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

57 restricted to base and sub-base courses of structural layers [4, 8]. Subsequently, the use of cold emulsified

58 asphalt as a structural layer is restricted because of the long period necessary for such material to reach its

59 full strength due to the UK weather and because of higher sensitivity to rainfall in the early stages of

60 application [9].

61 Those mixtures have been described as evolutive materials [6] in contrast to traditional hot mix asphalt

62 which gain strength rapidly as the material cools. They gradually achieve strength when the material

63 becomes dry, further developing increasing strength over time. There are continuously changing properties

64 exhibited by emulsion bound mixtures (stiffness modulus, permanent deformation resistance, water

65 sensitivity, etc.) which continue until they reach a steady state at a fully cured condition, though they may

66 still contain a low amount of residual water [6]. Jenkins [1] demonstrated that when moisture is expelled

67 from the mix, cold bituminous mixtures gain strength.

68 Conventional cement is commonly used in cold mix asphalt as an enhancement technique. One of the

69 benefits of this technique is that satisfactory strength can be reached in a short period of time [10, 11]. Brown

70 and Needham [12] examined cement modified emulsion mixtures, the primary aim to appraise the influence

71 of adding Ordinary Portland Cement (OPC) to said mixtures. They used a granite aggregate grading in the

72 middle of 20mm dense bituminous macadam with a single slow-setting emulsion. They concluded that the

73 addition of OPC enhanced the mechanical properties, namely: stiffness modulus, permanent deformation

74 resistance and the fatigue strength of the emulsified mixes. Oruc et al. [9] also carried out a study to assess

75 the mechanical properties of emulsified asphalt mixtures incorporating 0-6% Portland cement. Substantial

76 improvements were seen with the addition of higher quantities of Portland cement and they recommended

77 that such a mixture might be used as a structural pavement layer.

78 The re-use of waste materials in cold asphalt mixtures is generally promoted for two reasons: environmental

79 sustainability (cement manufacture is responsible for 5% of global greenhouse gases (GHG) [13]) and

80 economic benefits. Ellis et al. [14] investigated the performance of a range of storage-grade macadams

81 consisting of recycled aggregates from different sources bound by bitumen emulsion and Ground Granulated

82 Blastfurnace Slag (GGBS). The results revealed that stiffness and strength can develop when GGBS is added

83 in conditions of high humidity. Thanaya et al. [15] conducted experiments using pulverised fly ash (PFA)

84 as a filler in a cold mix at full curing conditions; the cold mix stiffness was found to be comparable to that

85 of hot mixes.

86 Al-Busaltan et al. [5] used waste materials (waste fly ash as the substitute for traditional mineral filler) to

87 develop a cold mix asphalt, a close graded cold bituminous emulsion mixture (CBEMs) that had outstanding

88 mechanical properties compared to traditional hot mix asphalt surface courses. The result was that an

89 additional binder was produced from the hydration process that took place between the waste fly ash and

90 the trapped water involved in cold mixtures.

91 Al-Hdabi et al. [16] applied two sorts of waste fly ash as filler substitutes to produce new gap-graded Cold

92 Rolled Asphalts (CRA). The new CRAs produced were found to have a comparable performance with

93 conventional Hot Rolled Asphalts (HRA) after short periods of curing in terms of stiffness modulus and

94 uniaxial creep tests. In addition, a considerable improvement in terms of resistance to water damage which

95 reported. The effect of chemical additives comprising OPC, hydrated lime (HL) and a combination of HL

96 and GGBS on recycled mixture performance with asphalt emulsion was examined by Du [17]. He concluded

97 that the level of performance enhancement depends on the type and content of chemical additives. Hydration

98 products can result in stiffness increments and cohesion of the asphalt mastic of the recycled mixture.

99 It was reported by Čížková et al. [18] that test conditions such as applied test temperature and loading speed,

100 effect the stiffness modulus of bitumen stabilized materials along with other cold recycled mixtures.

101 Viscoelastic material behaviour created from reclaimed asphalt material (RA/RAP) in addition to the type

102 of bituminous binder applied, can explain this. Zak and Valentin [19] stated that Bitumen Stabilized

103 Materials (BSM) have higher air voids, less adhesion and less cohesion in the internal structure in

104 comparison to HMA. Accordingly, the fatigue performance of such mixture is inferior to HMA. Recently,

105 Nassar et al. [20] used binary and ternary blended fillers (BBF and TBF) to see if there was any improvement

106 in Cold Asphalt Emulsion Mixtures (CAEMs). Various fillers sorts were used such as OPC, fly ash, GGBS

107 and silica fume. Their results indicated that the TBF was more appropriate than the BBF for CAEMs

108 manufacture claiming that the TBF mixture would be effective for use on road pavements subject to harsh

109 conditions, both hot and cold weather.

110 Sadique et al. [21] conducted research exploring the pozzolanic reactivity of calcium rich fly ash by blending

111 and grinding it in a cement-free system, finding that the hydration products and improvements in strength

112 in the new blend was comparable to cement. Mármol et al. [22] investigated the pozzolanic activity of FC3R

113 in Portland cement systems by examining the water/binder effect. They compared reactivity to Metakaolin,

114 a synthetic pozzolan, reporting that FC3R has a comparable chemical structure and high pozzolanic activity.

115 Accordingly, HCFA and FC3R have the potential to be employed as supplementary cementitious materials

116 (SCMs) to substitute traditional mineral filler. As further support, Pacewska et al. [23] revealed that both

117 spent catalyst and microsilica have the same potential to be combined with $Ca(OH)_2$, as the hydration process

118 is highly exothermic, resulting in rapid setting of the cement paste. These findings offer a potential impact

119 on sustainability by eliminating the need for cement in cold asphalt mixtures.

120 Despite extensive research into different types of CBEMs, to date, the problems relating to curing time, low

121 early strength, rutting resistance and water sensitivity of CBEMs for the binder course have not been

122 addressed. Therefore, developing a cold asphalt concrete for the binder course (CACB) mixture with high

123 early strength, resistance to water ingress and with minimal time requirements for structural loading would

124 be a breakthrough in CBEM research. Developing a novel binder to be used as a filler in CACB would

125 provide the enhanced properties mentioned above. Therefore, an investigation to establish an advanced

126 method for developing a cementitious binder suitable for CACB to be used as a filler has been carried out

127 in this study. A supplementary cementitious material derived from industrial waste has been used to enhance

128 the performance of the CACB mixtures. The indirect tensile stiffness modulus (ITSM) test, permanent

129 deformation resistance and tests of fatigue resistance were utilised to evaluate the effects of using high

130 calcium fly ash (HCFA) with a fluid catalytic cracking catalyst (FC3R) on the mechanical properties of

131 CACB mixtures. The effect of using these materials on moisture damage resistance was evaluated by

132 determining the Stiffness Modulus Ratio (SMR).

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134 **2**. **Materials and method**

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

136 below.

137 *2.1 Materials*

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139 *2.1.1 Aggregate*

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141 Coarse and fine aggregates were crushed granite from Carnsew Quarry at Mabe, Penryn in the UK, which

142 is generally used to produce hot asphalt concrete mixtures. A dense aggregate gradation for the asphalt

143 concrete binder course AC-20 was used in this research, as shown in Figure 1. The aggregate structure

144 permitted a curve to be established following the standard EN 13108-1 [24]. This gradation is normally used

145 on asphalt pavement binder course in the UK. The physical specifications of both the coarse and fine

146 aggregate have been detailed in Table 1.

147 Asphalt concrete is by far the most common mixture in use as a binder course and base in road pavements

148 in the UK. Having a continuous grade, it offers a good aggregate interlock giving it very good load-spreading

149 properties as well as high resistance to permanent deformation.

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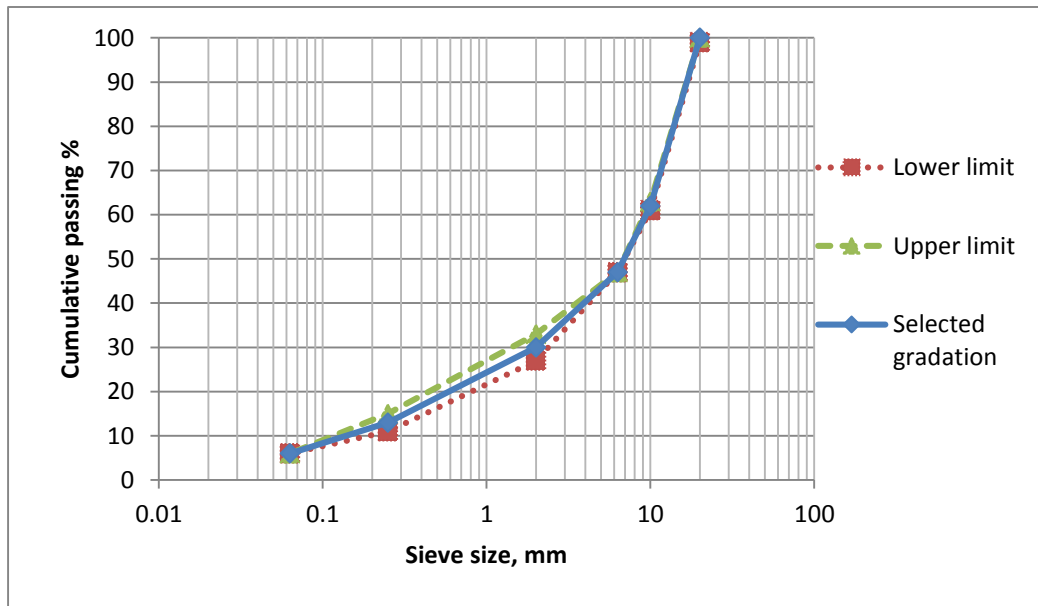
151   Figure 1. AC 20 mm dense binder course aggregate gradation

152

153   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

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155 *2.1.2 Bitumen emulsion and asphalt*

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157 Cationic slow-setting bitumen emulsion (C60B5) designed for use in road pavement and general

158 maintenance applications, was used to prepare the cold bituminous emulsion mixtures. Thanaya [25]

159 confirmed that cationic emulsion is preferred as a result of its ability to coat aggregate and to guarantee high

160 adhesion between aggregate particles. Table 2 illustrates the properties of the chosen bitumen emulsion.

161 Two grades of bitumen, soft bitumen of penetration grade (100/150) and hard bitumen of penetration grade

162 (40/60), were used to make the hot asphalt concrete binder course mixtures. Table 3 details the characteristics

163 of these binders.

164   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$_2$O$_3$	MgO	Fe$_2$O$_3$	SO$_3$	K$_2$O	TiO$_2$	Na$_2$O
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

182 As shown in Table 4, it can be seen that HCFA is mainly composed of CaO with a good amount of SiO_2 and

183 Al_2O_3. It was reported that the existence of Ca, Al and Si in fly ash are significant in developing a new

184 cementitious material from waste and by-product materials [21]. Sadique and Al-Nageim [26] reported a

185 lower CaO content (57%) in their investigation but the proportion of SiO_2 was higher (28%). The main

186 oxides in FC3R are Al_2O_3 and SiO_2, this consistent with those achieved by Mármol et al. [22], Mas et al.

187 [27].

188 Lea [28] stated that soluble SiO_2 and Al_2O_3 present in the glass phase of pozzolanic materials can react with

189 $Ca(OH)_2$ when released through cement hydration to make an extra calcium silicate hydrate (CSH) gel that

190 enhances the mechanical strength of the hardened concrete structure.

191 The powder pattern of HCFA in XRD shown in Figure 2 revealed that the HCFA sample is crystalline as it

192 contains sharp peaks without significant noise in the background. The major crystal peaks identified were:

193 lime (CaO), calcite ($CaCO_3$), mayenite ($Ca_{12}Al_{14}O_{33}$), merwinite ($Ca_3Mg[SiO_4]$) and gehlenite

194 ($CaAl[Al,SiO_7]$). A similar mineralogy was reported by Sadique and Al-Nageim [26], Sadique et al. [29],

195 however no merwinite was detected in the latter.

196 The powder diffraction in XRD shown in Figure 3 indicated that FC3R has very low crystalline peaks which

197 are amorphous in nature meaning that it will demonstrate high reactivity during the hydration process and

198 can be used as a pozzolanic material. The following crystalline peaks were identified: kyanite (Al_2O_5Si),

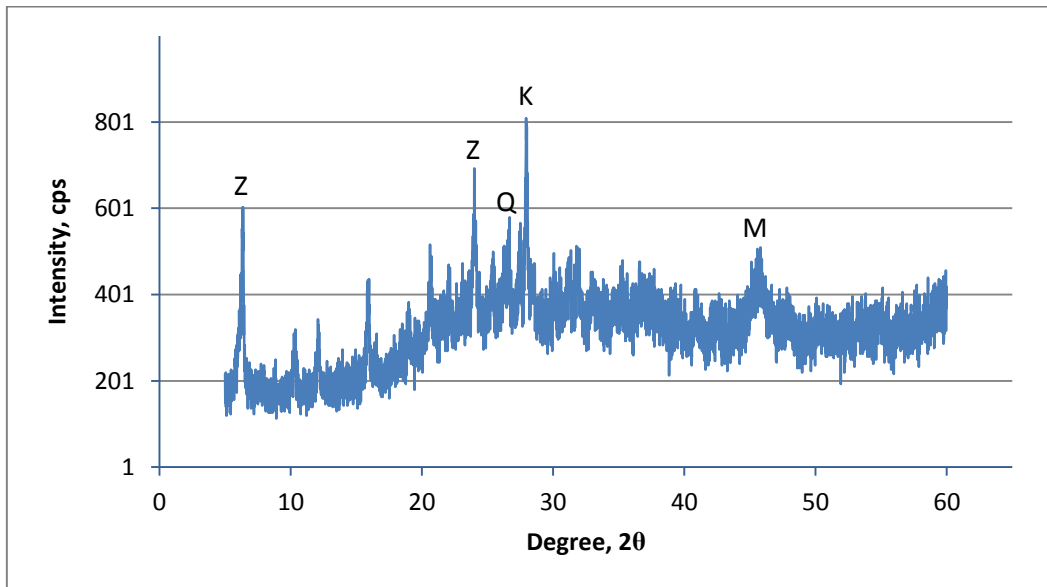199 quartz (SiO_2), mullite ($Al6Si_2O_{13}$) and dehydrated Ca-A zeolite ($Al_{96}Ca_{48}O_{38}4Si_{96}$).
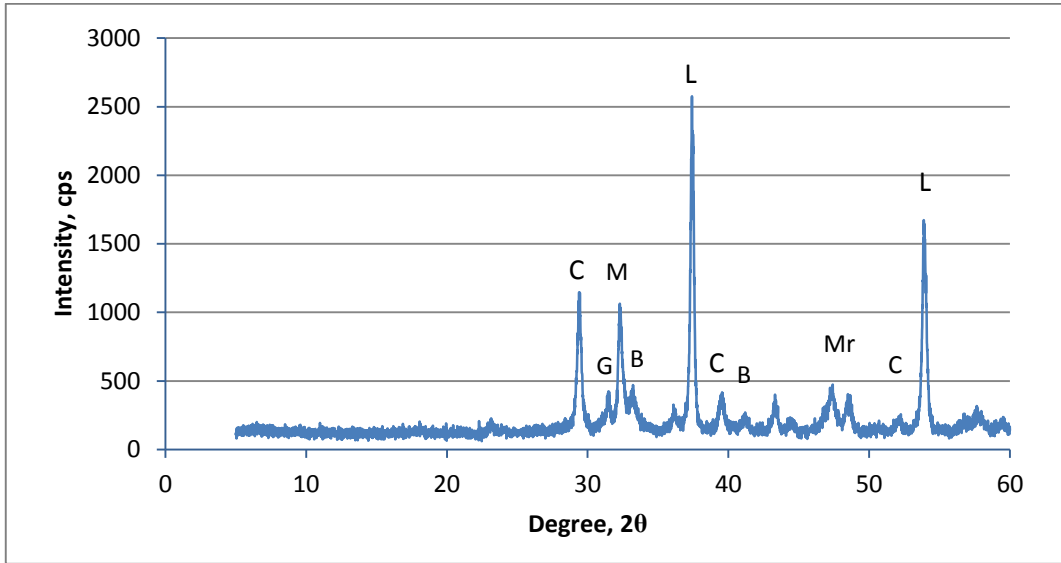
Figure 2. Powder XRD pattern of HCFA

(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.

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236   The proportions of the mixture by percentage of Marshall samples are summarized in Table 5. The

237   materials were mixed in a Hobart mixer. Afterwards, compaction was achieved by means of a Marshall

238   hammer with 100 blows, where 50 blows were applied to both faces of each specimen. It was reported by

239   Nassar et al. [20] that Marshall compaction is an appropriate method to use to manufacture an appropriately

240   dense mixture.

241   After compaction, the samples were left for 1 day at 20°C in the mould; the next day they were de-moulded.

242   All the specimens were then left in the lab at 20°C and tested at various ages, i.e. 1, 3, 7, 14 and 28 days.

243   Four additional reference mixtures were prepared and tested for comparison purposes. An untreated mixture

244   with traditional limestone filler (LF) was the first having the same design as other CACB mixtures. The

245   second mixture was treated with 6% OPC, while two grades of hot Asphalt Concrete-AC 20 (third and fourth

246   control mixtures), based on 100/150 pen and 40/60 pen, were tested at the same ages. The reference hot

247   mixtures also had the same aggregate type and gradation. The bitumen content was 4.6% in accordance with

248   the standard BS EN 13801-1 [24].

249   All the emulsion mixtures were fabricated and compacted at ambient temperature. The laboratory mixing

250   temperatures of the hot mixes were fixed at 150-160°C and 160-170°C for the 100/150 pen and 40/60 pen

251   respectively.

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262   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	-

263

264   Regarding the wheel track slabs, the sample mixtures for rutting tests were prepared in the same way as for

265   the stiffness tests. A slab sample with a 400mm length, 305mm width and 50mm thickness was compacted

266   at ambient temperature in the steel mould using a Cooper Technology Roller Compactor device following

267   the standard BS EN 12697-33 [33].

268   The slab specimens were kept in their moulds at lab temperature (20°C) for 24 hours before extraction, this

269   representing the first curing stage. Stage two involved curing the slabs at 40°C for 14 days, removing them

270   from the ventilated oven, cooling and subjecting to the wheel track test. This curing protocol was

271   recommended by Thanaya [25] to guarantee that a completely cured condition was reached. All the tests

272   were then performed on CACB mixtures at a fully cured condition. For the fatigue tests, slab samples were

273   prepared and cured in the same way as for the wheel track test samples. The slab samples were then cut with

274   a saw to provide a beam shape sample with 400mm length, 50mm height and 50mm width.

275   *2.3 Experimental program and tests performed*

276

277   The indirect tensile stiffness modulus (ITSM) test and rutting resistance were applied to evaluate the use of

278   the supplementary cementing material on the mechanical properties of CACB, while SMR was used to

279   assess moisture sensitivity. A Scanning Electron Microscopy (SEM) observation was applied to investigate

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280   the microstructure of the hydration products. Many researchers, for example Al-Busaltan et al. [2], Nassar

281   et al. [20], Thanaya [25], Monney et al. [34], Al-Hdabi et al. [35], have reported measuring the ITSM in

282   order to evaluate the mechanical performance of CAM. A wheel-tracking test was adopted by Ojum [36] to

283   characterize and assess the mechanism of failure of CBEMs. Four point beam-bending tests which evaluate

284   the fatigue performance of CBEMs, is recommended by Al-Hdabi et al. [10]. In addition, numerous

285   researchers such as Al-Busaltan et al. [2], Al-Busaltan et al. [5], Al-Hdabi et al. [37] have reported measuring

286   water sensitivity in terms of Stiffness Modulus Ratio (SMR) of CAMs following BS EN 12697-12 [38].

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288   *2.3.1 Indirect tensile stiffness modulus (ITSM) test*
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290   The ITSM test is a non-destructive test used to evaluate the ability of an individual layer of a pavement to

291   distribute traffic loads to the layer beneath. Currently, stiffness modulus is generally recognised as a

292   significant performance property of bituminous paving materials and is used as an indication of the load-

293   spreading ability of bituminous paving layers. The test was carried out on five samples for each mixture type

294   following the standard BS EN 12697-26 [31] using Cooper Research Technology HYD 25 testing apparatus.

295   Test conditions are shown in Table 6 below.

296

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

297

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

318 as equal to the number of load repetitions resulting in a 50% stiffness decrease. This test was performed

319 according to BS EN 12697-24 [40] using the controlled strain method at a temperature of 20°C and 10 Hz

320 frequency under sinusoidal loading with no rest period and a controlled strain criteria of 150 microstrain.

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322 *2.3.4 Water sensitivity*
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324 The ability of asphalt mixtures to resist moisture damage is critical to their long-term performance. Being

325 sensitive to moisture damage makes the asphalt mixture eventually fail in any of the failure modes, e.g.

326 rutting, fatigue, thermal cracking and ravelling [41]. A water sensitivity test was applied following the

327 standard EN 12697-12 [38] to evaluate the mixtures' sensitivity under the moisture effect. This test reveals

328 the effect of saturation and improved water conditioning on the indirect tensile stiffness modulus of

329 cylindrical specimens of CBEMs, performed here following EN 12697-26 [31].

330 The water sensitivity test identified that the interior bonding of the asphalt mixture was reduced due to water

331 existence. The specimens were divided into two sets; the first set of specimens were kept in the mould for 1

332 day before extraction and then left at 20°C for another 7 days prior to the stiffness modulus test performed

333 at 20°C; they represented a dry condition. The second set of samples were kept in the mould for 1 day,

334 extracted and left to cure at 20°C for 4 days before being subjected to a vacuum (with 6.7 kPa pressure for

335 30 minutes) and kept submerged in a glass jar for an additional 30 minutes. Following this, these samples

336 were conditioned at 40°C for 3 days before testing, representing a wet condition. Five specimens were tested

337 for each mixture type. The two sets were then tested using the ITSM test, where the water sensitivity was

338 measured by determining the stiffness modulus ratio (SMR) ratio as follows:

339   SMR = (wet stiffness / dry stiffness) $\times$ 100

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341 *2.3.4 Scanning Electron Microscopy (SEM) observation*

342 Scanning electron microscopy (SEM) is a technique for high resolution imaging of surfaces to reveal the

343 morphology and internal microstructure of the particles and surface characterization of materials. This

344 technique will allow changes to the hydration products as a result of using HCFA and BBCF fillers in the

345   CACB mixture to be examined. The tests were performed with an SEM resolution of 3-4 nm, high vacuum

346   and test voltage ranging from 5 kV to 25 kV using an Inspect scanning electron microscope.

347   Microstructural analyses were performed by employing SEM on selected paste samples (made with HCFA

348   and BBCF) taken from the centre of the crushed specimens. These specimens were used to detect changes

349   in the materials at various ages of curing. The pastes were moulded into cylinder samples which were kept

350   for 1 day at room temperature and then demoulded. Appropriate pieces were then taken off the cylinders at

351   due age, i.e. 3 and 28 days for SEM investigations. It was essential to ensure that the pieces were snapped

352   out of the specimens by impact without touching any tools otherwise the paste surface would not be a natural

353   one and would not accurately represent the features of the materials correctly. The pieces were mounted on

354   aluminium stubs using double-sided adhesive carbon disks and subjected to a vacuum. A palladium coating

355   was then applied to the sample, prior to taking the SEM images, using an auto fine sputter coater.

356   **2. Results and Discussion**

357   *3.1 Performance of CACB mixtures in ITSM test*

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359   The first phase of the research concerned the effect of the substitution of conventional mineral filler with

360   HCFA on the stiffness modulus of cold asphalt mixtures. The ITSM test was run in accordance with BS EN

361   12697-26 [31]. The results of ITSM tests for the HCFA replacement are shown in Figure 4, where it can be

362   seen that the ITSM for 6% HCFA replacement after 3 days is around 17 times the reference for untreated

363   cold mix asphalt (6% limestone filler- LF).

364   It is clearly demonstrated that the addition of HCFA as a substitute for limestone filler substantially enhanced

365   the stiffness modulus, this improvement due to two effects. The first is the generation of another binder

366   made from the process of hydration as a result of the hydraulic reaction of HCFA in addition to the bitumen

367   residue binder. Secondly, trapped water was lost during the hydration of the HCFA. Of note here,

368   conventional hot asphalt concrete binder course mixtures do not display visible differences in ITSM over
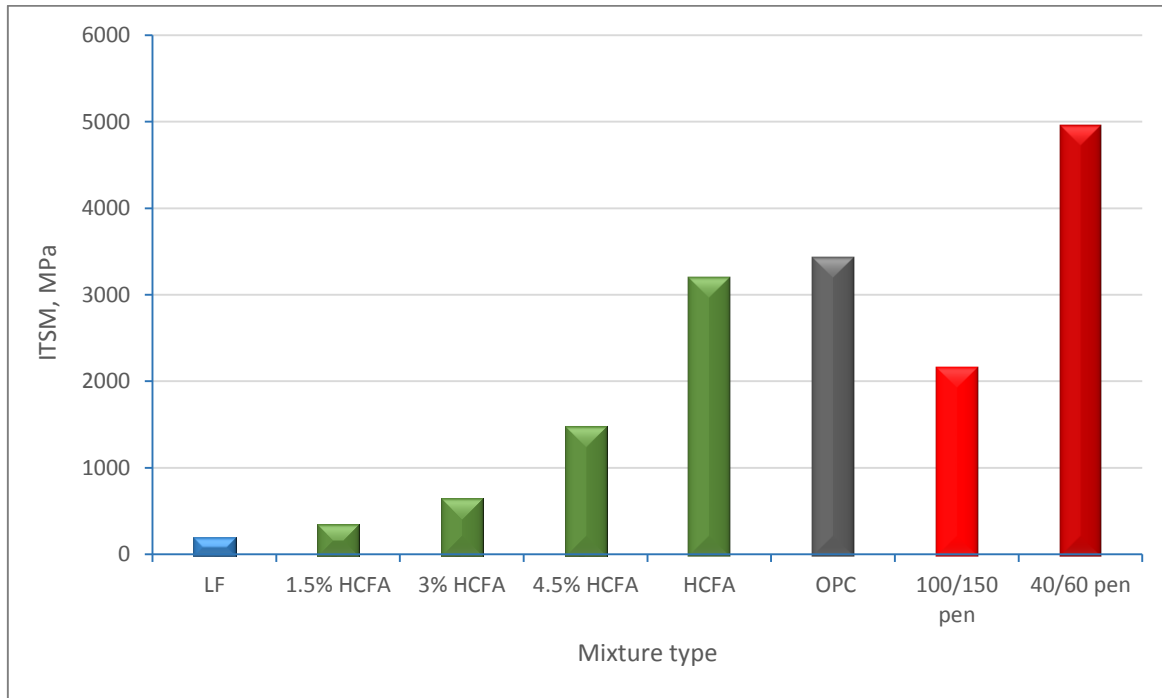
369   time.

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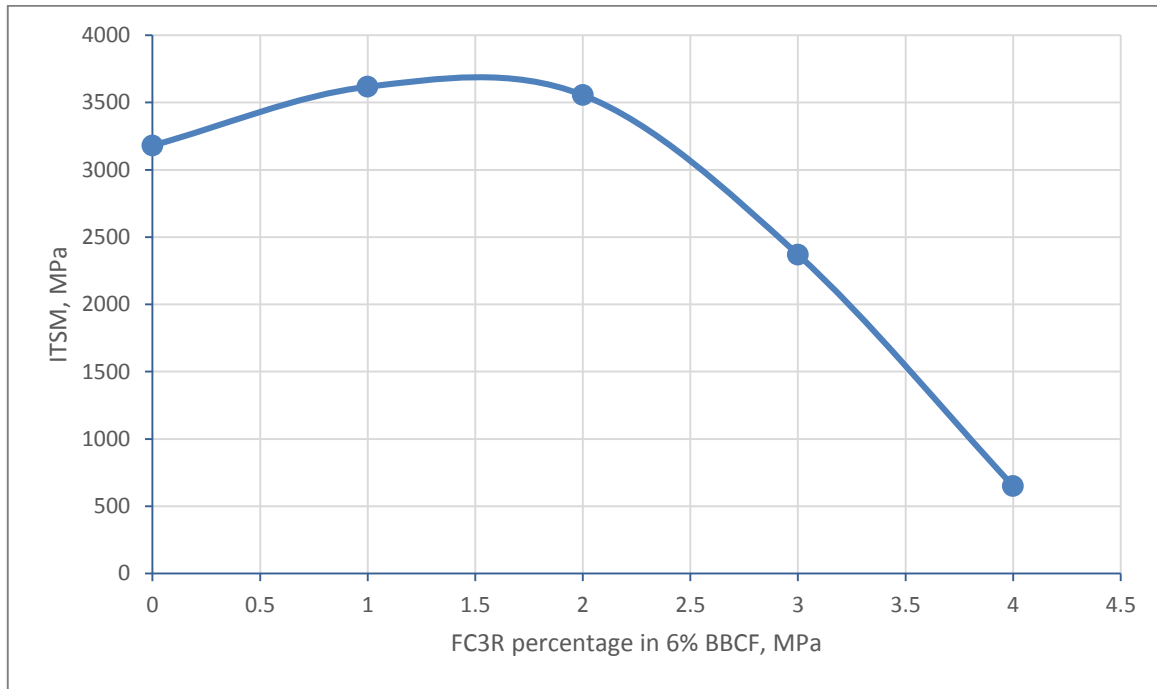
Figure 5. Influence of substitution of HCFA with FC3R on stiffness modulus after 3days

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.
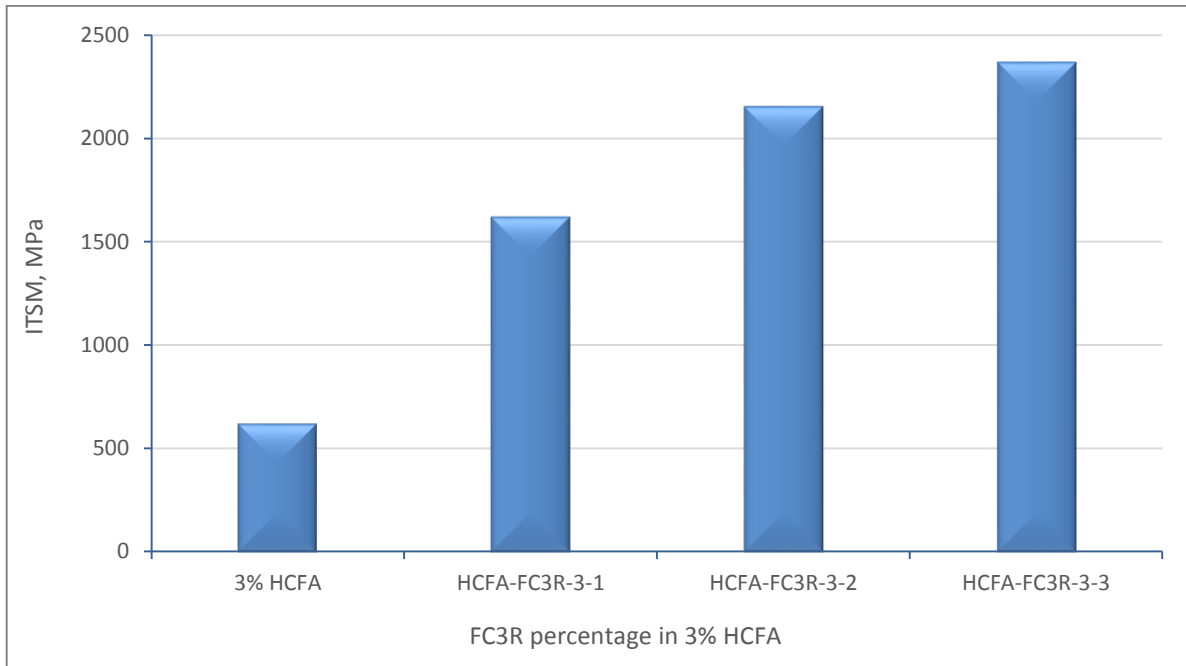
398

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

400   It is clear in Figure 7 which show the results at ages 1, 3, 7, 14 and 28 curing days, that increasing the

401   stiffness modulus of a BBCF treated mixture which has a stiffness modulus more than the OPC treated

402   mixture and also equal to or greater than traditional asphalt concrete hot mixes, will produce a suitable

403   material for use as a binder course layer for major heavy trafficked motorways, by reducing the loads

404   transmitted by traffic to the foundation.

405   In general, it can be seen from Figure 7 that when curing time increases, ITSM develops for HCFA, OPC

406   and BBCF mixtures as a result of the hydration process. The ITSM results improved greatly for BBCF

407   mixtures across all curing times. It worthy to note that BBCF offers more than a 25% increment in ITSM

408   after 1 day when compared to the HCFA as a result of FC3R activation producing more hydration products.

409   It can be seen that BBCF behaviour is the same as that of OPC, however the former offers more ITSM at all

410   curing times.

411   Therefore, BBCF treated mixtures allow early and temporary trafficking where in situ limitations prohibit

412   the installation of a surface course before elimination of traffic management. These materials also eliminate

413   restrictions applied to road engineers using traditional cold binder course by reducing the curing time to less

414   than 1 day.

415   It is worth mentioning that the air voids of the CABC mixtures were 10.53% and 10.27% for HCFA and

416   BBCF mixture while the reference cold LF mixture has 10.93%. These findings reveal an enhancement of

417   volumetric properties for CABC mixtures.

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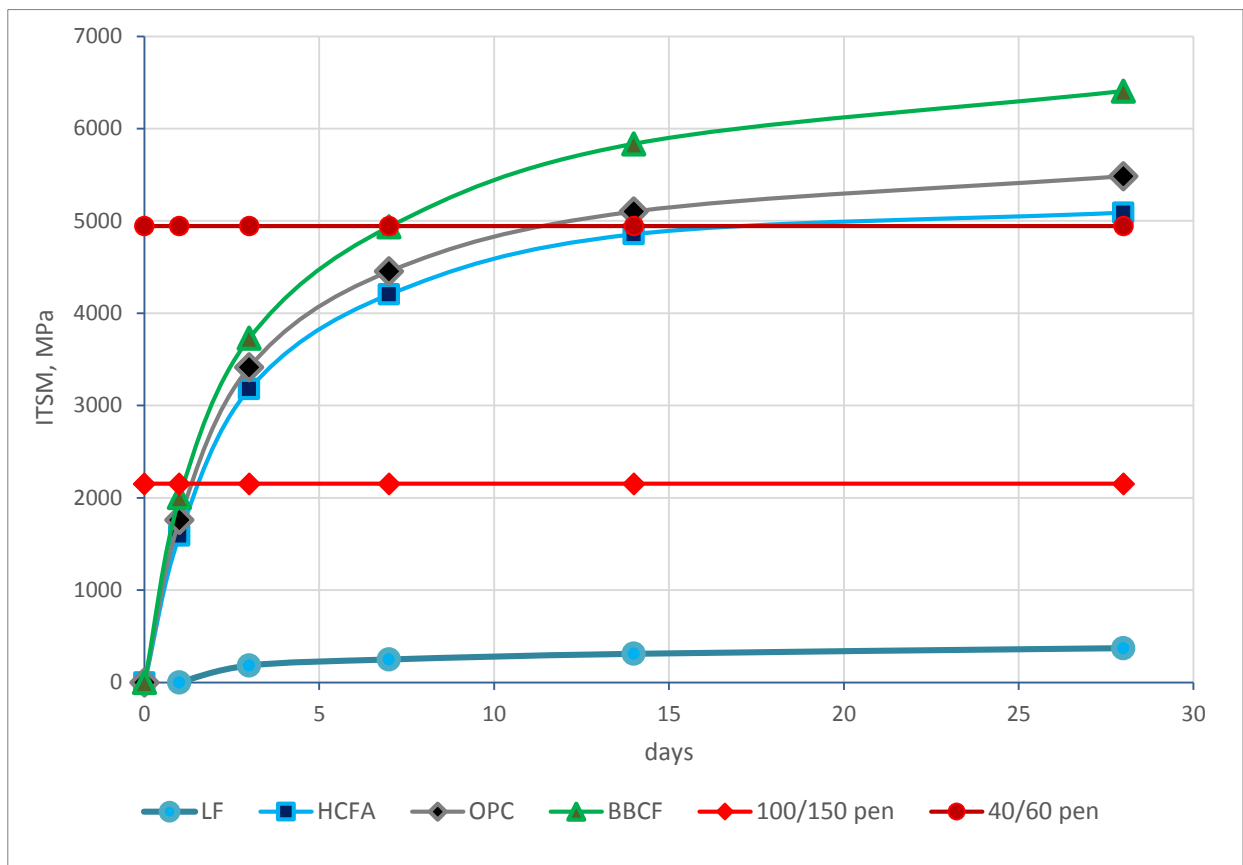


420   Figure 7. Influence of curing time on stiffness modulus

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422   *3.2 Performance in wheel-tracking tests*
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424   All samples were exposed to wheel tracking using the wheel-tracking device following BS EN 12697- 22

425   [39]. Figure 8 illustrates the rut depth at the central point of all slabs as a function of number of cycles.

426   Deformation against number of passes was plotted. From this, it is evident that CACB mixtures with BBCF

427   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
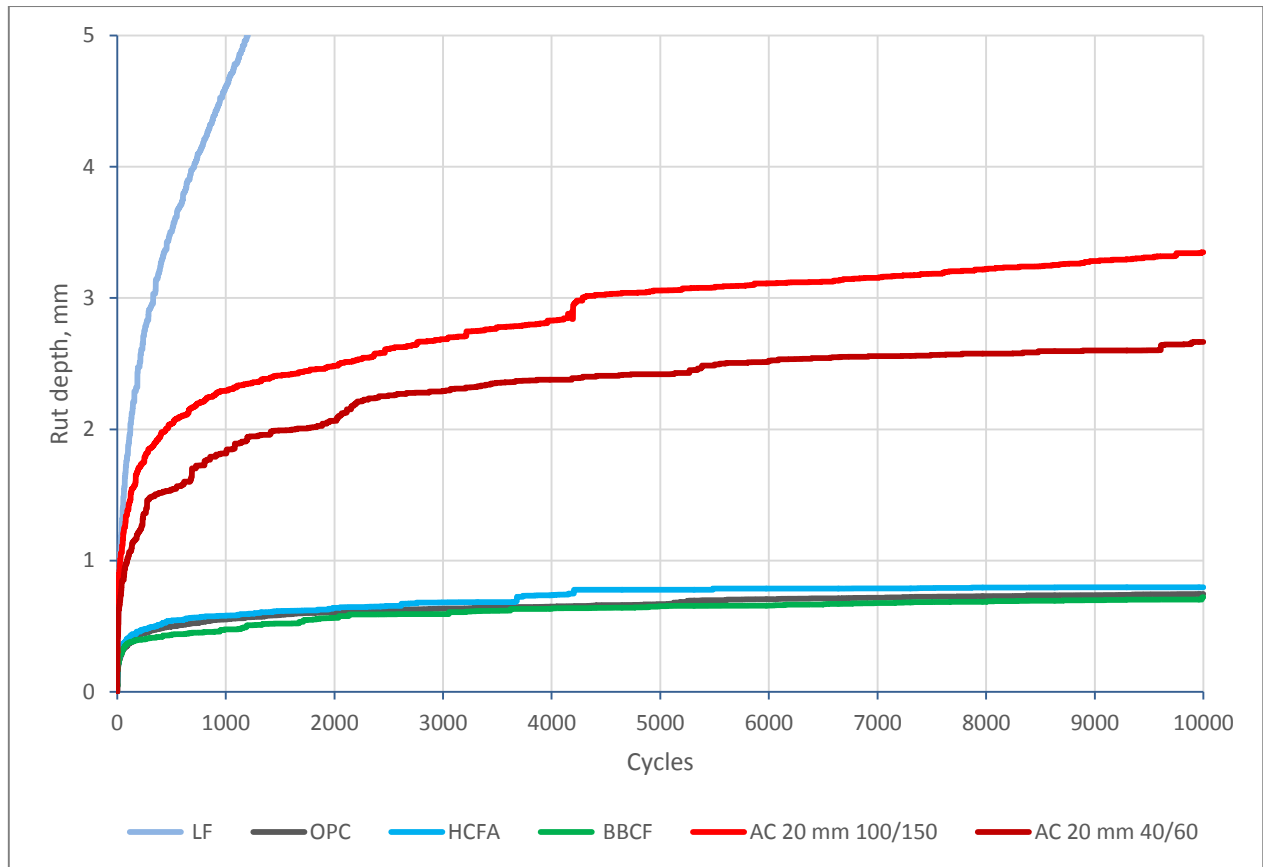
It appears that the rate of rutting in the CACB mixture with BBCF reduces considerably with time. This positive influence of BBCF on the rut resistance of CACB was revealed in specimens with 6% BBCF which had a considerably longer life under the wheel-track test when compared to control samples.

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.
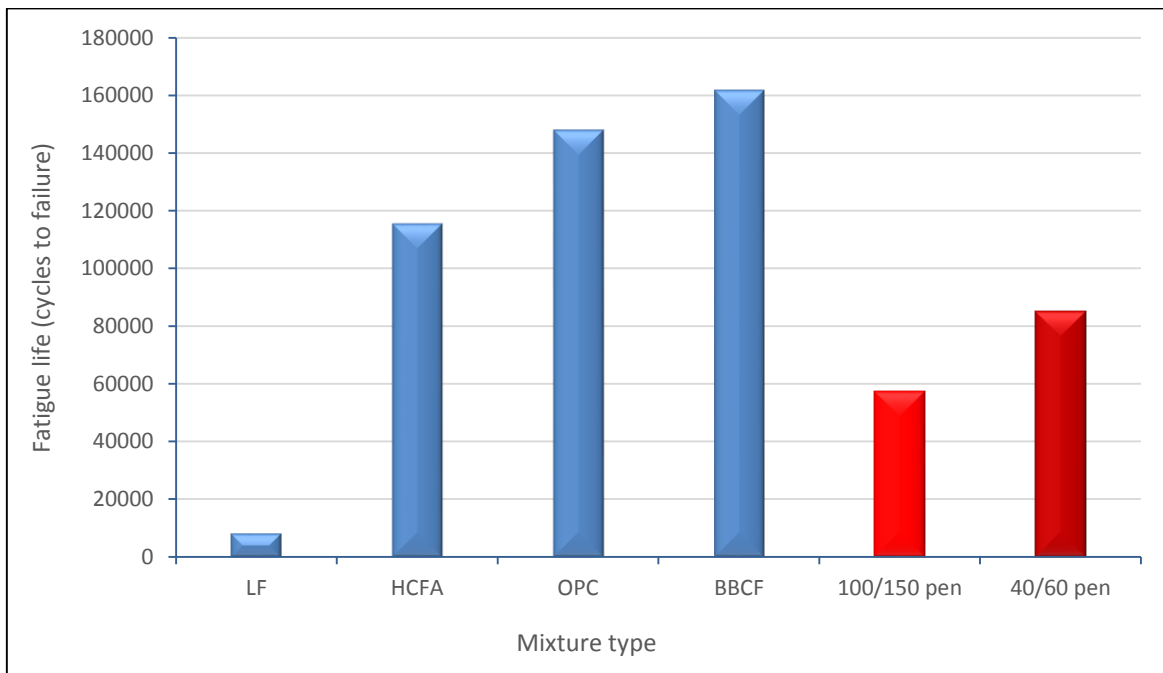

Figure 9. Four-point bending beam fatigue test results

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458   *3.4 Performance in water sensitivity*
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460   Water is the worst enemy of asphalt-concrete mixtures, as the existence of water may cause early failure of

461   a flexible pavement [45]. The water sensitivity of the cold asphalt concrete binder course mixtures was

462   calculated by finding the SMR in accordance with BS EN 12697-12 [38], to examine the impact of both

463   BBCF and HCFA substitutes for the conventional limestone filler. However, ITSM was used instead of

464   indirect tensile strength as recommended by many researchers such as Al-Busaltan et al. [2], Al-Busaltan et

465   al. [5], Al-Hdabi et al. [37].

466   Figure 10 shows that the SMR for CACB mixtures with 6% BBCF and 6% HCFA is more than 100%, which

467   indicates an excellent performance for these mixtures attributable to the hydration process of both fillers.

468   Accordingly, moisture sensitivity was eliminated through developing the bond between the binder and the

469   aggregate and generating a stronger bond with the asphalt binder. These results were better than those for

470   hot asphalt concrete binder course specimens and reached the requirements for bituminous materials. It is

471   worth noting that conditioning of the samples at high temperatures further activates the hydration process.
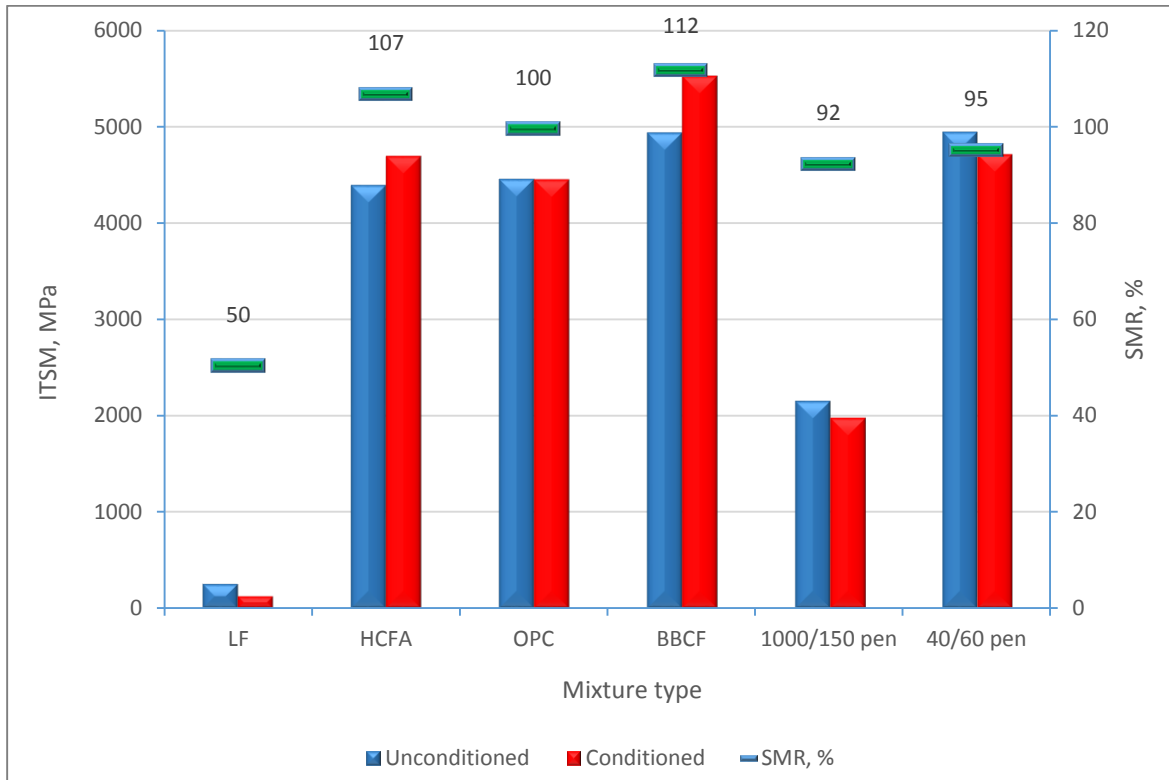
Figure 10. Water sensitivity results

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.

25

487   The SEM micrograph of the fracture surface of BBCF paste after28 days (Figure 11-f), reveals the generation

488   of a gel-like calcium silicate hydrate (CSH) that creates dense microstructure. As a consequence, the material

489   developed a high level of stiffness after 28 days. The CSH phase is the most significant since it creates the

490   essential cementitious or binding characteristics for the final product. The HCFA sample (Figure 11-e) also

491   produces good hydration products such as Portlandite (CH) and CSH gel, however the latter is lower than

492   in the BBCF sample. It was reported by Nassar et al. [20] that a higher degree of hydration in CBEMs as a

493   result of active fillers can produce a dense internal structure with less porosity.

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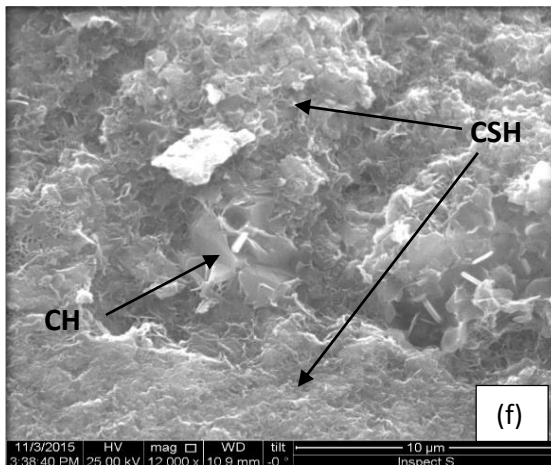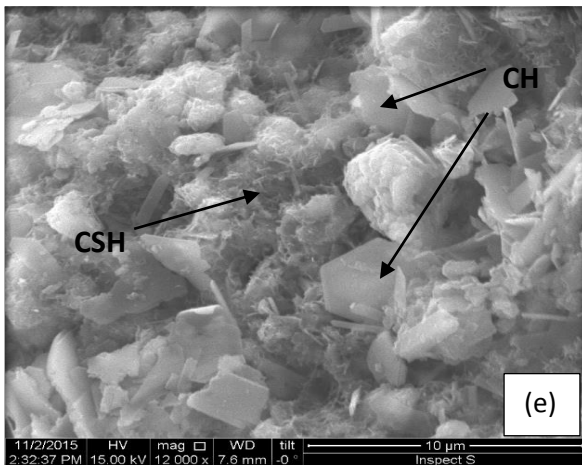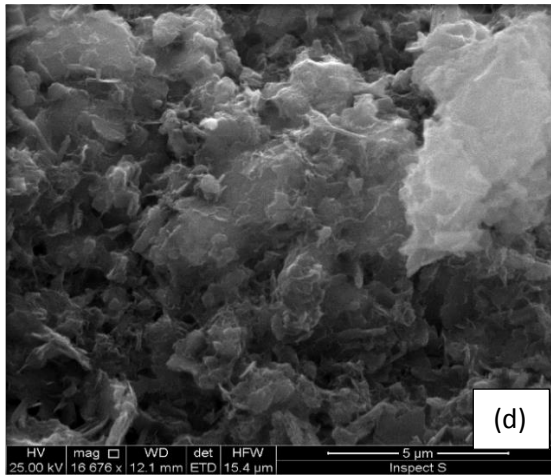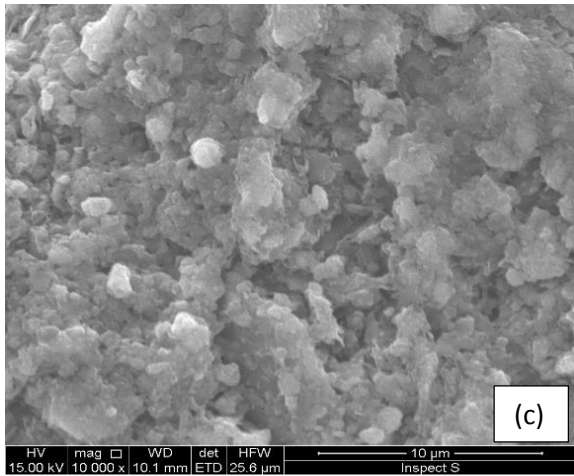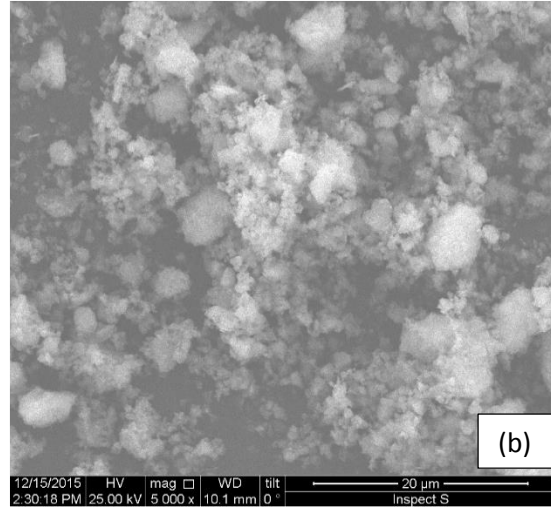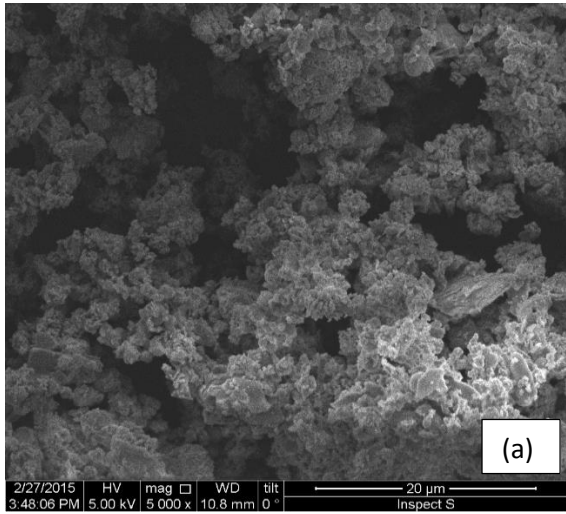
Figure 11. SEM images: (a) morphology of HCFA filler, (b) morphology of FC3R filler, (c) morphology of HCFA paste after 3 days, (d) morphology of BBCF paste after 3 days, (e) morphology of HCFA paste after 28 days, (f) morphology of BBCF paste after 28 days

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

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

536   development.

537   • Replacing conventional limestone filler with waste materials will decrease cement usage in

538   CBEMs and will offer a positive sustainability effect. In addition, the problems relating to carbon

539   emissions (during production) and mixture temperature maintenance (during transportation and

540   laying) in the case of hot mix asphalt, will be mitigated by using this novel CACB.

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