirements for cold mix, a set of laboratory tests are required which can be related to the field. Once data has been accumulated from field trials, laboratory test methods should be developed to enable laboratory data to be linked to field performance. These would include methods for measurement of stiffness, resistance to fatigue and permanent deformation and assessment of workability and compactability. Stiffness measurements should also be carried out in very early life in the order of 1 or 2 hours after laying as in a field situation, very early trafficking is is inevitable. This will require the use of some kind of triaxial test and development of a simple method, possibly based on the NAT, would be very useful for cold mix. As it was clearly indicated that cold mix compacted to low void content could achieve stiffness comparable with hot mix, the development of effective compaction methods would be very beneficial.

14.2.3 Performance criteria for cold mix

Several varieties of cold mix, including OGEM and Grave emulsion, are known to have very low stiffness in early life yet they have performed well in the field. Conversely, in these studies it was found that mixtures which had acceptable stiffness in the laboratory did not perform well in the field. Therefore, it seems evident that stiffness is not necessarily the only important factor in defining a good cold mix. At the moment, the HAUC specifications described in chapter 4 are heavily centred on stiffness and these values have been set from measurements on equivalent hot mix material. In the UK, therefore, we are in a situation in which cold mix systems which may have performed well in trials in terms of performance in situ are being excluded because they fail to meet the stiffness modulus criteria. An effort should be made to find out what performance criteria are important specifically for cold mix. It is envisaged that these should certainly include surface cohesion, resistance to rutting and durability.

14.2.4 New aggregate gradings

The studies reported here focused only on mixtures based on the BS 4987 designs for 20 mm DBM hot mix; or slight variations on them. These formulations were designed for hot mix and may not be ideal for cold mix which in some ways has quite different requirements. It would be of interest to look at other aggregate mixtures in an attempt to optimize a grading specifically for cold mix. It is suggested that Stone Mastic Asphalt mixtures, which contain a large stone matrix infilled with fines, may be suitable. The thinking being that the aggregate skeleton would provide strength and the high fines content would help to break the emulsion. Alternatively, more work should be done on open graded systems as these have been found to be very effective in a number of regions. However, completely new gradings should also be sought and it is not known what form these may take. In changing aggregate gradings, it should be realized that different emulsion types may be required and one emulsion will most likely not be suitable in all cases.

14.2.5 Development of new emulsifiers

Durability of emulsion mixtures is an inherent problem due to the possibility of re-emulsification of the binder, by the action of water, during or possibly even after curing. Cationic emulsion systems should be resistant to degradation from water but these emulsions are currently too rapid setting in nature for coating aggregate mixtures. Anionic or nonionic products are slow setting in combination with siliceous aggregates but these have no affinity for aggregate. Developments in the chemistry of the emulsifier are required to provide a specially designed molecule which is inherently cationic but masked in some way which will allow the manufacture of slow setting cationic emulsions. The difficulty in this area of development is that the chemistry of any products always remains the property of the manufacturer and it will, therefore, be hard for an independent researcher to work in this field.

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