HIGH STRENGTH COLD ROLLED ASPHALT SURFACE COURSE MIXTURES

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Declaration

The research reported in this thesis was conducted at Liverpool John Moores University, School of the Built Environment, between January 2011 and April 2014. I declare that the work is my own and has not been submitted for a degree at another university.

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Abstract

Cold Bituminous Emulsion Mixtures (BEMs) means manufacturing of asphalt at ambient temperature using bitumen emulsion as the binder. It has been widely utilised in many countries such as the USA and France. The use and development of BEMs were not brought forward in the UK due to the country's relatively wet/cold climatic conditions, which are not favourable to the application of cold BEM in terms of the long curing process and low early strength.

Decreasing wastes from aggregate production processes, reducing land-filling and reducing CO_2 emissions during hot bituminous mixture production and laying are the main target schemes for the environmentally friendly processes. Cold BEM is one of the attractive methods of producing bituminous mixtures to tackle the mentioned disadvantages when incorporating some waste and/or by-product materials individually or collectively into these mixtures. Recently, researchers have shown an increased interest in incorporating supplementary cementitious materials (SCM) in production of BEMs in the UK and around the world. Three benefits can be stated when using SCM in BEMs; these are upgraded mechanical properties, gaining economic advantage and the ecological advantage factor.

Mainly due to some inherent problems associated with the performance of the pavement produced by the BEMs process, they are regarded as "inferior" to conventional HMA. The major problems with this kind of application are the long curing time (evaporation of trapped water) required to achieve the required performance, the weak early life strength (because of the existence of water) and high air voids content. The full curing in the field of these mixtures may occur between 2−24 months depending on the mixture's ingredients and weather conditions.

Considering the above disadvantages, this study investigated the possible ways of developing a new BEM/s with gap graded mixtures similar to the conventional Hot Rolled Asphalt (HRA) gradation. HRA is extensively used for surfacing major roads in the UK because it provides a dense, impervious layer, resulting in a weather-resistant durable surface able to endure the demands of today's traffic loads and providing good resistance to fatigue cracking. The mentioned new product is termed Cold Rolled Asphalt (CRA).

The main aim of this study was to investigate producing high strength, fast curing and sustainable CRA mixtures for heavily trafficked road and highway surfacing layers by using different waste and by-product materials (normally used as SCM) individually and/or collectively as a replacement for conventional mineral filler. To achieve the above aim, four SCMs have been used which were: Waste Paper Sludge Ash (WPSA), which has high lime and gelenite content, Poultry Litter Fly Ash (PLFA), which has high alkali components, Silica Fume (SF), and Rice Husk Ash (RHA), which is high silica content and cost-plus material, collectively instead of conventional mineral filler. In addition, besides the production of the new high-quality CRA mixtures, the research includes a detailed comparison study of conventional HRA mixtures, CRA mixtures containing conventional mineral filler and CRA mixtures containing hydraulic filler, i.e. Ordinary Portland Cement (OPC).

This laboratory study was conducted by utilising different types of testing and curing and conditioning methods to characterise the mechanical properties and durability of the produced CRA mixtures. Indirect Tensile Stiffness Modulus (ITSM), Uniaxial Compression Cyclic Test (UCCT), Four Point Bending fatigue test on prismatic shaped specimens (4PB) and Semi-Circular Bending monotonic test (SCB) were used to assess the mechanical properties of these mixtures while Stiffness Modulus Ratio (SMR) and Long Term Oven Aging (LTOA) were used to investigate the main durability features, i.e. water sensitivity and long-term aging, respectively. Furthermore, Scan Electron Microscopy (SEM) technique and X-Ray Diffraction (XRD) analysis have been used to investigate the reasons behind the improvement in the mechanical properties of the novel mixtures.

By means of ITSM results, four high-qualities CRA mixtures have been optimised which are: CRA-WPSA (containing 6% WPSA), CRA-BBF (containing 4.5% WPSA+1.5% PLFA), CRA-TBF-1 (containing 3.75% WPSA+1.25% PLFA+1% SF) and CRA-TBF-2 (containing 3.375% WPSA+1.125%PLFA+1.5% RHA).

Stiffness modulus of CRA mixtures increases significantly by replacing the conventional mineral filler with WPSA, BBF, TBF-1 and TBF-2, especially in the early curing time (less than 7 days), which is the main disadvantage of the cold BEMs. Also, the target stiffness modulus, which is the ITSM for 100/150 HRA (approximately 2000MPa), was achieved after 4 hours for the produced fast-curing CRA mixtures, i.e. CRA-TBF-1 and CRA-TBF-2, under the normal curing method (24 hours in the mould then leave the samples at 20 ºC). In addition, the replacement of conventional mineral filler with WPSA, BBF, TBF-1 and TBF-2 greatly improves the permanent deformation resistance and fatigue life when compared with the control CRA and the traditional HRA mixtures.

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List of Abbreviations

4PB	Four-Point Bending Fatigue Test
AC	Asphalt Concrete
BBA	Biomass Bottom Ash
BBF	Binary Blended Filler
BEM	Bituminous Emulsion Mixture
BEMIX	Bitumen Emulsion cold-mixes
BSM-foam	Foamed Bitumen Stabilised mixtures
CAEC	Cement–Asphalt Emulsion Composite
CEN	European Committee for Standardization
C–H	Calcium hydrate
CRA	Cold Rolled Asphalt
C–S–H	Calcium silicate hydrate
ETB	Emulsion Treated Base
EVA	Ethyl Vinyl Acetate
FHWA	Federal Highway Administration
GEMs	Granular Emulsion Mixtures
GGBS	Ground Granulated Blast Furnace Slag
HAUC	Highway Authorities and Utilities Committee
HMA	Hot Mix Asphalt
HRA	Hot Rolled Asphalt
HVS	Heavy Vehicle Simulator
HWF	Half-Warm Foamed Mix
HWRA	Half-Warm Rolled Asphalt
IEC	Initial Emulsion Content
IRBC	Initial Residual Bitumen Content
ITS	Indirect Tensile Strength
ITSM	Indirect Tensile Stiffness Modulus
LCMT	Liverpool Centre for Materials and Technology

LD	Limestone Dust
LTOA	Long Term Oven Ageing
LVDTs	Linear Variable Differential Transducers
MSMR	Mean Stiffness Modulus Ratio
OPC	Ordinary Portland Cement
OPWwc	Optimum Pre-mixing water content
ORBC	Optimum Residual Bitumen Content
OTLC	Optimum Total Liquid Content at Compaction
PCSMs	Permanent Cold Lay Surfacing Materials
PFA	Pulverised Fuel Ash
PLFA	Poultry Litter Fly Ash
PSD	Particle Size distribution
RAP	Reclaimed Asphalt pavement
RHA	Rice Husk Ash
SBR	Styrene Butadiene Rubber
SCB	Semi-Circular Bending test
SCM	Supplementary Cementitious Materials
SEM	Scanning Electron Microscopy
SF	Silica Fume
SHRP	Strategic Highway Research Program
SMA	Stone Mastic Asphalt
SMR	Stiffness Modulus Ratio
SS	Steel Slag
STOA	Short Term Oven Ageing
STV	Standard Tar Viscometer
TBF	Ternary Blended Filler
UCCT	Uniaxial Cyclic Creep Test
VFB	Voids Filled with Bitumen
VMA	Voids in Mineral Aggregate
WPSA	Waste Paper Sludge Ash

XRF	X-Ray Fluorescence
XRPD	X-Ray Powder Diffraction

Chapter One

Introduction

A modern flexible pavement consists of a number of layers. These have various functions which contribute to the ability of the pavement to remain safe, stable and durable for a period of time and under the action of weather and a large numbers of vehicles.

The general structure on which all flexible pavements are based is shown in Figure 1-1.

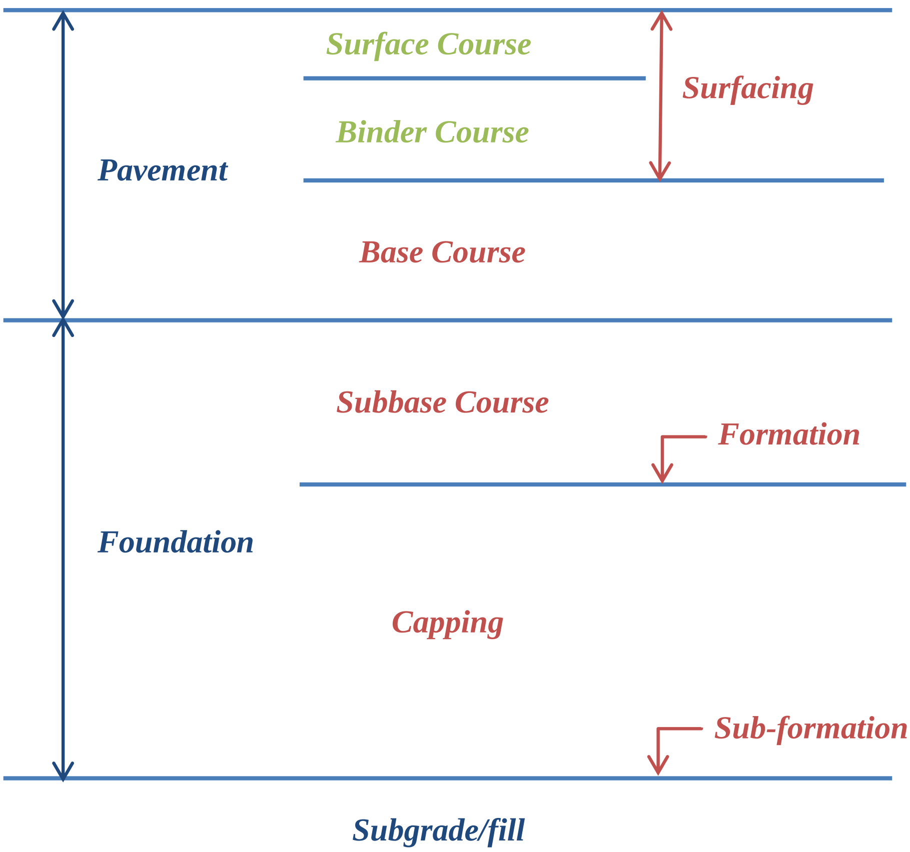


Figure 1-1: Typical flexible pavement structure

The subgrade (existing subsoil) is the naturally occurring soil upon which the road is constructed. This layer is sometimes compacted to improve its bearing capacity. The foundation is the platform upon which the more expensive layers are placed. It can be seen from the above figure that each succeeding layer is more expensive than its predecessor. The foundation carries the load-bearing layers of the pavement, namely the binder course and base course layers. A capping layer may sometimes be placed on top of the subgrade to improve the load-carrying capacity of the latter, and this is followed by the sub-base which consists of either unbound or bound materials. The layers above this form the main structural element of the pavement and are often composed of graded aggregate and binder based on various recipes. The lower load-bearing layer is termed the base course and this is followed by the surfacing layer or layers, i.e. binder course and surface course. The base course must be able to sustain the stresses and strains generated within itself without excessive or rapid deterioration of any kind. The base course might comprise hydraulically bounded granular materials. The binder course is present to allow asphalt contractors to achieve the high standards of surface regularity commonly required by current asphalt specifications. And, finally, the surface course has to provide a smooth, skid-resistant running plane having adequate resistance to the onset of defects, including most commonly deformation and cracking, to the extent that the lifespan of the layer does not fall below acceptable levels (Watson, 1989, Highway Agency, 1999a).

1.1 Hot mix asphalt and bituminous emulsion mixtures

The flexible pavement layers shown in Figure 1-1 consist of graded minerals held together by a binder. Bitumen is the more conventional binder used in construction of roads, city streets and highways. It might be noted here that the term ‘bitumen’ is used in UK pavement technical literature to describe what is termed ‘asphalt cement’ in the USA. In addition, the term ‘asphalt’ is reserved for materials containing a mixture of bitumen and graded aggregate, e.g. asphalt concrete or hot rolled asphalt. There are three primary technologies to produce asphalt, i.e. hot asphalt, cold asphalt, and warm asphalt technology (Read and Whiteoak, 2003).

Hot asphalt technology refers to the process of producing and laying asphalt at elevated temperatures using preheated bitumen, aggregate and filler. The production process consists of i) pre-heat the bitumen and the aggregate mixture, ii) mix hot bitumen with the preheated aggregate mixture in a kiln or a large blender until full coating of the aggregate is achieved. The bitumen and aggregate temperatures are in the region of 140 to 170 °C depending on the grade of the bitumen being used. The hot asphalt material is then conveyed to the laying site in insulated trucks and must be laid and compacted while still hot, at a temperature normally within the range of (110 to 140 °C). After laying and when the material has cooled to ambient temperature, the pavement can be open for traffic.

There are essentially three basic types of premixed hot bituminous surfacing materials (Highway Agency, 1999b). These are:

- A continuous grading (e.g. Asphalt Concrete AC), BS EN 13108-1: 2006;
- A rich mortar and a gap grading where the coarse aggregate does not interlock after compaction (e.g. Hot Rolled Asphalt (HRA)), BS EN 13108-4: 2006;
- High stone content, full coarse aggregate interlocking after compaction and a gap grading (e.g. Stone Mastic Asphalt SMA), BS EN 13108-5: 2006.

HRA surface course, which is under study in this work, is a gap graded mixture made from graded coarse aggregate, sand, mineral filler and bitumen (European Committee for Standardization, 2006). The mechanical properties of the mixture are dominated by the strength properties of the mortar (mineral filler, sand and bitumen). The material is extensively used for surfacing major roads in the UK because it provides a dense, impervious layer, resulting in a weather-resistant durable surface able to endure the demands of today’s traffic loads and providing good resistance to fatigue cracking.

However it may experience some weakness due to its low deformation resistance compared with other hot mix asphalt (Nichollas, 2004).

On the other hand, cold Bituminous Emulsion Mixtures (BEMs) (one of the cold asphalt technologies) for road pavements means manufacturing of asphalt using bitumen emulsion as the binder at ambient temperature. It has been widely utilised in many countries such as the USA and France, where such mixtures have been utilised since the 1970s and their scientists and road engineers have extensive experience in the performance of BEMs. Leech (1994) reported that, due to the relatively wet/cold climatic conditions in the UK, which are not favourable for the application of cold BEM in terms of the long curing process and low early strength, the use and development of BEM were not advanced.

However, the publication in the UK of "Specification for Reinstatement of Opening in Highways" in 1992 by the Highway Authorities and Utilities Committee (HAUC) allowed the use of cold laid asphalt instead of hot mixtures for reinstatement works in low volume roads and footways (Highway Authorities and Utilities Committtee, 1992).

BEMs include a large range of products, preparing procedures and laying techniques. This mixture may be made from: gap graded (such as HRA mixtures) or continuous dense graded (such as AC mixtures); virgin aggregates or Reclaimed Asphalt Pavement (RAP); cationic or anionic bitumen emulsion binder; normal emulsion or emulsion modified by polymer or solvent. Fibre and cement can also be used to enhance its properties. Also, BEM may be prepared using an asphalt plant or on site using special equipment or by hand mixing. BEM may or may not be stockpiled before final laying, spreading and laying by a) hand (in some cases or road-opening reinstatement) b) graders or asphalt pavers. BEMs might be used as structural layers in heavily trafficked base layers, low trafficked wearing courses, and as non-structural layers in surface treatment layers (National Cooperative Highway Research Program, 1975).

1.2 Advantages of using BEMs and incorporating waste materials into these mixtures

Reduction of adverse environmental impact, energy savings and safety during manufacturing and construction are the main issues encouraging the use of cold BEM instead of Hot Mix Asphalt (HMA) in the construction of roads and highways. Decreasing wastes from aggregate production processes, reducing land-filling and reducing CO_2 emissions during hot bituminous mixture production and laying are the main target schemes for the environmentally friendly processes (Thanaya, 2003). Cold BEM is one of the attractive methods of producing bituminous mixtures to tackle the mentioned disadvantages when incorporating some waste and/or by-product materials individually or collectively in these mixtures.

The advantages when adopting BEMs include but are not limited to:

1. Do not lead to gaseous emissions, which are potentially harmful to health and environment, because there is no need to heat mix materials in any steps of the production process. The CO_2 emission from cold mix manufacturing is approximately 14 % compared with hot mix asphalt. Manufacturing of the hot mixtures annually in the UK is around 30 million tonnes where approximate energy consumption is 6−7 million MWh with 1 million tonnes of CO_2 emissions (Kennedy, 1998),
2. Dust emissions are eliminated, as aggregates do not have to be dried for use in BEMs,
3. BEMs production is cheaper than conventional hot asphalt mix production. In terms of energy savings, Bouteiller (2010) reported that the manufacturing of each tonne of the cold mix needs energy equal to 13 % of the energy required for the hot mix. The said

energy saving is because of: firstly, BEMs production does not require drying and heating of aggregate mixture, while traditional HMA production requires drying and heating of aggregate mixtures to more than 140 °C as well as heating of the bitumen to a range of temperatures between 140 °C and 170 °C. Secondly, transport costs are generally higher for HMA as both raw materials and finished product have to be carried over longer distances because cold mix plants are simpler than conventional hot mix versions and therefore they are normally considered to be portable,

4. Safer to handle: skin contact is not eliminated by using BEMs and indeed they are more likely to be handled than hot or warm asphalt,
5. Offer potential improvements in performance, as the hardening of bitumen through oxidation and other processes is avoided because there is no heating process,
6. Have logistical advantages over hot mix, in that they can be stockpiled or transported over longer distances and it is not necessary to use insulated trucks for shorter journeys (Nikolaids, 1994a),
7. Virgin and recycled aggregates can be used to produce BEMs, as is the case with HMA,
8. No waste is expected from cold mixtures, as may happen with HMA when the losses in mixture temperature reach a certain unacceptable value.

The incorporation of waste and by-product materials into BEMs gives additional encouragement by supporting environmental conservation and can decrease raw materials' demand, mainly on virgin aggregate. Recently, researchers have shown an increased interest in incorporating Supplementary Cementitious Materials (SCMs) in production of BEMs in the UK and around the world. A considerable amount of literature has been published on using these materials to enhance the properties of BEMs. Three main benefits can be stated when using by-product materials in BEMs; these are:

- Upgraded mechanical properties: generally studies have reported an enhancement of ultimate strength due to the potential cementitious and/or pozzolanic properties,
- Gaining economic advantage as the cementitious and pozzolanic materials used are mostly industrial waste or by-products,
- The ecological advantage factor (Thanaya, 2003, Thanaya *et al.*, 2006, Cliff *et al.*, 2004, Al Nageim *et al.*, 2012, Al-Busaltan *et al.*, 2012).

1.3 BEMs' disadvantages and problem statement

Disadvantages are mainly due to some inherent problems associated with the performance of the pavement produced by the BEMs process which cause them to be regarded as "inferior" to conventional hot asphalt (Needhem, 1996, Thanaya, 2003, Leech, 1994) or as "Asphalt 2nd Class" as reported by Carswell (2004). Therefore, BEMs have been used for small-scale jobs such as reinstatement works and for works in remote areas with low to medium traffic conditions.

There are some concerns with using BEMs, as stated by the previous studies and applications. The major problems with this kind of application are the long curing time (evaporation of trapped water) required to achieve the maximum performance and the poor early life strength (because of the existence of water). The Chevron Research Company conducted laboratory and field studies to assess the performance of BEMs in California. They stated that the full curing in the field of these mixtures may occur between 2–24 months depending on the mixture's ingredients and weather conditions (Leech, 1994). High air voids for the compacted BEMs are reported to be another concern. There are also other concerns regarding BEMs, like: insufficient coating percentage because of the incompatibility between the aggregates and emulsion, binder drainage during storage because of the low viscosity of the emulsion, and binder stripping from the aggregate due to high water sensitivity and weak adhesion (Thanaya *et al.*, 2009, Leech, 1994).

1.4 Aims and objectives of the research

Considering the above disadvantages, the author – with his supervisory team and after a healthy discussion with Liverpool Centre for Materials and Technology (LCMT) industrial partners – decided to investigate possible ways of developing a new BEM/s with gap graded mixtures similar to the conventional HRA gradation which is suitable for surface course heavily trafficked pavements. The mentioned new product is termed throughout this study as Cold Rolled Asphalt (CRA). Although extensive research has been carried out on producing different types of BEMs, no single study exists which deals with producing a gap graded BEM suitable for heavily trafficked surface courses using standard slow setting cationic bitumen emulsion and incorporating SCMs individually or collectively. The new CRA is required to remove the currently imposed restriction on the use of cold asphalt by road engineers due to their poor early life strength, long curing time required and inherent high volume of voids.

The aim of this study is two-fold: 1) to investigate the possible ways of producing high strength, fast curing and sustainable CRA mixtures for heavily trafficked road and highway surfacing layers by i) using waste and by-product materials (normally used as SCM) separately or collectively as a replacement for conventional mineral filler and ii) applying different curing techniques such as microwave technique and curing of CRA mixtures at temperatures higher than the ambient temperature; and 2) providing an understanding of the early strength and stiffness development of the novel CRA mixtures by conducting SEM and XRD analysis.

To achieve the above aim, the development of the new CRA will incorporate the preparation of innovative mixtures and different curing techniques together with a comprehensive study to evaluate the potential optimisation of the use of different waste materials such as Pulverised Fuel Ash (PFA), Poultry Litter Fly Ash (PLFA), Waste Paper

Sludge Ash (WPSA), Silica Fume (SF), Rice Husk Ash (RHA) and Ground Granulated Blast Furnace Slag (GGBS) in terms of increasing the new CRA strength and reducing its curing time. The economic potential as well as the environmental benefit in terms of reducing carbon emissions and hazards to health, energy savings and recycling of waste materials will also be covered throughout this research work. Also, besides the production of the new high-quality CRA mixtures, the thesis will include a detailed comparison study of conventional HRA mixtures, CRA mixtures containing conventional mineral filler and CRA mixtures containing hydraulic filler, i.e. Ordinary Portland Cement (OPC).

Throughout this research work, the research aim will be achieved by the following objectives:

1. Carry out a detailed research review on: i) conventional HRA properties including testing and improvements achieved when additives are added to the bitumen and/or mixtures, ii) conventional Cold Mix Asphalt (CMA) and especially on BEMs manufacturing and construction technology, and iii) the available ways of improving the performance of BEMs in terms of using OPC, different virgin and waste materials as well as using different preparation techniques.
2. Investigate detailed chemical, mineralogical and physical properties of the prospective candidate wastes and by-products to replace conventional filler in CMA such as PFA, PLFA, WPSA, SF, RHA, APC and GGBS. Conduct a comparative study in terms of chemical properties of the candidate waste materials and critical analysis of their prospective uses as hydraulic and/or pozzolanic activators substituting the conventional mineral filler individually or collectively.
3. Technical development of high-quality CRA surface course mixtures using:
   a. Candidate filler resulting from (2) above as a replacement for filler; mechanical properties in terms of stiffness modulus will be covered in this part of the study,

b. Optimisation of CRA mixtures in (a) above,

c. Applying different preparation and curing techniques for enhancing the engineering properties of the newly developed CRA. The development of surface course CRA products in the lab will be based on the traditional Marshall compaction method together with curing of the samples at a temperature slightly more than the ambient temperature as well as an application of microwave technique to the CRA mixtures before compaction to minimise the material's curing time after laying, and allow opening of the road for traffic after construction within an acceptable duration.

4. Undertake a fundamental study of steady state permanent deformation behaviour of produced mixtures.
5. Evaluate the performance of the produced CRA mixtures in terms of fatigue life (crack initiation) using four-point load fatigue test and fatigue fracture using semi-circular bending monotonic test.
6. Evaluate the effect of replacement of the conventional mineral filler with the new fillers (Unary, Binary and Ternary) on the water sensitivity of the developed mixtures including a comparative study with the conventional HRA and CRA containing OPC as well as with the required standards.
7. Explain the reasons behind the properties enhancements of the new CRA using XRF, XRD and SEM analysis.
8. Investigate pre-compaction application of microwave conditioning of the CRA mixtures to improve the porosity property of these mixtures with regard to mechanical properties.
9. Writing the PhD thesis.

1.5 Thesis layout

The thesis comprises 10 chapters.

Chapter 1: covers briefly i) the details of traditional flexible pavements, ii) a description of the HMA and BEMs, iii) the main advantages and disadvantages of using BEMs, iv) aims and objectives of the research work, and v) justification of the reasons for the research work.

Chapter 2: details stage I of the literature review on bitumen emulsion.

Chapter 3: presents stage II of the literature review on bitumen emulsion mixtures.

Chapter 4: discusses the research methodology, candidate SCMs properties and experimental technique followed in the present laboratory study.

Chapter 5: shows a sequential optimisation process by means of stiffness modulus results to produce novel CRA mixtures. Also, the performance of these mixtures at different curing and testing temperature has been investigated.

Chapter 6: discusses the performance of the optimised CRA mixtures in terms of other mechanical properties, namely creep, fatigue and fracture toughness performance.

Chapter 7: details the durability of novel CRA mixtures by investigating water sensitivity and long-term oven-aging properties.

Chapter 8: covers an understanding of the progress of new CRA mixtures' strength by means of SEM observations and XRD analysis.

Chapter 9: introduces a new Half-Warm Rolled Asphalt by applying microwave heating in a pre-compaction method.

Chapter 10: presents the concluding summary of this study including recommendations for further works.

Chapter Two

Background Study I: A Review of Bitumen Emulsion

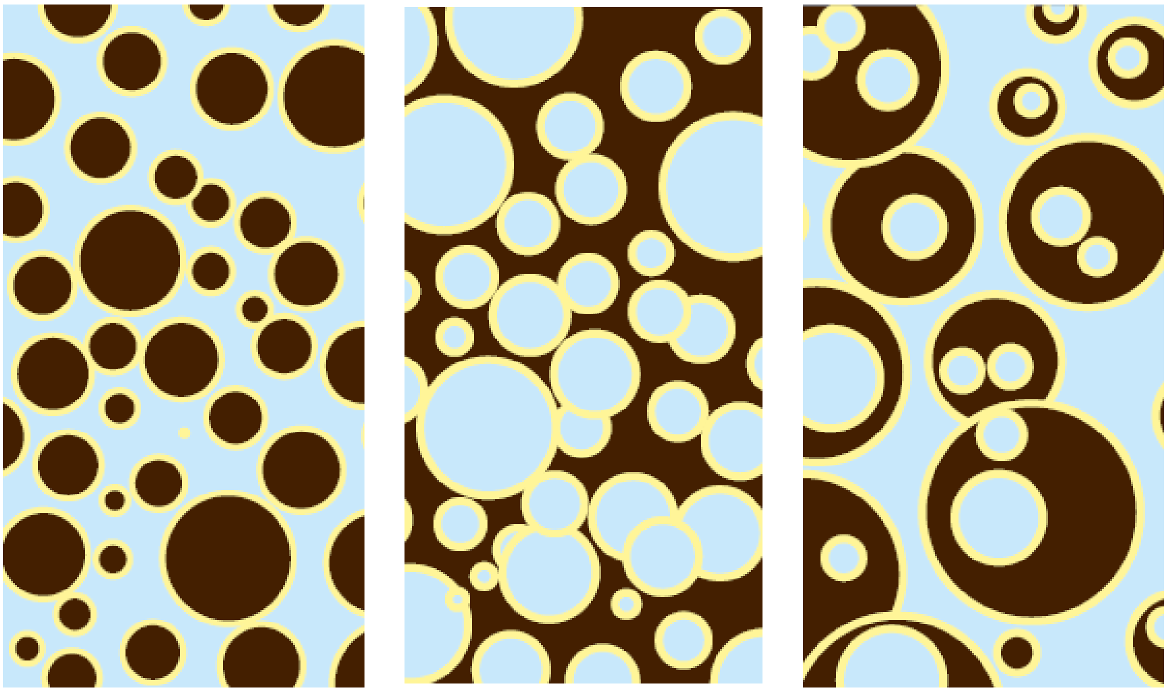
Cold Bituminous Emulsion Mixtures (BEMs) are primarily composed of bitumen emulsion as a binder and graded mineral aggregates that can be mixed and compacted with no need to heat, i.e. at ambient temperature. In this chapter, a detailed description of bitumen emulsion is provided.

2.1 Bitumen emulsion

An emulsion is a two-phase system that represents a dispersion of fine droplets of one fluid in another fluid. Emulsions can be formed by any two immiscible fluids but in most emulsions one of them is water (Akzonobel, 2008).

Akzonobel (2008) and James (2006) classified emulsions into three general groups: oil-in-water (O/W), water-in-oil (W/O) and multiple phase emulsions. O/W emulsions are those in which the dispersed phase is an oily liquid and the continuous phase is water. Conversely, W/O emulsions are those in which the continuous phase is oil and water represents the dispersed phase. Sometimes W/O emulsions are called inverted emulsions. Also, Akzonobel (2008) indicated that multiple phase emulsions can be formed in which the dispersed droplets themselves contain smaller globules of a third phase, commonly the same liquid as the continuous phase. Figure 2-1 illustrates the three broad groups of emulsions.

Bitumen emulsions are often of the oil-in-water group. The first phase in bitumen emulsion is bitumen droplets (dispersed phase as they are discrete droplets), while the second phase is water (continuous phase) (James, 2006). Needhem (1996) stated that, in contrast to solutions, the two liquids are coexistent rather than mutually mixed. The existence of the electrostatic charges given by an emulsifier and stabilised by stabiliser is the main reason to hold these globules in suspension.



a. Oil-in-water emulsion   b. Water-in-oil emulsion   c. Multiple emulsion

Figure 2-1: Emulsion types: (a) O/W emulsion, (b) W/O emulsion, and (c) multiple emulsions (Akzonobel, 2008), permission to reproduce this figure has been granted by AkzoNobel N.V.

Normally, the diameter of the bitumen droplets in bitumen emulsion is in the range of 0.1 to 20 microns, Figure 2-2. Bitumen emulsions comprising from 40 % – 80 % bitumen are brown fluids with consistencies ranging from that of milk to heavy cream (Akzonobel, 2008). The bitumen content within the emulsion depends on the proposed use of the emulsion. The upper limit of bitumen content depends on the relative volume of the two phases. There is insufficient room (adjacent to the upper limit) for more bitumen droplets without deforming them; therefore, the droplets may coalesce to each other.

Bitumen can be dispersed in water when it is distributed into fine droplets with the aid of chemical emulsifier (Asphalt Institute and Asphalt Emulsion Manufacturers Association, 1997); Figure 2-3 shows a micrograph of bitumen emulsion.

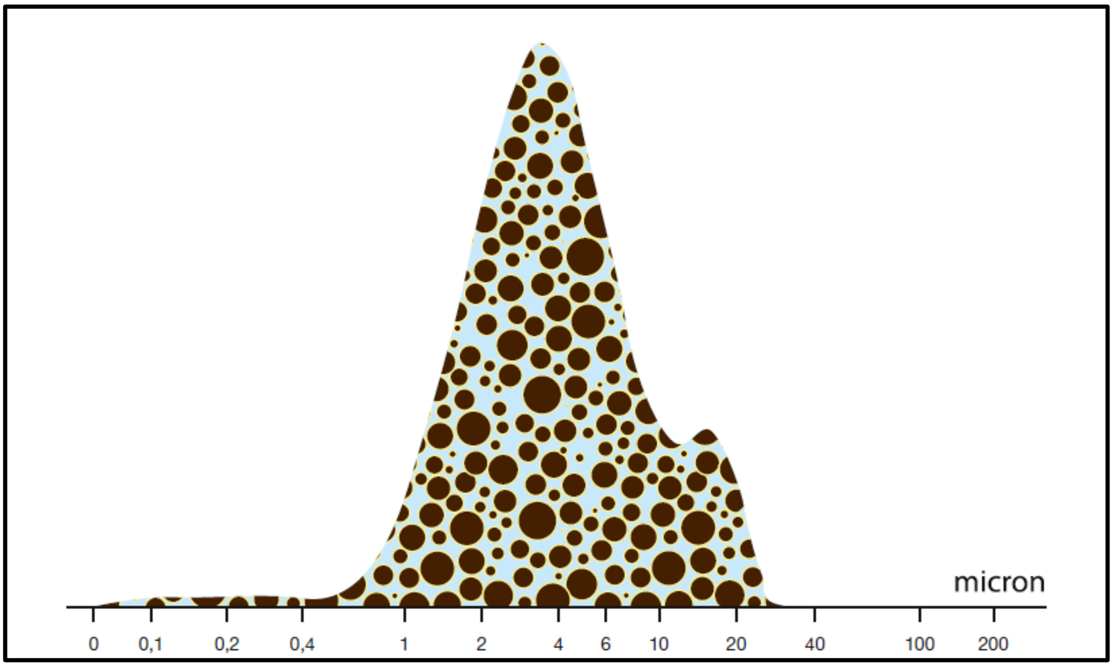


Figure 2-2: Typical particle size distribution of bitumen emulsion droplets (Akzonobel, 2008), permission to reproduce this figure has been granted by AkzoNobel N.V.

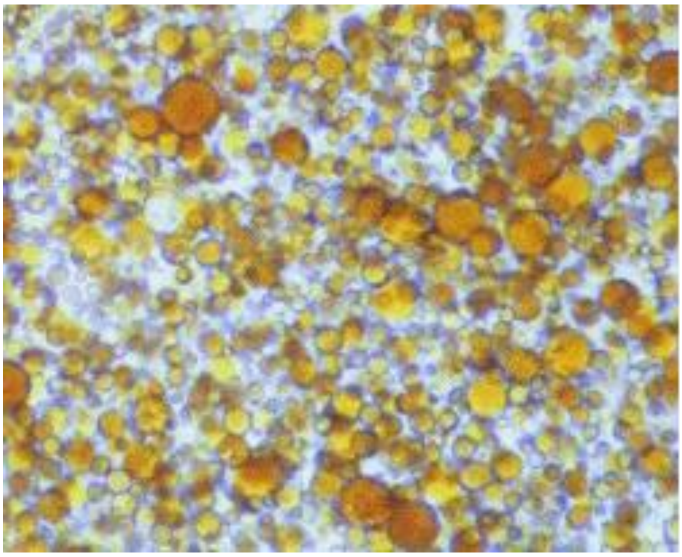


Figure 2-3: Micrograph of bitumen emulsion (James, 2006), permission to reproduce this figure has been granted by the Transportation Research Board

2.2 Constituents of bitumen emulsion

2.2.1 Bitumen and water

Bitumen is generally non-polar, even though it does comprise some polar components, and represents a very complex substance. The term “non-polar” means that there are no regions of charge concentration or deficiency as the electron distributions in the component molecules being regularly spread all over the structures. On the other hand, the consistency of different ionic types such as H_3O^+, OH^- and H^+ and the polar water molecule itself leads to the very polar medium for water. The polarity of the H_2O molecule is due to the existence of the very electronegative atom, i.e. oxygen, and the hydrogen, which is very electropositive (Needhem, 1996). Water and oil, such as bitumen, are completely immiscible under normal circumstances due to the chemistry of the two substances. If an attempt is made to mix bitumen and water, the two substances will separate as the molecules in the polar medium (water) prefer to be in contact with each other in order to cancel out positive and negative areas of charge.

2.2.2 Surfactant

Surfactant or emulsifier is a surface active agent necessary to produce a homogenous emulsion of two immiscible substances (such as bitumen and water) because their molecules concentrate and are active at the surface between these substances. A homogenous emulsion can be found as the emulsifier aids the dispersion of bitumen and keeps the bitumen droplets in permanent suspension. An emulsifier is commonly comprised of long hydrocarbon chains that terminate in either cationic or anionic functional sets. An absorbed film of emulsifier is formed around each bitumen globule in the emulsion because of the presence of an emulsifier. This absorbed film works as a protective coating and can resist the coalescence of the dispersed bitumen droplets.

Emulsifiers are water-soluble materials whose presence in a solution markedly changes the properties of the solvent and the surface they contact (Asphalt Institute and Asphalt Emulsion Manufacturers Association, 1997). They are classified according to the way they disperse or ionise in water. The emulsifier molecules possess a balance of a long lipophilic (oil loving), hydrocarbon tail and a polar hydrophilic (water loving) head, Figure 2-4.

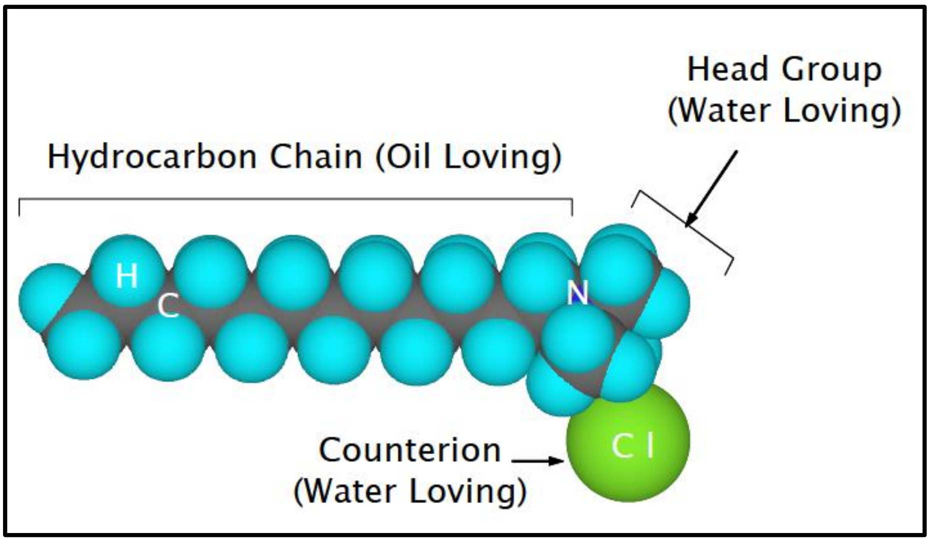
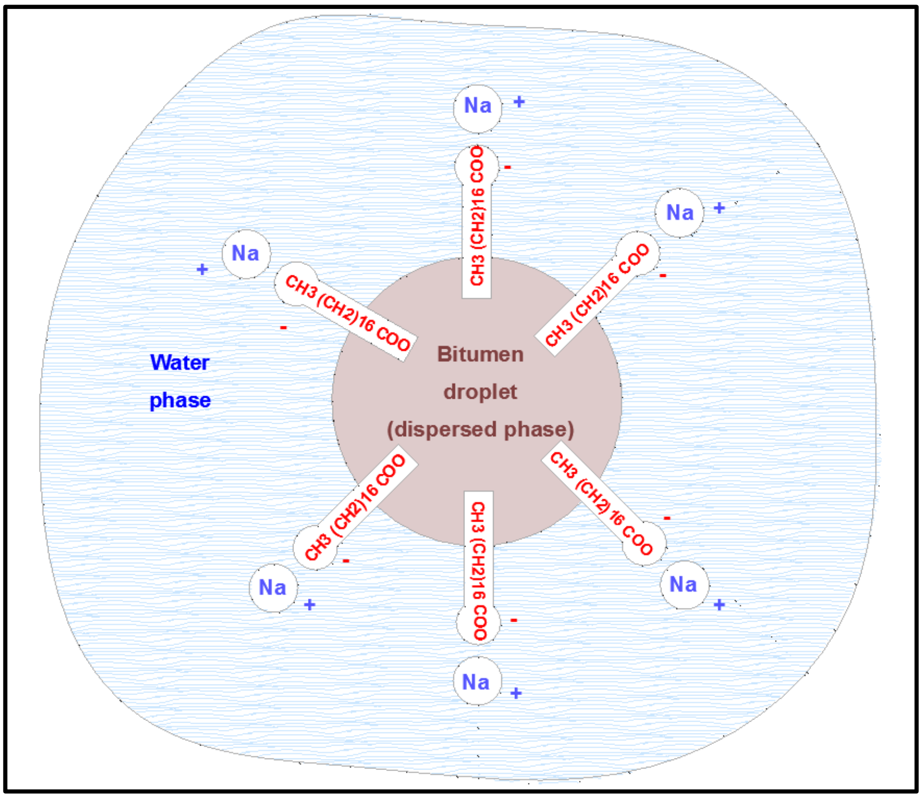


Figure 2-4: Structure of emulsifier (James, 2006), permission to reproduce this figure has been granted by the Transportation Research Board

The pH value of the system controls the type of electrical charge of the emulsifier. There is no possibility of mixing emulsions that are produced with anionic and cationic surfactants causing coalescence of the bitumen droplets. Accordingly, there are four primarily types of emulsifiers, namely: anionic, cationic, non-ionic, and colloidal. Anionic emulsions have a negative electrical charge and alkaline system (pH > 7). On the other hand, cationic emulsions have a positive electrical charge and acid system (pH < 7) (Thanaya, 2003).

Anionic emulsifiers are principally fatty acids, alkyl sulphates or sulphonates so they are normally alkaline solutions. One of these emulsifiers is sodium stearate; $CH_3(CH_2)16COONa$, which disassociates in water into stearate anions $CH_3(CH_2)16COO^-$

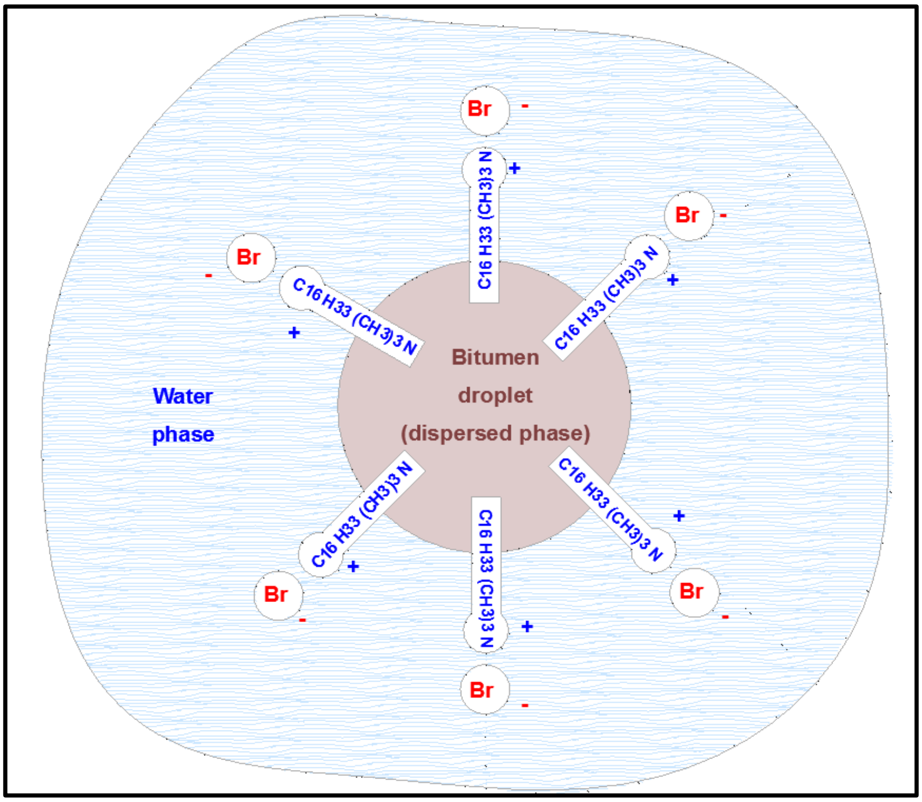
and sodium cations Na^+. Films of ions with negative charges on the surface of the bitumen droplets will be formed due to the ability of stearate anion to become soluble in bitumen with the carboxylic group that conveys the negative charge. Then, ‘electrical double layers’ are formed when positively charged ions are attracted to the negatively charged ions, Figure 2-5.



Anionic Emulsifier: Sodium Stearate: CH3 (CH2)16 COO Na
Dissociates in water and becomes:
► Sodium Cation, Na^+, in water (continous phase)
►Stearate Anion (large portion), CH3 (CH2)16 COO^-, in the bitumen particles (discontinuous phase)

Figure 2-5: Diagrammatic demonstration of the arrangement of anionic emulsifier around dispersed bitumen droplets.

Cationic emulsions have become the preferable type in most road applications, after first appearing in the middle of the twentieth century. Cationic emulsifiers are principally acidic solutions categorised by ‘cationic organic portions’ such as cetyl trimethyl-ammonium bromide $C_{16}H_{33}(CH_3)_3NBr$. This emulsifier dissociates into the positive cation $C_{16}H_{33}(CH_3)_3N^+$ and the negative anion Br^- when mixed with water, see Figure 2-6.



Cationic Emulsifier: Cetyl Trimethyl-Ammonium Bromide: $C_{16}H_{33}$ $(CH_3)_3$ N Br
Dissociates in water and becomes:

- ► Bromide Anion, Br^-, in water (continuous phase)
- ►Cetyl Trimethylammonium Cation, (large portion), $C_{16}H_{33}$ $(CH_3)_3$ N^+, in the bitumen particles (discontinuous phase).

Figure 2-6: Diagrammatic demonstration of the arrangement of cationic emulsifier around dispersed bitumen droplets.

The third type of emulsifiers (non-ionic) can be utilised in making esters and ethers which are necessary for acids and alcohol production. These emulsifiers are seldom used for manufacturing of road emulsions and they are not ionised.

Lastly, colloidal emulsifiers are frequently used for industrial purposes but not for road emulsions.

2.2.3 Other components

Several materials can be incorporated to enhance the properties of the produced bitumen emulsion such as calcium or sodium chloride, adhesion promoters, solvent and latex. The former is added to the emulsion to decrease the osmosis of water into the bitumen droplets due to the existence of salt in bitumen, and to minimise viscosity changes (James, 2006). Adhesion promoters, which are commonly surface active amine composite, can be incorporated into the bituminous mixtures or to the finished emulsion to overcome the insufficient adhesion between the curd bitumen film and aggregate; while solvents can be added to decrease emulsion settlement, enhance curing rate at low temperature, improve emulsification and/or provide the precise viscosity of bitumen after curing. Latex is a water-based dispersion of polymer which is mainly appropriate for emulsions' enhancement. The most conventionally utilised polymers in paving grade bitumen emulsions are Styrene Butadiene Rubber (SBR), polychloroprene, and natural rubber latex.

2.3 The manufacture of bitumen emulsion

Bitumen emulsions are usually produced utilising colloid mills. A colloid mill has two main parts: the first part is the rotor and the second one is the stator; the former revolving at about 1000 – 6000 rpm in a stator. Energy is applied to the system in the colloid mill by passing the mixture of hot bitumen and water phase between a rotating disc and stator. In order to create a turbulent flow, the rotor and stator might be grooved (Akzonobel, 2008).

Water phase and hot bitumen are fed under control temperatures individually but simultaneously into the colloid mill; the process is highly affected by the temperature of the two constituents. The temperature range of the hot bitumen entering the colloid mill is 100 – 140 ºC as the required viscosity should not exceed 0.2 Pa s (2poise). Also, the temperature of the water phase is adjusted to produce an emulsion with a temperature less than 90 ºC and avoid water boiling. As the bitumen and water phase enter the colloid mill they are exposed to extreme shearing forces that cause the bitumen to break into small droplets. Due to the resulting electrostatic forces there is no ability for droplets to coalesce (Read and Whiteoak, 2003).

Special components and technical solutions are required when different special additives like latex or SBS are recommended to be used. For example, SBS modified bitumen often requires the emulsion to be produced beyond the boiling point of water, which needs production under pressure and cooling before release to normal pressure in the storage tank (Akzonobel, 2008).

2.4 Classification and naming of bitumen emulsions

Bitumen emulsions can be classified into four classes based on the sign of the charge on the droplets. The first two are the most widely used:

- Anionic emulsions,
- Cationic emulsions,
- Non-ionic emulsions, and
- Clay-stabilised emulsions.

If an electrical potential is applied between two electrodes submerged in an emulsion comprising negatively charged particles of bitumen, they will move to the anode. In that case, the emulsion is termed as ‘anionic’. On the contrary, in a system comprising positively charged particles of bitumen, they will migrate to the cathode and the emulsion is termed as ‘cationic’, which is under the consideration of this study. For the non-ionic emulsion, the bitumen particles are neutral and will not move to either pole. These kinds of emulsions are seldom used in road applications. Clay-stabilised emulsions are not utilised for highways but for industrial applications. In these emulsions, fine powders, often natural or processed clays and bentonites, are used as emulsifiers. For these emulsions the particle size is much less than that of the bitumen droplets in the emulsion (Read and Whiteoak, 2003).

Also, bitumen emulsions can be classified according to their reactivity when in contact with aggregates or pavement surfaces. Consequently, there are three main types: rapid, medium and slow setting. Rapid setting emulsions principally are suitable for spray application such as surface dressing (the chippings used in chip seals) because they have little or no ability to mix with aggregates and they set quickly in contact with clean aggregates of low-surface area. Medium setting emulsions are mainly used for mixtures that mostly contain coarse aggregates with little or no fine particles such as those used in open graded mixes because they set sufficiently less quickly. The last type, i.e. slow setting emulsions, remain workable up to a certain time when mixed with aggregate particles, therefore are aiding a sufficient degree of coating for the production of mixtures (Thanaya, 2003). Generally, slow setting emulsions are unreactive so they are more suitable with reactive aggregates; conversely, rapid setting emulsions are reactive and are utilised with unreactive aggregates. Additionally, the technique and materials being used and the environmental conditions are the main factors affecting the actual setting and curing time in the field (Asphalt Institute and Asphalt Emulsion Manufacturers Association, 1997).

Accordingly, the Asphalt Institute and Asphalt Emulsion Manufacturers Association (1997) classified emulsion types by a series of numbers. Cationic emulsions are symbolised by the letter “C” in front of the emulsion type and the absence of “C” denotes anionic. The numbers in the classification specify the relative viscosity of the bitumen emulsion. In this case “1” means less viscous than “2”. Basically, the “s” that follows certain grades indicates that a softer base bitumen is used, whereas, “h” means a harder base bitumen. The “HF” previous used in some of the anionic grades means’ high float emulsion. Mainly, these grades are utilised for cold and warm mixes, seal coats and road applications, see Table 2-1.

Table 2-1: Typical classifications and uses of bitumen emulsions (Asphalt Institute and Asphalt Emulsion Manufacturers Association, 1997), permission to reproduce this table has been granted by Asphalt Institute and Asphalt Emulsion Manufacturers Association

Type of Construction	**ASTM D977 AASHTO M208**									**ASTM D2397 AASHTO M140**					
	RS-1	**RS-2**	**HFRS-2**	**MS-1, HFMS-1**	**MS-2, HFMS-2**	**MS-2h, HFMS-2h**	**HFMS-2s**	**SS-1**	**SS-1h**	**CRS-1**	**CRS-2**	**CMS-2**	**CMS-2h**	**CSS-1**	**CSS-1h**
Asphalt-Aggregate Mixtures															
Plant mix (Hot or Warm)						×[A]									
Plant mix (cold)															
Open graded aggregate					×	×						×	×		
Dense-graded aggregate							×	×	×					×	×
Sand							×	×	×					×	×
Mixed-in-place															
Open graded aggregate					×	×						×	×		
Dense-graded aggregate							×	×	×					×	×
Sand							×	×	×					×	×
Sandy soil							×	×	×					×	×
Asphalt aggregate applications															
Single and multiple surface treatments	×	×	×							×	×				
Sand seal	×	×	×	×						×	×				
Slurry seal							×	×	×					×	×
Micro surfacing															×[E]
Sandwich seal		×	×								×				
Cape seal		×									×				
Asphalt application															
Fog seal				×[B]				×[C]	×[C]					×[C]	×[C]
Prime coat					×[D]			×[D]	×[D]					×[D]	×[D]
Tack coat				×[B]				×[C]	×[C]					×[C]	×[C]
Dust palliative								×[C]	×[C]					×[C]	×[C]
Mulch treatment								×[C]	×[C]					×[C]	×[C]
Crack filler								×	×					×	×
Maintenance mix															
Immediate use							×					×	×		
Stockpile							×								

A Grades other than HFMS-2h may be used where experience has shown that they give satisfactory performance
B Diluted with water by the manufacture
C Diluted with water
D Mixed-in prime only
E Polymer must be added during or prior to emulsification

2.5 Breaking mechanism of bitumen emulsion

Bitumen emulsion must revert to a continuous bitumen film in order to act as binder in road materials. This includes flocculation and coalescence of the droplets and removal of the water. The speed of the coalescence process is often termed the breaking or setting rate of an emulsion. The main setting mechanism for very slow-setting emulsions may happen due to evaporation and absorption of water by the aggregate, but in most cases it is not necessary for all the water to evaporate before curing takes place because the chemical reactions between the aggregate and the emulsion contribute to the emulsion breaking. In many cases, the strength of the chemical reaction of emulsion with aggregate is enough to squeeze the water from the system. James (2006) stated that the breaking and curing processes are influenced by the reactivity of emulsion, the reactivity of aggregate and environmental factors, such as humidity, temperature, mechanical action and wind speed. Softer (less viscosity) base bitumens tend to give earlier coalescence. It may take several weeks in the case of a dense cold mix to a few hours in the case of a chip seal for the ultimate strength of the road application to be achieved.

2.5.1 Breaking mechanism of cationic bitumen emulsion

Wates and James (1993) stated three methods to explain the breaking mechanism of the cationic bitumen emulsion on the siliceous aggregates which are negatively charged, such as granite and quartzite, these are:

2.5.1.1 Emulsifier abstraction

In this situation, the emulsifier is taken out (withdrawn) from the bitumen-water interface by the aggregate surface. The bitumen becomes unstable due to the loss of emulsifier, leading to droplets' coalescence, Figure 2-7.

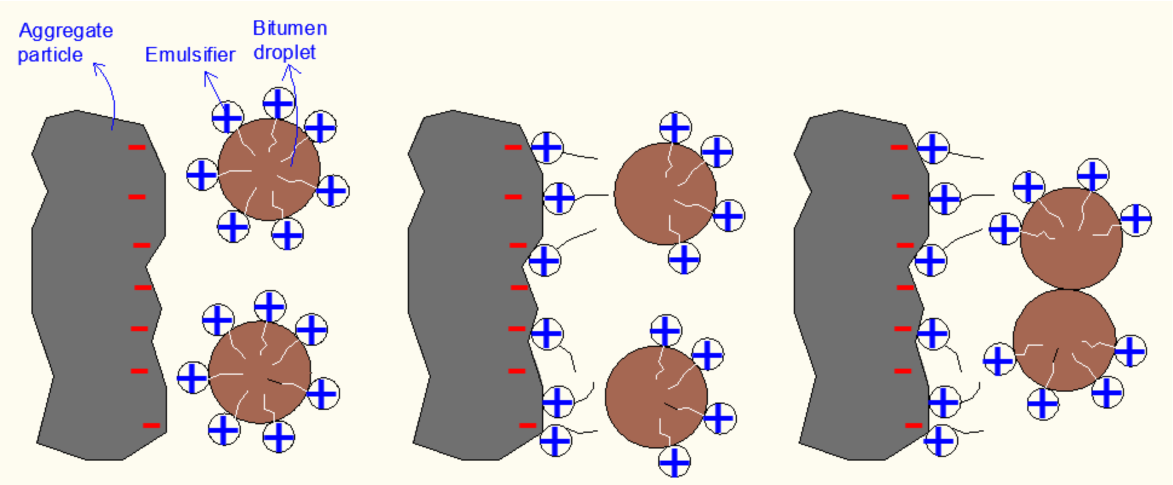


Figure 2-7: Breaking mechanism of cationic bitumen emulsion- emulsifier abstraction

2.5.1.2 Emulsifier Deprotonation

A second case comprises deprotonation of the acidified emulsifier in which the protons are adsorbed onto basic sites on the aggregate surfaces, deactivating the emulsifier then causing the emulsion to break, Figure 2-8.

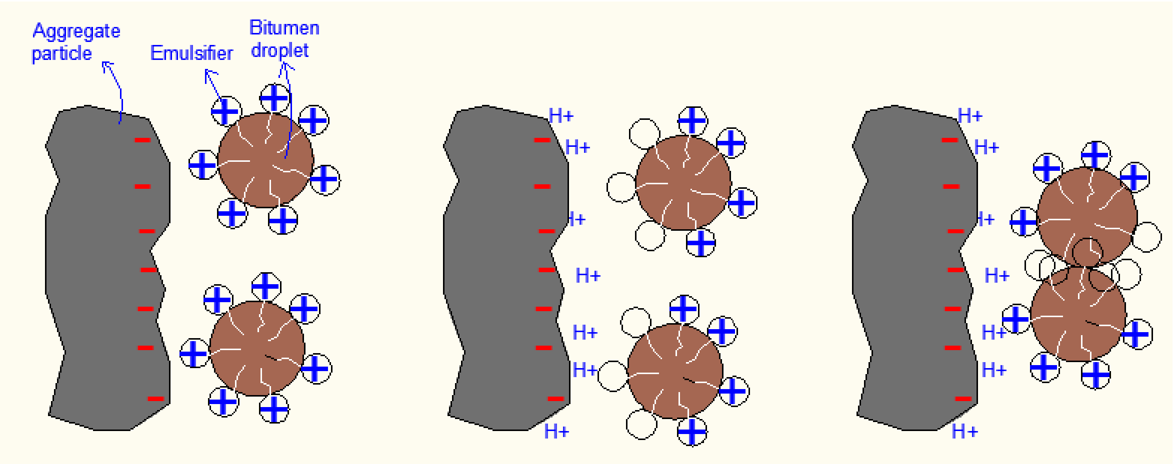


Figure 2-8: Breaking mechanism of cationic bitumen emulsion- emulsifier deprotonation

2.5.1.3 Droplet migration

The last case is considered to be the most important one and is termed the droplet migration process. Due to existence of emulsifiers on their surfaces, the positively charged bitumen droplets are attracted to the negatively charged aggregate surfaces. Then the bitumen droplets spread over the aggregate surfaces, aided by the emulsifier. Also, the surfactant acts as an anti-stripping or adhesion agent because the bitumen is bound to the surface by the surfactant molecules, Figure 2-9. This is the main reason why cationic emulsifiers have become more widely used than anionic ones in road construction applications.

Needhem (1996) stated that possibly all of the above three cases occur during the breaking of a bitumen emulsion to a greater or lesser extent, related to the present system's conditions.

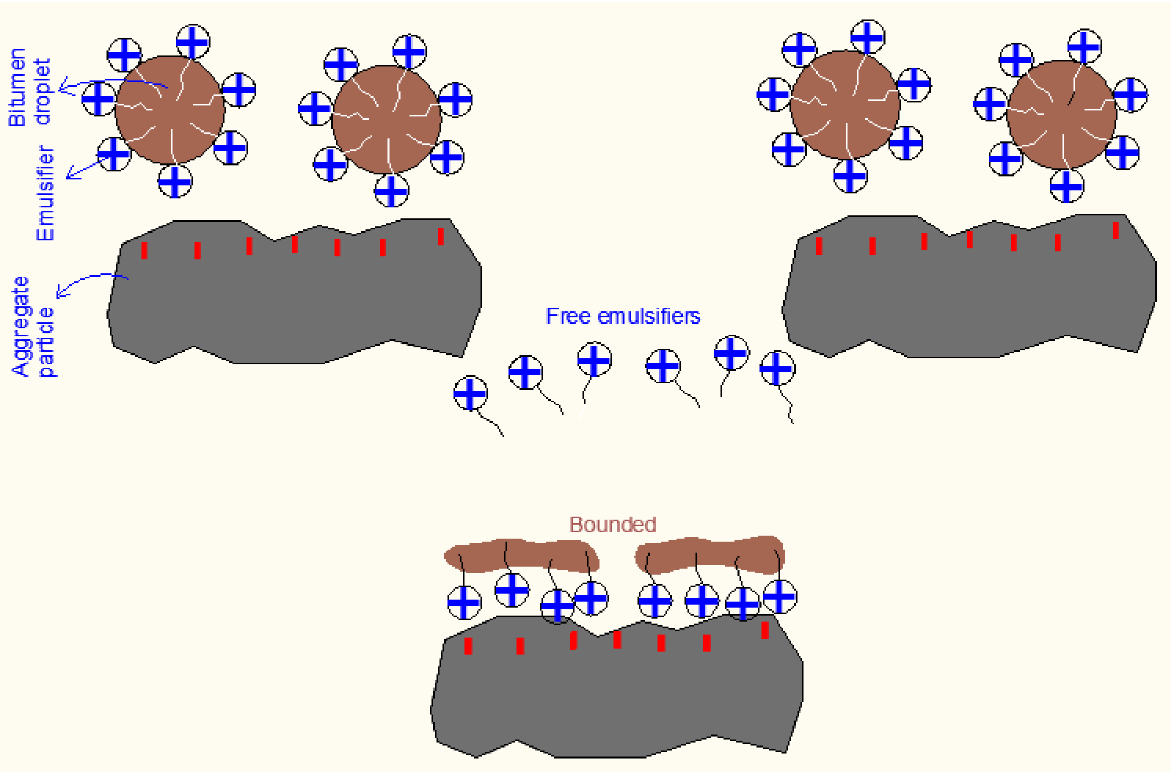


Figure 2-9: Breaking mechanism of cationic bitumen emulsion- droplet migration

Plotnikova (1993) introduced another theory on the breaking mechanism of cationic emulsion. In the case of siliceous aggregates such as granite and quartzite, he claimed that primarily the free emulsifiers are adsorbed onto the surfaces of aggregate. Then the emulsifiers are withdrawn from the bitumen droplets and adsorbed onto the aggregate surfaces which become hydrophobic (oil loving) due to adsorption of emulsifiers, as shown in Figure 2-10. The setting rate is proportional to the rate of the emulsifier's adsorption onto the aggregate, which is related to the surface area and chemical nature of the bitumen emulsion and the aggregate.

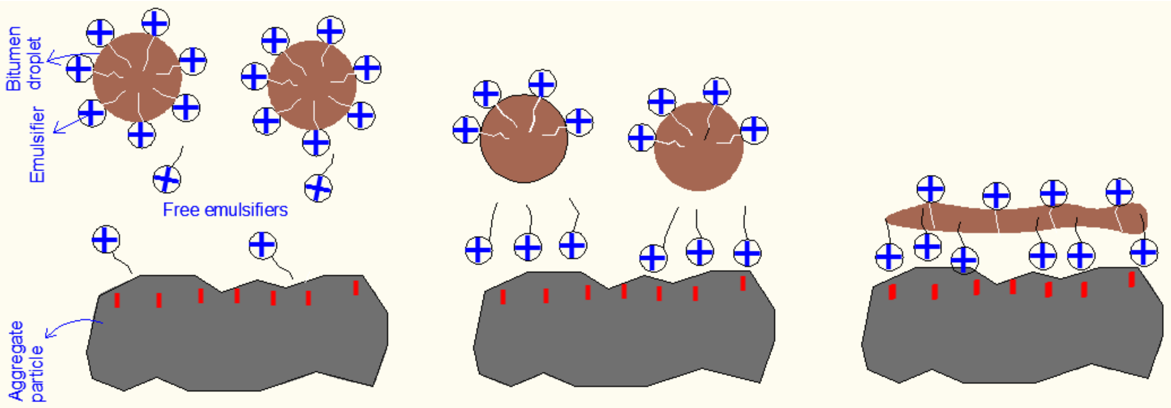


Figure 2-10: Breaking mechanism of cationic bitumen emulsion- emulsifier adsorption

2.5.2 Particle size and emulsion breaking

The breaking behaviour of an emulsion is affected by the particle size distribution of the binder droplets. The finer the emulsion, the larger the surface area at the binder water interfaces. There are two ways by which the emulsion behaviour is affected. Firstly, in terms of chemical reaction, an increase in surface area will increase the number of reaction sites thus speeding up the breaking process. Secondly, in terms of physical action, the larger surface area increases the capacity for surfactant adsorption at the bitumen water interface. On the contrary, if there is freer surfactant in the system, this will use up the active sites on an aggregate surface hence slowing down the breaking process according to the mechanism illustrated above.

If the binder is prevented from breaking onto the aggregate, the bitumen droplets coalesce away from the aggregate, leading to poor binding of the total mixture. This can result in poor adhesion, poor resistance to fatigue and ravelling. Otherwise, if the level of free surfactant is low, a good adhesion matrix will be formed due to the breaking of the bitumen globules onto the aggregate surfaces.

2.5.3 Influence of pH changes in breaking mechanism

Some types of calcareous aggregate such as limestone, lime filler or cement can neutralise the acid in cationic emulsion and raise the pH value. This can cause destabilisation of the bitumen emulsion. In other cases, a slower rise in the pH, but sufficient enough to destabilise the emulsion, may take place due to hydrogen ions' adsorption by the aggregates. On the other hand, some soluble aggregates such as limestone can provide calcium or magnesium ions to the solution which tend to neutralise the charges of anionic bitumen emulsions.

2.6 Characterisation and specification of cationic bitumen emulsion

Historically, in the UK, BS 434-1 defined the nomenclature and specifications of bitumen emulsions. For anionic emulsions, revised BS 434-1 (British Standard Institution, 2011) is still in use for anionic bitumen emulsion, but the definition of the grades of cationic bituminous emulsions has been replaced with the European Standard BS EN 13808 (European Committee for Standardization, 2013b).

BS EN 13808 identifies the requirements for performance characteristics of cationic bituminous emulsion classes which are suitable for use in the construction and maintenance of roads, airfields and other paved areas. It classifies emulsions in a more detailed way than BS 434-1, utilising up to seven characters, on the basis presented in Table 2-2. Thus, for example, a C70 B2 emulsion is a cationic bitumen emulsion with a 70 % nominal binder content, produced from paving grade bitumen (conforming to BS EN 12591) with a class 2 breaking value. Similarly, a C60 BPF6 emulsion is a cationic bitumen emulsion with a nominal binder content of 60 % containing polymer and flux and having a class 6 breaking value.

Table 2-2: Denomination of the abbreviation terms (European Committee for Standardization, 2013b), permission to reproduce this table has been granted by BSI Group

Position	Letters	Denomination	Supporting European Standard
1	C	Cationic bituminous emulsion	EN 1430 (particle polarity)
2 and 3	2 digits number	Nominal binder content in % (m/m)	EN 1428 (water content) or EN 1431 (recovered binder + oil distillate)
4, or 4 and 5, or 4,5 and 6	 B P F	Indication of type of binder Paving grade bitumen Addition of polymers Addition of more than 2 % (m/m) of flux based on emulsion	EN 12591 (specification for paving grade bitumen) EN 14023 (specification framework for polymer modified bitumens)
5 or 6 or 7	1 to 7	Class of breaking behaviour	EN 13075-1 (breaking value) (Classes 1 to 7)

EN 13808 developed as a framework specification, which includes a number of classes for each property of cationic bitumen emulsions to bring together all the existing CEN member country specifications into one standardised specification to apply throughout Europe. Also, all characteristics of cationic bituminous emulsions shall be classified in accordance with the appropriate parts of Tables AI-1, AI-2 and AI-3.

Below are the main cationic bitumen emulsion characterisations.

2.6.1 Particle charge

The particle charge is indicated according to BS EN 1430 in which a positive electrode (anode) and a negative electrode (cathode) are placed in the emulsion. Deposition of a layer of the bitumen on the cathode indicates positive polarity and that the emulsion is cationic, and vice versa (European Committee for Standardization, 2009a).

2.6.2 Breaking value

This property indicates the rate of breaking of the bitumen emulsion. Reference filler is added at a uniform rate to a specified quantity of stirred cationic bitumen emulsion. When the emulsion has broken completely, the amount of added filler is determined by weighing. The mass of filler (in grams) multiplied by 100 and divided by the amount of emulsion (in grams) is the breaking value (European Committee for Standardization, 2009d).

2.6.3 Binder content

Cationic bitumen emulsion contains from 38 % to more than 70 % of binder content. The binder content and base bitumen’s penetration grade is often varied to suit the particular use. The binder content can be determined by means of water content in accordance with BS EN 1428 (European Committee for Standardization, 2012b) or by distillation method in accordance with BS EN 1431 (European Committee for Standardization, 2009b).

2.6.4 Emulsion viscosity and efflux time

The efflux time is an indirect measure of the viscosity of bitumen emulsion and is also referred to as “pseudo-viscosity”. It is determined using an efflux viscometer known as the Standard Tar Viscometer (STV), which determines the time of efflux of a 50 ml sample through a 10 mm, 4 mm or 2 mm orifice at a specific temperature, i.e. 25, 40 or 50 ºC, depending on the emulsion type. Whatever temperatures or orifice diameters used, the efflux time shall not exceed 600 s (European Committee for Standardization, 2011a).

Bitumen emulsion viscosity should be low enough to ensure that the emulsion can coat aggregates sufficiently at ambient temperature. Otherwise, it should be sufficiently high, so that the binder does not drain when applied (Thanaya, 2003).

2.6.5 Residue on sieving

The sieve residue test as per BS EN 1429 is designed to indicate the percentage of binder above a certain sieve size, normally 0.5 or 0.16 mm (European Committee for Standardization, 2013a). For acceptable emulsion the results should comply with the requirements shown in appendix I (Table AI-1). The bitumen particles in the emulsion system tend to sediment because they are slightly heavier than water, particularly larger size particles. Therefore, this property affects the stability of the emulsion, which depends on the percentage of the oversize particles in the emulsion.

2.6.6 Settling tendency

The term “settling tendency” refers to the difference in water content of the top layer and the bottom layer of a prescribed volume of sample after standing for a specified time at ambient temperature. BS EN 12847 specifies a method for the determination of the settling tendency of bituminous emulsions (European Committee for Standardization, 2009c). In this test, the sample is allowed to stand for a specific time, e.g. 7 days at normal atmospheric temperature in a stoppered graduated cylinder, after which the water contents of the top and bottom layers are determined either as per EN 1428 or EN 1431. Then the settling tendency is calculated as the difference between the two water contents. When the emulsion is stored, it should not separate and should re-disperse into a homogenous condition by agitation. For the emulsions that are supplied in drums, the drums should be rolled or inverted at least once a month.

2.6.7 Adhesivity

The qualitative assessment of the measurement of the adhesion (the ability of a binder to coat the surface of an aggregate and to remain bonded over time in the presence of water) is called adhesivity. According to BS EN 13614 European Committee for Standardization (2011b), the bituminous emulsion is mixed thoroughly with the considered aggregate under specified conditions. To indicate the immediate adhesivity, the mixture is immediately washed under running water and the percentage of the aggregate surface covered with bitumen is indicated visually under specified circumstances. On the other hand, when testing water effect on binder adhesion, the mixture is first left to cure and then immersed under water under specified circumstances. The percentage of the aggregate surface covered with binder is indicated visually under specified circumstances.

2.6.8 Properties of the residual bitumen

The binders recovered by evaporation from cationic bituminous emulsions can be tested for their penetration grade, softening point, dynamic viscosity, etc. The properties should meet the requirements illustrated in Table AI-2.

2.7 Inversion of bitumen emulsion

The dispersion of an aqueous phase in a non-aqueous phase is called emulsion inversion. This can occur within the breaking process of bitumen emulsion. This means that the system inverts from being O/W, i.e. bitumen dispersed in water, to W/O, i.e. water dispersed in bitumen. Water loss (evaporation) from the O/W emulsion is the main reason for this phenomenon. At some point there will not be enough water to separate the bitumen droplets. Further water losses will end up in a situation where the water will be dispersed in a continuous oil phase, hence creating an emulsion inversion. At this condition, water will find it difficult to escape from the bituminous mixture as it is sealed off by binder droplets. Also, this situation can affect the softening of the bitumen, causing reduction in the mixture's strength (Needhem, 1996).

2.8 Application of bitumen emulsions

Bitumen emulsions' versatility makes them suitable for different applications. The Road Emulsion Association Ltd published a range of data sheets on emulsions' applications (Road Emulsion Association Ltd, 2013). In addition to utilising bitumen emulsion in cold BEMs (see Chapter 3), it has been used in different areas such as crack filling, grouting, tack/bond coats, slip layers and concrete curing, protective coats, soil stabilisation, surface dressing (chip seal), penetration macadam and slurry seals (Read and Whiteoak, 2003, Thanaya, 2003).

Chapter Three

Background Study II: A Review of Bitumen Emulsion Mixtures

A bituminous mixture is a combination of bituminous materials (as binders) and graded aggregates with or without additives. Since tar has been rarely used in bituminous mixtures in recent years, bitumen is the predominant binder material used. The term “asphalt” is now more commonly used to denote a combination of bituminous materials, aggregates and additives. 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 traffic and environment actions. Asphalt used in pavement applications are usually classified by their methods of production, composition and characteristics.

In terms of composition classification, bituminous mixtures can be classified mainly as dense, gap, open or uniform graded mixtures, see Figure 3-1. Dense or well graded refers to a gradation that is near the Federal Highway Administration’s (FHWA’s) 0.45 power curve for maximum density. The most common hot mix asphalt designs worldwide tend to use dense graded aggregate. Typical gradations are near the 0.45 power curve but not right on it. Gap graded refers to a gradation that contains only a small percentage of aggregate particles in the mid-size range. The curve is flat in the mid-size range. Open graded refers to a gradation that contains only a small percentage of aggregate particles in the small range, i.e. passing sieve number 2.36 mm. This results in more air voids because there are not enough small particles to fill in the voids between the larger particles during compaction of HMA. The curve is near vertical in the mid-size range and flat and near-zero in the small-size range, see Figure 3-1. Lastly, uniformly graded refers to a gradation that contains most of the particles in a very narrow size range i.e. all the particles are of the same size. The curve is steep and only occupies the narrow size range specified.

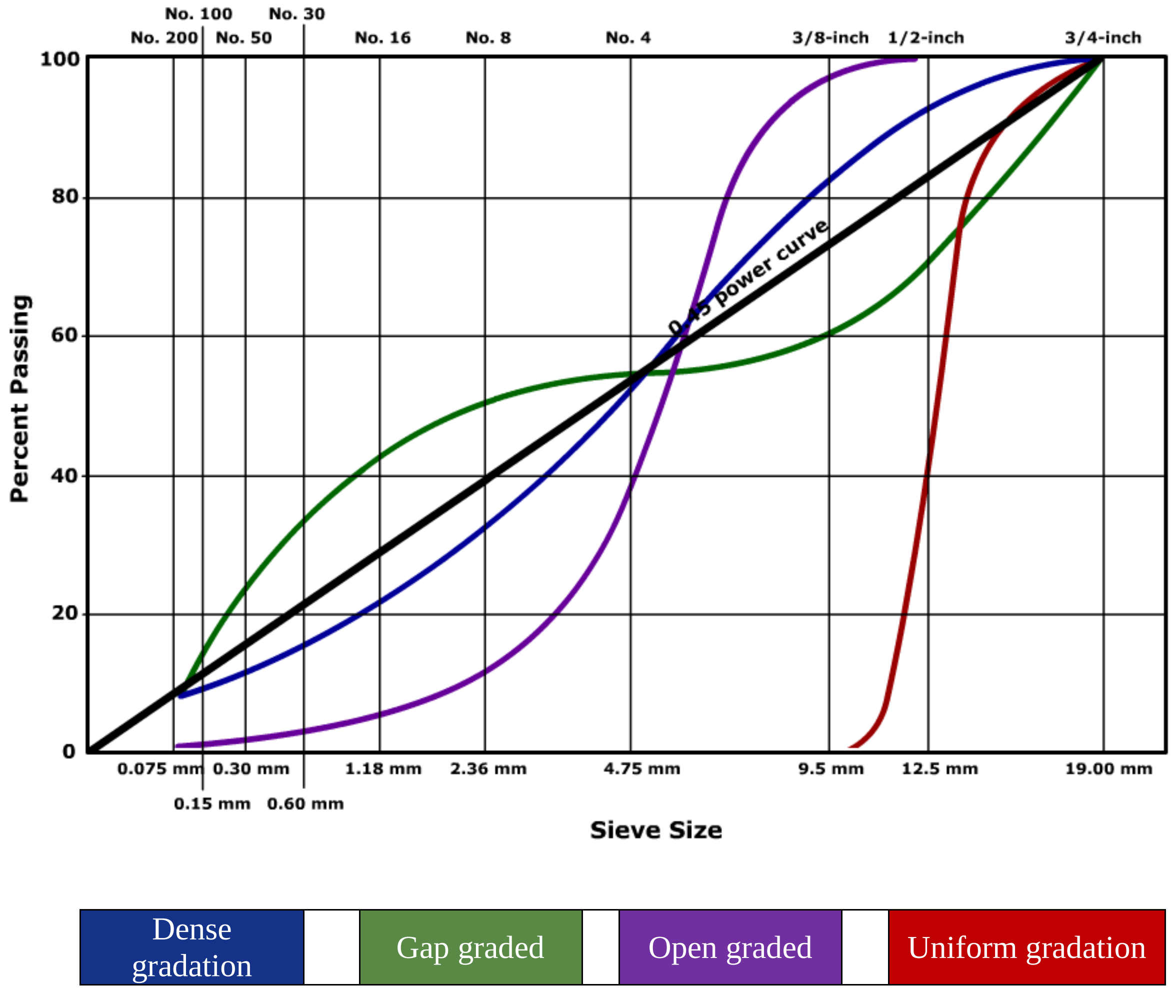


Figure 3-1: Federal Highway Administration (FHWA) gradation graph showing representative gradations (Pavement Interaction, 2013), permission to reproduce this figure has been granted by Pavia Systems, Inc.

Several different types of bituminous mixtures are utilised throughout the world, and in 1990 Venton (1990) stated that there were over 350 recipe-based mixture designs at that time in use in the UK alone. These mixtures have been produced to achieve certain requirements in terms of load-bearing capacity, durability, surface texture, permeability, etc. Recently, there have been moves to rationalise the methodology of mix design on a performance basis. This means that the mechanical properties of the material need to be fully known and utilised to design a pavement that will withstand predicted traffic levels for the desired period of time. The enhancements to current practice can improve mixture performance but implementation of the findings is in its early stages, as stated by Brown and Dawson (1992) and Brown *et al.* (1991).

3.1 Technologies involved in the production of bituminous mixtures

At ambient temperature, straight run penetration grade bitumens suitable for pavement applications generally have high viscosity, i.e. are relatively hard semisolids. There are different common ways to increase the workability and thus decrease the highly viscous bitumen into low viscosity liquid suitable for asphalt. Accordingly, there are three main techniques used to produce bituminous mixtures at different mixing and applying temperatures, these are hot asphalt, warm asphalt and cold asphalt technology, see Figure 3-2. More information on these techniques can be found in the following subsections, with more emphasis on cold asphalt since it is the focus of this study.

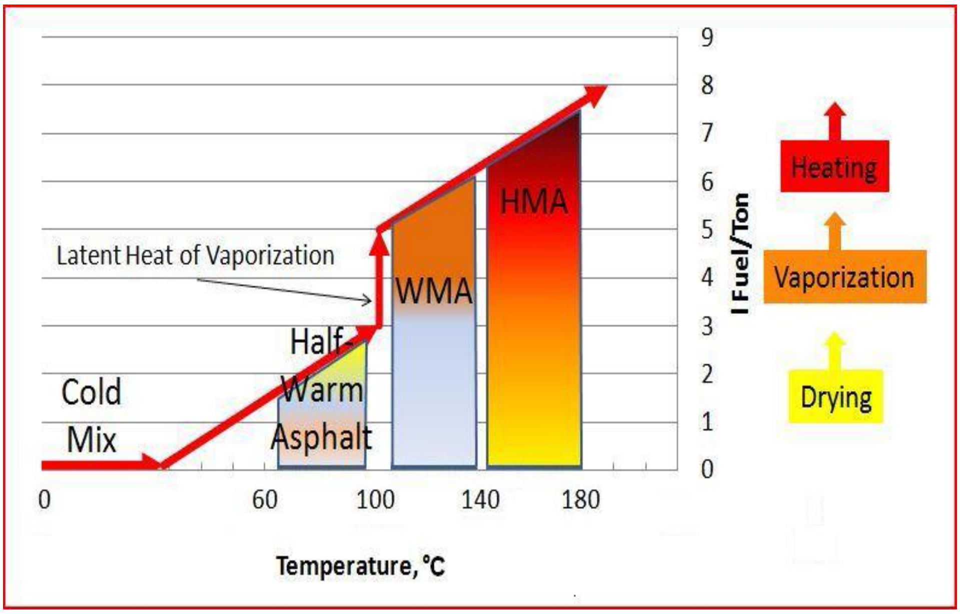


Figure 3-2: Different mixtures' production techniques

3.1.1 Hot mix asphalt technology

Hot asphalt mix refers to the techniques used to produce bitumen-aggregate mixtures at elevated temperatures (140 ºC to 180 ºC). In these mixtures, the compacting and mixing temperatures are determined according to the relationship between viscosity and temperature of the specified bitumen used in the mixture. In the production process, firstly, heat the bitumen and the aggregate particles; secondly, add hot bitumen to the preheated aggregate particles in a kiln or a large blender; thirdly, mix thoroughly until full coating of the aggregate is achieved. The bitumen and aggregate temperatures are in the region of the temperature depending on the grade of the bitumen being used. The hot asphalt material is then conveyed to the laying site in insulated trucks and must be laid and compacted while the mixture is still at the acceptable temperature range in the specification. After laying and when the material has cooled to ambient temperature, the pavement can be trafficked (Read and Whiteoak, 2003).

Mix designs for the hot bituminous mix have been well established over the last century. Three main mix design methods were developed; these are: Marshall, Hveem and Super-pave (Abbas and Ali, 2011). Presently, hot bituminous mix asphalt is the most extensively used technique to produce the materials for road pavements.

3.1.2 Warm mix asphalt technology

Warm mix asphalt technology comprises two main techniques depending on the production temperature. These are half-warm mix and warm mix asphalt technique.

3.1.2.1 Half-warm mix asphalt

The development of half-warm asphalt by using foamed bitumen as a binder happened after understanding the impact of aggregate temperature on the engineering properties of the cold mixes. Although the aggregates are heated to temperatures in the range between

75 ºC and 90 ºC and not more than 100 ºC, half-warm mixes can be described as cold mixes because they are produced in the same manner.

Jenkins (2000) stated that one of the main advantages of utilising half-warm foamed asphalt mixes is the improvement of aggregate coating. Figure 3-3 shows the relationship between aggregate temperature and aggregate coating for different maximum particle sizes of continuously graded aggregate. Three different coating regions are defined, namely practically no coating, partial coating and complete coating. The practically no coating region characterises 20 % or less particle coating, while the partial coating region characterises a partial coating range between 21 and 99 %, and the complete coating region characterises particles that are 100 % coated.

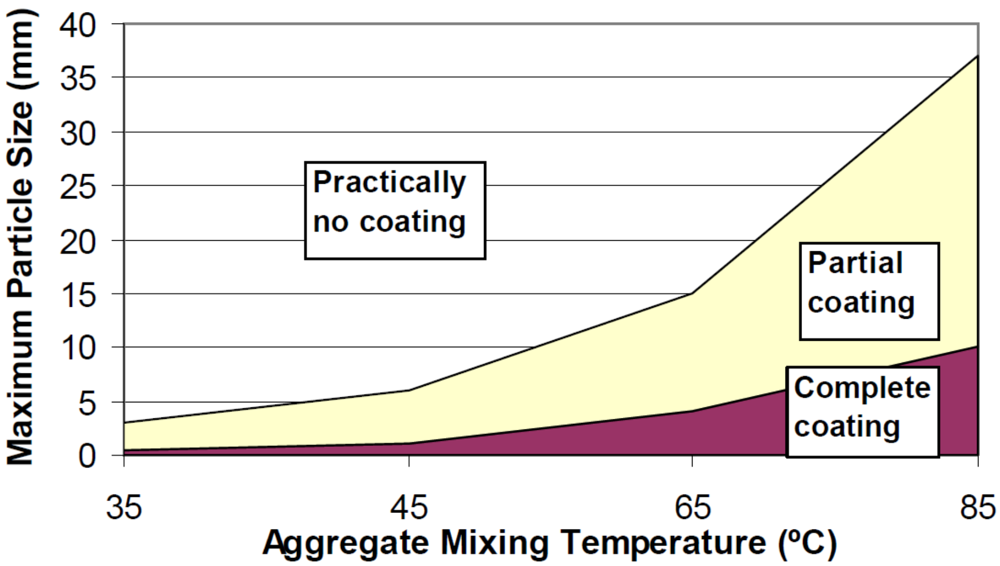


Figure 3-3: Particle coating in half-warm mixes (Jenkins, 2000), permission to reproduce this figure has been granted by Professor Kim Jenkins

3.1.2.2 Warm mix asphalt

This group of techniques are called warm mix asphalt and are utilised to produce bituminous mixtures at mixing and compacting temperatures lower than the conventional hot bituminous mixtures. The idea behind producing warm mix asphalt is to reduce bitumen viscosity either by addition of organic or chemical additives or by introducing cool water into the heated molten bitumen under controlled conditions of temperature and pressure (foamed bitumen). Sasobit, Asphaltan-B, Asphamin, Evotherm and WAM-Foam are examples of the available warm mix asphalt technologies (Abbas and Ali, 2011).

Using one of the warm mix asphalt technologies instead of traditional hot mix asphalt has several benefits, one of which is the reduction in mixing and compaction temperatures. D'angelo *et al.* (2008) reported other advantages including: decreased fuel and energy consumption, decreased emissions and odours from plants, decreased smoke, enhanced working conditions at the site, thus improving the quality of the work as well as the workers' productivity, longer hauling distances and extending the paving season. An overview of some warm mix asphalt techniques are given in the next paragraphs.

Sasobit®: is a synthetic wax that is manufactured in the coal gasification process. The melting point of these materials is in the range between 85 ºC and 115 ºC; therefore it is often blended with bitumen at temperatures above 115 ºC. Sasobit reduces the viscosity of the binder at temperatures above 115 ºC and helps improve the stability at ambient temperatures because it forms a crystalline structure within the bitumen binder at ambient temperatures. The optimum amount of Sasobit to be incorporated is about 3–4 % by weight of the bitumen as reported by the manufacturer, which in turn would result in reducing the production temperatures by approximately 8–30 ºC. Additionally, blending the solid with the bitumen during mixing is not recommended because it will result in an inhomogeneous distribution of Sasobit within the mix; hence it is blended within the hot bitumen stream to ensure homogenous distribution (Jamshidi *et al.*, 2013).

Asphaltan-B is a refined montan wax that is blended with a fatty acid amide. It is produced by solvent extraction of certain types of lignite or brown coal. It helps in decreasing the viscosity of bitumen binder at temperatures higher than its melting point, which is between 82 ºC and 99 ºC (Abbas and Ali, 2011).

Asphamin is a synthetic zeolite that contains about 20 % by weight of water of crystallisation. Asphamin addition to the asphalt is different from the previous techniques, i.e. Sasobit and Asphaltan-B, as it is added to the mix shortly after or at the same time as adding the bitumen binder into the mixer. The water contained inside its structure will be released when subjected to the higher temperatures. Due to the gradual release of water (in the form of steam), the bitumen starts to foam and its viscosity is decreased, making it suitable to produce asphalt mixes at lower production temperature. The addition of 0.3 % of Asphamin by total weight of the mixture is recommended by the manufacturer, Eurovia Service GmbH Germany. Also, they stated that this addition provides about 6 hours or more of enhanced workability if the mixture's temperature does not fall below 100 ºC. Furthermore, the manufacturer promotes that Asphamin can decrease the mixing temperature by between 20 ºC and 30 ºC, resulting in up to 30 % energy savings (Rubio *et al.*, 2012).

WAM-Foam was developed by Shell International Petroleum, UK and Kolo-Veidekke, Norway. This technique comprises a two stage-mixing process in which two different bitumen grades are introduced. In the first stage, a very soft bitumen binder is mixed with the heated aggregates at temperatures between 99 ºC and 121 ºC; hence completely coating the aggregate surfaces and ensuring that there is no ability for the aggregate to absorb any of the water used in foaming the hard bitumen. In the second stage, very hard bitumen is foamed by introducing cold water and then mixing with the pre-coated aggregate. The manufacturer reported that 30 % energy saving can result from using WAM-Foam processes to produce bituminous mixtures, which in turn can lead to a 30 % reduction in CO_2 emissions (Abbas and Ali, 2011). Foamed bituminous mixture is produced by introducing air and cold water into the heated bitumen binder in specially designed nozzles. Heat transfers from the hot bitumen to the cold water during the mixing of them, causing evaporation of the water, which in turn causes the bitumen to foam. Figure 3-4 illustrates an example of the laboratory foaming nozzle manufactured by Wirtgen, Inc.

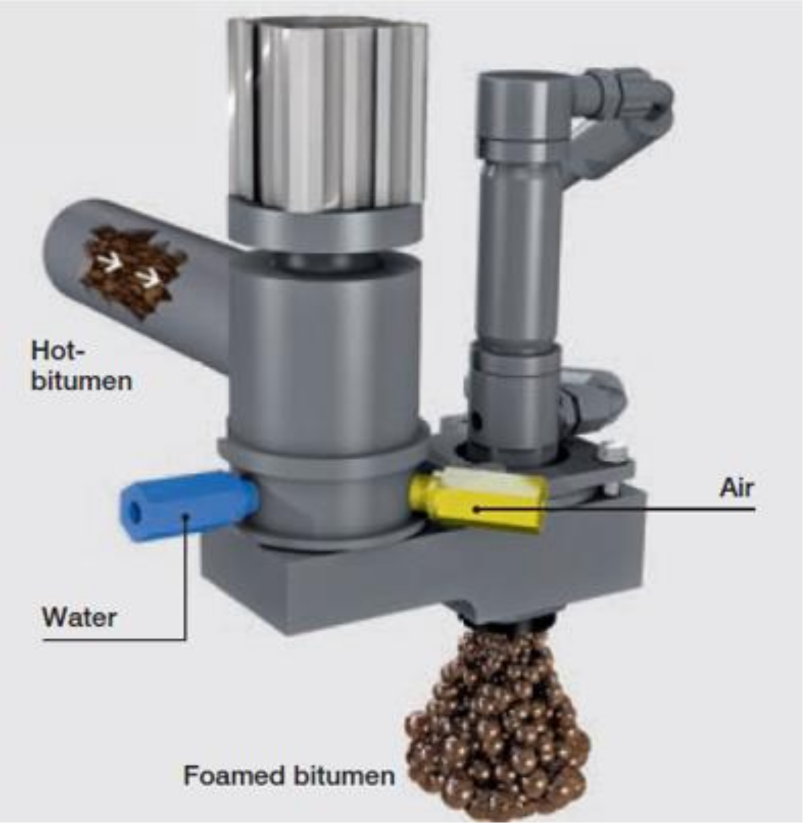


Figure 3-4: Bitumen foaming process (Wirngten Gmbh, 2001), permission to reproduce this figure has been granted by Wirtgen GmbH

3.1.3 Cold mix asphalt technology

There are several methods to achieve the required workability of bitumen at low temperatures, i.e. decreasing bitumen viscosity; namely by using: cut back bitumen produced from mixing the bitumen with flux oil, foamed bitumen produced from foaming process of the hot bitumen with cold water, and bitumen emulsion produced from emulsification of bitumen emulsion with water. This research study is focused on the application of cold mixtures produced using bitumen emulsion as binder.

A considerable amount of literature has reported that cold BEMs have benefits in terms of simplicity in production, being environmentally friendly as they produce almost no gas emissions into the atmosphere during production, and energy savings.

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

The most commonly used types of cold mix asphalts as listed by Thanaya (2003) are:

- Cold lay macadam (cutbacks),
- Grave emulsions (developed in France),
- Foamed bituminous mixtures, and
- Cold Bituminous Emulsion Mixtures (BEMs).

3.1.3.1 Cold lay macadam

Cold lay macadam is mixture of aggregate and cutback bitumen binder which is produced by adding a solvent or flux oil to the bitumen to reduce its viscosity. The solvent is a relatively non-volatile portion of petroleum utilised as a diluent to soften bitumen to a desired consistency. The performance of cutback bitumens depends on the solvent's evaporation through application and service; therefore the volatility of the solvent and the climatic conditions are the main factors affecting the performance of cold lay macadam. Consistency of cutback bitumen relies on the amount of flux oil.

Different types of solvents are available, namely: white sprit, kerosene, gasoil and may be a combination of them (Nichollas, 2004).

Surface dressing and macadam mixtures are the main applications of cutback bituminous mixtures. These mixtures can also be applied in reinstatement as impermanent fill materials. Generally, temporary reinstatements are applied to reduce potential delays and possible accidents, which in turn provide convenience for road users. Then these temporary mixtures are replaced by permanent reinstatement materials represented by hot asphalt. This is due to low stiffness values of cold lay bitumen macadam for cored samples because of the presence of flux oil (Robinson, 1997). In addition, the flux oils are generally expensive, flammable, and can pollute the environment. Several types of permanent cold lay macadam have recently been produced which utilise low viscosity modified bitumens and flexibilisers, which in turn reduce the brittleness of the base bitumen.

In the UK, cutback bitumens are specified and designated by the flow time (in seconds) through a Standard Tar Viscometer (STV). Three grades are available: 50, 100 and 200 s. The majority of cutback is used in surface dressing (Read and Whiteoak, 2003).

3.1.3.2 Grave emulsion

Grave Emulsion (GE) refers to the emulsion-stabilised aggregates' application. It was primarily developed in France and it was not specified until 1974 (Needhem, 1996). In this process, the bitumen emulsion is mixed with aggregate which is pre-wetted with water. In France, GE is used in the dryer and warmer areas due to the water sensitivity of these mixtures. However, since 1988, relatively large GE applications have been used in considerably wetter climatic conditions, and such contracts have met with a degree of success. GE can be an economical alternative to traditional hot bituminous mixtures, particularly in areas where there is a long distance to the nearest hot mix plant.

The process can be used in different applications:

- As base courses or wearing courses, using continuously graded virgin aggregate mixtures generally containing between 5 and 10 % passing 75 µm sieve.
- As strengthening and re-profiling light traffic roads.
- Overlaying cement base courses to prevent crack propagation.

The mixtures can be stockpiled for many days when a little quantity of flux oil is incorporated, then used for patching and re-profiling. The stiffness modulus of the produced mixtures is low because the bitumen becomes softer after flux oil incorporation, hence takes a longer time to cure.

The aggregate gradation utilised in GE normally is continuously dense gradation. The optimum design has a high sand content to provide high interlock between aggregate particles during curing and good surface texture. It is also important to minimize the susceptibility to rutting which can happen if bituminous/filler mastic overfilled the voids; therefore low filler content is preferred. This procedure indicated a very high maximum void content of 15 % compared with other densely graded mixtures. The aggregate may be either gravel or crushed rock with 10 mm, 14 mm, 20 mm or 31.5 mm nominal maximum size.

The bitumen content originally was only 3–3.5 % in the GE mixtures; therefore the partial coating of the aggregate is the most noticeable feature of these mixtures. Low bitumen contents were incorporated initially in order to promote a high level of aggregate contact to maximise internal interlock which resists permanent deformation.

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.

Traditionally, GE is followed with a surface dressing some days after laying to seal the surface and prevent ravelling (Akzonobel, 2002).

Satisfactory results have been reported from using GE in Ireland, despite the fact that core samples could only be taken about 2 years after compaction (Leech, 1994). Grave mixtures are usually prepared utilising cationic medium setting emulsions with 60–65 % residual bitumen content, i.e. may comprise some fluxing oil. Shortly after compaction, GE roads can be loaded with normal traffic. The trapped water within the compacted mixtures will evaporate gradually; therefore the full strength of these mixtures is achieved when all of this water has evaporated (Thanaya, 2003).

3.1.3.3 Foamed bitumen

In this process, foamed bitumen is used as binder to produce the cold bituminous mixtures, which is generated by mixing hot bitumen with water plus surfactant. The volume of the bitumen is expanded to fifteen times and forms foam due to the rapid boiling of the water. The surfactant works as a stabilising agent to the produced foam. When the said foam is incorporated with an aggregate mixture, it will give a high degree of coating due to the increased volume of the binder.

3.1.3.4 Bituminous emulsion mixtures

Bitumen Emulsion Mixtures (BEMs) are produced at ambient temperatures utilising bitumen emulsion as binder. However, some procedures can use warm emulsion up to around 60 °C. Although there is no need to dry the aggregate, the water content must be controlled because it strongly affects the performance of the produced mixtures. The main role of the pre-mixing water is to prevent premature breakage of the emulsion. Then the bitumen emulsion is gradually added to the pre-wetted aggregate and mixed until maximum aggregate coating is achieved by the binder. The mixing process can be operated by using a mixing device such as a pug mill (Figure 3-5), or a rolling drum mixer (Figure 3-6).

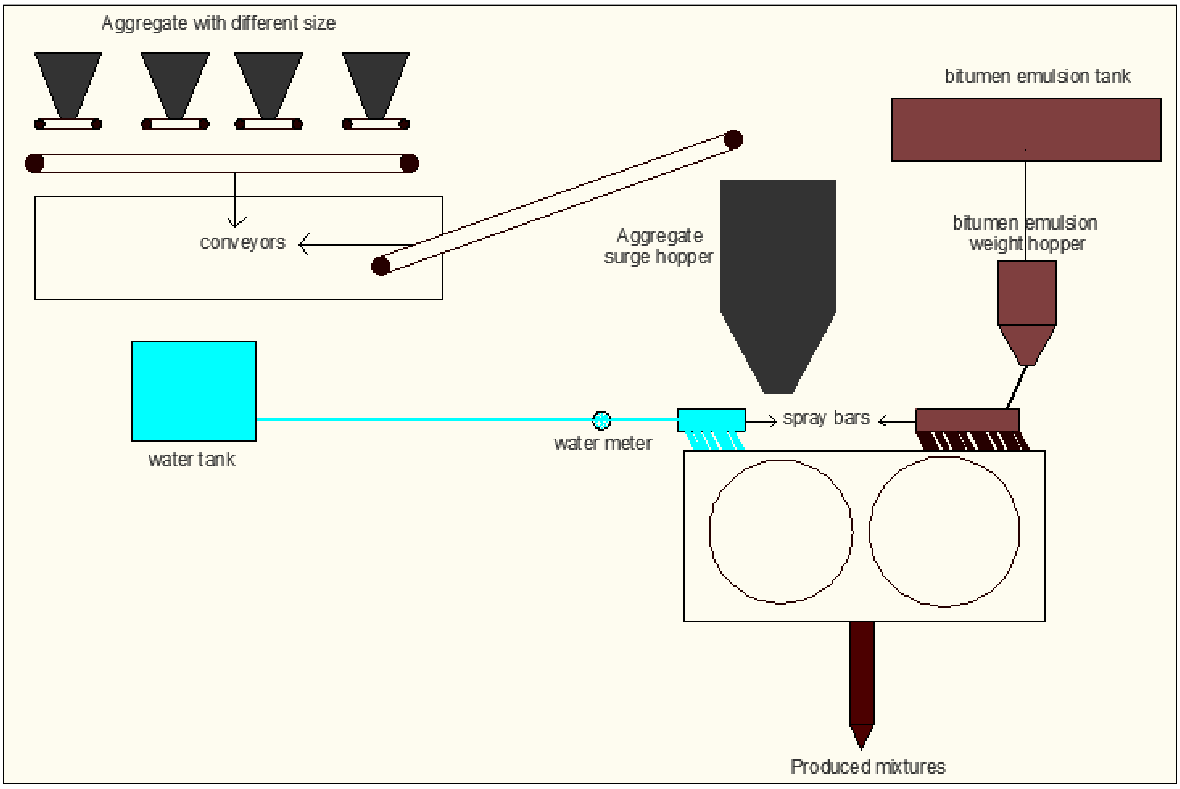


Figure 3-5: Batch cold mix plant

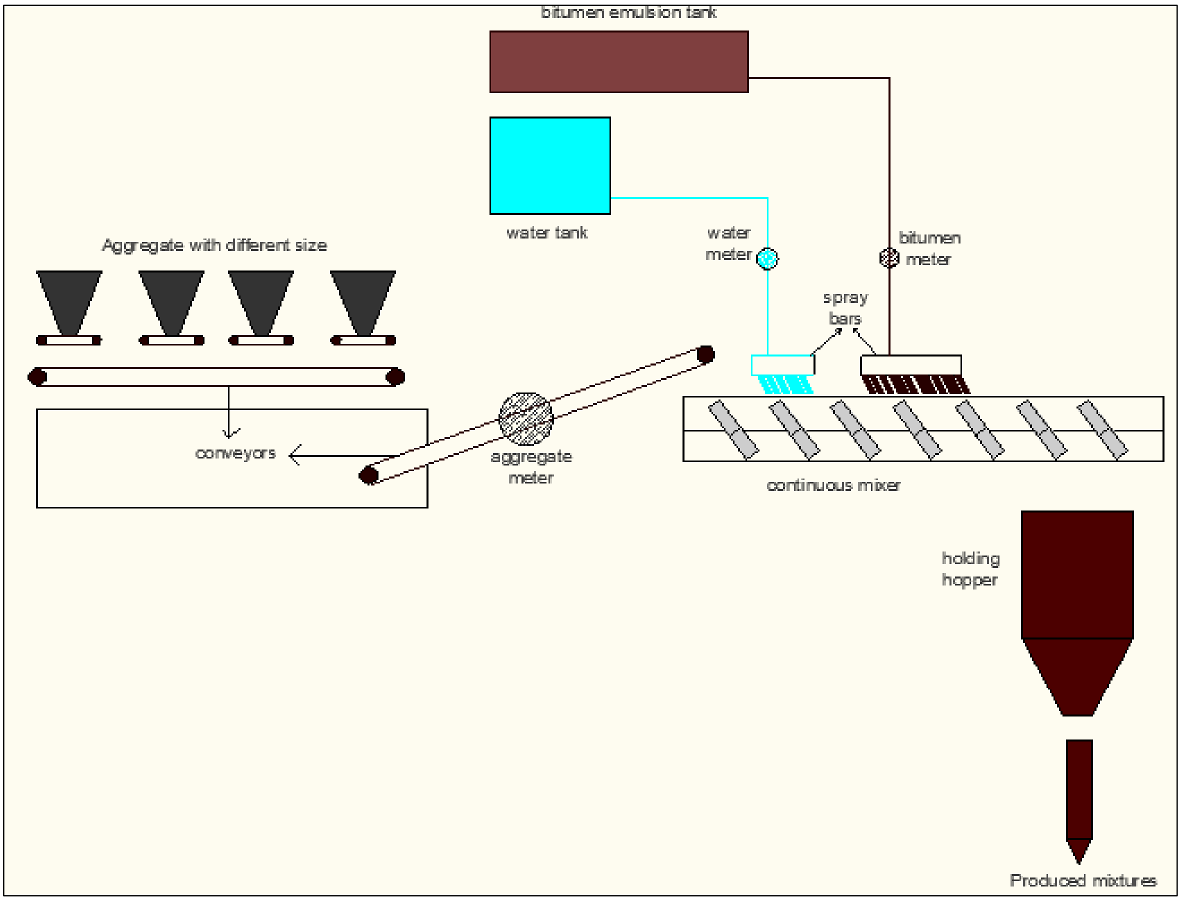


Figure 3-6: Continuous cold mix plant

BEMs are still limited to road pavements that accommodate low to medium traffic due to their intrinsic problems, especially insufficient early strength and long curing time required. Ibrahim and Thom (1997) reported that BEMs can be utilised in all pavement layers designed for low to medium traffic loads. However, for heavily trafficked roads, at least a 40 mm hot asphalt layer is needed to overlay the BEM layer.

BEMs can be applied in different ways starting from hand application, graders, and pavers to self-contained mixing and laying plants. Different compaction techniques are utilised for cold BEMs depending on the manufacturing company. However, Thanaya (2003) stated that steel rolling followed by a very heavy pneumatic tyred roller and finishing with a steel roller is the preferred technique. Over rolling at the mixing stage can lead to excessive emulsion breakage which can seal the surface, preventing curing and cracking; therefore, a degree of caution must be exercised by operators.

Previously, BEMs were produced with open graded or semi-dense graded mixtures to ensure better airflow, thus improving the curing process due to the high air voids in these mixtures. In line with improvement of emulsion technology and preparation techniques, currently cold BEMs can be produced even with dense or gap gradation mixtures (Ibrahim and Thom, 1997).

Suitable aggregate gradation, bitumen emulsion and pre-mixing water are required for the BEM mix design. Zizi and Sainton (1997) stated that the emulsion’s breaking mechanism and the compactability of the mixtures control the performance of the produced mixtures in the field. Bitumen emulsion’s breaking during curing covers the total evaporation of water followed by an effective distribution of the mixture's constituents and coating of the aggregate by the bitumen emulsions.

A number of processes have been developed and are in use to produce BEMs such as the two stage mixing process, the double mixing process and the double treatment process, and these are described below:

Two stage mixing

BEMs produced by this process are mainly used on lightly trafficked roads in remote regions, particularly in Sweden. This process comprises two stage mixing, which means that coating of coarse and fine aggregates is carried out in two stages to avoid preferential breaking of the emulsion onto the fine. Either virgin or recycled materials can be used as aggregate and the base binder of the bitumen emulsion is an extremely soft binder produced by adding heavy flux oil to normal penetration grade bitumen. They are often doped with fatty amine-based adhesion agents to improve durability. The bitumen types are characterised in terms of absolute viscosity at 60 °C, with grades ranging from 2000 to 10,000 centistokes. For comparison, 100-pen bitumen has 10,000 centistokes' absolute viscosity. Then the produced soft bitumen is emulsified with the aid of different levels of emulsifier to generate different breaking rates.

In this process, firstly, the coarse aggregate is charged into the mixer and pre-wetted with water. Then, a rapid setting bitumen emulsion is incorporated via a spray bar during the mixing process. The first mixing process is continued for 20 s. After the first step, the pre-wetted fine aggregate component is incorporated, shortly followed by a medium setting bitumen emulsion. Second mixing is continued for another 20 s. Figure 3-7 shows a schematic diagram of a two stage mixer. Two stage mixing plants are often mobile; therefore, they can be placed close to the region of application (Needhem, 1996).

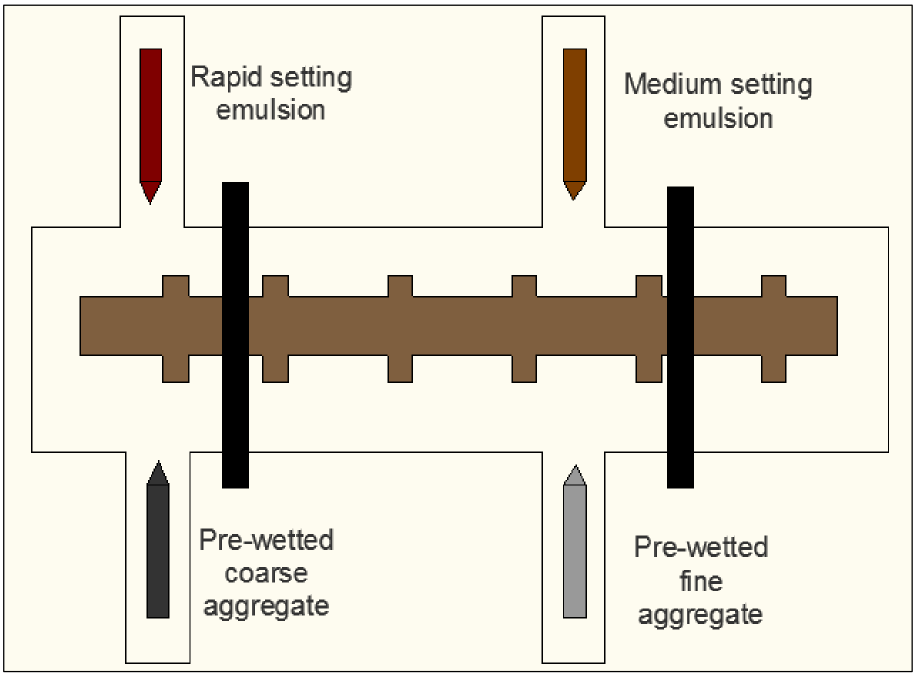


Figure 3-7: Two stage mixer: schematic diagram

Double mixing process

Mahesh (1990) invented a process for the production of stackable, dense road asphalts principally for use in making and repairing roads, aerodrome runways, etc. In this patent, the virgin aggregate materials with a diameter from essentially 0–30 mm are coated in a cutback or fluidified bitumen-based binder. The aggregate fractions are separated into at least two groups. The first comprises the smallest fractions, while the second one is a grouping of the remaining elements. The first group is coated with a cationic cutback or fluidified bitumen emulsion with a fracture index greater than zero and having a residual binder viscosity measured on a Standard Tar Viscosimeter STV (10 mm, 25 °C) of less than 50 s. On the other hand, the second portion of aggregate is coated with a cationic cutback or fluidified bitumen emulsion with a residual binder viscosity measured on a STV of between 1000 and 2000 seconds. Then, both coated materials are mixed together for a few minutes.

This method was adopted by the French company SCREG who refer to this process as by the trade name COMPOMAC. The same gradation of the grave emulsion is used with a 2 mm fine and coarse aggregate division size. In the double mixing process, firstly the fine aggregate is mixed with part of the bitumen emulsion or hot bitumen as an alternative binder type. The mixture is then stockpiled for later use. Secondly, the coarse aggregate is mixed with a second portion of emulsion and then added to the first group product, i.e. coated fine materials. The double mixing process products are richer than normal Grave Emulsion products in terms of the residual bitumen content, which lies from 5–5.8 %. Figure 3-8 shows a schematic diagram of the double mixing process (Needhem, 1996).

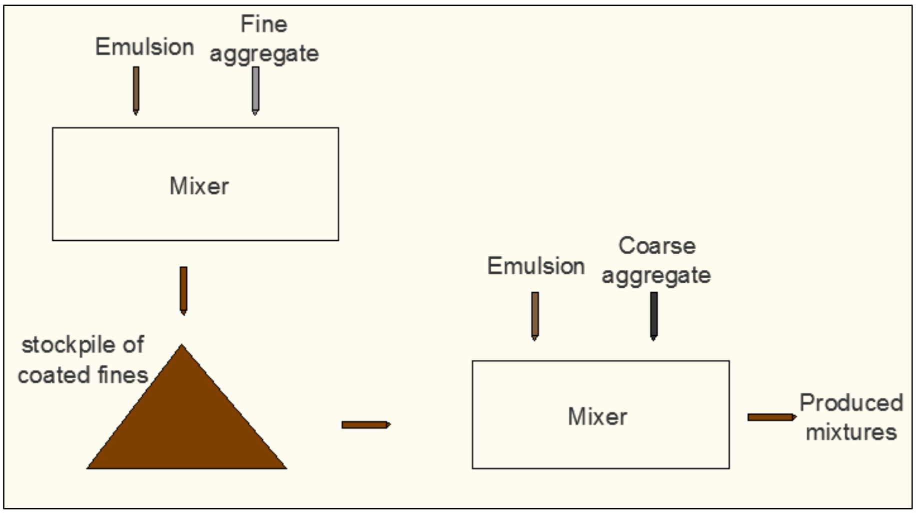


Figure 3-8: Double mixing process

Double treatment process

Cold BEMs produced by means of the double treatment process which are utilised in France, Germany, and recently in the Benelux countries for the base course. Cold BEMs are so called because of using two types of binders: bitumen and cement. These binders can be incorporated in different amounts to both virgin and recycled materials. The produced mixtures can give high resistance to cracking as well as permanent deformation at high temperatures due to the nature of the two binders. A slow setting bitumen emulsion is often used with cement as a hydraulic binder. The performance of the mixture is highly affected by the ratio of the bitumen emulsion and cement; therefore a careful mix design is needed to achieve the required specification (Sainton and Bourdrel, 1993).

3.2 Overview of BEMs' applications worldwide

Production and use of cold BEMs varies considerably in different countries. France and the USA had early applications of cold BEM for road works. A number of practices different from the processes discussed in the previous section have been developed and are in use around the world, as will now be discussed.

In Spain, utilisation of cold BEMs started in the late 1950s. Gravel and open graded mixtures began to be used in the 1970s and are still in use till now. In open graded mixtures, medium setting cationic or anionic emulsions with high viscosity have been used to ensure thick binder coatings on the aggregate surfaces. Further enhanced properties can be achieved when polymer modification of the binder has been added to these mixtures (Raz and Orue, 1993).

In Germany, there are two main methods of cold laying bituminous mixtures, which are mix in plant and mix in place, the latter of which is particularly useful for recycling. A considerable amount of recycled road materials or waste or by-product materials has been used with bitumen emulsion and cement as binders. Very high-quality roads have been developed with these bases. The nature and properties of the old road to be recycled must be known before any work starts. Old pavements are frequently milled to 30 cm depth. Millings are reduced in size and then mixed with slurry generated from cement, water and emulsion. Then the produced cold mixture is relayed and compacted using steel roller plus heavy pneumatic roller then steel roller procedure (Needhem, 1996).

Utilisation of cold BEMs in Italy was widespread for both reinforcement of existing roads and construction of new roads. Consequently, a set of regulations and guides to control cold mixtures and construction methods has been drawn up by the National Research Council. The common cold BEMs are utilised with normal design methods and laying techniques and it has been reported that the Italian climate favours these mixtures among the different types of cold bitumen mixtures (Needhem, 1996).

In France, a novel cold BEM has been developed by Jean Lefebvre using a gap graded aggregate combined with bitumen emulsion and two additive types, which were polyacrylonitrile fibres and a polymer called EVA (ethyl vinyl acetate); therefore it is termed as Gap Graded Cold Asphalt Concrete (Vivier and Brule, 1991). The fibres with 4 mm length are incorporated at a 0.1–0.2 % dosage by total mass of dry aggregate to decrease chip loss, decrease segregation of the mixture and enhance shear strength after cure. EVA, which is sometimes incorporated into bitumen in HMA to improve rutting resistance, is added in this procedure to the binder preceding emulsification to enhance cohesion, temperature susceptibility, rheological behaviour and adhesion properties. These mixtures are claimed to have better skid resistance, durability and wearing resistance in comparison with untreated cold mixtures.

Emulsified Asphalt Materials (EAMs) are referred to cold BEMs in the USA. According to the Asphalt Institute thickness design manual MS-1 (Asphalt Institute, 1991), EAMs are classified into three types: i) made with processed dense-graded aggregates, ii) made with semi-processed crusher-run, pit-run, or bank-run aggregates, and iii) made with silts or sands. The first type is considered to produce mixtures with strength as high as traditional asphalt concrete. The range of the layer thickness is recommended to be between 50 and 75 mm to ensure a suitable curing. However, 2 months to 24 months is the possible EAMs' curing time to achieve the recommended strength. Additionally, a good compaction degree for these mixtures is required and when the initial curing of the lower EAM layer is completed, then any other added layer can be laid. Hveem method and Marshall method with their recent modifications are the main detailed design procedures for cold BEMs (Asphalt Institute, 1989, Asphalt Institute and Asphalt Emulsion Manufacturers Association, 1997).

Bullen (1995) stated the appropriateness of utilising Grave Emulsion mixtures (firstly produced in France) in Australia. He reported the economic, environmental and logistical advantages of the produced GE according to Australia's circumstances and environment. Australian GE is cured more quickly compared with Europe because of the warmer and dryer climate. On the other hand, the resulted stiffness modulus of those mixtures is reduced due to the mentioned high temperature. Generally, GE mixtures can be considered as an economical and sustainable alternative to asphalt concrete as well as to crushed rock.

South Africa has utilised BEMs for several decades and calls the mixtures Granular Emulsion Mixtures (GEMs). Also, there is another bitumen emulsion application termed Emulsion Treated Base (ETB), which is produced from rehabilitation of cement treated base using bitumen emulsion. These mixtures contain very little residual binder content, i.e. 1 % on average. Due to particular concern, the long-term performance of emulsion-based mixtures has been assessed by conducting several evaluation studies. A Heavy Vehicle Simulator (HVS) which was recommended as an ideal tool for accelerated testing has been used to assess these mixtures. The HVS test results showed that ETB was a reasonable mixture for road construction and that its performance can be enhanced by incorporating cement or lime. Generally, resistance to cracking for ETB mixtures is more than concrete but less so than conventional hot mixtures.

Widodo (2007) reported that in Indonesia, as an example of a developing country, existence of hot asphalt mixing plants is still very rare, so that the road maintenance location is sometime too far from the location of these plants. This means that the temperature of the hot asphalt will have dropped by the time it reaches the work location; thus the quality of the mixture becomes unfavourable. Therefore, application of cold BEMs will be helpful and more economic.

In Tanzania, as another example of a developing country, the Ministry of Work has recently started considering Bitumen Emulsion cold-mixes (BEMIX) as an alternative pavement construction and maintenance strategy (Mgani and Nyaoro, 1999). In Morogoro Municipality, a trial project in cold BEMs was launched in August 1998. Cold BEMs mix design was conducted at the laboratory of the project. The specified Marshall Stability for the project samples did not comply with the predictable values.

3.3 Cold bitumen emulsion mixtures' future

Cold BEMs are gaining high interest in the USA, France and many other countries worldwide due to the positive aspects for the environment, energy saving and economy, health and safety of pavement construction and maintenance. In the UK, due to different causes such as low earlier strength, lower earlier rain resistance and no acceptance from the industry side to transfer to such new areas, cold BEMs have been used less widely. However, use of permanent cold-lay surfacing materials as an alternative to hot-mix asphalts for reinstatement work only on low-volume traffic roads and footways, as adopted by the Highway Authorities and Utilities Committtee (1992), opened the way to utilising cold BEMs in other medium and high trafficked roads.

3.3.1 Comparison between cold and hot mix technologies

A bituminous mixture is essentially a granular material with an extra ingredient, bitumen. A right understanding of the former is of considerable aid when trying to make sense of asphalt. The composition of the bituminous mixtures and the preparation and laying conditions are basically the most important factors which affect the performance of these mixtures under specified load and environmental conditions (Thom, 2008).

Generally there are two steps of asphalt mix design: i) choosing the aggregate and binder types according to their base properties, and ii) selecting aggregate gradation and then indicating the optimum binder content. Read and Whiteoak (2003) reported that obtaining a continuous and homogeneous binder film depends on the mixing process of these ingredients. Moreover, an effective compaction process is required to obtain the optimum void content to ensure a high performance mix.

Traditional hot bituminous mixture is an excellent material when properly designed and constructed. This degree of performance can be achieved when taking every last drop of water away from the aggregate particles; thus heating the aggregate is necessary, which is undesirable in economic and environmental terms. Also, the selected aggregate is restricted to its ability to withstand heating without being damaged due to the heating process. Cold BEMs' technology opens the door to a brave new world of possibilities. Section 3.1.3 has already introduced the principle of these mixtures.

In terms of energy consumption, cold BEMs' production needs considerably less than conventional bituminous mixtures. The manufacturing of each tonne of cold BEM needs 13 % of the energy required for hot mix, as stated by Bouteiller (2010). Moreover, Kennedy (1998) reported that CO_2 emission for cold BEMs is approximately 14 % compared with hot mix manufacturing. The annual production amount of hot mix, which is around 30 million tonnes, consumes 6-7 million MWh and generates about 1 million tonne of CO_2 emission. A general comparison between cold and hot mix technologies within production and application processes is shown in Table 3-1.

Table 3-1: General comparison between cold and hot mix technologies within production and application processes

Cold mix technology	**Hot mix technology**
No need for a special storage technique for the binder	Need special tanks to store the binder
Special binder and mixture transport is not required	Special tanks equipped with heating system for bitumen and sheeted lorries required for transporting mixtures
Simple storage of mixture at plant	Mixtures need heated lagging and pipes at plant
Mixing at ambient temperature with wet aggregate	Mixing at elevated temperatures with pre-heated aggregate
Mixed mixtures can be stored easily	Difficult to store mixed mixtures
Long time for laying and compaction	Construction time for laying and compaction depending on temperature space.
Consume about 36 (MJ/MT)	Consume about 277 (MJ/MT)
Generate approximately 3 (Kg/MT)	Generate approximately 21 (Kg/MT)

3.3.2 Deficiencies of cold BEMs

Although there are interesting benefits of cold BEMs, there are a number of deficiencies which make them inferior to conventional hot mixtures and have to be overcome within production stages and service life of such mixtures:

- Lacking in coating percentages due to the incompatibility between the aggregates and emulsion.
- Low bitumen film thickness on coarse aggregate due to the fines attracting emulsion particles more than coarse particles.
- Drainage of the binder in storage because of the low viscosity of the emulsion.
- Poor early life adhesion/cohesion properties because of the existence of water.

- High water sensitivity and poor adhesion of the produced mixtures cause binder stripping off the aggregate particles.
- Weak early life strength.
- Long curing periods are required to evaporate the trapped water, and breaking of bitumen emulsion.
- High residual air voids contents.

The full curing of cold BEMs on site may extend between 2–24 months depending on the weather conditions, as reported after a research study conducted by Chevron Research Company in California (Leech, 1994). Accordingly, there are two main challenges to adopting cold BEMs in UK, namely: i) long curing time is needed due to the weather conditions – it is humid, cold, and rainy most of the year, and ii) there are sufficient hot mix asphalt plants thus fewer remote areas.

3.3.3 Sequence of processes of cold BEMs' production

Mixing, storage and laying, and compaction are the main production processes of cold BEMs. In the mixing process, the emulsion must remain sufficiently stable and coat the coarse and fine aggregates uniformly. In the storage and laying process, the emulsion is required to remain workable and be partially broken; thus it can resist moisture and rain. The compaction process is the last stage, in which bitumen emulsion should set quickly and revert to its original base bitumen (Taylor, 1997).

Generally, a long curing time is required for most emulsions by which a complete break of the emulsion and the mixture will achieve its maximum strength.

3.4 Overview of BEMs' design procedures

Generally, there is no universally accepted mix design method for cold BEMs because public transport is a vital part of human mobility. Additionally, there is a lack of uniform procedure for laboratory evaluation because the correlation and assessment of conducted test results differ among investigators and institutions. In the UK, “Specification for Reinstatement of Openings in Highways” by the Highway Authorities and Utilities Committee, which allows the use of Permanent Cold Lay Surfacing Materials (PCSMs) instead of hot mix materials for reinstatement works in low trafficked roads and footpaths, promoted utilisation of cold BEMs (Highway Authorities and Utilities Committtee, 1992). The Highway Authorities and Utilities Committtee (1992) stated that PCSMs should work adequately after two-year guarantee ends which is becoming a major challenge, according to Leech (1994). Meanwhile, in Indonesia, the Ministry of Public Works provided a specification on Cold Asphalt Emulsion Mixtures (CAEMs) in 1990 (Ministry of Public Works Republic of Indonesia, 1990).

It is necessary to give a brief description about the main cold BEMs’ design procedures before showing the specifications of those mixtures. Five design procedures for cold BEMs are briefly described in the following subsections, namely: Asphalt Institute design procedure MS-14, Asphalt Institute design procedure MS-19, Ministry of Public Work-Republic of Indonesia, Nickolaids A. and Nynas method.

3.4.1 Asphalt Institute design procedure MS-14, 1989

The Asphalt Institute design procedure involves the following steps (Asphalt Institute, 1989): i) choosing of aggregate gradation, which can be dense or gap gradation; ii) determination of Initial Emulsion Content (IEC) and Initial Residual Bitumen Content (IRBC), by utilising empirical equations; iii) indicating Optimum Pre-Wetting water content (OPWwc) by conducting coating test; iv) indication Optimum Total Liquid Content at Compaction (OTLC) according to maximum dry density of cold mixtures; and v) indicating the Optimum Residual Bitumen Content (ORBC) which is related to the maximum soaked stability and the maximum dry density of cold BEMs with various RBC (Asphalt Institute, 1989).

3.4.2 Asphalt Institute design procedure MS-19, 1997

The Asphalt Institute in association with the Asphalt Emulsion Manufacturers Association (AEMA) published a new manual on asphalt emulsion in 1997 which is MS-19, "A Basic Asphalt Emulsion Manual", third edition (Asphalt Institute and Asphalt Emulsion Manufacturers Association, 1997). It largely follows the MS-14 design procedure discussed in 3.4.1, but with some additions and modifications which can be summarised by three main points, which are: 1) the bitumen emulsion can be used only if the degree of coating to the aggregate particles after a specific adhesion test procedure remains within acceptable range, 2) there is no requirement for OTLC such as MS-14, but the mixtures shall be air dried until neither too wet nor too dry for compaction, and 3) after compaction, the samples are conditioned with their mould in an oven at 60 ºC for 48 hours, then additional compaction with 178 KN load for 1 minute at the same temperature is applied by using a double plunger from both sides of the specimen.

3.4.3 Ministry of Public Works of Indonesia design procedure, 1990

The design procedure adopted by the Ministry of Public Works-Republic of Indonesia is principally similar to the Asphalt Institute MS-14 procedure with consideration of the regional and national conditions of Indonesia. This procedure covers two main areas: open graded emulsion mixtures and dense graded emulsion mixtures. Modified Marshall Stability test is required as a test procedure in which the samples are tested utilising a Marshall testing frame at ambient temperature; therefore preconditioning the specimens in water at 60 ºC for 30 minutes is not recommended (Ministry of Public Works-Republic of Indonesia, 1990).

3.4.4 Nikolaids' design procedure

Nikolaids worked in Indonesia as a consultant on road projects in 1990. His design procedure depends on Asphalt Institute and Ministry of Public Work-Republic of Indonesia procedures (Thanaya, 2007). Additionally, in this procedure Nikolaids has introduced a characterisation method for permanent deformation. The maximum permissible value of residual bitumen content is indicated according to the permanent deformation performance. Accordingly, a relationship between the residual bitumen content (RBC) and the creep stiffness coefficient (CSC) is needed to indicate this value, i.e. maximum RBC.

The CSC value is the slope of the relationship between mixture creep stiffness, which is obtained from a static creep test conducted at 40 ºC and 100 kPa vertically applied static load, and bitumen stiffness, which in turn can be indicated at any loading time for a specified grade of binder utilising Van der Poel nomograph or BANDS software from Shell Company, depending on softening point and testing temperature (Nikolaids, 1994b).

3.4.5 The Nynas tests procedure

The Nynas Company introduced three tests which are applied only to the loose mixtures during storage or before laying of cold mixtures; these are: runoff, washoff and workability tests.

The runoff value refers to the quantity of bitumen emulsion mixture material run-off in a specific time from a specific mesh; while the washoff test is conducted instantly after the runoff test to check that no bitumen has washed off during the runoff test. Finally, the workability test is conducted by using the Nynas workability tester shown in Figure 3-9. The test is carried out by scraping the top few mm of an uncompacted cold bituminous mixture during storage or just before laying, and the maximum force required to remove the top of the mixture by shear is measured.

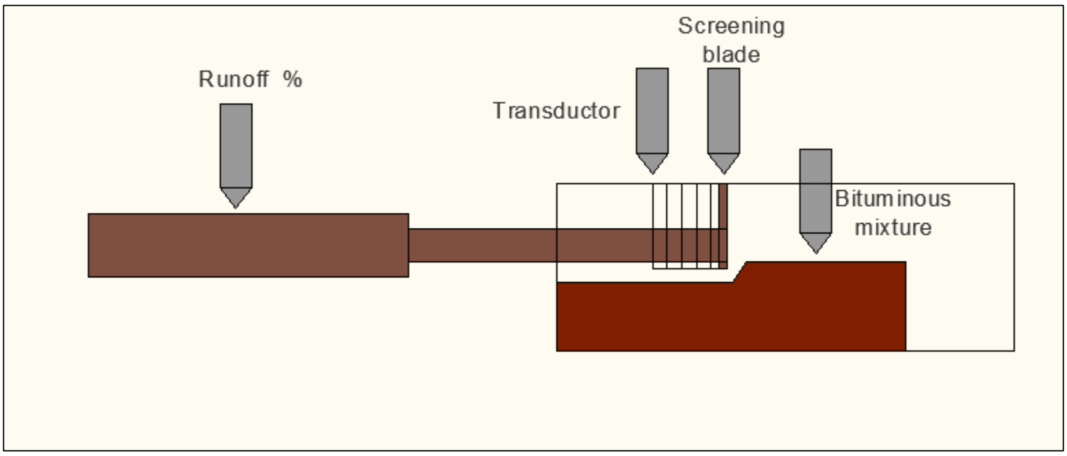


Figure 3-9: Basic principles of the Nynas workability tester

3.5 BEMs' specifications and design criteria

There is no universally agreed specification for cold BEMs' mix design or testing. Design of cold mixtures and the recommended type testing vary amongst the various road authorities, research institutes and pavement researchers. Three main global specifications are reviewed below. These are: Asphalt Institute; Ministry of Public Work (MPW)-Republic of Indonesia; the Highway Authorities and Utilities Committee-UK specifications. Table 3-2 shows a comparison summary between the first two procedures in terms of preparation and curing of samples because the procedure adopted in this work is close to these procedures.

Table 3-2: Summary of Asphalt Institute and MPW samples' preparation and curing

Description	Asphalt Institute, USA	MPW, Indonesia
Minimum degree of coating (%), for Base course Wearing course	 50 75	 75 75
Compaction (Marshall Hammer)	2 × 50 blows	2 × 50 blows
Curing in mould	24 hours at room temperature	24 hours at room temperature
Curing in oven	24 hours at 38 ºC	24 hours at 40 ºC
Samples' testing temperature	22.2 ºC	28 ±1 ºC
Soaking procedure	Step 1: Immerse the specimen in water with vacuum saturated at 100 mm HG for 1 hour, Step 2: immerse in water at 22.2 ºC (no vacuum) for another 1 hour	(capillary soaking) Step 1: Soak half of the specimen in water at room temperature (28 ± 1 ºC) for 24 hours, Step 2: invert the specimen and soak the other half for a further 24 hours.

3.5.1 Asphalt Institute specifications

The Asphalt Institute introduced two procedures, MS-14 in 1989 and MS-19, in 1997 as reviewed in section 3.4.

The MS-14 design criteria is applicable for low-volume traffic base course mixtures composed of up to 25 mm maximum size dense graded mineral aggregates. The cold BEMs' preparation should comply with the design criteria recommended by MS-14 manual and presented in Table 3-3. Furthermore, MS-14 does not recommend the construction of cold BEMs when the temperature is below 10 ºC, or in wet weather. On the other hand, MS-19 is applicable for either base or surface courses of low to medium trafficked roads with maximum aggregate size up to 25 mm.

Table 3-3: Cold BEMs' design criteria (Asphalt Institute, 1989), permission to reproduce this table has been granted by Asphalt Institute

Test Property	Minimum	Maximum
Soaked stability at 22 ºC, KN	2.225	---
Percent stability loss, after vacuum saturation and immersion at 22 ºC	---	50
Aggregate coating, %	50	---

3.5.2 Ministry of Public Work-Republic of Indonesia specifications

In this procedure, a modified Marshall test technique is utilised for open graded and dense graded cold emulsion mixtures. They introduced five continuous graded composition termed from I to V and one emulsion sand mixture termed as VI which is suitable for the base course. Continuous gradation types are specified depending on the maximum nominal aggregate size. Type V is the finest gradation mixture, which can be used in either base or wearing layers, types II-IV are appropriate for sub-base or base courses, whilst type I is the coarsest gradation for the sub-base course. Table 3-4 shows the design criteria and mixture property limits for the dense graded emulsion mixtures as adopted by MPW in Indonesia (Ministry of Public Works-Republic of Indonesia, 1990).

There are two main differences between this specification and the Asphalt Institute MS-14 as shown in Table 3-4; namely there is a requirement for porosity of 5–10 % and a minimum bitumen film thickness of 8 microns on the Indonesia specification, while there are no such requirements on the Asphalt Institute MS-14.

Table 3-4: Design criteria and mixture property limits for dense graded emulsion mixtures at ambient temperature (Ministry of Public Works-Republic of Indonesia, 1990)

Property	Value range	Types of dense graded emulsion mixtures					
		I/50	II/37.5	III/25	IV/19	V/12.5	VI
Soaked stability, kN	Min.	3	3	3	3	3	2.5
Retained stability, %	Min.	50	50	50	50	50	50
Porosity, %	Min.-Max	5-10	5-10	5-10	5-10	5-10	5-10
Moisture absorption, %	Max.	4	4	4	4	4	4
Bitumen Film Thickness (BFT), µm	Min.	8	8	8	8	8	8
Coating degree, %	Min.	75	75	75	75	75	75
Recommended layer thickness, mm	Min.-Max	80-150	50-100	40-100	30-75	25-75	25-75

3.5.3 The Highway Authorities and Utilities Committee (HAUC)-UK specification

In 1992 a code of practice “'Specification for the Reinstatement of Openings in Highways” was introduced by the Highway Authorities and Utilities Committee (HAUC) in response to UK government legislation. According to Appendix 10 of this code, the use of Permanent Cold Lay Surfacing Materials (PCSMs) is approved based on elastic stiffness criteria at the end of a 2-year guarantee period, see Table 3-5. The code does not cover a mix design procedure for the PCSMs. Robinson (1997) stated that total voids after compaction for footpath materials in the range 2–12 % and 2–10 % for carriageways are required. Additionally, an acceptable surface profile within the 2-year guarantee period should be achieved from using PCSMs.

Table 3-5: The minimum requirements of permanent cold laid surfacing materials (Highway Authorities and Utilities Committtee, 1992), permission to reproduce this table has been granted by Crown Copyright

Permanent Cold-Lay Surfacing Materials (PCSMs)	Minimum property requirement at 20 ºC equivalent to		
	50 pen hot laid elastic stiffness (MPa)	100 pen hot laid elastic stiffness (MPa)	200 pen hot laid elastic stiffness (MPa)
20 mm nominal size base course	4600	2400	900
10 mm nominal size wearing course	3800	1900	800
6 mm nominal size wearing course	2800	1400	600

3.6 Enhancing cold BEMs' performance

Wide studies have been done to understand the performance of cold BEMs and to enhance their performance. Two main aspects are related in this study to improve the performance of these mixtures, namely: i) incorporating different types of materials, which is the main consideration in this research, and ii) applying different preparation techniques, i.e. mixing, compaction and curing.

Ibrahim and Thom (1997) concentrated on the effects of curing procedures and compaction methods. They stated that indirect tensile stiffness modulus increases with curing time increment. The Chevron Research Company carried out laboratory and field studies to investigate the performance of BEMs in California. They reported that the full curing in the field of these mixtures may occur from 2–24 months depending on the mixture's constituents and weather conditions (Leech, 1994). Furthermore, some different preparation techniques have been discussed earlier in section 3.1.3.4, such as two stage mixing, double mixing process and double treatment process.

3.6.1 The role of mineral filler in cold bituminous emulsion mixtures

Mineral filler comprises the materials passing sieve 63 µm and may be active or inactive (inert) material when incorporated into cold BEMs depending on its chemical properties, fineness and surface characteristics. Reactive filler, such as cement, is a type of filler that reacts when in contact with emulsion and/or any added water as a hydration process occurs. Meanwhile, inert filler, such as limestone dust, will not react when mixed with emulsion.

The principal cementitious material in concrete and cold BEMs is Portland cement. Today, most concrete mixtures contain Supplementary Cementitious Materials (SCM) that make up a portion of cementitious components in concrete. These materials are generally by-products from other processes or natural materials. They may or may not need to be further processed for use in concrete. Some of these materials are called pozollans, which by themselves do not have any cementitious properties but when used with Portland cement or any cementitious material react to form cementitious compounds. Other materials, such as Waste Paper Sludge Ash (WPSA), do exhibit cementitious properties, i.e. generate cementitious compounds when they react with water.

The main benefits from incorporating SCMs in cold BEMs are: enhancing the mechanical properties and durability due to the cementitious and pozzolanic properties of those materials, gaining economic benefit as the cementitious and pozzolanic materials used are mostly industrial waste or by-products, and have ecological benefits as many of those by-product materials contain toxic elements which can be hazardous to human health when they are dumped in lakes, streams and landfills.

Thanaya (2003) stated that the chemical composition of SCMs used, for example in concrete, does not play a major role, but their mineralogical and particle characteristics, i.e.

particle size distribution, shape, texture and surface area, do affect the engineering properties of concrete. Generally, particle properties have more effect compared to their chemical composition.

Malhotra and Mehta (1996) reported that there is nothing that can be done to change the mineralogical properties of SCMs. Hence, controlling the particle size distribution of these materials is used to enhance their pozzolanic or cementitious characteristics. Accordingly, ASTM standards (C 618) indicated that the materials retained on sieve No. 325 (45μm) should be not more than 34 % because it has been found that the retained materials show little or no reactivity under normal hydration conditions.

Some examples of these materials are listed below:

3.6.1.1 Pulverised fuel ash

Pulverised Fuel Ash (PFA) is a pozzolanic material that is generally used in cement-based materials to improve long-term strength, workability, resistance to sulphate attack and durability in concrete. This industrial non-hazardous by-product results from the burning off of volatile matter and carbon in coal due to ignition in the furnace, which then helps the mineral impurities in the coal (e.g. feldspar and clay) to fuse in suspension and float out with the exhaust gases. The following process is the cooling and solidification of the fused substances into what it is called Pulverised Fuel ash. However, the last step is to collect the fly ash from the exhaust gases by bag filters or electrostatic precipitators, depending upon the collecting system (Owaid *et al.*, 2012). The PFA particles are fine-grained particles that are generally sized from > 1μm – < 100μm. However, it is worth mentioning that the diameter of the average PFA particles is smaller than 20 μm in size, and the particles retained on a 45 μm sieve are only 10–30 %. In general, the surface area of PFA typically varies from 300 to 500 m^2/kg and the specific gravity ranges from 1.9 to 2.8. The colour may vary from tan to dark grey depending on its chemical and mineral composition (Environment Agency, 2010).

3.6.1.2 Ground granulated blast furnace slag

Ground Granulated Blast Furnace Slag (GGBS) is a non-metallic by-product material produced by the blast furnaces used to make iron. A mixture of iron ore, coke and limestone operating at temperatures of approximately 1600 ºC in the furnace can produce molten iron and molten slag. Molten slag floats on top of the molten iron because of its light weight. Molten slag consists mostly of 30–40 % SiO_2 and approximately 40 % CaO. Then, cooled down by high-pressure water jets, it generates glassy and granular particles and, further, these particles are dried and ground to the required size to make a fine, glassy powder known as GGBS. GGBS sized less than 45 μm has a surface area of approximately 400–600 m^2/kg. Since its initial production in Germany, GGBS has been extensively utilised as a constituent of blended cement or a mineral admixture in cementitious materials due to its pozzolanic nature (Yazıcı *et al.*, 2010).

3.6.1.3 Steel slag

Steel Slag (SS) is a non-metallic by-product material generated from the steel refining process. It essentially consists of calcium silicates and ferrites combined with fused oxides of iron, aluminium, manganese, calcium and magnesium. Due to the variations in chemical composition and types of steel slag, a few are suitable for use in the construction industry. Although the steel slag ingredients are similar to those of blast furnace slag, the proportions are quite different, with the higher iron content being reflected in the high density of 3.2–3.5 mg/m^3 for steel slag compared to 2.2–2.5 mg/m^3 for air-cooled blast furnace slag.

3.6.1.4 Waste paper sludge ash

Waste Paper Sludge Ash (WPSA) is generated from incineration of paper mill sludge, which is a by-product from the pulp and paper making industry. Recently, incineration of paper sludge for energy recovery has become more attractive because of environmental restrictions and increased taxation for landfill; hence it is termed as waste material (Waste and Resources Action Programme, 2007). It was found that calcination of paper sludge at 700 ºC for 2 hours converts the kaolinite into reactive amorphous metakaolinite and increases the pozzolanicity of the product. WPSA main ingredients are CaO, SiO_2 and Al_2O_3. Annually, in the UK more than 1 million tonne from paper sludge is used in power generation plants. The main uses of WPSA are cement manufacturing and block production.

3.6.1.5 Silica fume

Silica Fume (SF) is a highly reactive pozzolanic material and is a by-product from the manufacture of silicon or ferro-silicon metal. It is collected from the flue gases from electric arc furnaces. SF is a very fine powder, with particles smaller than an average cement grain by about 100 times. SF is available as a densified powder or in a water-slurry form. It is commonly utilised at 5–12 % by mass of cementitious binder for concrete structures that need high strength or considerably reduced permeability to water. Special procedures are warranted when handling, placing and curing concrete produced with SF because of its extreme fineness (Owaid *et al.*, 2012).

3.6.1.6 Rice husk ash

Rice Husk Ash (RHA) is a by-product from burning of rice husk. About 20 % of a dried rice paddy is made up of rice husks. Rice paddy production worldwide is around 500 million tonnes hence 100 million tonnes of rice husks are generated. As the production rate of RHA is approximately 20 % of the dried rice husk, the amount of RHA generated yearly is about 20 million tonnes worldwide. Therefore, the material exists in huge amounts worldwide (Zemke and Woods, 2009). RHA with cellular microstructure and highly pozzolanic activity is formed when rice husk is burnt at temperatures lower than 700 ºC.

The most important property of RHA that determines pozzolanic activity is the amorphous phase content. RHA is a highly reactive pozzolanic material suitable for use in lime-pozzolana mixes and for Portland cement replacement. RHA contains a high amount of silicon dioxide, and its reactivity related to lime depends on a combination of two factors, namely the non-crystalline silica content and its specific surface. In the case of RHA, the compressive strength of blended concrete structures has been shown to be enhanced and water permeability to be decreased chemically and physically (Ganesan *et al.*, 2008).

The chemical reaction of amorphous silica with Ca^{+2}, OH^{-} ions and calcium hydroxide during the cement hydration forms more calcium silicate hydrate gel (C–S–H) which is known to contribute to the improvement in the strength and durability properties of concrete.

3.6.1.7 Poultry litter fly ash

Poultry Litter Fly Ash (PLFA), a type of biomass fly ash, is produced in thermoelectric power plants and is the result of combustion of poultry litter, wood chip and horse bedding in a fluidised bed combustor. Fluidised beds use CaO and activated carbon to capture sulphur and nitric oxide (NOx) released during combustion. The CaO is injected into the incinerator to precipitate out sulphate during combustion and trapped in the filter. Due to environmental agencies initiative for maximising the use of renewable energy sources, the use of biomass is increasing and the resulting wastes demand management through further uses.

Combustion of biomass results in two types of ash. These are Biomass Bottom Ash (BBA), primarily larger particles that fall through the grate during combustion, and Biomass Fly Ash (BFA), which refers to the very fine particles that are carried in the flue gases. BBA and BFA have been used with OPC as cement replacement (up to 40 %) in various studies such as Johnson *et al.* (2010) and Rajamma *et al.* (2009).

3.6.1.8 Flue gas desulphurisation gypsum

Flue Gas Desulphurisation (FGD) is the most extensively utilised system by air pollution control equipment in coal-fired power plants for exclusion of released sulphur. As a result of this process the by-product FGD gypsum is produced due to lime utilisation to absorb sulphur.

3.6.2 Enhancing cold BEMs by incorporating virgin materials

There are many investigations that have been undertaken to upgrade the mechanical properties of the cold BEMs utilising virgin materials. The most common hydraulic binders used in the UK comprise Portland cement, GGBS and lime. Cement and lime are the most extensively used cementitious components for cold BEMs.

An initial study conducted by Head (1974) concentrated on the development on Marshall Stability of the modified cold asphalt mix. He stated that Marshall Stability of modified cold asphalt mix increased by about three times with the addition of 1 % Portland cement compared with un-treated mix.

Milton and Earland (1999) indicated that cement may be required either as the primary binder or as a supplementary binder to act as an adhesion agent or help to improve the short-term properties of the compacted mixtures.

Needham (1996) and Brown and Needham (2000) showed that incorporation of cement into cold BEMs can enhance the stiffness modulus, resistance to permanent deformation, and resistance to fatigue cracking (at an initial strains below 200 micro strain), and improve resistance to water damage. Figure 3-10 shows the effect of addition different levels of OPC to the cold BEM, Figure 3-11 shows the creep performance in terms of percentage of axial strain treated cold mixtures, and Figure 3-12 illustrates fatigue performance of those mixtures.

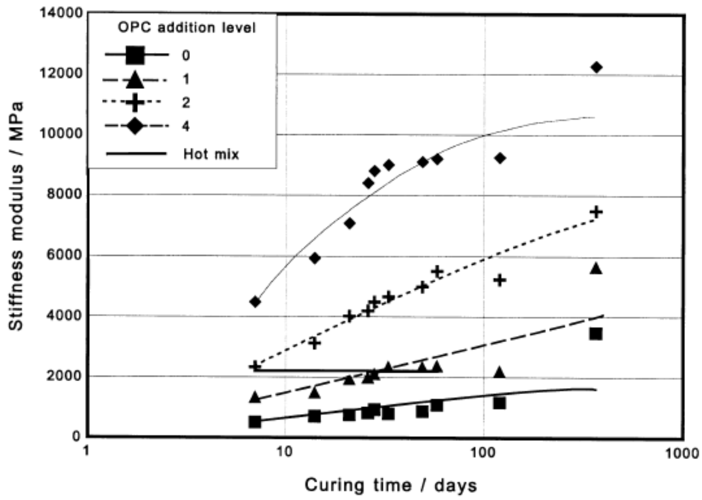


Figure 3-10: Effect of OPC on stiffness modulus (Brown and Needham, 2000), permission to reproduce this figure has been granted by Association of Asphalt Paving Technologists

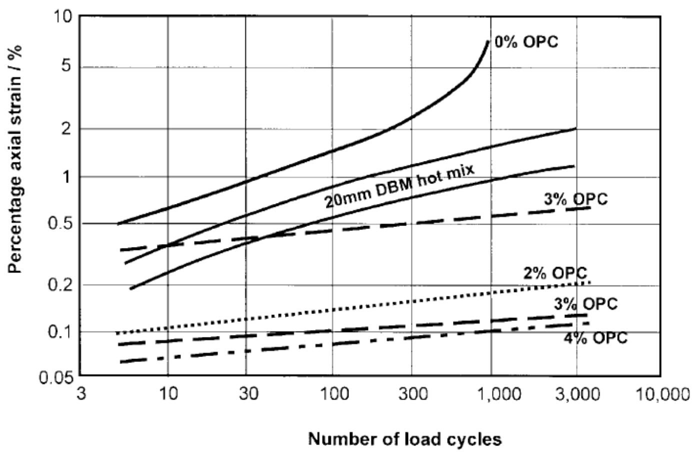


Figure 3-11: Resistance of permanent deformation of cold emulsion mixtures with different percentages of OPC (Brown and Needham, 2000), permission to reproduce this figure has been granted by Association of Asphalt Paving Technologists

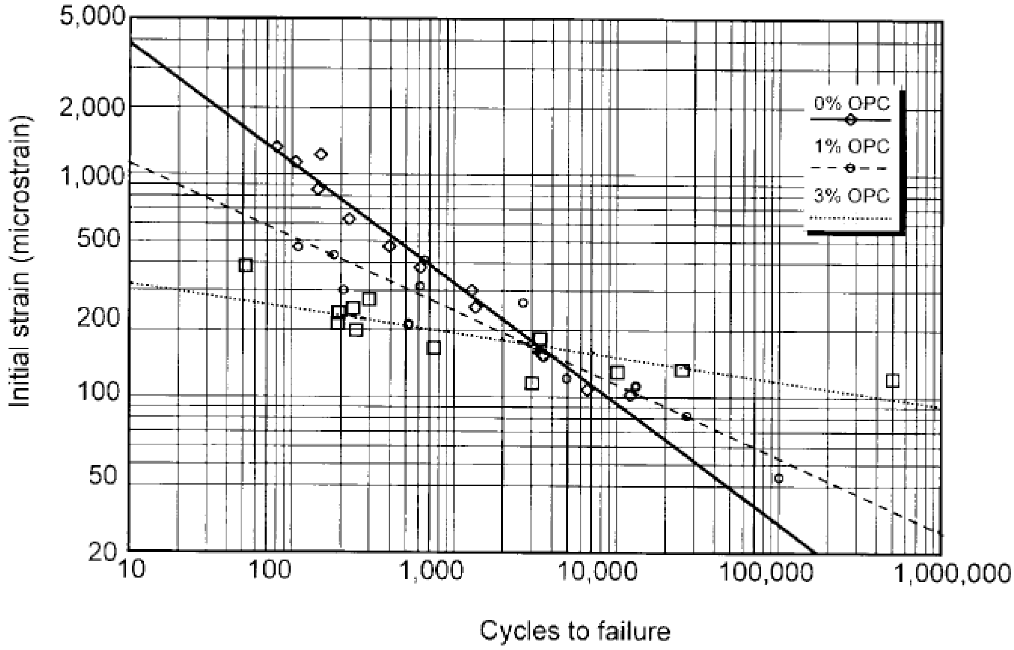


Figure 3-12: Cycles to failure Vs. initial strain for cold emulsion mixtures with different percentages of OPC (Brown and Needham, 2000), permission to reproduce this figure has been granted by Association of Asphalt Paving Technologists

An experimental study carried out by Li *et al.* (1998) assessed the mechanical properties of a three-phase Cement-Asphalt Emulsion Composite (CAEC). This study indicated that CAEC achieved most of the characteristics of cement and asphalt together, i.e. the longer fatigue life and lower temperature susceptibility of cement concrete, and the higher toughness and flexibility of asphalt concrete.

Laboratory studies carried out by Giuliani (2001) illustrated that incorporating cement into the emulsion improved the performance of the treated mixtures when compared with that of traditional hot mix asphalt. He stated that cement proved to be a regulating element of the emulsion breaking, by increasing the viscosity of the bitumen and contributing to the creation of new bonds in the mixture. Actually, physical and chemical properties of asphalt are influenced by cement addition, even though with small doses. Also, he observed that cement addition has a beneficial action in producing an excellent creep performance and a high stiffness modulus in laboratory samples, even after they were submerged in water.

Pouliot *et al.* (2003) introduced a detailed study in terms of hydration process, the microstructure and the mechanical properties of mortars with a new mixed binder which is prepared from a cement slurry and a small quantity of anionic bitumen emulsion (SS-1) or cationic bitumen emulsion (CSS-1). They concluded that the existence of a small quantity of emulsion had an effect on the hydration process of cement. In addition, they found that the cationic slow setting emulsion (CSS-1) showed higher mortar strengths and elastic modulus compared with anionic emulsion (SS-1).

Oruc *et al.* (2007) implemented experiments to assess the mechanical properties of emulsified asphalt having 0–6 % Portland cement. The test results revealed considerable improvement with high proportional additions of Portland cement, Figure 3-13. Furthermore, they recommended that the cement modified asphalt emulsion mixes might be used as a structural pavement layer.

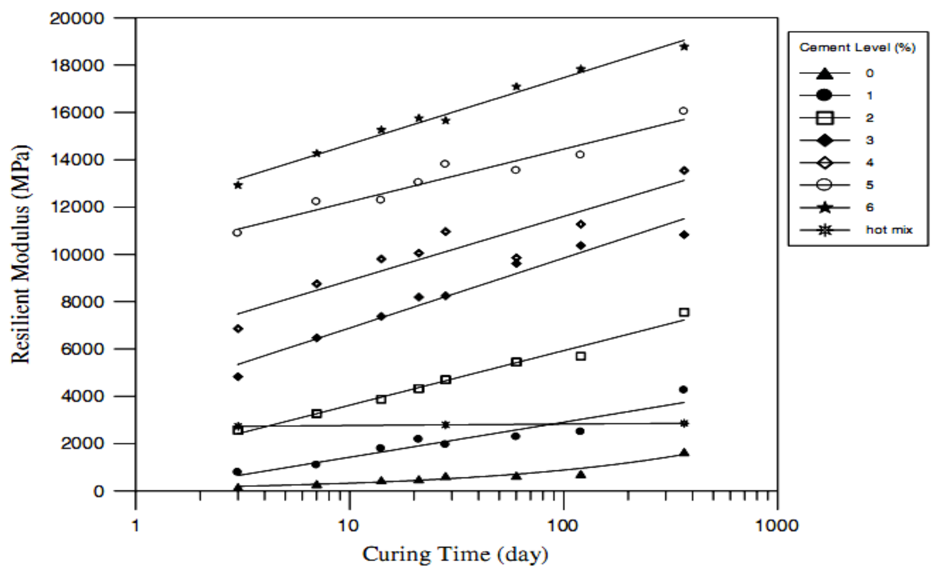


Figure 3-13: Enhancing stiffness modulus by addition of OPC (Oruc *et al.*, 2007), permission to reproduce this figure has been granted by Springer Science+Business Media

Another study, conducted by Thanaya *et al.* (2009), concluded that the addition of 1–2 % rapid-setting cement increased the strength development and improved the mechanical properties of the modified cold mixes, especially in the early days as shown in Figure 3-14.

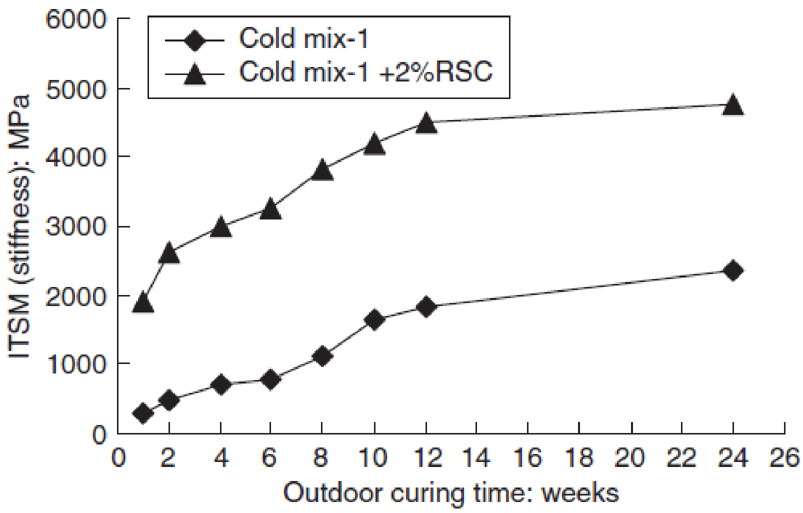


Figure 3-14: Rate of strength gain of outdoor cured cold mixtures (Thanaya *et al.*, 2009), permission to reproduce this figure has been granted by ICE Publishing

The study implemented by Wang and Sha (2010) determined that the rise of cement and mineral filler fineness has a progressive impact on the micro-hardness of the interface of aggregate and cement emulsion mortar. Also, they concluded that the limestone and limestone filler impact hardness values are higher than those for granite and granite filler.

Additionally, lime as a secondary binder mixed with road planing has been tried and proved successful (Cliff *et al.*, 2004). The main problem with binders such as hydraulic cement or lime is that they reduce the shelf life. Brown and Needham (2000) conducted a comparison study with different added materials, i.e. OPC, lime and $CaCl_2$. It can be seen from Figure 3-15 that hydrated lime and $CaCl_2$ do not have the same useful effect as OPC on stiffness modulus values. As lime has been seen to cause bitumen emulsion to break in other tests, these results suggest that OPC enhances the stiffness of a mixture by stiffening the binder but lime does not.

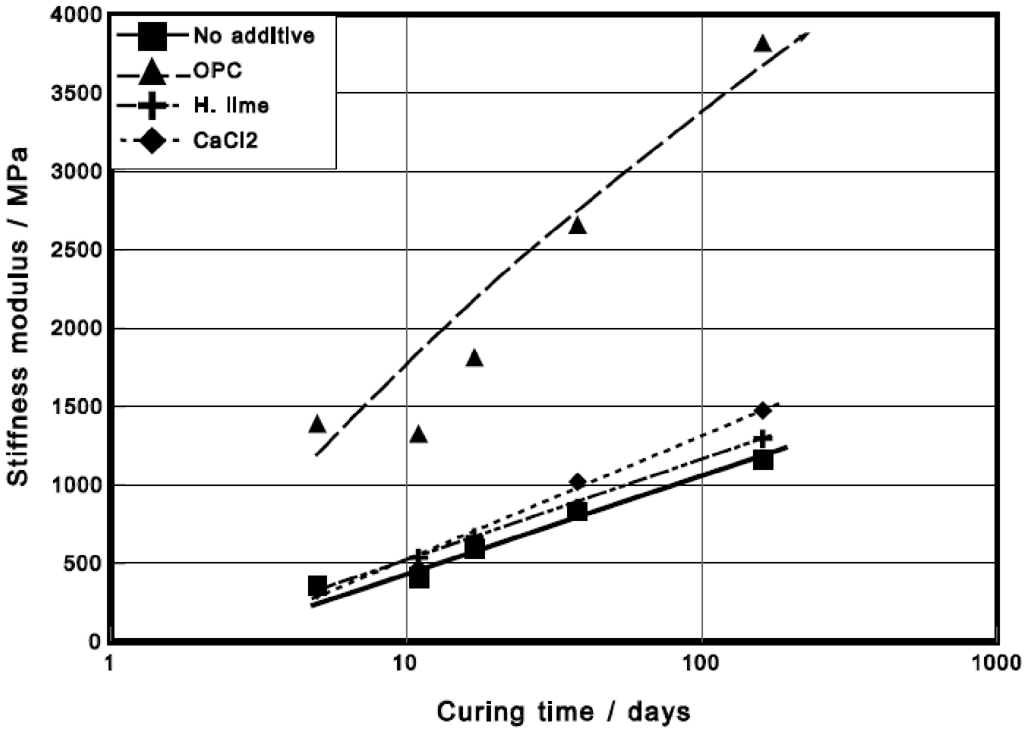


Figure 3-15: Stiffness modulus of cold emulsion mixtures with OPC, lime and $CaCl_2$ (Brown and Needham, 2000), permission to reproduce this figure has been granted by Association of Asphalt Paving Technologists

3.6.3 Enhancing cold BEMs by incorporating waste and by-product materials

Several attempts have tried the use of SCMs to improve the performance of cold BEMs; hence attractive benefits can be achieved when using these waste and by-product materials, as indicated in 3.6.1.

A cementitious material consists mainly of gelenite and lime; therefore it possesses cementing properties when mixed with water. On the other hand, a pozzolanic material has little or no cementitious behaviour, but when ground and mixed with water it reacts with calcium hydroxide produced from the hydration process between cementitious material and water, and therefore further activating the C–S–H generation. Some of the SCMs behave as cementitious material such as WPSA and GGBS and some of them as pozzolanic material such as PFA, SF and RHA.

Cliff *et al.* (2004) have studied a range of storage macadam composed of recycled aggregates from different sources and bound by bitumen emulsion and GGBS. They stated that the addition of GGBS to bitumen emulsion mixtures can give high levels of mechanical performance; especially over the long term and in conditions of high humidity.

Research conducted by Thanaya *et al.* (2006) indicated that the PFA can be used as a suitable filler in cold BEMs. Moreover, they found that the stiffness modulus of these mixtures is very comparable to hot mixtures after full curing conditions.

Studies conducted by Al-Busaltan *et al.* (2012) reported the upgrading of close graded cold BEMs to a stage whereby the mechanical properties are comparable to traditional asphalt concrete. This was due to the replacement of the conventional mineral filler with LJMU-FA1 (which is a waste domestic fly ash) and another binder generated from the hydration process between LJMU-FA1 and the trapped water included in cold mixtures.

Ameri and Behnood (2012) implemented a study to evaluate the effectiveness of steel slag as a supernumerary for virgin aggregates on mechanical properties of Cold In Place Recycling (CIR) mixes. The mixture's graduation requirements were satisfied by adding 20 % and 10 % of two types of new aggregates, i.e. limestone and steel slag. They concluded that the use of steel slag can improve Marshall Stability, resilient modulus, tensile strength, resistance to water damage and resistance to permanent deformation of CIR mixes.

The author of this thesis, together with his supervisory team, have published several journal and conference papers covering the utilisation of different SCMs in cold BEMs during his research period. All of these studies dependent on gap graded aggregate gradation normally used in the UK to produce Hot Rolled Asphalt (HRA) suitable for surface course mixtures. Also, it is worth noting that none of the previous studies covered the production of cold BEMs with this gradation although many advantages have been reported with HRA in comparison with different types of mixtures. Accordingly, the researchers, i.e. the author and his supervisory team have referred to their novel mixtures as Cold Rolled Asphalt (CRA).

Earlier trials dealt with producing CRA incorporating OPC as filler (Al-Hdabi *et al.*, 2013c). It has been concluded from these studies that replacement of the conventional mineral filler with OPC into the CRA mixtures significantly improves the mechanical properties of these mixtures.

Then this team tried to improve CRA mixtures containing OPC as filler by adding or replacing some of the OPC with one of the SCMs. The first attempt was conducted by addition of up to 3 % of the total aggregate of SF to the CRA containing from 0–6 % OPC (Al-Hdabi *et al.*, 2013c). The main conclusion was that the mechanical properties of the produced mixtures developed considerably and production of CRA mixtures with low levels of OPC can be achieved when adding SF. After that the idea of incorporating another very high silica content, i.e. Rice Husk Ash (RHA), to CRA mixtures was implemented (Al-Hdabi *et al.*, 2013e). Generally, the mechanical properties as well as water sensitivity of CRA mixtures containing OPC have been improved because the addition of high silica RHA activates the hydration process between OPC and the trapped water incorporated in those mixtures. Another CRA has been introduced (Al-Hdabi *et al.*, 2013b) using a new binary blended filler which is generated from blending different percentages of OPC and Poultry Litter Fly Ash (PLFA). In terms of stiffness modulus, the optimum blend was 4.5 % OPC plus 1.5 % PLFA. According to this study, the mechanical properties and water sensitivity results for the produced mixtures, i.e. CRA containing 6 % binary blended filler, are better than those for control CRA, CRA containing 6 % OPC and conventional HRA.

The last step was producing fast curing CRA surface course mixtures incorporating unary, binary and ternary blended filler which are generated totally from SCMs. The main guide for choosing unary filler from SCMs was investigating the cementitious reactivity of some waste and by-product material. According to Al-Hdabi *et al.* (2013a) and Al-Hdabi *et al.* (2012), replacement of conventional mineral filler with WPSA enhanced the mechanical properties and moisture resistance of the produced CRA mixtures due to the hydration process between WPSA and the trapped water incorporated in these mixtures. Also, the performance of those mixtures is better than control CRA as well as CRA containing OPC. Another study implemented two types of SCMs, namely WPSA and PLFA (Al-Hdabi *et al.*, 2013d). As per this research, a new Binary Blended Filler (BBF), which consists of 4.5 % WPSA plus 1.5 % PLFA, has been produced by means of optimum stiffness modulus results. In general, there is a significant improvement by means of stiffness modulus, creep performance and durability from using CRA containing 6 % BBF when compared with control CRA, CRA containing 6 % OPC and conventional HRA.

3.7 Summary of background literature review

A detailed review of literature was conducted in the previous chapter concentrating on manufacturing, components, characteristics, breaking mechanism and applications of bitumen emulsion especially cationic bitumen emulsion type; whereas this chapter has focused on the bitumen emulsion mixtures by means of types, production process, advantages, disadvantages and the available design procedures and specifications related to these mixtures.

Energy saving, environmental benefits and safety are the main advantages when adopting cold BEMs instead of conventional HMA. On the other hand, low early strength, long curing time to achieve the required properties and high air voids content were reported as the main challenges for these mixtures.

Furthermore, previous studies concluded that the incorporation of cementitious binder such as OPC as a replacement for the conventional mineral filler increases the early strength and decreases the curing time but causes an increase in the total cost of cold BEM product as it represent a cost minus material.

Therefore, it is proposed that there is an ability to replace the conventional mineral filler with different types of SCM individually or collectively to overcome the main deficiencies of cold BEMs and be comparable with the conventional HMA. Chapter 4 will focus on the methodology, candidate SCM properties, selected CRA ingredients, the available type testing and the research programme.

Chapter Four

Research Methodology and Characterisation of Materials

The main concept of this research work is to develop novel Cold Rolled Asphalt (CRA) as an alternative to the conventional Hot Rolled Asphalt (HRA) for use in heavily trafficked roads. After optimisation of their ingredients, and the mixing and compaction process, the new materials should have mechanical, durability and volumetric properties comparable with those of the conventional HRA and meet the current requirements of the European code of practice. It is worth stating that, although extensive studies have been conducted on developing different types of BEMs, no single study covered producing these mixtures with gap graded materials suitable for heavily trafficked surface courses and incorporating waste or/and by-product materials individually or collectively.

This chapter covers the characterisation of the primary materials, characterisation of the candidate SCMs and the research methodology.

4.1 Materials

4.1.1 Aggregate

The coarse and fine aggregate used in this investigation were crushed granite from Bardon Hill quarry, Leicestershire, UK, which are normally used to produce traditional HRA mixtures; Table 4-1 shows their physical properties. The aggregates were dried and sieved as per BS EN 933-1 (European Committee for Standardization, 2012a) to achieve the required gradation.

Table 4-1: Aggregates' physical properties

Material	Property	Value
Coarse aggregate	Bulk particle density, Mg/m^3	2.79
	Apparent particle density, Mg/m^3	2.83
	Water absorption, %	0.6
Fine aggregate	Bulk particle density, Mg/m^3	2.68
	Apparent particle density, Mg/m^3	2.72
	Water absorption, %	1.6
Mineral filler	Particle density, Mg/m^3	2.71

4.1.2 Bitumen emulsion and bitumen

Cationic slow setting bitumen emulsion (C56B7) was used to prepare all the new CRA mixtures with or without replacement of the conventional mineral filler. According to Nikolaids (1994b), cationic emulsion is more preferable due to its ability to coat the given aggregate and to ensure high adhesion between aggregate particles. Table 4-2 shows the properties of the selected bitumen emulsion.

Table 4-2: Properties of (C56B7) bitumen emulsion

Bitumen emulsion (C56B7)	
Property	**Value**
Appearance	Black to dark brown liquid
Relative Density at 15 ºC, g/ml	1.05
Residue by distillation, %	56
Residual bitumen penetration, 1/10 mm	100

On the other hand, two grades of bitumen have been used to produce HRA mixtures, which were 100/150 and 40/60. The properties of these bitumens are illustrated in Table 4-3.

Table 4-3: Properties of 100/150 and 40/60 bitumen binders

Bituminous binder 40/60		**Bituminous binder 100/150**	
Property	**Value**	**Property**	**Value**
Appearance	Black	Appearance	Black
Penetration at 25 ºC	43	Penetration at 25 ºC	122
Softening point, ºC	54	Softening point, ºC	43.6
Density at 25 ºC	1.02	Density at 25 ºC	1.05

4.1.3 Experimental techniques used for characterisation of filler materials

The utilisation of a new substance requires advanced analytical characterisation to describe its properties and the relationship between physical-chemical structures of the substance. Grasserbauer (1989) reported that utilising of highly integrated microelectronic, structure analysis of surfaces with laser beams and x-ray fluorescence aided devices is helpful for analysing materials at micro and molecular level. Also, they introduce important information such as interrelationships amongst physical, chemical, mechanical and durability properties of the generated products. In this research, four analytical techniques were used for the analysis, characterisation, and evaluation of filler materials in anhydrous state which are:

4.1.3.1 Particle size analysis

The influence of Particle Size Distribution (PSD) and particle shapes and sizes plays a considerable role in the development and implementation of sustainable BEMs. The grinding process and the properties of fresh and hardened paste of pozzolanic and hydraulic fillers are strongly affected by their PSD.

In this study, a Beckman coulter laser diffraction particle size analyser (LS 13 320, Figure 4-1) was utilised to indicate the grain size distribution of all the filler powders. This analyser utilises reverse Fourier optics incorporated in a patented fibre optic spatial filter system and a binocular lens system. Beckman Coulter (2012) stated that this aids the LS 13 320 to optimise light measurement across the widest dynamic range, from 0.040 to 2000 µm in a single scan in Aqueous Liquid Mode (ALM). Five consecutive runs were taken for each measurement of particle size analysis and the averages have been recorded to describe the material in this research.

Figure 4-1 : LS 13 320 particle size distribution analyser

4.1.3.2 X-ray fluorescent and X-ray powder diffraction

Angstrom range wavelengths of X-rays are well poised to examine the internal structure of solid substances because they are sufficiently energetic to penetrate these substances. According to this characteristics behaviour of X-rays, there are two extensively utilised techniques, namely Energy Dispersive X-Ray Fluorescence (EDXRF) for elemental analysis and X-Ray Powder Diffraction (XRPD) for structural and phase composition studies.

X-ray fluorescent

In this study, a Shimadzu EDX 720, Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer, shown in Figure 4-2, was used to determine the quantitative elemental composition, i.e. principal oxides and trace elements. The X-ray intensities at each energy were compared to values for known standards for quantitatively analysis of the identified specimen.

Figure 4-2: Shimadzu EDX 720 spectrometer

X-ray powder diffraction

A Rigaku Miniflex diffractometer (Miniflex gonimeter) with CuK X-ray radiation, voltage 30 kV, and current 15 mA at scanning speed of 2.0 deg./min in continuous scan mode with aluminium sample holder was utilised to indicate the phase composition for the selected filler materials as well as the paste produced from mixing these materials with water, Figure 4-3.

Figure 4-3: Rigaku Miniflex X-Ray diffractometer

The selected filler powders were investigated dry, whereas the paste samples at a specified age were dried then ground to a fine powder (passing sieve no. 200) before analysing in XRD tester. To identify the recorded diffraction pattern, it was compared with the standard patterns of various compounds available in the powder diffraction file database. Also, Mercury 2.3 software has been utilised to visualise, standardise and analyse the crystallisation of the XRD analysis.

4.1.3.3 Scanning electron microscopy

Scanning Electron Microscopy (SEM) is a method for high-resolution imaging of surfaces which is used to simultaneously examine the morphology of an object and analyse its elemental composition. The selected filler materials and the paste generated from mixing those materials with water need to be coated with a thin layer of electrically conductive material because they tend to be nonconductive. Therefore, in this investigation a gold coating was used on powders as well as fractured surfaces of paste specimens at different ages.

4.1.4 Characterisations of selected filler materials

4.1.4.1 Limestone dust

Conventionally, limestone dust is used as mineral filler in preparation of hot bituminous mixtures. Limestone dust is an inert material when incorporated in BEMs, i.e. there is no pozzolanic or/and cementitious reaction from this material and accordingly CRA mixtures prepared with limestone dust are referred to in this investigation as control CRA.

In the present study, limestone dust with 8.5μm mean diameter (d50) and about 1 % of its particles retained on sieve 45μm has been used, as shown in Figure 4-4. The chemical composition in EDXRF and the mineralogical configuration in XRPD analysis of limestone dust are shown in Table 4-4 and Figure 4-5, respectively. Also, Figure 4-6 shows the SEM view of limestone dust particles. The main crystal peaks identified in the XRPD pattern were quartz-Q (SiO_2), calcite-C ($CaCO_3$), and dolomite-D ($CaMg(CO_3)_2$).

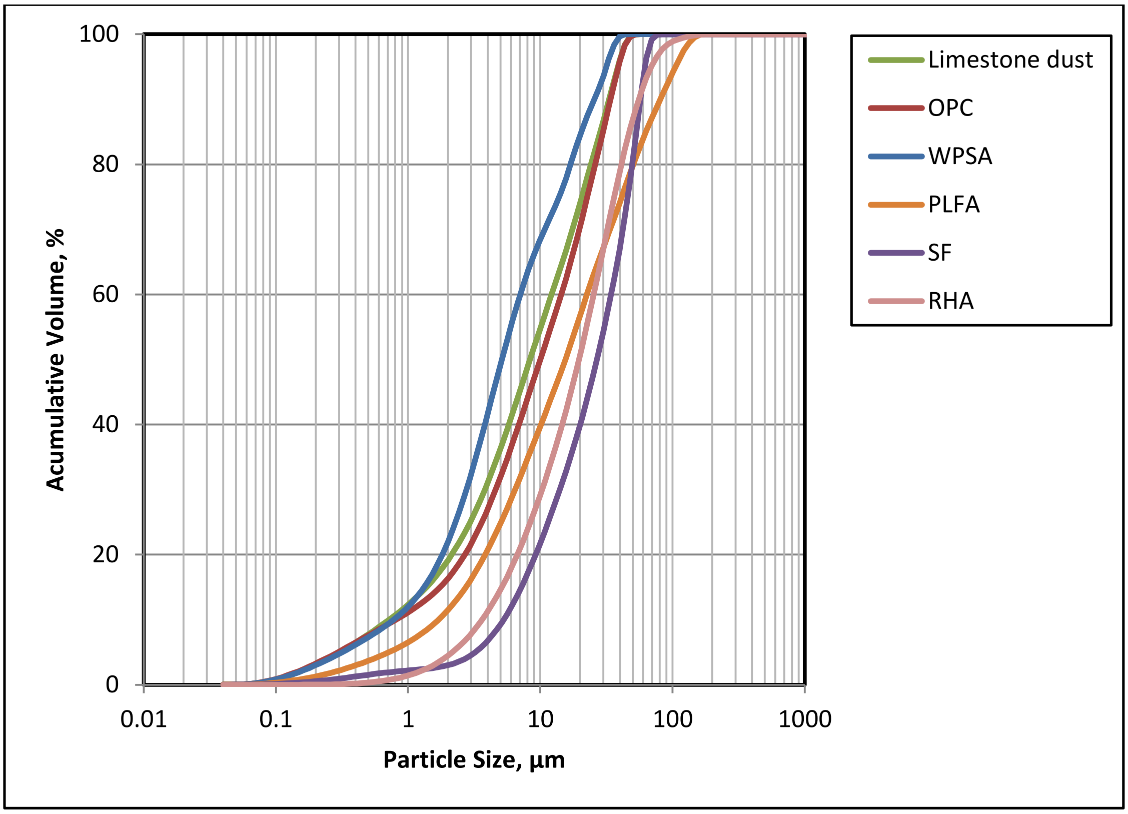


Figure 4-4: Particle size distribution of the selected filler materials

Table 4-4: Chemical composition of the selected filler materials, %

	CaO	**SiO_2**	**Al_2O_3**	**MgO**	**Fe_2O_3**	**K_2O**	**TiO_2**	**pH**
Limestone dust	74.95	15.06	---	1.00	0.26	0.234	0.18	11.10
OPC	61.55	21.98	4.65	4.27	2.27	1.04	0.40	12.54
WPSA	62.21	27.25	2.87	3.11	0.15	0.35	0.57	12.78
PLFA	9.91	14.38	---	0.60	0.01	21.33	0.16	12.39
SF	0.41	91.48	11.45	0.72	2.01	0.36	0.04	8.30
RHA	1.58	89.51	11.2	0.60	---	3.24	0.06	8.03

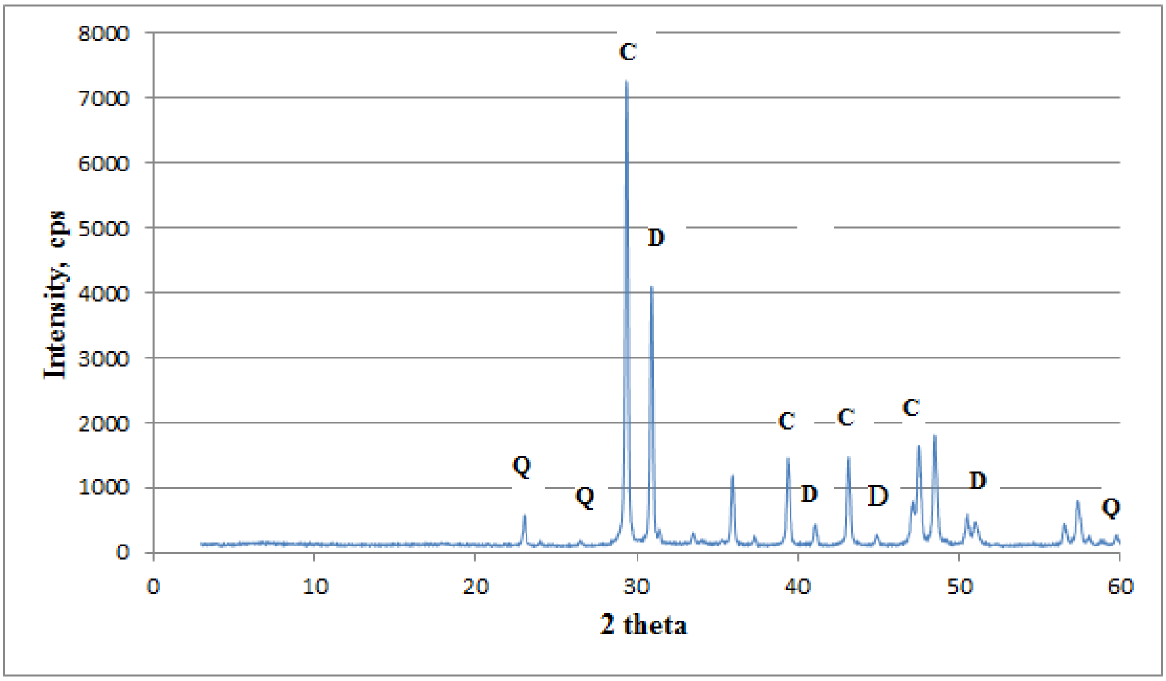


Figure 4-5: Experimental XRPD pattern of limestone dust with peaks corresponding to common minerals highlighted (quartz-Q, calcite-C, and dolomite-D)

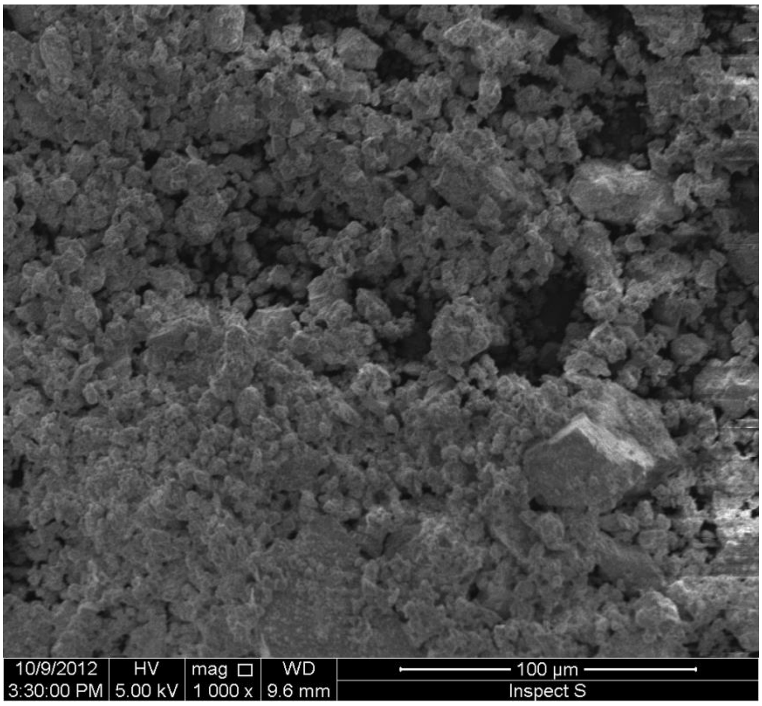


Figure 4-6: SEM view of limestone dust particles at 100 µm

4.1.4.2 Ordinary Portland cement

CEM-II/A/LL 42.5-N cement type was utilised throughout the present study as a respected hydraulic material to prepare CRA mixtures incorporating cementitious filler. Table 4-4 shows the chemical compositions of OPC which demonstrate the high CaO content. The XRPD analysis of OPC revealed that it is composed of calcite-C (CaCO3), alite-A (3CaOSiO2), gelenite-G (Ca2Al2SiO7), belite-B (2CaOSiO2), ferrite (4CaOAl2O3.Fe2O3), and periclase (MgO), as shown in Figure 4-7.

The particle size distribution, as shown in Figure 4-4, shows that the mean diameter d50 is 9.9μm and approximately 1 % of the particles were retained on 45μm sieve size. The crystal-shaped particles are revealed from the SEM view, Figure 4-8.

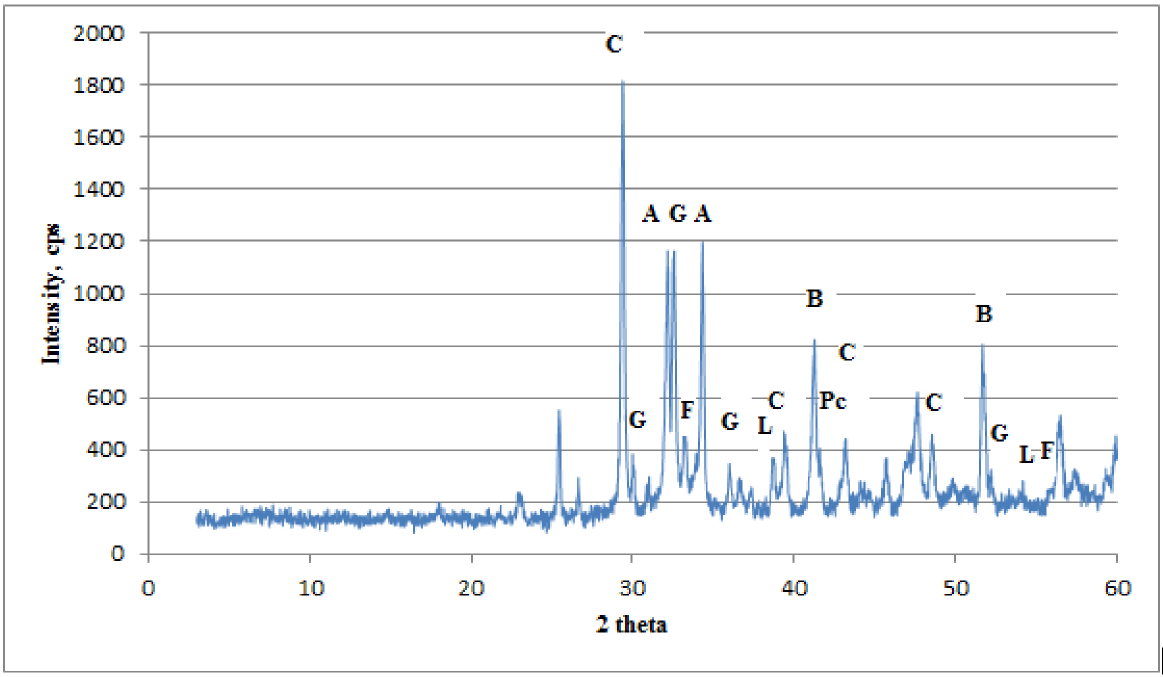


Figure 4-7: Experimental XRPD pattern of OPC with peaks corresponding to common minerals highlighted (calcite-C, alite-A, gelenite-G, belite-B, ferrite-F, and periclase-Pc)

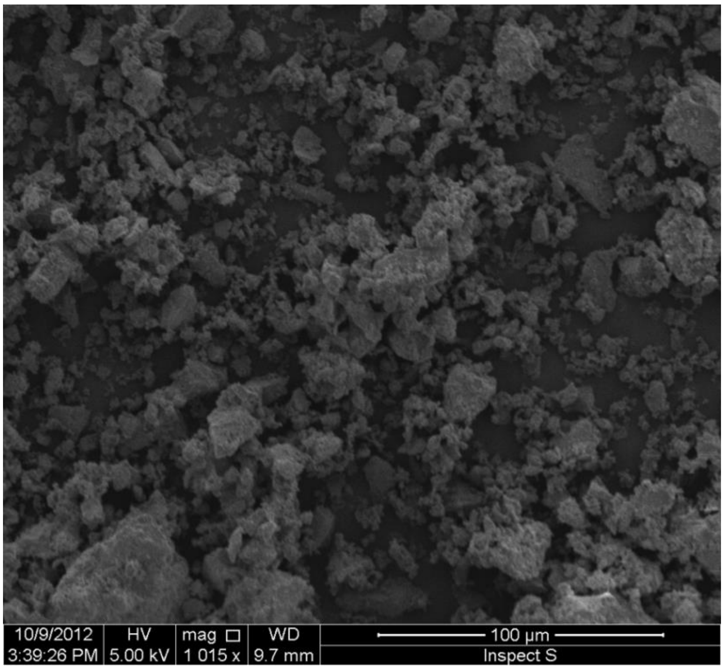


Figure 4-8: SEM view of OPC particles at 100 µm

4.1.4.3 Waste paper sludge ash

Combustion of waste paper sludge at about 850 ºC in power generation plants utilising fluidised bed combustion generates Waste Paper Sludge Ash (WPSA). Calcium hydroxide ($Ca(OH)_2$) and activated carbon is used to capture sulphur in fluidised beds and accordingly nitric oxide (NO_x) is released during this process. The generated solid ash is considered to comprise the inorganic fraction of the utilised fuel, extreme sorbent and the compound of sulphur generated from the desulphurisation reaction.

Annually more than 1 million tonne of WPSA is produced in Europe and about 125 thousands of this is produced in the UK alone. Presently, there are three paper mills producing WPSA from incineration of waste sludge in the UK. One of these mills, Aylesford Newsprint Ltd, is the biggest recycled newsprint mill in Europe and is the only supplier of WPSA to this research.

As shown in the particle size analysis, Figure 4-4, the d50 and d90 of ground WPSA particles are 4µm and 25µm, respectively. The properties are complying with the requirements of the active supplementary cementations materials, i.e. the materials retained on sieve No. 325 (45µm) should be not more than 34 %.

Chemical analysis of the used WPSA is presented in Table 4-4 whilst X-ray powder diffraction (XRPD) pattern of dry WPSA is shown in Figure 4-9. The latter shows the sample to be crystalline as it contains sharp peaks and low background. Major crystal peaks identified in the XRPD pattern were calcite ($CaCO_3$), lime (CaO), gehlenite ($CaAl[AlSiO_7]$), merwinite ($Ca_3Mg(SiO_4)_2$, belite (Ca_2SiO_4), and mayenite ($Ca_{12}Al_{14}$- O_{33}). It is expected that WPSA will play a vital role as a cementitious material due to the high concentration of CaO and the existence of lime and gehlenite in the XRPD pattern.

Figure 4-10 shows the SEM analysis of the selected WPSA. It reveals the thin and flaky nature of the WPSA particles with high void spaces and hence a low level of compactness with high porosity is expected. Also, the crystal-shaped particles are shown in SEM views.

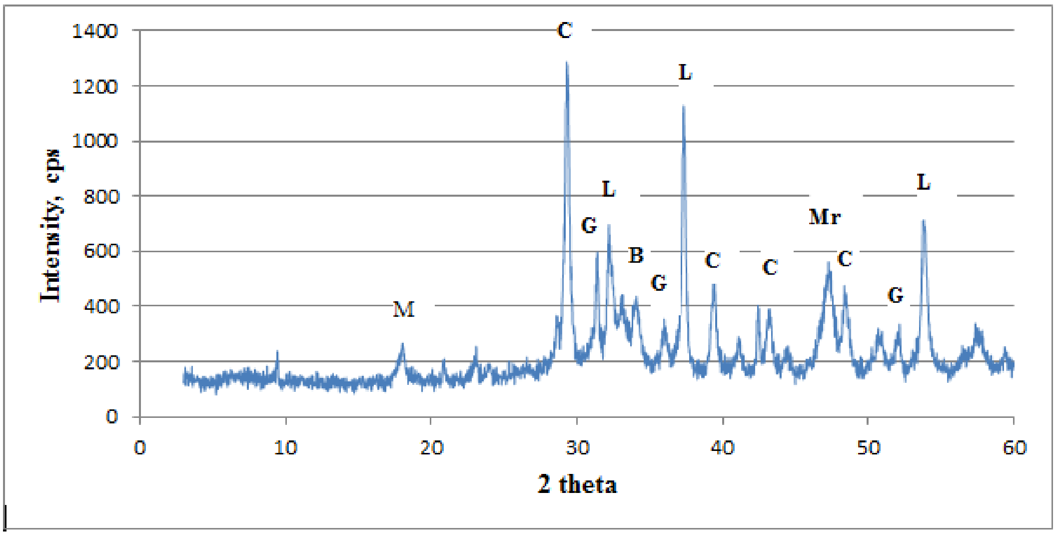


Figure 4-9: Experimental XRPD pattern of WPSA with peaks corresponding to common minerals highlighted (calcite-C, lime-L, gehlenite-G, merwinite-Mr, belite-B, and mayenite-M)

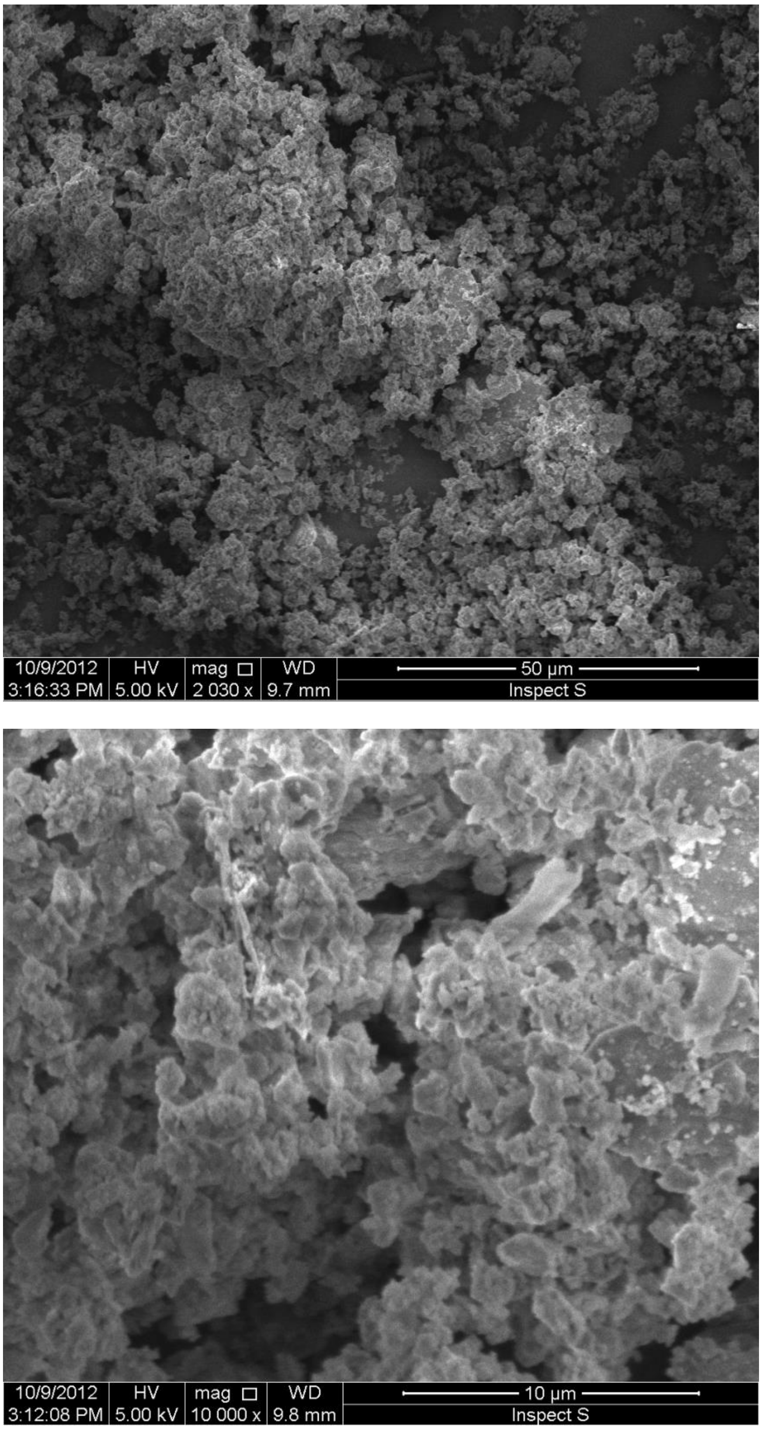


Figure 4-10**:** SEM views of WPSA particles at 50 μm and 10 μm

4.1.4.4 Poultry litter fly ash

Poultry Litter Fly Ash (PLFA) is produced from incineration of poultry litter in biomass power plants at a temperature between 900 to 1100 ºC with 2 seconds' residence time to generate electricity. Poultry litter consists of poultry manure and bedding material, predominantly straw, feathers and wood shavings and in some cases meat and bone meal. In this study, PLFA was collected from the Fibrowatt Thetford power station, which is one of the biggest power stations utilising biomass in the UK and consumes about 400,000 tonnes annually from poultry litter (Bioenergy Network on Socio-Economics, 2013).

Table 4-4 shows the major oxides for the used PLFA as identified by EDXRF, while Figure 4-11 illustrates the mineralogical profile according to XRPD analysis. The main crystalline phases are arcanite-K (K2SO4), sylvite-S (KCl), belite-B (2CaOSiO2), calcite-C (CaCO3) and orthoclase-O (KAlSi3O8).

The main difference between the chemical properties of PLFA and the conventional coal burning fly ash and WPSA is the high concentration of K_2O and associated arcanite which are expected to play a vital role. Poon *et al.* (2001) reported that the content of arcanite in PLFA is expected to provide an ambient environment for breaking the glass phase of cementitious materials such as OPC and WPSA. Also, it will play a catalytic role during the hydration process, especially in the early stage (Rodrigues *et al.*, 1999).

The PSD and SEM of PLFA are shown in Figures 4-4 and 4-12, respectively. The mean particle size d50 is 15.65µm, while the percentage of the materials retained on sieve 45µm is 23 %. The SEM micrograph reveals the non-agglomerated and non-spherical regular-shaped particles of PLFA.

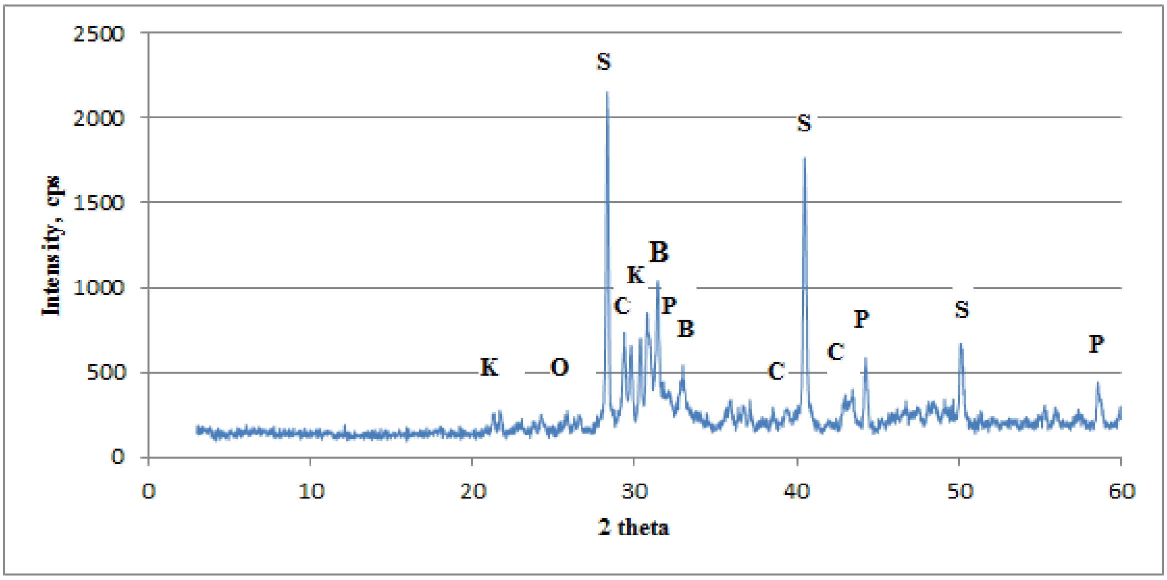


Figure 4-11: Experimental XRPD pattern of PLFA with peaks corresponding to common minerals highlighted (sylvite-S, belite-B, calcite-C, arcanite-K, pervoskite-P, and orthoclase-O)

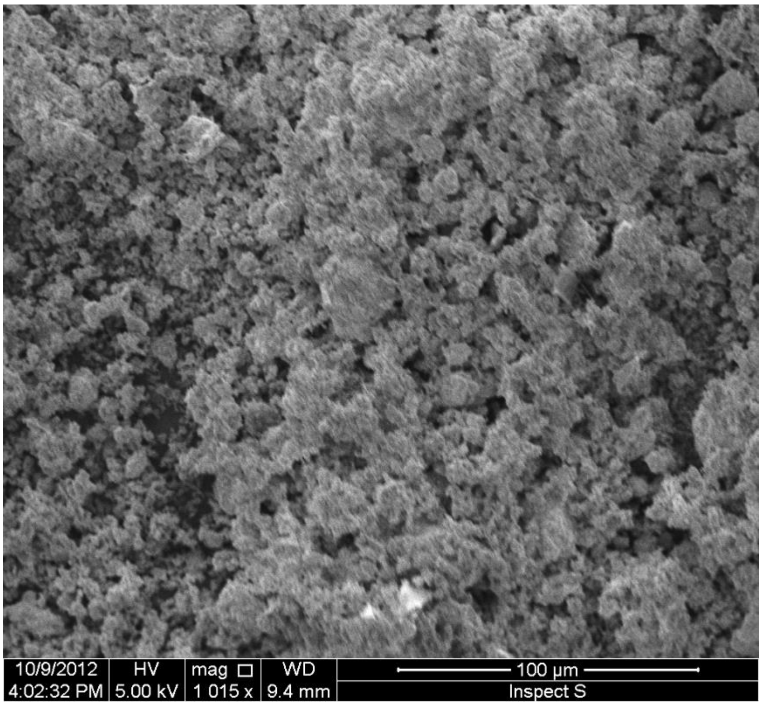


Figure 4-12: SEM view of PLFA particles at 100 µm

4.1.4.5 Silica fume

Silica Fume (SF) is a by-product from the manufacture of silicon or ferro-silicon metal and in this study it is collected from Elkem materials. Its chemical properties according to EDXRF analysis, as shown in Table 4-4, specified that it is composed of 91.48 % SiO_2 and complies with the data provided by the supplier. The amorphous silica particles are clearly shown in the mineralogical analysis by powder diffraction in XRPD, as illustrated in Figure 4-13.

The superfine particles of SF are clearly shown from the PSD diagram shown in Figure 4-4 with a mean diameter d50 of 27.1µm and about 25 % of the particles were retained on sieve 45µm. Figure 4-14 shows the agglomeration of these particles in a SEM micrograph.

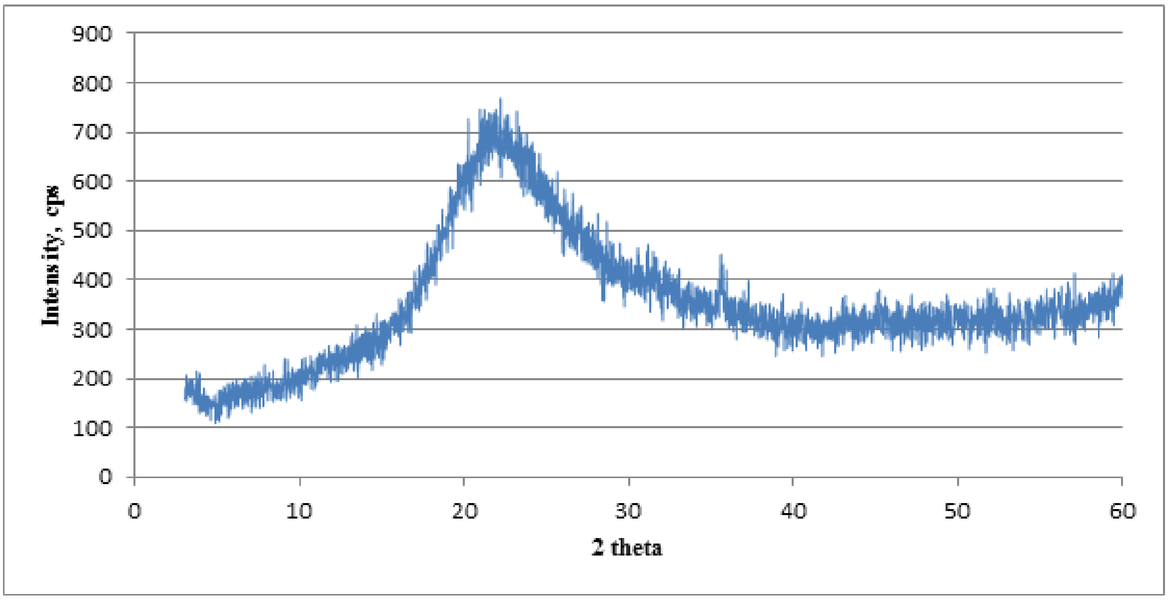


Figure 4-13: Experimental XRPD pattern of SF

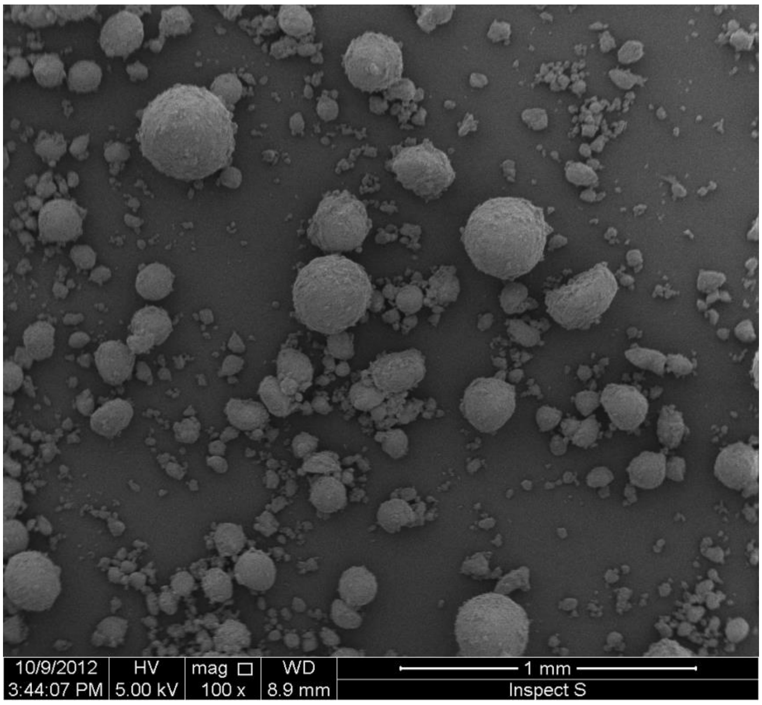


Figure 4-14: SEM view of SF particles at 1 mm

4.1.4.6 Rice husk ash

RHA can be generated from combustion of rice husk to generate energy or under controlled process (temperature and duration) to produce high silica RHA. In this study, RHA produced under controlled temperature (700–800 ºC) and duration (about 2 hours) has been used and was supplied by one of the RHA production companies.

The EDXRF analysis as shown in Table 4-4 indicates the presence of a high content of SiO_2 (89.51 %) whereas the XRPD analysis is similar to the SF one, Figure 4-15.

The PSD of RHA as shown in Figure 4-4 indicated that the mean diameter d50 is 19.4µm and 17 % of the materials are retained on sieve 45µm. The SEM micrograph, Figure 4-16, shows the non-spherical and non-agglomerated regular-shaped particles of RHA which represent the main difference to SF particles in terms of SEM micrograph. As the RHA has high silica content it is predicted to be a very promising and cheap pozzolanic material to activate the hydration process of cementitious material.

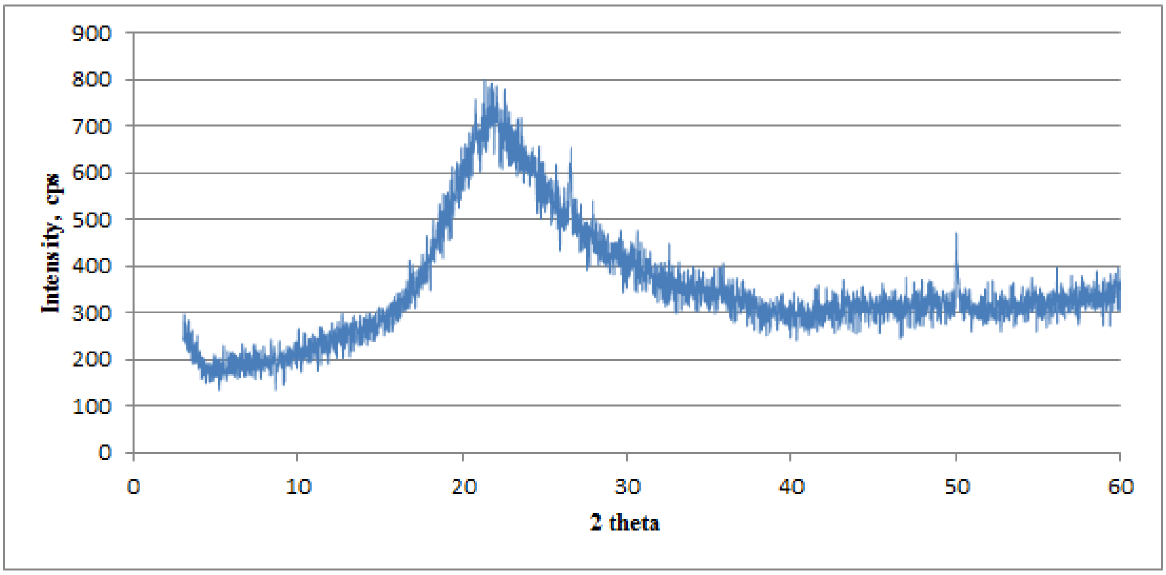


Figure 4-15: Experimental XRPD pattern of RHA

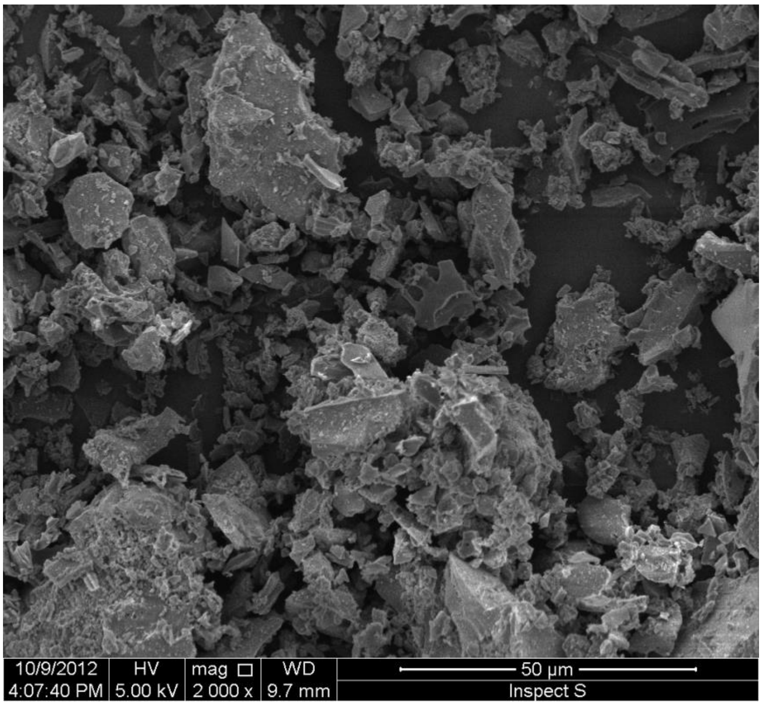


Figure 4-16: SEM view of RHA particles at 50 µm

4.2 Selected CRA and HRA gradation

Hot Rolled Asphalt (HRA) surface course is a gap graded mixture of coarse aggregate, sand, mineral filler and bitumen. The mechanical properties of the mixture are dominated by the strengthening properties of the mortar components, i.e. mineral filler, sand and bitumen. The material is extensively used for surfacing major roads in the UK because it provides a dense, impervious layer, resulting in a weather-resistant durable surface able to endure the demands of modern traffic loads and providing good resistance to fatigue cracking (Child, 1998).

Due to the high performance of HRA surface course mixtures, especially high stiffness values with low air voids content which are the main disadvantages of the produced BEMs, the author with his supervisory team and after a healthy discussion with Liverpool Centre for Materials and Technology (LCMT) industrial partners decided to develop a new BEM/s with a gap gradation conventionally used for HRA surface course mixtures. Accordingly, a 55/14C gap graded surface course mixture gradation was used to prepare CRA and HRA mixtures based on BS EN 13108-4 (European Committee for Standardization, 2006) for HRA; Figure 4-17 shows the selected gradation.

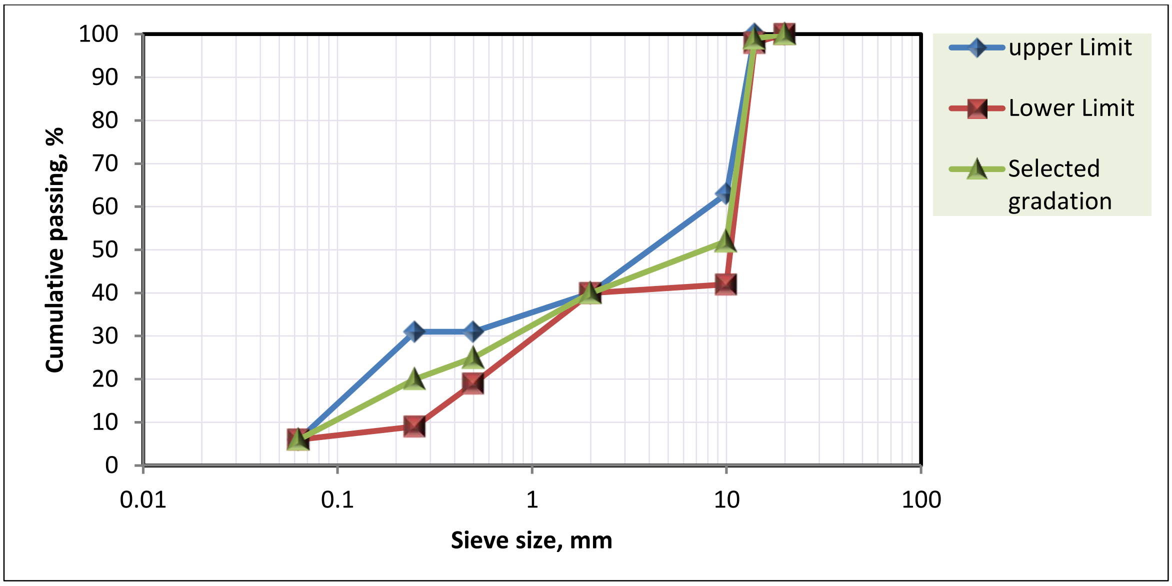


Figure 4-17: 55/14C Gap graded surface course aggregate gradation

4.3 CRA mixtures' preparation

4.3.1 Initial mix design

All CRA mixtures produced in this research were prepared based on the mix design procedure adopted by the Asphalt Institute (Asphalt Institute, 1989), as summarised in section 3.4.1. A brief description about the indicating of pre-mixing water content, Optimum Residual Bitumen Content (ORBC) and Optimum Total Liquid at compaction (OTLC) is given below.

- Pre-mixing water content

  The coating degree of the aggregate and bitumen emulsion is primarily controlled by the pre-mixing water content, particularly when the aggregate gradation contains a high percentage of filler such as the gap gradation under study. Thanaya (2003) reported that the best bitumen coating on aggregate can be achieved when the mixture is not too sloppy or too stiff. Different pre-mixing water contents were incorporated, i.e. 3–6 % by mass of aggregate with Initial Emulsion Content (IEC), calculated from equations 4-1 and 4-2 (IEC=11.75 %), to indicate the lowest pre-mixing water content with adequate coating. According to the visual inspection, 3 % pre-mixing water content was chosen in preparation of CRA mixtures.

$$p = (0.05\,A + 0.1\,B + \ 0.5\,C) \ \times (0.7) \qquad 4\text{-}1$$

Where:

P = the percentage of Initial Residual Bitumen Content (IRBC) by mass of total mixture,

A = the percentages of coarse aggregate (retained on sieve 2.00 mm),

B = the percentages of fine aggregate (passing sieve 2.00 mm and retained on sieve no. 0.063 mm), and

C = the percentages of filler (materials passing 0.063 mm)

While the second step is determination of IEC from another empirical formula as show below:

$$IEC = \left({}^{P}\!/_{X}\right) \qquad 4\text{-}2$$

Where:

IEC = Initial Emulsion Content by mass of total mixture, and

X = the bitumen content of the emulsion.

- Optimum Residual Bitumen Content (ORBC)

  Indirect Tensile Strength (ITS) was used to determine the ORBC. Two sets of samples have been prepared with different RBC, i.e. 5.5, 6, 6.5, 7, and 7.5 %, by total mass of aggregate. The first set was tested dry while the second one was tested after soaking. Accordingly, ORBC was chosen to produce CRA mixture with optimum soaked ITS which was 7 %, i.e. Optimum Emulsion Content (OEC) = 12.5 %.

- Optimum Total Liquid Content (OTLC)

In this study, all the produced CRA mixtures were compacted directly after mixing; therefore OTLC was 15.5 % by total mass of aggregate. The possibility of producing high-quality BEMs without any delay between mixing and compaction which is required to produce high-density cold mixtures has been concluded in this research and is discussed in the next chapters.

4.3.2 Preparation of bituminous mixtures

4.3.2.1 Cold rolled asphalt

Specimens of CRA were mixed using a Hobart mixer. The aggregate and filler material with pre-wetting water content (3 %) were added and mixed for 1 min at low speed. Then bitumen emulsion (12.5 %) was added gradually during the next 30 s of mixing, and the mixing was continued for 1.5 min at the same speed. This mixing method is termed as normal mixing throughout this study. Also, standard Marshall Hammer (impact compactor) was utilised as the general compacting process with 50 blows to each face of the 100 mm diameter specimens.

4.3.2.2 Hot rolled asphalt

The two types of HRAs were prepared with different bitumen grade for comparison with the produced CRA mixtures, which were 100/150 and 40/60 penetration grade. HRA mixtures were prepared with the same aggregate type and gradation; 5.5 % optimum binder content for each type of bitumen was added based on the BS 594987 Annex H for the 55/14C HRA surface course design mixtures (British Standard Institution, 2010). 100/150 and 40/60 HRA mixtures were mixed at (150–160 ºC) and (165– 175 ºC), respectively. The same compaction effort and number of blows for CRA mixtures has been used to compact HRA mixtures.

4.4 Experimental type testing and research methodology

Bituminous mixtures' properties should be appraised using appropriate testing methods and there exists a wide range of different type testing and configurations to assess these properties. Some testing modes do not take into consideration the elastic properties of the bituminous mixtures, such as the Marshall Stability and the Indirect Tensile Strength (ITS) tests, whilst in other testing modes, such as the Indirect Tensile Stiffness Modulus (ITSM) test, the elastic properties of the bituminous mixtures play a significant role.

Currently, there is no single mixture design procedure; however, testing protocol or end result specifications for BEMs are universally accepted. Basically, most testing methods routinely used for characterising hot bituminous mixtures can be adopted for testing BEMs. However, some modifications are required, in respect of the nature of those mixtures, such as the influence of curing time and test temperature on mixture performance. Table 4-5 shows a summary of the experimental testing which were conducted throughout the present investigation.

Table 4-5: Test methods utilised for analysis of samples

Property tested	Test/Technique used
Mechanical Properties:	
➢ Indirect Tensile Stiffness Modulus (ITSM)	BS EN 12697-26: 2012
➢ Uniaxial Cyclic Creep Test (UCCT)	BS EN 12697-25: 2005
➢ Fatigue Test (Four-point bending test on prismatic shaped specimens (4PB))	BS EN 12697-24, Annex D: 2012
➢ Semi-Circular Bending test (SCB)	BS EN 12697-44: 2010
Durability Tests:	
➢ Water Sensitivity	BS EN 12697-12: 2008
➢ Long Term Ageing Test	SHRP A-003A
Volumetric properties:	
➢ Bulk Density	BS EN 12697-6: 2012
➢ Air Voids, Voids in Mineral Aggregate (VMA) and Voids Filled with Bitumen (VFB)	BS EN 12697-8: 2003

4.4.1 Mechanical properties tests

In this research, four types of tests were applied on respective specimen to assess the mechanical properties; namely, Indirect Tensile Stiffness Modulus (ITSM), Uniaxial Cyclic Creep Test (UCCT), Four-point bending test on prismatic shaped specimens (4PB) and Semi-Circular Bending test (SCB).

4.4.1.1 Indirect Tensile Stiffness Modulus

Stiffness modulus has been measured by applying indirect tension to cylindrical specimen as per BS EN 12697-26:2012 and the resultant modulus was named Indirect Tensile

Stiffness Modulus (ITSM) (European Committee for Standardization 2012). Cooper Research Technology HYD 25 testing apparatus was used to determine the ITSM results, Figure 4-18. Specimens of 100 mm diameter and 63±3 mm height were exposed to a transient load pulse across their vertical axis and the resultant deformations along the horizontal axis were measured using Linear Variable Differential Transducers (LVDTs). Normally, the measurements were taken in two orthogonal orientations. The test was conducted at 20 ºC as per the mentioned standard despite in case it is mentioned different than this temperature. To ensure that the specimens' temperature was at the required temperature, the specimens were conditioned at 20 ºC inside the temperature-controlled cabinet of the machine at least 4 hours before the test.

In this method, the selected details of applied load parameters were:

- The rise-time, which is the time taken for the applied load to increase from zero to maximum value, which was (124 ± 4) microstrain;
- Transient peak horizontal deformation was (5±2) µm;
- The pulse repetition period was (3.0 ± 0.1) s;
- The Poisson's ratio, a value of 0.35 shall be assumed for all temperatures;
- Number of conditioning load pulses was 5; and
- Number of test pulses was 5.

Figure 4-18: ITSM test using Cooper Research Technology HYD 25 apparatus

4.4.1.2 Uniaxial Cyclic Compression Test

The UCCT describes the method for indicating the permanent deformation characteristics of asphalts by means of a uniaxial cyclic compression test with the existence of a little confinement. In this test, the diameter of the loading platen is taken smaller than that of the sample to ensure a certain confinement under the applied cyclic axial stress. It is worth noting that, for gap graded mixes with a large stone portion which is the same gradation under study, confinement of the sample is necessary to predict realistic permanent deformation behaviour (European Committee for Standardization, 2005).

A schematic illustration of the test device is given in Figure 4-19, while Figure 4-20 shows the HYD 25 Cooper Technology test apparatus used here. A cylindrical test sample with a 150 mm diameter was maintained at 40 ºC conditioning temperature. This specimen is located between two plane parallel loading platens in which the upper platen has a 100 mm diameter. There is no further lateral confinement pressure utilised.

Throughout the test, the variation in height of the sample is recorded at a certain number of pulses. Accordingly, the relationship between the accumulative axial strains (ε) of the test sample with number of load applications (pulses) can be drawn to produce a creep curve, as given in Figure 4-21, which is required to investigate the creep performance of bituminous mixtures.

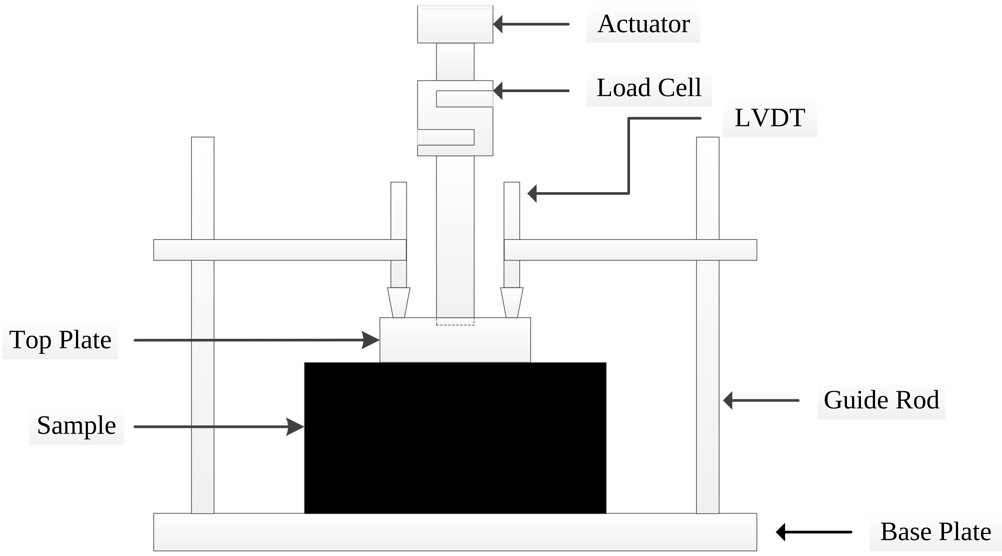


Figure 4-19: Schematic illustration of creep test

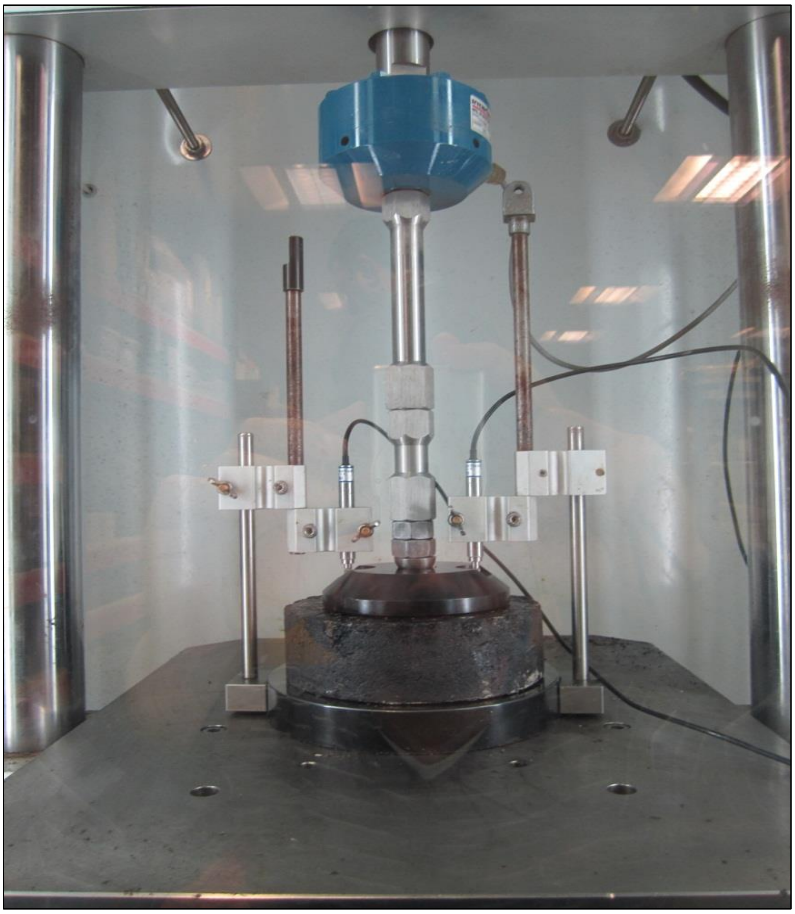


Figure 4-20: Test apparatus for UCCT test

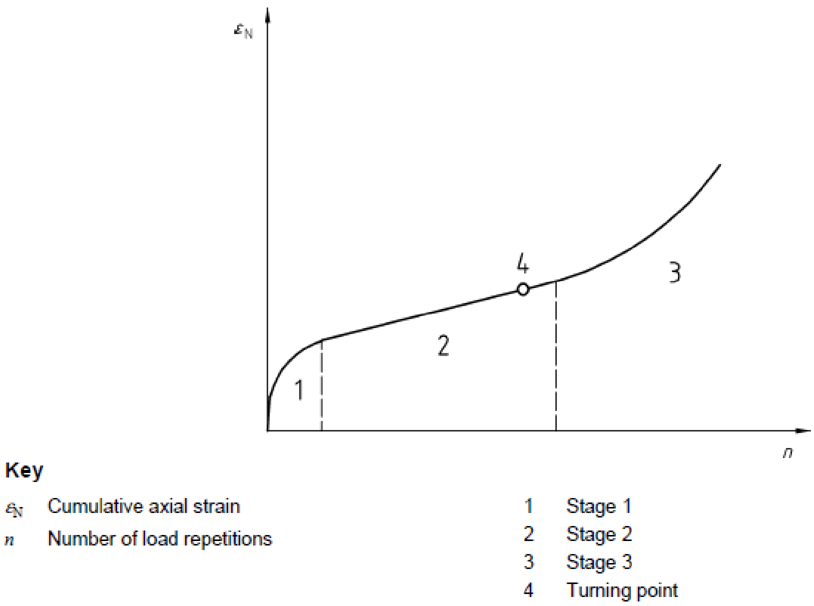


Figure 4-21: Creep curve example (European Committee for Standardization, 2005), permission to reproduce this figure has been granted by BSI Group

As shown in Figure 4-21, there are three stages of creep performance that might be recognised from this test; namely, stage 1, stage 2 and stage 3. Stage 1 represents the initial part of the creep curve in which the slope of the curve decreases with the increasing number of pulses, while stage 2 is the middle part of the creep curve where the curve is almost linear, ending with a turning point. Finally, stage 3 represents the last part where the slope increases with the increasing number of pulses. Depending on the testing conditions and on the mix, one or more stages may be absent.

Normally, creep stiffness modulus is the applied stress test over the accumulative strain at a defined temperature and loading time and its value is indicative of the resistance to permanent deformation. Creep stiffness modulus can be determined from the equation below:

$$E_n = \frac{\sigma}{\varepsilon_n} \times 1000 \qquad 4\text{-}3$$

Where:

E_n is the creep stiffness modulus after n load applications, MPa

σ is the applied stress, kPa

ε_n is the cumulative axial strain of the specimen after n load applications, %

Another indicator can be determined from UCCT test, which is the creep rate of the linear line at stage 2. The smaller the creep rate, the better the mixtures' resistance to permanent deformation. Equation 6-2 gives the formula to determine the creep rate.

$$f_c = \frac{\varepsilon_{n1} - \varepsilon_{n2}}{n_1 - n_2} \qquad 4\text{-}4$$

Where:

f_c is the creep rate, microstrain/loading pulse,

ε_{n1}, ε_{n2} is the cumulative axial strain of the specimen after n_1, n_2 load applications, microstrain, and

n_1, n_2 is the number of repetitive load applications, 3600 and 1200 pulses, respectively.

The details of applied load parameters are listed below:

- A preload of 20 KPa was applied for 2 mins.
- The UCCT is normally terminated at 3600 pulses which requires a total time of 2 hours. The periodic load shall be applied with a loading time for each pulse (1 ± 0.05) s. Every rest period between the pulses shall be (1 ± 0.05) s as well; therefore the total time for the test is about 2 hours.
- A typical value of (100 ± 2) KPa was used for the repeated load.

Poisson's ratio of 0.35 for 40 ºC has been used.

4.4.1.3 Four-point load fatigue test

The behaviour of bituminous mixtures is characterised in this method under four-point bending fatigue loading. The equipment used in this test has symmetrically placed inner and outer clamps and prismatic-shaped beams are used. The rectangular beams were subjected to four-point cyclic bending with free rotation and translation at all load and reaction points. The two inner clamps were loaded in the vertical direction while the end clamps were fixed in the vertical position. According to this configuration, a constant movement will be created with constant strain between the inner loading points.

A Four-Point Bending (4PB) test, Figure 4-22, was used to investigate the fatigue performance of the produced CRA and HRA samples in accordance with BS EN 12697-24 (European Committee for Standardization, 2012c). The applied load and vertical displacement were utilised to calculate the stresses and strains for 400×50×50 mm prismatic-shaped specimens which were manufactured in the laboratory.

All tests were conducted at a temperature of 20 °C and 10 Hz frequency under sinusoidal loading with no rest period and controlled strain criteria of 150 μstrain. Fatigue failure has been arbitrarily defined as the number of cycles, N_f, at which the initial stiffness (stiffness modulus at 100 load repetition) is reduced by 50 %.

Additionally, the relationship between fatigue life and different controlled strain levels, i.e. 100, 125 and 150 μstrain, has been investigated for control CRA and a superior CRA incorporating TBF-2 instead of conventional mineral filler. According to the results representing the life length (N_f) for the selected fatigue criteria, the fatigue line can be drawn by making a linear regression between the natural logarithms of N_f and the natural logarithms of the initial strain amplitude (ε). The following formula demonstrates the shape of the fatigue line:

$$\ln N_f = A_0 + A_1 \ln(\varepsilon) \qquad 4\text{-}5$$

Where:

A_0 represents intercept with y-axis, and

A_1 represents the slope of the fatigue line.

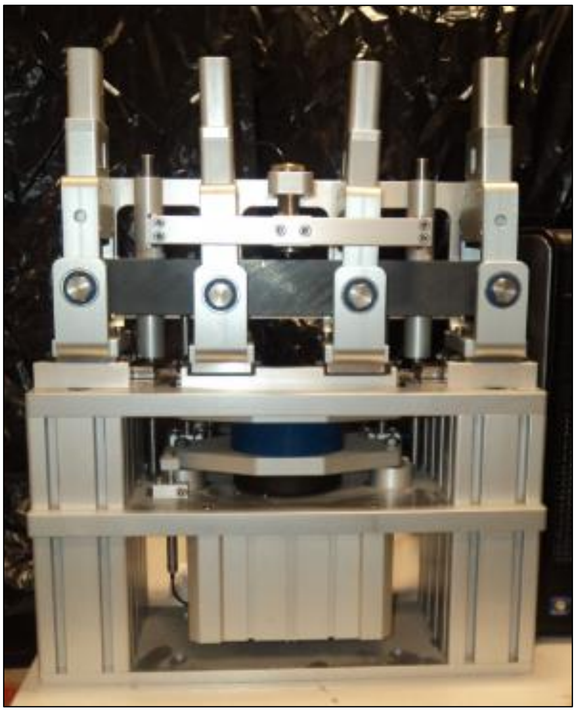


Figure 4-22: Configuration of Four-point load fatigue test (4PB)

4.4.1.4 Semi-Circular Bending (SCB) test

As specified by European Committee for Standardization (2010), the Semi-Circular Bending (SCB) test was used to determine the tensile strength or fracture toughness of a bituminous mixture to investigate the potential for crack propagation. It is worth noting that the crack propagation phase describes the second part of failure mechanism during dynamic loading, while the crack initiation phase represents the first phase as covered in section 4.4.1.3 by conducting the fatigue test.

A half-cylindrical sample with 150 mm diameter and a centre crack of 10 mm depth was loaded in three-point bending in such a way that the centre of the base sample was exposed to a tensile stress. Throughout the test, the rate of deformation was increased at a constant value of 5 mm/min; correspondingly, the load was increased to a maximum value, F_{max}, which is directly related to the sample's fracture toughness. In Figure 4-23 an example of the test frame and specimen configurations are given. The maximum load that the specimen containing a notch (crack) can resist before failure was recorded.

As per the European Committee for Standardization (2010), the maximum stress at failure (σ_{max}) and the fracture toughness (K_{IC}) have been calculated in accordance with equations 4-6 and 4-7, respectively.

$$\sigma_{max} = \frac{4.263 \times F_{max}}{D \times t} \ N/mm^2 \qquad 4\text{-}6$$

Where:

D is the diameter of specimen, mm

t is the thickness of specimen, mm

F_{max} is the maximum force of specimen, Newtons

$$K_{IC} = \sigma_{max}.f\left(\frac{a}{W}\right) \ N/mm^{3/2} \qquad 4\text{-}7$$

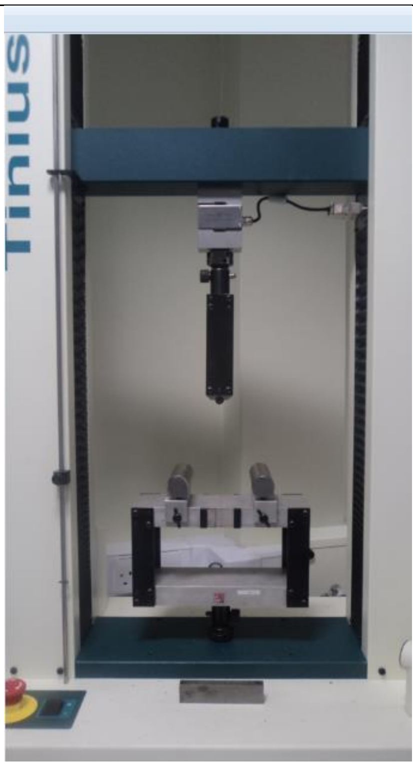
Where:

W is the height of specimen, mm

a is the notch depth of specimen, mm

σ_{max} is the stress at failure of specimen, N/mm^2

f (a/W) is the geometric factor of specimen, for $9 < a < 11$mm and $70 < W < 75$ mm, then *f (a/W)* = 5.956



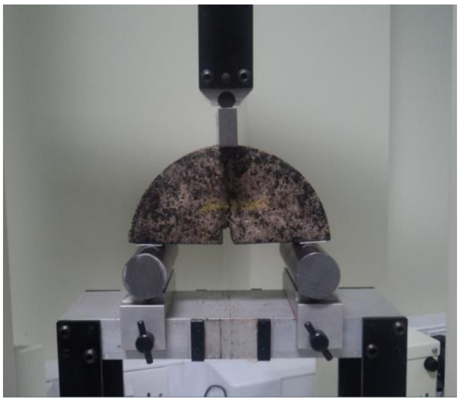


Figure 4-23: SCB test frame and specimen configuration

4.4.2 Durability test

Water damage and failure due to age hardening are the main factors affecting the mechanical properties and durability of asphalts.

4.4.2.1 Water sensitivity test

Stiffness Modulus Ratio (SMR) was used to investigate the water sensitivity of CRA and HRA mixtures according to BS EN 12697-12 (European Committee for Standardization, 2008). Two sets of specimens were prepared and separated. The first set of specimens, dry samples, were left in the mould for 24 hours before being extruded, then stored in the laboratory at 20 °C for 7 days, followed by testing for ITSM at 20 °C. The second set, wet samples, were left in the mould for 24 hours before extrusion, cured at 20 °C for 4 days, and then conditioned as follows (see Figure 4-24):

- Place samples in the vacuum, which is filled with distilled water to a level at least 20 mm above the upper surface of the test samples, at (20 ± 5) °C. Then apply the vacuum to achieve an absolute pressure of (6.7 ± 0.3) kPa within (10 ± 1) minutes;
- Maintain the vacuum for (30 ± 5) min. then let the atmospheric pressure gradually into the vacuum desiccator. Reduce the pressure slowly to avoid damage to the specimens due to expansion;
- Leave the specimens soaking in water for another (30 ± 5) min.; and
- Place the specimens in a water bath at (40 ± 1) °C for a period of 72 hours.

Finally, the ITSM for the second set was conducted at 20 °C and the ratio of the wet to dry ITSM was obtained to determine the SMR.

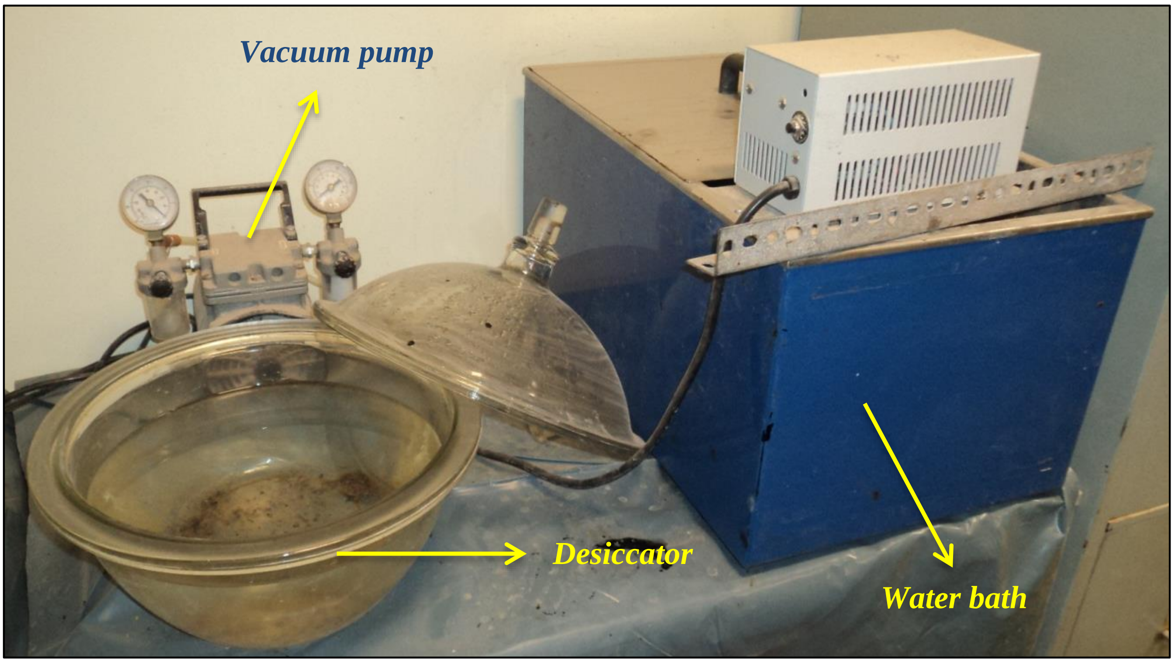


Figure 4-24: Vacuum saturation apparatus and water bath

4.4.2.2 Long tern ageing test

There are two kinds of age hardening investigations in terms of hot mixtures, namely Short Term Oven Ageing (STOA) and Long Term Oven Ageing (LTOA). The former simulates mixture age hardening within the mixture preparation stage, whereas the latter simulates age hardening through road use. The STOA test procedure recommended that loose bituminous mixtures be aged at 135 °C for 4 hours before compaction. While, the LTOA procedure as adopted by the Strategy Highway Research Program (SHRP) A-003A recommended that the compacted samples are cured in an oven at 85 °C for 2 or 5 days to simulate 5 or 10 years' age hardening in the field, respectively (Kliewer *et al.*, 1995).

The short-term aging investigation for CRA is not appropriate as these mixtures are produced at ambient temperature with the aid of substantial amounts of water (water from emulsion plus pre-mixing water) to achieve suitable workability and bitumen coating. Long-term age hardening may take place after full curing condition of the specimens has been reached, but it is not considered as urgent an aspect as long curing time, low early strength and high air voids content. Accordingly, LTOA has been investigated for both produced CRA and conventional HRA mixtures to assess the performance of the optimised mixtures, i.e. mixtures containing WPSA, BBF, TBF-1 and TBF-2 fillers. All the mixtures are conditioned in an oven at 85 °C for 5 days to simulate the age-hardening effects after 10 years, as indicated earlier. Then the specimens are tested in accordance with BS EN 12697-26 (European Committee for Standardization 2012) to indicate ITSM values after ageing at 20 °C.

4.4.3 Volumetric properties' measurement

Air voids content, dry bulk density, voids in mineral aggregate and voids filled with bitumen were determined in accordance with Asphalt Cold Mix Manual-MS 14 adopted by the Asphalt Institute (1989). Accordingly, wet bulk density for CRA mixtures was obtained from equation 4-8 while dry density has been calculated from equation 4-9.

$$wet\ density = \frac{weight\ in\ air}{SSD\ weight - weight\ in\ water} \qquad 4\text{-}8$$

$$dry\ density = \frac{(100+RBC)}{(100+RBC+w)} \times wet\ density \qquad 4\text{-}9$$

Where, SSD weight = saturated surface dry condition which was obtained by towel drying the soaked samples,

RBC = residual bitumen content, and

w = water content after 1 day which was the time of testing.

Then the air voids content was determined from the equation below:

$$Air\ voids, \% = \left(1 - \frac{dry\ density}{SG_{max}}\right) \times 100\ \% \qquad 4\text{-}10$$

Where SG_{max} is maximum specific gravity for the mixture and is calculated from equation 4-11.

$$SG_{max} = \frac{100}{\frac{CA}{SG_{CA}}+\frac{FA}{SG_{FA}}+\frac{F}{SG_F}+\frac{B}{SG_B}} \qquad 4\text{-}11$$

Where, CA, FA, F and B are percentages of coarse aggregate, fine aggregate, filler and bitumen by weight of total mix, respectively.

While the Voids in Mineral Aggregate (VMA) and Voids Filled with Bitumen (VFB) are calculated by using equations 4-12 and 4-13, respectively.

$$VMA = air\ voids + \frac{bitumen\ content}{SG_B} \qquad 4\text{-}12$$

$$VFB = \frac{(VMA - air\ voids)}{VMA} \times 100 \qquad 4\text{-}13$$

A summary of the experimental types that have been followed throughout the present study is shown previously in Table 4-5, while Figure 4-25 shows a schematic diagram of the proposed research methodology.

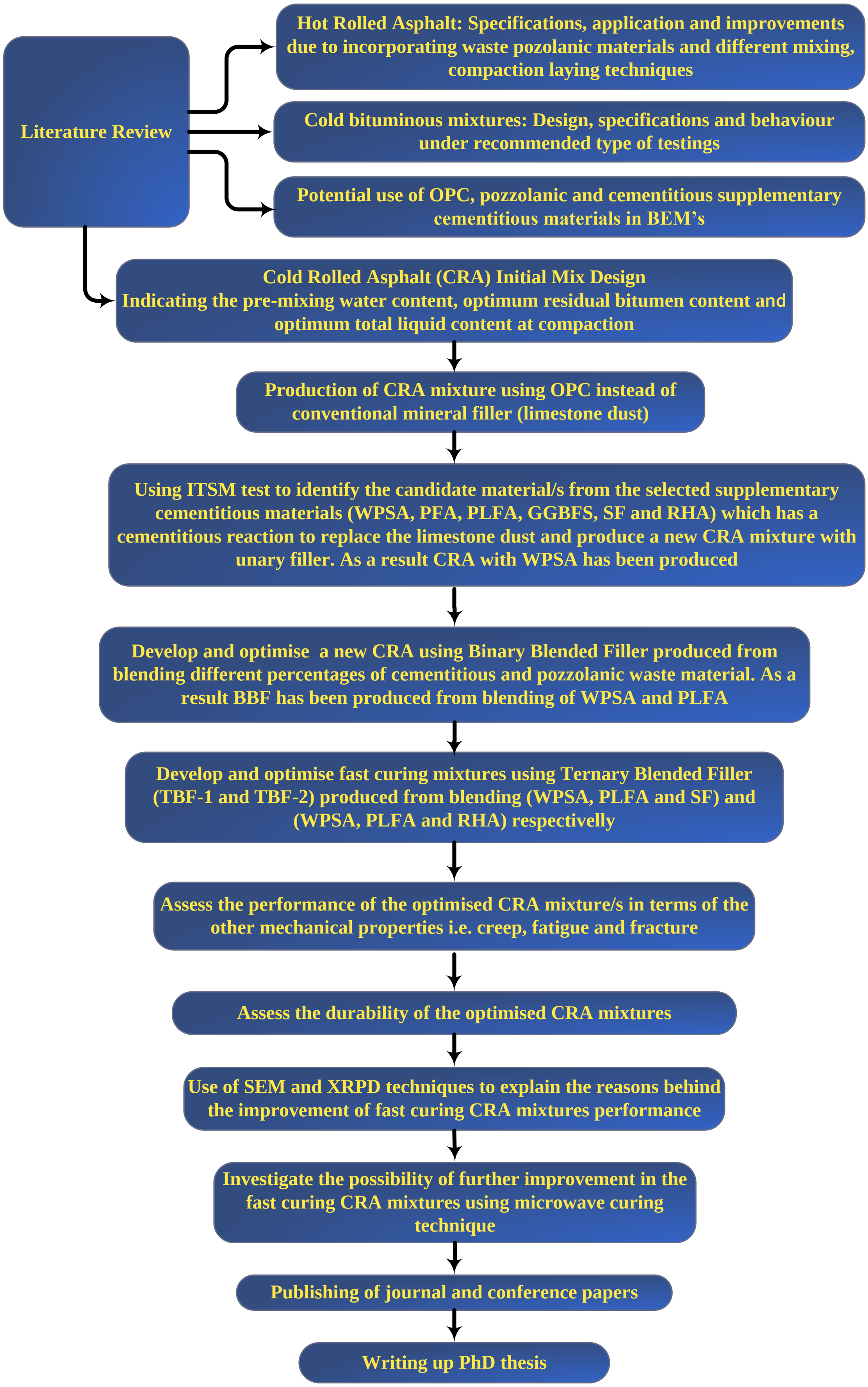


Figure 4-25: Research methodology flowchart

4.5 Summary

This chapter covered the characterisation of candidate SCMs and the research methodology which is adopted to investigate the properties of the produced mixtures.

Cold Rolled Asphalts (CRAs) are recommended to produce new BEMs suitable to use as surface course materials in heavily trafficked roads and airfields by 1) incorporating different SCMs individually or collectively instead of conventional mineral filler; and 2) applying different curing techniques. Their gradation is similar to the conventional HRA mixture which is conventionally used as surface course mixtures in heavily trafficked UK roads.

The physio-chemical characteristics of various candidate waste and by-product materials, i.e. WPSA, PLFA, SF and RHA, were revealed and a comparative study between the materials and conventional mineral filler, i.e. limestone dust as well as OPC, was carried out. The strong hygroscopic nature and high lime concentration of WPSA introduce it as an attractive choice to replace the conventional mineral filler. Also, the existence of strong crystal peaks of lime and gelenite for WPSA was expected to influence the hydraulic property of bituminous mortar. On the other hand, a potential activation of the hydration process of WPSA is expected when incorporating PLFA due to the presence of arcanite as a source of alkali sulphate activator. Furthermore, the utilisation of highly reactive SF for activating the hydraulic fillers and creation of a dense microstructure is expected. Finally, the high pozzolanic characterisation and economic aspects of RHA make it a favourite candidate to replace SF to activate the hydration process of cementitious fillers and thus reduce the free water responsible for high voids in conventional CMA.

Essentially, most of the types of testing which are routinely utilised to assess the conventional hot mixtures can be implemented for BEMs. The stiffness modulus test which is recommended by industrial partners as a major fundamental property to optimise the novel CRA mixtures is detailed in the next chapter.

Chapter Five

High Strength and Fast Curing CRA Mixtures

As reported by the preceding investigations and applications on BEMs, the long curing time required to achieve the maximum performance and the poor early life strength are the main concerns about these mixtures. Chevron Research Company stated that the full curing of those mixtures may occur between 2 months and 2 years depending on their ingredients and weather conditions (Leech, 1994). Thanaya *et al.* (2006) indicated that the stiffness modulus of BEMs containing PFA instead of conventional mineral filler is very comparable to hot mixtures after full curing conditions (more than 2 years).

This chapter presents the process technology for the production of CRA mixtures containing different types of filler: the first mixture represents a control CRA containing conventional limestone dust which is the traditional mineral filler for HRA mixtures and complies with the BS EN 13108: Part 4 standards requirements (European Committee for Standardization, 2006). While the second mixture represents a CRA containing OPC instead of mineral filler to investigate the ability of producing a high-performance gap graded CRA mixture containing OPC. Due to the economic potential as well as the environmental benefit in terms of carbon emissions and hazards to health, energy savings, and recycling of waste materials, supplementary cementitious materials have been used individually or collectively instead of OPC to produce sustainable CRA mixtures. Therefore, the third mixture was CRA with WPSA (which is a SCM with high lime and gehlenite content) as a replacement for the conventional mineral filler.

To improve the activity of the secondary binder (i.e. binder generated from the hydration process between filler and the trapped water incorporated in CRA mixtures), an optimisation has been made to produce a Binary Blended Filler (BBF) which was generated from blending WPSA with PLFA, which is another SCM with high alkali content. The last two steps were focused on producing fast curing CRA mixtures with Ternary Blended Filler (TBF) which were generated from blended WPSA, PLFA and high silica SCM. The first TBF-1 was produced from blending WPSA and PLFA with SF (which is a high silica content by-product material). Finally, as SF is a cost-minus material, the idea of replacing this material with another, cost-plus, material (with similar activity) has been proven successful by using RHA which is rich in silica content waste material to produce the second TBF i.e. TBF-2.

Stiffness modulus of the bituminous mixtures is one of the main fundamental properties of these mixtures; therefore it has been used as a primary property to indicate the ultimate or optimum performance of the candidate CRA mixtures.

5.1 Stiffness modulus

The behaviour of bituminous mixtures is particularly elastic, although these mixtures are viscoelastic materials (Nunn, 1997). The elastic stiffness modulus (ESM) of bituminous mixtures is a respectable indicator of its ability for load spreading. Furthermore, high ESM controls the level of tensile strain created in the underlying courses. Consequently, a mixture with a high elastic stiffness modulus will have a good load-spreading ability which decreases the deflection caused by the passing traffic loads and the tensile strains in the underlying layers.

In this study, stiffness modulus has been indicated by applying indirect tension to cylindrical specimen as per BS EN 12697-26 and the resultant modulus was named Indirect Tensile Stiffness Modulus (ITSM) as described in section 4.4.1.1.

5.1.1 CRA specimens' curing

The normal curing process adopted in this investigation consisted of two stages. The first stage was to maintain the sample in the mould for 24 hours at lab temperature, i.e. 20 ºC. After that time the sample was extruded and kept at lab temperature until testing took place at different ages, i.e. 1/6, 1, 3, 7, 14, 28, 90 and 180 days to determine the ITSM. Most of the previous investigations such as Thanaya (2003) and Ibrahim (1998) were focused on curing of BEMs at high temperature such as 40 ºC due to the weak strength of these mixtures. But in this study, use of 20 ºC as the normal curing temperature was adopted to prove the possibility of producing high performance cold mixtures at a normal lab temperature.

5.2 Production of CRA mixtures

An attractive laboratory optimisation process based on the ITSM test results using SCMs individually or collectively instead of the conventional mineral filler has been conducted and is presented in the subsections below.

5.2.1 Control CRA mixtures

A control CRA mix is prepared from control blended composition, i.e. with limestone dust as conventional mineral filler (CRA–LD). These mixtures were prepared as detailed in section 4.2.3 with 3 % pre-mixing water content and 12.5 % bitumen emulsion content and cured as illustrated in section 5.1.1. As CRA with LD samples were not suitable to withstand the test loads before 3 days, ITSM tests were conducted after 3, 7, 28, 90 and 180 days. Figure 5-1 shows the progress of ITSM results for control CRA (LD) mixtures within these curing duration times. It is clearly shown that there is an increment in the ITSM results for LD mixtures with curing time especially within the first 28 days, but still their results are relatively low in comparison with the target ITSM for 100/150 HRA (approximately 2000MPa). These higher values are due to the evaporation of the trapped water incorporated in these mixtures with time. On the other hand, there is no noticeable change in 100/150 and 40/60 HRA results with curing time.

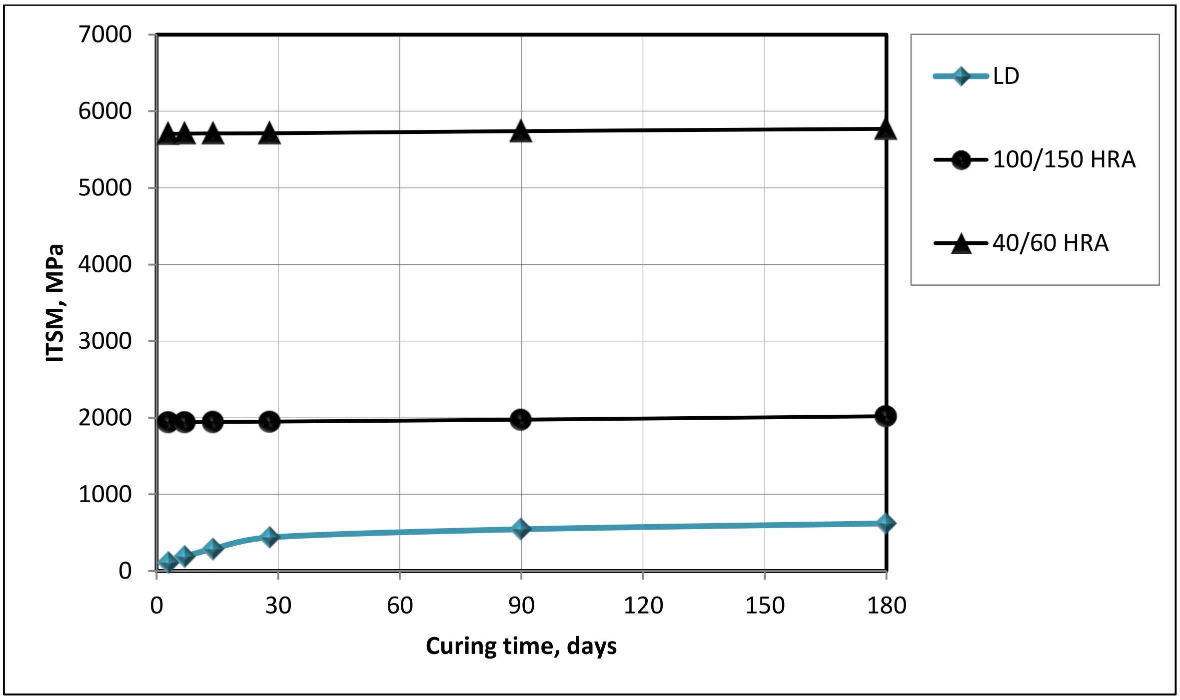


Figure 5-1: ITSM results for control CRA (LD)

5.2.2 CRA containing OPC

As described in chapter 3, OPC has been assumed in a number of instances over the years to improve the mechanical properties of a range of BEMs. After detailed laboratory and field studies, Needhem (1996) stated that OPC can have a beneficial effect on BEMs. In the work described in this subsection, the aim was to produce a new CRA containing OPC instead of the conventional mineral filler. Therefore, conventional mineral filler has been replaced with different amounts of OPC, i.e. 0, 1.5, 3, 4.5 and 6 % by total mass of aggregate, as shown in Figures 5-2 and 5-3.

The first step of this stage was to determine the influence of the replacement of the mineral filler with different proportions of OPC up to 6 % of the total mass of aggregate on ITSM results after 3 days, Figure 5-2. From this figure, it is clearly shown that there is a significant increase in the stiffness modulus with the increase of OPC content. Therefore, full replacement of conventional mineral filler with OPC is recommended, i.e. 6 %, to achieve a fast curing CRA. CRA containing 6 % OPC instead of mineral filler is termed as OPC mixture within this investigation.

Consecutively, there is a considerable improvement in ITSM results for OPC mixtures with the increase of curing time, as shown in Figure 5-3. Generally, OPC mixtures had ITSM higher than the control CRA (LD) mixtures for the whole curing time. Also, the rate of increment of stiffness modulus values for these mixtures with time was very high (in the early days), i.e. up to 7 days. After that time the rate of increment was comparatively low. Also, it can be concluded that the target ITSM (2000 MPa) can be achieved after less than 1 day which leads to the ability to open a road constructed with these mixtures after several hours of curing, while the conventional BEMs only gain the required strength after 2–24 months, according to Leech (1994). The main reason behind this performance can be due to the generation of an additional binder from the hydration process of OPC with trapped water incorporated in the CRA mixtures. Also, this process will activate the trapped water loss which is incorporated in these mixtures and affects the improvement of their strength.

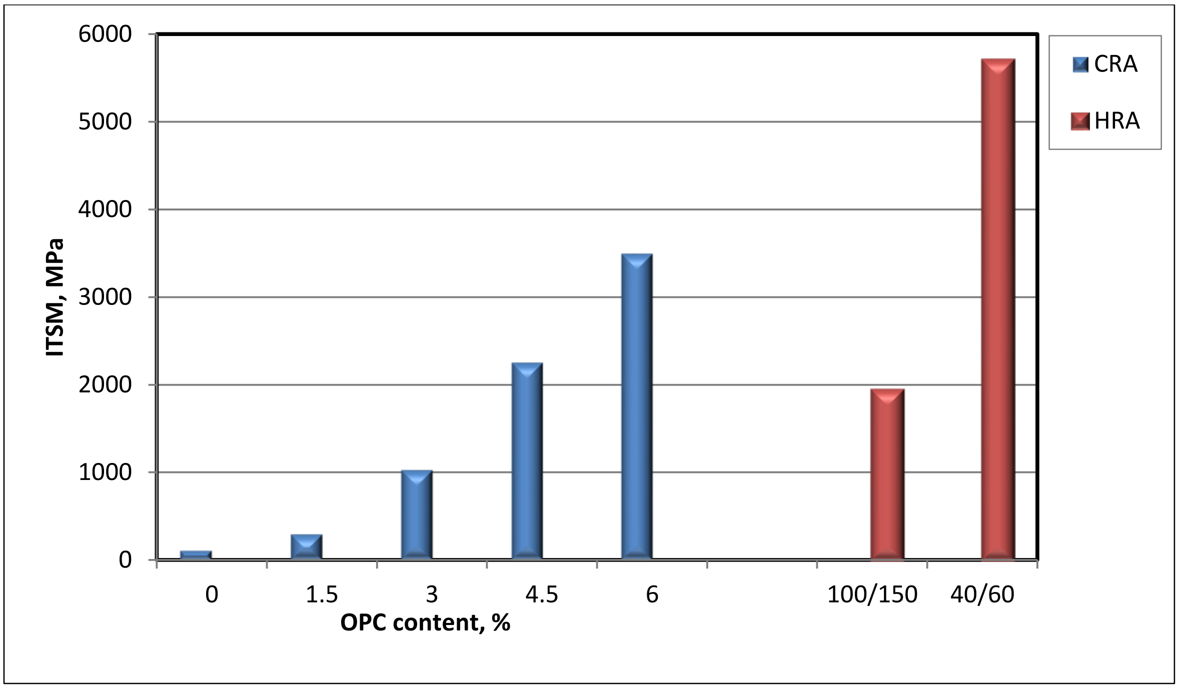


Figure 5-2: Effect of OPC percentage on ITSM results after 3 days for CRA mixtures

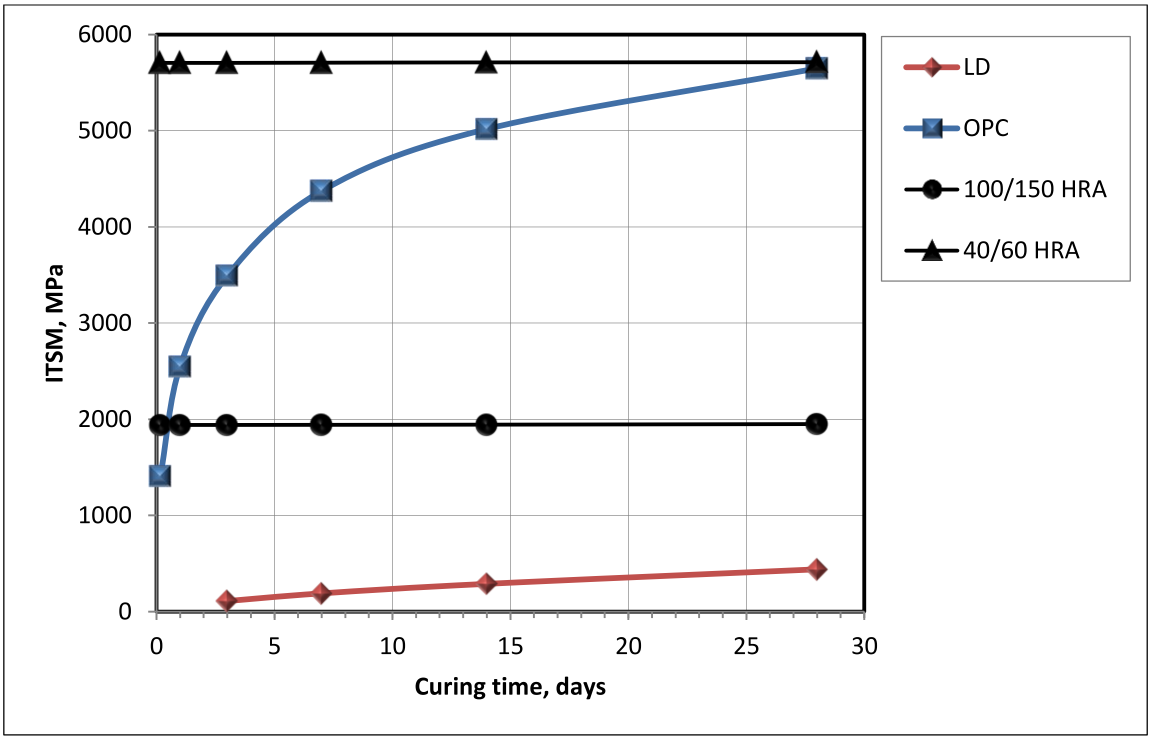


Figure 5-3: Effect of curing time on ITSM results for OPC mixtures

5.2.3 CRA containing WPSA

Due to the high price of OPC, the main aim of this study was to produce fast curing CRA mixtures by using SCMs individually or/and collectively to replace the conventional mineral filler. Accordingly, wide range of physical and chemical tests have been done for the selected candidate wastes and by-product materials (PFA, WPSA, GGBS, PLFA, APC, SF, and RHA) to explore cementitious and pozzolanic characteristics of these materials. From these candidate materials listed above, WPSA with high contents of gelenite-G and lime-L crystalline phases (see Figure 4-9) is recommended to work as a cementitious material and to replace the conventional mineral filler.

WPSA was added at 0, 1.5, 3, 4.5, and 6 % by mass of aggregate, i.e. replacement percentage equivalent to 0, 25, 50, 75, and 100 %, to assess the effect of replacement of conventional mineral filler with WPSA on ITSM results and the results are illustrated in Figures 5-4 and 5-5.

According to the results shown in Figures 5-4 and 5-5, several points can be concluded: 1) the ITSM results of the CRA mixtures increased considerably when the percentage of WPSA increased and reached the ultimate strength when all the conventional mineral filler is replaced with WPSA, i.e. 6 % by mass of aggregate, Figure 5-4; 2) CRA mixtures with 6 % WPSA can provide a stiffness modulus of approximately 30 times that of LD mixtures at a curing time of 3 days; 3) CRA containing 6 % WPSA reveals an ultimate ITSM result and is termed as WPSA mixtures within this thesis; 4) the ITSM results increased dramatically for WPSA mixtures with curing time especially before 14 days, whereas HRA does not show noticeable variations in ITSM with time; 5) the target stiffness modulus can be achieved easily after 1 day within the normal curing process (20 ºC); and 6) the strength-gaining behaviour of WPSA mixtures is similar to that of OPC mixtures and this confirms the cementitious action of WPSA filler within cold BEMs.

This enhancement in ITSM results is due to firstly, the fact that another binder being generated from the hydration process of the WPSA powder in addition to the bitumen–filler residue binder and, secondly, loss of the trapped free water which is absorbed by WPSA (has extremely hygroscopic nature) and evaporated during curing time.

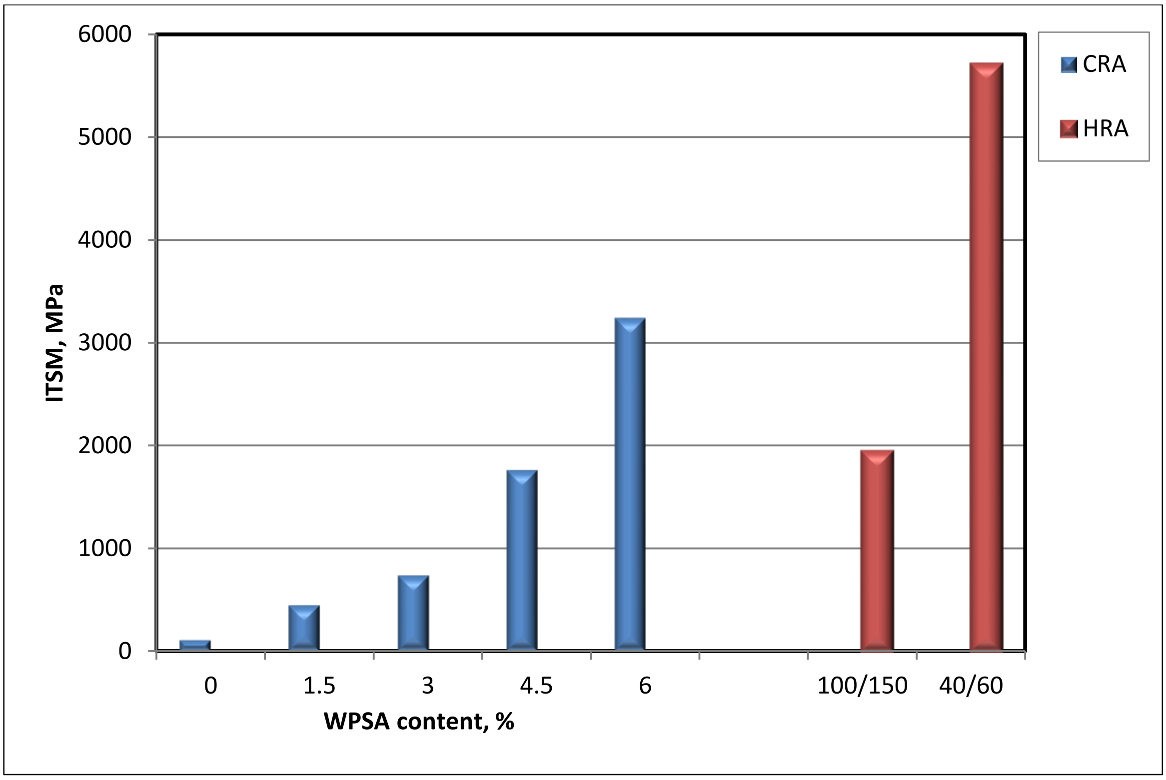


Figure 5-4: Effect of WPSA percentage on ITSM results after 3 days for CRA mixtures

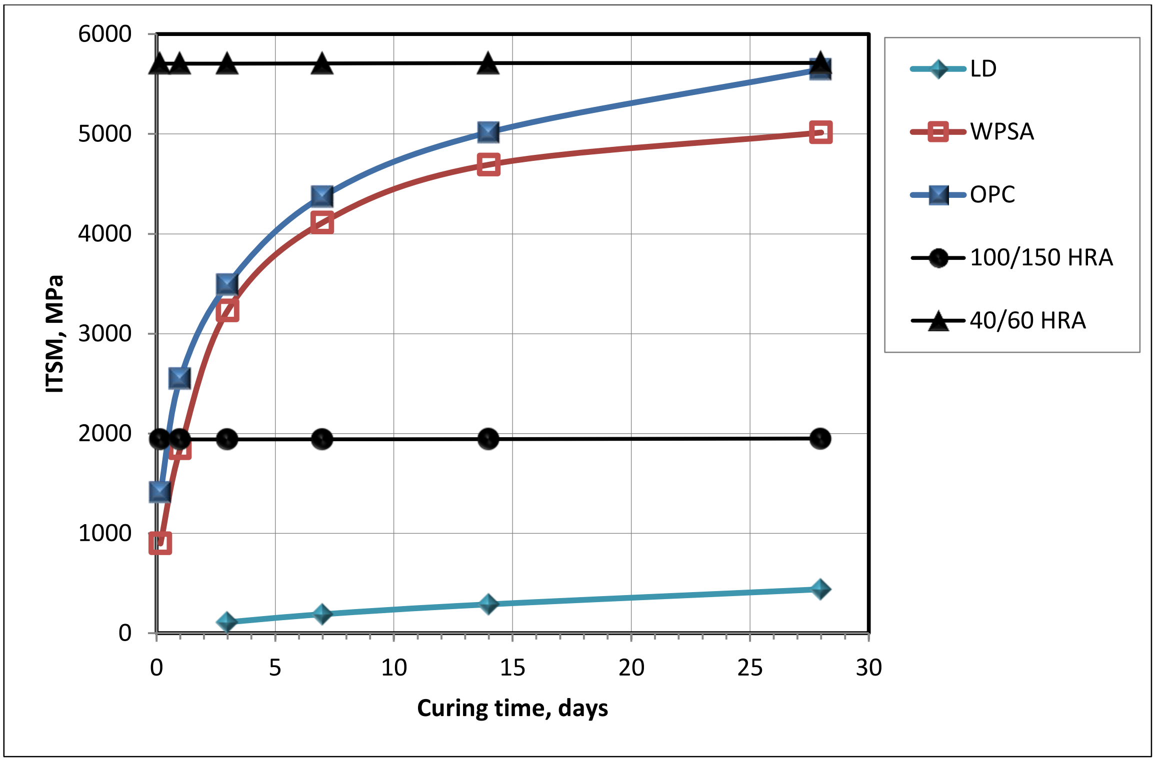


Figure 5-5: Effect of curing time on ITSM results for WPSA mixtures

5.2.4 CRA containing BBF

At this stage, the idea of activating the hydration process for the incorporated filler substances is started to produce a new filler material with high hydration activity, especially in the early curing time. This activation process is based on blending two or three types of the selected SCMs depending on their physical and chemical properties.

Accordingly, a new fast curing gap graded CRA has been produced by replacing the conventional mineral filler with a Binary Blended Filler (BBF). BBF is produced from blending different compositions of WPSA (cementitious waste material) and PLFA which is rich in arcanite-K component (Figure 4-11). It is expected that the existence of arcanite-K in PLFA will provide an ambient environment to activate the hydration process of the WPSA, which is rich in cementitious components such as lime and gelenite (Rodrigues *et al.*, 1999).

CRA samples were prepared with different percentages of WPSA and PLFA in place of mineral filler comprising 6 % of the total aggregate mass and then tested after 3 days to indicate the ITSM results, Figure 5-6. From this figure, it is clearly shown that there is a significant increase in the stiffness modulus samples containing up to 50 % PLFA. For samples with >50 % PLFA, the stiffness modulus values decreased sharply. In addition, this performance reveals the cementitious action of WPSA and the pozzolanic role of PLFA, as reported by Rodrigues *et al.* (1999). The binary blend of 4.5 % of WPSA with 1.5 % PLFA was found to show the highest stiffness modulus after 3 days' curing time and this is recommended as a Binary Blended Filler (BBF). CRA mixture incorporating 6 % BBF represents another optimum CRA and is termed as BBF within this study.

The ITSM for the prepared BBF mixtures at different curing time, i.e. 1/6, 1, 3, 7, 14 and 28 days, are shown in Figure 5-7. Generally, these mixtures had ITSM results higher than the control CRA, OPC, and WPSA mixtures for the whole curing time. Also, the rate of increment of stiffness modulus values for these mixtures with time was very high in the early days, i.e. up to 3 days, after that time the rate of increment was lower. Interestingly, the ITSM of BBF mixtures after just 4 h (2040 MPa) is more than the ITSM of the target stiffness and achieves the British and European requirements in terms of ITSM. From the last point, the produced environmental and sustainable BBF mixtures can be opened to traffic very quickly, while the traditional cold mixtures only gain the required strength after 2–24 months, as reported before; this would be an important advantage. One more interesting point that can be concluded is that the stiffness modulus for the BBF mixtures after just 4 h is more than those for OPC mixtures by about 40 %, which represents a significant economic advantage because the selected materials are classified as waste materials according to the UK Environment Agency.

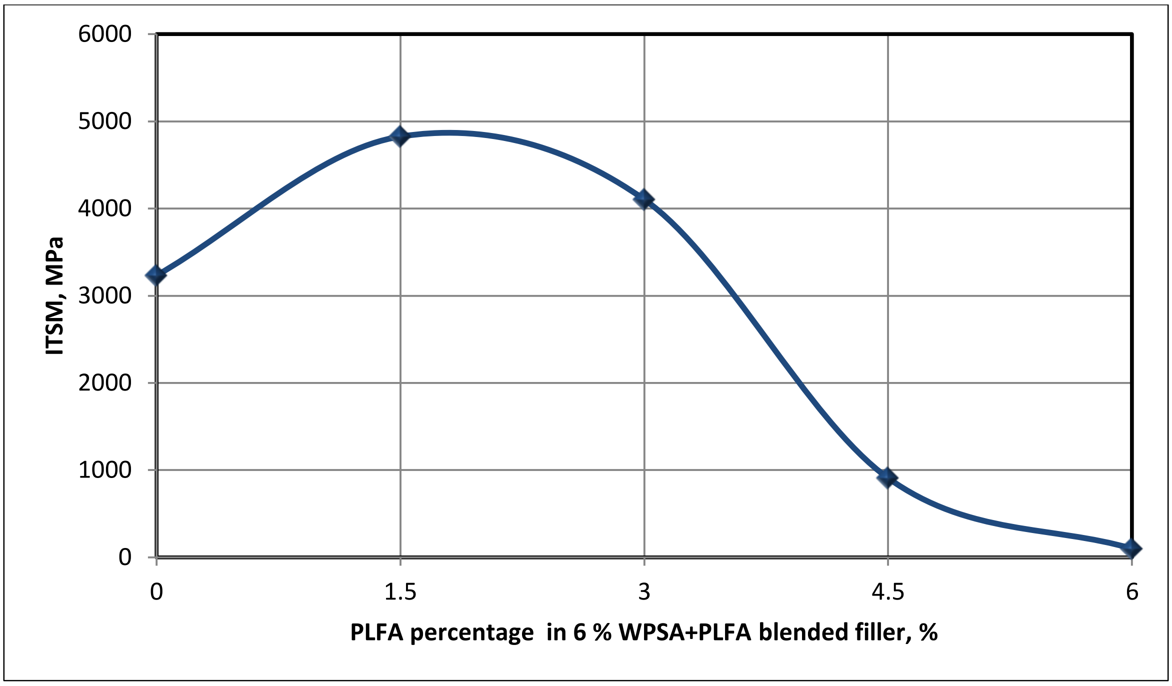


Figure 5-6: Effect of replacement of WPSA with PLFA on stiffness modulus of CRA after 3 days

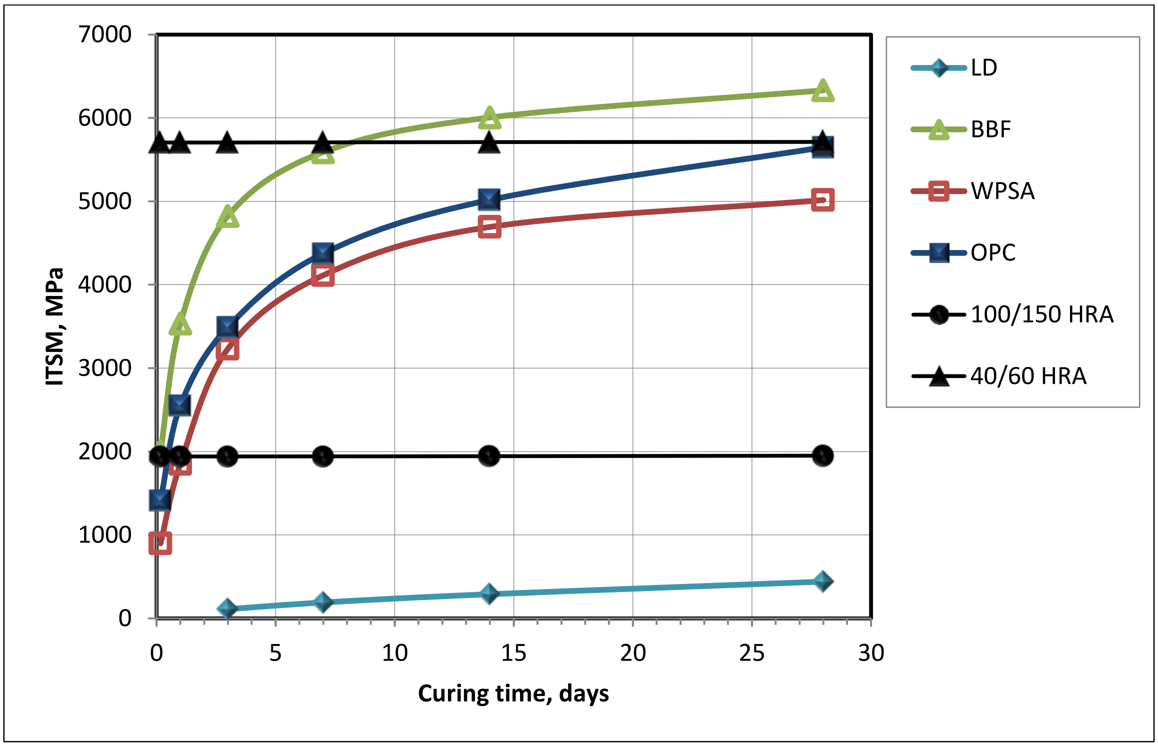


Figure 5-7: Effect of curing time on stiffness modulus of BBF mixtures

5.2.5 CRA containing TBF-1

The resultant new cementitious filler from the blending of two types of supplementary cementitious materials, i.e. WPSA and PLFA, in the previous subsection leads to further activate the hydration process by using another high silica waste or by-product material. It is expected to convert soluble calcium hydroxide (C–H), generated from hydration reaction of BBF filler material, into dense calcium silicate hydrate (C–S–H) through pozzolanic reaction when adding a high silica material (Durand *et al.*, 1990, Lothenbach *et al.*, 2011, Sadique *et al.*, 2012).

SF is a by-product material containing amorphous silica particles with a high surface area. A new Ternary Blended Filler (TBF-1) was generated from incorporation of SF within the BBF system produced from the stage outlined in the previous subsection. CRA specimens were prepared by incorporating different amounts of BBF and SF to specify the optimum composition which gives the maximum ITSM after 3 days, Figure 5-8. This figure indicates the positive role of SF when incorporated in BBF filler materials, as there is a considerable increase in the ITSM results when 1 % of the BBF is replaced with SF. At this point, i.e. 5 % BBF plus 1 % SF, maximum ITSM was achieved with about a 15 % increase in comparison with BBF mixtures; therefore a new TBF has been generated. The optimum CRA mixture produced here is named as TBF-1 mixture within this investigation.

Figure 5-9 shows the ITSM results for TBF-1 mixtures at 1/6, 1, 3, 7, 14, and 28 curing time. These results reveal the positive effect of adding SF as there is a further increase at all testing times in comparison with the whole optimised mixtures. The role of SF in TBF-1 was to create a dense impermeable secondary binder structure; plus, a successful hydration reaction could be expected with mechanically activated filler in a high pH CRA mixture environment. Therefore, the C–H generated from WPSA+PLFA converted into C-S-H, which is the main strength-generating item.

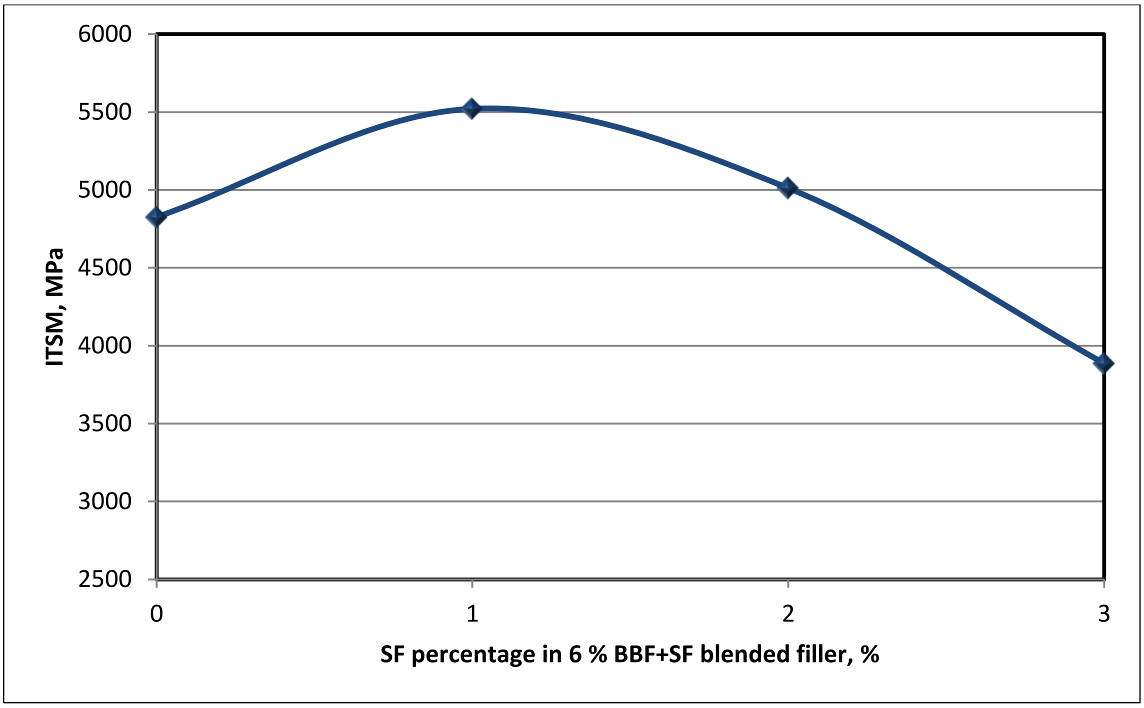


Figure 5-8: Effect of replacement of BBF with SF on ITSM of CRA after 3 days

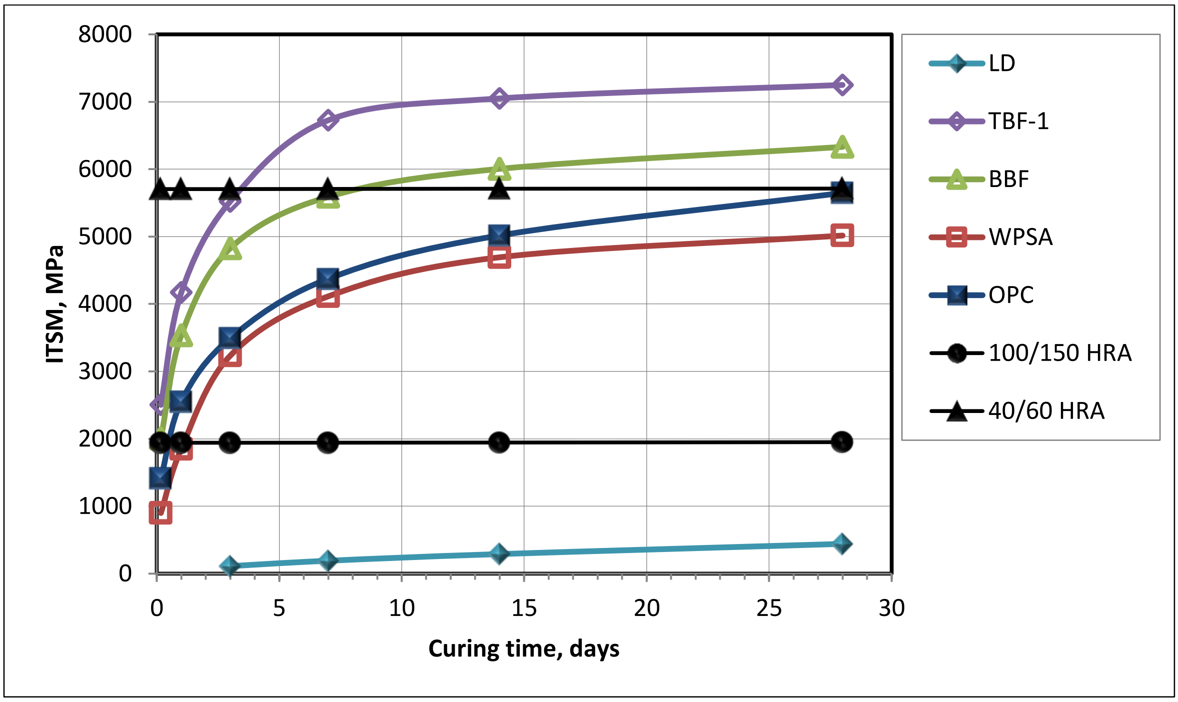


Figure 5-9: Effect of curing time on stiffness modulus of TBF-1 mixtures

5.2.6 CRA containing TBF-2

At the final optimisation step, RHA has been used to produce CRA mixtures with ternary blended filler instead of SF. The positive effect of using high silica RHA as a supplementary cementitious material with binary or ternary blended systems, as reported by many previous studies such as Cordiero *et al.* (2012) and Naji Givi *et al.* (2010) as well as the high price of SF, encouraged the use of RHA instead of SF.

Non-spherical-shaped particles with a high content of silica (89 %) RHA were used to produce another Ternary Blended Filler (TBF-2). Different percentages of BBF and RHA have been added to replace the 6 % conventional mineral filler to indicate the optimum composition considering the maximum ITSM result after 3 days and the results are illustrated in Figure 5-10. There is a significant increase in ITSM results when BBF was replaced with RHA until reaching the 1.5 %, then the values decreased. Accordingly, a superior TBF has been generated from 4.5 % BBF plus 1.5 % RHA to produce a fast curing CRA, termed within this study as TBF-2. The ITSM value is increased by more than 30 % in comparison with BBF mixtures after 3 days’ curing time.

To investigate the behaviour of the produced TBF-2 mixtures within different curing times, ITSM test was conducted at different ages, i.e. 1/6, 1, 3, 7, and 28 days, to show their performance, Figure 5-11. Several interesting points can be reported from the stiffness modulus results; these are: 1) TBF-2 mixtures had a significant early strength and their strength was higher than all the previous mixtures, i.e. LD, OPC, WPSA, BBF, and TBF-1, for the whole curing time, 2) it is clearly shown that the rate of stiffness modulus increase was extensively high until 7 days then a remarkable decrease in this rate can be recognised after the said age , 3) the stiffness modulus of TBF-2 mixtures after just 4 hours is 2750MPa, which is much higher than the target stiffness modulus (2000MPa) as well as all the previous mixtures and achieves the BS and European requirements, 4) more interestingly, TBF-2 stiffness modulus after 4 h is about double that for OPC mixtures, and 5) a new binder totally from waste and by-product materials (cost-plus materials), i.e. WPSA, PLFA and RHA, has been produced by this study as a new cementitious material which is expected to be used in all the application fields of OPC and introduces significant economic, environmental and sustainable advantages.

As a result, the produced environmentally, sustainable and fast curing TBF-2 mixtures can be opened to traffic very quickly after preparation and compaction and these mixtures are expected to perform without any possible problems. This would be a remarkable benefit in comparison with the 2–24 months' curing time which is required for the conventional BEMs.

The further improvement in ITSM results for TBF-2 mixtures in comparison with the previous optimised mixtures can be due to the generation of dense C–S–H through pozzolanic reaction when adding high silica RHA and the high ability of the new filler material to absorb the trapped water incorporated in CRA mixtures without affecting the mixing workability.

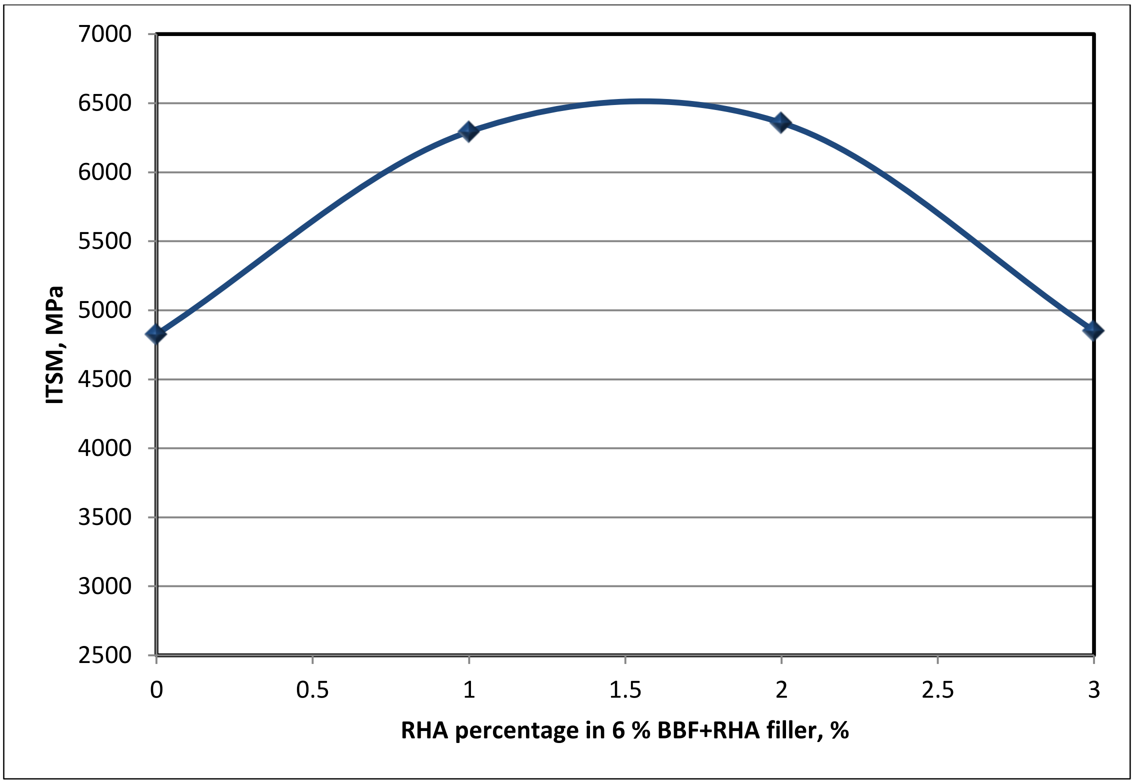


Figure 5-10: Effect of replacement of BBF with RHA on ITSM of CRA after 3 days

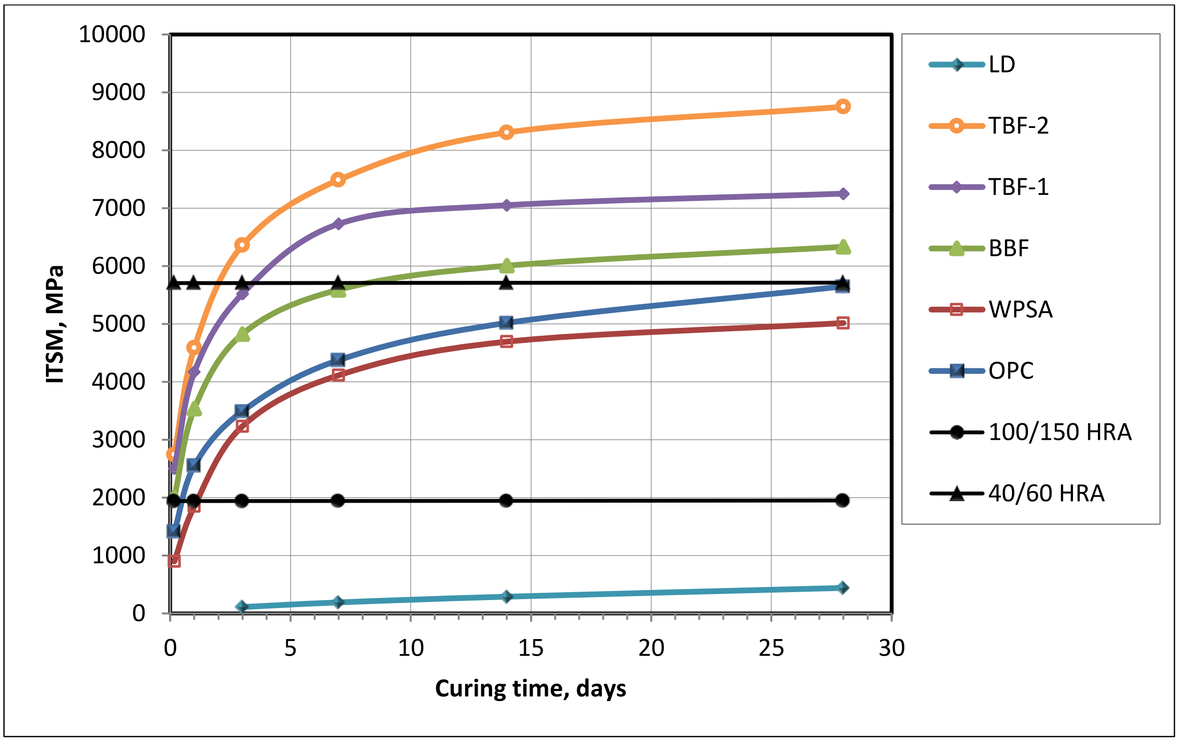


Figure 5-11: Effect of curing time on stiffness modulus of TBF-2 mixtures

5.3 Performance of the optimum CRA mixtures cured at high temperature

Cold BEMs are very sensitive to conditioning temperature and curing time. Therefore, these mixtures are more suitable for regions where the ability to evaporate the trapped water is fast. Jenkins (2000) conducted a detailed study on the performance of cold mixtures with different curing criteria. He concluded that there is a considerable improvement in terms of strength enhancement when these mixtures are cured at higher temperature. Also, he stated that 1 day @ 20 ºC plus 1 day @ 40 ºC curing criteria represents 7–14 days in the field. In this study, the said curing criteria were adopted to identify the evolution of the stiffness modulus with higher temperature of the produced CRA mixtures. Also, this temperature is a normal temperature in different regions around the world and it is the common summer temperature of the author's country of origin.

All the optimised CRA mixtures, i.e. CRA with LD, OPC, WPSA, BBF, TBF-1 and TBF-2, were prepared according to this curing method and tested at 20 ºC to indicate the stiffness modulus value with different curing times (1, 3, 7, 14, and 28 days) and the results are shown in Figures 5-12 and 5-13. A significant enhancement in ITSM results was recognised at curing with high temperature for all CRA mixtures in comparison with normal curing results. For example, LD mixtures cannot conduct at normal temperature because it was not suitable to withstand applied load but with 40 ºC curing for 1 day, a noticeable result can be achieved. Also, CRA with OPC, WPSA, BBF, TBF-1 and TBF-2 results after 1 day (Figure 5-12) were increased by about 26, 81, 31, 49 and 21 %, respectively when compared with normal cured mixtures. This attractive enhancement might encourage use of CRA mixtures due to high early strength especially when incorporated in hot regions and/or hot seasons. From Figure 5-13 it looks very easy to achieve the required stiffness modulus, i.e. 2000MPa, for all modified CRA mixtures just after 1 day of 40 ºC curing method.

Kobayakawa *et al.* (1998) and Haneharaa *et al.* (2001) studied the effect of curing on the cementitious and pozzolanic fly ashes' reaction at high temperature. They concluded that the hydration process will accelerate when these materials are cured at high temperature. Therefore, the activity of the secondary binder, generated from the hydration process between the different types of cementitious fillers used to produce OPC, WPSA, BBF, TBF-1 and TBF-2 CRA mixtures, was accelerated within the adopted curing method.

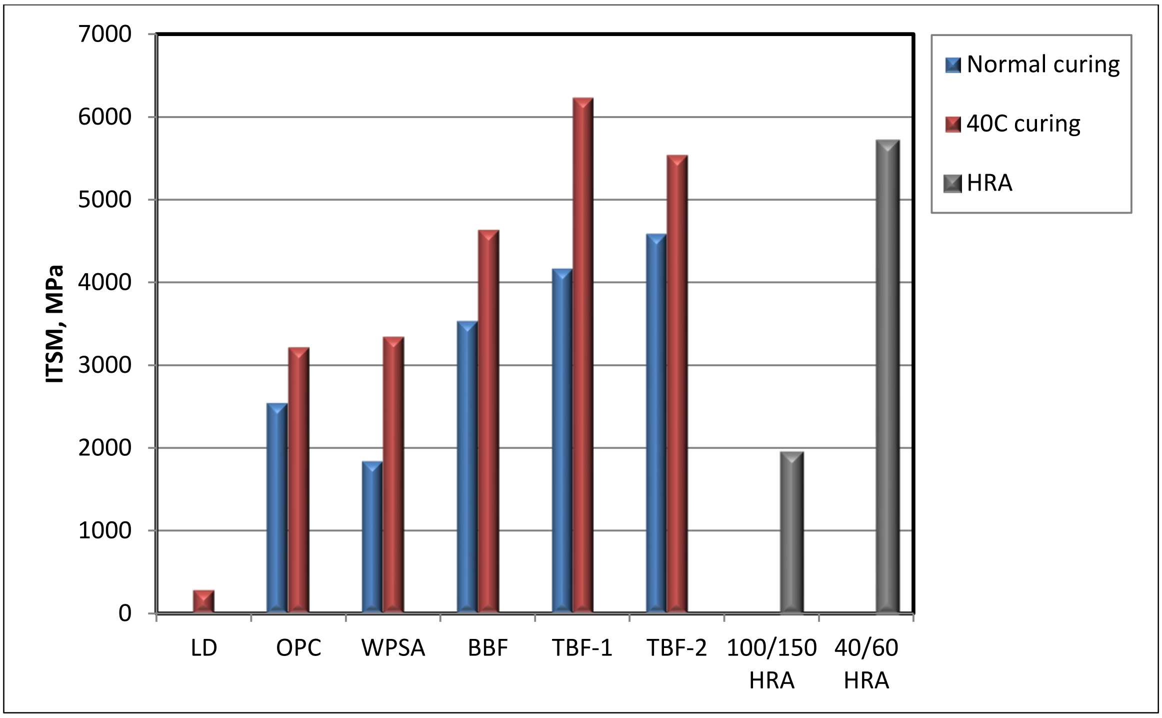


Figure 5-12: ITSM results of 20 ºC and 40 ºC curing temperature after 1 day

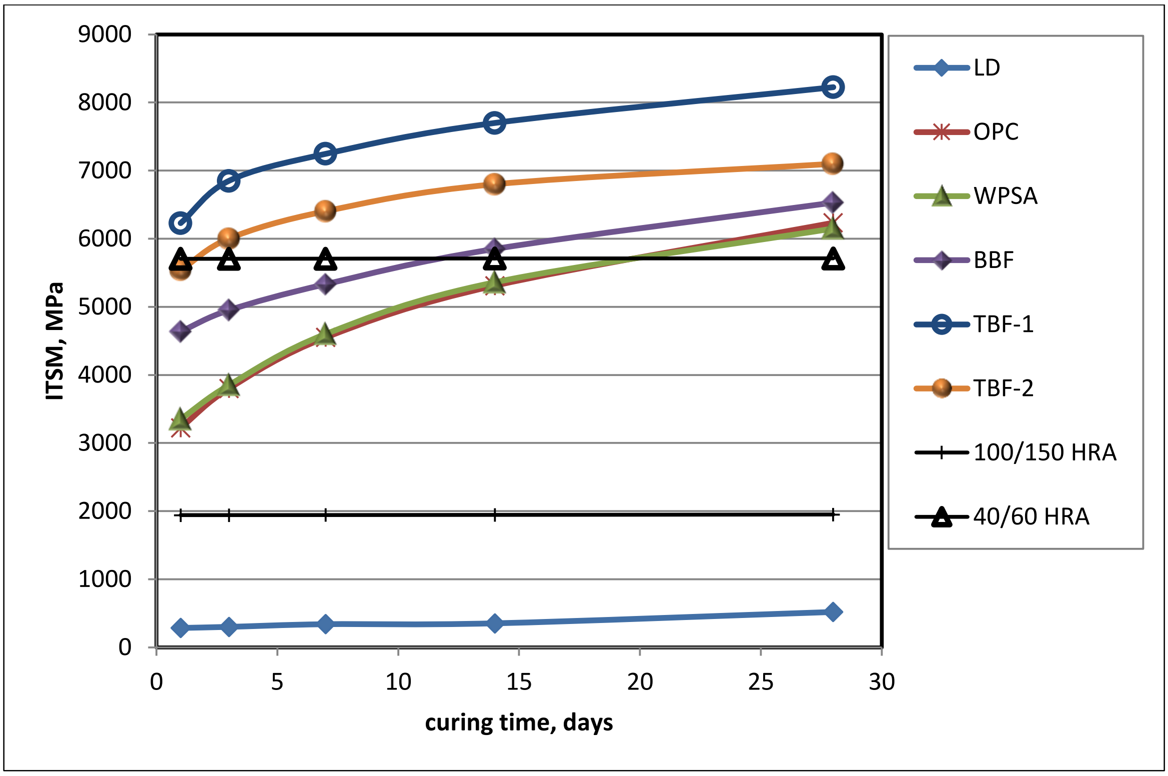


Figure 5-13: Effect of curing time on ITSM results for 1 day @ 40 ºC curing method

5.4 Temperature susceptibility of the optimum CRA mixtures

To indicate the temperature susceptibility of all the optimised CRA mixtures as well as HRAs, ITSM test was conducted at 28 days with different testing temperatures, namely 5, 20 and 40 °C and the results are illustrated in Figure 5-14. The slope of the curve in a semi-logarithmic plane can represent temperature sensitivity, as mixtures with a higher rate of change have more temperature sensitivity.

As shown in this figure, it was not possible to conduct the test for control CRA mixtures at 40 ºC due to the weak strength of those mixtures at high temperature. Also, the results confirmed the thermo-dependence of CRA mixtures, showing a decrease in ITSM with the increase in temperature. However, CRA mixtures with OPC, WPSA, BBF, TBF-1 and TBF-2 showed a significant lower thermal sensitivity than conventional HRA. The presence of strong bonds due to the cementitious action of modified CRA mixtures' filler and the residual internal friction between aggregate particles guarantees high stiffness properties at high temperatures but these mixtures preserve the temperature-dependent behaviour.

Therefore, it is expected that modified CRA mixtures suffer less distortion and rutting as compared to 100/150 and 40/60 HRA during hot seasons in addition to the possibility of lower mixtures' fracture at low temperature.

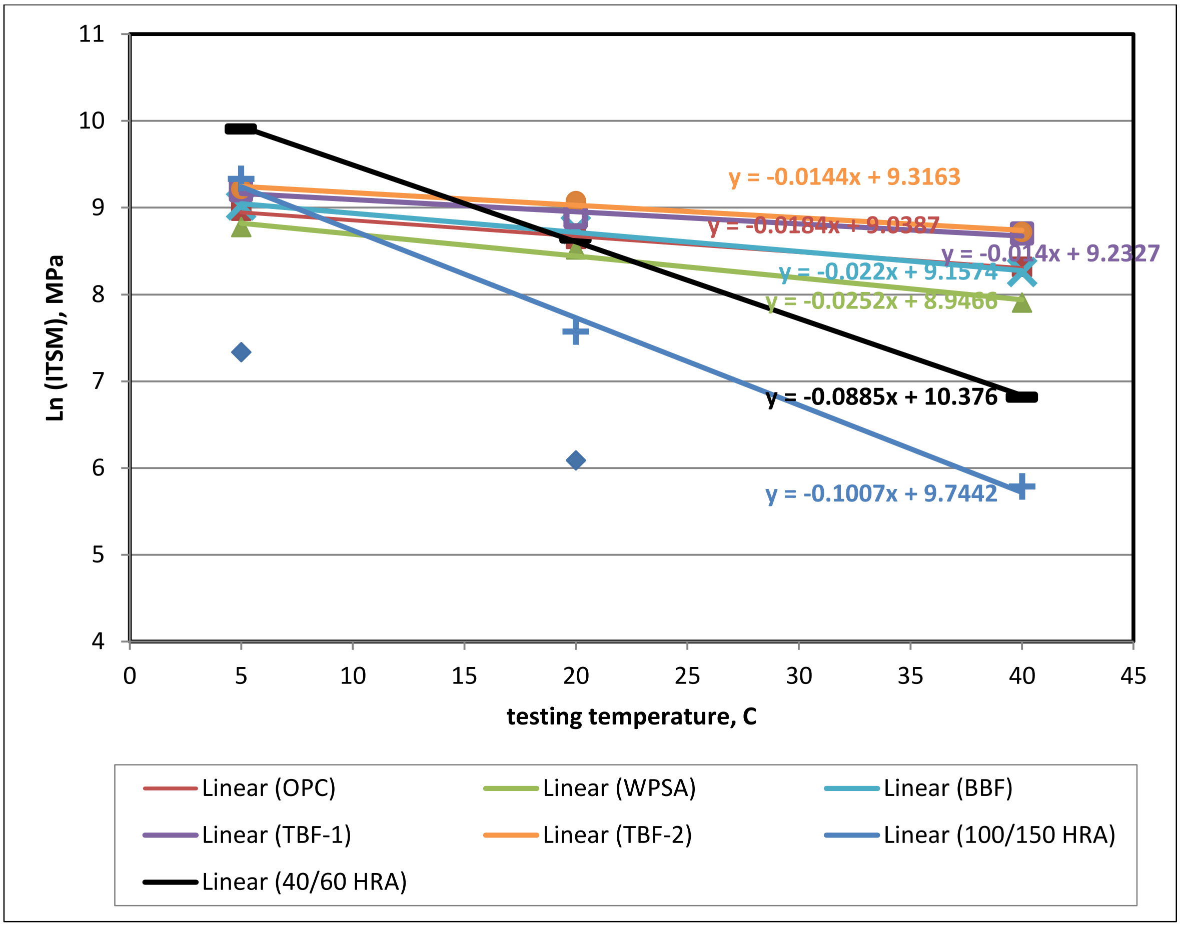


Figure 5-14: Temperature sensitivity of CRA and HRA mixtures

5.5 Performance of outdoor specimens

To ensure a better simulation of the site conditions, all the control and optimised CRA mixtures were manufactured and cured under rain and real temperature profile in outdoor exposure conditions. The samples were kept in their moulds for 24 hours @ 20 ºC prior to extrusion. Then, the sides of the samples were taped with plastic adhesion tape and placed on a flat surface outdoors on the car park area outside the lab building of the School of the Built Environment, Liverpool John Moores University. It is worth noting that the outdoor curing started on the 15/04/2013, which was a rainy day with a 7.9 ºC monthly temperature on average; Table 5-1 shows Liverpool's average weather by month while Figure 5-15 illustrates the mean monthly temperature in 2013. ITSM at 20 ºC and different curing times namely, 1, 3, 7, 14, 28, 90 and 180 days was used as a respected fundamental property to assess the performance of outdoor samples; the results are illustrated in Figures 5-16 and 5-17.

Table 5-1: Liverpool average weather by month for 2013 (Tutiempo, 2013)

Month	Average Temperature		Average daily rainfall, mm	Average snow days	Mean Humidity, %	Average fog days
	max	min				
January	6.4	2.3	2.2	3	88.1	5
February	6.8	1.7	2.1	4	81.9	3
March	6.3	0.4	1.9	2	78.1	1
April	11.7	4	1.7	1	69.6	1
May	14.3	7.3	1.5	0	72.8	1
June	18.2	10.6	2.2	0	72.5	2
July	23.1	14.1	2.6	0	70.8	1
August	20.2	14	2.1	0	74.3	1
September	17.3	10.9	2.5	0	77.9	2
October	15.3	10.1	3.2	1	82.1	2
November	9.7	5.1	2.7	1	82.1	3
December	6.8	2.6	2.8	4	87.8	5

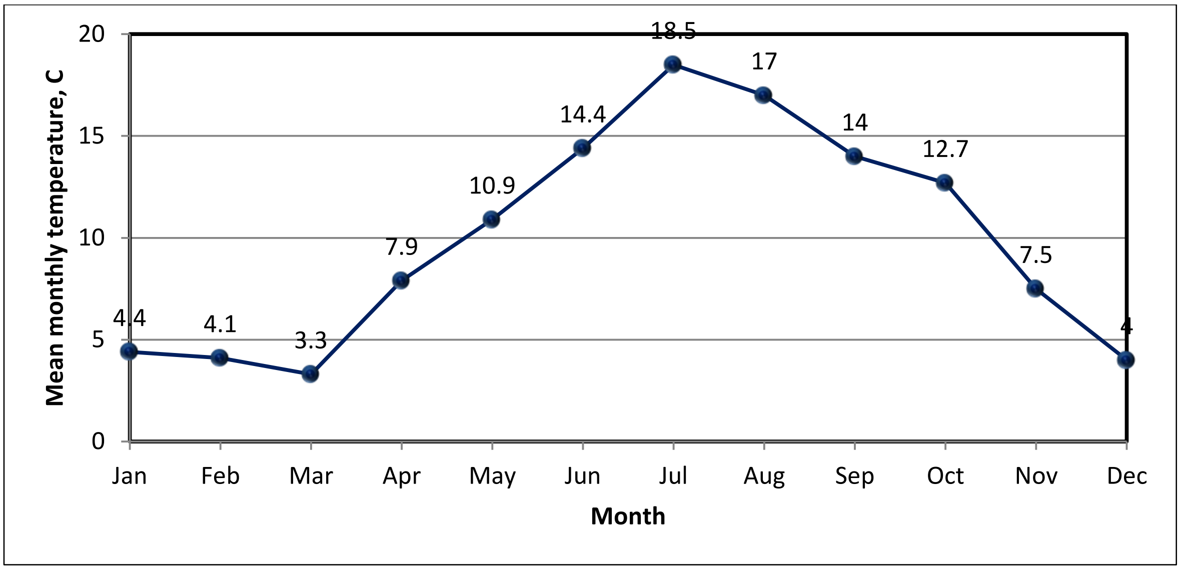


Figure 5-15: Average monthly temperature, ºC (Tutiempo, 2013)

As shown in Figure 5-16, the results of the modified CRA mixtures containing OPC, WPSA, BBF, TBF-1 and TBF-2 are very close to those for CRA conditioned under the normal curing method. On the other hand, outdoor CRA mixtures with OPC and TBF-2 samples still have the same greatly improved performance, since they required less than 1 day to meet the target 2000MPa, Figure 5-17. Also, the rate of gain of stiffness modulus values was considerably faster than untreated CRA mixtures and represents fantastic progress when compared with some BEM site trials without any addition, where 2 to 24 months is required (Leech, 1994). The performance of outdoor samples with real climatic condition and rainfall confirmed the positive activity of the new CRA mixtures due to increasing the early strength and adhesion properties between the primary and secondary binders with aggregate.

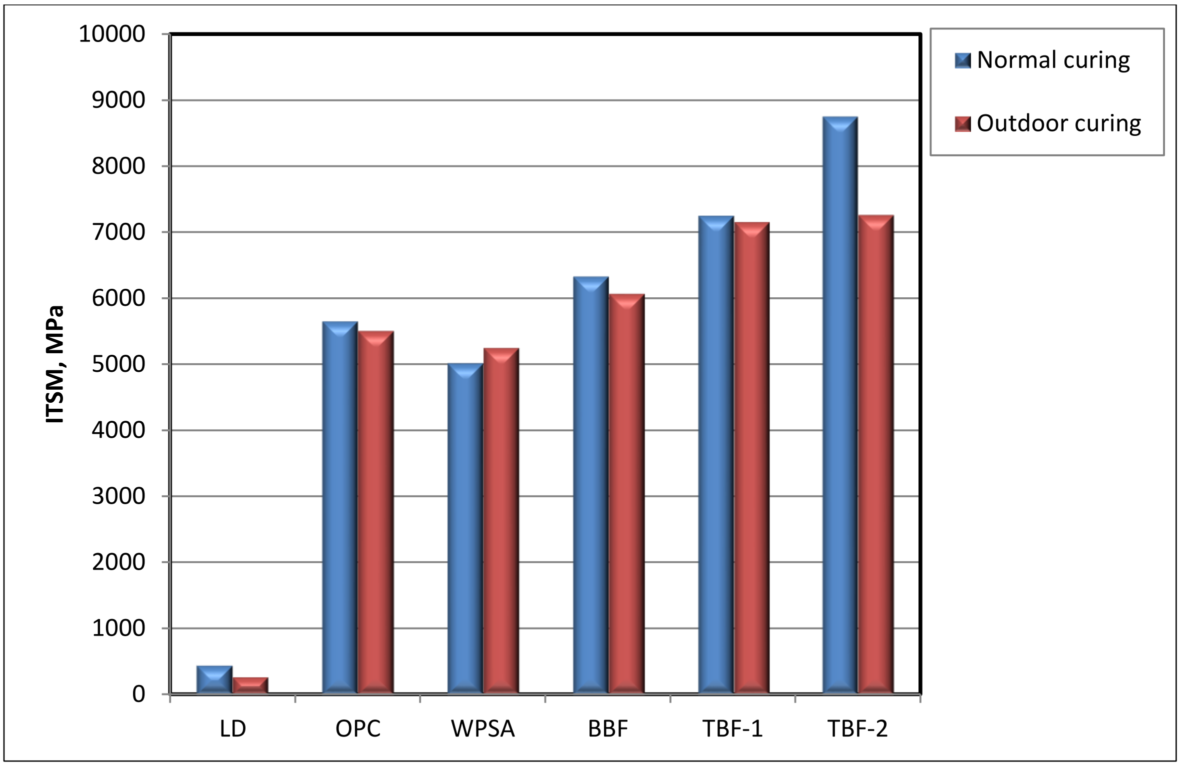


Figure 5-16: ITSM results for indoor and outdoor CRA mixtures after 28 days

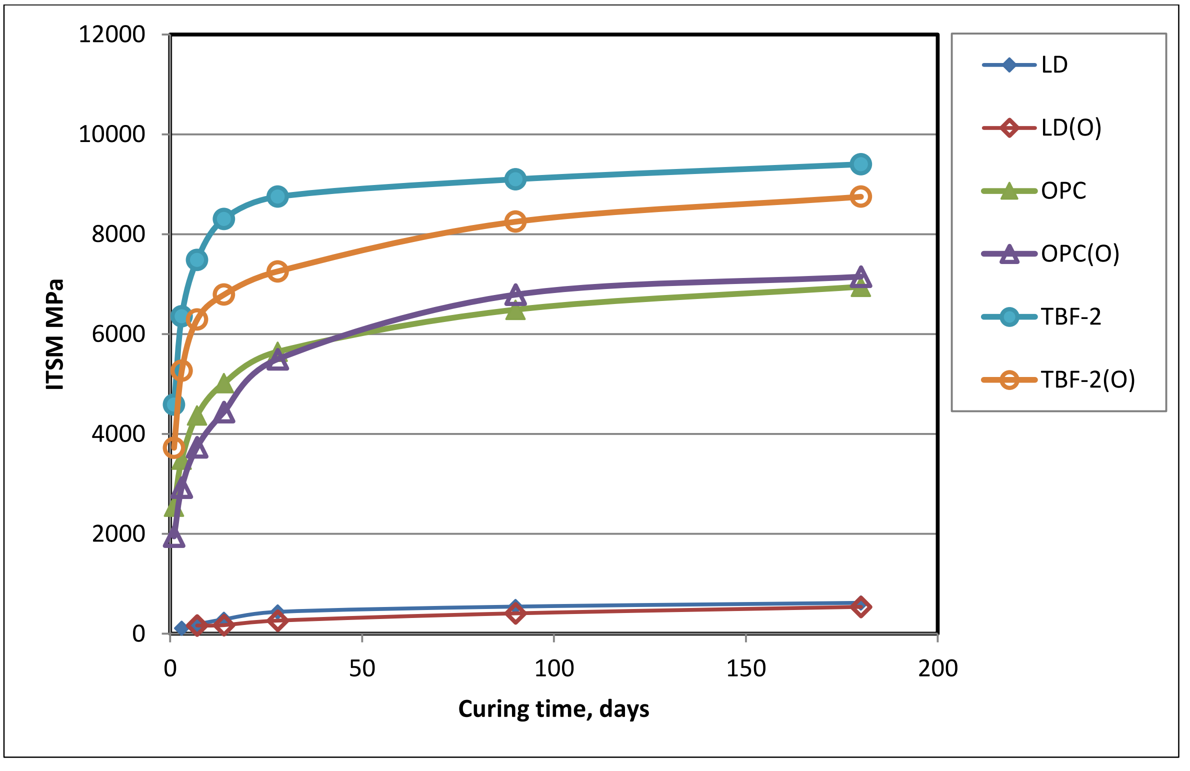


Figure 5-17: Rate of stiffness modulus gain of outdoor LD, OPC and TBF-2 samples

5.6 Water loss of CRA mixtures

To indicate the water loss of different CRA mixtures, all these mixtures were manufactured and left in the moulds for 24 hours before being extruded, then cured at 20 ºC in the lab room. Simply, water loss for each specimen was taken as the loss in mass at different ages, i.e. 1, 3, 7, 14 and 28 days. Figure 5-18 shows the results of water loss.

The following points are worthy of note from Figure 5-18:

- The rate of water loss for all mixtures before 7 days is higher than after that age,
- In general, it can be concluded that LD mixtures have high water loss in comparison with the other mixtures which incorporated hydrated filler,
- The period of investigation may be too short to indicate the progress of water loss for long-term studies.

The behaviour of CRA mixtures with cementitious filler consumes some of the trapped water incorporated in these mixtures, especially in the early days. Also, the evaporation process and absorption of the filler and aggregate materials to this water increased the total water loss of these mixtures. These results complement the finding of previous studies conducted by Puzinauskis and Jester (1983) and Brown and Needham (2000).

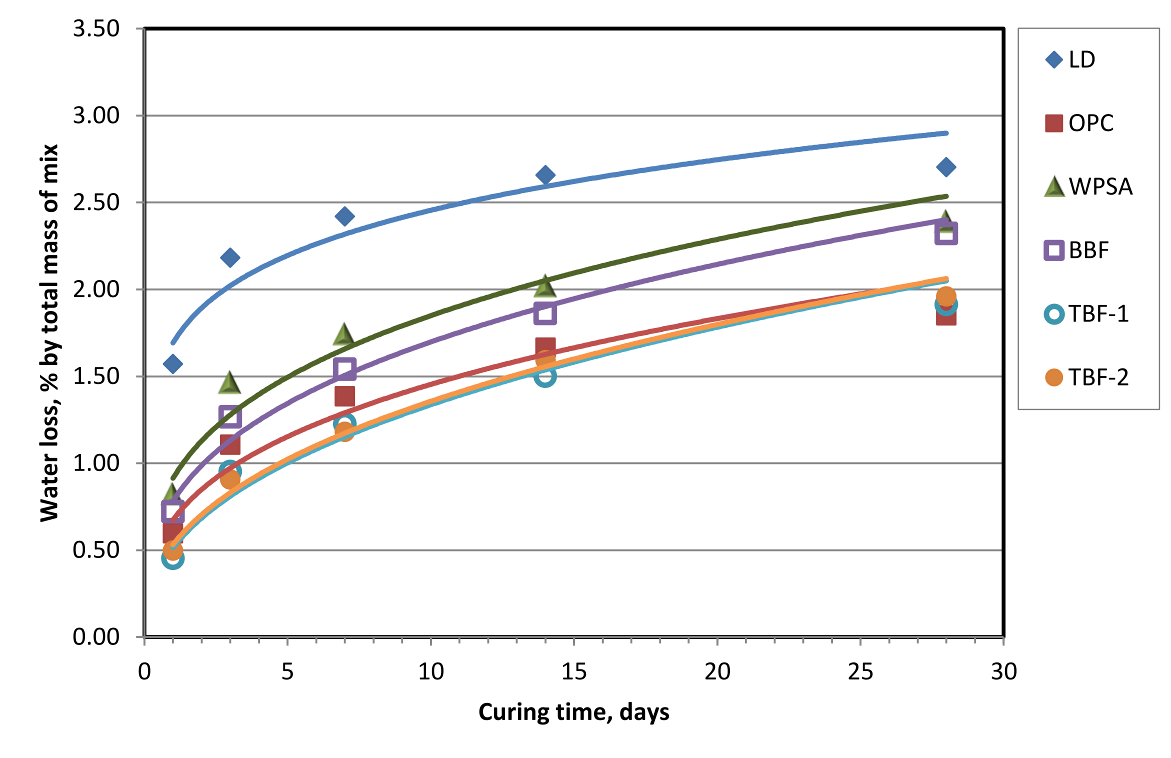


Figure 5-18: Effect of curing time in water loss of CRA mixtures

5.7 Summary

The main concerns of cold BEMs are the long curing time required to achieve the maximum performance and the inferior early life strength. A number of previous investigations indicated that the full curing of BEMs may occur after 2 months to 2 years depending on their ingredients and weather conditions.

This chapter has presented the optimisation sequence of the novel CRA mixtures in terms of stiffness modulus results, as a respected fundamental property, when different SCMs were incorporated individually or collectively instead of the conventional mineral filler. Accordingly, a number of CRA mixtures have been produced, as summarised below:

- Control CRA (LD) is produced with limestone dust as mineral filler and the results show inferior strength in comparison with the target stiffness modules, i.e. 100/150 HRA values;

- CRA containing OPC (OPC) mixtures is produced by replacing LD with 6 % OPC and the results reveal the positive performance of these mixtures;
- CRA containing 6 % WPSA (WPSA) mixtures are generated when LD has been replaced with WPSA, which is a cementitious material nominated as waste material; the results are promising due to the hydration process for WPSA with the trapped water incorporated in CRA mixtures as well as because of its hygroscopic nature;
- Another novel CRA mixture was produced when PLFA was used with WPSA to produce a new cementitious binary blended filler as the resultant filler contained 4.5:1.5 (WPSA:PLFA) proportion and is named BBF throughout this research. The results of CRA containing BBF (BBF mixtures) revealed the positive effect of replacement of LD with BBF and the stiffness modulus was increased incredibly;
- Further improvement from incorporating SF, which is a pozzolanic material, to the WPSA plus PLFA system revealed that a fast curing CRA is produced by generating ternary blended filler (named as TBF-1 mixtures) produced from blending these three materials as the stiffness modulus increased significantly and achieved the target strength after just 4 hours of normal curing;
- RHA, which is a bottom ash produced from burning rice husk, has a pozzolanic activity and is classified as waste material, is used instead of SF, which is a cost-minus material, to produce other ternary blended filler, named as TBF-2. The stiffness modulus of this mixture is as high as TBF-1 mixtures and more than those for CRA containing OPC filler.

Also in this chapter, temperature susceptibility of the produced mixtures has been assessed in terms of indicating the slope of the stiffness modulus vs temperature curves as the lower slope is less sensitive to the temperature changes. The results revealed a considerably lower thermal sensitivity for the whole CRA with cementitious filler, i.e. OPC, WPSA, BBF, TBF-1 and TBF-2, in comparison with the conventional HRA mixtures. Therefore, it can be expected that these CRA mixtures suffer less rutting in a hot weather and less fracture at low temperature in comparison with 100/150 and 40/50 HRA. The next chapter will investigate the performance of the produced mixtures in terms of the other fundamental mechanical properties, i.e. creep, fatigue and fracture behaviour.

Chapter Six

Other Mechanical Properties of the Optimised Mixtures

Chapter six is divided into three main areas of performance: investigating the permanent deformation, investigating fatigue life and investigating fracture toughness, each area contains a method, results and discussions. The experiments that were chosen for each abovementioned property were considered to provide a measure of the performance of the control CRA, i.e. LD, the other optimised CRA mixtures, i.e. OPC, WPSA, BBF, TBF-1 and TBF-2, and conventional HRA mixtures.

6.1 Permanent deformation performance

Rutting or permanent deformation is one of the main failure manners in flexible road pavements and thus affects pavement service time (Barksdale, 1967). It can be defined as the irrecoverable cumulative deformation that happens in the wheel path due to the cyclic traffic loading, mostly at elevated temperatures. Therefore, the depressions on the road surface along the wheel path related to other surface points are caused by permanent deformation. Two main directions of movement might be caused in the asphalt pavement due to permanent deformation, namely, downward movement (mainly due to compaction) and lateral movement (occurs as a result of shear failure).

The said cumulative permanent deformation, which represents all or some of the pavement structure layers' deformation because of high stresses caused in close surface asphalt layers due to the tyre pressure and axle load increment. Brown and Cross (1992) reported that the majority of rutting depth resulting from these stresses can be inspected in the top 75 to 100 mm of the asphalt layers. Additionally, on European roads, the permanent deformation created in asphalt layers is the most common pavement deterioration mode (European

Commission, 1999). Accordingly, investigating the characteristics of permanent deformation has become a focus of research as one of the most important fundamental properties.

Binder properties, aggregate properties, volumetric composition of mixtures and lab and field conditions highly influence the permanent deformation of asphalt layers. The effect of aggregate gradation is one of the main factors that affects the resistance of creep deformation, as reported by several previous studies such as Oliver *et al.* (1997), Brown and Cooper (1984) and Dukatz (1989). Oliver *et al.* (1997) conducted a laboratory and field study to assess the performance of rutting with different mixtures. They concluded that there is a substantial influence on rutting resistance in terms of aggregate gradation. Tarefder *et al.* (2003) concluded that aggregate gradation comes in the third place, after binder content and temperature, amongst the most important factors that affect permanent deformation potential of asphalts. Brown and Cooper (1984) conducted a study to assess the resistance of permanent deformation for continuously and gap graded mixtures. They concluded that the former has higher resistance than gap graded mixtures, but high fraction coarse aggregate gap graded mixtures (>70 %) had high rutting resistance because of their high aggregate interlocking.

Since the early 1970s, simple uniaxial creep tests have been utilised to investigate creep performance of asphalts; conversely, repeated load tests have been developed instead of these tests to comply with the actual case in the field and therefore are considered to distinguish between different bituminous mixtures (Read and Whiteoak, 2003).

The Uniaxial Cyclic Compression Test (UCCT) was comprehensively utilised to assess the resistance to creep performance in the design of bituminous mixtures as well as retrospective investigations. Repeated load triaxial cell and the wheel trucking tests can be used as well to study the creep performance. The latter tests are preferred when a degree of confinement is required, especially when the aggregate interlock is the main component to resist the permanent deformation (Vaughan, 1999).

The aim of this section is to contribute towards understanding permanent deformation in CRA mixtures containing different types of filler as optimised in the previous chapter. The Uniaxial Cyclic Compression Test (UCCT), which is detailed in section 4.4.1.2, was used in this study to determine the resistance to permanent deformation of a cylindrical specimen of CRA mixtures and conventional HRA mixtures as per BS EN 12697-25:2005 (European Committee for Standardization, 2005). The effect of different filler types on the behaviour of the produced CRA mixtures was highlighted and a detailed study in comparison with untreated CRA and conventional HRA has been done.

Alderson (1995) determined the creep slope at stage 2 from testing asphalts in repeated load axial creep tests and introduced typical minimum slope values for this stage, as illustrated in Table 6-1.

Table 6-1: Typical laboratory determined minimum dynamic stage 2 creep slope (Alderson, 1995), permission to reproduce this table has been granted by ARRB Group Ltd

Average annual pavement temperature (ºC)	Heavy traffic > 10^6 ESAL	Medium traffic 5xl0^5 to 10^6 ESAL	Light traffic < 5x10^5 ESAL
>30	< 0.5	0.5–3	> 3–6
20–30	< 1	1–6	> 6–10
10–20	< 2	2–10	Not applicable

6.1.1 CRA samples' preparation and conditioning

CRA samples with a 150 mm diameter were prepared according to the mixing process stated at section 4.3 and compacted to achieve (50 ± 2) mm final thickness.

Creep test was carried out on CRA mixtures at full curing condition. The full curing condition consists of two stages: the first stage was achieved by leaving the specimens in their mould for 1 day at lab temperature (20 ºC) before being extruded, while stage two was achieved by placing the specimens in an oven at 40 ºC for 14 days to reach their constant mass. At the end, the specimens were cooled at 20 ºC. After this conditioning process, the samples were tested at 40 ºC in HYD 25 Cooper Technology test apparatus, as shown in Figure 4-20.

6.1.2 Test results and discussion

The first step on the presentation of permanent deformation results will cover the performance of the control CRA and a comparison study with OPC as well as the conventional HRA mixtures. The relationship between cumulative axial creep strain and number of pulses of these mixtures is shown in Figure 6-1, while Figures 6-2 and 6-3 illustrate their ultimate creep stiffness modulus after 3600 pulses and the creep rate, respectively. It is obviously shown that control CRA mixtures complete the test with cumulative axial strain higher than conventional HRA mixtures. Generally, the behaviour of the control CRA mixtures is different than hot mixtures as their performance within stage 1 is somehow between the soft and hard bitumen base HRA. However, this behaviour shows a big difference at stage 2 as the rate of change of control CRA creep strain (5.03) is much higher than those for HRA mixtures (1.88 and 1.65 for 100/150 HRA and 40/60 HRA respectively). Also, the ultimate creep stiffness for these mixtures is less than the conventional HRA. Accordingly it is expected that control CRA mixtures display inferior permanent deformation performance more than the HRA mixtures.

On the other hand, OPC mixtures had remarkably lower cumulative axial strain in comparison with LD and conventional HRA mixtures. In terms of ultimate creep stiffness, OPC mixtures illustrate high values (38.4 MPa), which are much higher than control CRA (more than 10 times) and conventional HRA mixtures. As shown in Figure 6-3, it was revealed that creep rates for OPC mixtures (0.02) were extremely low and smaller than all the other mixtures, even HRA with 40/60 binder grade. When comparing the results with the typical laboratory minimum stage 2 slope which was specified in Table 6-1 earlier, it can be concluded that control CRA mixtures are not suitable for heavily trafficked pavement for all the ranges of annual pavement temperature i.e. >30, 20–30 and 10–20 ºC. However, OPC mixtures comply with all the heavily trafficked requirements even for high average annual pavement temperatures (>30 ºC).

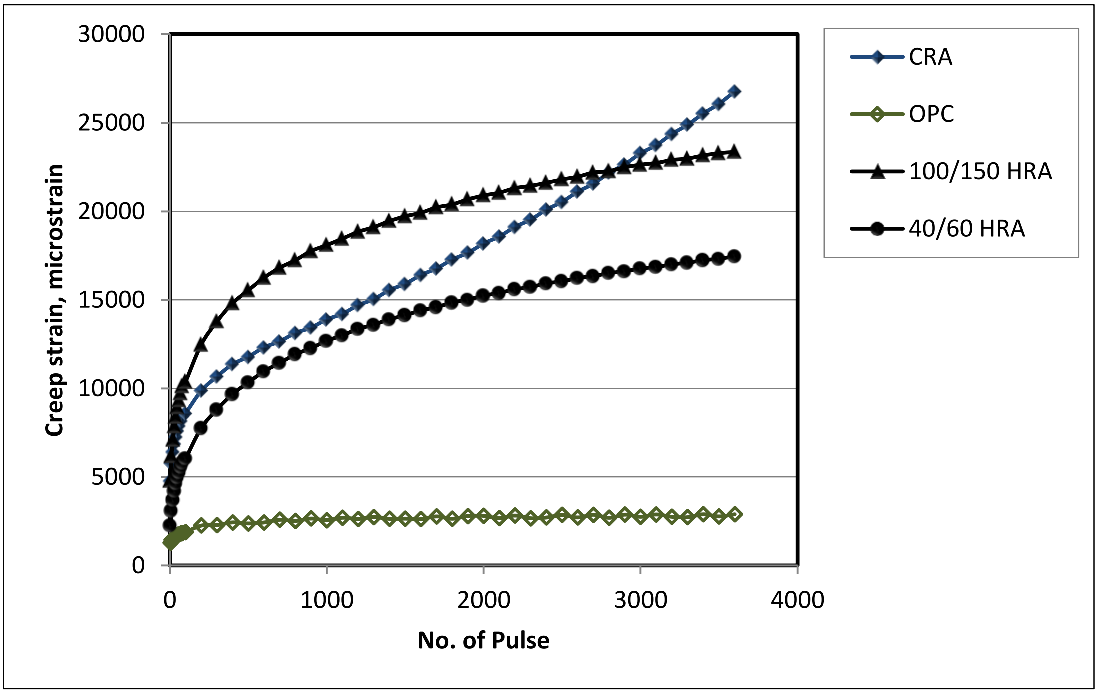


Figure 6-1: Creep strain versus number of pulse applications of specimens for control CRA (LD), OPC and HRA mixtures

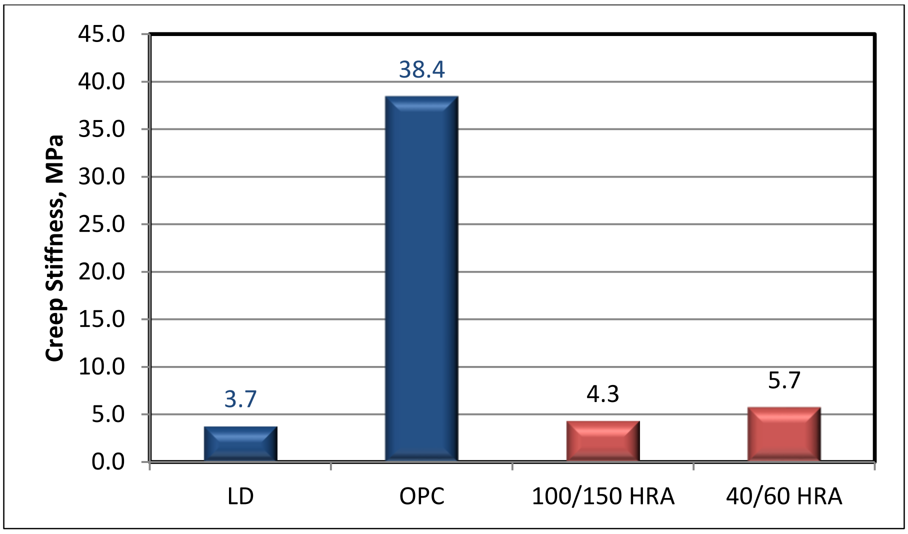


Figure 6-2: Ultimate creep stiffness for LD, OPC and HRA mixtures

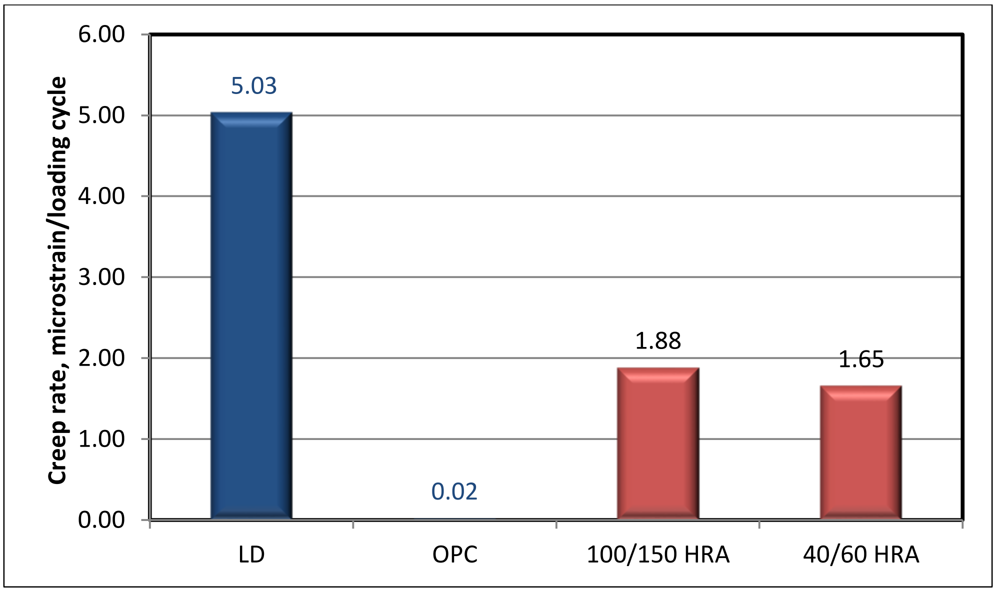


Figure 6-3: Creep rate for LD, OPC and HRA mixtures

The second step of the creep results comprised investigating the performance of CRA mixtures with unary, binary and ternary blended fillers produced totally from SCMs as optimised in chapter five to produce four sustainable fast curing CRA mixtures, i.e. WPSA, BBF, TBF-1 and TBF-2. The results are illustrated in Figures 6-4, 6-5 and 6-6 to assess the performance of these mixtures by determining the cumulative creep strain vs. number of pulses' relationship, ultimate creep stiffness and creep rate, respectively.

Figure 6-4 shows that the accumulative creep strain behaviour is extensively low in comparison with the LD and conventional HRA mixtures and it is similar to OPC mixtures' behaviour. Also, it is obvious from Figure 6-5 that ultimate creep stiffness increased dramatically for the optimised mixtures with interesting increments of approximately 7, 12, 10 and 11 times those for control CRA for WPSA, BBF, TBF-1 and TBF-2, respectively. Finally, the creep rate decreased significantly for these mixtures in comparison with untreated mixtures as well as HRA mixtures, Figure 6-6. More interestingly, it can be indicated that these mixtures can be used for heavily trafficked pavements (> 10^6 ESAL) even with worst climatic temperatures when compared with Table 6-1 requirements.

To sum up, the candidate CRA mixtures, i.e. WPSA, BBF, TBF-1 and TBF-2, have a superior performance in terms of creep test results and represent a further enhancement in addition to their dramatic stiffness modulus improvements as described in the previous chapter, Table 6-2. This enhancement of creep performance of CRA mixtures with different cementitious fillers, i.e. WPSA, BBF, TBF-1 and TBF-2, complies with the role of OPC when incorporated in CRA and strongly agrees with the previous studies such Li *et al.* (1998), Brown and Needham (2000) and Giuliani (2001). Moreover, it is expected that these fast curing CRA mixtures, i.e. WPSA, BBF, TBF-1 and TBF-2, will overcome most of the characteristics of bitumen and the new cementitious binder together, i.e. low permanent deformation, long fatigue life and lower temperature susceptibility of cementitious concrete and higher flexibility and toughness of bituminous concrete.

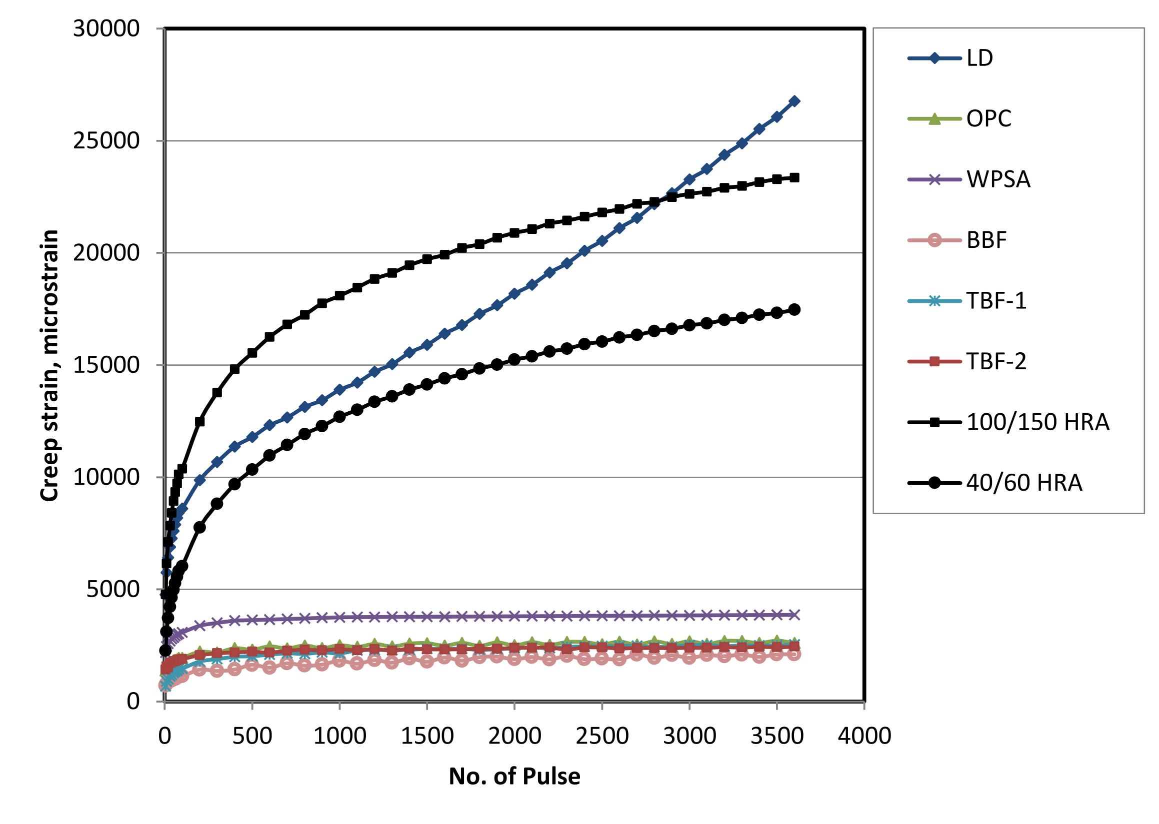


Figure 6-4: Creep strain versus number of pulse applications of the optimised mixtures

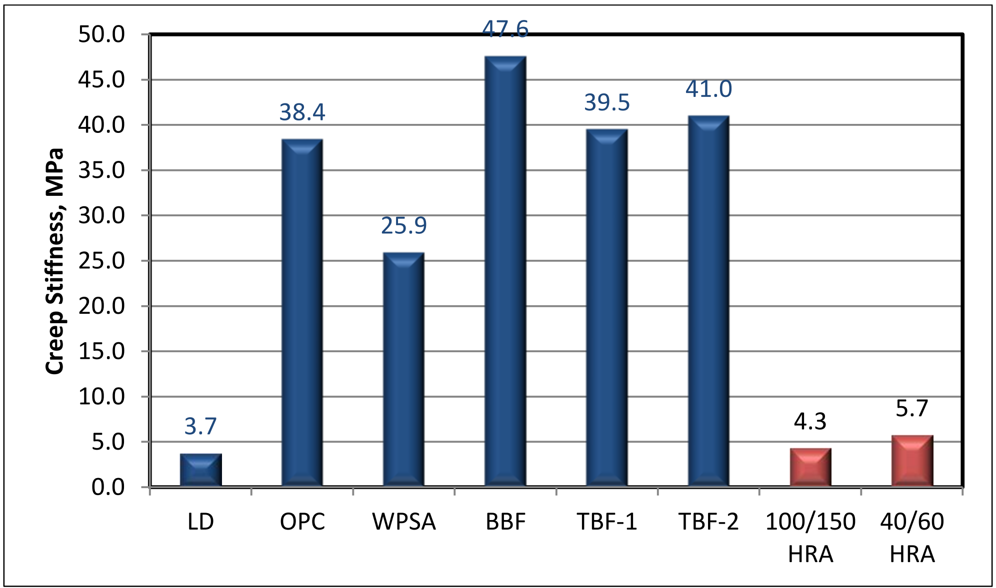


Figure 6-5: Ultimate creep stiffness for the optimised mixtures

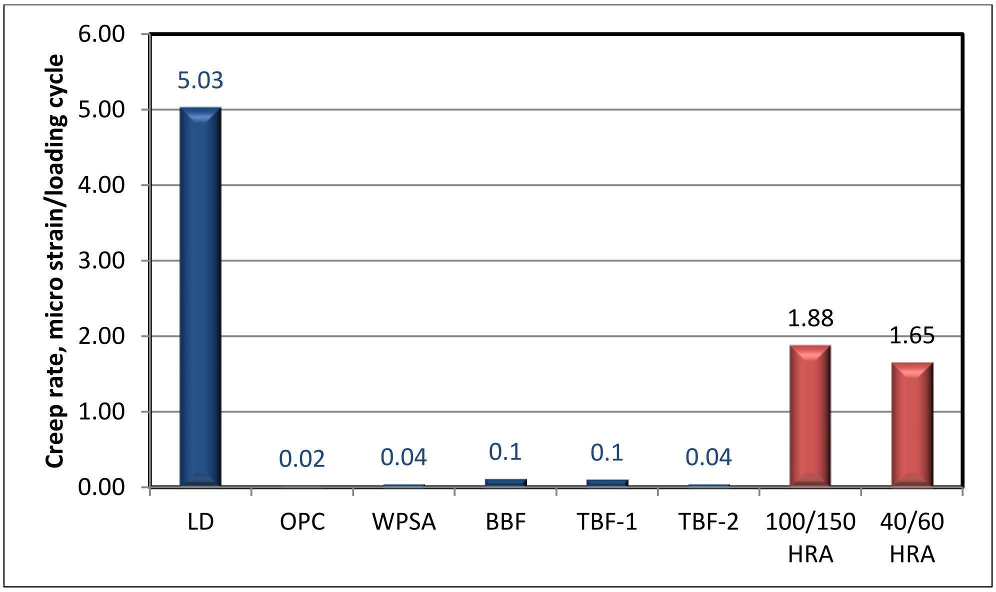


Figure 6-6: Creep rate for the optimised mixtures

Table 6-2: Summary of creep test results

Mix type	UCCT test results				Notification
	Accumulative axial strain after 3600 pulses, mm/mm	Ultimate creep stiffness, MPa	% increase compared with untreated CRA	Creep rate, micro strain/ loading cycle	ITSM after 3 days with normal curing, MPa
CRA mixtures					
LD	26767	3.7	---	5.03	110
OPC	2603	38.4	938	0.02	3492
WPSA	3860	25.9	600	0.04	3233
BBF	2100	47.6	1186	0.1	4826
TBF-1	2532	39.5	968	0.1	5521
TBF-2	2441	41	1008	0.04	6358
HRA mixtures					
100/150	23353	4.3	---	1.88	1941
40/60	17463	5.7	---	1.65	5705

6.2 Fatigue life investigation

Bituminous mixtures are commonly exposed to cracking caused by thermal cycling, repeated traffic loads or a combination of the two types. Cracking can be considered as one of the main modes of distress in flexible pavements. Tensile stresses at the bottom of the pavement layer are generated due to temperature changes and applied traffic load, and remain under the tensile strength of the pavement layer for a specific time, after which cracks initiate at weak points of the pavement layer. Then, fracture occurs when these cracks propagate towards the surface of the asphalt pavement layer.

Evdorides *et al.* (2006) studied the influence of the existence of cracking in pavement. They stated that the effective modulus of the asphalt is significantly affected by these cracks, which in turn reduces the effect of temperature on pavement performance. Also, water penetration inside these cracks will cause weakening of the foundation of the pavement structure.

Two stages of cracking can be considered: crack initiation and crack propagation. Forming of a macro crack from an accumulation of micro cracks due to the cyclic load application and thermal changes represents the first stage. Also, this stage can be investigated to indicate the fatigue life using different types of testing such as Indirect Tensile Fatigue Test (ITFT) and Four-Point Load Fatigue Test (4PT). Additional applications of traffic load and temperature variation cause the growth of macro cracks through the asphalt layer in a phenomenon known as crack propagation, which represents the second stage of cracking failure. The latter can be investigated by different methods and it is covered in the next section by conducting a Semi-Circular Bending (SCB) monotonic test.

Cracking resistance of asphalts can be defined as their ability to resist cyclic traffic loading without fracture. In terms of mixtures' characterisation, mixture stiffness, bitumen properties and content and air voids are the main variables that affect crack resistance of asphalts (Pell, 1973). One of the most important of the above factors is stiffness of the bituminous mixture. Pell reported that a mixture with higher stiffness has longer fatigue life when tested in stress-controlled mode, whereas there is a reduction in fatigue life when using a control strain mode of loading.

Also, the phenomenon of fracture under cyclic stress whose magnitude is often less than the tensile strength of the mixture is called fatigue. Fatigue cracking of bituminous mixtures can be caused with tensile strains in the range of 30 to 200 microstrains, as reported by Thanaya (2003).

Stress magnitude, loading frequency and duration of rest period between load applications on the test procedure strongly affect fatigue life (Croney and Croney, 1998).

Characteristics of bitumen have a significant role in crack resistance properties of asphalts as, in a stress-controlled mode, higher crack resistance can be achieved with higher bitumen stiffness. On the other hand, in a strain mode of loading the opposite is correct, as reported by Cooper and Pell (1974). Also, it was concluded that crack resistance properties improved when bitumen content increased and air voids decreased (Gibb, 1996).

Aggregate gradation has a considerable effect on fatigue life of bituminous mixtures. Asphalts prepared with finer gradation were better than mixtures that incorporating coarser gradations in terms of fatigue performance (Thanaya, 2003).

Fatigue life is often investigated in the laboratory by cyclic load tests. The European Committee for Standardization (2012c) identifies the methods for characterising the fatigue of bituminous mixtures using alternative test procedures, comprising bending tests such as two-point bending test on trapezoidal specimens, two-point bending test on prismatic-shaped specimens, three-point bending test on prismatic-shaped specimens, four-point bending test on prismatic-shaped specimens and indirect test represented by indirect tensile test on cylindrical-shaped specimens. All of these tests are performed on compacted asphalt materials under a sinusoidal loading or other controlled loading utilising different kinds of specimens and supports.

There are two conventional modes normally used to carry out fatigue test; these are: controlled stress mode and controlled strain mode. In the controlled stress method, the stress is maintained constant, which increases the strain during the test, whereas in controlled strain the strain is kept constant by decreasing the stress during the test. Generally, controlled stress is more related to a relatively thick pavement layer (> 150mm), as high stiffness modulus is the essential parameter that supports fatigue life. On the other hand, for thin traditional asphalt pavements the controlled strain mode is more relevant, as the elastic recovery properties have an essential effect on their fatigue life.

Fatigue life of bituminous mixtures can be defined using different approaches. Reduction of 50 % of the initial stiffness modulus has been extensively utilised to indicate the fatigue life in controlled strain mode, while, for controlled stress mode, it is considered to occur when the stiffness modulus is at 10 % of its initial value.

Based on the dissipated energy concept, Pronk (1997) and Hopman *et al.* (1989) introduced an energy ratio, Rn, and defined fatigue failure as the number of repetitions, N_1, when cracks are considered to initiate. Moreover, they specify that at this stage, i.e. N_1, coalescence of micro cracks have occurred to form a macro crack, which then propagates; hence it represents a transition point between micro crack formation and the propagation of a macro crack. According to the Rn approach, indication of N_1 is dependent on the mode of test, i.e. controlled stress or controlled strain mode. For the former, when Rn is plotted versus the number of repetitions, N_1 is the point where the slope of Rn versus N deviates from a straight line, while, for the latter, it corresponds to the peak value of Rn. This approach of analysis is different from the first approach as N_1 represents the mixture in the same state of damage that is corresponding to initiation of a macro crack.

Another approach has been proposed by Dibenedetto *et al.* (2003) to characterise fatigue, as they identified the existence of a stage during a fatigue test which accounted for most of the fatigue life, where there is approximately linear stiffness reduction in the stiffness modulus with repeated load cycles. Accordingly, a damage parameter (D) has been introduced to characterise fatigue life. Damage due to fatigue was depicted by studying the change in D with number of cycles.

In this investigation, the four-point bending test on prismatic-shaped specimens, which is detailed in section 4.4.1.3, was used to assess the fatigue characteristics of all the CRA and HRA mixtures. The controlled strain mode was used and fatigue life was defined as the number of cycles required to reduce the initial stiffness modulus by 50 %.

6.2.1 CRA samples' preparations and curing

Prismatic samples for the optimised CRA mixtures, i.e. control CRA (LD), OPC, WPSA, BBF, TBF-1 and TBF-2, with approximately 400 x 50 x 50 mm (length, wide and height) were prepared according to the mixing method detailed at section 4.3 and compacted by means of a static compaction using a compressive machine under a gradual application of 0.4 MPa/sec static load for 2 min.

4BP tests were carried out on CRA mixtures such as creep test at full curing condition. As specified earlier at 6.1.1, this curing procedure comprises three steps. Firstly, the samples were left in their moulds for 24 hour at 20 ºC. Secondly, the samples were placed in an oven at 40 ºC for 14 days to achieve their constant mass. Finally, the samples were cooled at lab temperature, i.e. 20 ºC. Then the samples were tested at 20 ºC in a 4BP testing machine as specified in the previous subsection.

6.2.2 Results and discussion

The resistance to fatigue cracking of all the CRA mixtures, i.e. LD, OPC, WPSA, BBF, TBF-1 and TBF-2, as well as HRA mixtures was determined using the 4PB as described earlier. This test procedure can give useful data on the relative fatigue performance of mixtures; therefore the effect of the new high-performance CRA mixtures, i.e. WPSA, BBF, TBF-1 and TBF-2, could be quantified. The fatigue life (N_f) for CRA mixtures and traditional HRA mixtures is shown in Figure 6-7 while Figure 6-8 illustrates the fatigue line for the control CRA and the superior TBF-2 CRA mixture.

The results presented in Figure 6-7, which is based on 150 μstrain controlled strain criteria, show that fatigue life increased incredibly for CRA mixtures with treated fillers in comparison with untreated CRA mixture and conventional HRA mixtures. Therefore, replacement of limestone dust with these fillers, i.e. WPSA, BBF, TBF-1 and TBF-2, extends the fatigue life more than 8, 20, 24 and 22 times those for control mixtures, respectively. These results conform to the performance of all the mixtures at different temperatures discussed earlier in 5.4. Furthermore, the produced mixtures are performing much better than CRA mixtures with OPC in terms of fatigue life. The performance of OPC, WPSA, BBF, TBF-1 and TBF-2 is logical, in particular when considering the high stiffness modulus after full curing of these mixtures, which is much higher than the control CRA and conventional HRA mixtures.

On the other hand, Figure 6-8 shows the fatigue line for control CRA and TBF-2 mixtures. The spread of data for untreated and treated CRA mixtures was quite narrow as the R^2 was 0.99 and 0.93 respectively. Moreover, it is clearly shown that the fatigue life for TBF-2 mixtures is extensively higher than that of the control CRA mixtures for all the tensile strain under study. Brown and Needham (2000) stated that the possible strain levels that might be experienced in a pavement structure are below 200 microstrain and therefore are most important in practical situations. Subgrade and mixture stiffness, layer thickness and load are the main parameters affecting by the actual strain value. Accordingly, incorporating TBF-2 in CRA mixtures significantly extends the fatigue life.

The results of this investigation show that a further improvement can be reported in terms of fatigue performance for the candidate CRA mixtures in addition to their significant stiffness modulus and creep performance characteristics, as described earlier and summarised in Table 6-3.

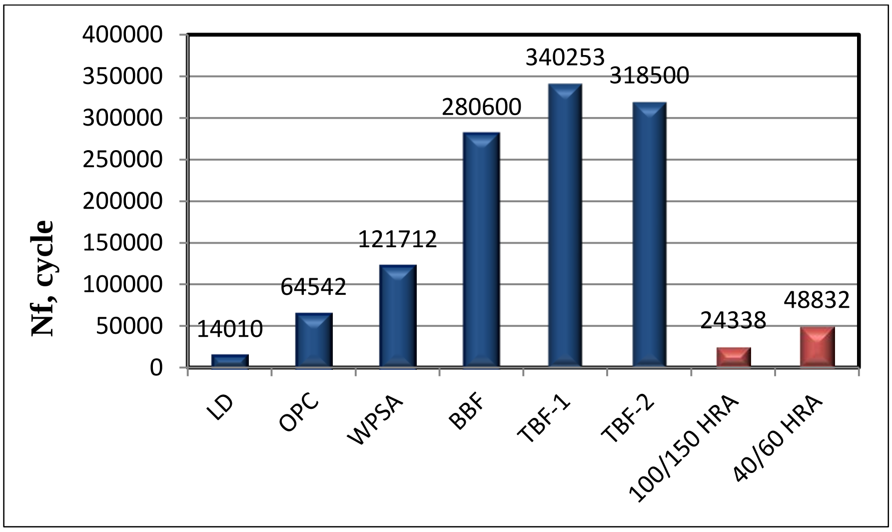


Figure 6-7: Fatigue lives for the optimised CRA mixtures and traditional HRA mixtures

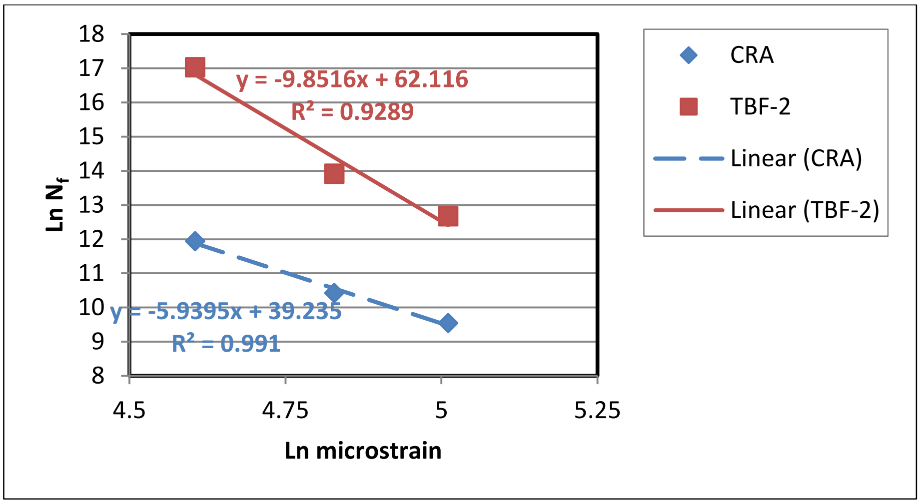


Figure 6-8: Fatigue line for control CRA and TBF-2 mixtures

Table 6-3: Summary of 4BP fatigue test results

Mix type	4BP test results			Notification	
	N_f at 100 microstrain	N_f at 125 microstrain	N_f at 150 microstrain	Ultimate creep stiffness, MPa	ITSM after 3 days with normal curing, MPa
CRA mixtures					
LD	153546	33670	14010	3.7	110
OPC	---	---	64542	38.4	3492
WPSA	---	---	121712	25.9	3233
BBF	---	---	289600	47.6	4826
TBF-1	---	---	340253	39.5	5521
TBF-2	2501504	1102034	318500	41	6358
HRA mixtures					
100/150	---	---	24338	4.3	1941
40/60	---	---	48832	5.7	5705

6.3 Fracture toughness performance

Cracking comprises crack initiation and crack propagation. The former represents the accumulation of micro cracks to form a macro crack because of the cyclic loading and it can be investigated by finding out fatigue life, as detailed in the previous subsection; while growth of a macro crack through the material due to further application of cyclic loading is known as crack propagation and it can be assessed by utilising a monotonic test, as described in this subsection.

Artamendi and Khalid (2006) stated that there are three fracture modes, namely: mode I (opening or tensile mode), mode II (sliding mode) and mode III (tearing mode), which can be followed individually or collectively by crack propagation depending on the configuration of the load, Figure 6-9. The tensile mode is caused when the crack tip is exposed to a vertical stress as the surfaces of the crack move directly apart resulting in perpendicular displacement of these surfaces to the crack plane. In sliding mode, the crack tip is exposed to an in-plane shear stress, causing a sliding of the crack surfaces over one another and crack surfaces to displace in the crack plane vertical to the crack front. Lastly, in tearing mode an out of plane shear stress is exposed on the crack tip, therefore the crack surfaces move parallel to the crack front and remain in the crack plane. Generally, the tensile mode (mode I) is the most frequently faced and it is related to a local displacement as the surfaces of the crack move directly apart. In this subsection, tensile mode is considered as the aim was to assess the performance of the control and optimised mixtures on crack propagation properties.

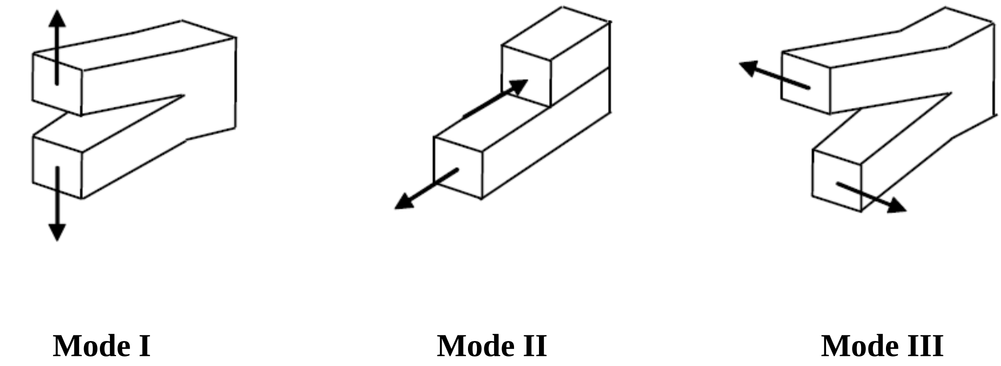


Figure 6-9: Asphalts' fracture modes

Usually, comparing calculated stresses with tensile strength of asphalt was used to characterise fracture behaviour of the material. According to this method, the tensile strength is adopted to characterise the fracture criteria, thus the higher resistance to fracture can be achieved with higher tensile strength. Nevertheless, fracture mechanics theory found that a very susceptible material to fracture in the presence of cracks can be established with high-strength material because these cracks act as stress concentrators. Generally, a pavement containing cracked asphalt layers will collapse at a much lower level of stress than it can withstand without the crack.

The first application of fracture mechanics theory to depict crack propagation of asphalt was based on the Linear Elastic Fracture Mechanism (LEFM) (Irwin, 1957). He introduced fracture toughness, known as stress intensity factor as well, to describe the stress field at the crack tip. Paris and Erdogan (1963) stated that the crack propagation rate is related to the stress intensity factor of the pure tension loading mode and this relationship is well known as Paris' Law.

On the other hand, Abdulshafi and Majidzadeh (1985) applied the critical strain energy release rate, known as critical J-Integral as well, to asphalt based on Elastic-Plastic Fracture Mechanics (EPFM). They gained a load-displacement relationship for notched samples with different primary crack length tested under load-controlled mode and the J-Integral value represented the slope of this relationship.

In a recent study, Paris' Law was used by Hassan (2009), using stress intensity factor, to investigate the influence of Incinerator Bottom Ash Aggregate (IBAA) content level on crack propagation resistance of asphalts. Furthermore, he examined the applicability of Paris' Law using J-Integral to IBAA bituminous mixture. He concluded that Paris' Law was suitable to characterise crack growth properties of IBAA bituminous mixtures using stress intensity factor or J-Integral.

Hence, fracture toughness can be defined as a measure of a bituminous mixture's resistance to crack propagation when the stress state near the crack tip is predominantly plane strain and tensile mode monotonic loading is applied (Artamendi and Khalid, 2006). Consequently, the critical load at which a construction with a certain crack length fails can be estimated from fracture toughness.

Numerous types of tests can investigate the determination of mixture crack characteristics. These characteristics are often assessed utilising the total response of a specimen when exposed to a particular mode of loading. The geometry and mixture factors are the main aspects that affect the response of the material. Thermal Stress Restrained Specimen (TSRST), recommended by Monosmith *et al.* (1965), Indirect Tension Test (ITT), recommended by Hadipour and Anderson (1988), Direct Tension Test (DTT), recommended by Bolzan and Huber (1993), and Semi-Circular Bending (SCB) test, recommended by Molenaar *et al.* (2002), are the most common types of testing used to investigate material crack propagation.

Considerable attention has recently been paid to the latter test as a respected laboratory type testing to assess the resistance of asphalts to crack propagation. Molenaar *et al.* (2002)

reported several advantages from using the SCB monotonic test. One of the main advantages that can be observed is that a clear crack progresses without smashing near the loading strip, as can be recognised in the ITT, which reveals the failure mode to be due to tension, rather than any other cause, Figure 6-10. Furthermore, the SCB monotonic test is a simple and cheap test that can be simply implemented on half-cylindrical samples.

In this research, SCB monotonic test, which is described previously in section 4.4.1.4, has been used to investigate the influence of incorporating of different cementitious fillers, i.e. OPC, WPSA, BBF, TBF-1 and TBF-2, in CRA mixtures on the crack propagation resistance of these mixtures. Also, a comparative study with the control CRA and conventional HRA mixtures was conducted.

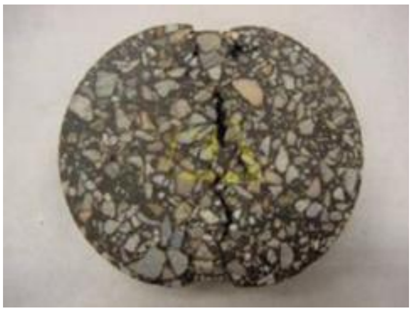


Figure 6-10: Crack shape in SCB test and ITT (Molenaar *et al.*, 2002), permission to reproduce this figure has been granted by Association of Asphalt Paving Technologists

6.3.1 Sample preparation and curing

The mixing process detailed at section 4.3 was used to prepare all the CRA mixtures and they were compacted to achieve (150 ± 1) mm diameter and (50 ± 3) mm thickness. SCB tests were carried out on CRA mixtures at full curing condition such as creep and fatigue test; therefore the curing procedure is the same procedure as described in sections 6.1.1 and 6.2.1 previously.

Each sample was then cut, perpendicular to the axis, in half to obtain semi-circular pieces. These were then notched: notch length (a) was 10 mm, at mid-point in the direction of the load using a diamond-tip saw tile cutter. Metal bearing strips, with a length of at least the thickness of the sample, a width of (10,0 ± 0,2) mm and a height of (20,0 ± 0,5) mm, were glued to the samples where they rested on the rollers. Then all CRA and HRA half samples were cured in a temperature-controlled cabinet at 5 ºC for at least 4 hours before the test, as shown in Figure 6-11.

Figure 6-11: Samples curing in a temperature-controlled cabinet at 5 ºC

6.3.2 Results and discussion

The load-deformation relationships of notched monotonic SCB samples corresponding to the six CRA mixtures and two HRA mixtures tested are presented in Figure 6-12. Generally, two load-deflection responses can be recognised, namely linear and non-linear relationships. The linear response converted to non-linear a short time before the ultimate load was reached due to a generation of plastic zone ahead of the crack tip. The non-linear behaviour of CRA mixtures was dependent on the type of the filler. When the ultimate load was reached, the crack propagated until failure.

It is noticeable that the behaviour of control CRA is similar in shape to the 100/150 HRA and it is obvious in the formation of the non-linear region and in the crack propagation rate after the ultimate load is reached. In the same manner, treated CRA mixtures, i.e. OPC, WPSA, BBF, TBF-1 and TBF-2, are similar to 40/60 HRA in terms of load-deformation relationship shape. The non-linear response for these mixtures starts at deformation close to the deformation corresponding to the maximum load then, after the maximum load, cracks propagated very rapidly to cause a sudden failure. This similarity in the trend behaviour might be influenced by the stiffness value of each sort of mixture, i.e. cold or hot, as the low stiffness mixtures such as control CRA (with low stiffness value after full curing) and 100/150 HRA which have wide non-linear zone while the other mixtures (high stiffness CRA and 40/60 HRA) have slightly rapid failure within the narrow non-linear zone.

On the other hand, Figure 6-13 shows the calculated fracture toughness, by using equations 4-6 and 4-7, for all the mixtures, i.e. CRA and HRA mixtures, which is linked to the maximum load presented in the previous figure. It is clearly shown that all CRA mixtures have less fracture toughness in comparison with conventional HRA mixtures despite they have high stiffness. This can be due to the high air voids of CRA mixtures (which will be described in detail in chapter nine), which highly affect the crack propagation mechanism. Furthermore, it can be concluded that there is no further enhancement in terms of crack propagation resistance when new cementitious fillers have been incorporated instead of conventional mineral filler. This can be attributed to the effect of high voids content being larger than the effect of mixture stiffness increase.

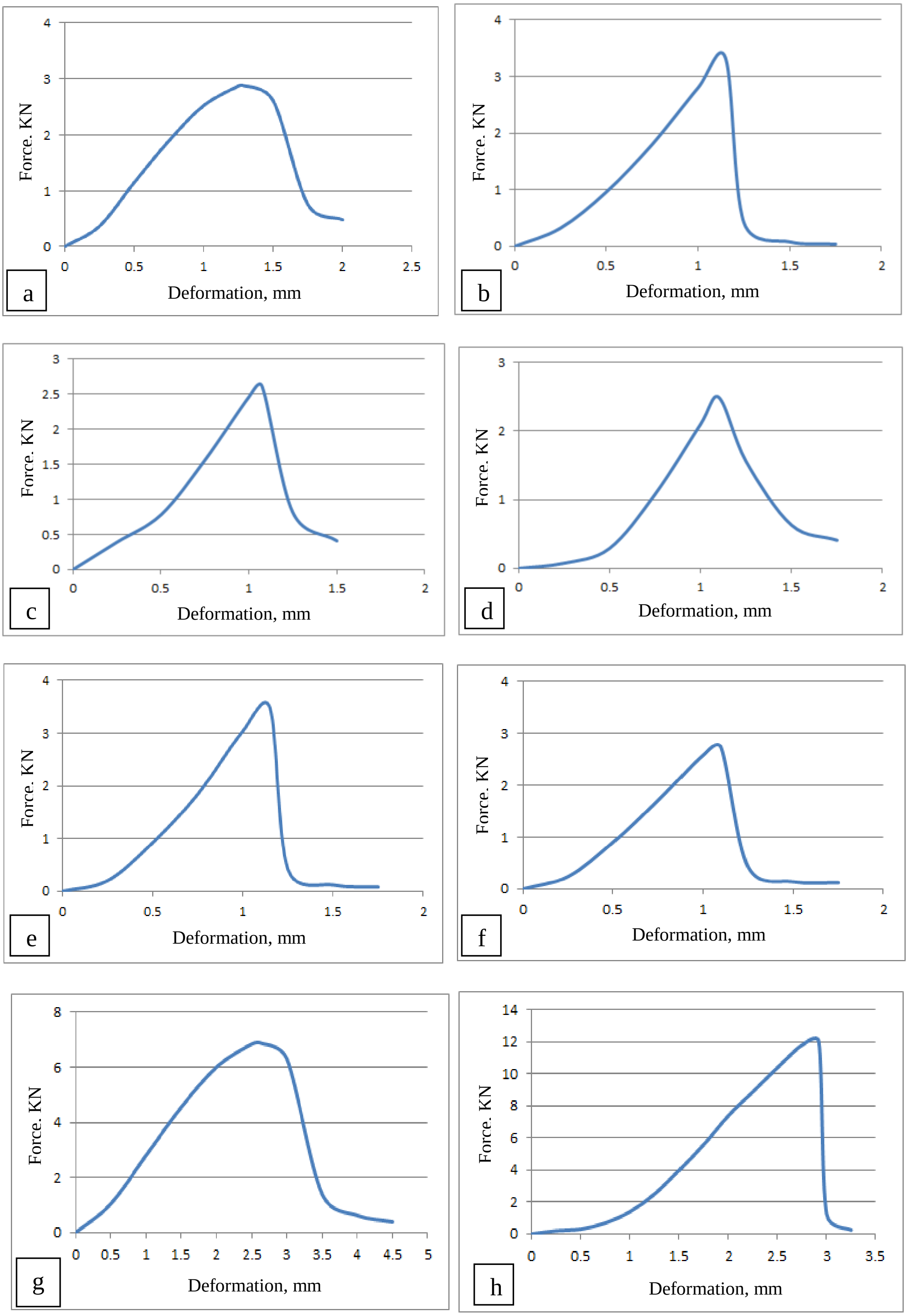


Figure 6-12: Load-deflection relationship for mixtures: a. LD, b. OPC, c. WPSA, d. BBF, e. TBF-1, f. TBF-2, g. 100/150 HRA and h. 40/60 HRA

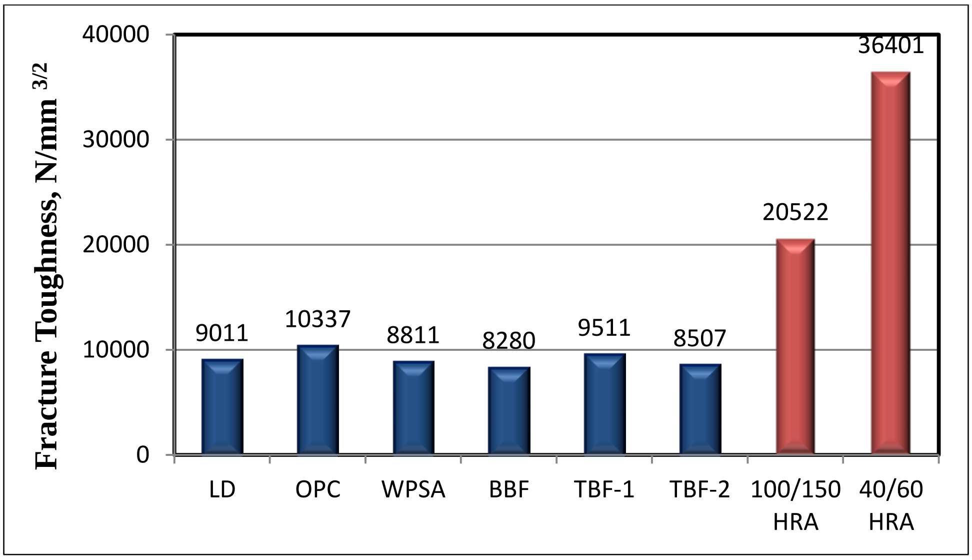


Figure 6-13: Fracture toughness for optimised CRA mixtures and HRA mixtures

6.4 Summary

As the CRA mixtures produced with cementitious fillers, i.e. WPSA, BBF, TBF-1 and TBF-2, showed a significant early stiffness modulus in the previous chapter, therefore this chapter has investigated the other fundamental mechanical properties, i.e. creep, fatigue life and fatigue fracture.

In general, different creep performance behaviour with lower ultimate creep stiffness for control CRA mixtures can be observed in comparison with the conventional HRA mixtures. However, when CRA mixtures were treated with SCMs to produce WPSA, BBF, TBF-1 and TBF-2 a remarkable enhancement has been observed, as the ultimate creep stiffness increased significantly and the creep rate decreased dramatically. For example, creep stiffness increased more than 11 times the control CRA for TBF-2 mixtures and is comparable with OPC mixtures.

On the other hand, there is a considerable increase in fatigue life, which is linked with the crack initiation phase, for CRA mixtures when limestone dust has been replaced with WPSA, BBF, TBF-1 and TBF-2. Another study has been done to indicate the fatigue life of the control CRA and TBF-2 mixtures with different micro tensile strain. The results illustrated that fatigue life is very high when CRA mixtures were treated with TBF-2 filler materials for all the tensile strain under study, i.e. 100, 125 and 150 μstrain.

Lastly, the crack propagation phase has been studied by conducting a monotonic SCB test to indicate fracture toughness. Two main points can be summarised here: firstly there is no further improvement in terms of fracture toughness for the produced fast curing CRA mixtures in comparison with the control one, which might be due to the high air voids for these mixtures as the crack propagation mechanism is highly affected by air voids content. Secondly, despite the high stiffness of the fast curing CRA mixtures, less fracture toughness was observed for them when compared with traditional HRA mixtures.

Generally, it is obvious that incorporating different types of SCMs collectively improved the mechanical properties of the produced mixtures, as detailed in chapters five and six. These results confirm that these fast curing mixtures overcome most of the properties of bitumen and the novel cementitious binder together such as low permanent deformation, long fatigue life and lower temperature susceptibility of cementitious concrete and higher flexibility and toughness of bituminous asphalt. In the next chapter, the durability of the new mixtures will be investigated and compared with control CRA as well as traditional HRA.

Chapter Seven

Optimised CRA Mixtures: Durability Investigations

Durability of the bituminous mixtures is defined as the ability of the materials to resist the effect of water, aging and temperature variations without significant deterioration for a specific period and for a given amount of traffic loading. Water damage and failure due to age hardening are the main factors influencing the mechanical properties and durability of asphalts (Read and Whiteoak, 2003).

In terms of water damage, moisture can affect the bitumen-aggregate interface bending strength by causing cohesion and stiffness loss of the bitumen and attacking the adhesive bond between the bitumen and the aggregate, which is often called stripping. Accordingly, these two mechanisms decrease the strength of the bituminous layer and the whole pavement structure as well. Suparma (2001) stated that permeable bituminous mixtures are more exposed to stripping; therefore, the mixtures with higher air voids are expected to have a higher risk of stripping.

On the other hand, bitumen aging during mixing, construction stages and whilst the pavement is in service affects the durability of bitumen-aggregate integrity. This is because of the oxidation of the bitumen, which chemically affects its chemical characteristics, and it becomes steadily harder, less flexible and more viscous (Read and Whiteoak, 2003).

Read and Whiteoak also stated that the temperature, time and bitumen film thickness are the main factors that affect the degree of oxidation. Accordingly, age hardening can be classified to short-term and long-term age hardening: the former represents the age hardening during construction process while the latter represents the age hardening whilst the mixtures are in service (Suparma, 2001). Exudation and physical hardening represent other forms of bitumen hardening. The movement of any oily component from the bitumen into the mineral aggregates causes exudation hardening while the latter occurs at ambient temperatures and results from reorientation of molecules and the slow crystallisation of waxes (Suparma, 2001).

This chapter describes the investigations carried out on the durability of the CRA mixtures which are optimised, and their mechanical properties are detailed in the previous chapters. To cover the durability investigations, this chapter will assess the produced mixtures containing the following fillers: OPC, WPSA, BBF, TBF-1 and TBF-2 in terms of water sensitivity and long-term age hardening. A comparison study is conducted for the results of these mixtures with the control CRA and conventional HRA mixtures as well as the requirements of British and European specifications.

7.1 Water sensitivity

Cold BEM design procedures depend on water resistance results as water is considered one of the main damaging reasons that affect the response of these mixtures. To simulate environmental influences in the field in a short time, many procedures of water conditioning of bituminous samples have been recommended in the previous literature (Thanaya, 2007, Lanre, 2010). The curing times of the sample at which water conditioning is carried out for these procedures is different. Generally, cold BEMs are categorised as higher water sensitive mixtures in comparison with hot mixtures.

Specimen curing procedures before conditioning of cold BEMs comprised: i) specimen curing at ambient temperature in or out of their moulds for 2 to 3 days, ii) specimen curing in the oven at different temperatures and durations have been adopted, and iii) specimen vacuum desiccation (Ibrahim, 1998). On the other hand, water conditioning of samples in the laboratory included: i) vacuum saturation with different periods and levels, ii) capillary soaking, and iii) specimen immersion in water at ambient temperature (Thanaya, 2003).

The capillary soaking procedure is adopted from the design procedure introduced by the Ministry of Public Works-Republic of Indonesia (1990). In this procedure, half the thickness of each sample is saturated in water at lab temperature for 24 hours; the sample is then reversed and the other half is saturated for a further 24 hours. Throughout the sample soaking, approximately 15 to 20 mm coarse sand is used as a bed to rest the samples on, to ensure full contact with water. Then, the sample is towel-dried and tested for Marshall Stability at lab temperature to indicate the soaking stability value.

On the other hand, the Asphalt Institute (1989) adopted the vacuum saturation procedure to assess the influence of exposure of the cold BEMs to subsurface water. The equipment comprises a desiccator linked to a vacuum pump. Through the conditioning process, the sample is located in the desiccator and fully submerged with water. Subsequently, the desiccator is evacuated at 100 mm of Hg for 1 hour, and then the sample is soaked in water for another 1 hour. Finally, the sample is removed, towel-dried and tested for its indirect tensile strength.

In this research, the test procedures and guides, which is described in section 4.4.2.1, have been applied as per BS EN 12697-12 to assess the water sensitivity of CRA and HRA mixtures (European Committee for Standardization, 2008). Method A in the standard using Stiffness Modulus Ratio (SMR) was adopted.

7.1.1 Results and discussion

The responses of control CRA mixtures (LD) in terms of water sensitivity investigation with detailed results for the conventional HRA mixtures are shown in Figure 7-1. The figure also shows the SMR, dry ITSM and wet ITSM for CRA mixtures produced by replacing the conventional mineral filler with 6 % OPC. A close inspection of the results indicates that LD mixtures have the poorest SMR in comparison with the two HRA types. This result can be attributed to the low stiffness modulus, which experiences a decrease in its values after conditioning of the samples. Therefore, the results indicate that the control CRA mixtures have low water resistance, and provision must be made to enhance their properties when such mixtures are utilised as a pavement's structural layer.

Therefore, it is clearly shown in the same figure that OPC mixtures can overcome the negative performance of these mixtures, i.e. LD, as the SMR (105 %) increased incredibly to a level which is better than the conventional HRA mixtures (91 % and 94 % for 100/150 and 40/60 HRA, respectively).

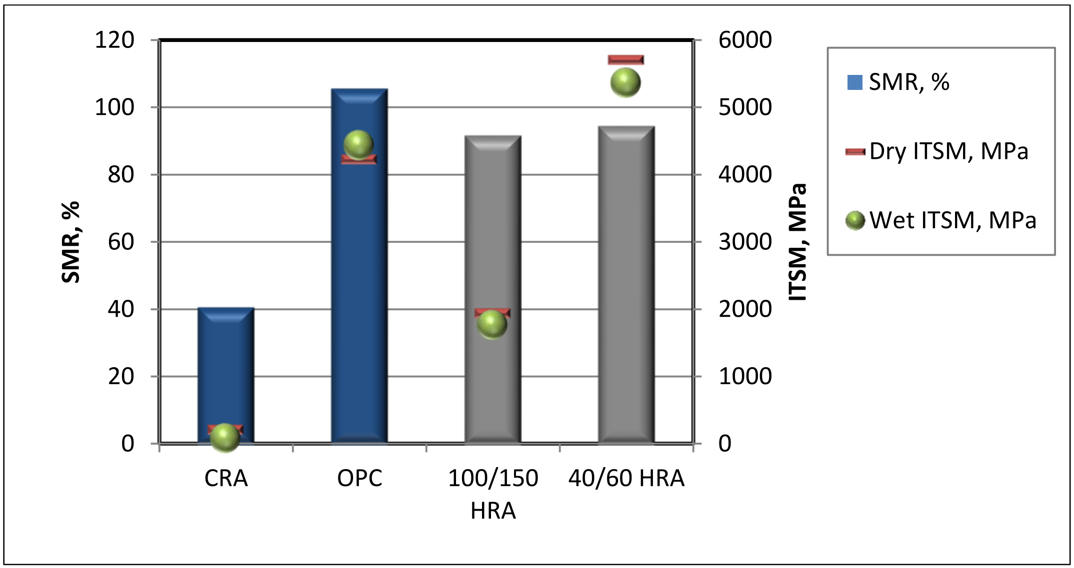


Figure 7-1: Water sensitivity results for control CRA, OPC and conventional HRA mixtures

The performance of CRA mixtures incorporating the optimised cementitious fillers, i.e. WPSA, BBF, TBF-1 and TBF-2, have been investigated and are presented in Figure 7-2 and summarised in Table 7-1. It is obvious that the influence of replacement of conventional mineral filler with WPSA, BBF, TBF-1 and TBF-2 revealed outstanding water resistance compared with CRA, 100/150 HRA and 40/60 HRA.

SMR for WPSA, BBF, TBF-1 and TBF-2 were higher than the control CRA, 100/150 HRA and 40/60 HRA with values more than 100 % because the wet ITSM values were more than dry ITSM. Also, these results are compliant with the required specifications recommended by the British and European standards. This promising behaviour of the conditioning WPSA, BBF, TBF-1 and TBF-2 samples for the water sensitivity test can be attributed to the hydration process of the new cementitious filler in the presence of trapped water. Furthermore, the conditioning of the samples at high temperatures (40 °C) in water further activates this process.

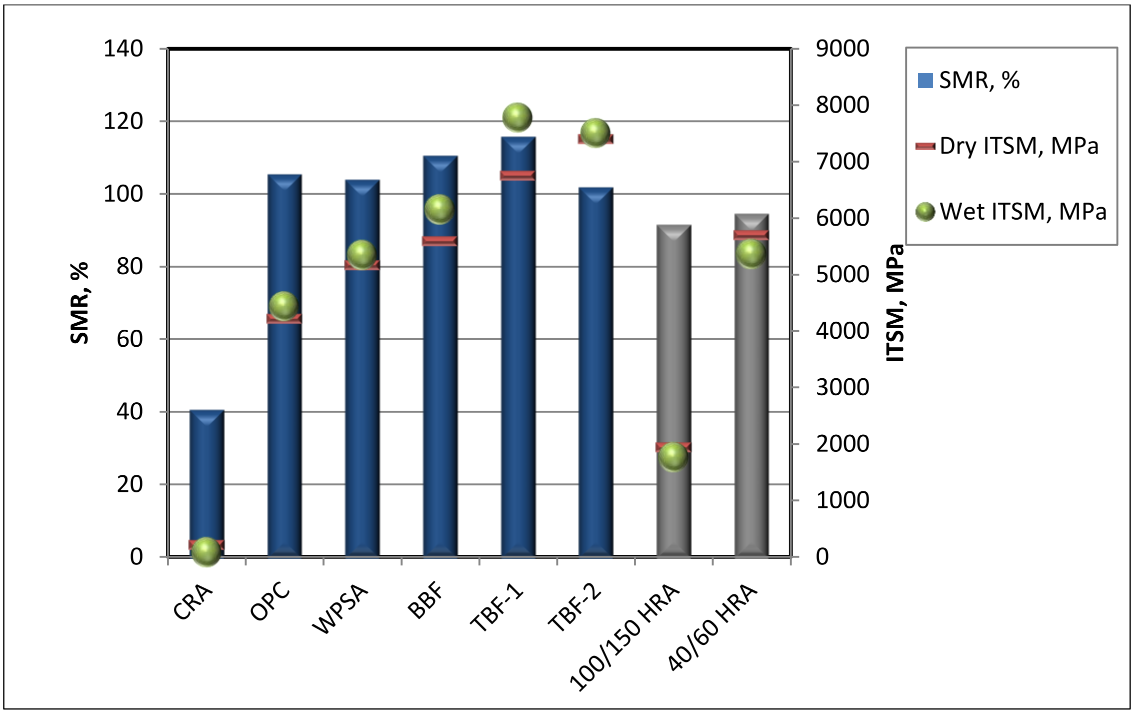


Figure 7-2: Water sensitivity results for all the mixtures

Table 7-1: Dry ITSM, wet ITSM and SMR results of water sensitivity test

Mix type	Dry ITSM, MPa	Wet ITSM, MPa	SMR, %
CRA mixtures			
LD	210	84	40
OPC	3594	3774	105
WPSA	5167	5348	103
BBF	5590	6149	110
TBF-1	6750	7781	115
TBF-2	7400	7498	101
HRA mixtures			
100/150	1941	1766	91
40/60	5705	5363	94

7.2 Long-term age hardening

Age hardening is often investigated for hot bituminous mixtures, while for cold BEMs it is not considered as the main challenge due to its mix preparation and compaction features. The evolution of these mixtures depends on the progress of trapped water evaporation to improve their strength; therefore, their long-term strength is higher than their early strength. As reported earlier, reducing curing time and improving early life strength are the main aims of this research.

Two kinds of age hardening in terms of hot mixtures can be investigated, namely Short Term Oven Ageing (STOA) and Long Term Oven Ageing (LTOA). These ageing types are detailed in section 4.4.2.2 and adopted to investigate LTOA for cold BEM's in accordance with the procedure adopted by the Strategy Highway Research Program (SHRP) A-003A.

7.2.1 Results and discussions

In this research, LTOA has been investigated for both produced CRA and conventional HRA mixtures to assess the performance of the optimised mixtures, i.e. mixtures containing WPSA, BBF, TBF-1 and TBF-2 fillers. All the mixtures are conditioned in an oven at 85 °C for 5 days to simulate the age-hardening effects after 10 years, as indicated earlier. Then the specimens are tested in accordance with BS EN 12697-26 (European Committee for Standardization 2012) to indicate ITSM values after ageing at 20 °C. Figure 7-3 shows the results in terms of: i) Mean Stiffness Modulus Ratio (MSMR), which is the ratio between ITSM after ageing and ITSM before ageing, ii) ITSM values before and after ageing, and iii) compared with the stiffness modulus values for CRA mixtures after 3 days under normal curing process and conventional HRA mixtures values.

Several points can be reported from ageing test investigation, these are: 1) there is a significant enhancement in stiffness modulus ratio for control CRA mixtures as the MSMR is more than 13, 2) generally, all CRA mixtures improved their strength after aging, which was expected and is compliant with the previous studies (Thanaya, 2003), 3) OPC, WPSA, BBF, TBF-1 and TBF-2 mixtures had a stiffness modulus more than the target stiffness modulus, i.e. 2000 MPa, and 4) control CRA mixtures' (LD) stiffness modulus after ageing (1431 MPa) are now comparable with the stiffness modulus for 100/150 HRA (1941 MPa).

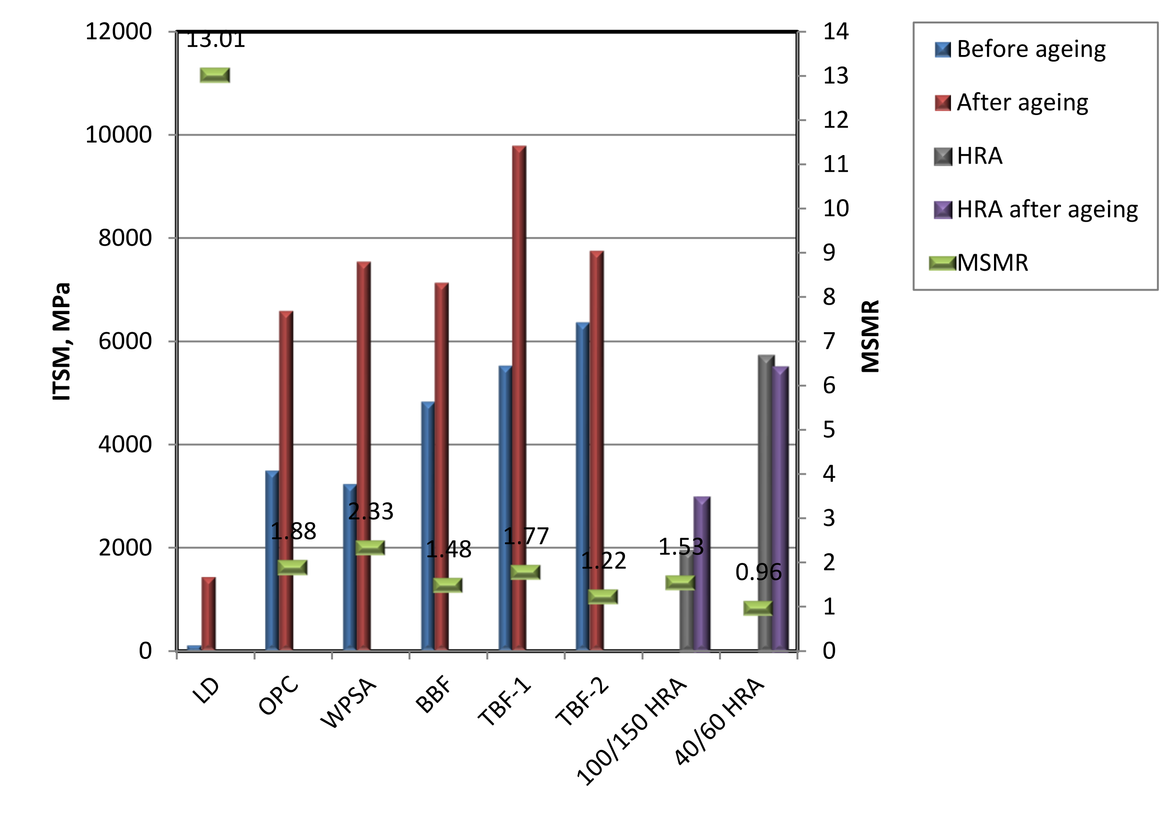


Figure 7-3: Effect of LTOA on CRA and HRA mixtures

7.3 Summary

The promising mechanical properties of the novel CRA mixtures, i.e. WPSA, BBF, TBF-1 and TBF-2, are detailed in chapters five and six by indicating the stiffness modulus, creep performance, fatigue life and fracture toughness.

For all optimised CRA mixtures a considerable improvement in terms of water sensitivity has been observed in comparison with the control CRA and conventional HRA mixtures.

Water sensitivity was specified by indicating stiffness modulus ratio, which is the ratio between wet and dry samples, as the wet samples were exposed to vacuum pressure then conditioned in a water bath at 40 °C for 72 hours before testing. This behaviour of the produced CRA mixtures is attributed to the high wet stiffness modulus values due to conditioning the samples at a high temperature, which further activates the hydration process between the trapped water and the novel cementitious fillers, i.e. WPSA, BBF, TBF-1 and TBF-2. The reasons behind such improvement are detailed in section 8.3 of chapter 8.

Cold BEMs' short-term ageing assessment is not considered for these mixtures as they are prepared at ambient temperature. Long-term ageing was investigated by placing the samples in an oven at 85 °C for 5 days. The results revealed the positive effect of this ageing conditioning for all the CRA mixtures especially the control CRA mixtures as the MSMR was more than 13, which meant that the ageing ITSM value increased by more than 13 times the ITSM results after 3 days of the normal curing process.

It is clearly shown that the performance of the new CRA mixtures with new cementitious fillers in terms of mechanical and durability characteristics is promising and introduces novel CRA mixtures with structural, economic, sustainable and environmentally positive points. The main idea behind this improvement is attributed to producing new cementitious fillers and generating another binder in addition to the bitumen. Therefore, there is a need to indicate the reasons behind gaining strength by means of chemical aspects by conducting Scan Electron Microscopy (SEM) and X-Ray Diffraction (XRD), which will be covered in the next chapter.

Chapter Eight

SEM Observations and XRPD Analysis on CRA Fillers' Microstructure

It is expected that the enhancement of the mechanical properties and durability of CRA mixtures produced by replacing conventional mineral filler with different cementitious fillers, i.e. OPC, WPSA, BBF, TBF-1 and TBF-2, is due to the generation of a new binder (in addition to the primary bituminous binder) from the hydration process between these fillers and the trapped water incorporated in CRA mixtures. Chemical reactions between these cementitious fillers and the trapped water in CRA mixtures change the microstructure of the components of the filler. The material generates a hard mass which develops inter-particle bonding with significant shear strength.

In this chapter, the microstructure of the secondary binder which is generated from the hydration process between TBF-2 and the water has been studied using two approaches: Scanning Electron Microscopy (SEM) and X-Ray Powder Diffraction (XRPD). At the same time, a comparison study with the conventional mineral filler was investigated to indicate the positive points from replacing this filler with TBF-2. The selection of TBF-2 among the other optimised CRA mixtures is due to the high performance of these mixtures in terms of mechanical and durability properties, as reported in the previous chapters, as well as the fact that it is generated from mixing three types of SCM classified as waste materials, i.e. WPSA, PLFA and RHA.

SEM technique is used to magnify the surface of the material for detailed inspecting. It has been utilised extensively in the cement research field for microstructure analysis of hardened products and for the appraisal of the degree of hydration. Also, elemental composition and distinguishion between different phases can be indicated by this technique (Goldstein *et al.*, 2003).

On the other hand, X-Ray Diffraction analysis is the other technique used to track the changes in the material mineralogy as it records the intensity of the material crystals. The appraisal of the XRPD patterns of the filler materials before and after reaction with water can reveal whether crystallisation has taken place in the course of chemical reactions.

8.1 Filler microstructure study programme

The performance of CRA mixtures containing conventional mineral filler and TBF-2 in terms of ITSM under normal curing process (24 hours in the mould then left at 20 ºC in the lab before the designed test age) over four weeks is presented in Figure 8-1. Consequently, using SEM and XRPD techniques during this duration of material life will picture the journey of limestone dust and TBF-2 mastics to create a new binder. The new binder compound is expected to be the main reason for the performance of the materials especially for TBF-2 mixtures, as shown in Figure 8-1. Also, SEM and XRPD analysis from dry powders were used to distinguish the changes at different curing time.

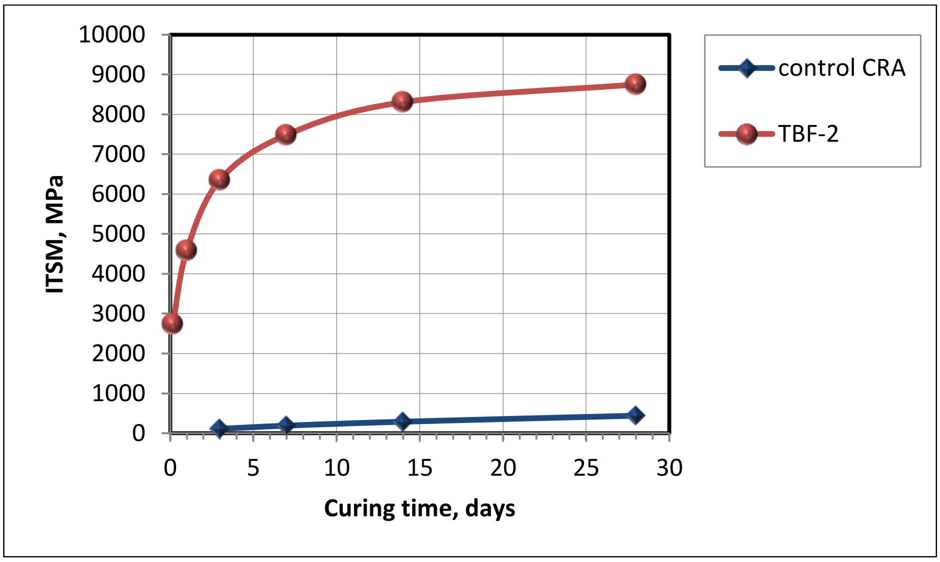


Figure 8-1: ITSM development over 28 days with limestone dust and TBF-2 filler types

8.2 SEM observations

The definition of hydration as a chemical term, in the cement chemistry field, is a reaction of an anhydrous cementitious material or one of its ingredients with water producing a hydrate as a new compound. Chemical and physic-mechanical alterations of the system correspond to the hydration process particularly with regard to setting and hardening (Hewlett, 2004).

Sarkar *et al.* (2001) summarised the shape of the main strength phases that might be generated from the hydration process between a cementitious material and water. They indicated that the calcium silicate hydrate (C–S–H) phase in cementitious paste is performed by a gel structure, while the calcium hydrate, portlandite (C–H) crystals are presented by many different shapes and sizes starting from massive platy crystals with distinctive hexagonal prism microstructure or large thin elongated crystals. On the other hand, the ettringite (Aft) might appear during early hydration as needle-like crystals in vacant spaces, whereas hexagonal platy crystals represent the existence of monosulphate (AFm).

C–S–H was characterised as the most important component that provides binding property and contributes strength. Simultaneously, SEM has been found to be very beneficial for describing the microstructure of the C–S–H phase, as reported by Sarkar *et al.* (2001) and Hewlett (2004).

In this research, SEM observation was performed for the purpose of taking images of the microstructure of the limestone dust and TBF-2 before and after hydration. The idea of this stage was to obtain as clear photographs as possible from the powder and the paste generated from the hydration process of these materials.

Limestone dust and TBF-2 have been mixed individually with the expected trapped water for CRA mixtures, i.e. 8.5 % from the total mass of aggregate, to produce the paste for the secondary binder. Pieces suitable for SEM observation were taken off the paste at the designated ages, i.e. 2, 7 and 28 days, by impact applied with care to produce as many undisturbed pieces as possible. Then the samples were oven-dried, as the sample that is going to be put in the machine should be totally dry. Dry samples were put onto the studs which were placed in a machine utilising a sample holder to coat them with gold. Sample studs were then put in the sample holder in the SEM machine to apply a vacuum to the sample chamber. After this stage, the machine sends some electrons which help in seeing the particulars of the material surface in detail. Coating in gold significantly improves the spread of electrons; therefore better images are obtained.

Figure 8-2 shows the microstructure of limestone dust powder while Figures 8-3, 8-4 and 8-5 show the microstructure of the paste produced from mixing limestone dust with water. It is obvious from Figure 8-2 that limestone dust particles have an un-spherical shape with a dense mass of calcium carbonite crystals. After mixing with water, the morphology of the paste did not show any remarkable change in the microstructure during the curing time, i.e. 2, 7 and 28 days. Accordingly, the slight progress in the stiffness modulus of the control CRA mixtures (shown in Figure 8-1) can be attributed to the evaporation of the trapped water and confirms that limestone dust can be classified as inert filler.

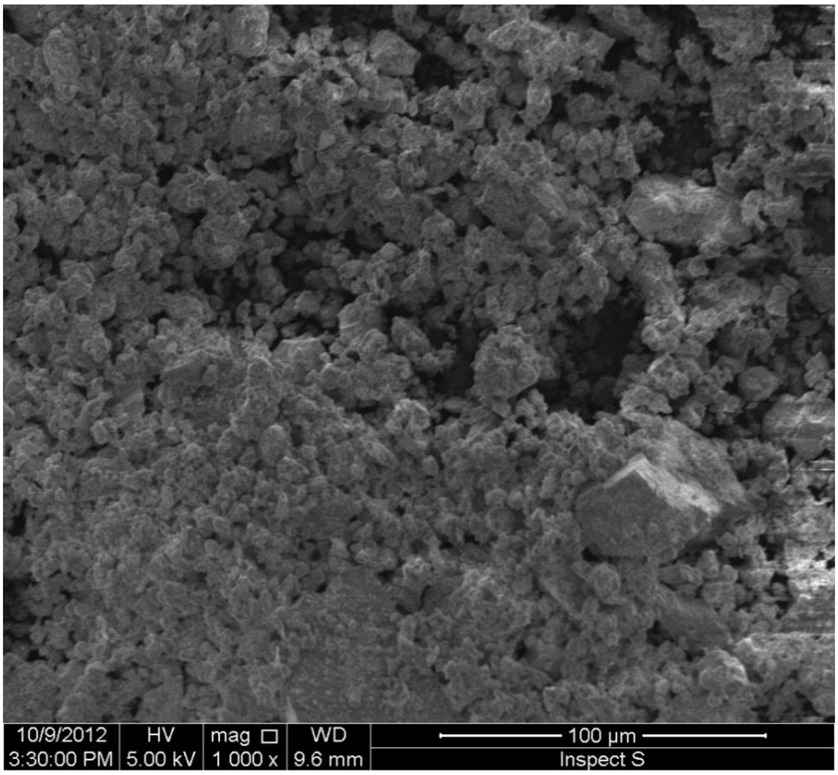


Figure 8-2: Limestone dust particles (1000x)

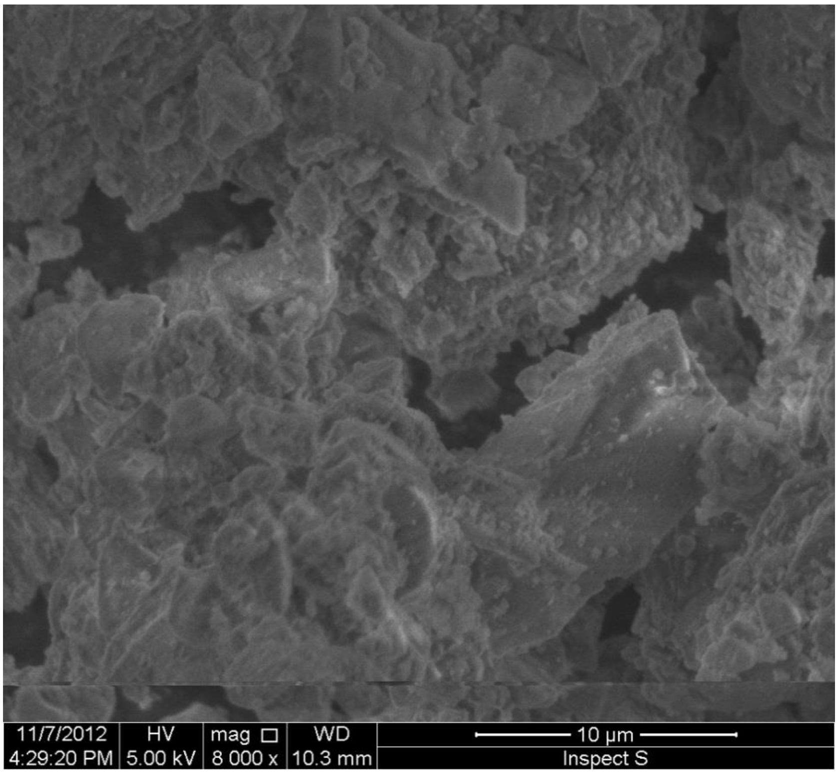


Figure 8-3: 2 days limestone dust paste (8000x)

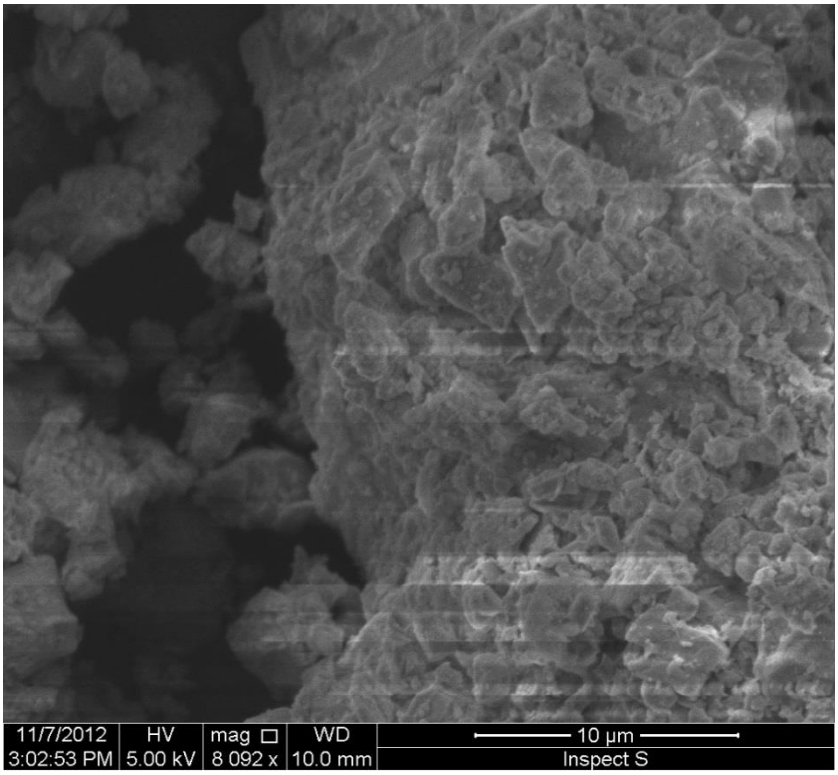


Figure 8-4: 7 days limestone dust paste (8092x)

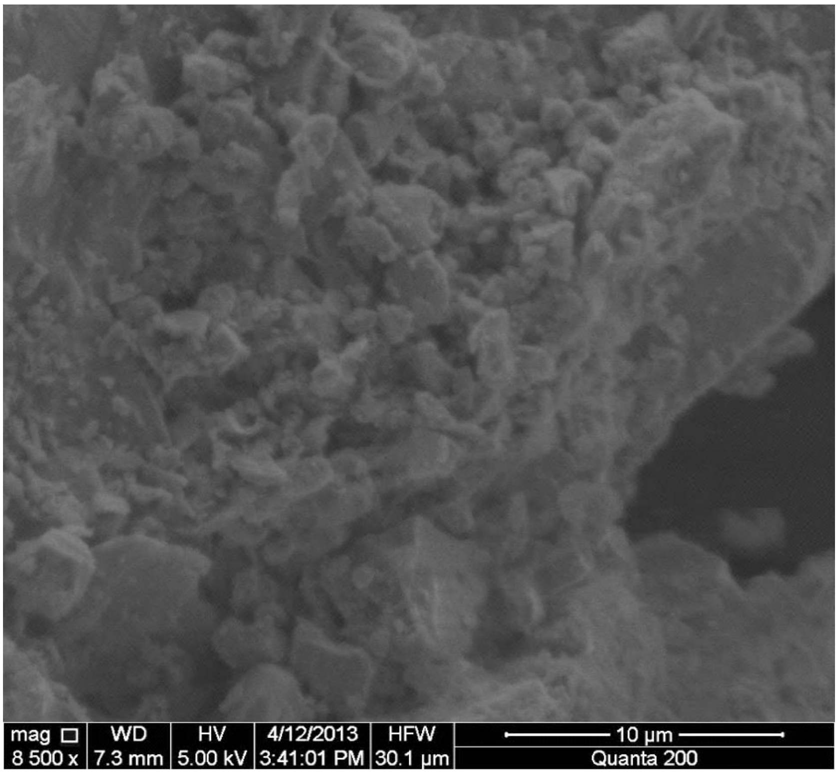


Figure 8-5: 28 days limestone dust paste (8500x)

On the other hand, Figure 8-5 illustrates the microstructure of the TBF-2 powder and Figures 8-6, 8-7 and 8-8 show the microstructures of the fractured faces of the paste produced from mixing TBF-2 with water after 2, 7 and 28 days of curing. The morphology of TBF-2 shown in Figure 8-5 reveals the different characterisations of the blended supplementary cementitious materials, i.e. WPSA, PLFA and RHA, as presented in chapter four, while the SEM observations for the TBF-2 mastics reveal the dense microstructure. It is obviously shown that the density of these mastics increased extensively during curing time especially between 2 and 7 days, which complies with the stiffness modulus increase within the same period as shown in Figure 8-1.

The 2 days image of TBF-2 paste (Figure 8-7) shows the formation of C–S–H gel which is termed as the most important in cement science as it provides binding characteristics and contributes strength. Similar image observations have been reported by Hewlett (2004). The formation of C–S–H gel may be attributed to the hydration of lime and belite components with water.

Figure 8-8 provides a detailed view of the 7 days TBF-2 paste, which demonstrated remarkable progress in the microstructure of this filler type in terms of the density of the structure and appearance of new crystal materials. The new crystals are believed to be portlandite (C–H) as it complies with the microstructure of previous studies such as Hewlett (2004) and Sarkar *et al.* (2001).

Then the progress of the hydration process increased slightly as demonstrated in the microstructure of the TBF-2 paste after 28 days (Figure 8-9). This progress can be attributed to the continuous generation of C–S–H and portlandite minerals from the hydration process.

Generally, the crystalline features in the TBF-2 paste were much more defined than in the limestone dust paste, which generates harder binder. Furthermore, it can be noted that the hydration process of the secondary binder, i.e. TBF-2, is not affected significantly when incorporated in cold bituminous mixtures as the stiffness modulus in Figure 8-1 increased considerably with curing time due to the generation of new binding material besides the bitumen.

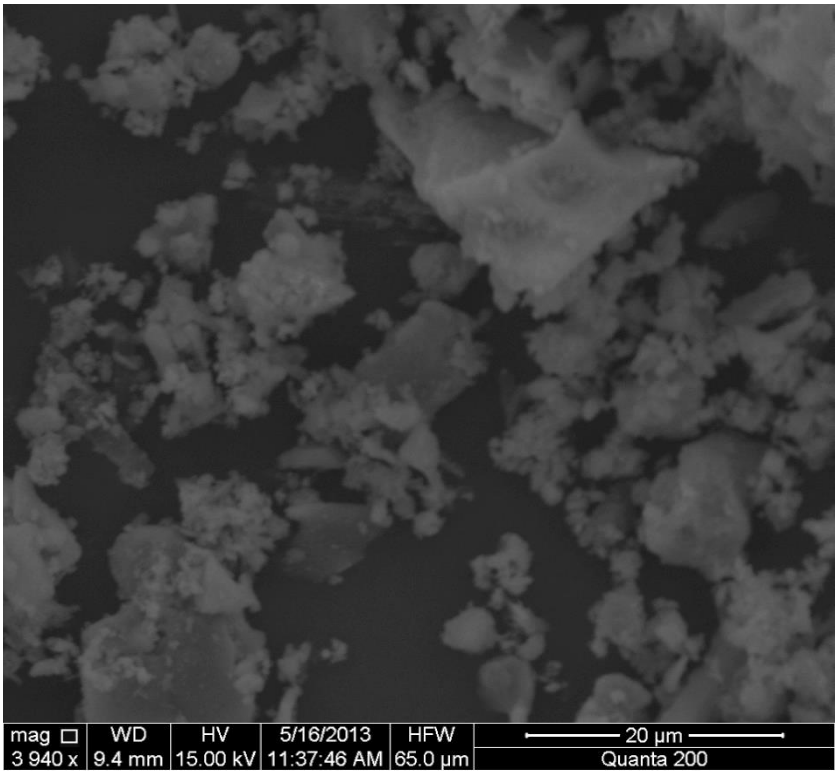


Figure 8-6: TBF-2 particles (3940x)

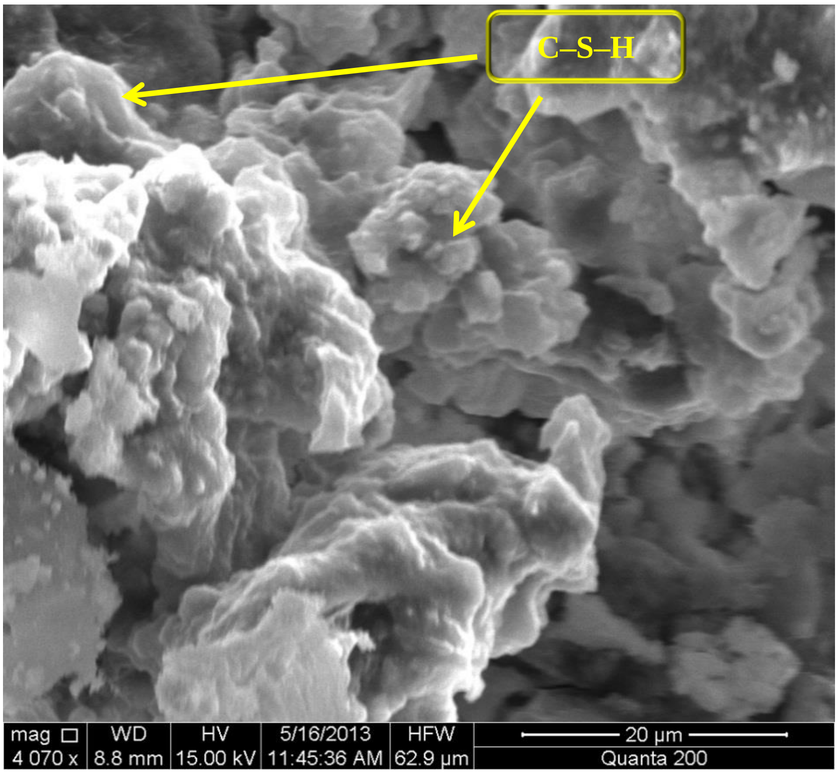


Figure 8-7: 2 days TBF-2 paste (3914x)

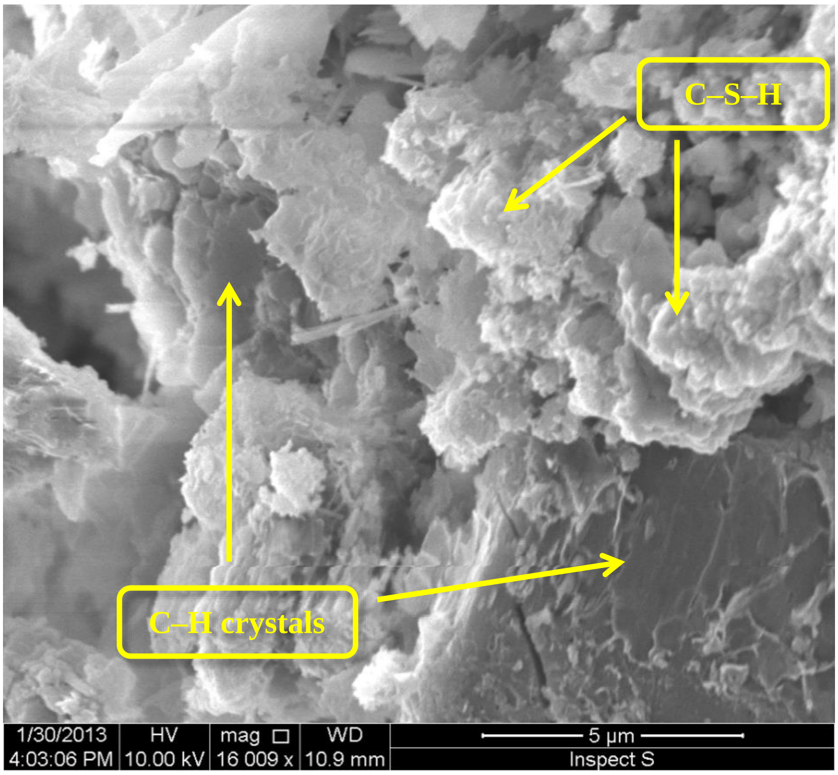


Figure 8-8: 7 days TBF-2 paste (16009x)

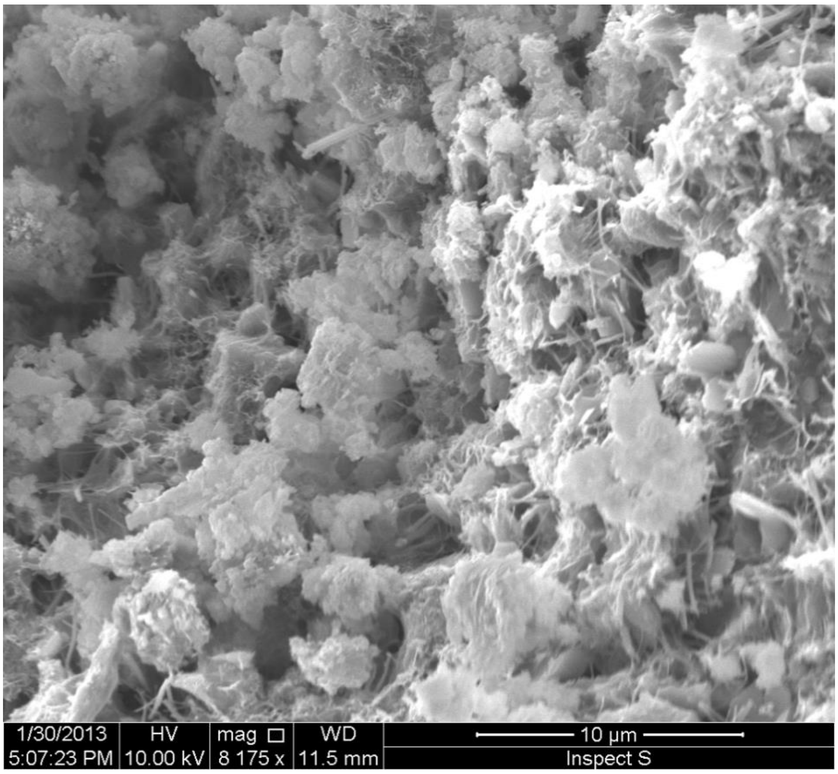


Figure 8-9: 28 days TBF-2 paste (16000x)

8.3 XRPD analysis

Since the invention of X-rays in the 19^{th} century by Wilheim Conrad Roentgen, they have been used for different purposes. Angstrom range wavelengths of X-rays are sufficiently energetic to penetrate solids, and are well poised to probe their internal structure. Based on these characteristics, there are two main techniques extensively used for material evaluations: x-ray fluorescence (XRF) for elemental analysis and x-ray diffraction (XRD) for structural and phase composition studies (Chatterjee, 2001).

The XRD technique has been used extensively to identify the chemical phases of cementitious materials in the cement technology field. In order to specify an unidentified material, the XRD pattern is recorded with the aid of a camera or a diffractometer and a list of *d*-values and the relative intensities of the diffraction lines are prepared. Then, the resultant data are compared with the standard patterns existing for different compounds in a database called Powder Diffraction File (PDF).

Chatterjee (2001) discussed the general features of reaction sequence and hydrates of cement using XRD analysis. He reported that unreacted phases (such as calcite) in different proportions coexist alongside a number of reactants at different ages. Also, the silicate phases (such as belite and alite) lead to the calcium silicate hydrate (C–S–H) and portlandite (P) formation; the former being amorphous or poorly crystalline. Furthermore, he stated that ettringite (AFt) can be generated during early hydration of cement from the reaction of the aluminite and ferrite phases in the presence of gypsum, and the latter is converted to monosulphate (AFm). Furthermore, Esteves (2011) investigated the utilisation of XRD analysis for tracking the consumption of mineral phases and their subsequent reduction of crystal nature during the hydration reaction.

C-S-H may be considered to be gel-like and not necessarily amorphous, although it is believed that it is amorphous. Figure 8-10 shows the diffraction pattern from samples prepared with only C–S–H generated either from C_3S or CaO-SiO_2 hydration, as reported by Nonat (2004).

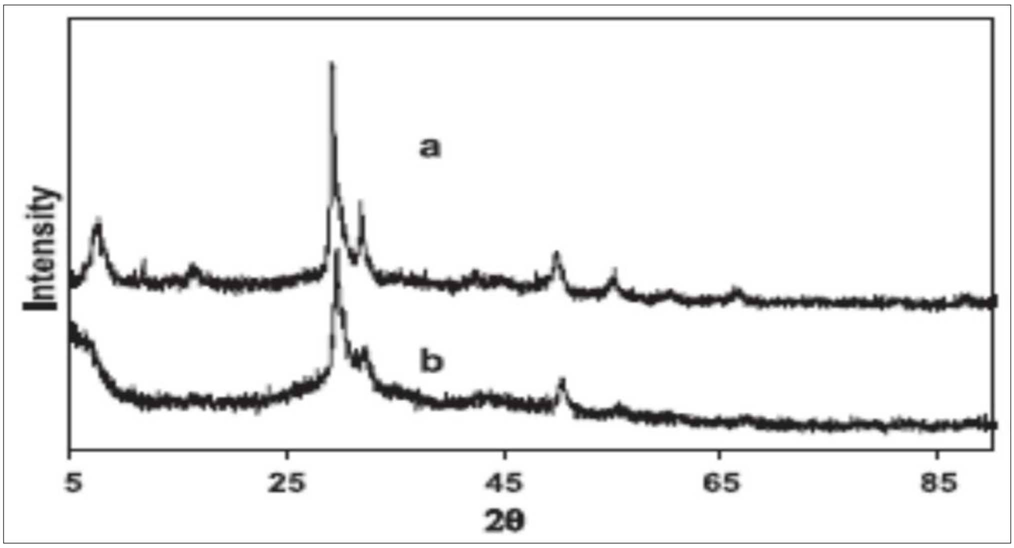


Figure 8-10: XRD patterns of C–S–H (a) mix of CaO and SiO_2 (w/c = 20), (b) hydration of a mix of fine C_3S and SiO_2 in paste (w/c = 1.5) (Nonat, 2004), permission to reproduce this figure has been granted by Elsevier B.V

Tracking the mineral phases of the hydration reaction of a supplementary cementitious material can be grouped into five types: new peak appearance, previous peak disappearance, previous peak expansion, previous peak reduction and unchanged peaks.

In this section, the phase composition of limestone dust and TBF-2 before and after hydration was indicated by X-Ray Powder Diffraction (XRPD), utilising a Rigaku Miniflex diffractometer with CuK X-ray radiation, voltage 30 kV, and current 15 mA at scanning speed of 2.0 deg./min in continuous scan mode with cylindrical aluminium sample holder. To conduct an XRPD analysis on dry powder, the paste of limestone dust and TBF-2 has been prepared as mentioned before in the preparation procedure for SEM paste samples (see section 8.2). Then on the design age, i.e. 2, 7 and 28 days, the paste has been dried and ground to prepare powder suitable for the test. A standard small cylindrical aluminium sample holder was filled with this powder to run the XRPD machine.

In the case of limestone dust XRPD analysis, Figure 8-11 illustrates the XRPD pattern for the limestone dust powder before it reacting with water while Figures 8-12, Figure 8-13 and Figure 8-14 illustrate these patterns for limestone paste after 2, 7 and 28 days, respectively. The main crystalline components of the limestone dust powder, see Figure 8-11, are calcite-C, dolomite-D and quartz-Q. There is no significant change in chemical phases, i.e. calcite, dolomite, and quartz have been observed after 2, 7 and 28 days of hydration reaction. It can be concluded that the mineralogical phases of limestone dust are inert phases therefore there is no reaction with water to produce new chemical phases especially those which introduce higher strength. Moreover, this finding strongly complies with SEM investigation findings in the previous section which indicates, in terms of limestone dust microstructure before and after hydration, that there is no considerable change in the original microstructure. Also, it confirms that the slight progress of CRA stiffness modulus results shown in Figure 8-1 is due to evaporation of the trapped water that is incorporated in these mixtures.

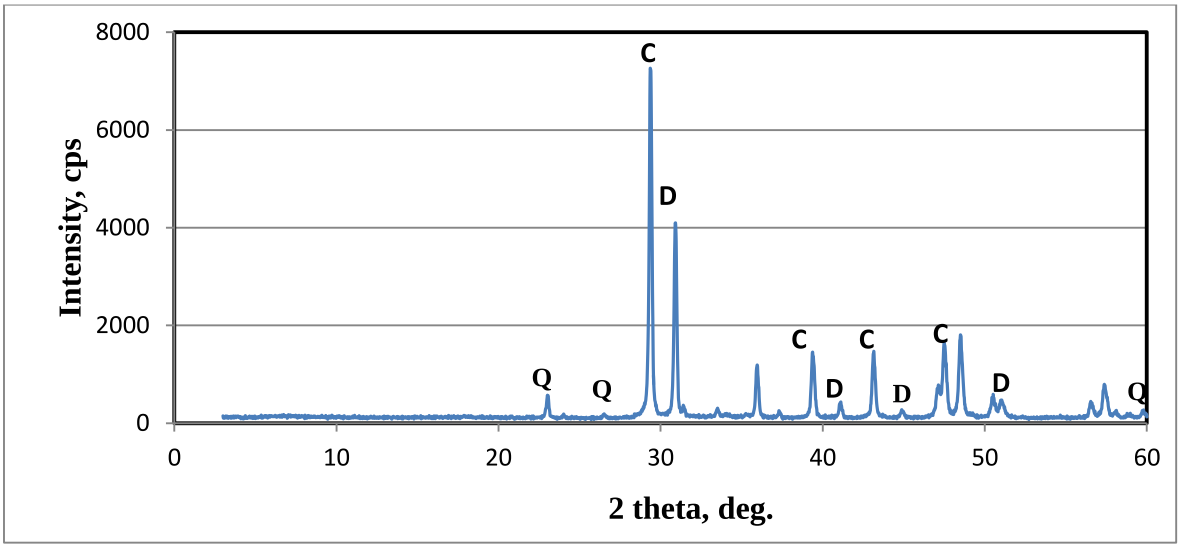


Figure 8-11: XRPD pattern of limestone dust before hydration with peaks corresponding to common minerals highlighted (quartz-Q, calcite-C, and dolomite-D)

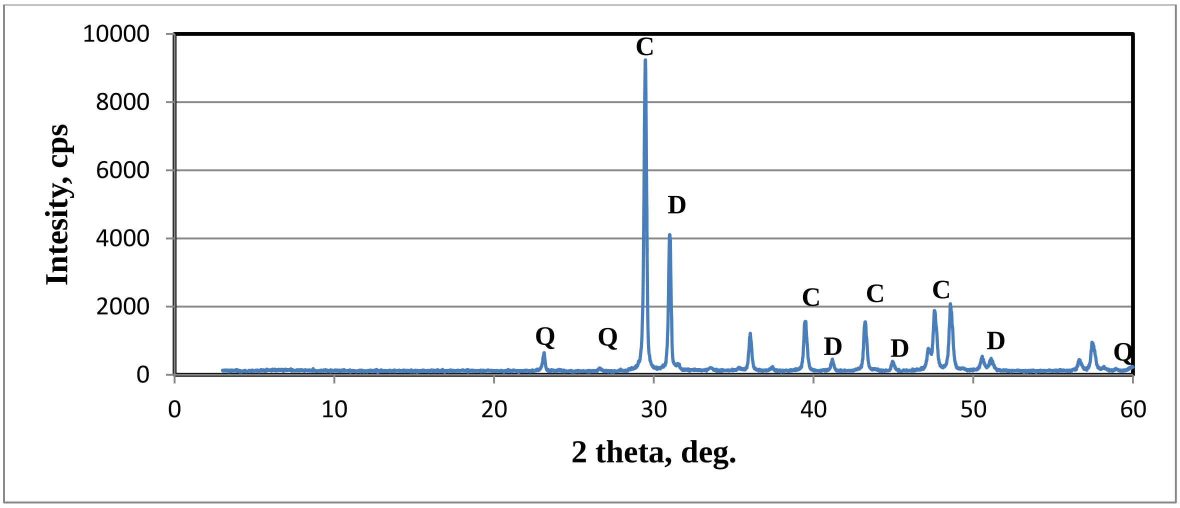


Figure 8-12: XRPD analysis on limestone dust paste at 2 days (quartz-Q, calcite-C, and dolomite-D)

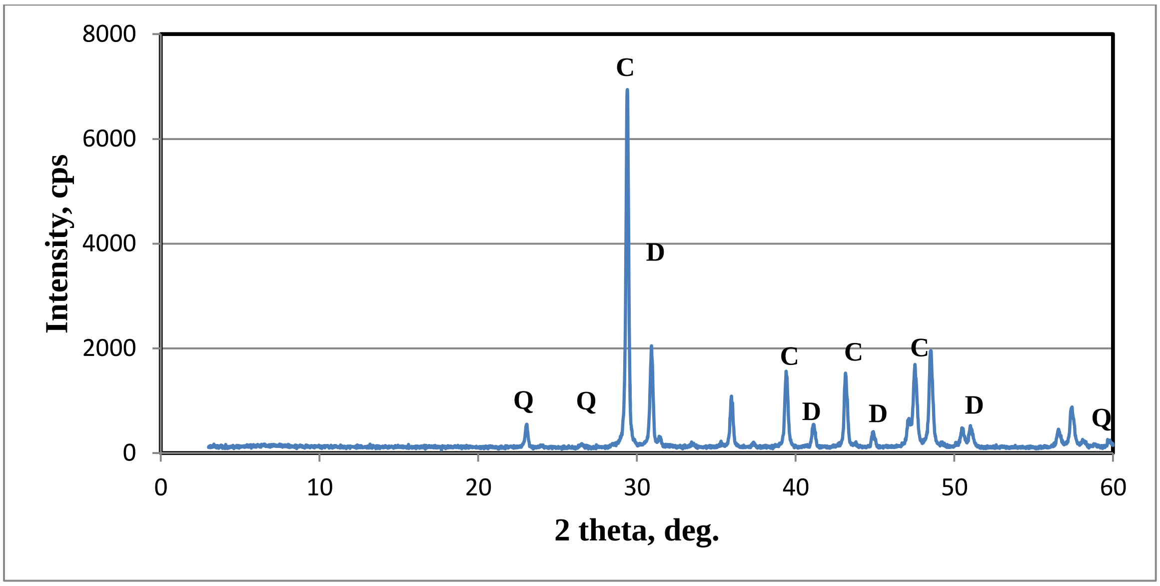


Figure 8-13: XRPD analysis on limestone dust paste at 7 days (quartz-Q, calcite-C, and dolomite-D)

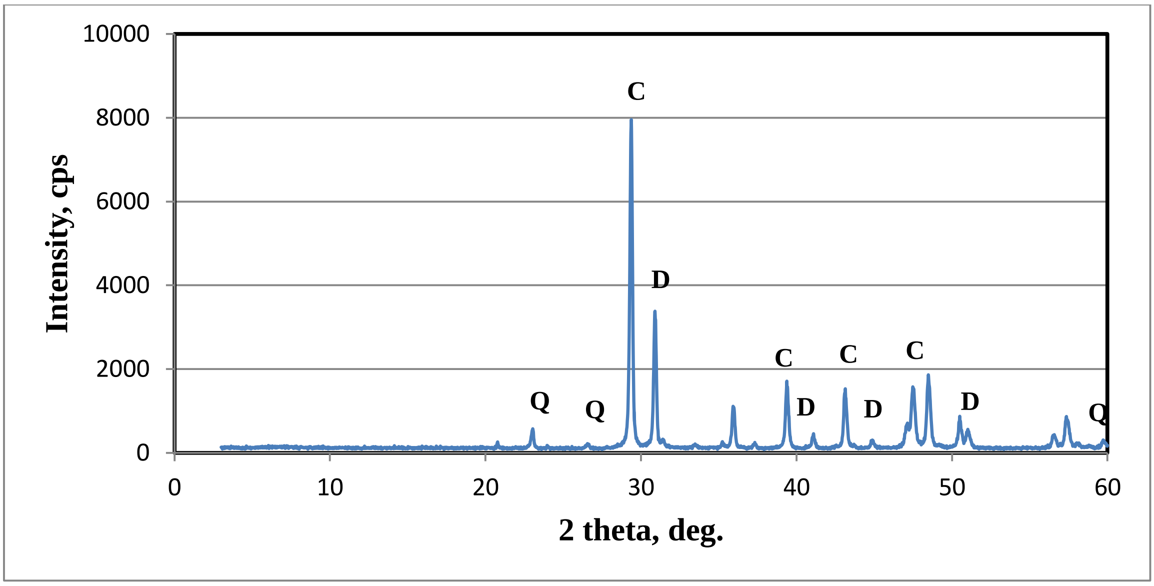


Figure 8-14: XRPD analysis on limestone dust paste at 28 days (quartz-Q, calcite-C, and dolomite-D)

On the other hand, Figure 8-15 demonstrates the mineralogical phases of TBF-2 powder whereas Figures 8-16, 8-17 and 8-18 show these phases of TBF-2 paste after 2, 7 and 28 days. Figure 8-15 reveals that the XRPD pattern of the TBF-2 powder mainly consists of lime-L, calcite-C, belite-B, gehlenite-G and sylvite-S.

Generally, substantial changes have been observed within the TBF-2 paste over time as some of the chemical phases of TBF-2 powder were released at early age or with time, such as lime and belite. At the same time, other mineralogical phases have increasingly appeared, such as C–S–H and portlandite, while, some chemical phases appeared with no significant changes, such as calcite as it is classified as an inert component.

Based on the above observation with the aid of Figures 8-15 to 8-18, it can be concluded that the appearance of C–S–H and portlandite give the strength of the TBF-2 paste which in turn produces a secondary binder, in addition to the bitumen binder, when incorporated in CRA mixtures, as shown in Figure 8-1. More interestingly, the dense microstructure which is observed in the previous section for TBF-2 paste after 2, 7 and 28 days can be attributed to these two components, i.e. C–S–H and portlandite.

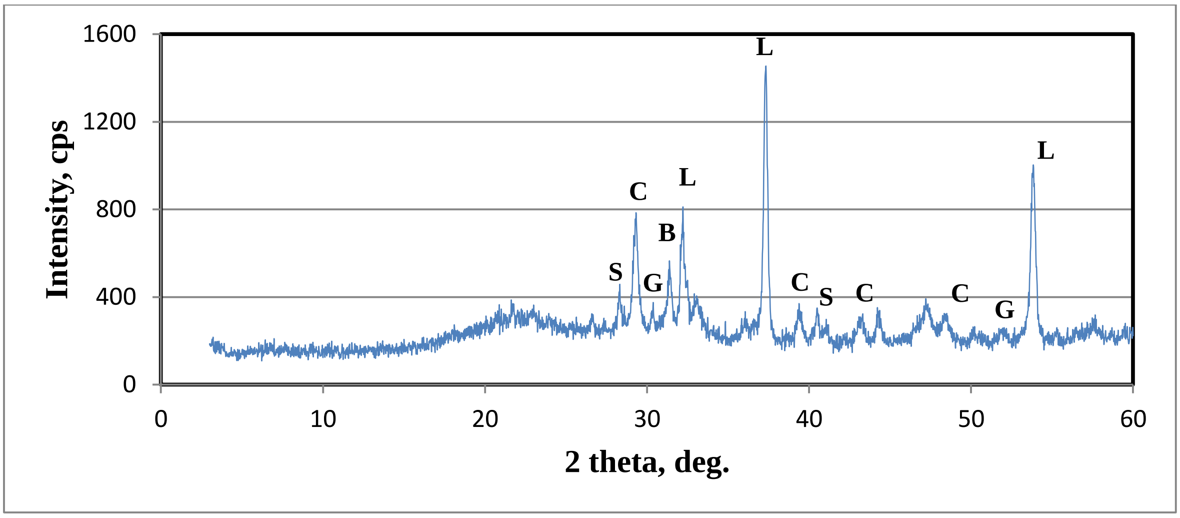


Figure 8-15: XRPD pattern of TBF-2 before hydration with peaks corresponding to common minerals highlighted (lime-L, calcite-C, belite-B, gehlenite-G and sylvite-S)

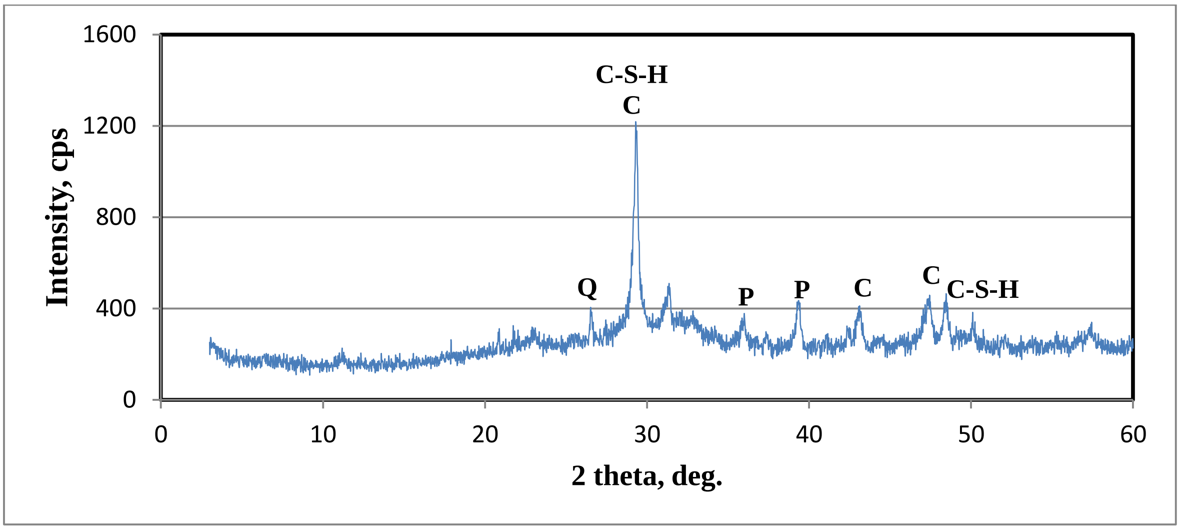


Figure 8-16: XRPD analysis on TBF-2 paste at 2 days (calcium silicate hydrate C-S-H, portlandite-P, calcite-C, and quartz-Q)

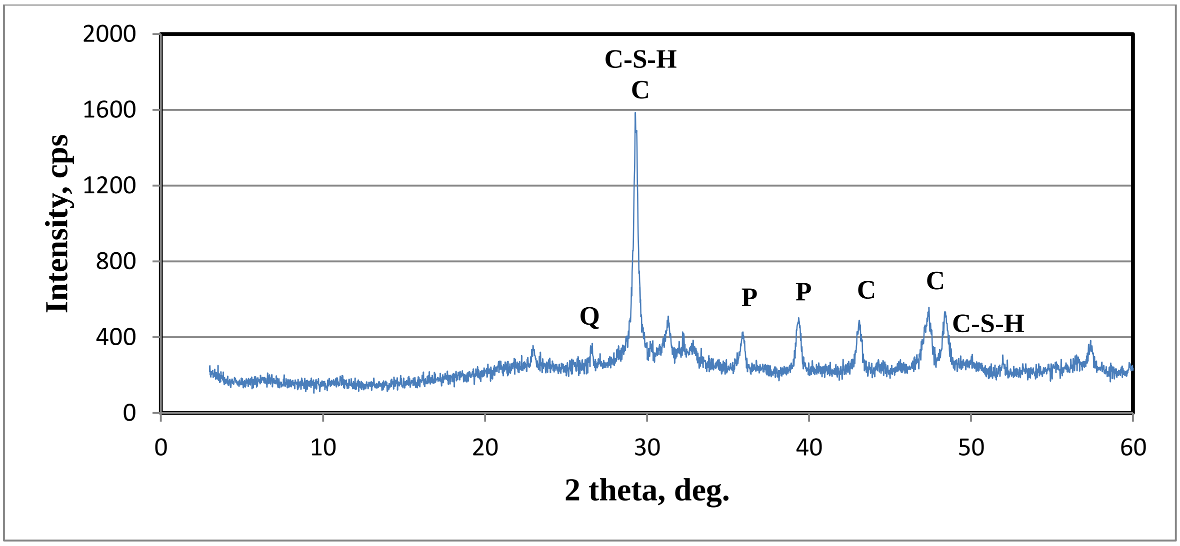


Figure 8-17: XRPD analysis on TBF-2 paste at 7 days (calcium silicate hydrate C-S-H, portlandite-P, calcite-C, and quartz-Q)

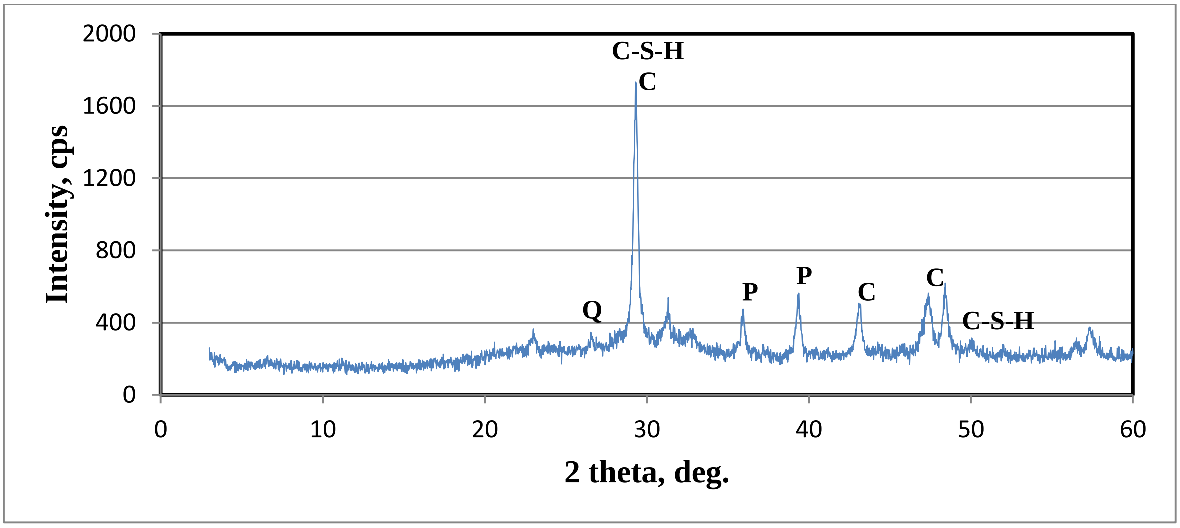


Figure 8-18: XRPD analysis on TBF-2 paste at 28 days (calcium silicate hydrate C–S–H, portlandite-P, calcite-C, and quartz-Q)

8.4 Summary

Following successful experimental work on the performance of the new CRA mixtures in terms of mechanical properties and durability in the previous chapters, reasons for achieving such high stiffness were investigated in this chapter by concentrating on the differences in the morphology and mineralogy of the samples containing limestone dust and TBF-2 at different mixture ages. In order to find out the effective filler components, each filler, i.e. limestone dust and TBF-2, was investigated in terms of SEM to study the morphology before and after hydration, while the mineralogy has been studied by XRD analysis.

The SEM technique was found to clarify the reason why the CRA mixtures containing TBF-2 had gained stiffness modulus with time. It was clearly shown that TBF-2 mastic reveals a dense microstructure which is increased extensively during curing time, especially between 2 and 7 days. This dense microstructure can be attributed to formation of C–S–H gel which in turn represents the main component that is offer strength according to the cement research technology. On the other hand, the microstructure of limestone dust after mixing with water did not show any remarkable change. Also, when comparing the progress of TBF-2 paste improvement with the enhancement of stiffness modulus of CRA mixtures containing TBF-2, it is very clear that TBF-2 is not considerably affected when incorporated in CRA mixtures.

Meanwhile, XRPD analysis was utilised to track the mineralogical phases of the limestone dust and TBF-2 paste. The XRPD patterns show that the most significant change of TBF-2 paste happened during the first 7 days and then there was a further outstanding change with slight enhancement. This enhancement can be attributed to the appearance of C–S–H and portlandite due to the reaction between lime and belite which represent the main components of the TBF-2 filler. Also, calcite, which is classified as an inert component, has appeared in the TBF-2 powder and paste XRPD patterns. On the other hand, there was no remarkable change in XRPD patterns between limestone dust and its paste as all the chemical phases are almost inert components.

Therefore, the XRPD findings strongly comply with those of the SEM observations and together confirm that the stiffness enhancement of CRA mixtures containing TBF-2 was due to generation of a secondary binder, in addition to the bitumen-aggregate-filler adhesion, from the hydration process between the new filler and trapped water incorporated in these mixtures.

Chapter Nine

Application of Microwave Technique on Preparation of HWRA Mixtures

The major problems that have plagued cold BEMs since their introduction are their slow curing rate, low early strength and high air voids. Within the previous chapters, the first and second problems have been improved significantly but the latter, i.e. high air voids content, is still very high in comparison with conventional hot bituminous mixtures. Therefore, this chapter will focus on this property and the attempts that have been applied to decrease the air voids of the produced new mixtures will be explained.

9.1 Influence of air voids content on asphalts' performance

Many studies have been conducted to investigate the influence of air void content on the properties of hot asphalts. Generally, the stiffness modulus of hot asphalts is considerably affected by the increase in air void content (Tayebali *et al.*, 1994). Also, the road engineers limit the air voids contents of the produced hot mixtures to be within the BS EN specifications. Their value also must be not less than 3 % to prevent permanent deformation at early life. Suparma (2001) reported that permeable bituminous mixtures are more exposed to stripping, therefore the mixtures with higher air voids are expected to have higher risk of stripping.

Several studies have been conducted to investigate such relationships between air voids and stiffness modulus for cold BEMs such as Dash (2013), Lanre (2010) and Ibrahim (1998). These studies were focused on the main parameters which are affecting the air voids contents in these mixtures such as aggregate gradation, type and level of compactive effort and curing mechanism and time.

Ibrahim (1998) studied the influence of different emulsion mixture variables on materials' compactibility. Three types of compaction methods – Marshall, vibration and gyratory – have been examined for use in compaction of these mixtures. In terms of compaction method, he prepared a set of samples which were compacted to approximately similar bulk density using Marshall Hammer, vibrating compaction and gyratory compaction and then tested to indicate stiffness modulus at different curing times. He concluded that the compaction method affected the stiffness modulus of cold BEMs significantly as the vibratory compaction showed much higher stiffness modulus than the other compaction methods after being cured due to a higher coalescence of bitumen droplets onto aggregate particles. On the other hand, he stated that stiffness modulus increased considerably with increasing of the compaction effort as the air voids decreased and in turn the interaction between the binder and the aggregate particles increased.

Generally, the target air voids content range for cold BEMs is between 5-10 %, as reported by Thanaya (2007), therefore he recommended a heavy compaction effort for these mixtures to achieve the said target. Further he stated that consideration in the selection of an appropriate compaction method is required to achieve both the volumetric and the engineering characteristics. Accordingly, he stated that increasing the compaction effort can easily lead to reducing the air voids of cold BEMs to comply with a pre-selected target value. In more detail, he reported that, in order to achieve the target air voids, the cold BEMs need to be compacted using 240 gyros, which is designed in his study as extra heavy compaction. Compaction at 80 gyros represented the medium compaction effort which is considered to be corresponding to 50 blows utilising the Marshall Hammer, whereas heavy compaction is carried out at 120 gyros, corresponding to 75 blows.

Also, the Asphalt Institute and Asphalt Emulsion Manufacturers Association (1997) stated that drying or aeration preceding compaction may be required especially for gradations containing considerable fine materials. This process leads to the production of cold BEM with lower air voids, higher density, and higher strength in comparison with cold mixtures compacted at the same mixing water content.

Lanre (2010) reported that mixing and compaction temperature is very significant to produce cold BEMs with low air voids as he observed that cold BEMs prepared at 32 ºC gave better results, lower air voids content and higher stiffness in comparison with those mixtures prepared at 20 ºC.

Recently, Dash (2013) conducted a comparative study between two types of cold BEMs with different gradations, namely dense bituminous concrete and stone mastic bituminous mixtures. One of the studied parameters in this study was to investigate the effect of compaction effort and type which in turn consisted of Marshall Hammer and Gyratory compaction. He found that the latter type of compaction was highly active in decreasing the air voids content despite not indicating much influence on the Marshall Stability.

The volumetric measurements has been determined in accordance on the details described in section 4.4.3.

9.2 Overview of microwave heating

The development of microwave technology primarily began at the time of developing high definition radar during World War II. The potential of microwaves to heat substances was accidentally discovered when conducting random experimentations utilising a microwave generator. Since that time, there has been huge progress in microwave heating in industrial applications and the consumer market as well. The rapid growth of microwave technology in the 1970s was due to its versatility over conventional heating more than its affordability. However, some socio-technical events have been observed questioning about the safety of exposure to microwaves (Osepchuk, 1984).

Kobusheshe (2010) stated the main differences between microwave heating and conventional heating which are shown in Table 9-1.

Table 9-1: Summary of the differences between the microwave and conventional convection heating

	Microwave heating	Conventional convection heating
Source	Energy transfer	Heat transfer via conduction
Start up	Immediate	Related to heating chamber
Rate	Rapid heating possible	Related to thermal diffusivity
Uniformity	Volumetric and selective heating	Temperature gradient from surface
Energy loss	Waveguide to reduce loss	Loss due to radiation externally

The microwave oven has been commonly used in most kitchens in the last 25 years. As shown in the table above, energy savings, cooking time and volumetric heating are the basic advantages over the conventional heating system. Consequently, Thostenson and Chou (1999) reported that microwave heating for processing materials can possibly offer similar advantages, i.e. decrease processing time and energy saving. Meredith (1998) stated that time of heating can often be decreased to 1 % with less than 10 % energy variation in the workload.

Microwaves are a kind of electromagnetic wave between infrared radiation and radio waves, as shown in Figure 9-1, with wavelengths between 0.001 and 1 m and 900 and 2450 MHz frequencies (Meredith, 1998). In terms of efficiency, Meredith (1998) reported that the overall efficiency of a well-designed microwave heating systems is very high, 80 % at 2450 MHz, while conventional heating methods operate at around 30 % efficiency.

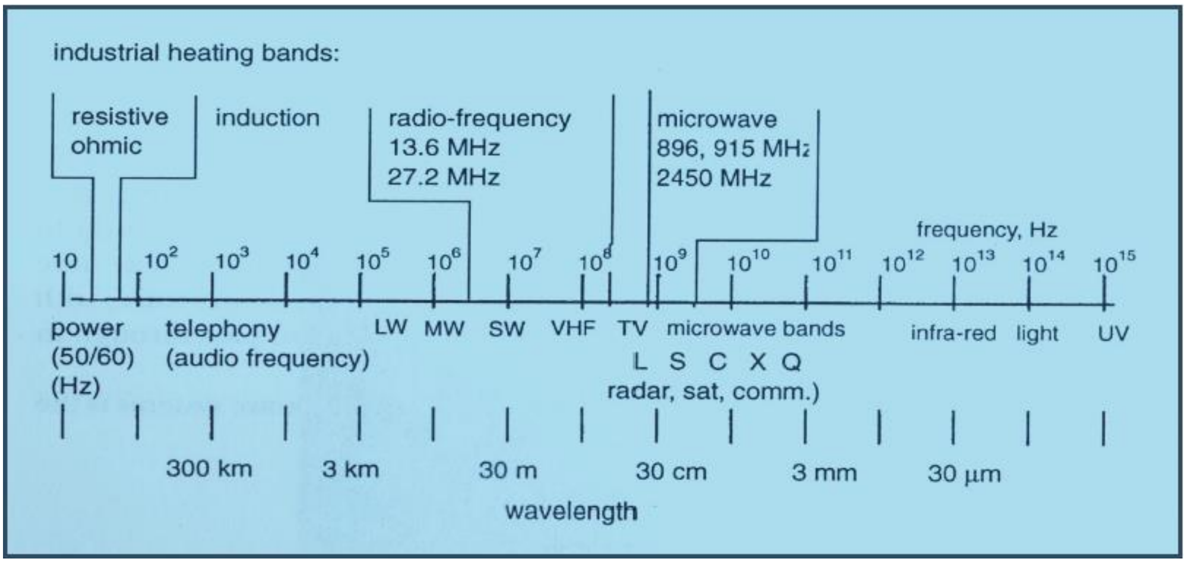


Figure 9-1: Frequency band of microwaves in relation to other electromagnetic waves (Meredith, 1998), permission to reproduce this figure has been granted by © The Institution of Engineering and Technology

Microwaves can penetrate materials and deposit energy throughout the volume of the material as microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field, as reported by Thostenson and Chou (1999). Thus, there is a possibility to achieve fast and uniform heating of whole materials by using microwave energy.

The microwave technology can process mineral materials and carbon-related material, as reported by previous studies (Haque, 1999, Menedez *et al.*, 2010). Also, using the microwave heating for the pre-heating process in the bituminous materials' mixing process prevents the oxidation of bitumen during this process in addition to the previous advantages. Furthermore, Jeon *et al.* (2012) stated that there is a possibility that the microwave can break up the larger molecules in the bitumen and generate light oil fractions.

Jenkins (2000) investigated the possibility of preparing bituminous mixtures at temperatures higher than ambient temperature but below 100 °C, and termed this mix as a Half-Warm Foamed mix (HWF). He stated many benefits that can be achieved when adopting such mixtures, namely improved tensile strength, particle coating and durability, in comparison with BSM-foam, which is produced at ambient temperature. The main HWF advantages in comparison to WMA and HMA are its shear strength and the potential energy-saving element over WMA and HMA, as the extra energy required by the latent heat of water vapour when exceeding its boiling point could be avoided.

Engineering properties of pavement materials are directly related to their density. Eggers *et al.* (1990) reported that, when preparing HWF mixtures at approximately 90 °C, a reduction of air voids of about 2 % has been observed for each 42 °C increase in compaction temperature. Similarly, Jenkins (2000) concluded that air voids reduced while compacting mixtures below 90 °C. The compaction of HWF mixtures at raised temperature, 45 to 90 °C, soon after mixing can reduce air voids by up to 30 %.

Nieftagodien (2013) conducted a study to investigate the suitability of using microwave application to heat-reclaimed bituminous mixtures and crushed aggregates as an energy-efficient technique to produce a Half-Warm Mix (HWM). The advantages when using this method of heating comprise energy saving due to its volumetric heating capability and fast heating, which in turn improves productivity when using suitable materials. The properties of Half-Warm Foamed (HWF) mixtures are a compromise between HMA and Foamed Bitumen Stabilised mixtures' (BSM-foam) properties. He produced a new HWA utilising microwave heating technique to improve the engineering characteristics of BSM-foam. He evaluated the HWF properties and BSM-foam by implementing Indirect Tensile Strength (ITS) and monotonic triaxial tests on specimens as well as by means of pavement analysis. He concluded that the compaction density increased for HWF mixtures and a huge reduction in water contents is observed after curing in comparison with BSM-foam mixtures.

In this thesis, the microwave technique has been used to produce a new Half-Warm Rolled Asphalt (HWRA) in the pre-heating process before compaction of the materials. The next subsection will cover the method of preparation of these mixtures, optimisation of HWRA mixtures, the results and discussion.

9.3 Preparation and properties of Half-Warm Rolled Asphalt

As reviewed in the previous section, there is a promising reduction in air voids of cold bituminous mixtures when compacted at raised temperatures, below 100 °C. On the other hand, using microwave heating instead of the conventional heating procedure has many benefits in terms of energy saving and volumetric heating. However, the most important challenges of using cold BEMs are their low early strength and the long curing time needed to achieve the required strength due to the existence of trapped water in addition to the high air voids content. Therefore, microwave heating is proposed in this study to be applied after mixing CRA materials, i.e. a pre-compacting process to reduce air voids content and to increase the evaporation process of the trapped water.

In this investigation, the domestic microwave apparatus, which is illustrated in Figure 9-2, has been utilised to heat CRA mixtures at the pre-compacted stage. The wavelength frequency was 2450MHz and the output power was set as 300W. The mixing procedure for CRA was the normal procedure, which is detailed in section 4.3.2. After conditioning mixtures in the microwave, the materials were moulded and compacted to indicate their mechanical properties in terms of ITSM, UCCT, 4PB fatigue test and SCB monotonic test and durability by means of SMR. The normal curing process, i.e. leave the sample in the mould for 24 hours and then cure it at 20 °C, has been adopted in this section unless another curing process is mentioned.

Figure 9-2: Domestic-type microwave apparatus

9.4 Optimisation of microwave application

As indicated previously, microwave heating has been applied, with 2450MHz frequency and 300W output power, in different duration times, namely 1, 2, 3, 4, 5 and 6 minutes for TBF-2 mixtures which have very promising properties, as indicated in the previous chapters. Then, the volumetric properties for Marshall size samples and mechanical properties have been indicated in terms of air voids for dry samples and ITSM after 1 day for these mixtures, respectively, to indicate the optimum microwave conditioning time. Also, the temperature of the mixtures has been reported after the mentioned microwave conditioning time. Figure 9-3 shows the air voids and ITSM after 1 day results while Table 9-2 reports the summary of the volumetric properties, ITSM and the temperature which are observed within this investigation. Furthermore, Figure 9-4 displays TBF-2 samples photo within the different conditioning times of microwave application.

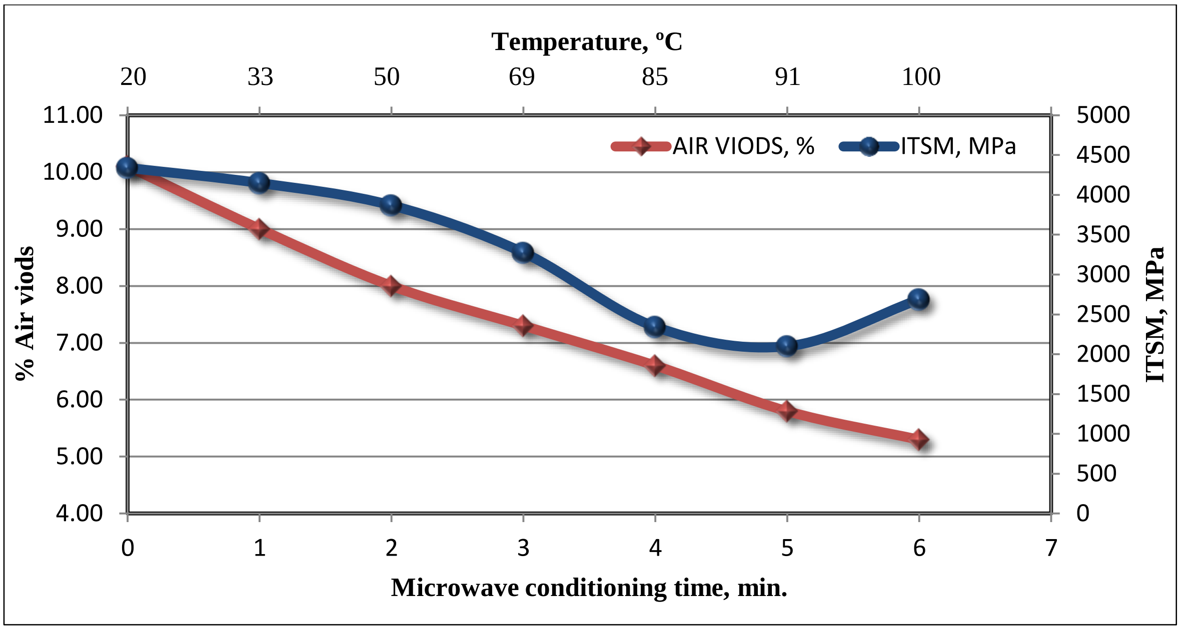


Figure 9-3: Air voids and ITSM results after microwave heating for TBF-2 mixtures

Table 9-2: Summary of volumetric and ITSM results of TBF-2 with different microwave conditioning times

Microwave conditioning time, min.	Compaction temperature, °C	Air voids, %	Dry density, mg/m^3	VMA, %	VFB, %	ITSM after 1 day, MPa
0	20	10.1	2.15	24	58	4340
1	33	9.20	2.18	23	60	4150
2	50	8.00	2.20	22	64	3869
3	69	7.30	2.21	22	66	3275
4	85	6.60	2.23	21	68	2343
5	91	5.80	2.25	20	71	2100
6	100	5.30	2.26	20	73	2688

Without conditioning

2 min. conditioning

3 min. conditioning

6 min. conditioning

Figure 9-4: Samples photos with different microwave conditioning times

From Figure 9-3, it is obvious that air voids decreased, from 10.1 % to 5.3 %, with the increase of microwave conditioning time from 0 to 6 minutes and at the same duration the temperature of the mixes increased from 20 °C to 100 °C. While, stiffness modulus decreased slightly until 3 minutes of microwave conditioning, then the rate of decrease increased sharply between 3 and 4 minutes of microwave curing. Finally, stiffness modulus values increased after 5 minutes of microwave curing when the temperature of the mixtures was close to 100 °C. The air voids decrease can be attributed to the loss of trapped water due to the increment in mixture temperature. Stiffness modulus behaviour is more complicated than that of air voids. But, generally, the early decrease in their values might be due to the negative effect of temperature increase on the progress of the hydration process of the secondary binder, which is generated from the hydration process between the TBF-2 filler and the trapped water; while the later increment of the stiffness modulus values, after 5 minutes of microwave curing, can be attributed to: i) the densification of the mixture which leads to increase the interlock between the mixture particles and ii) the high temperature, which is more than 90 °C, leads to decrease the viscosity of the bitumen (improve the consistency) of the primary binder, i.e. bitumen, and motivated the bonding reaction between the bitumen film and aggregate particles.

An optimisation study can be conducted according to the air voids, stiffness modulus and compaction temperature to indicate the more suitable microwave conditioning time for TBF-2 mixtures. Compaction of TBF-2 mixtures after 3 minutes of microwave conditioning is selected as the best one for several reasons, namely: i) it decreases the air voids from 10.1 % to 7.3 % with a 28 % decrease, ii) the stiffness modulus value is slightly decreased in comparison with the control TBF-2 mixtures, then decreased sharply, and iii) compaction temperature for these mixtures is 69 °C, which represents a very moderate temperature. The new optimised CRA mixtures are termed within this thesis as Half-Warm Rolled Asphalt (HWRA) because they are produced at moderate temperatures.

9.5 Mechanical properties and durability of Half-Warm Rolled Asphalt

The mechanical properties of HWRAs have been investigated by conducting ITSM with different curing time, UCCT, 4PB fatigue test and SCB monotonic test; while SMR has been measured to identify the water sensitivity performance of these mixtures.

9.5.1 ITSM results for Half-Warm Rolled Asphalt

Figure 9-5 shows the influence of curing time at normal curing temperature, i.e. 20 °C, on stiffness modulus results for the novel HWRA mixtures. The ITSM test was conducted at 1/6, 1, 3, 7, 14 and 28 days after de-moulding. Generally, it can be stated that stiffness modulus of HWRA mixtures decreased for all the curing times in comparison with those for TBF-2 mixtures. Interestingly, these results are still higher than OPC mixtures for the same duration of curing and stiffness modulus at 4 hours (2024MPa), are more than those for the 100/150 HRA mixture (1941MPa) and achieves the target stiffness modulus, i.e. 2000MPa.

On the other hand, Figure 9-6 illustrates the stiffness modulus results for the outdoor samples for HWRA, TBF-2 and control CRA mixtures. The procedure of curing the samples and the climatic details were presented earlier, in section 5.5, see Table 5-1 and Figure 5-15. The results show that the stiffness modulus for outdoor HWRA is close to the normal curing HWRA mixtures especially at the early stage of curing, i.e. before 14 days, which is the most important challenge for cold BEMs. Furthermore, their results at 1 day achieve the requirements. The positive performance of the novel HWRA mixtures under real rainfall and climatic conditions can be attributed to the adhesion properties between the bitumen and TBF-2 binder as well as to the increment in the particle interlock due to the decrease in the air voids of those mixtures.

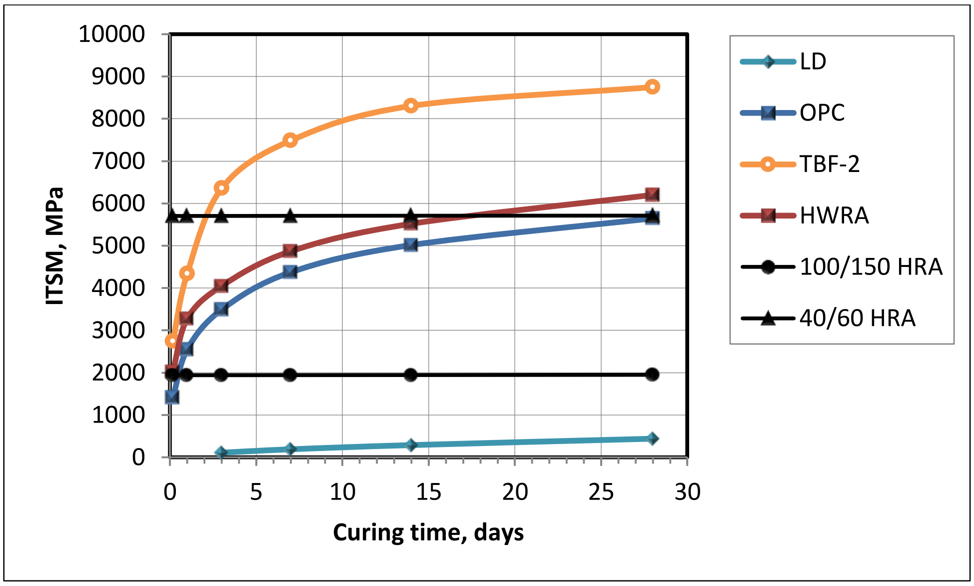


Figure 9-5: Effect of curing time on ITSM of HWRA mixtures

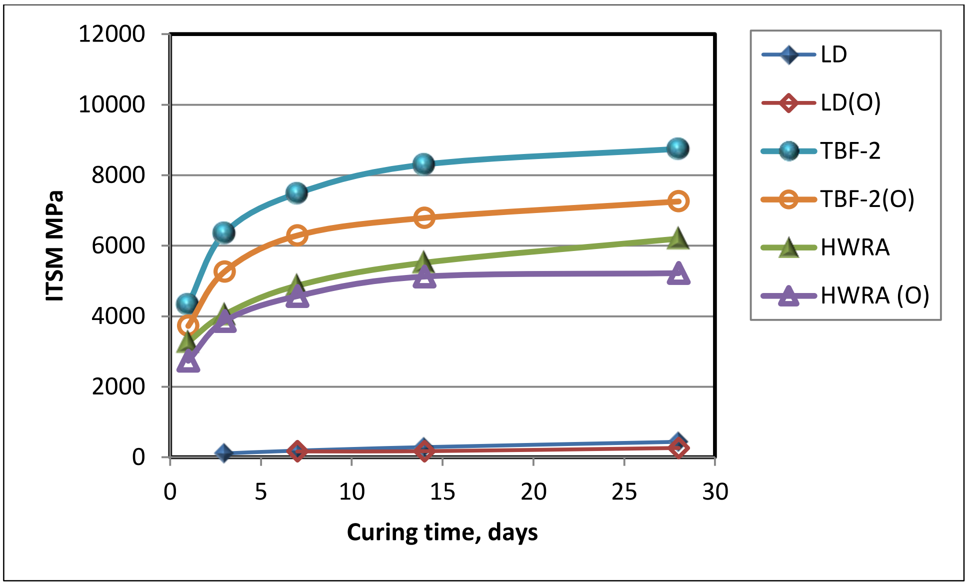


Figure 9-6: Outdoor results of HWRA mixtures

9.5.2 Creep performance of Half-Warm Rolled Asphalt

The Uniaxial Compressive Cyclic Test (UCCT) was conducted at 40 °C in accordance with the European Committee for Standardization (2005) to investigate the creep performance of HWRA mixtures. The samples' preparations and curing has been detailed before, in section 6.1. Figure 9-7 below shows the creep strain vs. number of pulses relationship while Figure 9-8 shows the creep stiffness for HWRA mixtures in comparison with control CRA, TBF-2, and conventional HRA mixtures. It is obvious that HWRA behaves much better than the TBF-2, control CRA (LD) and conventional HRA mixtures. Again, these results identify that the positive effect from increasing particle interlock is more than the negative one due to decrease in the activity of the secondary binder as the trapped water decreased in HWRA mixtures.

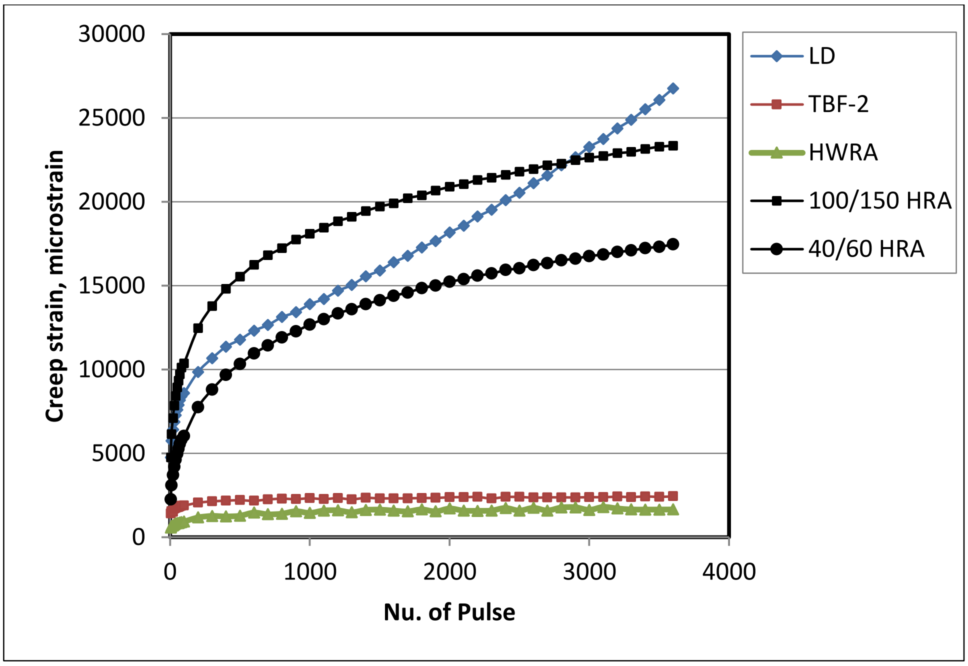


Figure 9-7: Creep strain vs. number of pulses for HWRA mixtures

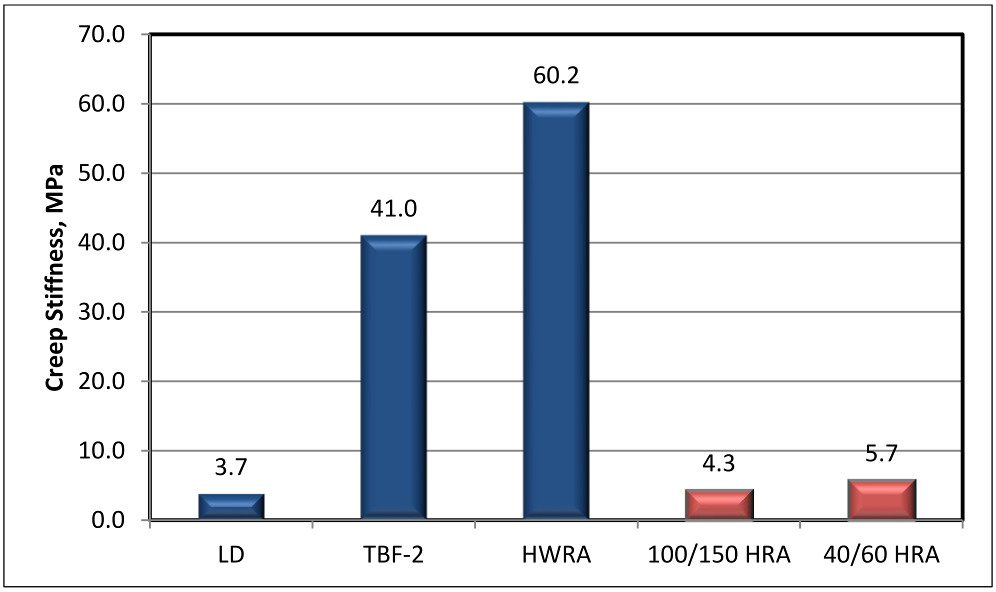


Figure 9-8: Creep stiffness of HWRA mixtures

9.5.3 Fatigue performance of HWRA mixtures

The two stages of cracking, which are crack initiation and crack propagation, have been considered to assess the fatigue performance of the novel HWRA mixtures. As discussed in sections 6.2 and 6.3, fatigue life, which represents the crack initiation stage, has been indicated by conducting the Four-Point Bending test on prismatic shaped specimens (4PB) while Semi-Circular Bending (SCB) test has been used to assess stage two (crack propagation) by indicating the fracture toughness of the produced mixtures.

Figure 9-9 displays the fatigue lives of the novel HWRA mixtures with LD, TBF-2 and conventional HRA mixtures. Interestingly, these mixtures have more fatigue life than all the other mixtures, even TBF-2 mixtures. The fatigue life is (850858), which is more than that for control CRA mixtures (14010) by approximately 25 times.

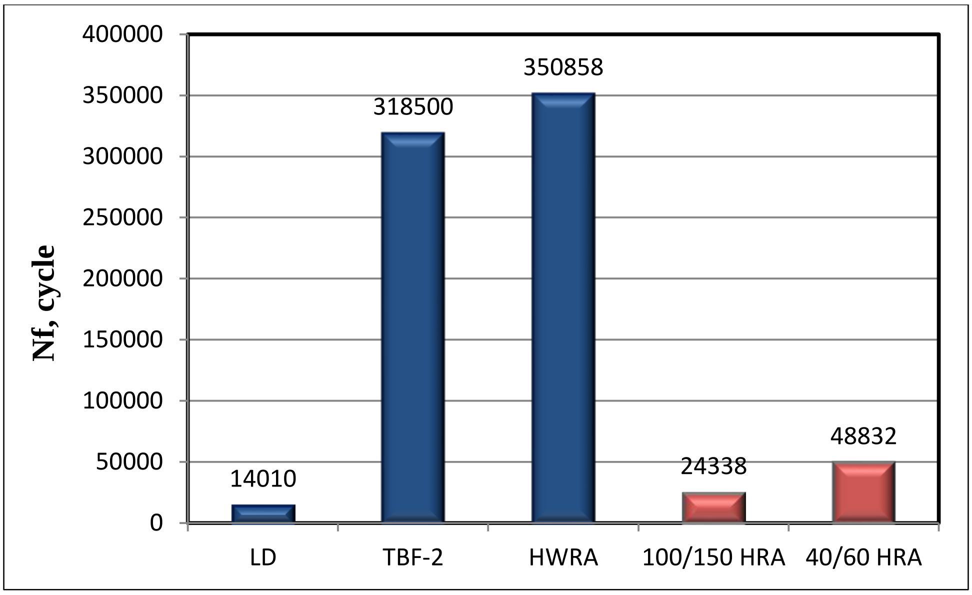


Figure 9-9: Fatigue lives for the LD, TBF-2, HWRA and conventional HRA mixtures

On the other hand, Figure 9-10 shows fracture toughness results for the mentioned mixtures. The fracture toughness of HWRA mixtures is more than control CRA and TBF-2 mixtures together but still less than the results of conventional HRA mixtures. This behaviour of HWRA in comparison with TBF-2 can be attributed to the low air voids content and that the microwave conditioning increased the consistency (decreased the viscosity) of the bitumen which in turn improved the interlock between mixture ingredients. In general, it is believed that the performance of control CRA, TBF-2 and HWRA in comparison with HRA mixtures is due to their higher air voids when compared with HRA mixtures, as the effect of air voids is one of the main parameters influencing crack propagation performance.

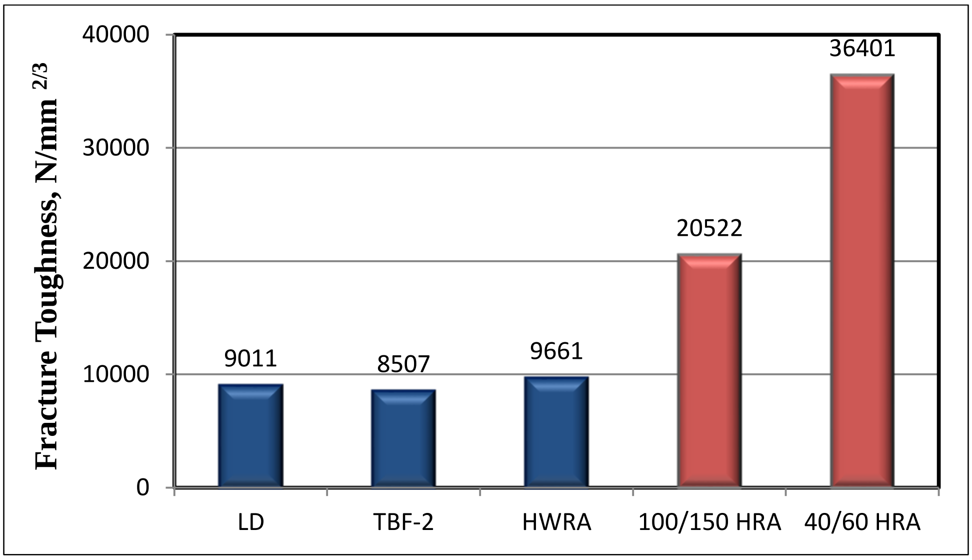


Figure 9-10: Fracture toughness of HWRA mixtures

9.5.4 Durability of HWRA mixtures

Finally, there is a need to investigate the durability of HWRA mixtures, which comprises assessing the long-term aging water sensitivity on the mechanical properties of these mixtures as these are classified as the main factors affecting the durability of bituminous mixtures.

In this study, water sensitivity of all cold, HWRA and HRA mixtures has been investigated as per BS EN 12697-12 to assess the water sensitivity of CRA and HRA mixtures by indicating SMR (European Committee for Standardization, 2008). Figure 9-11 illustrates water sensitivity results of the novel HWRA with the other mixtures, i.e. control CRA, TBF-2, 100/150 HRA and 40/60 HRA mixtures. It is clearly shown that SMR for HWRA mixtures is higher than conventional HRA and complies with BS and European standards. There is a slight reduction in these values in comparison with TBF-2 mixtures, which might be attributed to the loss of some of the trapped water which in turn affected the activity of the secondary binder.

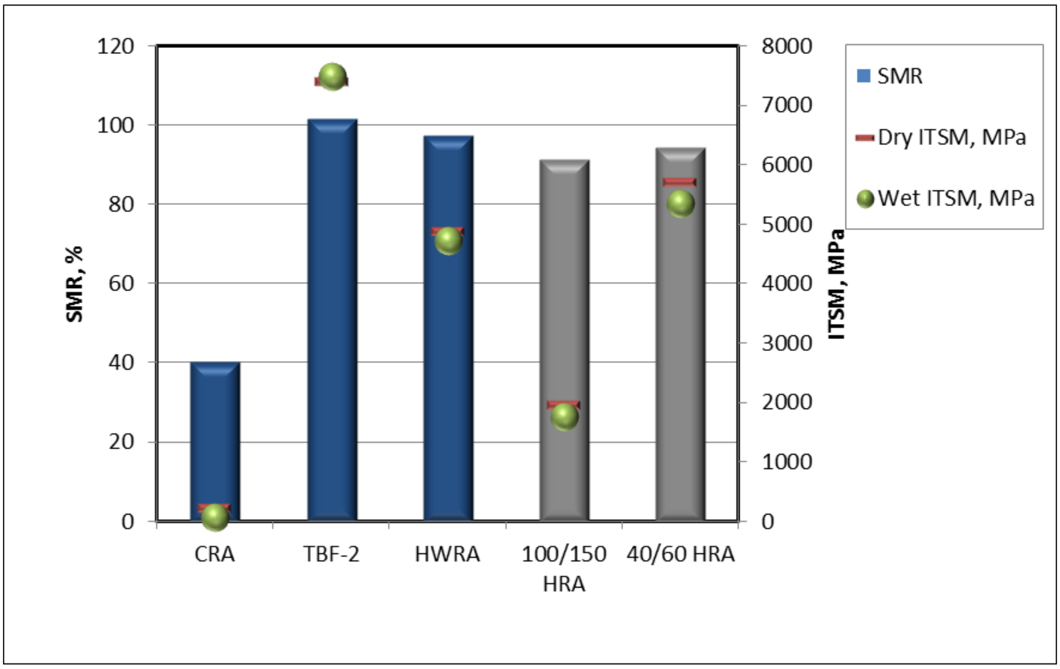


Figure 9-11: Water sensitivity of HWRA mixtures

The long-term aging of the new HWRA mixtures has been assessed by indicating Long Term Oven Aging (LTOA) in accordance with the procedure adopted by Strategy Highway Research Program (SHRP) A-003A and detailed in section 7.2. Therefore, LTOA has been applied for HWRA mixtures and compared with the control mixtures, as shown in Figure 9-12. An interesting point can be reported from these results, as the Mean Stiffness Modulus Ratio (MSMR) for HWRA (1.87) is higher than that for TBF-2 mixtures (1.22), which indicates that the rate of strength progress of HWRA is much better than TBF-2 mixtures.

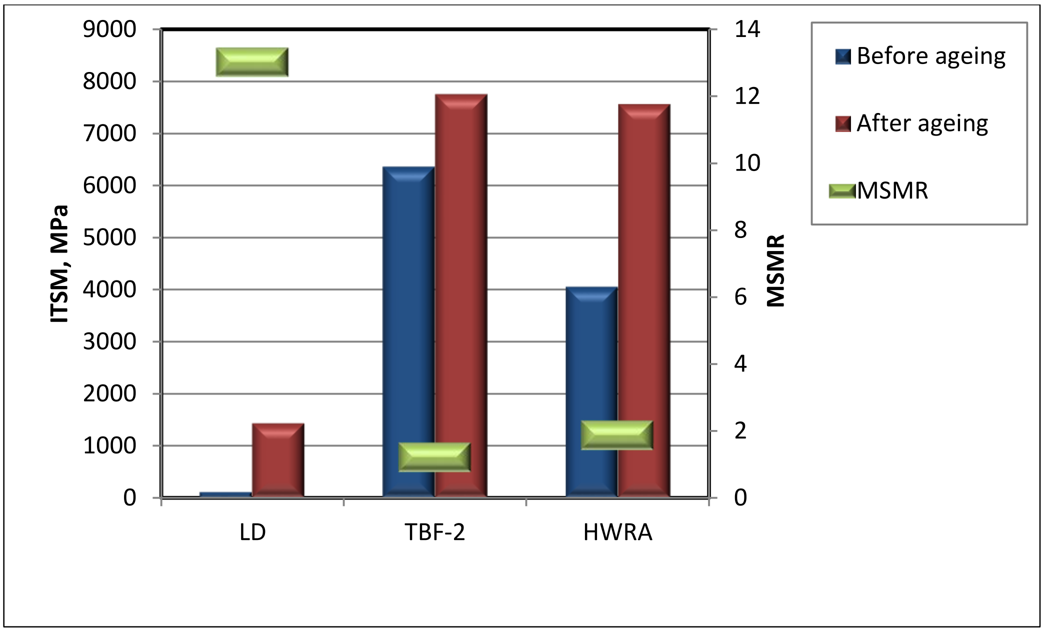


Figure 9-12: Influence of LTOA of HWRA mixtures

9.6 Summary

Promising mechanical properties and durability of the novel CRA mixtures, i.e. WPSA, BBF, TBF-1 and TBF-2, have been adopted from the previous chapters. In terms of volumetric characterisations, the early investigation on air voids of these mixtures revealed high air voids contents, more than 10%. Air voids content has been named as one of the cold BEMs' challenges by previous researchers. Accordingly, a laboratory application has been conducted, and presented in this chapter, to enhance the performance of these mixtures in terms of air void contents whilst maintaining the mechanical properties and durability in the acceptable range.

Bituminous mixtures can be prepared at temperatures higher than ambient temperature but below the boiling temperature, i.e. <100 °C. Jenkins (2000) was successful in the preparation of Half-Warm Foamed mix (HWF) by using a conventional heating process while Nieftagodien (2013) produced a Half-Warm Mix (HWM) utilising microwave application to heat-reclaimed bituminous mixtures and crushed aggregate. Microwave heating has several advantages in comparison with the conventional heating method such as: it is volumetric heating, which represents an energy-saving point, and rapid curing, which might enhance the productivity. Therefore, microwave heating is used to produce a novel Half-Warm Rolled Asphalt by means of a pre-compacting microwave heating method.

Consequently, CRA mixtures containing TBF-2 have been mixed and then microwave heating, with 2450MHz frequency and 300W power, was applied in different durations, i.e. 1, 2, 3, 4, 5, and 6 minutes (the resultant temperature was 33, 50, 69, 85, 91 and 100 ºC, respectively) in the pre-compaction method. The optimum microwave conditioning time was indicated in terms of stiffness modulus, air voids content and temperature. Accordingly, 3 minutes microwave conditioning was chosen as the air voids decreased from 10.1 % to 7.3 %, the stiffness modulus decreased slightly and within acceptable range and, finally, the temperature was in the moderate range (69 °C). Also, at this temperature the consistency of the bitumen is improved by decreasing its viscosity in comparison with 20 °C, which in turn increases the bonding between mixture ingredients. The produced novel mixtures are called Half-Warm Rolled Asphalt (HWRA).

The mechanical properties of HWRA have been investigated in terms of stiffness modulus, creep performance, fatigue life and fatigue fracture while water sensitivity and Long Term Oven Aging (LTOA) were conducted to investigate their durability. Generally, their performance in terms of mechanical properties and durability is promising due to high early stiffness modulus, long fatigue life and high SMR in comparison with control CRA, OPC and conventional HRA mixtures. Finally, the high performance of HWRA mixtures can be attributed to the increase of interlock between the aggregate particles due to the decrease in air voids and, in addition to the activity of the secondary binder, which is generated from the hydration process between TBF-2 and trapped water.

Chapter Ten

Conclusions and Recommendations for Further Works

10.1 Conclusions

Environmentally friendly, economic and sustainable Cold Rolled Asphalts (CRAs) have been satisfactorily produced at Liverpool John Moores University (LJMU) lab using different Supplementary Cementitious Materials (SCMs) individually and/or collectively as a replacement for the conventional mineral filler (limestone dust). The gradation of these mixtures is the same as those mixtures traditionally used to produce Hot Rolled Asphalt (HRA), which is suitable for surface course heavily trafficked pavements.

The current study was focused on overcoming the main concerns regarding cold BEMs, which are the long curing time required to achieve the maximum performance, the inferior early life strength and high air voids content.

In this study, the utilisation of different types of testing and curing and conditioning methods to characterise the mechanical properties and durability of the produced CRA mixtures has been accomplished. Indirect Tensile Stiffness Modulus (ITSM), Uniaxial Compression Cyclic Test (UCCT), Four-Point Bending test on prismatic shaped specimens (4PB) and Semi-Circular Bending monotonic test (SCB) were used to assess the mechanical properties of these mixtures while Stiffness Modulus Ratio (SMR) and Long Term Oven Aging (LTOA) were used to investigate the main durability features, i.e. water sensitivity and long-term aging, respectively. Furthermore, Scan Electron Microscopy (SEM) technique and X-Ray Diffraction (XRD) analysis have been used to investigate the reasons behind the improvement in the mechanical properties of the novel mixtures.

Finally, microwave heating has been applied in the post-mixing system to improve the volumetric properties of the produced CRA mixtures.

A summary of the main conclusions for the research work presented in this thesis is listed as follows:

1. In this study, by means of stiffness modulus results after 3 days, different SCMs have been incorporated and optimised to produce Unary Filler (UF), Binary Blended Filler (BBF) and two Ternary Blended Filler (TBF-1 and TBF-2). UF comprises Waste Paper Sludge Ash (WPSA), BBF comprises a blend of WPSA and Poultry Litter Fly Ash (PLFA) (4.5 %WPSA:1.5 %PLFA), TBF-1 comprises a blend of WPSA, PLFA and Silica Fume (SF) (3.75%WPSA:1.25%PLFA:1%SF), and the last one is TBF-2, which comprises a blend of WPSA, PLFA and Rice Husk Ash (RHA) (3.375%WPSA:1.125%PLFA:1.5%RHA).
2. The cementitious reaction of WPSA due to its mineralogical properties, existence of lime and gelinite, contributes to the appearance of a secondary binder from the hydration process with trapped water incorporated in CRA mixtures, while the presence of alkali components in PLFA act as a potential SCM activates WPSA to generate the BBF. Moreover, incorporation of amorphous silica in SF or RHA form further activates the hydration process of the BBF to produce TBF-1 and TBF-2, respectively.
3. Stiffness modulus of CRA mixtures increases significantly by replacing the conventional mineral filler with WPSA, BBF, TBF-1 and TBF-2 especially in the early curing time, less than 7 days, which is the main disadvantage of the cold BEMs. The target stiffness modulus, which is the ITSM for 100/150 HRA (approximately 2000MPa), was achieved after 1 day of de-moulding for WPSA mixtures and 4 hours for BBF, TBF-1 and TBF-2 mixtures under normal curing method (24 hours in the

mould then leave the samples at 20 °C for the designed age). This means that a road laid with these materials can be opened less than 1 day after application, not 2–24 months, as previously reported for the traditional cold mixtures.

4. The performance of CRA with WPSA, BBF, TBF-1 and TBF-2, in terms of ITSM, is comparable to or much better than those with 6 % OPC, which represents a very promising point as all the materials are waste or by-product material.
5. Temperature susceptibility results reveal a considerably lower thermal sensitivity for OPC, WPSA, BBF, TBF-1 and TBF-2 mixtures in comparison to the conventional HRA mixtures. Accordingly, it can be estimated that the novel CRA mixtures suffer less rutting during hot weather and fracture at low temperature, when laid in a road pavement, in comparison with 100/150 and 40/60 HRA.
6. Conditioning of the optimised CRA mixtures at high temperatures instead of normal temperature considerably increases the stiffness modulus values, which provides us with an attractive choice, especially in slightly hot regions and/or seasons.
7. The replacement of conventional mineral filler with WPSA, BBF, TBF-1 and TBF-2 can also improve incredibly the permanent deformation resistance when compared with control CRA, OPC and traditional HRA mixtures. The creep stiffness increased more than 7, 12, 10 and 12 times more than control CRA for WPSA, BBF, TBF-1 and TBF-2, respectively.
8. It was shown experimentally that the utilisation of WPSA, BBF, TBF-1 and TBF-2 as a replacement for the limestone dust can dramatically increase fatigue life, which is related to the crack initiation time, for the produced mixtures after using the full curing method. Also, fatigue life for TBF-2 mixtures is very high when compared with the control CRA mixtures for a wide range of controlled tensile strain values; while, in terms of crack propagation assessment, by means of indicating fracture toughness, there is no further improvement when utilising novel CRA instead of control mixtures.

9. The water sensitivity of CRA mixtures can be improved significantly by incorporating the novel filler materials, i.e. WPSA, BBF, TBF-1 and TBF-2; their water sensitivity, by means of SMR, are approximately 3 times those for untreated CRA mixtures. Furthermore, there is a significant improvement for these mixtures, as the stiffness modulus for the conditioned sample is higher than the unconditioned sample and therefore the SMR becomes more than 100 %.
10. A positive effect can be expected when CRA mixtures are exposed to long-term aging conditioning for all the mixtures. In this line of research, control CRA revealed the highest stiffness modulus percentage increment among the other mixtures, which increased at a lesser ratio.
11. From a comparative study between limestone dust and TBF-2 morphology before and after being treated with water, the reasons for the behaviour of the mixtures manufactured with these fillers are reported. By utilising SEM, TBF-2 mastic reveals a dense microstructure which is increased extensively during curing time especially between 2 and 7 days, while there is no remarkable change when mixing limestone dust with water.
12. Another comparative study was conducted using XRD analysis to indicate the mineralogy of limestone dust and TBF-2 fillers. XRD analysis confirms the SEM findings through indicating the released mineralogical components such as lime and gelinite and appearance of new components such as C–S–H, which is the main component that the improved strength can be attributed to.
13. It was found that heating of CRA mixtures by means of microwave application and in the post-mixing method considerably decreased air voids contents for TBF-2 mixtures and produces a novel mixture in a moderate temperature (69 °C), which can be named Half-Warm Rolled Asphalt (HWRA). The mechanical properties and durability of

HWRA are encouraging due to their high early strength and lower water sensitivity when compared with control CRA, OPC, 100/150 and 40/60 HRA mixtures.

14. It can be concluded that TBF-2 can be introduced as a new cementitious material which is generated totally from cost-plus materials and might be suitable for other civil engineering applications such as normal concrete, lightweight concrete, masonry products, road base, road subbase, road subgrade, plastering board, and structural elements etc.

10.2 Recommendations for further works

Based on the experience gained during the course of this research, a number of possible future studies can be recommended, as listed below:

1. Study the ability of applying chipping materials onto Cold Rolled Asphalts with different gradation types.
2. Incorporate the optimised fillers adopted from this study with percentages more than the maximum filler content.
3. Investigate the performance of different gradation types, e.g. asphalt concrete, stone mastic asphalt and porous asphalt, with the produced fillers. Also, investigate the performance of binder course and base course with cold BEMs containing the new fillers.
4. Study the idea of adding other available supplementary cementitious materials around the world to produce cold BEMs, such as cement kiln dust, metakaolin and APC.
5. Study the possibility of incorporating the same fillers in foamed bituminous mixtures and warm bituminous mixtures.
6. Investigate the performance of CRA mixtures in real cases of loading and climatic conditioning by constructing a trial road section.

7. Investigate the performance of Half-Warm Bituminous Mixtures with different frequency and power of microwave heating.

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Appendices

Appendix I: Requirements and performance classes for cationic bitumen emulsion according to BS EN 13808 2013

Table AI-1: Specification framework for the requirements & performance classes of bituminous emulsions (European Committee for Standardization, 2013b)

		Performance classes for the technical requirements of cationic bituminous emulsions										
Technical requirements	**Document**	**Unit**	**Class 0**	**Class 1[a]**	**Class 2**	**Class 3**	**Class 4**	**Class 5**	**Class 6**	**Class 7**	**Class 8**	**Class 9**
Perceptible properties	EN 1425	None	NPD	TBR	-	-	-	-	-	-	-	-
Particle polarity	EN 1430	None	-	-	Positive	-	-	-	-	-	-	-
Breaking value	EN 13075-1	None	NPD	TBR	≤ 80	50 to 100	70 to 130	120 to 180	170 to 230	≥ 220	-	-
Mixing stability with cement	EN 12848	g	NPD	TBR	≤ 2	> 2	-	-	-	-	-	-
Fines mixing time	EN 13075-2	S	NPD	TBR	≥ 180	≥ 300	--	-	-	-	-	-
Penetration power	EN 12849	Min	NPD	TBR	-	-	-	-	-	-	-	-
Binder content (by water content)	EN 1428	Per cent by mass	NPD	TBR	38 to 42	48 to 52	53 to 57	58 to 62	63 to 67	65 to 69	67 to 71	≥ 70
Recovered binder content (by distillation)	EN 1431	Per cent by mass	NPD	TBR	≥ 38	≥ 48	≥ 53	≥ 58	≥ 63	≥ 65	≥ 67	≥ 70
Oil distillate content	EN 1431	Per cent by mass	NPD	TBR	≤ 2.0	≤ 3.0	≤ 5.0	≤ 8.0	≤ 10.0	5-15	> 15	-
Efflux time 2 mm at 40 ºC	EN 12846	s	NPD	TBR	≤ 20	15 to 45	35 to 80	70 to 130	-	-	-	-
Efflux time 4 mm at 40 ºC	EN 12846	s	NPD	TBR	-	-	-	-	10 to 45	30 to 70	50 to 100	-
Efflux time 4 mm at 50 ºC	EN 12846	s	NPD	TBR	-	-	-	-	-	-	-	25 to 50
Dynamic viscosity at 40 ºC	prEN 14896	M pa.s	NPD	TBR	DV	-	-	-	-	-	-	-
Residue on sieving	EN 1429											
0.5 mm sieve		Per cent by mass	NPD	TBR	≤ 0.1	≤ 0.2	≤ 0.5					
0.16 mm sieve		Per cent by mass	NPD	TBR	≤ 0.25	≤ 0.5	-	-	-	-	-	-
Residue on sieving (7 days storage)	EN 1429											
0.5 mm sieve		Per cent by mass	NPD	TBR	≤ 0.1	≤ 0.2	≤ 0.5	-	-	-	-	-
Settling tendency (7 days storage)	EN 12847	Per cent by mass	NPD	TBR	≤ 5	≤ 10	-	-	-	-	-	-
Adhesivity	EN 13614	Per cent coating	NPD	TBR	≥ 75	≥ 90	-	-	-	-	-	-
a class 1, TBR, may not be used for regulatory declaration and marking purposes.												

Table AI-2: Specification framework for the technical requirements and performance classes of binders recovered by evaporation from cationic bituminous emulsions (European Committee for Standardization, 2013b)

			Performance classes for the technical requirements of binders recovered by evaporation from cationic bituminous emulsions.							
Technical requirements	**Document**	**Unit**	**Class 0**	**Class 1[a]**	**Class 2**	**Class 3**	**Class 4**	**Class 5**	**Class 6**	**Class 7**
Method of recovery: by evaporation	EN 13074	-	-	-	-	-	-	-	-	-
Penetration at 25 ºC	EN 1426	0.1 mm	NPD	TBR	≤ 50	≤ 100	≤ 150	≤ 200	≤ 330	> 330
Softening point	EN 13357	ºC	NPD	TBR	≤ 55	≤ 50	≤ 43	≤ 39	≤ 35	≥ 35
Efflux time (10 mm cup at 25 ºC or 40 ºC as appropriate)	EN 12596	s	NPD	TBR	DV	-	-	-	-	-
Dynamic viscosity at 60 ºC	EN 12596	Pa.s	NPD	TBR	≤ 18.0	≤ 12.0	≤ 7.0	≤ 4.0	≤ 2.0	-
Kinematic viscosity at 60 ºC	EN 12595	mm^2/s	NPD	TBR	≥ 1600	≥ 8000	≥ 6000	≥ 4000	≥ 2000	≤ 2000
Cohesion (modified binders only)										
Cohesion energy by tensile test at 5 ºC (low speed)	EN 13587 EN 13703	J/cm^2	NPD	TBR	≥ 1	≥ 2	≥ 3	-	-	-
Cohesion energy by force ductility at 5 ºC	EN 13589 EN 13703	J/cm^2	NPD	TBR	≥ 1	≥ 2	≥ 3	-	-	-
Cohesion by pendulum test	EN 13588	J/cm^2	NPD	TBR	≥ 0.5	≥ 0.7	≥ 1.0	≥ 1.2	≥ 1.4	-
Elastic recovery at 10 ºC (for elastic polymer binders)	EN 13398	Per cent	NPD	TBR	≥ 30	≥ 40	≥ 50	≥ 75	-	-
Elastic recovery at 25 ºC (for elastic polymer binders)	EN 13398	Per cent	NPD	TBR	≥ 30	≥ 40	≥ 50	≥ 75	-	-
a class 1, TBR, may not be used for regulatory declaration and marking purposes.										

Table AI-3: Framework specification for the technical requirements and performance classes of binders recovered by evaporation from cationic bituminous emulsions and subjected to a stabilising procedure possibly followed by an ageing procedure

			Performance classes for the technical requirements of binders recovered by evaporation and subjected to a stabilising procedure (pr EN 14895)		**Performance classes for the technical requirements of binders recovered by evaporation and subjected to a stabilising and ageing procedure (prEN 14895 followed by EN 14769)**		
Technical requirements	**Document**	**Unit**	**Class 0**	**Class 1[a]**	**Class 0**	**Class 1[a]**	**Class 2**
Stabilisation of binder	prEN 14895	None	-	-	-	-	-
Method of aging: Accelerated long term aging by PAV	prEN 14769	None	Not relevant	Not relevant	-	-	-
Penetration at 25 ºC	EN 1428	0.1 mm	NPD	TBR	NPD	TBR	DV
Softening point	EN 1427	ºC	NPD	TBR	NPD	TBR	DV
Dynamic viscosity at 60 ºC	EN 12596	Pa.s	NPD	TBR	NPD	TBR	DV
Kinematic viscosity at 60 ºC	EN 12595	Mm^2/s	NPD	TBR	NPD	TBR	-
Cohesion (modified binders only)							
Cohesion energy by tensile test at 5 ºC (low speed)	EN 13589 EN 13703	J/cm^2	NPD	TBR	NPD	TBR	DV
Cohesion energy by force ductility at 5 ºC	EN 13588 EN 13703	J/cm^2	NPD	TBR	NPD	TBR	DV
Cohesion by pendulum test	EN 13588	J/cm^2	NPD	TBR	NPD	TBR	DV
Elastic recovery at 10 ºC (for elastic polymer binders)	EN 13398	Per Cent	NPD	TBR	NPD	TBR	-
Elastic recovery at 25 ºC (for elastic polymer binders)	EN 13398	Per Cent	NPD	TBR	NPD	TBR	-
a class 1, TBR, may not be used for regulatory declaration and marking purposes.							

Appendix II: List of Journal Publications

Published journal papers:

1. Al-Hdabi, A., Al Nageim, H. and Seton, L. "Performance of Gap Graded Cold Asphalt Mixtures Containing Cement Treated Filler", Journal of Construction and Building Materials, ISSN: 0950-0618, Volume 69, pp. 362-369, October 2014.
2. Al-Hdabi, A., Al Nageim, H, and Seton, L. "Superior Cold Rolled Asphalt Mixtures Using Supplementary Cementations Materials", Journal of Construction and Building Materials, ISSN: 0950-0618, Volume 64, pp. 95-102, August 2014.
3. Al-Hdabi, A., Al Nageim, H, Ruddock, F. and Seton, L. "Laboratory Studies to Investigate the Properties of Novel Cold Rolled Asphalt Containing Cement and Waste Bottom Ash" Journal of Road Materials and Pavement Design, ISSN 1468-0629 (Print), 2164-7402 (Online), Volume 15, Issue 1 pp. 78-89, 2014.
4. Al-Hdabi A., Al Nageim H., Ruddock F., Seton L., "A Novel Cold Rolled Asphalt Mixtures for Heavy Trafficked Surface Course", Journal of Construction and Building Materials, ISSN: 0950-0618, Volume 49, pp. 598-603, December 2013.
5. Al-Hdabi A., Al Nageim H., Ruddock F., Seton L., "Enhancing the Mechanical Properties of Gap Graded Cold Asphalt Containing Cement Utilising by-product Material", Journal of Civil Engineering and Architecture, ISSN 1934-7359 e-ISSN: 1934-7367, Volume 7, number 8, pp. 916-921, August 2013.
6. Al-Hdabi A., Al Nageim H., Ruddock F., Seton L., "Development of Sustainable Cold Rolled Surface Course Asphalt Mixtures Using Waste Fly Ash and Silica Fume". ASCE's Journal of Materials in Civil Engineering, e-ISSN: 1943-5533, http://dx.doi.org/10.1061/(ASCE)MT.1943-5533.0000843 , April 2013.

Book chapter:

1. Al-Hdabi, A., AL Nageim, H., Ruddock, F. & Seton, L. 2014. Improving the Mechanical Properties of Cold Rolled Asphalt Containing Cement Utilising by Product Material. Construction and Building Research, pp 487-496, Springer Netherlands.

Appendix III: List of Conference Publications

Presented conference papers:

1. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock, Linda Seton, 2014 "Performance of Cold Rolled Asphalt with Different Conditioning Processes". Proceeding of the 9th Annual Built Environment and Natural Environment Conference, Liverpool, UK.
2. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock and Linda Seton, 2014 "Superior Cold Rolled Asphalt for Surface Course Pavement Construction". Presented in: the 13th Annual International Conference on Pavement Engineering and Infrastructure, Liverpool, UK. ISBN 978-0-9571804-2-0.
3. Hassan Al Nageim, Abbas Al-Hdabi, Felicite Ruddock and Linda Seton, 2014 "Mechanical Properties and Durability of Cement Treated Gap Graded Cold Bituminous Mixtures". Presented in: the 13th Annual International Conference on Pavement Engineering and Infrastructure, Liverpool, UK. ISBN 978-0-9571804-2-0.
4. Abbas Al-Hdabi, Hassan Al Nageim and Linda Seton, 2014 "High Strength Cold Rolled Asphalt for Pavement Construction". Presented in: the International Conference of Engineering Science, University of Mustansiriyah, 26-27 March, Baghdad, Iraq.
5. Hassan Al Nageim, Abbas Al-Hdabi and Linda Seton, 2014 "A New Novel Cold Rolled Asphalt for Roads and Highways". Presented in: the International Conference of Engineering Science, University of Mustansiriyah, 26-27 March, Baghdad, Iraq.
6. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock and Linda Seton, 2013 "Durability and Stiffness Modulus of New Cold Rolled Asphalt Containing Cement and Waste Fly Ash". Proceeding of the 12th Annual International Conference on Pavement Engineering and Infrastructure, Liverpool, UK. ISBN 978-0-9571804-2-0.

7. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock and Linda Seton, 2013 “Development of Sustainable Cold Rolled Asphalt Using Binary Blended Filler”. Proceeding of the 8th Annual Built Environment and Natural Environment Conference, Liverpool, UK.
8. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock, Linda Seton, 2012 “Cold Rolled Asphalt Surface Course Containing Waste Materials” Proceeding of the Creative Construction Conference 2012, June 30 – July 3, 2012, Budapest, Hungary. ISBN 978-963-269-297-5.
9. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock, Linda Seton, 2012 “Development of Cold Rolled Asphalt Surface Course”. Proceeding of the 11th Annual International Conference on Pavement Engineering and Infrastructure, Liverpool, UK. ISBN 978-0-9571804-0-6.
10. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock, Linda Seton, 2012 “Improving the Mechanical Properties of Cold Rolled Asphalt Containing Cement Utilising by Product Material”. Proceeding of the second International Conference on Construction and Building Research, 14th-16th November, 2012, Valencia, Spain.
11. Abbas Al-Hdabi, Hassan Al Nageim, Felicite Ruddock, Linda Seton, 2012 “Stiffness Modulus and Durability of Cold Rolled Asphalt Surface Course Mixture”. Proceeding of the 7th Annual Built Environment and Natural Environment Conference, Liverpool, UK.

Appendix IV: Awards, honours, and recognitions

1. An Honourable Mention Award by the Association of British Turkish Academics (ABTA) in engineering category of the 2014 ABTA Doctoral Researcher Awards competition, May 2014.
2. A recognition on the 14^{th} of March 2014 at the Iraqi cultural attaché in London by the Iraq Minister of Higher Education, due to publish more than 10 journal and conference papers (until that time) within the author PhD research.

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25 MAY 2014

In honour and recognition of your academic excellence
Association of British Turkish Academics (ABTA)
hereby presents

Abbas Al-Hdabi

Honourable Mention

2014 ABTA Doctoral Researcher Awards

On this 25th day of May 2014
On behalf of ABTA Executive Committee
Dr Hasan Yardimci

2014 ABTA DOCTORAL RESEARCHER AWARDS

الملحقية الثقافية العراقية - لندن

تثمين جهود

السيد عباس طالب محسن المحترم

تقديراً لإنجازاتكم العلمية ومشاركتكم في المُؤتمرات العالمية ونَشْركم البحوث في المجلات الرصينة ورفع اسم العراق في المحافل الدولية، نتقدم اليكم بالشكر والتقدير متمنين لكم الموفقية في خدمة الوطن العزيز والى مزيد من العطاء.

علي محمد الحسين الأديب

وزير التعليم العالي والبحث العلمي

2014/3/14