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High performance cold asphalt concrete mixture for binder course using alkali-activated binary blended cementitious filler

High Performance Cold Asphalt Concrete Mixture for Binder Course Using Alkali-Activated Binary Blended Cementitious Filler

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Abstract

A slow rate of curing and the long time necessary to achieve full strength has led cold asphalt mixes (CAM) to be considered poorer in comparison to hot mix asphalt over the last decades. This piece of research aimed to develop a new fast-curing and environmentally friendly cold asphalt concrete for binder courses mixture (CACB). It has the same gradation as that of traditional hot asphalt concrete mixtures but incorporates a binary blended cementitious filler (BBCF) containing waste, high calcium fly ash (HCFA) and fluid catalytic cracking catalyst residue (FC3R) activated by a waste alkaline NaOH solution. The research concludes that incorporating an alkali activated binary blended cementitious filler (ABBCF) with CACB significantly improves the mechanical properties and water susceptibility. In addition, the high performance ABBCF mixture has a substantial lower thermal sensitivity than traditional hot asphalt concrete binder course mixtures. SEM analysis revealed that the main crystallisation had taken place at an early stage of the new ABBCF. More significantly, the new CACB mixture has a comparable stiffness modulus with the traditional asphalt concrete binder course after a very short curing time (less than one day).

Keywords:

Alkali activation

Binder course

Cold asphalt mix

Emulsion

Fluid catalytic cracking catalyst

High calcium fly ash

Microstructure

Rutting

Stiffness modulus

Water sensitivity

1. Introduction

Eco-friendliness, energy efficiency and cost effectiveness associated with safety are significant drivers accountable for the development of cold bituminous emulsion mixtures (CBEM) as a substitute for hot mix asphalts (HMA). CBEMs are popular types of cold asphalt mixtures (CAMs) that are produced with no application of heat in comparison to traditional HMA. Consequently, this technology contributes to the protection of environmental and occupational health and safety Needham [1], [2]. In contrast to HMAs, CBEMs do not achieve their ultimate strength and other associated properties as quickly after application. CBEMs are identified to have low early strength, long curing times, the resultant mixtures having quite high porosites [3]. When comparing emulsion mixtures to HMA in general, they are of a relatively low quality as demonstrated by Ibrahim and Thom [4].

CBEM technology for road pavements has been employed in several countries. The USA and France have been using CBEMs since the 1970's and seem to have a substantial bank of knowledge about the performance of these mixtures [5]. The annual levels of manufacture has reached 1.5 million tonnes in France [6]. However, using cold emulsified asphalt as structural layers which require a longer curing time for such materials to reach their full strength after construction is restrictive, mainly in the UK, because of high sensitivity to rainfall by these mixes in the early stages of installation [7]. Characteristics exhibited by emulsion bound mixtures continuously change (stiffness modulus, rutting resistance, water sensitivity, fatigue resistance, etc.) until they reach a steady state at a fully cured condition, although they may still contain a low amount of residual water. However, evolutional characteristics have been exhibited by CAMs, especially during their early life, where early cohesion is low, increasing gradually [8].

The mechanical properties of CBEMs have been examined by many researchers such as Terrel and Wang [9] who found that the addition of cement to emulsion-treated mixes resulted in an acceleration in the rate of the development of resilient modulus. Another study was implemented by Head [10] in order to improve the Marshall stability of modified cold asphalt mix. He found that with the addition of 1% Ordinary Portland Cement (OPC), the Marshall stability of modified cold asphalt mixes improved approximately 3-fold compared with un-treated mixes. An examination of the role of cement in emulsion-treated mixes to enhance the slow improvement of strength of these mixtures was carried out by Schmidt et al. [11]. They concluded that when cement was added to the aggregate at the time the asphalt emulsion was combined, mixes cured faster, additional resilient modulus (Mr) developed more quickly and there was a higher water damage resistance. Previous studies on the mechanical properties of three-phase cement–asphalt emulsion composites (CAEC) reported that most of the properties of both cement and asphalt were present in CAEC; longer fatigue life and low sensitivity to temperature in cement concrete and higher toughness and flexibility in asphalt concrete [12]. Brown and Needham [13] carried out a study on cement modified emulsion mixtures where the prime aim was the evaluation of the influence of adding OPC in emulsified mixes. They used a granite aggregate grading in the middle of 20mm dense bituminous macadam with a single slow-setting emulsion. They concluded that the OPC addition enhanced the mechanical properties namely: stiffness modulus, resistance to permanent deformation and the fatigue strength of the emulsified mixes. Oruc et al. [7] performed an investigation to evaluate the mechanical properties of emulsified asphalt mixtures including 0-6% OPC which was substituted for mineral filler. A significant improvement was revealed with the addition of a high percentage OPC, leading them to speculate that cement modified asphalt emulsion mixtures might be utilized as structural layers. Al-Hdabi et al. [14] carried out experiments on the mechanical properties and water damage resistance of cold-rolled asphalt (CRA) incorporating OPC as a substitute to conventional filler and waste bottom ash (WBA). The results showed a considerable enhancement in stiffness modulus and uniaxial creep tests in addition to water sensitivity.

Other research implemented by Fang et al. [15] investegated the effect of cement on the rheology and stability of rosin-emulsified anionic bitumen emulsions. They used optical microscopy to examine how bitumen emulsion breaks and bitumen droplets morphology in cement and filler. They concluded that cement, unlike limestone filler, reacts with rosin emulsifiers leading to flocculation and the partial coalescence of bitumen emulsions. Further to this, Gómez-Meijide and Pérez [16] proposed a new methodology for the global study of the mechanical properties of CAMs. They found that bitumen materials stabilized with emulsion and recycled aggregates from construction and demolition (C&D) are more flexible, showing improved resistance to permanent deformation and similar stress failures in comparison to mixtures with natural aggregates. That said, a higher water and bitumen content is needed [17].

Cement has been used widely in CBEMs, but cement production is accountable for 5% of global greenhouse gases (GHG) [18]. However, CBEMs can be further developed when manufactured with waste materials thus addressing environmental and economic concerns. That said, it is necessary to replace cement with waste materials that has the same or better performance. Research by Ellis et al. [19] considered a range of storage grade macadams consisting of recycled aggregates from different sources bound by bitumen emulsion and Ground Granulated Blastfurnace Slag (GGBS). They concluded that stiffness and strength can develop when GGBS is incorporated in high humidity conditions. Thanaya et al. [20] conducted experiments to use pulverized fly ash (PFA) as a filler in cold mix at full curing conditions, finding the cold mix stiffness equivalent to HMA. Al Nageim et al. [21] studied the addition of OPC and fly ash to CBEMs as a filler replacement. They conducting an experiment to show the development of mechanical properties in CBEM's and to identify the possibility of replacing OPC with fly ash. Recently, Nassar et al. [22] conducted investigations to improve the performance of Cold Asphalt Emulsion Mixtures (CAEMs) using binary and ternary blended fillers (BBF and TBF). They used OPC, fly ash and GGBS for the BBF while TBF was obtained by incorporating silica fumes with BBF. They concluded that the mechanical and durability properties indicated that TBF was more appropriate than BBF for the manufacture of CAEMs. In addition, they stated that a TBF mixture would be effective in road pavements which were subjected to harsh conditions both in hot and cold weathers.

Sadique et al. [23] aimed to develop a new cementitious material through the activation of a high calcium fly ash by a different alkali sulphate rich fly ash. They found that the cement free activation of fly ash was very effective. They revealed that the presence of a structure comprising Ca, Al, K and Si with high pH in two types of fly ashes, has the ability to break the glassy phase in the cement free system. In addition, Sadique et al. [24] performed a study to explore the pozzolanic reactivity of calcium rich fly ash by blending and grinding it in a cement-free system. They reported that the hydration effects and strength enhancement in the new blend were comparable to cement.

Fluid catalytic cracking catalyst residue (FC3R) is an industrial by-product generated from the fluid catalytic cracking process in petrol refineries. Pacewska et al. [25] investigated the hydration of cement paste as a function of adding spent catalyst residue to address catalytic cracking, reporting on the pozzolanic nature of the spent catalyst. They found both spent catalyst and microsilica to be similar when combined with $Ca(OH)_2$, and that the process of hydration was highly exothermic promoting fast setting of the cement paste. Mas et al. [26] studied the mechanical properties of mortars and roof tiles using a fluid catalytic cracking catalyst residue with various mixtures, varying the proportions of Na OH and waterglass. They concluded that the use of geopolymers in the design of a new product with reduced CO_2 emissions was feasibly and sustainable in the construction sector.

Chemical activation suggests that some chemicals can be used to activate the reactivity of cementitious components [27]. Alkaline activated materials have been shown to have enhanced higher level mechanical characteristics in comparison to cement. Consequently, the alkali activation of fly ash offers potential financial and environmental cost savings when used as a cement replacement [28]. Al-Hdabi et al. [29] stated that the incorporation of high alkali waste material as a filler replacement in CBEMs provides an ambient environment to activate the hydration process of the incorporated cementitious constituents.

There is demand for the development of sustainable novel CBEMs which use waste filler materials activated by alkali waste solutions and as such the main aim of this study has been to develop a fast-curing Cold Asphalt Concrete for Binder course (CACB) mixtures, to examine the effect of waste filler as a filler substitution on the performance of CACB and subsequently to compare the characteristics of this with conventional hot asphalt concrete binder course mixtures. There is limited research on the incorporation of waste materials in the production of CBEM for binder courses in road pavements. None of these studies has investigated waste binary filler systems activated by waste alkali solutions. Such binary blended cement filler (BBCF) can be activated to achieve higher strength values within a short period of time, eliminating the problems relating to curing time and low early strength of the CBEMs for binder courses.

This research has been carried out using 100% replacement of traditional mineral filler (limestone filler) by waste materials as the use of these materials is beneficial both in terms of being environment friendly as well as offering substantial economic advantages. Enhancements in mechanical properties were evaluated by using the indirect tensile stiffness modulus, a high-temperature wheel tracking test and thermal susceptibility. At the same time, a water susceptibility test was performed to examine the durability of the new CACB. Scanning Electron Microscopy (SEM) observation has also been applied to investigate the microstructure of the new CACB mixtures.

2. Materials

2.1. Aggregates

The coarse and fine aggregate utilized in this research to manufacture all the mixtures comprised of crushed granite from Carnsew Quarry at Mabe, Penryn, UK. This is usually used to produce hot asphalt concrete mixtures, so the selection of aggregate gradation followed hot mix asphalt guidelines. The physical properties of the coarse aggregate were: apparent density 2.67 Mg/m^3 and water absorption 0.8% while for the fine aggregate: apparent density 2.65 Mg/m^3 and water absorption 01.7%. A sieve analysis according to the standard BS EN 933-1 [30] was performed on the aggregate. The aggregate structure permitted a curve to be established following EN 13108-1 [31]. Figure 1 shows the particle size distribution curve of the aggregate where a dense aggregate gradation for asphalt concrete binder course AC-20 was used.

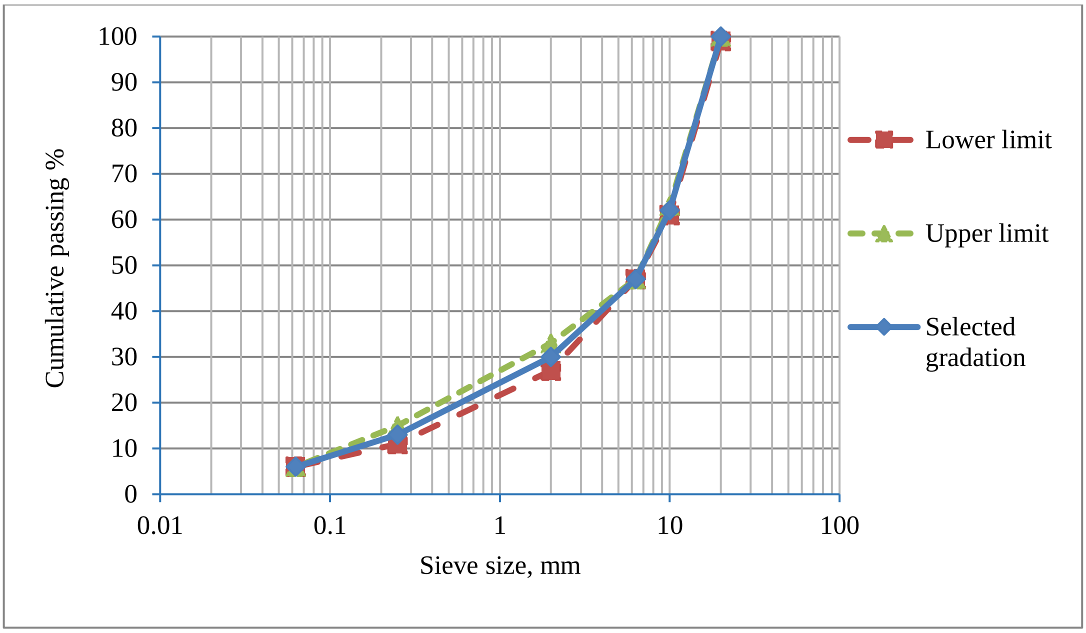

Figure 1. AC 20 mm dense binder course aggregate gradation

2.2. Chosen gradation

After discussion with the Liverpool Centre for Materials Technology (LCMT) industrial partners', the author and supervisory team aimed to develop a new CBEM with a continuously grade, traditionally used for asphalt concrete binder course mixtures. Asphalt concrete AC 20 dense binder course mixture was used to produce reference mixtures (cold and hot). Asphalt concrete is a continuously graded mixture, a prominent type of mixture used as a binder course and base in road pavements in the UK. Its strength is derived from the interlocking of coated aggregates which provides the principal mechanism for the material to transmit load.

2.3 Bitumen emulsion and asphalt

To prepare the CACB mixtures, cationic slow setting bitumen emulsion (C60B5) was used. This kind of emulsion is designed for use in road pavements and common maintenance applications. Thanaya [32] confirmed that cationic emulsion is preferred because of its capability to coat the aggregate and to guarantee high adhesion between aggregate particles. The bitumen base of the emulsion is 100/150 pen while the bitumen residual content is 60%. In addition, two traditional binders consisting of 100/150 and 40/60 penetration-grade bitumen with a softening point of 43.5°C and 51.5°C respectively were utilized for the control hot asphalt concrete binder course mixture preparations.

2.4. Filler materials

Two different wastes from the industry were employed and analysed as filler replacements in this research: high calcium fly ash (HCFA) which is obtained from power generation plants through combustion between 850°C and 1100°C using a fluidised bed combustion (FBC) system. A second high aluminosilicate waste material, fluid catalytic cracking catalyst residue (FC3R), was also used. A typical commercial limestone filler (LF) and a commercial Ordinary Portland cement (OPC) were utilized as control mixtures for comparison purposes during the research. Chemical and mineralogical analyses were carried out on the waste materials in order to make a qualitative assessment of the geometric features of the filler particles. The chemical compositions by energy dispersive X-ray fluorescence (EDXRF) spectrometer are given in Table 1.

Table 1. Chemical analysis of the chosen filler materials, %.

Filler type	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

Table 1 illustrates the high calcium content of the HCFA used, showing a good ratio of SiO_2 and $Al2O_3$. These outcomes are consistent with those of Sadique and Al-Nageim [33]; however, the reported quantity of CaO in the current research is higher. The main oxides in the FC3R are Al_2O_3 and SiO_2, this in agreement with the results of Mas et al. [26], Mármol et al. [34]. It has been reported that calcium hydroxide reacts with pozzolanic materials (SiO_2 and Al_2O_3) in the moisture present at normal temperatures to form calcium silicate hydrate (CSH) gel [35]. Lea [36] stated that soluble SiO_2 and Al_2O_3 present in the glass phase of pozzolanic materials can react with $Ca(OH)_2$ released through the hydration of cement to make an extra CSH gel that improves the mechanical strength of hardened concrete structures.

The two waste material's mineralogy was assessed using an X-ray diffraction (XRD) method (Rigaku Miniflex diffractometer). Figure 2 shows that the sample of HCFA is crystalline as it contains sharp peaks without significant background noise. The major crystal peaks identified were: lime (CaO), calcite ($CaCO_3$), mayenite ($Ca_{12}Al14O_{33}$), merwinite ($Ca_3Mg[SiO_4]$) and gehlenite ($CaAl[Al,SiO_7]$). The diffraction pattern of FC3R illustrates that the material has very low crystalline peaks with an amorphous nature. Consequently, it will show high reactivity during the hydration process and can be used as an activator material as shown by the diffraction pattern in Figure 3.

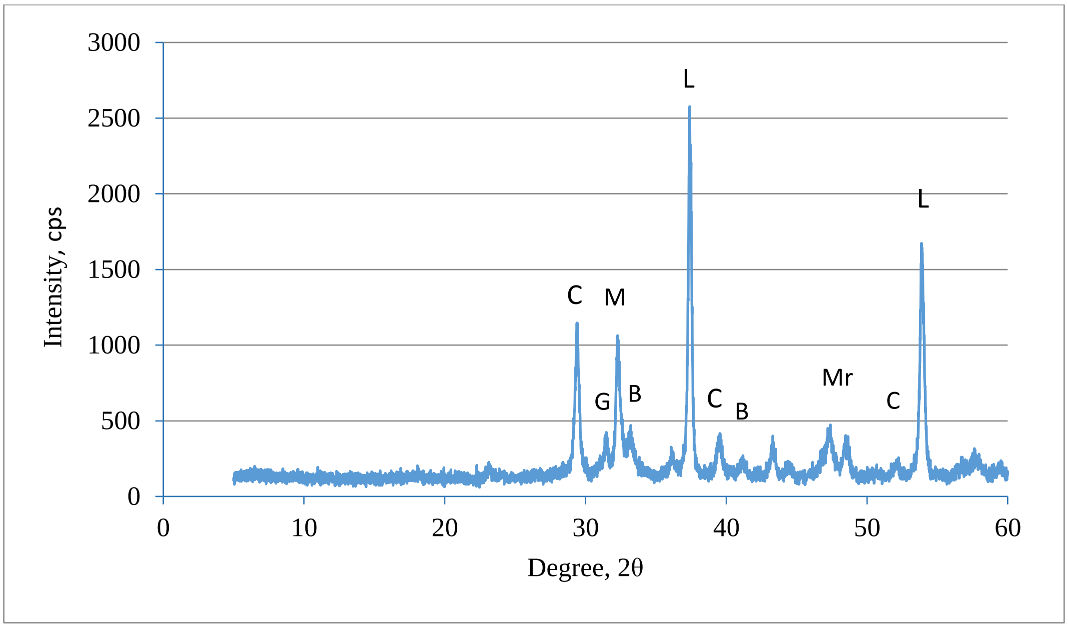

Figure 2. X-ray diffraction pattern of the HCFA

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

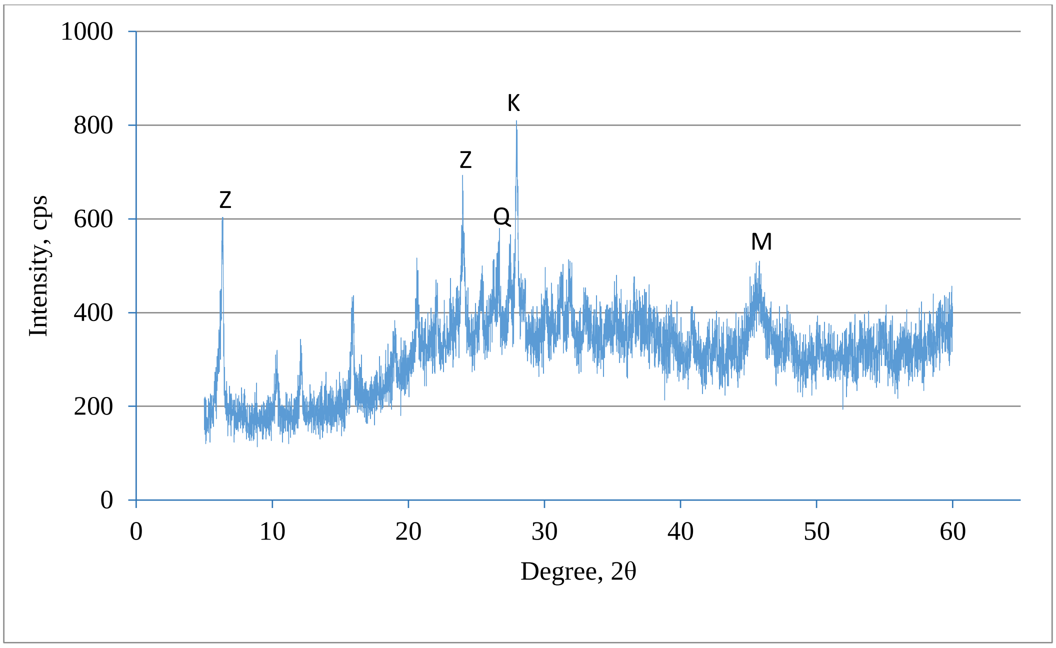

Figure 3. X-ray diffraction pattern of the FC3R

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

2.5. Sodium hydroxide (NaOH) alkali waste

A sodium hydroxide (NaOH) alkali waste solution produced from an acid neutralisation plant containing ≤ 8% NaOH in water, was used as the alkali activator. This caustic waste product was supplied by Lambson Ltd originating at Magnesium Electron Ltd. It is a waste by-product of the extraction of magnesium from sea water.

3. Samples preparation and conditioning

Currently, there is no agreed design mixture for CBEM neither in the UK nor worldwide, but mix design procedures for CBEM have been presented by some authorities and researchers [32, 37, 38]. The Marshall mix design procedure, as specified by the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14)) [37], was used in this investigation for designing the cold asphalt mixtures.

Firstly, aggregate gradation was chosen as outlined in section 2.2. Next, the initial emulsion content was determined by using an empirical equation that governs the aggregate gradation as recommended by the Asphalt Institute Manual MS-14. This was followed by determination of the pre-wetting water content (PWWC) where the coating ability of the bitumen emulsion to aggregates is extremely sensitive to the PWWC. Various pre-wetting water contents were examined, the lowest ratio consistent with satisfactory coating then adopted. In addition, indirect tensile stiffness modulus tests (ITSM) were performed to choose the optimum emulsion content following the standard BS EN 12697-26 [39]. This is the only change to the previously mentioned method: substitution of the Marshall test by the ITSM test. Finally, a mix density test was carried out to decide the optimum total liquid content at compaction (OTLCC) (i.e. emulsion plus pre-wetting water contents providing the highest mix indirect tensile strength and density). As a result, the PWWC, OTLCC and optimum residual bitumen content were found to be 3.5%, 14% and 6.3%, respectively. These findings are comparable to those published by Al-Busaltan et al. [40], Al-Hdabi et al. [41].

The materials were mixed in a Hobart mixer as shown in Figure 4. Aggregate, filler and pre-wetting water were incorporated and mixed for 60 seconds at a low speed. After that, Bitumen emulsion was introduced at a steady rate over the next 30 seconds, the mixing process continuing for a further 120 seconds at the same speed. Following this the mixed materials were placed into moulds and immediately subjected to compaction with 100 blows of a standard Marshall hammer (impact compactor), 50 on each side of the samples. It was reported by Nassar et al. [22] that Marshall compaction is an accepted procedure used to create a suitably dense material. The samples were left for 24 hours at 20°C in the moulds and de-moulded the next day. All the specimens were left in the lab at 20°C and tested for Indirect Tensile Stiffens Modulus (ITSM) test at various ages, i.e. 1, 3, 7, 14 and 28 days. In addition, four control mixtures were prepared and tested for comparison purposes. The first control mixture was an untreated mix with traditional limestone filler (LF) which has the same design as other CACB mixtures. A mixture treated with 6% OPC composed the second. Two traditional asphalt concrete binder course mixtures were also produced for comparison purposes. To manufacture the hot asphalt concrete binder course, the laboratory mixing temperatures were fixed at 150-160°C and 160-170°C for the 100/150 pen and 40/60 pen, respectively. The proportions of the mixture by percentage of Marshall samples are summarized in Table 2.

Figure 4. Photograph of mixer

Table 2. Details of the mix proportions of CACBs.

Mixture types	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%
ABBCF mix	4.5% HCFA +1.5% FC3R	10.5%	3.5% waste NaOH solution
<u>Control mixtures</u>			
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	-

For wheel track tests, slab samples were produced measuring 400 mm long, 305 mm wide and 50 mm thick compacted at an ambient temperature in a steel mould using a Cooper Technology Roller Compactor device following the standard BS EN 12697-33 [42]. Wheel-track tests were performed for all cold mixtures at full curing conditions in two stages: slab samples were left in their mould for 1 day at lab temperature 20°C, this the first stage. Stage two involved placing the slab samples in a ventilated oven at 40°C for 14 days to reach their constant mass. This curing protocol was recommended by Thanaya [32] to guarantee that an entirely cured condition was reached. Finally, the slabs samples were allowed to cool at lab temperatures before starting the process of conditioning. Table 3 illustrates the abbreviations used in this research and their meaning.

Table 3. List of abbreviations

Abbreviations	Meaning
100/150 pen	Hot asphalt concrete binder course with 100/150 pen
40/60 pen	Hot asphalt concrete binder course with 40/60 pen
ABBCF	Alkali activated binary blended cementitious filler
BBCF	Binary blended cementitious filler
CACB	Cold asphalt concrete for binder courses mixture
CAM	Cold asphalt mix
CBEM	Cold bituminous emulsion mixture
FC3R	Fluid catalytic cracking catalyst residue
HCFA	High calcium fly ash
ITSM	Indirect tensile stiffness modulus
LF	Limestone filler
OPC	Ordinary Portland cement
OTLCC	Optimum total liquid content at compaction
PWWC	Pre-wetting water content

4. Experimental program and tests performed

A variety of laboratory tests were carried out to evaluate the performance of the new CACB. The main laboratory programme covered the stiffness modulus, temperature susceptibility, rutting resistance and resistance to moisture damage assessed using the indirect tensile stiffness modulus test, wheel tracking tests at high temperatures and stiffness modulus ratio, respectively. In addition, scanning electron microscopy was employed to investigate the microstructure of the new binder paste.

4.1 Indirect tensile stiffness modulus (ITSM) test

To assess the load bearing capacity of the layer manufactured from the asphalt mix, the indirect tensile stiffness modulus was determined. The ITSM tests were carried out at 20°C and were conducted on cylindrical specimens following the BS EN 12697-26 [39] using a Cooper Research Technology HYD 25 testing machine as shown in Figure 5. Numerous researchers such as Al-Hdabi et al. [14], Nassar et al. [22], Monney et al. [43], Al-Busaltan et al. [44] have measured ITSM in order to evaluate the stiffness modulus of CBEMs. At minimum of five samples have been used for each mixture type. The modulus defines the vertical force under controlled stress. The test conditions were as shown in Table 4. Incorporation of the HCFA was achieved through full replacement of the conventional limestone filler. Consequently, HCFA was selected for 100% replacements, while FC3R was used as supplementary cementitious material to produce a binary blended cement filler (BBCF). Sodium hydroxide (NaOH) alkali waste was then incorporated as a replacement for the pre-wetting water content to produce an alkali activated binary blended cement filler (ABBCF). Following their preparation, the samples were kept in the lab up to the time of testing. ITSM test was conducted at 1, 3, 7, 14, 21 and 28 days.

The ITSM test was also performed at various testing temperatures, namely 5, 20 and 45 °C to investigate the susceptibility of CACB mixtures and control mixtures to temperature.

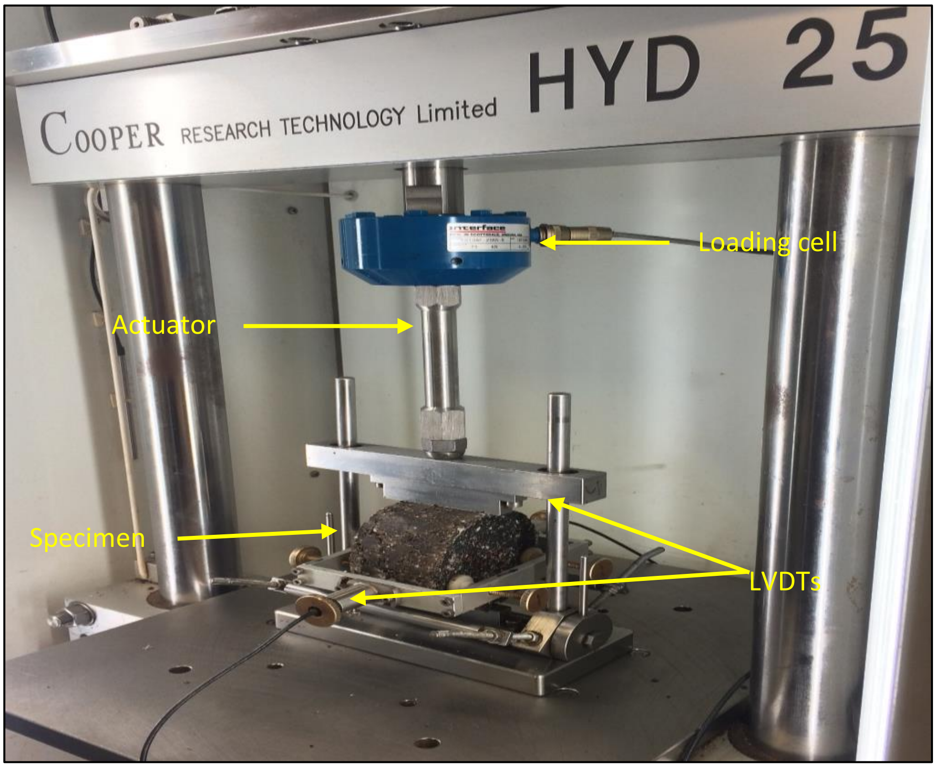

Figure 5. ITSM Apparatus machine

Table 4. Conditions of the ITSM Test

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
Specimen thickness, (mm)	63± 3

4.2 Rutting resistance

The evaluation of rutting resistance was achieved using the wheel-tracking test which is a simulative test to predict rut depth in accordance with the standard BS EN 12697-22 [45]. The test was performed on slab specimens which were mixed and compacted by a roller compactor following the BS EN 12697-33 [42]. The wheel-tracking test adopted by Ojum [46] was used to assess the rutting resistance of the CBEMs. Before the test, slab samples were conditioned at 60°C for at least 4 hours. The wheel-tracking test involves the application of a wheel pressure (0.7 MPa) on the slab specimens (400 x 305 x 50 mm) through repeated passes of a loaded wheel (10000 cycles). The traveling distance was 230 ± 10 mm at a speed of 42 ± 1 cycles/min. at a temperature of 60°C. The resulting deformation on the slab is measured in each of the wheel passes. Figure 6 shows the HYCZ-5 small size wheel-tracking equipment used by LCMT labs while Table 5 illustrate the test conditions. The tests were performed with five specimens per mix type.

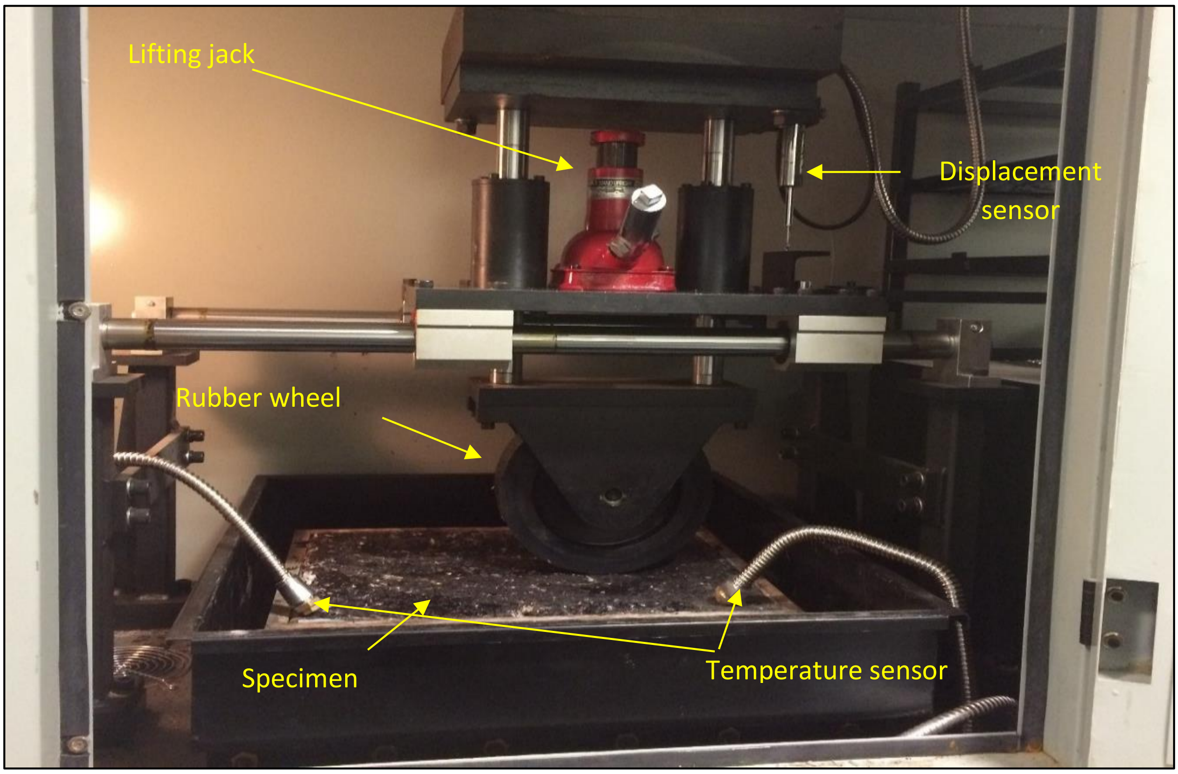

Figure 6. Wheel-tracking test equipment

Table 5. Wheel-track 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
Contact pressure (MPa)	0.7 ± 0.05
Poisson's ratio	0.35
No. of conditioning cycles	5
No. of test cycles	10000
Test temperature, (°C)	60
Compaction	Roller compactor
Specimen temperature conditioning	4hr before testing

4.3 Water sensitivity

Water sensitivity has a vital role in the mix design criteria from a pavement engineer's point of view. BS EN 12697-12 [47] was adopted to assess the moisture susceptibility of the CACB mixtures along with cold and hot control mixtures. The stiffness modulus ratio (SMR) is expressed as the ratio of wet stiffness to dry stiffness in the specimens. This was used to evaluate the resistance to water as recommended by Al-Busaltan et al. [40], Al-Busaltan et al. [44], Al-Hdabi et al. [48].

In this test, two sets of cylindrical specimens were fabricated and separated using a Marshall hammer with five parallel specimens in each set of samples. The first set of samples known as the dry set (unconditioned), were kept dry at room temperature (20°C) for 24 hours in their mould. They were de-moulded the following day and left in the lab for 7 days. The second set, known as the wet set (conditioned), were left at lab temperature (20°C) for 24 hours, de-moulded and kept in the lab for another 4 days. Following this, a vacuum (6.7 kPa pressure) was applied to the specimens for 30 minutes, after which they were left immersed for 30 minutes and submerged in a water bath for 3 days at 40°C. The two sets were then tested for ITSM whereby water damage resistance was evaluated by determining the SMR ratio of the samples in each set calculated as follows:

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

4.4. Scanning electron microscopy (SEM) observation

The microstructure of the original raw materials and fracture surfaces from the binder pastes were examined using a scanning electron microscopy (SEM). SEM is a technique for high-resolution imaging of surfaces used to examine the morphology of the object. The binder pastes of the ABBCF were prepared and dry samples were investigated with the aid of inspect scanning electron microscopy with accelerating voltages 5-25 kV. Proper fragments were taken off from the core of the paste at due age, i.e. 3 and 28 days for SEM observation. It was very important to guarantee that the fragments were snapped out of the cylinders by impact without touching any tools; if not, the paste surface would not be a natural one and would not represent the materials features correctly. Prior to carrying out SEM observations, the samples were dried in a vacuum pump to eliminate any evaporable water. They were then mounted onto aluminium stubs by means of double-sided adhesive carbon disks. A thin layer of Palladium was used to coat the fracture samples using a sputter coater to improve visibility.

5. Results and discussion

5.1. Performance of CACB in ITSM

Figure 7 shows the evolution of the indirect stiffness modulus test over 1, 3, 7, 14, 28 days for the activated mixtures by replacing the commercial limestone filler with HCFA. A significant improvement in ITSM of the CACB with HCFA was due to the hydration process that produced another binder resulting in additions to the bitumen residue binder. Consequently, the two binders generated microstructural integrity within the HCFA-emulsion mixtures. This resulted in the CACB mixture with HCFA becoming 17+ times higher than the reference LF mixture after just 3 days, offering a stiffness which overcomes that of traditional hot asphalt concrete binder courses 100/150 penetration grade in less than three days. However, the stiffness of the HCFA treated mixture is less than the stiffness of OPC by approximately 7% at 3 days. In contrast, there were no noticeable changes in stiffness for the two grades of hot asphalt concrete binder courses while the control LF showed the lowest ITSM at all ages.

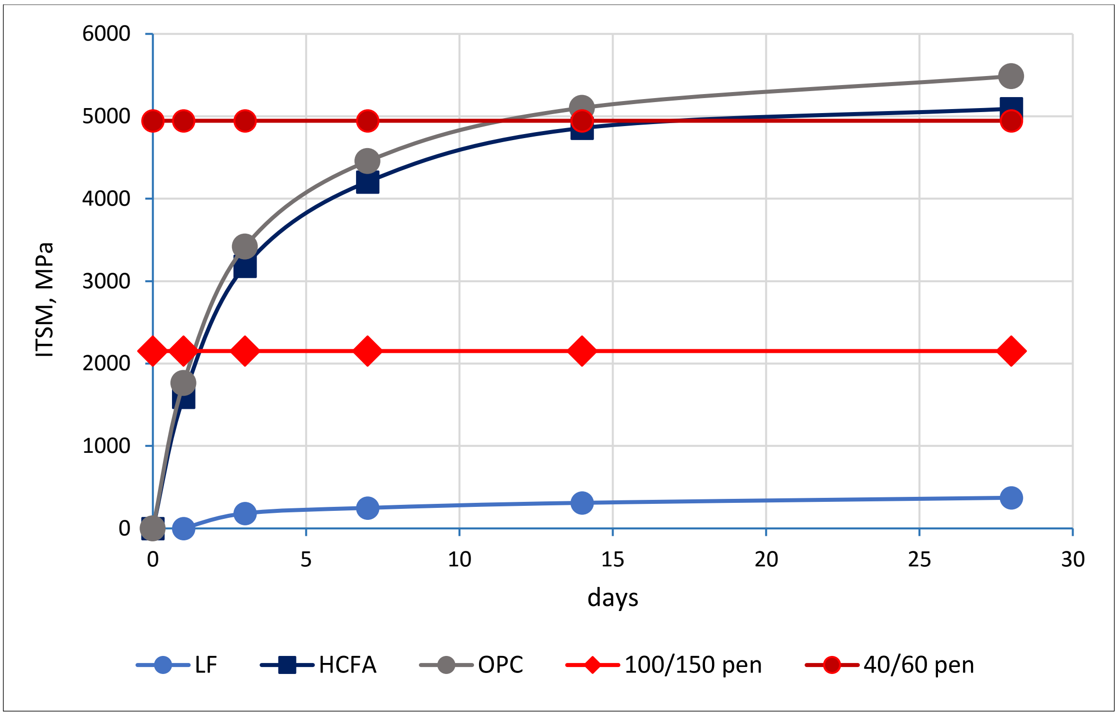

Figure 7. Influence of curing time on ITSM results

The second stage aimed to activate HCFA with FC3R to generate a binary blended cement filler (BBCF). The presence of pozzolanic particles in FC3R help to speed up hydration of the HCFA particles which leads to a more hydrated product. Figure 8 illustrates the behaviour of the CACB mixtures with different percentages of HCFA and FC3R. As a result, a new binary blended cement filler (BBCF) was recommended with 4.5% of HCFA and 1.5% of FC3R. The performance of FC3R reveals its pozzolanic activity which was reported by Payá et al. [49]. The reason for this enhancement is that the pozzolanic particles of FC3R react with $Ca(OH)_2$ released during the hydration process and help to speed up hydration of the HCFA particles. Consequently, more hydrated products were created. A balanced oxides composition was expected to be generated in this composition within the BBCF.

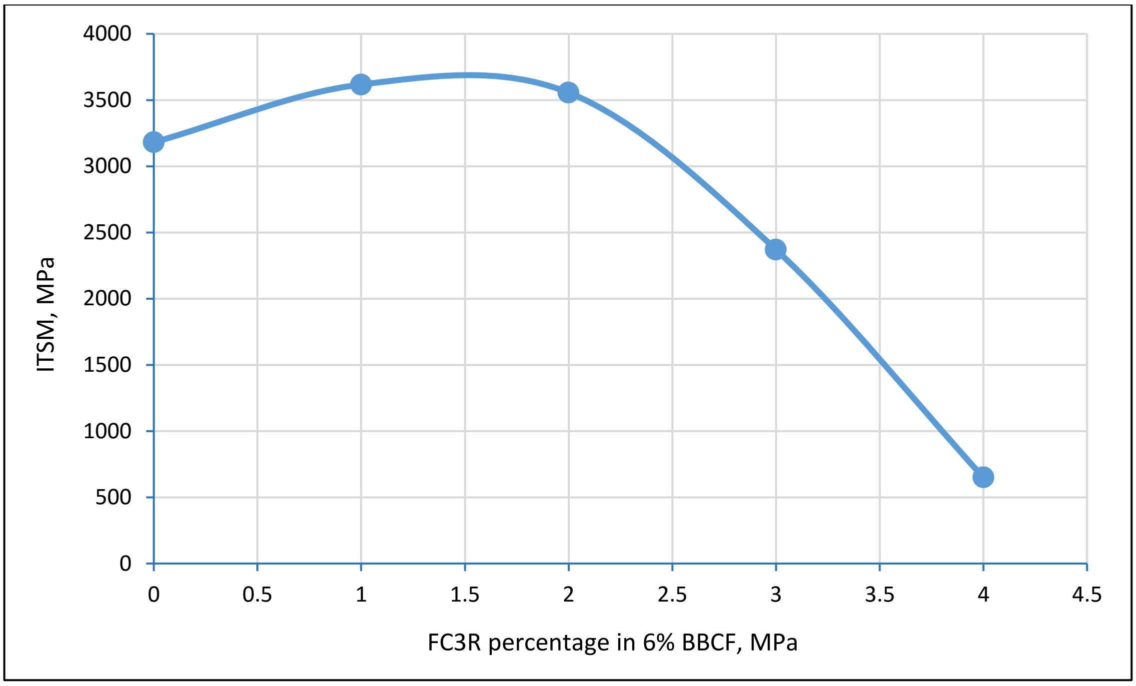

Figure 8. Influence of replacement of HCFH with FC3R on stiffness modulus after 3 days

The third stage aimed to employ alkali activation for further development of the BBCF by using waste alkali sodium hydroxide (NaOH) as a replacement of 3.5% of the pre-wetting water content. Alkali-activation offers the opportunity to employ waste materials, because the material properties based on alkali-activated binders are often greater than those of concrete and mortar prepared from standard Portland cement [50]. Some studies have found that the addition of alkali-activators raise the pH of the medium of hydration which improves breaking and dissolution of the glassy phase of pozzolanic material [51, 52]. Li et al. [53] stated that fly ash can be activated by breaking down the glass phases of particles through a rise in the alkalinity of the mixture. The usual technique used to increase alkalinity is by adding a NaOH solution. However, in that alkaline environment, it is expected that glass phases of fly ash particles will be broken and react with $Ca(OH)_2$ creating CSH gel. Figure 10 shows that CACB with an alkali activated binary blended cement filler (ABBCF), developed a higher stiffness (approximately 33%) than BBCF mixtures after three days with 100% pre-water replacement by the waste NaOH solution.

It can be observed from Figure 9 that CACB with ABBCF offers a significant stiffness modulus in comparison to all other cold mixtures. In addition, the rate of stiffness modulus development was high through to the 7 day point when before a reduction in rate was detected. The target stiffness (2152 MPa) for the hot asphalt concrete 100/150 pen can be achieved in 1 day by curing with the ABBCF mixture. It will then reach British and European requirements in terms of ITSM however, as reported by Leech [5], traditional cold mixtures only achieve the necessary strength after 2–24 months. As a result, a new cementitious material made completely from waste materials has been recommended for application in CBEMs. These results are consistent with those achieved by Al-Busaltan et al. [40], Al-Hdabi et al. [54]. In addition, the stiffness values obtained in this research are greater than those achieved in the afore mentioned studies. Aggregate and emulsion type and the method of activation in this research may be the factors that have led to this improvement.

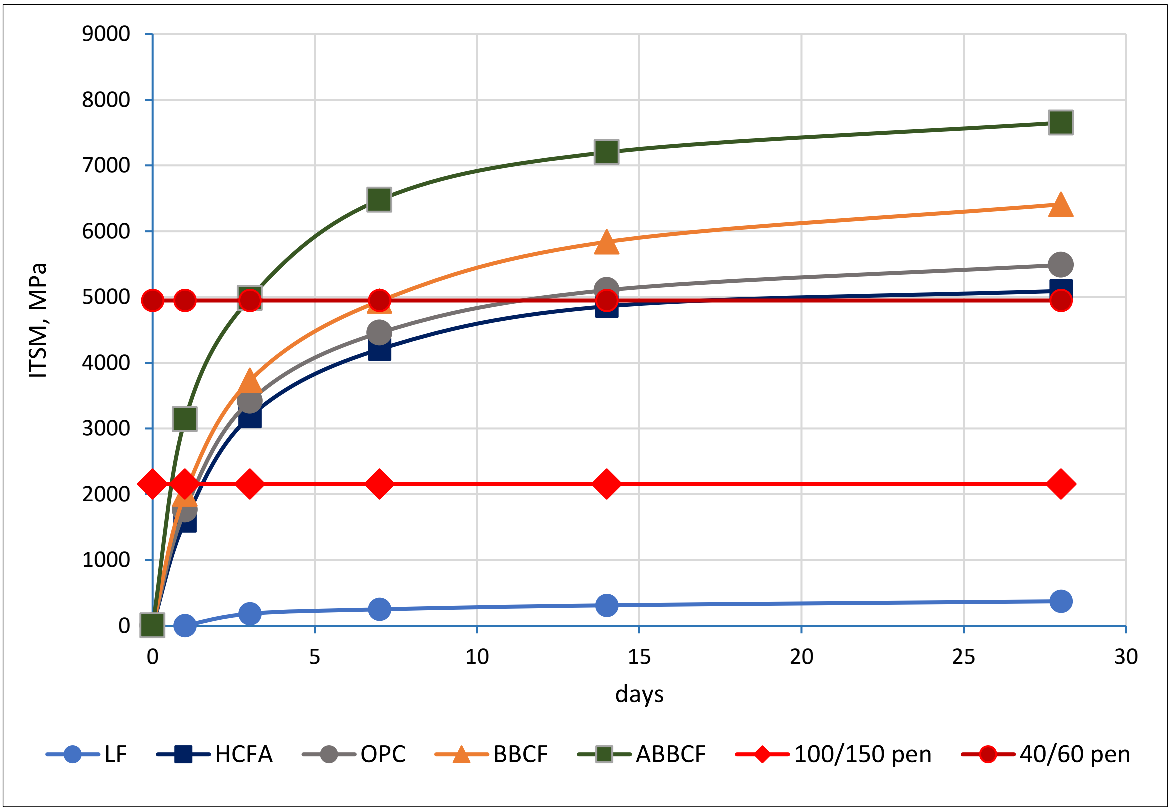

Figure 9. Influence of curing time on stiffness modulus

5.2. Temperature sensitivity performance

Studying the temperature sensitivity of CACBs can offer a useful insight into the stabilization mechanisms of cold asphalt mixes. Figure 10 illustrates the temperature susceptibility results of all the cold and hot mixtures. The slope of the curve in a semi-logarithmic plane characterizes temperature susceptibility where the greater the rate of change, the more temperature sensitive the mixture. The results of ITSM for the cold LF mixture is highly dependent on the test temperature applied; these mixtures fail at 45°C. The stiffness modulus of the LF mixture decreases with the increase in temperature. In addition, there is a strong trend apparent for both the hot asphalt concrete binder course mixtures where they lost about 97% of their stiffness when heating from 5°C to 45°C. Nevertheless, CACB mixtures with OPC, HCFA and BBCF and ABBCF showed a substantial lower thermal sensitivity than the LF mixture and both hot asphalt concrete binder course mixtures. The ABBCF mixture has an excellent performance potential regarding use in a hot climate. These findings are comparable to those published by other authors [54, 55].

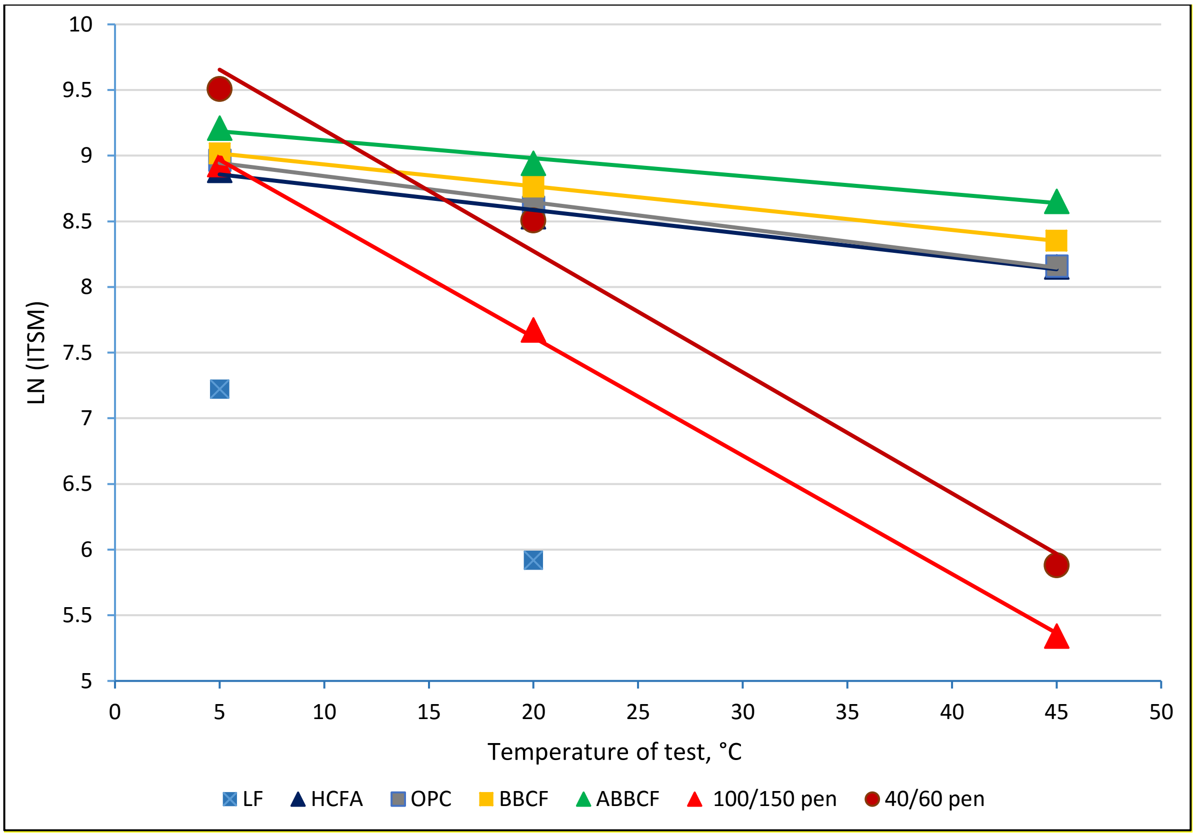

Figure 10. Temperature sensitivity results

5.3. Performance of rutting in wheel track

The susceptibility to permanent deformation of the cold asphalt concrete mixtures in comparison to the hot asphalt concrete mixtures were evaluated based on the rut resulting from repeated tracking of a loaded wheel across slab specimens at a high temperature (60°C). Figure 11 shows the rutting test results using the wheel-track. There was a remarkable decrease in rutting depth for CACB mixtures with HCFA, OPC and BBCF. This might be due to the production of the new binder from the hydration process which makes the new CACB mixtures more rut resistant. The LF mixture has the highest rut depth (occurring during the first 1000 cycles) indicating that this mixture is more prone to rutting, revealing the weakness of such mixtures in summer weather and hot regions.

It is of interest to note that the ABBCF mixture dramatically reduced rut depth and exhibits considerably higher rutting resistance than LF, OPC, HCFA, BBCF and both control hot asphalt concrete mixtures. This might be related to the role of waste NaOH creating a dense microstructure activated through the hydration process. Accordingly, the new ABBCF mixture will be able to withstand a considerable traffic loading typical of road structures today indicating the potential advantage of applying this mixture on heavily trafficked roads.

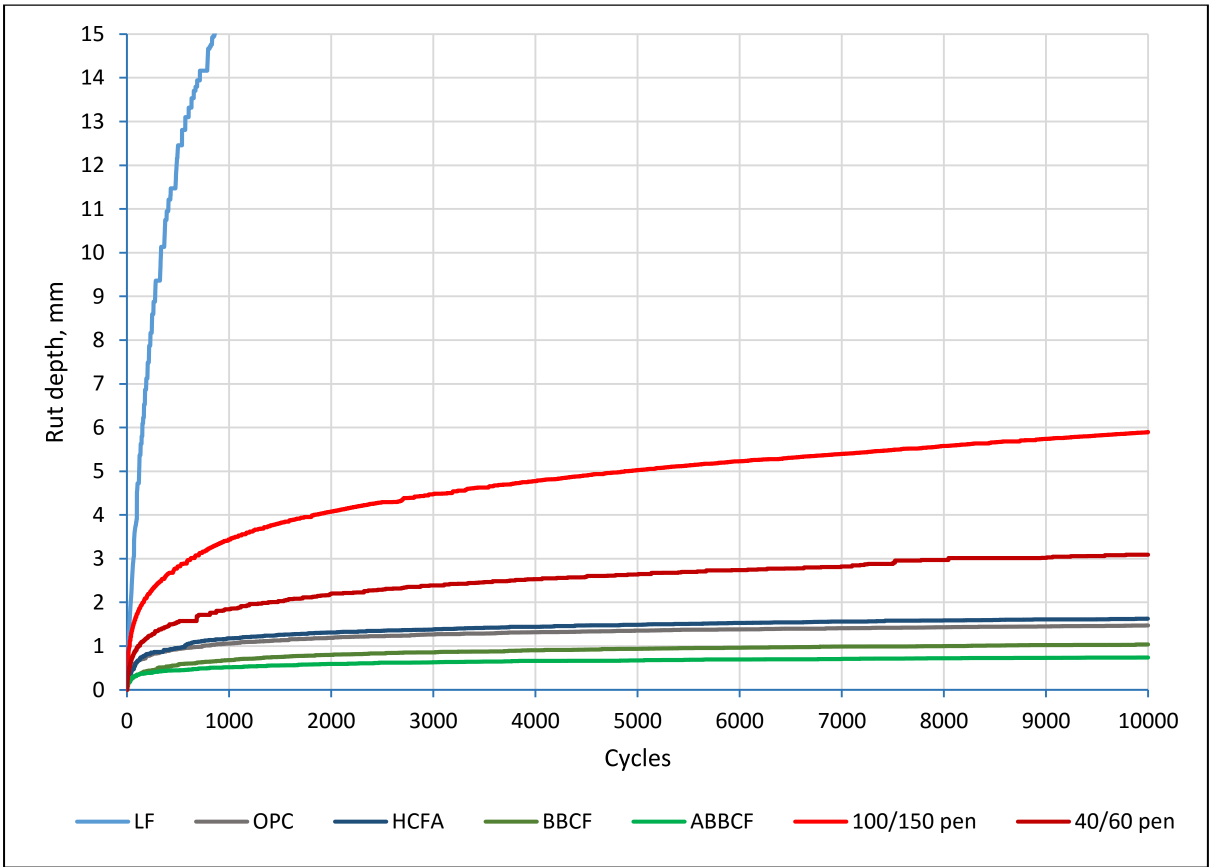

Figure 11. Comparison of rut depth

5.4. Water sensitivity performance

The water sensitivity results for CACB mixtures with different filler materials and the hot asphalt concrete binder course mixtures are shown in Figure 12. Here it can be observed that the CACB with

HCFA, OPC, BBCF and ABBCF exhibit higher values than the reference LF mixture and both grades of hot asphalt concrete binder course mixtures. It seems clear that a lack of cohesion is the main reason for the inferior performance against water action in the cold mixture made with LF. It can be observed that the stiffness for the immersed samples for CACB with HCFA, BBCF and ABBCF is higher than the stiffness of the dry samples. Samples immersed in water show an improved hydration process this due to high water temperature (40°C). Heating accelerated the hydration process and more hydration products were produced. Accordingly, CACB mixtures with HCAF, OPC, BBCF and ABBCF are less susceptible to moisture damage.

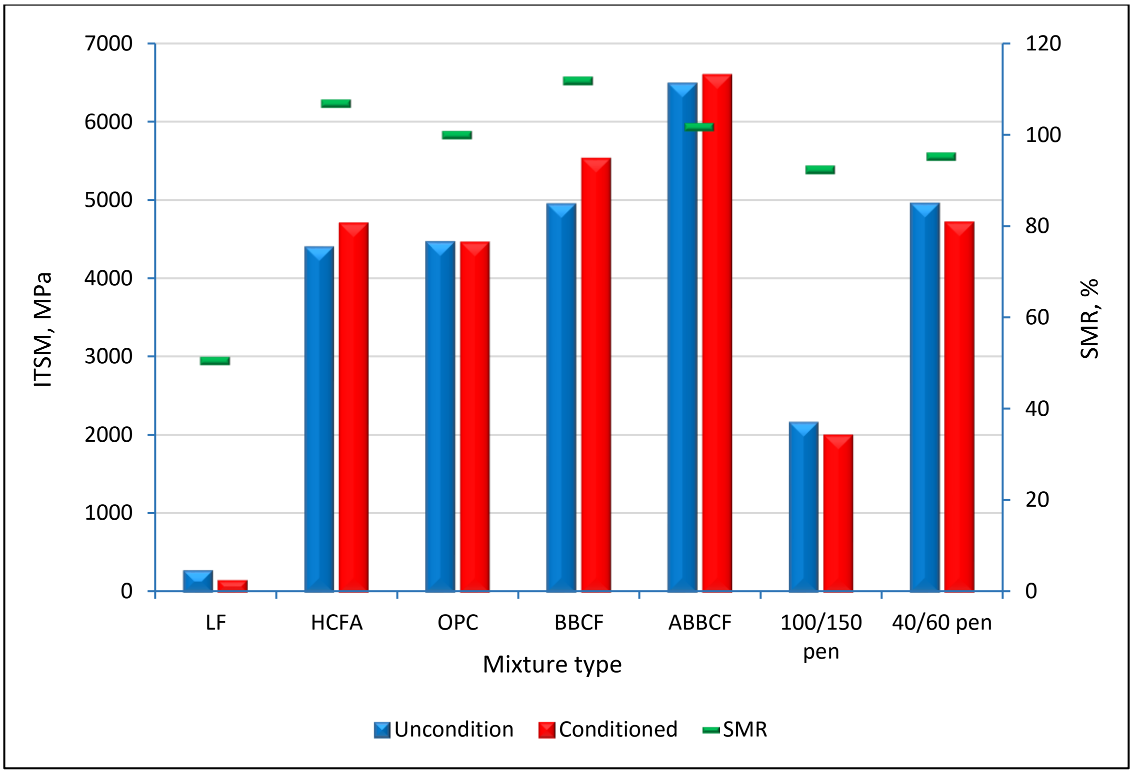

Figure 12. Water sensitivity Performance results

5.5. Scanning electron microscopy (SEM) observation

Figure 13 shows the SEM photographs after 3 and 28 days of curing for the paste sample and the original raw materials (HCFA and FC3R in their dry state). Significant amounts of hydrates were formed at an early stage of curing within the alkali activated binary blended filler (ABBCF). SEM analysis revealed a more pronounced micro-structural evolution after 3 days; no intact filler particles can be detected after 3 days curing, the HCFA and FC3R powder particles found to be converted in to hydrates due to successive hydration reaction. These hydration products created a dense material with high mechanical properties which is consistent with the development of the stiffness modulus of the ABBF samples. After 28 days, the surface of ABBF is covered by CSH gel and Portlandite (CH). The structure of the ABBCF is dense and crystalline products were mainly found in pore areas of this sample due to the reaction of the active BBCF and NaOH. Consequently, the ABBCF mixture can be said to have improved properties.

It is worth mentioning that the air voids present in the ABBCF mixture were 10.25% incomparison to 10.93% in the reference cold LF mixture revealing an enhancement of volumetric properties for the ABBCF mixture. These findings are consistent with those obtained by Nassar et al. [22] and Dulaimi et al. [56]. Serfass [57] reported that the higher air void content in compacted CAM mixtures was the result of water evaporation. If a comparison is made between CMA and HMA, many tiny voids are present in the former due to the film made by coalescence and because the viscosity of the bitumen is higher at ambient temperature. Nassar et al. [22] recently reported that the presence of hydration products such as Ettringite, as a result of the use of OPC and fly ash (as a filler replacement) in the capillary voids of CBEM, can enhance the volumetric properties (less porosity) by decreasing both the pore size and their continuity. This prevents the movement of water and other aggressive fluids into the mixture.

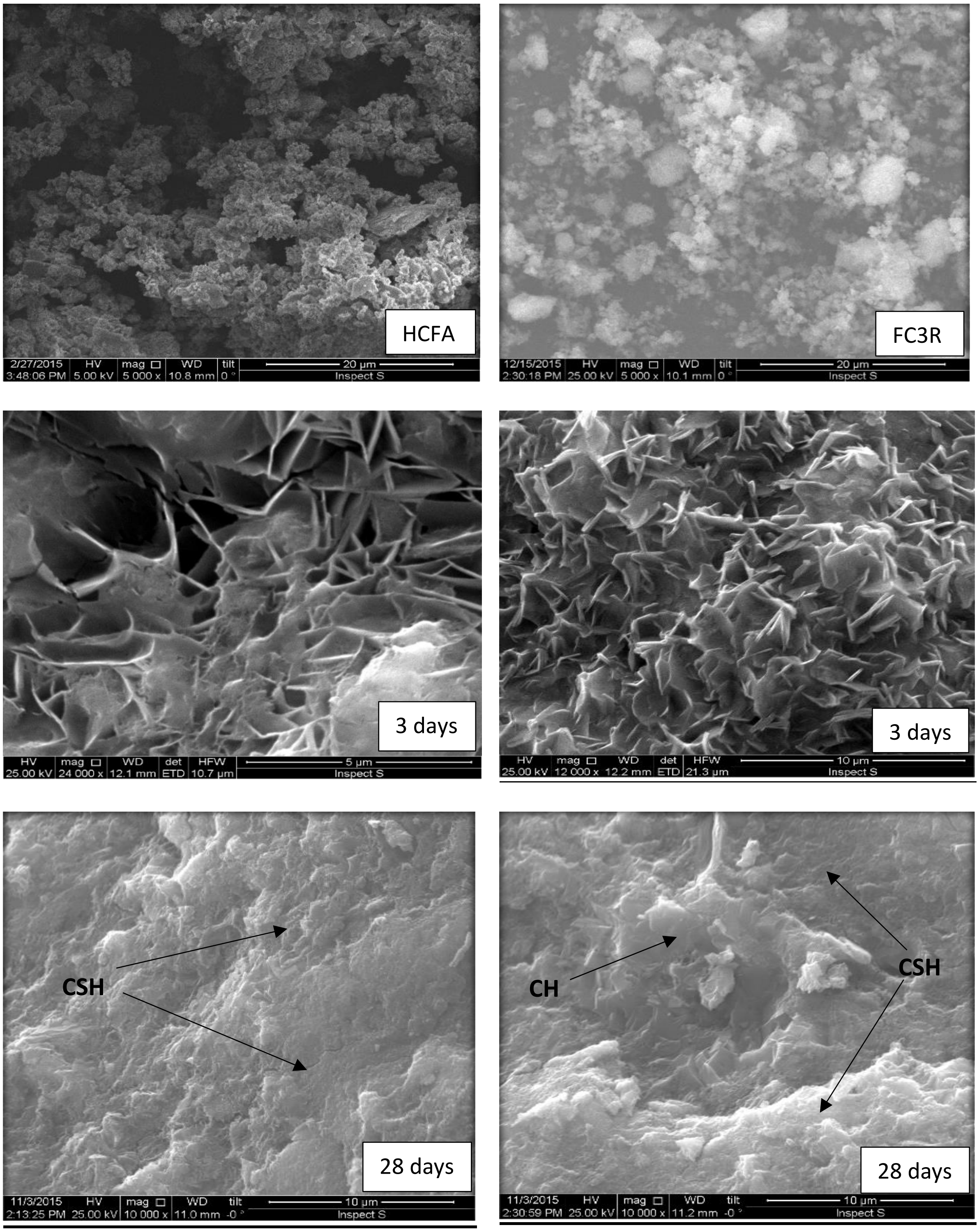

Figure 13. The microstructures of the original raw materials and ABBCF paste after 3 and 28 days

6. Conclusions

A new fast-curing and environmentally friendly cold asphalt concrete for a binder course mixture (CACB) with high performance properties was developed at the Liverpool Centre for Material Technology (LCMT). In this mixture, a novel alkali activated binary blended cement filler (ABBCF) from waste materials was used as a substitution for commercial mineral filler. Based on the results achieved in the research performed, the following conclusions can be drawn:

1. A new binary blended cement filler (BBCF) from waste material was developed from 4.5% HCFA and 1.5% FC3R. This BBCF was activated by a waste NaOH solution to produce a novel alkali activated binary blended cement filler (ABBCF).
2. In terms of stiffness modulus, the new ABBCF mixture offers a stiffness modulus 27 times more than a mixture with commercial limestone dust after 3 days, this a result of the improved hydration products of the ABBCF.
3. The new ABBCF achieved the required stiffness for the conventional hot asphalt concrete binder course 100/150 pen (2152 MPa) in less than one day. This will overcome restrictions around the time required to achieve acceptable stiffness for traditional CBEMs.
4. The new ABBCF mixture has significant resistance to rutting in wheel-track tests at high temperatures. These results are much better than the two grade hot asphalt concrete binder course meaning it can carry heavy traffic loads in hot climate conditions.
5. In terms of water susceptibility, the ABBCF offers a conditioning stiffness modulus which is more than the unconditioned stiffness and this result is more than 100% in SMR which is better than the result for the two grades of hot asphalt concrete binder course. Progressive curing with ABBCF was accountable for the high water damage resistance.
6. The new ABBCF is significantly improved with reference to resistance to temperature sensitivity. It will therefore provide an appropriate solution in resistance to temperature variations.
7. SEM provides evidence for the existence of hydrate products which are responsible for ITSM development in the ABBCF mixture.

8. Decreasing waste disposal and saving raw materials will be ensured and will contribute to sustainable development.

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