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New Developments with Cold Asphalt Concrete Binder Course Mixtures Containing Binary Blended Cementitious Filler (BBCF)

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Abstract

A weakness in early strength and the need for longer curing times in the case of cold bituminous emulsion mixtures (CBEMs) compared to hot mix asphalt have been cited as barriers to the wider utilization of these mixtures. A binary blended filler material produced from high calcium fly ash (HCFA) and a fluid catalytic cracking catalyst (FC3R) was found to be very effective in providing microstructural integrity with a novel fast-curing cold asphalt concrete for the binder course (CACB) mixture. Balanced oxide compositions within the novel filler were identified as responsible for an enhanced hydration reaction, resulting in a very high early strength and a significant improvement in permanent deformation and fatigue resistance. Improved water sensitivity for progressive hydration with the new binary filler was also established while SEM analysis confirmed the formation of hydration products after various curing ages.

Keywords:
Binder course
Cold asphalt mix
Emulsion
Fluid catalytic cracking catalyst
High calcium fly ash
Microstructure
Rutting
Stiffness modulus
Water sensitivity

1. Introduction

Currently, attempts to save energy and decrease emissions from asphalt paving applications have led to a growth in the usage and acceptance of sustainable pavement design practices. One of these practices is the use of cold asphalt mixtures (CAMs), as less energy is used when producing its main constituents. CAMs can be defined as bituminous materials mixed using aggregates and binders at ambient temperatures [1]. Both economic and environmental advantages can be achieved by removing the need to heat substantial volumes of aggregate compared to traditional hot mix asphalt [2, 3]. However, these mixtures are inferior in terms of their mechanical properties, high air voids, rain sensitivity and the long curing time needed to achieve their final strength [4, 5]. It is generally accepted that hot mix asphalt should approach its full design strength in a relatively short period of time, this enabling its characteristics to be measured almost immediately after manufacture. For a number of reasons, the same approach cannot be applied to CAMs [6]. The CAMs technology adopted in the pavement industry has been widely utilised in many countries for example, France and Turkey in recent years. Annual production is 1.5 million tonnes in the former and 2 million tonnes in the latter [7]. In contrast, due to the weather conditions in the UK, the use of CAMs is restricted to base and sub-base courses of structural layers [4, 8]. Subsequently, the use of cold emulsified asphalt as a structural layer is restricted because of the long period necessary for such material to reach its full strength due to the UK weather and because of higher sensitivity to rainfall in the early stages of application [9].

Those mixtures have been described as evolutive materials [6] in contrast to traditional hot mix asphalt which gain strength rapidly as the material cools. They gradually achieve strength when the material becomes dry, further developing increasing strength over time. There are continuously changing properties exhibited by emulsion bound mixtures (stiffness modulus, permanent deformation resistance, water sensitivity, etc.) which continue until they reach a steady state at a fully cured condition, though they may still contain a low amount of residual water [6]. Jenkins [1] demonstrated that when moisture is expelled from the mix, cold bituminous mixtures gain strength.

Conventional cement is commonly used in cold mix asphalt as an enhancement technique. One of the benefits of this technique is that satisfactory strength can be reached in a short period of time [10, 11]. Brown and Needham [12] examined cement modified emulsion mixtures, the primary aim to appraise the influence of adding Ordinary Portland Cement (OPC) to said mixtures. They used a granite aggregate grading in the middle of 20mm dense bituminous macadam with a single slow-setting emulsion. They concluded that the addition of OPC enhanced the mechanical properties, namely: stiffness modulus, permanent deformation resistance and the fatigue strength of the emulsified mixes. Oruc et al. [9] also carried out a study to assess the mechanical properties of emulsified asphalt mixtures incorporating 0-6% Portland cement. Substantial improvements were seen with the addition of higher quantities of Portland cement and they recommended that such a mixture might be used as a structural pavement layer.

The re-use of waste materials in cold asphalt mixtures is generally promoted for two reasons: environmental sustainability (cement manufacture is responsible for 5% of global greenhouse gases (GHG) [13]) and economic benefits. Ellis et al. [14] investigated the performance of a range of storage-grade macadams consisting of recycled aggregates from different sources bound by bitumen emulsion and Ground Granulated Blastfurnace Slag (GGBS). The results revealed that stiffness and strength can develop when GGBS is added in conditions of high humidity. Thanaya et al. [15] conducted experiments using pulverised fly ash (PFA) as a filler in a cold mix at full curing conditions; the cold mix stiffness was found to be comparable to that of hot mixes.

Al-Busaltan et al. [5] used waste materials (waste fly ash as the substitute for traditional mineral filler) to develop a cold mix asphalt, a close graded cold bituminous emulsion mixture (CBEMs) that had outstanding mechanical properties compared to traditional hot mix asphalt surface courses. The result was that an additional binder was produced from the hydration process that took place between the waste fly ash and the trapped water involved in cold mixtures.

Al-Hdabi et al. [16] applied two sorts of waste fly ash as filler substitutes to produce new gap-graded Cold Rolled Asphalts (CRA). The new CRAs produced were found to have a comparable performance with conventional Hot Rolled Asphalts (HRA) after short periods of curing in terms of stiffness modulus and uniaxial creep tests. In addition, a considerable improvement in terms of resistance to water damage which reported. The effect of chemical additives comprising OPC, hydrated lime (HL) and a combination of HL and GGBS on recycled mixture performance with asphalt emulsion was examined by Du [17]. He concluded that the level of performance enhancement depends on the type and content of chemical additives. Hydration products can result in stiffness increments and cohesion of the asphalt mastic of the recycled mixture.

It was reported by Čížková et al. [18] that test conditions such as applied test temperature and loading speed, effect the stiffness modulus of bitumen stabilized materials along with other cold recycled mixtures. Viscoelastic material behaviour created from reclaimed asphalt material (RA/RAP) in addition to the type of bituminous binder applied, can explain this. Zak and Valentin [19] stated that Bitumen Stabilized Materials (BSM) have higher air voids, less adhesion and less cohesion in the internal structure in comparison to HMA. Accordingly, the fatigue performance of such mixture is inferior to HMA. Recently, Nassar et al. [20] used binary and ternary blended fillers (BBF and TBF) to see if there was any improvement in Cold Asphalt Emulsion Mixtures (CAEMs). Various fillers sorts were used such as OPC, fly ash, GGBS and silica fume. Their results indicated that the TBF was more appropriate than the BBF for CAEMs manufacture claiming that the TBF mixture would be effective for use on road pavements subject to harsh conditions, both hot and cold weather.

Sadique et al. [21] conducted research exploring the pozzolanic reactivity of calcium rich fly ash by blending and grinding it in a cement-free system, finding that the hydration products and improvements in strength in the new blend was comparable to cement. Mármol et al. [22] investigated the pozzolanic activity of FC3R in Portland cement systems by examining the water/binder effect. They compared reactivity to Metakaolin, a synthetic pozzolan, reporting that FC3R has a comparable chemical structure and high pozzolanic activity. Accordingly, HCFA and FC3R have the potential to be employed as supplementary cementitious materials (SCMs) to substitute traditional mineral filler. As further support, Pacewska et al. [23] revealed that both spent catalyst and microsilica have the same potential to be combined with $Ca(OH)_2$, as the hydration process is highly exothermic, resulting in rapid setting of the cement paste. These findings offer a potential impact on sustainability by eliminating the need for cement in cold asphalt mixtures.

Despite extensive research into different types of CBEMs, to date, the problems relating to curing time, low early strength, rutting resistance and water sensitivity of CBEMs for the binder course have not been addressed. Therefore, developing a cold asphalt concrete for the binder course (CACB) mixture with high early strength, resistance to water ingress and with minimal time requirements for structural loading would be a breakthrough in CBEM research. Developing a novel binder to be used as a filler in CACB would provide the enhanced properties mentioned above. Therefore, an investigation to establish an advanced method for developing a cementitious binder suitable for CACB to be used as a filler has been carried out in this study. A supplementary cementitious material derived from industrial waste has been used to enhance the performance of the CACB mixtures. The indirect tensile stiffness modulus (ITSM) test, permanent deformation resistance and tests of fatigue resistance were utilised to evaluate the effects of using high calcium fly ash (HCFA) with a fluid catalytic cracking catalyst (FC3R) on the mechanical properties of CACB mixtures. The effect of using these materials on moisture damage resistance was evaluated by determining the Stiffness Modulus Ratio (SMR).

2. Materials and method

The properties of the materials that have been utilised in the preparation of laboratory samples are given below.

2.1 Materials

2.1.1 Aggregate

Coarse and fine aggregates were crushed granite from Carnsew Quarry at Mabe, Penryn in the UK, which is generally used to produce hot asphalt concrete mixtures. A dense aggregate gradation for the asphalt concrete binder course AC-20 was used in this research, as shown in Figure 1. The aggregate structure permitted a curve to be established following the standard EN 13108-1 [24]. This gradation is normally used on asphalt pavement binder course in the UK. The physical specifications of both the coarse and fine aggregate have been detailed in Table 1.

Asphalt concrete is by far the most common mixture in use as a binder course and base in road pavements in the UK. Having a continuous grade, it offers a good aggregate interlock giving it very good load-spreading properties as well as high resistance to permanent deformation.

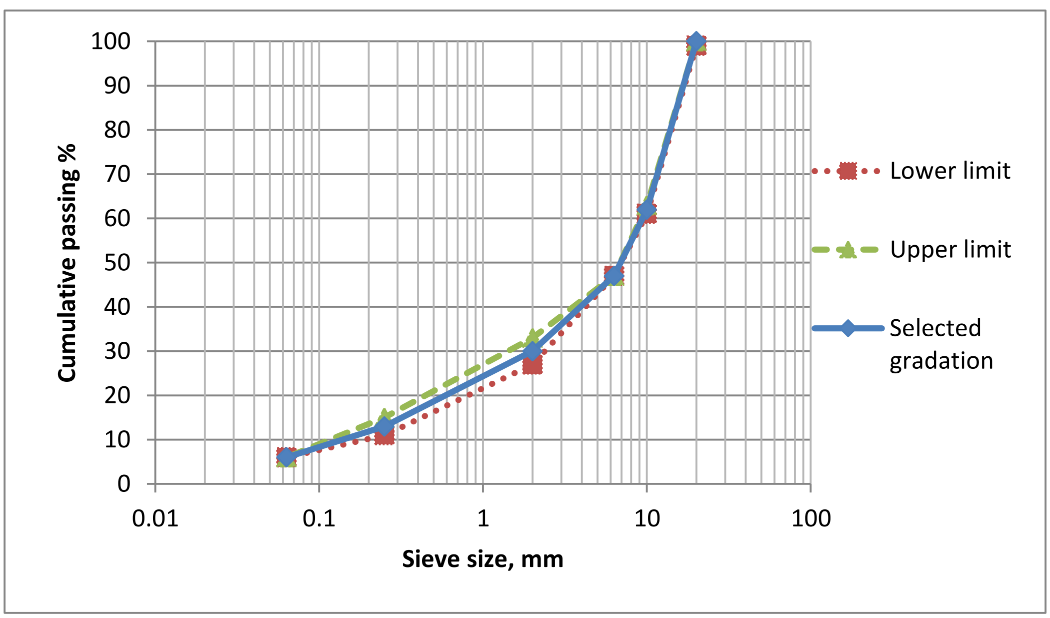

Figure 1. AC 20 mm dense binder course aggregate gradation

Table 1. Aggregate physical properties

Material	Property	Value
Coarse aggregate	Bulk particle density, Mg/m^3	2.62
	Apparent particle density, Mg/m^3	2.67
	Water absorption, %	0.8
Fine aggregate	Bulk particle density, $Mg/\ m^3$	2.54
	Apparent particle density, $Mg/\ m^3$	2.65
	Water absorption, %	1.7
Traditional mineral filler (limestone filler)	Particle density, $Mg/\ m^3$	2.57

2.1.2 Bitumen emulsion and asphalt

Cationic slow-setting bitumen emulsion (C60B5) designed for use in road pavement and general maintenance applications, was used to prepare the cold bituminous emulsion mixtures. Thanaya [25] confirmed that cationic emulsion is preferred as a result of its ability to coat aggregate and to guarantee high adhesion between aggregate particles. Table 2 illustrates the properties of the chosen bitumen emulsion. Two grades of bitumen, soft bitumen of penetration grade (100/150) and hard bitumen of penetration grade (40/60), were used to make the hot asphalt concrete binder course mixtures. Table 3 details the characteristics of these binders.

Table 2. Properties of (C60B5) bitumen emulsion

Description	(C 60 B5) bitumen emulsion
Type	Cationic
Appearance	Black to dark brown liquid
Base bitumen	100/150 pen
Bitumen content, (%)	60
Particle surface electric charge	Positive
Boiling point, (°C)	100
Relative density at 15 °C, (g/ml)	1.05

Table 3. Properties of 40/60 and 100/150 bitumen binders

Bituminous binder 40/60		Bituminous binder 100/150	
Property	Value	Property	Value
Appearance	Black	Appearance	Black
Penetration at 25 °C, (0.1 mm)	49	Penetration at 25 °C, (0.1 mm)	131
Softening point, (°C)	51.5	Softening point, (°C)	43.5
Density at 25 °C, (g/cm^3)	1.02	Density at 25 °C, (g/cm^3)	1.05

2.1.3 Chosen fillers

Two waste materials were used as filler replacement in this study: high calcium fly ash (HCFA) which is generated from power generation plants by combustion at between 850°C and 1100°C using a fluidised bed combustion (FBC) system, and a spent fluid catalytic cracking catalyst (FC3R) a waste material generated via fluid catalytic cracking processes in petrol refineries from the fluidised bed process. FC3R is a silica-aluminous based material similar to metakaolin which makes this material a promising prospect with reference to the activation of the HCFA.

HCFA was blended with FC3R in different percentages to produce a new binary blended cement filler (BBCF) for the cold bituminous emulsion mixture. Limestone filler (LF) was utilised as the traditional mineral filler whereas a commercially available Ordinary Portland Cement was used for comparison purposes during the study. The chemical analysis by energy dispersive X-ray fluorescence (EDXRF) spectrometer of all the fillers can be seen in Table 4.

Table 4. Chemical analysis of the selected filler materials, %

Chemical composition	CaO	SiO_2	Al_2O_3	MgO	Fe_2O_3	SO_3	K_2O	TiO_2	Na_2O
HCFA	67.057	24.762	2.430	2.845	0	0.340	0.266	0.473	1.826
FC3R	0.047	35.452	44.167	0.684	0.368	0	0.049	0	0
OPC	62.379	26.639	2.435	1.572	1.745	2.588	0.724	0.385	1.533
LF	76.36	16.703	0	0.981	0	0.096	0.348	0.185	2.258

As shown in Table 4, it can be seen that HCFA is mainly composed of CaO with a good amount of SiO_2 and Al_2O_3. It was reported that the existence of Ca, Al and Si in fly ash are significant in developing a new cementitious material from waste and by-product materials [21]. Sadique and Al-Nageim [26] reported a lower CaO content (57%) in their investigation but the proportion of SiO_2 was higher (28%). The main oxides in FC3R are Al_2O_3 and SiO_2, this consistent with those achieved by Mármol et al. [22], Mas et al. [27].

Lea [28] stated that soluble SiO_2 and Al_2O_3 present in the glass phase of pozzolanic materials can react with $Ca(OH)_2$ when released through cement hydration to make an extra calcium silicate hydrate (CSH) gel that enhances the mechanical strength of the hardened concrete structure.

The powder pattern of HCFA in XRD shown in Figure 2 revealed that the HCFA sample is crystalline as it contains sharp peaks without significant noise in the background. The major crystal peaks identified were: lime (CaO), calcite ($CaCO_3$), mayenite ($Ca_{12}Al_{14}O_{33}$), merwinite ($Ca_3Mg[SiO_4]$) and gehlenite ($CaAl[Al,SiO_7]$). A similar mineralogy was reported by Sadique and Al-Nageim [26], Sadique et al. [29], however no merwinite was detected in the latter.

The powder diffraction in XRD shown in Figure 3 indicated that FC3R has very low crystalline peaks which are amorphous in nature meaning that it will demonstrate high reactivity during the hydration process and can be used as a pozzolanic material. The following crystalline peaks were identified: kyanite (Al_2O_5Si), quartz (SiO_2), mullite ($Al6Si_2O_{13}$) and dehydrated Ca-A zeolite ($Al_{96}Ca_{48}O_{38}4Si_{96}$).

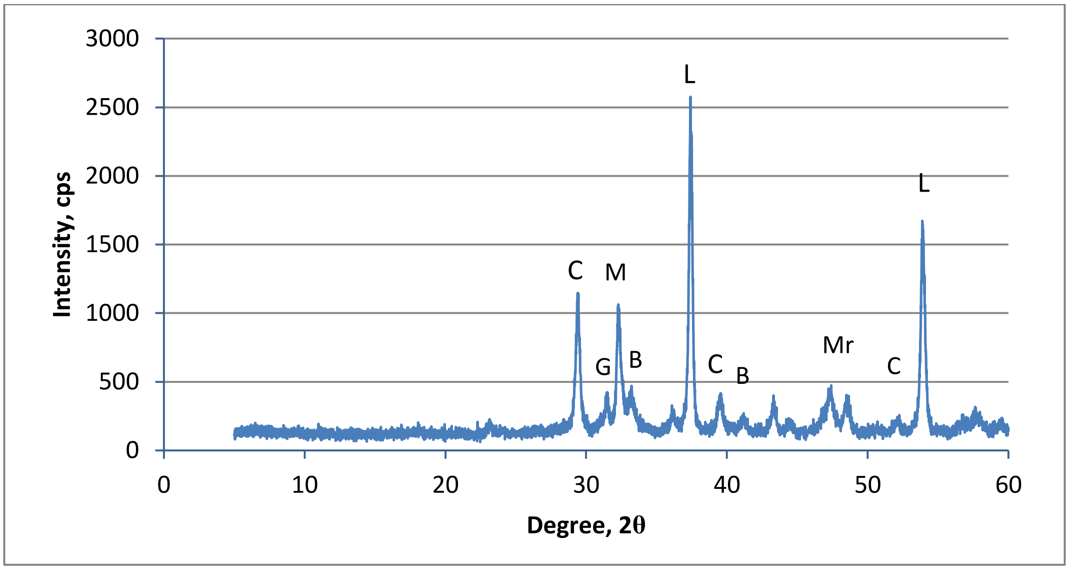

Figure 2. Powder XRD pattern of HCFA

(lime-L, calcite-C, gehlenite-G, belite-B, mayenite-M, merwinite-Mr)

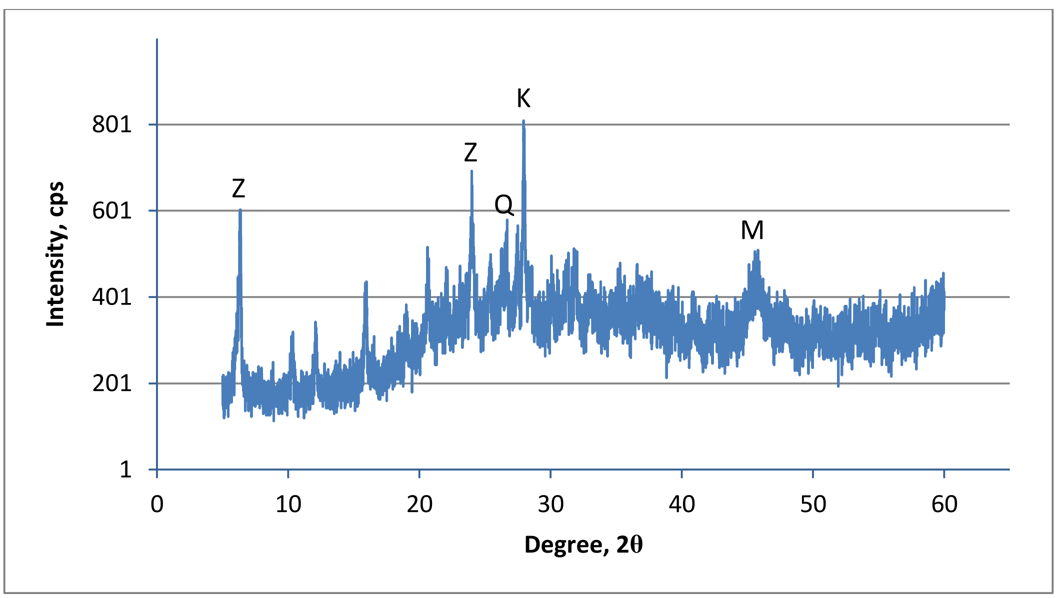

Figure 3. Powder XRD pattern of FC3R

(K- kyanite (Al_2O_5Si), Q – quartz (SiO_2), M- mullite ($Al_6Si_2O_{13}$), Z- dehydrated Ca-A zeolite ($Al_{96}Ca_{48}O_{384}Si_{96}$))

2.2 Sample preparation and conditioning

In this research, OPC, HCFA and FC3R were utilized to substitute traditional limestone filler in the CACB mixtures. To date, there is no universally accepted design mixture for CBEMs although some mix design procedures for CBEMs have been suggested by some authorities and researchers [1, 25, 30]. The design procedure for the current study followed the method implemented by the Asphalt Institute [30], the Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14). Preparation according to the design method was carried out as follows:

The first step was to decide the gradation of the aggregate as stated in Section 2.1.1. Next, the initial emulsion content was determined by using an empirical equation as recommended by the Asphalt institute manual MS-14. The aggregate gradation has a major effect on the initial emulsion content according to this equation. Determining the pre-wetting water content (PWC) has to be considered in light of the fact that the coating ability of the bitumen emulsion to the aggregate is dependent on the pre-wetting water content. This is even more pertinent when the aggregate gradation comprises a high proportion of materials passing a 63 µm sieve. Various pre-wetting water contents were examined to select the lowest ratio and ensure that the coating was satisfactory. The optimum bitumen emulsion content (OBEC) was decided by the indirect tensile stiffness modulus test according to BS EN 12697-26 [31]. Finally, a mix density test was utilised to decide the optimal total liquid content at compaction (OTLCC) (i.e. emulsion plus pre-wetting water contents providing the highest mix indirect tensile strength and density). Accordingly, PWC, OTLCC and OBEC were 3.5%, 14% and 10.5%, respectively. These findings are comparable to those published in other research [5, 32].

Cold asphalt concrete binder course mixtures were produced by substituting the mineral filler with HCFA and adding FC3R in different proportions as a supplementary cementitious material. The indirect tensile stiffness modulus test was carried out to evaluate the influence of HCFA and FC3R substitution, the results compared with those for conventional hot asphalt concrete binder course mixtures. Serfass et al. [6] found that cold mixes have evolutional characteristics mainly in their early life, where early cohesion is low and builds up slowly.

The proportions of the mixture by percentage of Marshall samples are summarized in Table 5. The materials were mixed in a Hobart mixer. Afterwards, compaction was achieved by means of a Marshall hammer with 100 blows, where 50 blows were applied to both faces of each specimen. It was reported by Nassar et al. [20] that Marshall compaction is an appropriate method to use to manufacture an appropriately dense mixture.

After compaction, the samples were left for 1 day at 20ºC in the mould; the next day they were de-moulded. All the specimens were then left in the lab at 20ºC and tested at various ages, i.e. 1, 3, 7, 14 and 28 days.

Four additional reference mixtures were prepared and tested for comparison purposes. An untreated mixture with traditional limestone filler (LF) was the first having the same design as other CACB mixtures. The second mixture was treated with 6% OPC, while two grades of hot Asphalt Concrete-AC 20 (third and fourth control mixtures), based on 100/150 pen and 40/60 pen, were tested at the same ages. The reference hot mixtures also had the same aggregate type and gradation. The bitumen content was 4.6% in accordance with the standard BS EN 13801-1 [24].

All the emulsion mixtures were fabricated and compacted at ambient temperature. The laboratory mixing temperatures of the hot mixes were fixed at 150-160°C and 160-170°C for the 100/150 pen and 40/60 pen respectively.

Table 5. Details of the mix proportions of CACBs.

Types of mixtures	Filler types	Bitumen emulsion, %	Pre-wetting, %
1.5% HCFA mix	1.5% HCFA + 4.5% LF	10.5%	3.5%
3% HCFA mix	3% HCFA + 3% LF	10.5%	3.5%
4.5% HCFA mix	4.5% HCFA + 1.5% LF	10.5%	3.5%
HCFA mix	6% HCFA	10.5%	3.5%
BBCF mix	4.5% HCFA +1.5% FC3R	10.5%	3.5%
HCFA-FC3R-3-1 mix	3% HCFA+1% FC3R+2%LF	10.5%	3.5%
HCFA-FC3R-3-2 mix	3% HCFA+2% FC3R+1%LF	10.5%	3.5%
HCFA-FC3R-3-3 mix	3% HCFA+3% FC3R	10.5%	3.5%
Control mixtures			
LF mix	6% LF	10.5%	3.5%
OPC mix	6% OPC	10.5%	3.5%
Hot AC 100/150 mix	6% LF	4.6% base binder 100/150	-
Hot AC 40/60 mix	6% LF	4.6% base binder 40/60	-

Regarding the wheel track slabs, the sample mixtures for rutting tests were prepared in the same way as for the stiffness tests. A slab sample with a 400mm length, 305mm width and 50mm thickness was compacted at ambient temperature in the steel mould using a Cooper Technology Roller Compactor device following the standard BS EN 12697-33 [33].

The slab specimens were kept in their moulds at lab temperature (20°C) for 24 hours before extraction, this representing the first curing stage. Stage two involved curing the slabs at 40°C for 14 days, removing them from the ventilated oven, cooling and subjecting to the wheel track test. This curing protocol was recommended by Thanaya [25] to guarantee that a completely cured condition was reached. All the tests were then performed on CACB mixtures at a fully cured condition. For the fatigue tests, slab samples were prepared and cured in the same way as for the wheel track test samples. The slab samples were then cut with a saw to provide a beam shape sample with 400mm length, 50mm height and 50mm width.

2.3 Experimental program and tests performed

The indirect tensile stiffness modulus (ITSM) test and rutting resistance were applied to evaluate the use of the supplementary cementing material on the mechanical properties of CACB, while SMR was used to assess moisture sensitivity. A Scanning Electron Microscopy (SEM) observation was applied to investigate the microstructure of the hydration products. Many researchers, for example Al-Busaltan et al. [2], Nassar et al. [20], Thanaya [25], Monney et al. [34], Al-Hdabi et al. [35], have reported measuring the ITSM in order to evaluate the mechanical performance of CAM. A wheel-tracking test was adopted by Ojum [36] to characterize and assess the mechanism of failure of CBEMs. Four point beam-bending tests which evaluate the fatigue performance of CBEMs, is recommended by Al-Hdabi et al. [10]. In addition, numerous researchers such as Al-Busaltan et al. [2], Al-Busaltan et al. [5], Al-Hdabi et al. [37] have reported measuring water sensitivity in terms of Stiffness Modulus Ratio (SMR) of CAMs following BS EN 12697-12 [38].

2.3.1 Indirect tensile stiffness modulus (ITSM) test

The ITSM test is a non-destructive test used to evaluate the ability of an individual layer of a pavement to distribute traffic loads to the layer beneath. Currently, stiffness modulus is generally recognised as a significant performance property of bituminous paving materials and is used as an indication of the load-spreading ability of bituminous paving layers. The test was carried out on five samples for each mixture type following the standard BS EN 12697-26 [31] using Cooper Research Technology HYD 25 testing apparatus. Test conditions are shown in Table 6 below.

Table 6. ITSM test conditions

Item	Range
Specimen diameter, (mm)	100 ± 3
Rise time, (ms)	124 ± 4
Transient peak horizontal deformation, (µm)	5
Loading time, (s)	3-300
Poisson’s ratio	0.35
No. of conditioning plus	10
No. of test plus	5
Test temperature, (°C)	20 ± 0.5
Specimen thickness, (mm)	63± 3
Compaction	Marshall 50 blows/face
Specimen temperature conditioning	4hr before testing

2.3.2 Wheel-tracking tester

Laboratory wheel-tracking tests were applied to evaluate the rutting resistance of the cold bituminous emulsion mixtures following BS EN 12697-22 [39]. Wheel-tracking tests usually measure the rut produced by the repeated passage of a wheel over asphalt concrete slab samples. Slab samples of dimensions 400mm length and 305mm width were prepared for the cold asphalt concrete binder course bituminous emulsion mixtures and control mixtures. These samples were then tested for rutting susceptibility in a small size wheel-tracking device. The samples were tested at 45°C under application of 10,000 load passes of a 700N axle load. The longitudinal distance that the wheel travelled through on each pass was approximately 230 mm. The small HYCZ-5 wheel-tracking equipment used by the Liverpool Centre for Material Technology (LCMT) labs was used, Table 7 illustrating the test conditions. Five slab samples have been tested for each mixture type.

Table 7. Wheel-tracking test conditions

Item	Range
Tyre of outside diameter, (mm)	200-205
Tyre width, (mm)	50 ± 5
Trolley travel distance, (mm)	230 ± 10
Trolley travel speed, (s/min)	42 ± 1
Frequency load cycles per 60 s	26.5 ± 1.0
Poisson's ratio	0.35
No. of conditioning cycles	5
No. of test passes	10000
Test temperature, (°C)	45
Compaction	Roller compactor
Specimen temperature conditioning	4hr before testing

2.3.3 Four-point beam-bending test

The fatigue life of asphalt mixtures indicates its ability to resist repeated traffic loads without suffering failure. Because of this, fatigue resistance is considered a main principle in design methods of flexible pavements and was performed here using a standard four-point beam fatigue test. The fatigue life measured as equal to the number of load repetitions resulting in a 50% stiffness decrease. This test was performed according to BS EN 12697-24 [40] using the controlled strain method at a temperature of 20°C and 10 Hz frequency under sinusoidal loading with no rest period and a controlled strain criteria of 150 microstrain.

2.3.4 Water sensitivity

The ability of asphalt mixtures to resist moisture damage is critical to their long-term performance. Being sensitive to moisture damage makes the asphalt mixture eventually fail in any of the failure modes, e.g. rutting, fatigue, thermal cracking and ravelling [41]. A water sensitivity test was applied following the standard EN 12697-12 [38] to evaluate the mixtures' sensitivity under the moisture effect. This test reveals the effect of saturation and improved water conditioning on the indirect tensile stiffness modulus of cylindrical specimens of CBEMs, performed here following EN 12697-26 [31].

The water sensitivity test identified that the interior bonding of the asphalt mixture was reduced due to water existence. The specimens were divided into two sets; the first set of specimens were kept in the mould for 1 day before extraction and then left at 20°C for another 7 days prior to the stiffness modulus test performed at 20°C; they represented a dry condition. The second set of samples were kept in the mould for 1 day, extracted and left to cure at 20°C for 4 days before being subjected to a vacuum (with 6.7 kPa pressure for 30 minutes) and kept submerged in a glass jar for an additional 30 minutes. Following this, these samples were conditioned at 40°C for 3 days before testing, representing a wet condition. Five specimens were tested for each mixture type. The two sets were then tested using the ITSM test, where the water sensitivity was measured by determining the stiffness modulus ratio (SMR) ratio as follows:

$$SMR = (\text{wet stiffness} / \text{dry stiffness}) \times 100$$

2.3.4 Scanning Electron Microscopy (SEM) observation

Scanning electron microscopy (SEM) is a technique for high resolution imaging of surfaces to reveal the morphology and internal microstructure of the particles and surface characterization of materials. This technique will allow changes to the hydration products as a result of using HCFA and BBCF fillers in the

CACB mixture to be examined. The tests were performed with an SEM resolution of 3-4 nm, high vacuum and test voltage ranging from 5 kV to 25 kV using an Inspect scanning electron microscope.

Microstructural analyses were performed by employing SEM on selected paste samples (made with HCFA and BBCF) taken from the centre of the crushed specimens. These specimens were used to detect changes in the materials at various ages of curing. The pastes were moulded into cylinder samples which were kept for 1 day at room temperature and then demoulded. Appropriate pieces were then taken off the cylinders at due age, i.e. 3 and 28 days for SEM investigations. It was essential to ensure that the pieces were snapped out of the specimens by impact without touching any tools otherwise the paste surface would not be a natural one and would not accurately represent the features of the materials correctly. The pieces were mounted on aluminium stubs using double-sided adhesive carbon disks and subjected to a vacuum. A palladium coating was then applied to the sample, prior to taking the SEM images, using an auto fine sputter coater.

2. Results and Discussion

3.1 Performance of CACB mixtures in ITSM test

The first phase of the research concerned the effect of the substitution of conventional mineral filler with HCFA on the stiffness modulus of cold asphalt mixtures. The ITSM test was run in accordance with BS EN 12697-26 [31]. The results of ITSM tests for the HCFA replacement are shown in Figure 4, where it can be seen that the ITSM for 6% HCFA replacement after 3 days is around 17 times the reference for untreated cold mix asphalt (6% limestone filler- LF).

It is clearly demonstrated that the addition of HCFA as a substitute for limestone filler substantially enhanced the stiffness modulus, this improvement due to two effects. The first is the generation of another binder made from the process of hydration as a result of the hydraulic reaction of HCFA in addition to the bitumen residue binder. Secondly, trapped water was lost during the hydration of the HCFA. Of note here, conventional hot asphalt concrete binder course mixtures do not display visible differences in ITSM over time.

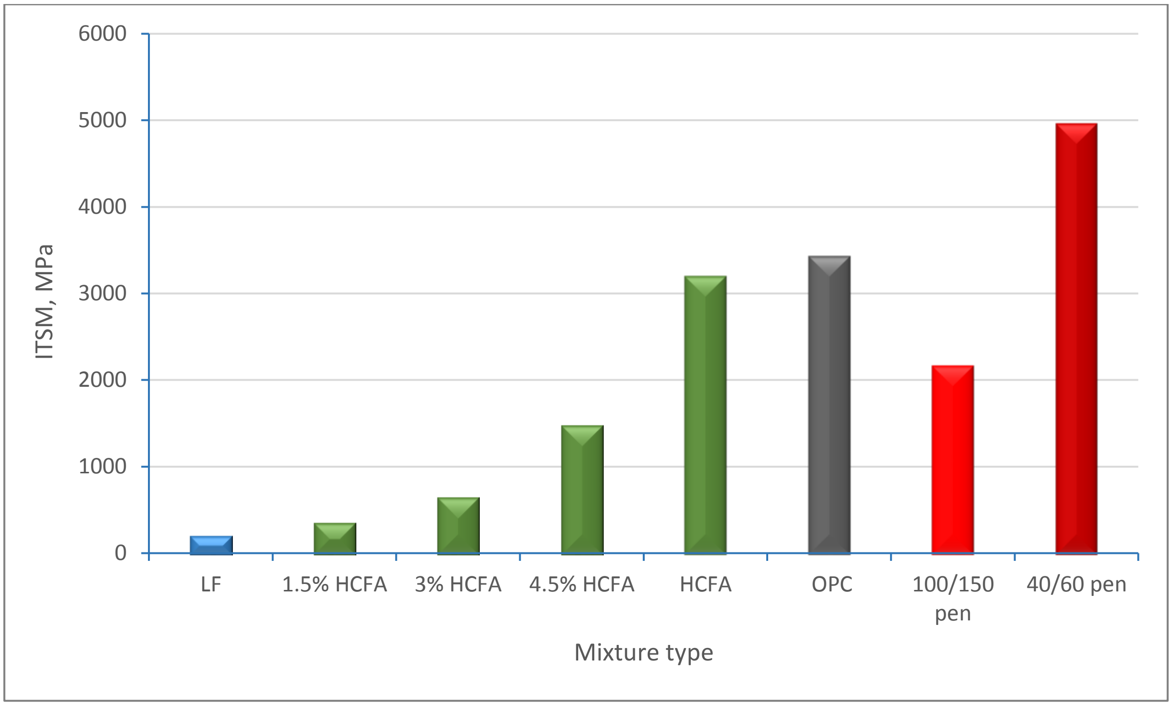

Figure 4. ITSM results after 3 days

The second phase was achieved by adding FC3R in a binary filler as a substitute for HCFA with different percentages (0%, 1%, 2% 3% and 4%) by the dry aggregate weight. The optimum composition within the binary blended filler was found to be 4.5% HCFA and 1.5% of FC3R as displayed in Figure 5, this creating the highest stiffness modulus after 3 days. A balanced oxide composition was expected to be formed in this composition within the binary blended filler. The presence of pozzolanic particles helped to expedite the hydration of the HCFA particles, resulting in more hydrated products. It is expected that adding pozzolanic materials with a high silica material will convert soluble calcium hydroxide (C–H), produced from the hydration reaction of HCFA filler, into dense calcium silicate hydrate (C-S-H) because of the pozzolanic reaction [29, 42]. Nevertheless, in cases where the pozzolanic materials comprise significant quantities of Al_2O_3, the creation of hydrous silicates is accompanied by the creation of hydrous calcium aluminates [43]. Therefore, changes in the materials' structure led to enhancements in their mechanical strength [44]. The utilisation of pozzolanic materials in the BBCF is notable due to its energy-saving potential and from an ecological point of view.

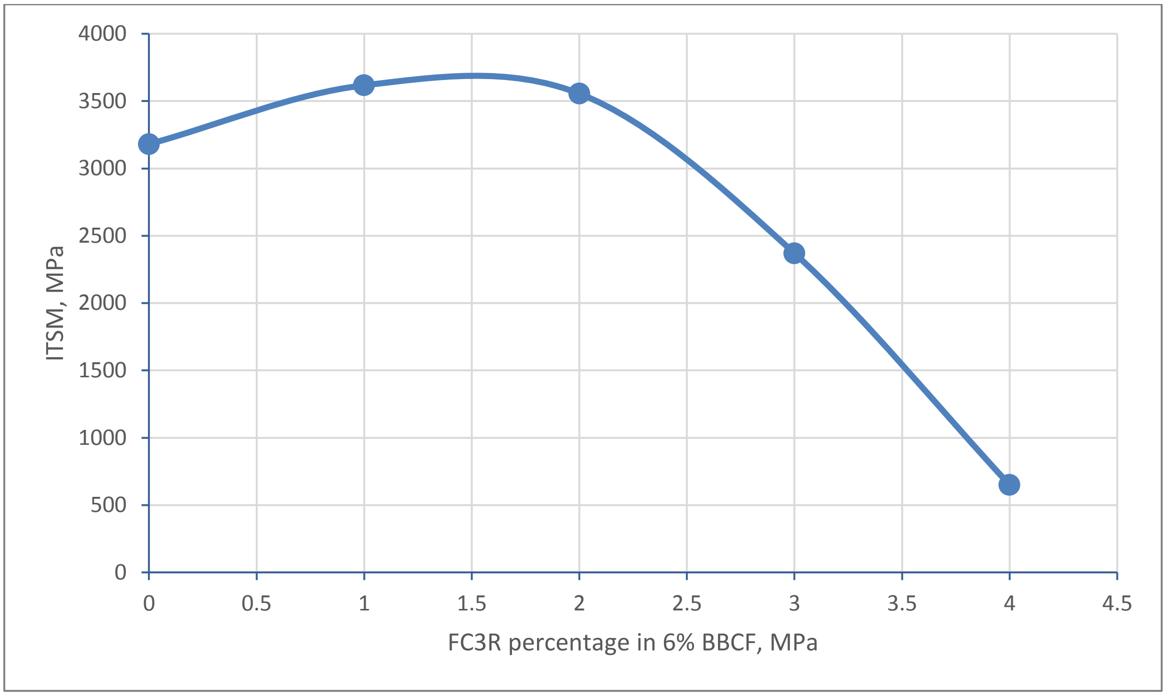

Figure 5. Influence of substitution of HCFA with FC3R on stiffness modulus after 3days

To explore the effect of different percentages of FC3R on 3% HCFA, Figure 6 shows that a significant enhancement was achieved in the stiffness modulus by the inclusion of FC3R at an early age (3 days). The inclusion of 1% of FC3R to the mixtures containing 3% HCFA improved the ITSM by around 160% within 3 days. In addition, mixtures containing 3% HCFA activated by two different percentages of FC3R, i.e., 2% and 3%, achieved more ITSM by approximately 245% and 280% in 3 days, respectively. Moreover, the stiffness modulus for mixtures comprising 3% HCFA with 2% and 3% FC3R exceeded the target value for a 100/150 hot asphalt concrete binder course after 3 days. This development from the HCFA hydration process was enhanced when the high silica-alumina waste material, i.e. FC3R, was applied as it behaved as an activating agent in the hydration process of HCFA.

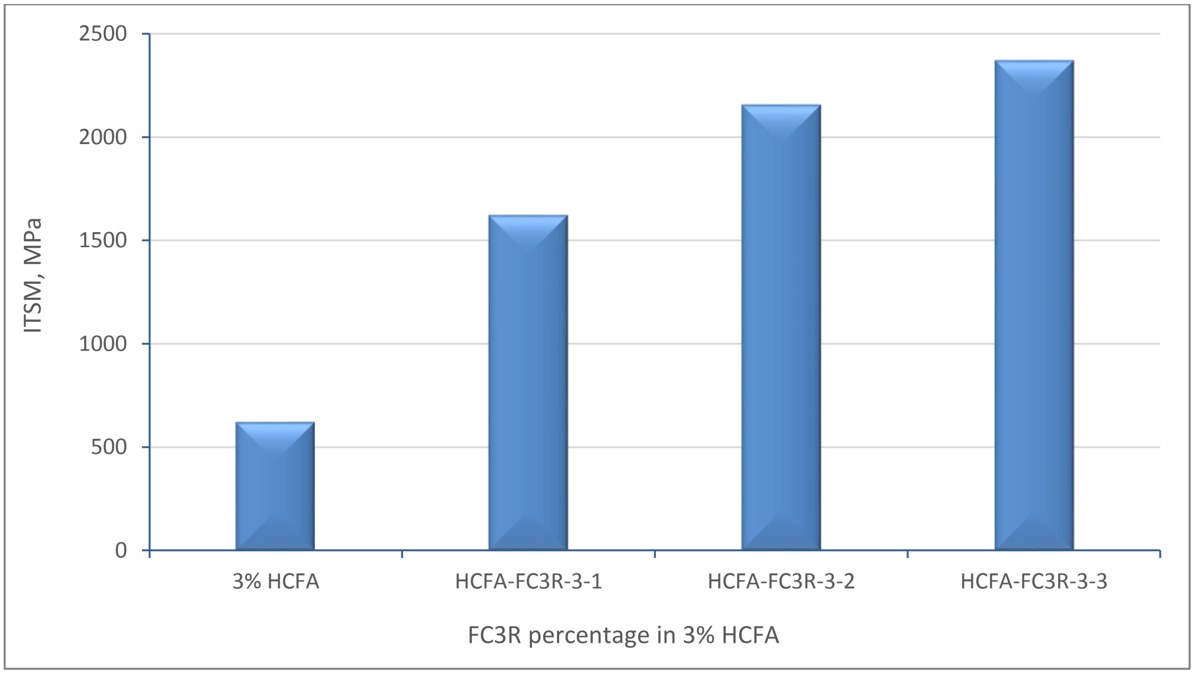

Figure 6. Influence of activating of 3% HCFA by FC3R on stiffness modulus after 3days

It is clear in Figure 7 which show the results at ages 1, 3, 7, 14 and 28 curing days, that increasing the stiffness modulus of a BBCF treated mixture which has a stiffness modulus more than the OPC treated mixture and also equal to or greater than traditional asphalt concrete hot mixes, will produce a suitable material for use as a binder course layer for major heavy trafficked motorways, by reducing the loads transmitted by traffic to the foundation.

In general, it can be seen from Figure 7 that when curing time increases, ITSM develops for HCFA, OPC and BBCF mixtures as a result of the hydration process. The ITSM results improved greatly for BBCF mixtures across all curing times. It worthy to note that BBCF offers more than a 25% increment in ITSM after 1 day when compared to the HCFA as a result of FC3R activation producing more hydration products. It can be seen that BBCF behaviour is the same as that of OPC, however the former offers more ITSM at all curing times.

Therefore, BBCF treated mixtures allow early and temporary trafficking where in situ limitations prohibit the installation of a surface course before elimination of traffic management. These materials also eliminate restrictions applied to road engineers using traditional cold binder course by reducing the curing time to less than 1 day.

It is worth mentioning that the air voids of the CABC mixtures were 10.53% and 10.27% for HCFA and BBCF mixture while the reference cold LF mixture has 10.93%. These findings reveal an enhancement of volumetric properties for CABC mixtures.

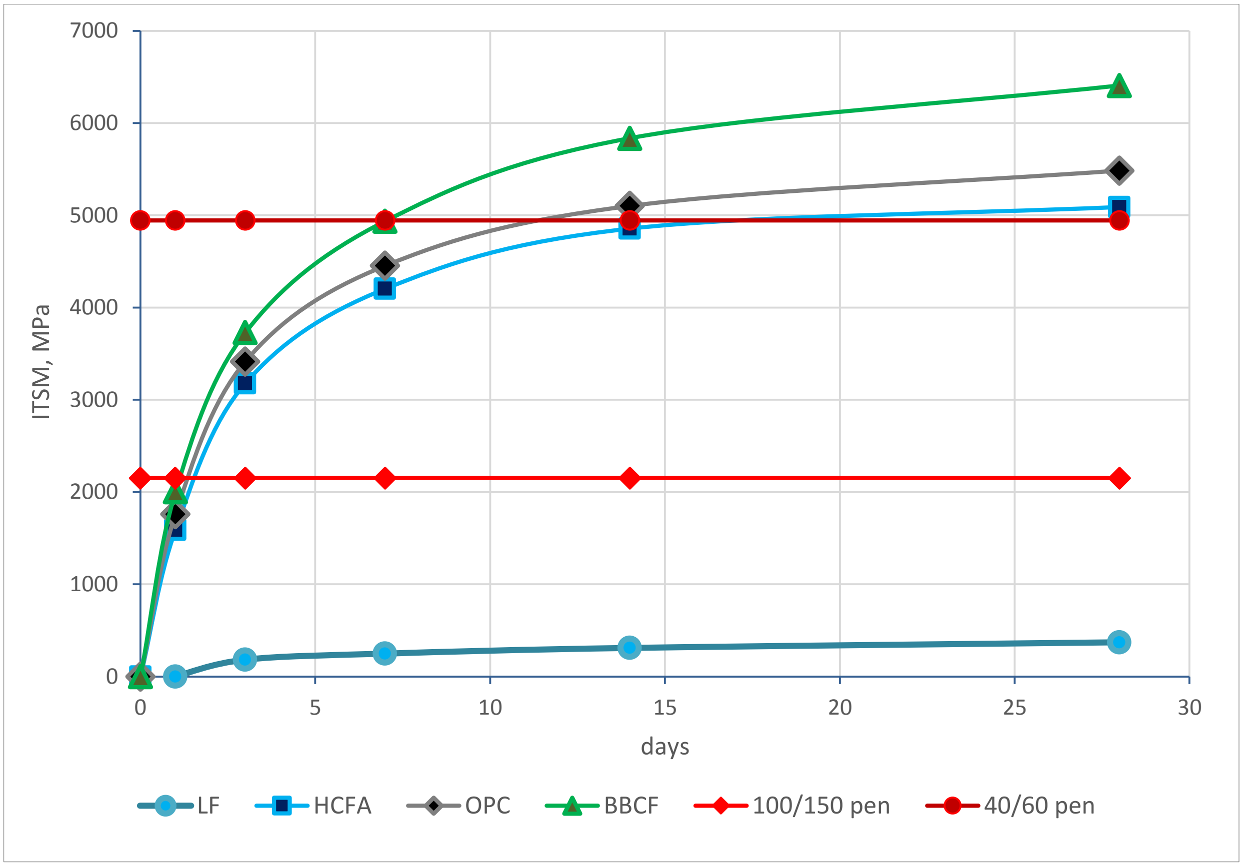

Figure 7. Influence of curing time on stiffness modulus

3.2 Performance in wheel-tracking tests

All samples were exposed to wheel tracking using the wheel-tracking device following BS EN 12697- 22 [39]. Figure 8 illustrates the rut depth at the central point of all slabs as a function of number of cycles. Deformation against number of passes was plotted. From this, it is evident that CACB mixtures with BBCF and HCFA evidenced a maximum proportional rut depth of 1.44 % and 1.59 % after 10,000 wheel passes, which is considerably lower than that of the untreated cold mix asphalt, which had a maximum proportional rut depth of 23.611 % after 10,000 wheel passes. CACB mixtures with HCFA and BBCF have better long-term rut performances than those of the cold mix asphalt treated with OPC, hot AC 20 dense bin 100/150 and hot AC 20 dense bin 40/60, which exhibited maximum proportional rut depths of 1.49 %, 6.697 % and 5.331 % after 10,000 wheel passes, respectively.

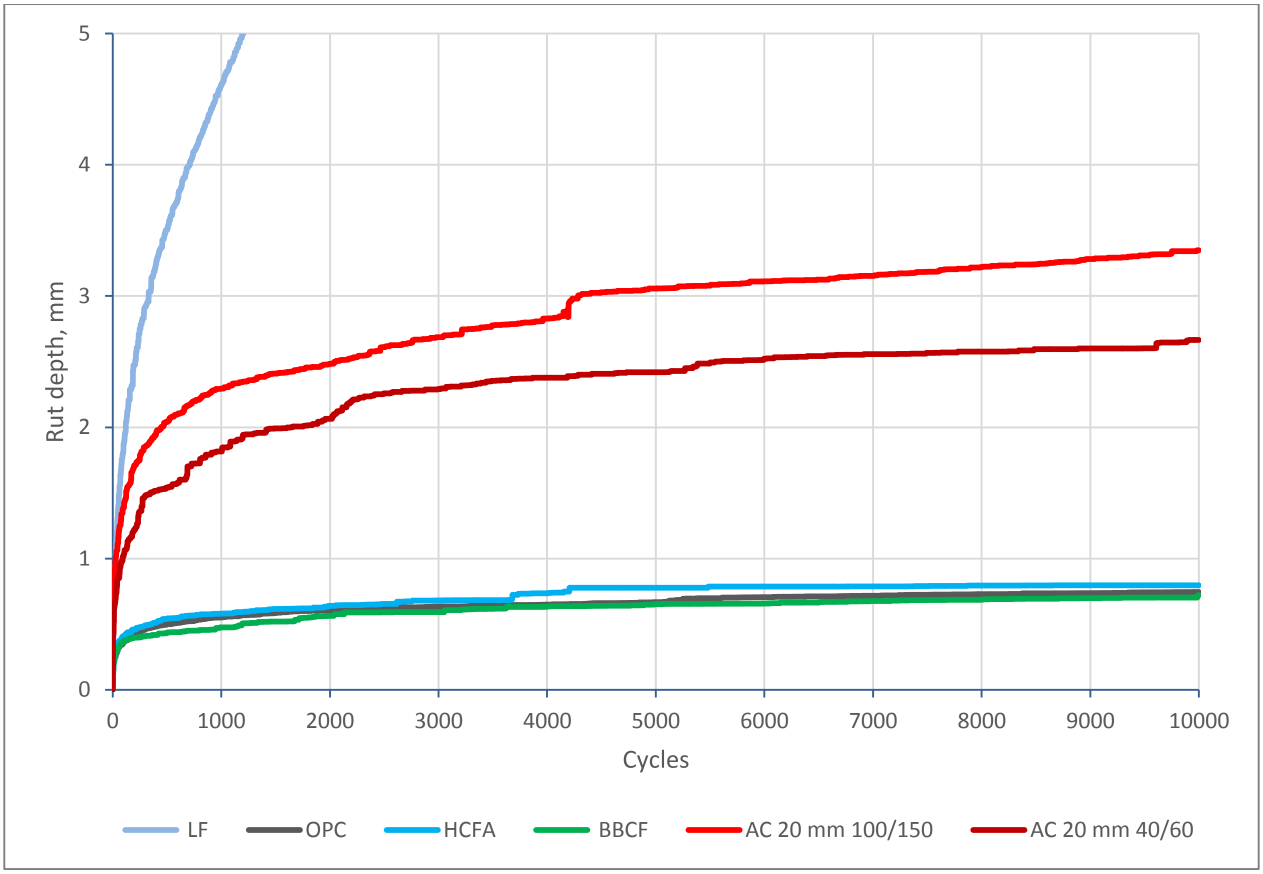

Figure 8. Rut depth evolution

It appears that the rate of rutting in the CACB mixture with BBCF reduces considerably with time. This positive influence of BBCF on the rut resistance of CACB was revealed in specimens with 6% BBCF which had a considerably longer life under the wheel-track test when compared to control samples.

3.3 Fatigue test results

The resistance of the cold mixtures LF, HCFA, OPC and BBCF as well as the hot asphalt concrete binder course mixtures to fatigue cracking were assessed by using the flexural beam fatigue test following the standard BS EN 12697-24 [40]. Constant strain tests were performed at a 150 microstrain level using sinusoidal loading at a frequency of 10 Hz as recommended by Al-Hdabi et al. [10].

The fatigue tests for all mixtures were carried out at a lab temperature of 20°C. Initial flexural stiffness was measured at the 100th load cycle while fatigue life was defined as the number of cycles corresponding to a 50% decrease in the initial stiffness. According to the results presented in Figure 9, it is seen that the BBCF mixture exhibited higher fatigue failure cycles in comparison to their cold counterparts displaying average fatigue failure cycles of 161782, which is 19 times greater than that of the control LF that fails at 8322 cycles. Likewise, the HCF had fatigue failure cycles of 115613, which was 14 times higher than that of the control cold binder course with limestone. The BBCF performance in fatigue tests is logical taking into consideration the stiffness modulus for such mixtures after full curing which is much higher than the reference LF and traditional HMA mixtures.

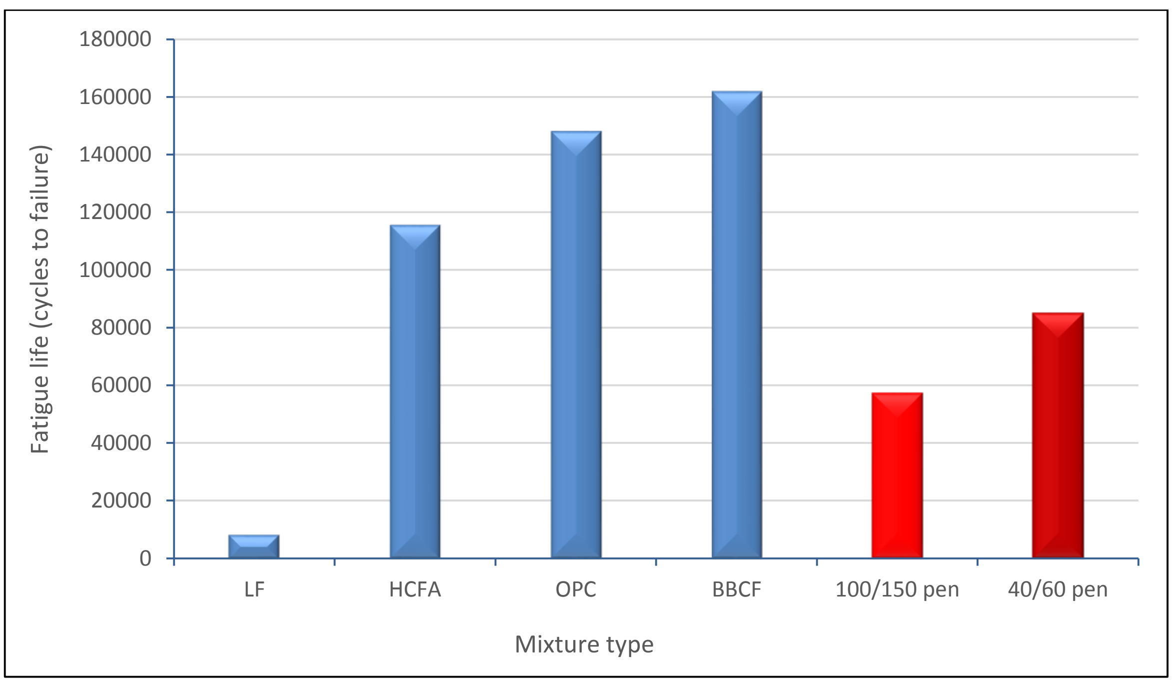

Figure 9. Four-point bending beam fatigue test results

3.4 Performance in water sensitivity

Water is the worst enemy of asphalt-concrete mixtures, as the existence of water may cause early failure of a flexible pavement [45]. The water sensitivity of the cold asphalt concrete binder course mixtures was calculated by finding the SMR in accordance with BS EN 12697-12 [38], to examine the impact of both BBCF and HCFA substitutes for the conventional limestone filler. However, ITSM was used instead of indirect tensile strength as recommended by many researchers such as Al-Busaltan et al. [2], Al-Busaltan et al. [5], Al-Hdabi et al. [37].

Figure 10 shows that the SMR for CACB mixtures with 6% BBCF and 6% HCFA is more than 100%, which indicates an excellent performance for these mixtures attributable to the hydration process of both fillers. Accordingly, moisture sensitivity was eliminated through developing the bond between the binder and the aggregate and generating a stronger bond with the asphalt binder. These results were better than those for hot asphalt concrete binder course specimens and reached the requirements for bituminous materials. It is worth noting that conditioning of the samples at high temperatures further activates the hydration process.

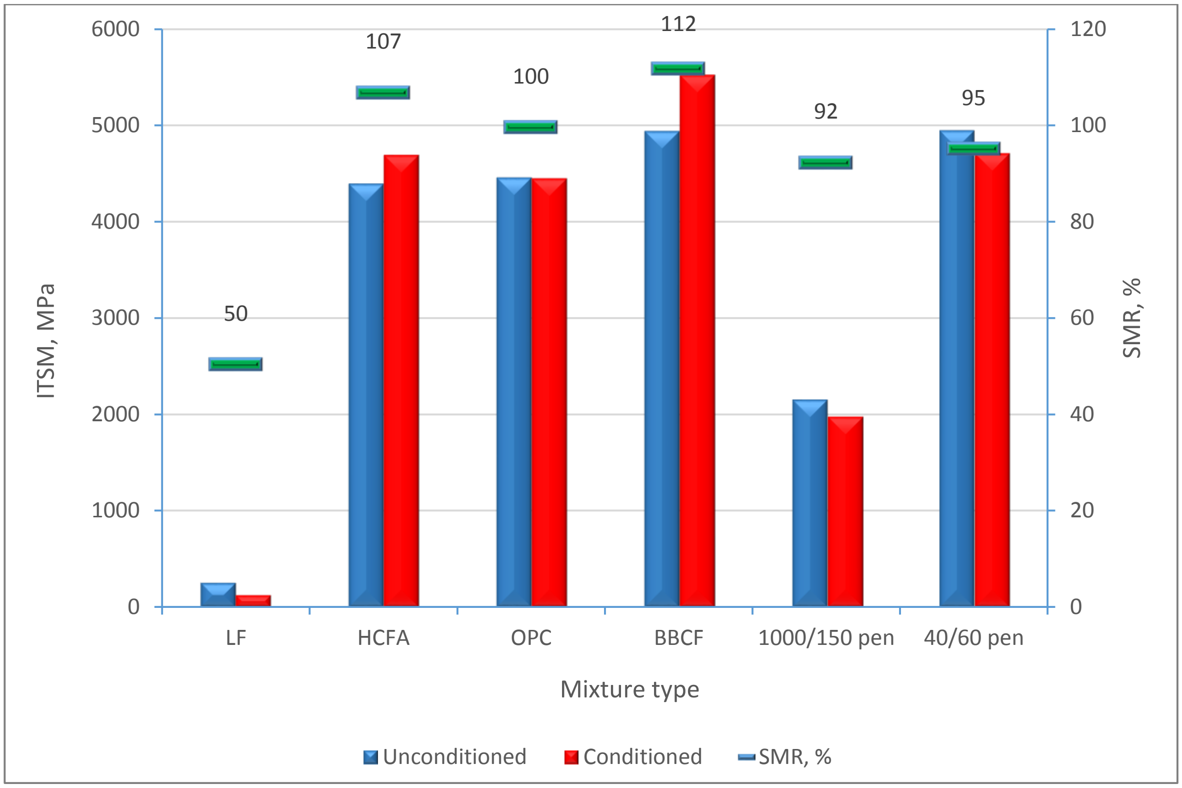

Figure 10. Water sensitivity results

3.3 SEM observation

Firstly, both dry powdered HCFA and FC3R were investigated under SEM in order to identify changes in the material during hydration. The SEM view of HCF particles in Figure 11-a shows that they are flaky and thin, while the morphology for the FC3R particles (Figure 11-b) is agglomerated and non-spherical. Figures 11-c and 11-d display the SEM images of the HCFA and BBCF pastes after 3 days of curing. Significant variations in the microstructural configuration within the hydrates influenced by FC3R is evident in these two figures. In addition, there are distinctions in the morphology of the BBCF sample; the particles started reacting in the BBCF sample. This means that when the HCFA was activated by FC3R, hydration was speeded up. The high stiffness exhibited by samples formulated with BBCF can be associated with a high degree of reaction of this material. However, it is clear that many HCFA particles had not reacted at this early age and acted as a filler material.

The SEM micrograph of the fracture surface of BBCF paste after28 days (Figure 11-f), reveals the generation of a gel-like calcium silicate hydrate (CSH) that creates dense microstructure. As a consequence, the material developed a high level of stiffness after 28 days. The CSH phase is the most significant since it creates the essential cementitious or binding characteristics for the final product. The HCFA sample (Figure 11-e) also produces good hydration products such as Portlandite (CH) and CSH gel, however the latter is lower than in the BBCF sample. It was reported by Nassar et al. [20] that a higher degree of hydration in CBEMs as a result of active fillers can produce a dense internal structure with less porosity.

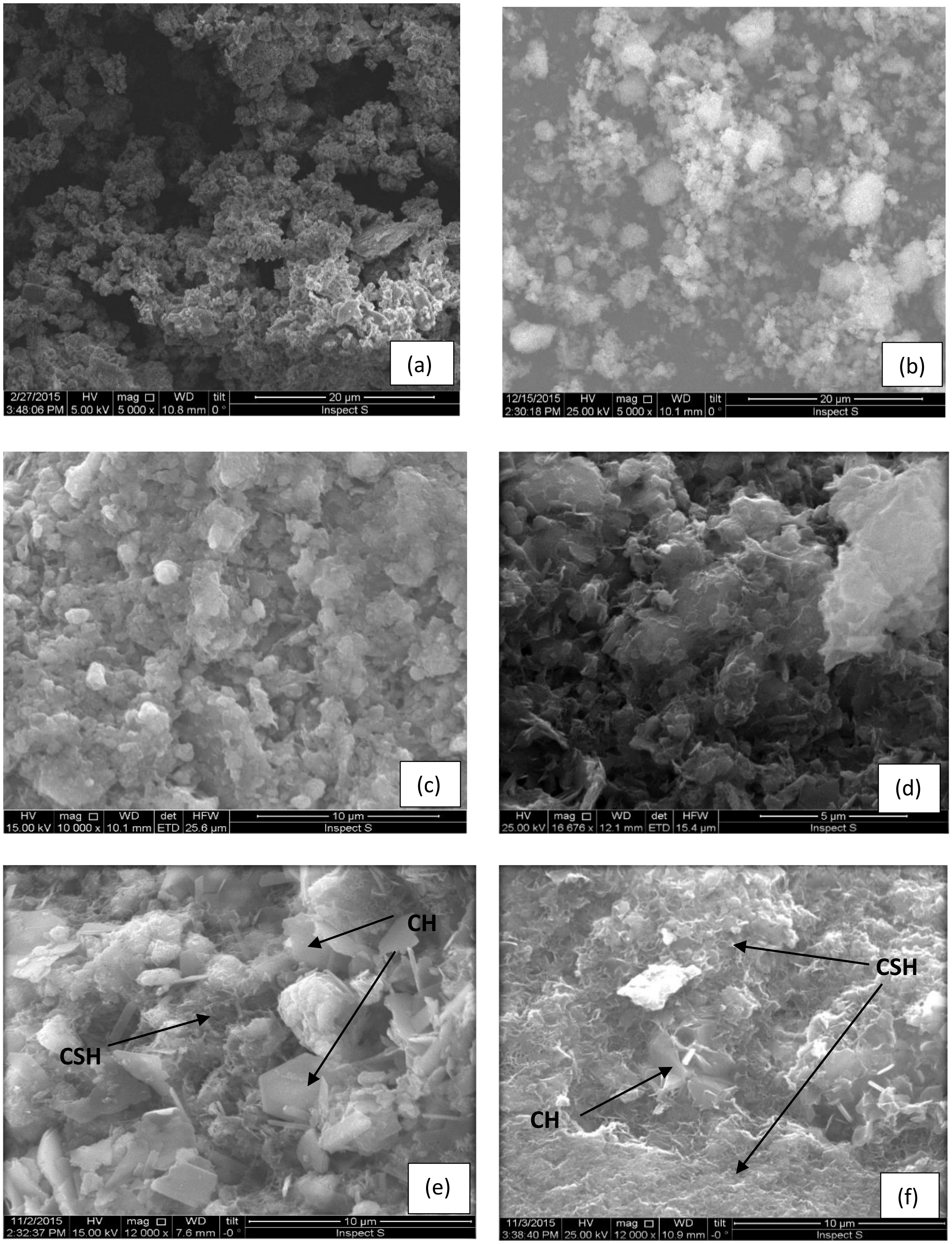

Figure 11. SEM images: (a) morphology of HCFA filler, (b) morphology of FC3R filler, (c) morphology of HCFA paste after 3 days, (d) morphology of BBCF paste after 3 days, (e) morphology of HCFA paste after 28 days, (f) morphology of BBCF paste after 28 days

3. Conclusions

The following conclusions can be drawn:

- Substantial improvements were achieved in the stiffness modulus by replacing the traditional limestone filler with by-product fillers: high calcium fly ash (HCFA) and fluid catalytic cracking catalyst (FC3R). The binary blended cement filler (BBCF) comprising 4.5% HCFA and 1.5% FC3R significantly improved the ITSM in both early and later ages for the BBCF mixture. When compared with the control LF mixture, the stiffness modulus increased more than 17 times after just 3 days. In addition, the new CACB is found to be equivalent to the traditional hot asphalt concrete binder course after short periods of curing.
- A balanced oxide composition in the binary blended cement filler (BBCF) was responsible for advanced pozzolanic reactivity achieved by activating high calcium fly ash with high aluminosilicate waste material (FC3R).
- The BBCF and HCFA treated mixtures have high resistance to water damage. Improved performance in the ITSM test for conditioning samples results in an SMR of more than 100%. The water sensitivity of CACB mixtures containing BBCF is more than two times that of untreated mixtures (LF); this also better than traditional soft and hard hot mixtures.
- The BBCF mixture offered a significantly longer life under the wheel-tracking test when comparing the results with the untreated LF mixture, which showed a high rut depth in the wheel-tracking test reflected in poor permanent deformation resistance. The successful hydration with the binary blended cement filler was responsible for creating advanced stiffness ability in addition to high resistance to permanent deformation demonstrating the possible advantages of using this material on heavily trafficked roads.
- The BBCF mixture revealed a substantial improvement in fatigue life which was 19 times greater in comparison to the reference LF mixture.
- The morphology of the BBCF sample varies considerably with age. BBCF was observed to create larger amounts of hydrated products than HCFA. According to the results achieved in this

research, the formation of hydration products can be noticed at early ages which explains stiffness development.

- Replacing conventional limestone filler with waste materials will decrease cement usage in CBEMs and will offer a positive sustainability effect. In addition, the problems relating to carbon emissions (during production) and mixture temperature maintenance (during transportation and laying) in the case of hot mix asphalt, will be mitigated by using this novel CACB.

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References

[1] Jenkins, K.J., *Mix design considerations for cold and half-warm bituminous mixes with emphasis on foamed asphalt*. 2000, University of Stellenbosch, Stellenbosch: South Africa.

[2] Al-Busaltan, S., Al Nageim, H., Atherton, W. and Sharples, G., *Mechanical Properties of an Upgrading Cold-Mix Asphalt Using Waste Materials.* Journal of materials in civil engineering 2012. 24(12): p. 1484-91.

[3] Doyle, T.A., McNally, C., Gibney, A. and Tabaković, A., *Developing maturity methods for the assessment of cold-mix bituminous materials.* Construction and Building Materials, 2013. 38: p. 524-29.

[4] Thanaya, I.N.A., Forth, J.P. and Zoorob, S.E., *A laboratory study on cold-mix, cold-lay emulsion mixtures.* Proceedings of the ICE - Transport, 2009. 162(1): p. 47-55.

[5] Al-Busaltan, S., Al Nageim, H., Atherton, W. and Sharples, G., *Green Bituminous Asphalt relevant for highway and airfield pavement.* Construction and Building Materials, 2012. 31: p. 243-250.

[6] Serfass, J., Poirier, J., Henrat, J. and Carbonneau, X., *Influence of curing on cold mix mechanical performance.* Materials and Structures, 2004. 37: p. 365-68.

[7] European Asphalt Pavement Association (EAPA), *Asphalt in figures.* EAPA, 2012.

[8] Khalid, H.A. and Monney, O.K., *Moisture damage potential of cold asphalt.* International Journal of Pavement Engineering, 2009. 10(5): p. 311-318.

[9] Oruc, S., Celik, F. and Akpinar, V., *Effect of cement on emulsified asphalt mixtures.* Journal of Materials Engineering and Performance, 2007. 16(5): p. 578-583.

[10] Al-Hdabi, A., Al Nageim, H. and Seton, L., *Performance of gap graded cold asphalt containing cement treated filler.* Construction and Building Materials, 2014. 69: p. 362-69.

[11] Fang, X., Garcia, A., Winnefeld, F., Partl, M.N. and Lura, P., *Impact of rapid-hardening cements on mechanical properties of cement bitumen emulsion asphalt.* Materials and Structures, 2015.

[12] Brown, S.F. and Needham, D., *A study of Cement Modified Bitumen Emulsion Mixtures*, in *Association of Asphalt Paving Technologists Conference Proceeding (AAPT)*. 2000: Reno, Nevada p. 92-121.

[13] Sadique, M., Atherton, W., Dempster, N., Seton, L. and Al-Nageim, H., *A laboratory study for full cement replacement by fly ash and silica fume.* Magazine of Concrete Research, 2012. 64(12): p. 1135-1142.

[14] Ellis, C., Zhao, B., Barnes, J. and Jones, N., *Properties of GGBS-Bitumen Emulsion Systems with Recycled Aggregates.* Road Materials and Pavement Design, 2004. 5:3: p. 373-383.

[15] Thanaya, I.N.A., Zoorob, S.E. and Forth, J.P., *Utilisation of Coal Ashes in Hot and Cold Bituminous Mixtures*, in *International Coal Ash Technology Conference. The Birmingham Hippodrom Theatre, Birmingham, West Midlands, UK*. 2006.
[16] Al-Hdabi, A., Nageim, H.A., Ruddock, F. and Seton, L., *A novel Cold Rolled Asphalt mixtures for heavy trafficked surface course.* Construction and Building Materials, 2013. 49: p. 598-603.
[17] Du, S., *Performance Characteristic of Cold Recycled Mixture with Asphalt Emulsion and Chemical Additives.* Advances in Materials Science and Engineering, 2015. 2015: p. 1-8.
[18] Čížková, Z., Valentin, J., Suda, J., Krpálek, O., Simnofske, D., Mollenhauer, K. and Batista, F., *Report on Durability of cold-recycled mixes: Test procedures for stiffness determination.* CEDR Transnational Road Research Programme. Call 2012: Recycling: Road construction in a post-fossil fuel society, 2015.
[19] Zak, J. and Valentin, J., *Fatigue characterization applicable to cold recycled asphalt mixes.* CEDR Transnational Road Research Programme. Call 2012: Recycling: Road construction in a post-fossil fuel society, 2015.
[20] Nassar, A.I., Mohammed, M.K., Thom, N. and Parry, T., *Mechanical, durability and microstructure properties of Cold Asphalt Emulsion Mixtures with different types of filler.* Construction and Building Materials, 2016. 114: p. 352-363.
[21] Sadique, M., Al-Nageim, H., Atherton, W., Seton, L. and Dempster, N., *Mechano-chemical activation of high-Ca fly ash by cement free blending and gypsum aided grinding.* Construction and Building Materials, 2013. 43: p. 480-489.
[22] Mármol, G., Savastano, H., Bonilla, M., Borrachero, M.V., Monzó, J.M., Soriano, L. and Payá, J., *Ternary Blended Cementitious Matrix for Vegetable Fiber Reinforced Composites.* Key Engineering Materials, 2015. 668: p. 3-10.
[23] Pacewska, B., Wilinska, I. and Kubissa, J., *Use of spent catalyst from catalytic cracking in uidized bed as a new concrete additive.* Thermochimica Acta, 1998. 322: p. 175-181.
[24] European Committee for Standardization, *BS EN 13108: Part 1. Bituminous mixtures materials specification-Asphalt Concrete.* London, UK: British Standards Institution, 2006.
[25] Thanaya, I.N.A., *Improving the performance of cold bituminous emulsion mixtures incorporating waste materials*. 2003, University of Leeds, UK.
[26] Sadique, M. and Al-Nageim, H., *Hydration Kinetics of a Low Carbon Cementitious Material Produced by Physico-Chemical Activation of High Calcium Fly Ash.* Journal of Advanced Concrete Technology, 2012. 10(8): p. 254-263.
[27] Mas, M.A., Tashima, M.M., Payá, J., Borrachero, M.V., Soriano, L. and Monzó, J.M., *A Binder from Alkali Activation of FCC Waste: Use in Roof Tiles Fabrication.* Key Engineering Materials, 2016. 668: p. 411-418.
[28] Lea, F., *The chemistry of cement and concrete*. Vol. 3rd. ed. 1970, London: Edward Arnold.
[29] Sadique, M., Al Nageim, H., Atherton, W., Seton, L. and Dempster, N., *A new composite cementitious material for construction.* Construction and Building Materials, 2012. 35: p. 846-55.
[30] Asphalt Institute, *Asphalt cold mix manual, manual series no. 14(MS-14)* third edition, Lexington, KY 40512-4052, USA., 1989.
[31] European Committee for Standardization, *BS EN 12697: Part 26. Bituminous mixtures-test methods for hot mix asphalt- stiffness.* London, UK: British Standards Institution, 2012.
[32] Al-Hdabi, A., Al Nageim, H., Ruddock, F. and Seton, L., *Development of Sustainable Cold Rolled Surface Course Asphalt Mixtures Using Waste Fly Ash and Silica Fume.* Journal of Materials in Civil Engineering, 2014. 26(3): p. 536-543.
[33] European Committee for Standardization, *BS EN 12697: Part 33.Bituminous mixtures - Test methods for hot mix asphalt - Specimen prepared by roller compactor.* London, UK: British Standards Institution, 2003.
[34] Monney, O.K., Khalid, H.A. and Artamendi, I., *Assessment of Fracture Properties of Emulsified Asphalt Mixtures.* Road Materials and Pavement Design, 2007. 8(1): p. 87-102.
[35] Al-Hdabi, A., Al Nageim, H., Ruddock, F. and Seton, L., *Enhancing the Mechanical Properties of Gap Graded Cold Asphalt Containing Cement Utilising By-Product Material.* Journal of Civil Engineering and Architecture, 2013. 7(5): p. 916-921.
[36] Ojum, C.K., *The Design and Optimisation of Cold Asphalt Emulsion Mixtures*. 2015, University of Nottingham: Nottingham, UK.
[37] Al-Hdabi, A., Nageim, H.A., Ruddock, F. and Seton, L., *Laboratory studies to investigate the properties of novel cold-rolled asphalt containing cement and waste bottom ash.* Road Materials and Pavement Design, 2013. 15(1): p. 78-89.

[38] European Committee for Standardization, *BS EN 12697: Part 12. Bituminous mixtures-test methods for hot mix asphalt-determination of the water sensitivity of bituminous specimens.* London, UK: British Standards Institution, 2008.
[39] European Committee for Standardization, *BS EN 12697: Part 22. Bituminous mixtures -Test methods for hot mix asphalt - Wheel tracking test methods for hot mix asphalt* London, UK: British Standards Institution, 2003.
[40] European Committee for Standardization, *BS EN 12697-24. Bituminous mixtures-Test methods for hot mix asphalt- Part 24: Resistance to fatigue.* London, UK: British Standards Institution, 2012.
[41] Sebaaly, P.E., Hajj, E.Y., Sathanathan, T. and Shivakolunthar, S., *A comprehensive evaluation of moisture damage of asphalt concrete mixtures.* International Journal of Pavement Engineering, 2015: p. 1-14.
[42] Lothenbach, B., Scrivener, K. and Hooton, R.D., *Supplementary cementitious materials.* Cement and Concrete Research, 2011. 41(12): p. 1244-1256.
[43] Morsy, M.S., Galal, A.F. and Abo-El-Enein, S.A., *Effect of temperature on phase composition and microstructure of artificial pozzolana-cement pastes containing burnt kaolinite clay.* Cement and Concrete Research, 1998. 28(8): p. 1157-1163.
[44] Chan, S.Y.N. and Xihuang, J., *Comparative study of the initial surface absorption and chloride diffusion of high performance zeolite, silica fume and PFA concretes.* Cement & Concrete Composites, 1999. 21: p. 293-300.
[45] Sharma, V., Chandra, S. and Choudhary, R., *Characterization of Fly Ash Bituminous Concrete Mixes.* Journal of Materials in Civil Engineering, 2010. 22(12).