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Performance analysis of a Cold Asphalt Concrete Binder 1 **Course Containing High Calcium Fly Ash Utilizing Waste** 2 Material 3 4 Anmar Dulaimi ^{1,2}, Hassan Al Nageim³, Felicite Ruddock⁴ and Linda Seton⁵ 5 6 ¹ PhD student, Department of Civil Engineering, Liverpool John Moores University, Henry Cotton Building, 7 8 Webster Street, Liverpool L3 2ET, UK, A.F.Dulaimi@2013.ljmu.ac.uk. 9 ² Kerbala University, Kerbala, Iraq. ³ Professor of Structural Engineering, Head of the Liverpool Centre for Materials Technology, Department of 10 Civil Engineering, Liverpool John Moores University, Peter Jost Centre, Byrom Street, Liverpool L3 3AF, UK, 11 12 H.K.Alnageim@ljmu.ac.uk. ⁴ Programme Leader, Department of Civil Engineering, Liverpool John Moores University, Peter Jost Centre, 13 14 Byrom Street, Liverpool L3 3AF, UK, F.M.Ruddock@ljmu.ac.uk. ⁵ Reader, School of Pharmacy and Biomolecular Science, Liverpool John Moores University, James Parsons 15 16 Building, Byrom Street, Liverpool, L3 3AF, UK, L.Seton@ljmu.ac.uk. 17 18 ABSTRACT 19 20 It has been established that cold bituminous emulsion mixtures (CBEMs) have a comparatively low initial strength in comparison to hot mix asphalt (HMA), however its superior performance with regard 21 to carbon emissions, is a significant driver regarding its manufacture. In this research, high calcium fly 22 ash (HCFA) together with a fluid catalytic cracking catalyst (FCC) - a rich silica-alumina waste material 23 24 - have been incorporated to develop a new cold asphalt concrete binder course (CACB) bituminous emulsion mixture. HCFA was used as a substitute for traditional limestone filler while FCC was the 25 additive used to activate the HCFA. The mixtures' performance was assessed using the indirect tensile 26 stiffness modulus test (ITSM), assessment of resistance against permanent deformation, temperature 27 28 and water sensitivity tests. Surface morphology was tested using a scanning electron microscopy (SEM). A considerable improvement was identified by the ITSM test in addition to a substantial enhancement 29 in rutting resistance, temperature susceptibility and water sensitivity. It was also established that the 30 addition of FCC to CACB mixtures was found to improve early strength as well as long-term strength, 31 32 rutting resistance, temperature sensitivity and durability.

33

35 KEYWORDS

- 36 Binder course
- 37 Cold bituminous emulsion mixtures
- 38 Fluid catalytic cracking catalyst
- 39 High calcium fly ash
- 40 Indirect tensile stiffness modulus
- 41 Rutting
- 42 Water sensitivity

43 INTRODUCTION

There are certain restrictions associated with the use of hot mix asphalt (HMA), such as the emission 44 of greenhouse gases and problems in maintaining its temperature when hauling it long distance. 45 Conversely, cold asphalt mixtures (CAMs) defined as bituminous materials mixed utilising cold 46 47 aggregates and binder (Jenkins 2000), can be handled at ambient air temperature. They are produced and compacted at ambient temperature, considered to have a low environmental impact and to be cost-48 effective thus safer for pavement construction (Needham 1996; Oruc et al. 2006). The more popular 49 types of CAMs are cold bitumen emulsion mixtures (CBEMs) the use of which offers advantages in 50 51 terms of material and energy conservation as well as reductions in cost compared to HMA (Thanaya 2003; Gómez-Meijide and Pérez 2014). 52

That said, CAMs have rarely been used as a structural layer for heavy-duty pavements due to their inferior performance in comparison to HMAs (Al-Busaltan et al. 2012b; Doyle et al. 2013). This is generally due to the curing time required to reach their full ultimate strength, leading to unsatisfactory performance represented by inferior early strength and high porosity (Needham 1996; Thanaya 2003; Gómez-Meijide and Pérez 2014). Accordingly, CAM utilisation is still limited to surface treatment and reinstatement work on low-trafficked roads and pavements. However, it should be noted that, CAMs show evolutionary characteristics (Serfass et al. 2004) especially in their early life, where low earlycohesion gradually improves.

61 Needham (1996) reported that the aggregate in CAMs can be used without drying it although the water 62 content must be measured as this has a major effect on the nature of the mixture. Consequently, the 63 major difference between hot mix plants and cold mix plants is the absence of facilities required for 64 heating and drying. In the latter, the aggregate mixtures are fed into a mixing device such as a pug mill 65 or a rolling drum mixer where pre-water is added to wet the aggregate to avoid early break of the 66 emulsion. Bitumen emulsion is then added and mixed until it achieves maximum coating. Over-mixing 67 will cause the emulsion to break as a result of mechanical energy meaning that care must be taken to 68 avoid the production of a stripped or unworkable mixture.

The technology for producing CBEMs for use in the pavement industry has been available in several countries such as the USA and France since the 1970s, these countries building a solid knowledge base about the performance of these mixtures (Leech 1994). However, in the UK, due to the weather conditions which are not optimal for the curing process of emulsion mixtures, this technology has only recently been introduced.

74 Traditional cement has been widely used in CAMs as an enhancement technique as this process 75 produces sufficient strength in a short period of time (Thanaya et al. 2009; Al-Hdabi et al. 2014). Early 76 research conducted by Head (1974) on cement modified asphalt cold mixes revealed that the incorporation of cement had a substantial influence on mix stability; the addition of 1% cement 77 increased stability by 250-300% over that of untreated samples. Furthermore, he reported that asphalt 78 79 cold mix samples prepared without cement, collapsed in water after 24 hours, whereas cement-treated 80 samples showed no deterioration. Research by Oruc et al. (2007) evaluated the mechanical properties of emulsified asphalt mixtures incorporating 0-6% Ordinary Portland Cement (OPC). Their results 81 82 showed significant enhancement with a high OPC addition ratio. In terms of temperature susceptibility, they demonstrated that the amount of cement added results in a substantial increase in the resilient 83 84 modulus and reduction in vulnerability to changes in temperature. They consequently recommended that cement modified asphalt emulsion mixes might be used as a structural pavement layer. Fang et al. 85

(2015) conducted research to accelerate the enhancement of the mechanical properties of cement
bitumen emulsion asphalt (CBEA) by including rapid-hardening cements as a filler replacement in 3
percentages: 0%, 3% and 6% by weight of dry aggregate. They found that after 1 day of curing,
inclusion of calcium sulfoaluminate and calcium aluminate cement to these mixes produced mechanical
properties similar to those realized with Portland cement after 1-week of curing.

However, the cement industry is a material and energy intensive activity which results in an impact on the environment. For example, the manufacture of 1 tonne of cement involves the use of 1.5 tonnes of quarry material involving an energy consumption of 5.6 GJ/tonne which results in emissions approximating 0.9 tonnes of CO_2 representative of 5% of total anthropogenic CO_2 emissions (O'Rourke et al. 2009).

With this in mind, there have been several attempts to use fly ash and waste materials to enhance the 96 properties of CAMs. Thanaya (2003) conducted a study using pulverized fly ash (PFA) finding that it 97 98 can be used as an appropriate filler for CAMs as it produces comparable stiffness to hot mixtures at full 99 curing conditions. An experimental study conducted by Ellis et al. (2004) on a variety of storage macadam comprising reprocessed aggregates bound by bitumen emulsion and Ground Granulated 100 101 Blastfurnace Slag (GGBS), concluded that the incorporation of GGBS to bitumen emulsion mixtures 102 may enhance stiffness and improvements in strength in conditions of high humidity. Another study by 103 Al-Hdabi et al. (2013a) revealed that the mechanical properties of gap graded CRA (cold rolled asphalt) 104 with OPC as the replacement for conventional filler, can be improved by the addition of by-product materials. Gómez-Meijide et al. (2016) investigated the use of 100% recycled construction and 105 demolition waste materials (CDW) to improve the performance of cold asphalt mixtures. They 106 107 concluded that although cold asphalt mixtures with CDW lost water because of longer curing times, greater stiffness could be achieved with CDW in comparison to natural aggregates at any curing time. 108

109 The urgent need to reuse waste materials is driven by environmental factors as well as by economic 110 considerations. Therefore, the replacement of cement with waste materials such as fly ash would be a 111 significant move to improving both economic and environmental impacts. Research conducted by 112 Sadique et al. (2013) examining the pozzolanic reactivity of a calcium rich fly ash through mechanochemical activation with another alkaline ash in a cement-free system, found that the hydration productsand relative development in strength in the new blend was similar to those of cement.

Fluid catalytic cracking catalyst (FCC), is a petrochemical industry waste rich in silica and alumina with similar properties to metakaolin having excellent pozzolanic properties. It has been used in the preparation of geopolymers (Mas et al. 2016) and in the production of alkali-activated binders (Tashima et al. 2014). Both fluid catalyst and microsilica have the same potential to be combined with Ca(OH)₂, as the hydration process is highly exothermic leading to rapid setting of the cement paste (Pacewska et al. 1998).

An asphalt concrete binder course is a continuous graded mixture providing a good aggregate interlock, meaning that this material has very good load-spreading properties as well as a high resistance to permanent deformation (Read and Whiteoak 2003; O'Flaherty 2007). Read and Whiteoak (2003) reported that it is commonly used as a binder course and base in UK road pavements. Its strength originates from the interlocking of coated aggregates providing the fundamental base for the material to transfer load.

Despite extensive research on the manufacture of different types of CBEM, there is currently no specific 127 research into the fabrication of a cold asphalt concrete bituminous emulsion mixture which would be 128 129 adequate for a binder course using bitumen emulsion, containing HCFA activated by FCC. The aim of 130 this study is to develop a new cold asphalt concrete binder course (CACB) mixture incorporating high calcium fly ash as filler replacement and various levels of waste activation material, i.e. FCC, and to 131 examine its mechanical and durability properties. This new CACB mixture will remove constraints 132 imposed by road engineers on the use of CAMs, namely the need for a long curing time ranging from 133 134 2 to 24 months. In addition, the use of these two waste materials will bring about a reduction in cement 135 use in CAMs as well as a decrease in waste disposal which will be of benefit to the environment.

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139 TESTING MATERIAL AND EXPERIMENTAL PLAN

140 **Testing Material**

141 Aggregate

Both coarse and fine crushed granite aggregates from Carnsew Quarry at Mabe in Penryn in the UK were used in this investigation. These are usually used to produce hot asphalt concrete mixtures. Table 144 1 shows the main properties of the aggregate together with the traditional mineral filler which is used. 145 Limestone filler was employed as the traditional mineral filler. A dense aggregate gradation for asphalt 146 concrete binder course AC-20 was used in this research, as shown in Figure 1, for both hot and cold 147 mixtures in accordance with BS EN 13108-1 (European Committee for Standardization 2006).

148 Bitumen emulsion and asphalt

A cationic slow-setting bituminous emulsion (C60B5) produced from paving grade bitumen (100/150) with a residual bitumen content of 60% by mass of emulsion, was used in this research. Nikolaides (1994) indicated that this type of emulsion, i.e. cationic emulsion, is favoured because of its ability to coat the specified aggregate and to create high adhesion between aggregate particles. In addition, 40/60pen and 100/150-pen bitumen grades were used to fabricate two types of hot asphalt concrete binder course mixtures. Tables 2 and 3 illustrate the physical properties of the chosen bituminous emulsion and bituminous binders.

156 Chosen fillers

Two filler types were used in this research: conventional mineral filler, i.e. limestone filler (LF), and high calcium fly ash (HCFA). A waste fluid catalytic cracking catalyst (FCC) was also used for the first time along with HCFA to develop a new cold asphalt concrete binder course (CACB) bituminous emulsion mixture with various proportions (ranging from 1% to 3%) of dry aggregate weight. In addition, a commercially available Ordinary Portland Cement type CEM-II/A/LL 42.5-N was used as the comparison material.

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165 Energy Dispersive X-ray Fluorescence (EDXRF)/XRD analysis

The X-ray fluorescence technique (XRF) was applied to provide elemental analysis of the materials 166 used as filler replacements. The equipment used to carry out the analysis was a Shimadzn EDX 720, 167 energy dispersive X-ray fluorescence spectrometer. X-ray diffraction (XRD) of powdered samples was 168 169 performed using a Rigaku Miniflex diffractometer. The machine uses CuK X-ray radiation and was 170 operated using the following parameters: acceleration voltage 30 kV and current 15 mA at a scanning speed of 2.0 deg. /min in continuous scan mode. The chemical analysis by EDXRF of all the fillers is 171 172 illustrated in Table 4. In summary, the composition of the HCFA was 67.057% of CaO and 24.762% 173 silicon oxide while FCC contained 35.452% silicon oxide and 44.167% aluminium oxide.

Figure 2 reveals that the main crystal peaks identified in XRD of HCFA were lime, calcite, mayenite, merwinite and gehlenite. A similar mineralogy was reported by Sadique et al. (2012a) except that they did not find any merwinite. The powder diffraction in XRD shown in Figure 3 indicates that FCC has very low crystalline peaks of an amorphous nature meaning that it will display high reactivity during the hydration process and can be used as an activator. The powder XRD pattern of the reference OPC as shown in Figure 4, reveals that it was composed of alite, belite, ferrite, calcite and periclase. Figure 5 shows that the main components of the limestone filler are calcite and quartz.

181 Particle size distribution (PSD) and Scanning electron microscopy (SEM) analyses

Particle shapes and sizes play a substantial role in the development of sustainable CBEMs technology. 182 A Beckmen Coulter Laser diffraction particle size analyser LS 13 320 was used to determine the Particle 183 184 Size Distribution (PSD) of the filler materials. From the PSD detailed in Figure 6, it can be seen that the fineness of HCFA is approximately the same as that of OPC with the exception of the range 14-47 185 μ m where it is a little finer than OPC. OPC has more fine particles than HCFA in the range 1.5 to 0 μ m. 186 Most of the FCC particles are in the region of 0.8 μ m to 60 μ m having d₅₀ and d₉₀ equal to 9.16 μ m and 187 188 40.52 μ m respectively. The d₅₀ and d₉₀ for OPC were measured as 11.90 μ m and 41.10 μ m respectively 189 with major particles in the range of 4µm to 60µm. LF composed of 24.22µm and 96.48µm d50 and d90 respectively, meaning that the range of these particles is $3 \mu m$ to $130 \mu m$. 190

Scanning electron microscopy (SEM) analyses was carried out by equipment manufactured by the Inspect S and Guanta to observe the microstructures characteristics. Test conditions were an SEM resolution of 3-4 nm, high vacuum and test voltage 5 kV to 25 kV. The SEM view of HCFA and FCC in Figure 7 shows that they are flaky and agglomerated, whereas it can be seen that OPC and limestone filler particles are irregular in shape with sharp angles. Segui et al. (2012) indicated that highly porous fillers with an agglomerated morphology of the particles will absorb more water, whilst Thanaya (2003) stated that sharp and irregularly shaped particles will interrupt workability.

198 Mix design and specimen preparation

199 Until now, there is no accepted design mixture for CBEMs, neither in the UK nor internationally, but various mix design procedures for CBEMs have been proposed by several authorities and researchers 200 (Asphalt Institute 1989; Jenkins 2000; Thanaya 2003). In this study, the design procedure was 201 established using the method adopted by the Asphalt Institute (Marshall Method for Emulsified Asphalt 202 Aggregate Cold Mixture Design: (Asphalt Institute 1989) for designing cold asphalt concrete binder 203 204 course bituminous emulsion mixtures. Following this procedure, pre-mixing water content, optimum total liquid content at compaction and optimum residual bitumen content were 3.5%, 14% and 6.3%, 205 206 respectively. All the samples were mixed by means of a Hobart mixer. Firstly, coarse and fine aggregate 207 together with the filler material were mixed at low speed for 1 minute with pre-wetting water content 208 (3.5%). Following this, bitumen emulsion (10.5%) was added progressively over the next 30 seconds 209 of mixing after which the mixing process continued for 1 minute 30 seconds at the same speed.

The influence of the substitute conventional limestone filler with HCFA and FCC was tested using the indirect tensile stiffness modulus (ITSM) test. ITSM samples compaction was achieved with 100 blows of a standard Marshall hammer (impact compactor), 50 on each side of the samples, this representing a medium compaction effort according to Thanaya (2003). The compaction equipment setup is illustrated in Figure 8.

The mixtures have been contrasted with two variants of standard asphalt concrete; AC 20 dense binder course 100/150 and AC 20 dense binder course 40/60 have been used throughout the study with the same gradation and type of aggregate. Following the requirements of PD 6691:2010 (European Committee for Standardization 2015), a 4.6% optimum binder content by weight of aggregate was used for each AC 20 dense binder course. All the cold samples were mixed and compacted at room temperature, while the 100/150 and 40/60 hot mixtures were mixed at 150–160°C and 160–170°C respectively, according to the bitumen viscosity and based on PD 6691 (European Committee for Standardization 2015). A cold asphalt concrete binder course containing limestone filler was also used for comparison purposes.

224 Sample curing conditioning

After compaction, the samples for the ITSM test were extracted the following day and were left in the lab at 20°C for normal conditioning at ambient temperature then subjected to ITSM testing at different ages; 3, 7, 14 and 28 days.

Regarding the wheel track slab samples, the slabs were prepared for a mix type of dimensions of 400 × 305 × 50mm and were compacted using a roller compactor according to BS EN 12697-33 (European Committee for Standardization 2003b). The curing conditioning for the slab samples was undertaken in two stages following Thanaya (2003) recommendations. Stage one was performed when the samples were left in their moulds for 24 hours at 20°C, whilst the second stage entailed placing the samples in an oven for 14 days at 40°C ensuring that they reached their full curing condition.

For the water sensitivity samples conditioning, two groups of three specimens for each filler type were prepared and separated. The first group was prepared for the dry conditional test, the specimens left at 20°C for 8 days after preparation. The second group was prepared for the wet conditional test, left at 20°C for 5 days. Following this, a vacuum (6.7 kPa pressure) was applied to the samples for 30 minutes after which they were left submerged for the next 30 minutes where the pressure was decreased slowly to avoid damage to them, thereafter submerged in a water bath for 72 hours at 40°C.

240 Laboratory Testing Program

241 Indirect tensile stiffness modulus (ITSM) test

The stiffness of bituminous mixtures is a significant factor in the analysis and design of flexible pavements, and is directly associated with the capacity of the material to distribute loads (Pasetto and Baldo 2010). The test was carried out on cylindrical specimens following the standard procedure 245 according to BS EN 12697-26 (European Committee for Standardization 2012) using a Cooper Research Technology HYD 25 testing device, as shown in Figure 9. The conditions of the test were as 246 shown in Table 5; the test conducted at a controlled temperature of 20°C. Measuring the ITSM in order 247 to assess the mechanical performance of CAM has been reported by several researchers (Thanaya 2003; 248 249 Monney et al. 2007; Al-Busaltan et al. 2012b; Dulaimi et al. 2016). All indirect tensile stiffness modulus test values are the average of 3 specimens to ensure reliability. It should be noted that Poisson's ratio of 250 0.35 has been adopted as recommended by (Al Nageim et al. 2012; Nassar et al. 2016; Dulaimi et al. 251 2016) for such types of mixtures. All CACB mixture samples were subjected to ITSM testing at ages 3, 252 7, 14 and 28 days. The two reference hot AC mixtures were also tested at the same ages for comparison 253 purposes. The ITSM test was also performed on samples that were 28 days old at different temperatures, 254 namely 5°C, 20°C and 45°C, to explore their susceptibility to temperature changes. 255

256 Wheel-track test

In the lab, wheel-tracking devices are usually utilized to assess the rutting performance of asphalt 257 mixtures subject to environmental and loading conditions in order to simulate actual field conditions. 258 In the current research, the rutting measurement was achieved using the wheel-tracking test in 259 260 accordance with BS EN 12697-22 (European Committee for Standardization 2003a). This test was used 261 to characterize and assess the mechanisms of failure of the CBEMs under set controlled conditions 262 (Ojum 2015; Dulaimi et al. 2016). Five slabs of each mixture type were tracked using a wheel tester. Figure 10 shows a photograph of the HYCZ-5 wheel-tracking equipment used by the Liverpool Centre 263 for Material Technology (LCMT) labs while the test conditions are listed in Table 6. The test was 264 carried out at a temperature of 45°C for 10,000 load cycles. 265

266 Water Sensitivity

Moisture damage is commonly defined as the degradation of the mechanical properties of asphalt mixtures as a result of the presence of moisture in their microstructure (Caro et al. 2008). The principle behind the water sensitivity test is to determine the saturation influence on the specimen as water generates a loss of adhesion within the mastic and the surface of aggregates. Measuring water sensitivity

- 271 in terms of Stiffness Modulus Ratio (SMR) of CAM has been reported by numerous researchers (Al-
- 272 Busaltan et al. 2012b; Al-Busaltan et al. 2012a; Al-Hdabi et al. 2013b).

The test involves the application of the ITSM test in cylindrical specimens subjected to 50 blows per side by means of a Marshall hammer. Two groups of three samples for each level of filler proportion were prepared and separated. Both groups of specimens were tested at 20°C. All samples were tested for indirect stiffness modulus where water sensitivity was evaluated by determining the stiffness modulus ratio (SMR) as the proportion of wet to dry, in accordance to with BS EN 12697-12: 2008 (European Committee for Standardization 2008).

- 279 The SMR was calculated according to Eq. (1):
- 280 $SMR = (ITSM_w / ITSM_d) * 100,$
- $\label{eq:smaller} 281 \qquad \text{where SMR is the stiffness modulus ratio, ITSM_w is the indirect tensile stiffness modulus of the wet}$

specimens while ITSM_d is the indirect tensile stiffness modulus of the dry specimens.

283 Performance Test Results and Discussion

284 Performance of CACB using HCFA and FCC in ITSM test

The first step of this research was to develop new cold asphalt concrete binder course mixtures (CACB) by substituting traditional limestone filler with HCFA in five different substitution percentages, 0%, 1.5%, 3%, 4.5%, and 6% by dry aggregate weight. FCC was added as an extra percentage at 1%, 2% and 3% by dry weight of aggregate as an activator.

From the analysis of the results, it is seen that the stiffness modulus as shown in Figure 11, increased dramatically when the HCFA percentage was increased, achieving its ultimate values when a level of 6% was used. Mixtures with 6% HCFA have the ability to offer a stiffness modulus of around 17 times that of the control mixture which uses limestone filler (0% HCFA) at 3 days. Another interesting point is that the target ITSM for the soft AC 20 dense binder course (100/150 pen bitumen) was achieved within 3 days for the CACB mixtures treated with 6% HCFA. ITSM results also indicated a considerably improvement for HCFA mixtures specifically with 4.5% and 6% replacement of HCFA. There was a considerable improvement of ITSM with time for all the mixtures with HCFA replacements, whereas both grades of HMA showed nonsignificant changes in ITSM over time.

ITSM improvement is due to the generation of an additional binder to the bitumen residue binder as a 298 result of the process of hydration because of the hydraulic reaction of HCFA. This additional binder, 299 300 working together with the bitumen residue binder, improves the strength of the ITSM. A point of interest is that the trapped water which is accountable for the mixtures' weakness, was lost due to HCFA 301 absorption during the hydration process. HCFA particles react in the mix and the subsequent hydration 302 reaction of the HCFA is responsible for the ITSM improvement after 3 days. This provides the 303 additional binder enabling fast curing of the HCFA treated mixture. However, the ITSM for the HCFA 304 mixtures after 3 days was less than that of the OPC treated mixture by approximately 7%. 305

306 The results of ITSM tests in the second stage of testing incorporated another waste material, FCC, which 307 was used as an additive to activate HCFA in different percentages, 1%, 2% and 3% by dry aggregate weight. As shown in Figures 12 and 13, this resulted in further activation of the process of hydration. 308 309 It is expected that soluble calcium hydroxide (C–H), produced from the hydration reaction of HCFA 310 filler, will be converted into dense calcium silicate hydrate (C-S-H) by pozzolanic reaction when 311 adding material with a high silica content (Sadique et al. 2012a). These results revealed considerable 312 improvement in ITSM for all the FCC percentages after 3 days compared with the reference mixtures. 313 Soluble SiO₂ and Al₂O₃ in the glass phase of the pozzolanic materials reacts with Ca(OH)₂ released during hydration to generate an extra calcium silicate hydrate (CSH) gel that is responsible for the 314 mechanical strength of the hardened concrete structure (Lea 1970). 315

Overall, the results are outstanding. The samples exhibited a considerable enhancement in stiffness modulus according to the percentage of FCC added to the HCFA mixtures. As is shown Figure 12, the addition of 1% of FCC to the 6% HCFA mixtures led to an approximate 45% improvement in the stiffness modulus. The addition of 2% and 3% of FCC improved the stiffness modulus by approximately 83% and 102% respectively. Moreover, all of these gains using FCC exceeded the value for the AC 20 mm 100/150-pen after 3 days.

From Figure 14 it can be seen that a considerable improvement was achieved in the stiffness modulusby the addition of FCC to the mixtures having 3% HCFA at an early age. The addition of 1% of FCC

to the mixtures containing 3% HCFA enhanced the ITSM by approximately 160% within 3 days. Mixtures containing 3% HCFA activated by two different percentages of FCC, 2% and 3%, gained approximately 245% and 280% more ITSM in 3 days, respectively. In addition, the stiffness modulus for mixtures having 3% HCFA with 2% and 3% FCC, exceeded the target value for a 100/150 hot asphalt concrete binder course after 3 days. This improvement in the hydration process of HCFA was further enhanced when high silica and alumina FCC waste material was applied as an activating agent in the process of hydration of HCFA.

Figure 15 details the performance of ITSM under different testing temperatures. It can be observed that the control mixture using limestone filler (0% HCFA) failed at 45°C as a result of the weakness of the mixture at high temperature. The rate of change in terms of ITSM for the HCFA-treated mixture and the mixture treated with both HCFA and FCC, was less than both grades of HMAs. These mixtures will perform better than traditional HMAs when temperature changes occur.

336 *Performance of CACB in wheel-track tests*

The assessment of resistance to permanent deformation was achieved thorough the wheel-tracking test in accordance with BS EN 12697-22 (European Committee for Standardization 2003a). Figure 16 illustrates the rut development at the central point of all slabs as a function of the number of cycles. It can be seen that the rut under the wheel path for the reference LF mixture develops rapidly with time, said rut depth after 10,000 cycles at 45°C already exceeding 11mm. In contrast, the rut under the wheel path for the mixtures treated with HCFA and FCC evolves very slowly over time.

As seen in Figure 16, over a comparable cycling scope, rut development is much faster in untreated CAM (6% limestone filler) in comparison to CACB treated with HCFA and FCC indicating the positive impact of HCFA on the rut resistance of CACB. The addition of FCC offered better resistance than AC 20 bin 100/150 and AC 20 bin 40/60.

347 The reactions between HCFA particles and between HCFA with FCC particles were responsible for 348 generating hydration products resulting in rutting improvement. The microstructural integrity produced 349 by successful hydration with the samples treated with HCFA as well as samples treated with HCFA and FCC, accounts for an advanced stiffness capability in addition to a higher resistance to permanentdeformation.

352 Water sensitivity results

As seen in Figure 17, mixtures with HCFA and FCC gave an outstanding performance in terms of water 353 354 sensitivity when compared to reference mixtures, i.e. mixtures with conventional limestone filler, mixtures with OPC and the two grades of conventional hot asphalt concrete binder course mixtures. 355 Mixtures with HCFA and FCC had SMR values of more than 100%, which confirms the improved 356 performance of the two filler materials under wet conditions. The HCFA hydration process, promoted 357 by the addition of FCC during the conditioning period, may have caused this change in material 358 response and thus caused this enhancement. A point of interest is that immersing the specimens in water 359 increased the hydration process and that specimen conditioning at high temperatures (40°C) provides 360 361 good conditions by which to activate the hydration process.

362 Scanning Electron Microscopy (SEM) analyses

363 Scanning electron microscopy (SEM) is a technique for high resolution imaging of surfaces; it reveals the microstructure morphology of particles and the surface characterization of the materials. In the 364 current research, tests were conducted after 3 days and 28 days on selected paste samples taken from 365 the centre of the crushed specimens. Figure 18 presents the SEM photos of the paste samples after 3 366 367 and 28 days for the HCFA and HCFA + 2% FCC pastes. It can be observed that after 3 days there is variation in the absence and presence of FCC and there are considerable variations in the morphology 368 of HCFA +2% FCC paste. This suggests that when the HCFA was activated by FCC, hydration was 369 accelerated and thus responsible for the increased stiffness exhibited by this mixture. In addition, a 370 371 denser structure was developed by adding the FCC compared to the HCFA only sample at 28 days. This dense structure is responsible for the improvement because it forms a bond inside the system. The CH 372 (Portlandite) and CSH (calcium silicate hydrate) phases provide important cementitious binding as well 373 as cohesion characteristic to the final product (Sadique et al. 2012b). 374

376 CONCLUSIONS

377 In this study, several points can be concluded:

The new cold asphalt concrete binder course mixtures enhance pavement performance in terms
 of ITSM, rut resistance and water sensitivity while controlling costs by using obtainable waste
 filler materials that provide cementitious and pozzolanic activity. The use of both HCFA and
 FCC will decrease pollutant waste quantity deposits and their harmful effects on the
 environment.

- The new CACB mixtures comprised of waste filler materials, will remove restrictions on the
 use of CBEMs imposed by road authorities because of lengthy curing time. Replacing
 conventional limestone filler with waste filler materials will decrease cement usage in CBEMs
 and offer enhanced sustainability.
- 387
 3. The tests have established that the use of HCFA results in a substantial improvement in stiffness
 388 modulus. Furthermore, using FCC as an activator for HCFA means that the stiffness modulus
 389 can exceed the stiffness modulus of both grades of HMAs within 3 days.
- 4. Mixtures treated with HCFA and FCC revealed lower thermal susceptibility than traditional hot
 asphalt concrete binder course mixtures. This is considered an outstanding improvement in the
 performance of pavements in hot weather.
- 393 5. CACB mixtures with HCFA and FCC offer considerable improvements to permanent
 394 deformation resistance. The performance of these mixtures was better than two comparative
 395 grades of HMA. The untreated cold binder course exhibited a high rut depth in the wheel-track
 396 test, which indicates poor resistance to permanent deformation.
- Additional encouraging results were found in terms of water sensitivity as the wet stiffness
 modulus results were better than the dry ones. SMR is better than that of conventional HMAs
 and mixtures with OPC and, as a result, meet the requirements for the bituminous mixtures.
- The addition of FCC to HCFA accelerated the hydration of HCFA as evaluated by the ITSM
 test. This was confirmed by the SEM observation that provided evidence of the presence of
 hydrated products.

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540 Figure captions

Figure no.	Title	Conditioning stage
Figure 1	AC 20 mm dense binder course aggregate gradation	/
Figure 2	Powder XRD pattern of HCFA	/
Figure 3	Powder XRD pattern of FCC	/
Figure 4	Powder XRD pattern for OPC	/
Figure 5	Powder XRD pattern for limestone filler	/
Figure 6	Comparative PSD of candidate materials	/
Figure 7	SEM view of filler particles	/
Figure 8	Marshall compaction apparatus	/
Figure 9	ITSM Apparatus	/
Figure 10	A wheel-tracking equipment	/
Figure 11	Effect of HCFA percentage on ITSM results (after 3 days)	4 hr before testing
Figure 12	Effect of adding FCC on ITSM results (after 3 days)	4 hr before testing
Figure 13	Effect of curing time and FCC proportion on ITSM results	4 hr before testing
Figure 14	Effect of adding FCC to 3% HCFA (after 3 days) on ITSM results	4 hr before testing
Figure 15	Temperature sensitivity results	4 hr before testing
Figure 16	Rut depth evolution	4 hr before testing
Figure 17	Water sensitivity results	4 hr before testing
Figure 18	SEM observation	/

546 List of tables

Table no.	Title
Table 1	Physical properties of the aggregate
Table 2	Properties of (C60B5) bitumen emulsion
Table 3	Properties of 40/60 and 100/150 bitumen binders
Table 4	EDXRF analysis of the chosen filler materials, %
Table 5	Conditions of the ITSM Test
Гable 6	Wheel-track test conditions

Material	Property	Valu
	Bulk particle density, Mg/m ³	2.62
Coarse aggregate	Apparent particle density, Mg/m ³	2.67
	Water absorption, %	0.8
	Bulk particle density, Mg/ m ³	2.54
Fine aggregate	Apparent particle density, Mg/ m ³	2.65
	Water absorption, %	1.7
Traditional mineral filler	Particle density, Mg/ m ³	2.57

Table 2. Properties of (C60B5) bitumen emulsion

Description	(C60B5) bitumen emulsion
Туре	Cationic
Appearance	Black to dark brown liquid
Base bitumen	100/150 pen
Bitumen content, (%)	60
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	49	Penetration at 25 °C	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	
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Chemical composition	CaO	SiO ₂	Al ₂ O ₃	MgO	Fe ₂ O ₃	SO_3	K ₂ O	TiO ₂	Na ₂ C
HCFA	67.057	24.762	2.430	2.845	0	0.340	0.266	0.473	1.826
FCC	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	2.25

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	5
No. of test plus	5
Test temperature, (°C)	20 ± 0.5
Specimen thickness, (mm)	63 ± 3
Compaction Marshall	50×2
Specimen temperature conditioning	4 hr before testing

Table 5. Conditions of the ITSM test

Item	Range
Tyre of outside diameter, (mm)	200-205
Tyre width, (mm)	50 ± 5
Total distance of travel, (mm)	230 ± 10
Trolley travel speed, (time/min)	42 ± 1
Contact pressure (MPa)	$0.7{\pm}0.05$
Poisson's ratio	0.35
No. of conditioning cycles	5
No. of test passes	10000
Test temperature, (°C)	45
Specimen dimension, (mm)	$400\times 305\times 50$
Compaction	Roller compactor
Specimen temperature conditioning	4 hr before testing

Table 6. Wheel-track test conditions





Figure 1. AC 20 mm dense binder course aggregate gradation





Figure 2. Powder XRD pattern of HCFA

(L-lime (CaOH)₂, C-calcite (CaCO₃), G-gehlenite (Ca₂Al[Al,SiO₇]), B-belite (2(CaOSiO₂),M-

mayenite (Ca12Al14O33), Mr-merwinite (Ca3Mg[SiO4]))





Figure 3. Powder XRD pattern of FCC

(K- Kyanite (Al₂O₅Si), Q- Quartz (SiO2), M- Mullite(Al₆Si₂O₁₃), Z- Dehydrated Ca-A Zeolite (Al₉₆Ca₄₈O₃₈₄Si₉₆))





Figure 4. Powder XRD pattern for OPC

(A-alite (3CaOSiO₂), B-belite (2CaOSiO₂), C-calcite (CaCO₃), F-ferrite (4CaOAl₂O₃.fe₂O₃), Pc-periclase (MgO))





Figure 5. Powder XRD pattern for limestone filler (C-calcite, Q-quartz)



Figure 6. Comparative PSD of candidate materials



Figure 7. SEM view of filler particles



Figure 8. Marshall compaction apparatus



Figure 9. ITSM Apparatus



Figure 10. A wheel-tracking equipment



Figure 11. Effect of HCFA percentage on ITSM results (after 3 days)



Figure 12. Effect of adding FCC on ITSM results (after 3 days)





Figure 13. Effect of curing time and FCC proportion on ITSM results





Figure 14. Effect of adding FCC to 3% HCFA (after 3 days) on ITSM results



Figure 15. Temperature sensitivity results





Figure 16. Rut depth evolution





Figure 17. Water sensitivity results



Figure 18. SEM observation