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Laboratory Studies to Examine the Properties of a Novel Cold-Asphalt Concrete Binder Course Mixture Containing Binary Blended Cementitious Filler (BBCF)

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29 modulus test by indirect tension to cylindrical samples, wheel-tracking tests and water sensitivity.

Regarding environmental issues, a toxicity characteristic leaching procedure (TCLP) test was 30 performed to analyse the leachate from various specimens comprising concentrations of heavy metal. 31 The findings of these tests have demonstrated that CACB performs extremely well compared to 32 traditional hot mixtures. The stiffness modulus of the BBCF treated mixture - 3730 MPa after 3 days -33 34 is higher than the traditional hot mixture (100/150 pen). In addition, the BBCF treated mixture offered a superior performance regarding rutting resistance, fatigue resistance and water susceptibility as well 35 as revealing a considerably lower thermal sensitivity. More significantly, the BBCF treated mixture was 36 found comparable to the traditional asphalt concrete binder course after a very short curing time (1 day). 37 Finally, the concentration of heavy metals in the specimens incorporating the BBCF was observed to 38 be less than the regulatory levels determined for hazardous materials and so requirements were satisfied. 39 Consequently, this BBCF treated mixture has significant potential with reference to its application as a 40 41 binder course in asphalt pavement.

42 Keywords: Cold bituminous emulsion mixtures, indirect tensile stiffness modulus, rutting, TCLP,
43 waste material and water susceptibility.

44 Introduction

Hot mix asphalt production is accountable for a great deal of energy consumption as a result of the need 45 to heat its constituent parts, aggregates and binder, meaning that greenhouse gas emissions are generated 46 from burning fossil fuels (Rubio et al., 2013). Cold mix asphalt is a technology by which the asphalt 47 mix is produced and laid at normal temperatures. One of the popular types of cold asphalt mixtures is 48 cold bitumen emulsion mixtures (CBEMs). The application of CBEMs in the construction of asphalt 49 pavements has attracted attention in the past few years as it can provide an alternative to traditional hot 50 51 mix asphalt. It has several advantages including environmental protection, economical benefits and meeting health and safety requirements as these mixtures are characterised by production at ambient 52 temperature. Nevertheless, some disadvantages have resulted in cold mix asphalt being considered the 53 poorer option to hot mix asphalt (Thanaya et al., 2009). The long curing time required to achieve full 54 strength, which is generally between 2-24 months (Leech, 1994), weak early strength and possible early 55

distress due to rainfall water intrusion, are regarded as major disadvantages (Brown and Needham, 2000;
Thanaya et al., 2009).

CBEM technology has been applied in several countries including the USA; France has been using CBEMs since the 1970s, annual manufacture in France reaching 1.5 million tonnes in 2014 (European Asphalt Pavement Association (EAPA), 2014). Nevertheless the use of cold emulsified asphalt as a structural layer is very restricted as a result of the longer curing time essential for such materials to reach their full strength after placing, and because of their high sensitivity to rainfall (mainly in the UK) at the early stages of placement (Oruc et al., 2007).

Cold mix asphalt is termed as an evolutive material (Serfass et al., 2004), which means that after it has 64 been laid, this mixture passes through a number of stages in which binder cohesion, binder-aggregate 65 adhesion and mixture shear strength take place (Khalid and Monney, 2009). Enhancements of the 66 mechanical properties and moisture susceptibility of cold asphalt mixtures are limited by the use of 67 cement. An initial study performed by Terrel and Wang (1971) revealed that emulsion mixes treated 68 69 with the addition of cement resulted in an acceleration of the enhancement rate of the resilient modulus. Head (1974) noted that when adding 1% Ordinary Portland Cement (OPC), the Marshall Stability of a 70 71 modified cold asphalt mix was superior (approximately three times better) in comparison to an 72 untreated mix. They also concluded that cement helped the modified mixture to cure under cold and 73 damp conditions. Cement-modified emulsion mixtures were studied by Brown and Needham (2000), 74 their main objective to assess the positive influence of adding OPC to the emulsified mixes. A granite aggregate grading was used in the middle of 20mm dense bituminous macadam with a single slow-75 setting emulsion. From the results, it was concluded that the addition of OPC improved the mechanical 76 77 properties, namely stiffness modulus, permanent deformation resistance and fatigue strength. Oruc et al. (2007) also carried out laboratory investigations to assess the mechanical properties of emulsified 78 asphalt mixtures, including 0-6% OPC, replaced with mineral filler. They concluded that using a high 79 80 percentage of additional OPC resulted in a significant improvement suggesting that cement-modified asphalt emulsion mixtures might be applied as a structural layer. A novel gap-graded Cold Rolled 81 Asphalt (CRA) was developed by Al-Hdabi et al. (2014) by utilising OPC instead of traditional 82

83 limestone mineral filler. They identified a substantial improvement in mechanical properties, namely 84 stiffness modulus, four-point load fatigue, uniaxial creep and semi-circular bending tests in addition to 85 an improvement in water sensitivity. However, Portland cement manufacture consumes a great deal of 86 resources and energy causing CO₂, SO₂ and NOx emissions, this acid rain contributing to the 87 greenhouse effect. Consequently, there will be a significant environmental improvement if industrial 88 by-products can be utilised instead (Li et al., 2008).

Various waste and by-product materials have attracted attention due to their chemical composition as 89 the re-use of different kinds of waste materials in CBEMs is a growing research area promising 90 economic, technical and ecological advantages. The relatively low environmental impact connected 91 with CBEM manufacture can be further decreased while, at the same time, improvements to the quality 92 of CBEMs can be further improved with the application of industrial wastes. Ellis et al. (2004) 93 94 investigated a variety of storage grade macadams composed of recycled aggregates from many sources, bound by bitumen emulsion and Ground Granulated Blastfurnace Slag (GGBS). Their results indicated 95 that stiffness and strength can develop when GGBS is added at high humidity. Thanaya et al. (2006) 96 performed laboratory investigations to assess the performance of a cold mix at full curing conditions by 97 using pulverised fly ash (PFA) as filler, finding that the cold mix stiffness was comparable to that of 98 99 hot mixes. Al Nageim et al. (2012) studied the addition of fly ash and OPC to cold bituminous emulsion 100 mixtures as a filler replacement to examine the development of the mechanical properties of CBEMs 101 and to investigate the possibility of replacing the OPC with fly ash. This new CBEM achieved 102 significant results in comparison to conventional CBEM with and without OPC addition. Al-Hdabi et 103 al. (2013) also investigated the mechanical characteristics and water susceptibility of cold-rolled asphalt 104 (CRA) by using cement as a filler replacement and waste bottom ash (WBA). They concluded that using 105 WBA as an additive enhanced the development of mechanical properties vis a vis stiffness modulus and uniaxial creep tests in addition to water sensitivity. 106

Likewise, Thanaya et al. (2014) investigated the properties of cold asphalt emulsion mixtures (CAEMs)
including milled old road pavement, with and without the inclusion of cement and compaction delays.
They found that improved strength at an early age under tropical room temperatures was governed more

by the evaporation of water than by the inclusion of cement. Gómez-Meijide and Pérez (2014) conducted a study looking to enhance the properties of cold asphalt mixtures in terms of the environmental and economics, by using construction and demolition waste aggregates in such mixtures. From the experimental results it was revealed that the indirect tensile strength, unconfined compression strength, stiffness modulus and susceptibility to moisture were tolerable, not only in comparison to a control mix with 100% natural aggregates (NA), but also with values provided by various standards and recommendations.

117 Fluid catalytic cracking catalyst residue (FC3R) is an industrial by-product produced by the fluid 118 catalytic cracking processes in petrol refineries. Some studies have revealed that FC3R has the ability 119 to increase mechanical properties in mortars or concretes because of a densification of the cementitious 120 matrix generated by pozzolanic reaction. Pacewska et al. (1998) studied the hydration of cement paste 121 as a function of adding a spent catalyst to address catalytic cracking. Their research detailed the 122 pozzolanic nature of the spent catalyst. They found that the spent catalyst and microsilica had a similar potential to be combined with Ca(OH)₂, and that the hydration process was highly exothermic, 123 facilitating rapid setting of the cement paste. Payá et al. (1999) revealed that the substitution of cement 124 by FC3R in mortars created a significant increase in compressive strength, one that overcomes plain 125 126 cement mortar, possibly as a result of a pozzolanic reaction. Consequently, research indicates that FC3R has the potential to be used as a supplementary cementitious material (SCM) which can be substituted 127 128 for cement.

Therefore, from the point of view of economics, environmental concerns and safety, CBEMs are useful 129 in the construction of asphalt pavements. However, the mechanical properties and water damage 130 131 resistance of material using waste materials has not been well documented. Only a few researchers have examined the possibility of applying waste materials as filler replacement in CBEMs but there is no 132 research examining high calcium fly ash activation by fluid catalytic cracking catalyst (FC3R) in order 133 to produce a binary blended cement filler (BBCF). The current research aimed at developing fast-curing 134 cold emulsion asphalt mixtures for binder course materials in road pavements, with the goal of 135 decreasing the disposal of waste and raw materials and contributing to the improvement of sustainable, 136

137 cleaner production practices. Since the success of this technology is subject to performance, this study aimed to assess the performance of CACB, with or without BBCF, when compared to the hot asphalt 138 concrete binder course mixtures. A laboratory programme covered the stiffness modulus, rutting 139 resistance, fatigue resistance and moisture damage resistance, assessed through indirect tensile stiffness 140 141 modulus tests on cylindrical specimens, wheel-track tests, four-point bending tests on prismatic shaped samples and stiffness modulus ratio tests, respectively. Testing of waste material characteristics such as 142 morphology using a scanning electron microscope (SEM), chemical and mineralogical composition 143 utilising X-ray fluorescence (XRF) and X-ray diffraction (XRD) techniques, have been carried out. 144

145 Materials and methods

146 Aggregates

A commercial granite aggregate of coarse and fine fractions supplied by Carnsew Quarry at Mabe, Penryn in the UK, was used in this research to manufacture all the hot and cold mixtures. As shown in Figure 1, a dense gradation for binder course AC-20 mm was used. The aggregate gradation in this research is based on EN 13108-1 (European Committee for Standardization, 2006), the common properties of the aggregates presented in Table 1.

152 Emulsion and binder

The binder was a standard cationic bitumen emulsion (C60B5) (60% bitumen content) with 100/150 pen. This kind of emulsion is designed for use in road pavements and common maintenance applications. Nikolaides (1994) confirmed that cationic emulsion is preferred because of its ability to coat aggregates and to guarantee high adhesion between the particles of said aggregates. With reference to the conventional hot asphalt mixture, a 40/60 and 100/150 penetration-grade bitumen was used. Tables 2 and 3 detail the primary properties of these binders.

159 Fillers

160 Two fillers which are considered industrial waste, were analysed in this research: high calcium fly ash 161 (HCFA) which is produced from power generation plants by combustion between 850°C and 1100°C 162 using the fluidised bed combustion (FBC) system, and fluid catalytic cracking catalyst (FC3R), which is a by-product material produced in petrol refineries from the fluidised bed process. In addition, typical
traditional limestone filler (LF) was used for the reference cold mixture.

165 Filler characterisation

The chemical composition of the fillers was examined using X-ray fluorescence (XRF) while the 166 mineralogical properties were inspected using X-ray diffraction (XRD) techniques. Table 4 details the 167 168 chemical structure of the three fillers tested as revealed by the energy dispersive X-ray fluorescence (EDXRF) spectrometer test while Figures 2 and 3 present the mineralogical composition from the XRD 169 test results. It can be seen from Table 4 that HCFA is composed of CaO with a suitable quantity of SiO₂ 170 and Al₂O₃. Conversely, FC3R contains Al₂O₃ and SiO₂ as the main oxides. Lea (1970) reported that 171 soluble SiO₂ and Al₂O₃ present in the glass phase of pozzolanic materials, have an important role to 172 play in that they react with the Ca(OH)₂ released through the hydration of cements to form an extra 173 calcium silicate hydrate (CSH) gel that enhances the mechanical strength of the hardened concrete 174 175 structure.

176 The powder XRD pattern of HCFA in XRD shown in Figure 2 indicates that the HCFA is crystalline as it contains sharp peaks without substantial noise in the background; the major crystal peaks identified 177 were lime (CaO), calcite (CaCO₃), mayenite (Ca₁₂Al₁₄O₃₃), merwinite (Ca₃Mg[SiO₄]) and gehlenite 178 179 (CaAl[Al,SiO₇]). The powder diffraction in XRD shown in Figure 3 indicates that FC3R has very low crystalline peaks which are amorphous in nature. This means that it will demonstrate high reactivity 180 during the hydration process and can be used as a pozzolanic material, making this material a potential 181 precursor in the production of a new BBCF. The crystalline peaks identified comprised kyanite 182 183 (Al₂O₅Si), quartz (SiO₂), mullite (Al6Si₂O₁₃) and dehydrated Ca-A zeolite (Al₉₆Ca₄₈O₃₈4Si₉₆).

184

185 Mixture production

The Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14) procedure, as
specified by the Asphalt Institute (1989), was used to design the cold asphalt concrete binder course
bituminous emulsion mixtures.

Various pre-wetting water contents (PWWC) were examined to select the lowest pre-water ratio and as a result, adequate coating can be confirmed. Indirect tensile stiffness modulus tests were used to determine the optimum emulsion content (OEC), while the mix density test was employed to define the optimum total liquid content at compaction (OTLC). Consistent with this procedure, PWWC, OTLC and optimum residual bitumen contents were 3.5%, 14% and 6.3%, respectively.

Incorporation of the HCFA was carried out with a partial substitution of the conventional mineral filler with various proportions of HCFA (0%, 1.5%, 3%, 4.5% and 6%) by dry aggregate total mass, while FC3R was used as the activator in four ratios (1%, 2%, 3% and 4%) by dry aggregate total mass to replace the HCFA in order to generate a binary blended cement filler (BBCF).

The materials were mixed in a Hobart mixer where the aggregate, filler and PWWC were added and mixed for 60 seconds at low speed. The bitumen emulsion was slowly introduced over the following 30 seconds of mixing, the mixing process continuing for the next 120 seconds at the same speed. Samples were mixed and placed in moulds then compacted using a Marshall hammer with 50 blows per face. The samples were left in their moulds at room temperature (20±1°C) for 24 hours then demoulded. The samples were then cured at room temperature before they were used for ITSM tests.

204 The indirect tensile stiffness modulus test has been performed to investigate the influence of the 205 substitution of LF with HCFA and of FC3R inclusion, the results compared to a standard AC 20mm hot 206 dense binder course. Two types of hot binder course mix, namely AC 20mm dense binder course 207 100/150 and AC 20mm dense binder course 40/60, have been used throughout this research with the 208 same aggregate type and gradation. 4.6% optimum binder content by weight of aggregate was used 209 according to the PD 6691:2010 (European Committee for Standardization, 2015) for the AC 20mm 210 dense binder course. Specimens were fabricated and compacted at lab temperature (20°C), while the 100/150 and 40/60 hot mixtures were mixed at 150-160°C and 160-170°C, respectively. In addition, a 211 cold asphalt concrete binder course containing limestone filler (LF) was used for the purpose of 212 comparison. Every indirect stiffness modulus test value is the average of 5 specimens to ensure the 213 reliability of the results. 214

215

217 Laboratory testing programme

218 The performance tests covered the stiffness modulus, temperature susceptibility, rutting resistance, 219 fatigue resistance and moisture susceptibility, assessed through the indirect tension of cylindrical 220 specimens, wheel-track tests, four-point bending tests and stiffness modulus ratio tests respectively.

221 Indirect tensile stiffness modulus (ITSM) test

222 Stiffness of bituminous mix is associated with the capacity of such material to distribute traffic loads, meaning it can be considered as a synthetic indicator of their structural properties. The stiffness modulus 223 of asphalt mixtures is one of the most significant characteristics in the design of flexible pavements. 224 The test used here applied indirect tension to cylindrical samples in accordance with BS EN 12697-26 225 226 (European Committee for Standardization, 2012b), using a Cooper Research Technology HYD 25 testing machine, as shown in Figure 4, performed under the conditions given in Table 5. ITSM is 227 determined by applying 5 repeated loads, preceded by a pre-loading of 10 repetitions of load, which has 228 the function of correcting the load application system to the sample. All ITSM tests were conducted at 229 230 20°C, the samples conditioned for a 4-hour period to guarantee the test temperature as stipulated in the 231 above-mentioned standard EN 12697-26. Many researchers such as Monney et al. (2007); Al-Busaltan et al. (2012); Nassar et al. (2016) and (Dulaimi et al., 2016) have used ITSM in order to assess the 232 233 stiffness modulus of CBEMs as this test is straightforward and can be executed quickly in comparison 234 to other conventional methods of testing (Oke, 2010). ITSM tests were performed at various testing temperatures, 5, 20 and 45°C, to gain a measure of the temperature sensitivity of the CACB mixtures 235 236 and the control mixtures.

237 Wheel-tracking test

The wheel-tracking test has been utilised by numerous researchers to assess asphalt mixtures' resistance to rutting (Bodin et al., 2009; Ma et al., 2015). In this study, wheel-tracking tests were conducted at 45°C to assess rutting resistance. The test slab specimen with a length of 400mm, width of 305mm and thickness of 50mm was compacted by a roller compactor in accordance with BS EN 12697-33 (European Committee for Standardization, 2003b), as shown in Figure 5. Wheel-track tests were conducted for all cold mixtures at full curing condition, which comprises two stages. The first stage was completed by leaving the slabs in their moulds for 1 day at lab temperature, 20°C. Stage two required the slab samples to be placed into a ventilated oven at 40°C for 14 days' curing to achieve their constant mass as recommended by Thanaya (2003) and Al-Hdabi et al. (2014). The curing temperature used is important as it needs to be below the softening point of the bitumen and thus stop the bitumen from ageing (Cardone et al., 2014; Kuna, 2015). The slab samples were then cooled at lab temperature before testing commenced.

In this test, a rubber tyre is moved back and forth in the centre of the slab sample at a speed of 42 passes/min., with a contact pressure of 700 N and contact width of 50mm. The vertical displacement of the slab sample along the wheel path was measured by linear variable differential transformers (LVDTs), the final vertical deformation an indicator of resistance to rutting. The wheel-track machine type HYCZ-5 shown in Figure 6, is used by the Liverpool Centre for Materials Technology (LCMT) to perform the tests following BS EN 12697-26 (European Committee for Standardization, 2003a). Table 6 details the test conditions.

257 Fatigue resistance

Fatigue cracking is one of the main structural distress modes found in layers of bituminous road 258 259 pavement due to the repeated application of traffic-induced stresses which can lead to a substantial decrease in the serviceability of flexible pavements. In this research, an investigation of fatigue 260 performance of the CACB mixtures and hot mixtures was performed using a four point bending test 261 following the standard BS EN 12697-24 (European Committee for Standardization, 2012a) at a test 262 temperature of 20°C using prismatic shaped samples (400 x 50 x 50mm) dimensions samples. Slab 263 specimens were prepared in accordance with the same mixing method detailed in Section 3.2, subject 264 to full curing conditions detailed earlier for wheel track samples after which five prismatic shaped 265 specimens were produced from each slab using a machine saw. The frequency was 10Hz under 266 sinusoidal loading with no rest period and 150 microstrain controlled strain as recommended by Al-267 268 Hdabi et al. (2014).

Fatigue failure has been defined as cycles number (Nf), at which the initial stiffness is decreased by 50%. A Linear variable differential transformer (LVDT) located at the top of the beam was used to measure the vertical deflection at the centre of the beam together with the applied load to determine stresses and strains. The test set up is illustrated in Figure 7.

273 Moisture damage resistance

274 The presence of moisture in an asphalt mixture can result in deterioration of the bonds between the aggregate and bitumen. This can lead to adhesive as well as cohesive weakening of the mixture which 275 in turn results in a reduction in its strength, load-carrying capacity and durability of the pavement. 276 Therefore, a water sensitivity assessment is essential because it is directly associated with the durability 277 performance of mixtures throughout the pavement life. Water sensitivity characterisation of the 278 bituminous mixtures followed the standard procedure in BS EN 12697-12 (European Committee for 279 Standardization, 2008). The production of cylindrical specimens consists of manufacturing a set of 280 samples which is divided into two equal subsets. The first subset is kept dry (dry samples) at a 281 temperature of 20°C for 7 days after 1 day inside the mould during preparation, whereas the other subset 282 is wet (wet samples). These specimens were left at 20°C for 4 days after 1 day in the mould. They were 283 then subjected to a vacuum at 20°C, left for 30 minutes under an absolute pressure of 6.7 kPa and then 284 285 left immersed for another 30 min. Following this, they were soaked and kept in water at 40°C for 3 days. 286 The two sets of samples underwent an ITSM test following EN 12697-26 (European Committee for 287 Standardization, 2012b). From these test results, the stiffness modulus ratio (SMR) was determined as follows: 288

289 SMR = (wet stiffness / dry stiffness) \times 100

290 Effect on the environment – leaching of metals into water

The two industrial wastes used, HCFA and FC3R, were measured using the toxic characteristic leachability procedure (TCLP) standard in terms of the release of heavy metals by leaching. TCLP is one of the major leaching procedure tests used to examine the risk of heavy metal leachability from stabilised layers (Xue et al., 2009). In this research, a TCLP test was applied to measure the leached concentrations of cadmium (Cd), chromium (Cr), lead (Pb), arsenic (As), barium (Ba), nickel (Ni),
copper (Cu) and zinc (Zn) from both the BBCF and HCFA treated mixtures. The tests were carried out
following the procedure adopted by Xue et al. (2009) and Modarres and Ayar (2014).

The BBCF and HCFA treated mixtures were prepared in the laboratory. A stock of TCLP leachant was 298 299 prepared by mixing stoichiometric amounts of deionised water and acetic acid (pH 2.88). Weighted 300 amounts (10 grams) of the crushed samples were placed in bottles containing 200 mL of the TCLP 301 leachant. These bottles were then shaken using a rotary extractor at 30 rpm for 18 hours. All experiments 302 were conducted at a temperature of 20°C. After the extraction process, the solutions were filtered using 303 a 47mm glass fibre filter then acidified using acetic acid to a pH below 2. The concentrations of heavy 304 metals were measured using an atomic adsorption spectrophotometer (type: Thermo, Model: ICE 3300), 305 as shown in Figure 8.

306 Results and discussion

307 Performance in the indirect tensile stiffness modulus (ITSM) test

This study examined the effects of HCFA and BBCF with cationic slow-setting bitumen emulsion on the short- and long-term performance of binder course properties using laboratory mechanistic assessment. The method used followed the European standard BS EN 12627-26 (European Committee for Standardization, 2012b). Five specimens were prepared for each mixture type.

High calcium fly ash (HCFA) was included as a substitute for conventional limestone filler (LF) of 312 313 various proportions (0%, 1.5%, 3%, 4.5% and 6%) by total mass of dry aggregate for ITSM testing. 314 The results of these tests are illustrated in Figure 9 where it can be seen that the stiffness modulus of the HCFA treated mixtures was higher than that of the cold emulsion mixture with 6% LF. Stiffness 315 levels depended on the HCFA percentage. The highest ITSM was 3181 MPa for 6% HCFA treated 316 317 mixture after 3 days, which is a 17 fold increase compared to the reference limestone treated mixture. 318 Comparing this with the hot asphalt concrete binder course mixture, it can be seen that using HCFA 319 gives a stiffness modulus 47.8% higher than that obtained for the traditional AC 20mm with 100/150 320 pen.

The improvements seen in the ITSM test are due to the consumption of trapped water and formation of an additional binder from the process of hydration of HCFA. Using HCFA in CACB had a positive effect on the stiffness modulus, particularly within the range of 4.5% to 6% filler replacement. When the HCFA content was 6%, the influence on the stiffness modulus was more noticeable.

325 Waste material with a high aluminosilicate (FC3R) was included as the supplementary cementitious material and worked as an activator in four proportions (1%, 2%, 3% and 4%) by dry aggregate weight 326 as a substitute for HCFA. FC3R is rich in pozzolanic particles which help to speed up the hydration of 327 328 the HCFA particles, leading to the production of more hydrated products. It can be noted from Figure 329 10 that the addition of FC3R to the HCFA improved both early as well as long-term strength. As 330 mentioned above, the FC3R pozzolanic particles reacted with the Ca(OH)₂ released during the hydration process resulting in an accelerated hydration of the HCFA particles. Consequently, more hydrated 331 332 products were formed. A balanced oxide composition was expected to be formed within the BBCF. The 333 new BBCF treated mixture was also found to have comparable ITSM to that of the conventional hot asphalt concrete binder course mixture AC 20 mm with 100/150 pen, this in less than 1 day of curing. 334

335 The hydration of the HCFA particles were been accelerated by existence of the pozzolanic particles provided by the FC3R which generated more hydrated products. The inclusion of pozzolanic materials 336 337 with a high silica material transformed the soluble calcium hydroxide (C-H) formed from the HCFA 338 hydration reaction, into a dense calcium silicate hydrate (C-S-H) through a pozzolanic reaction (Lothenbach et al., 2011; Sadique et al., 2012). In addition, the formation of hydrous silicates was 339 accompanied by the formation of hydrous calcium aluminates in the pozzolanic materials which include 340 substantial amounts of Al₂O₃ (Morsy et al., 1998). These changes in the materials' structure led to 341 342 improvements in their mechanical strength (Chan and Xihuang, 1999).

It has been demonstrated that there is a considerable development in the stiffness modulus of the specimens containing up to 2% FC3R replacement. This function uncovered its pozzolanic activity, something which was reported by Payá et al. (1999). Therefore, a new binary blended cement filler (BBCF) with 4.5% of HCFA and 1.5% of FC3R may be recommended. This BBCF mixture generated more than a 17% increment in stiffness modulus compared with the HCFA treated mixture after 3 days. However, increasing the percentages of FC3R more than 2% lead to a reduction in ITSM due to the reduction in HCFA percentage and thus decreased the amount of Ca(OH)₂ released as a function of the hydration process which results in some of the FC3R particles showing no reaction.

Figure 11 shows that the treated BBCF mixture offers a substantial stiffness modulus in comparison to the HCFA and cold limestone mixtures. Furthermore, the rate of stiffness modulus improvement was clearly higher up to 7 days, when a reduction in this rate was detected. The target stiffness (2000 MPa) can be achieved in 1 day's curing using the BBCF treated mixture, this meeting the British and European requirements in terms of ITSM. Consequently, a new cementitious material made completely from waste materials has been recommended for use in CBEMs.

357 Temperature sensitivity performance

Figure 12 presents the temperature susceptibility results of all the cold and hot mixtures. The slope of the curve in a semi-logarithmic plane characterises temperature susceptibility where the greater the rate of change, the more temperature sensitive the mixture. The results of ITSM for the LF treated mixture depends significantly on the test temperature applied, which makes these mixtures fail immediately at 45°C. However, BBCF and HCFA treated mixtures revealed a considerable lower thermal sensitivity than the two conventional hot asphalt concrete binder course mixtures.

364 Rutting resistance performance

The sensitivity of a bituminous material to rut is measured by a wheel-tracking test through a loaded 365 366 wheel making repeated passes at a fixed temperature, this simulating the effect of traffic. The BBCF treated mixture, HCFA mixture, reference limestone mixture and hot asphalt concrete mixtures were 367 subjected to this test. As seen in Figure 13, the rutting resistance for the limestone reference mixture 368 369 dropped considerably; the cold limestone mixture is especially susceptible to rutting due to the long 370 time this mixture needs to achieve an acceptable performance. Nevertheless, when the BBCF filler was used, the resistance to rutting increased notably to a level even greater than that of the hot asphalt 371 concrete mixture. In detail, the proportional depths of the mixtures' ruts after 10,000 cycles are 1.4%, 372 1.6%, 23.6 %, 5.3% and 6.7% for the BBCF, HCFA, LF, hot AC 20 40/60-pen and hot AC 100/150-373

pen mixtures respectively. This shows that the rutting resistance of the HCFA treated mixture is enhanced after the addition of FC3R. In terms of the rutting resistance required in practice, it is obvious that the rutting resistance of the BBCF treated mixture is better than both the hot asphalt concrete mixtures. The major reason for this is the higher stiffness of this mixture. Without the new BBCF or even the HCFA, cold limestone is more prone to rutting. This can be attributed to the increase in hydration products being generated by adding FC3R to the BBCF treated mixture.

380 Fatigue performance

The fatigue cracking resistance of the LF, HCFA, BBCF and the two reference hot mixtures were 381 382 achieved by using the four-point bending test following BS EN 12697-24 (European Committee for Standardization, 2012a). Fatigue life is defined as the total amount of cycles which produce a 50% 383 reduction in initial stiffness. The results in Figure 14 show the significant variance in fatigue life 384 obtained from the 150 µstrain level at 20°C for both BBCF and LF which was analogous to the outcomes 385 for permanent deformation performance discussed earlier in section 4.3. The fatigue life values are 386 much higher than the corresponding two traditional hot mixtures; the inclusion of BBCF extends fatigue 387 life more than 19 times in comparison to the reference LF mixture, while HCFA extends fatigue life 14 388 times more in comparison to the control LF mixture. Consequently, including BBCF in CACB mixtures 389 390 considerably extends the fatigue life.

391 It was reported that the potential strain levels that might be experienced in a pavement structure are 392 below 200 microstarin (Brown and Needham, 2000) and they also stated that subgrade, stiffness of the 393 mixture, layer thickness and load represent the major factors affecting the actual strain value.

394 Moisture damage resistance results

The moisture susceptibility or moisture damage resistance assessment of the cold asphalt concrete binder course mixtures in this research was performed through an indirect tensile stiffness ratio (SMR) test, according to the standard BS EN 12697-12 (European Committee for Standardization, 2008) to examine the impact of both BBCF and HCFA as replacements for the LF. The ITSM values for all types of mixtures are illustrated in Figure 15 for both dry and conditioned samples. It is clearly seen that the 400 stiffness modulus ratio (SMR) for both the BBCF and HCAF treated mixtures is more than 100%; the 401 moisture susceptibility of these mixtures increasing after conditioning the specimens. These results 402 were greater than those for the hot asphalt concrete binder course specimens and reached the 403 requirements for bituminous mixtures. When BBCF was used, the stiffness modulus of both the dry and 404 wet specimens increased significantly. Conversely, it was observed that the cold mixture with limestone 405 filler had a lower stiffness modulus in both dry and wet conditions.

406 The results of the SMR test can be interpreted as follows. When the traditional mineral filler is fully 407 substituted by BBCF, this filler has a stronger bond than the mineral filler. Furthermore, because BBCF 408 contains hydration products, when it is immersed in water this activates the hydration process, resulting in the mixture being less susceptible to moisture damage. In addition, the moisture susceptibility of the 409 BBCF mixture can further activate the hydration process after samples are conditioned at high 410 411 temperatures. Therefore, a BBCF treated mixture would perform well in terms of moisture susceptibility 412 and could satisfy specification requirements. It is evident that moisture damage is not an issue for the mixes containing BBCF and HCFA, while in the case of the cold limestone mixture, the reduction in 413 stiffness is due to trapped water and the weak early strength of this mixture. 414

415 Effect on the environment – leaching into water

416 The results for the heavy metals, namely nickel (Ni), copper (Cu), lead (Pb), chromium (Cr), zinc (Zn), 417 strontium (Sr), barium (Ba) and cadmium (Cd), can be observed in Table 7, where it can be seen that 418 the concentrations of all the elements presented were zero, except for Cr and Sr, which had a very slight increase as compared to the reference blank water. This means that adding HCFA and FC3R to CACB 419 does not have any negative impact on the environment. In addition, it is established that not did only 420 421 the application of HCFA and FC3R in CACB have technical benefits but this also reduced the 422 leachability of the waste and its harmful effects on the environment, meeting the requirements of the standard limits and standing within regulatory levels (Modarres and Nosoudy, 2015; Modarres et al., 423 2015). 424

426 Conclusions

A novel, fast-curing cold asphalt concrete for the binder course (CACB) mixture has been developed
which incorporates waste materials as the filler replacement. This mixture has benefits for the road
industry, in particular with regard to its contribution to sustainability, since this is an environmentally
friendly mixture. The main conclusions in this investigation are as follows:

- This research presents an environmentally friendly CACB mixture with substantial engineering
 properties for use as a binder course in road pavements. It can be stated that the BBCF treated
 mixture is a new technology with a mechanical performance comparable to traditional hot mix
 asphalt binder course mixtures.
- A new binary blended cement filler (BBCF) comprising 4.5% HCFA and 1.5% FC3R was
 created. A balanced oxide composition in the binary blend was responsible for the advanced
 pozzolanic reactivity displayed. In addition, using FC3R as a pozzolanic material rapidly
 enhanced the ITSM of the BBCF treated mixture.
- The BBCF treated mixture was found to have a higher stiffness modulus than the cold limestone
 mixture and the relevant traditional hot asphalt concrete binder course AC 20mm with 100/150
 pen. In addition, the BBCF treated mixture showed significant temperature susceptibility
 resistance.
- The BBCF treated mixture can be used in the asphalt binder layer to offer significant rutting resistance because of its high stiffness modulus as well as stability at high temperature.
- The BBCF mixture accomplished a significant improvement in fatigue life, extending fatigue
 life more than 19 times that of the control LF mixture.
- In terms of water sensitivity, the BBCF treated mixture has an SMR of more than 100%, which
 is higher than conventional hot mixtures and cold limestone mixture. Progressive curing with
 the BBCF treated mixture accounted for higher resistance to water damage.
- The problems relating to carbon emissions (during production) and mixture temperature
 maintenance (during transportation and laying) in the case of hot mix asphalt will be mitigated
 by using this fast-curing CACB.

An environmental investigation of the influence of using HCFA and FC3R in CACB mixtures
 reveals that there are no negative impacts on the environment as the heavy metals will not leach
 into the environment, thus there will be no pollution as a result of using this mixture. Analysis
 of the TCLP demonstrates that these two wastes are non-hazardous and can therefore be
 categorised as general industrial waste. This will encourage pavement and asphalt agencies to
 promote their use.

459

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584 Figure captions

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Figure 3	Powder XRD pattern of FCER
Figure 4	ITSM Apparatus machine
Figure 5	Roller compactor machine
Figure 6	Wheel-tracking equipment used by LCMT lab
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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
Table 6	Wheel-tracking test conditions
Table 7	Concentrations of heavy metals in leachate water, (mg/L)

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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

Boiling point, (°C)	Cationic Black to dark brown liquid 100/150 pen 60 Positive 100
Base bitumen Bitumen content, (%) Particle surface electric charge Boiling point, (°C)	100/150 pen 60 Positive
Bitumen content, (%) Particle surface electric charge Boiling point, (°C)	60 Positive
Particle surface electric charge Boiling point, (°C)	Positive
Particle surface electric charge Boiling point, (°C) Relative density at 15°C, (g/ml)	
	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
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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 O
	HCFA	67.057	24.762	2.430	2.845	0.000	0.340	0.266	0.473	1.826
	FC3R	0.047	35.452	44.167	0.684	0.368	0	0.049	0	0
	LF	76.36	16.703	0	0.981	0	0.096	0.348	0.185	2.258
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Table 5.	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	5
No. of test plus	5
Test temperature, (°C)	20 ± 0.5
Specimen thickness, (mm)	63 ± 3
Compaction	Marshall 50 blows/face
Specimen temperature conditioning	4 h before testing

7	13
7	14

733	Table 6. 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
	Contact pressure, (MPa)	0.7 ± 0.05
	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
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	Ba	Cu	Cd	Pb	Zn	Cr	Ni	Sr
Reference water quality	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Leachate water (HCFA mix)	0.0	0.0	0.0	0.0	0.0	0.009	0.0	0.367
Leachate water (BBCF mix)	0.0	0.0	0.0	0.0	0.0	0.008	0.0	0.257
TCLP regulatory level	100	25	1	5	25	5	25	-

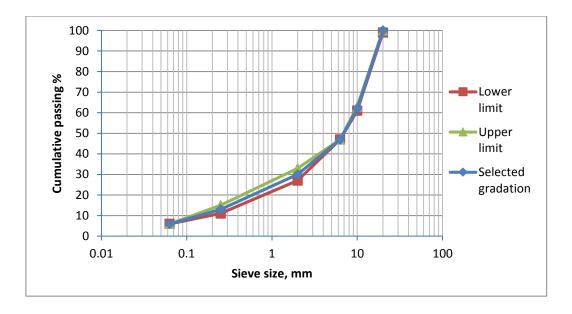


Figure 1. Gradation of aggregates



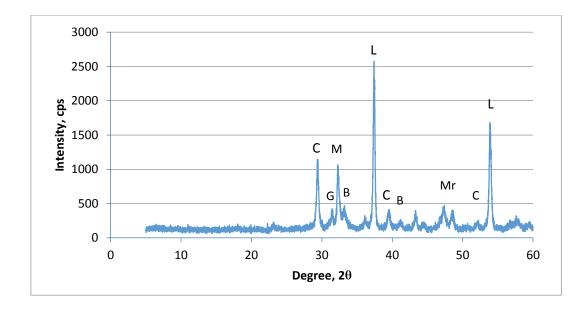


Figure 2. Powder XRD pattern of HCFA

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

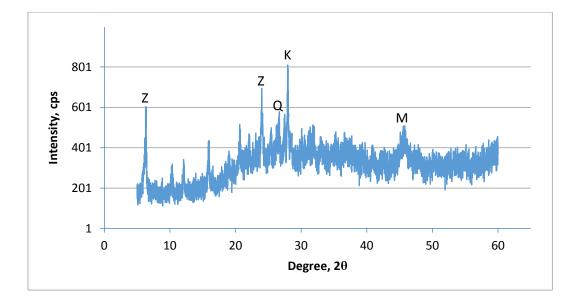


Figure 3. Powder XRD pattern of FCER

(K- kyanite (Al₂O₅Si), Q – quartz (SiO₂), M- mullite (Al₆Si₂O₁₃), Z- dehydrated Ca-A zeolite (Al₉Ca₄₈O₃₈₄Si₉₆))

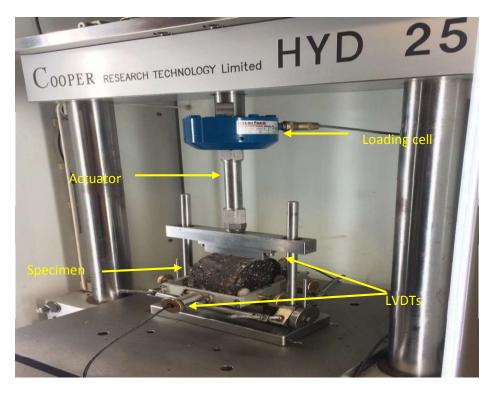


Figure 4. ITSM Apparatus machine



Figure 5. Roller compactor machine

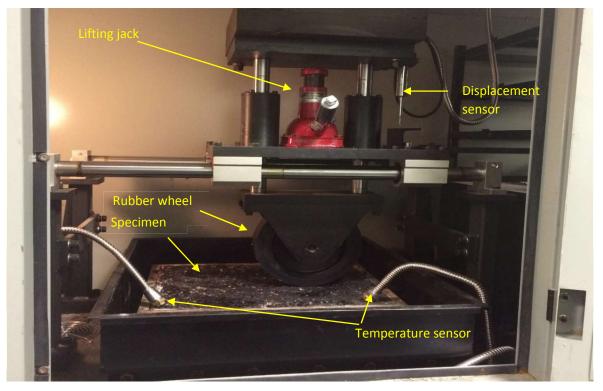


Figure 6. Wheel-tracking equipment used by LCMT lab

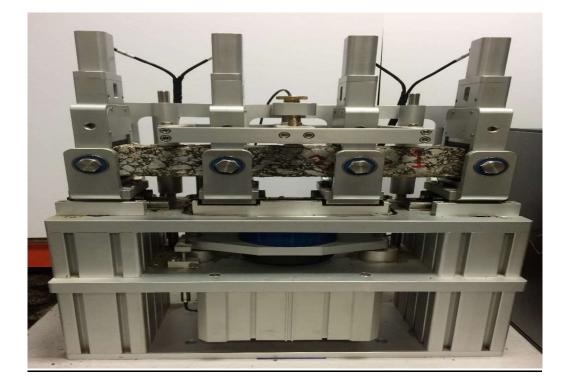


Figure 7. Test set up for the four point bending test



Figure 8. Atomic adsorption spectrophotometer



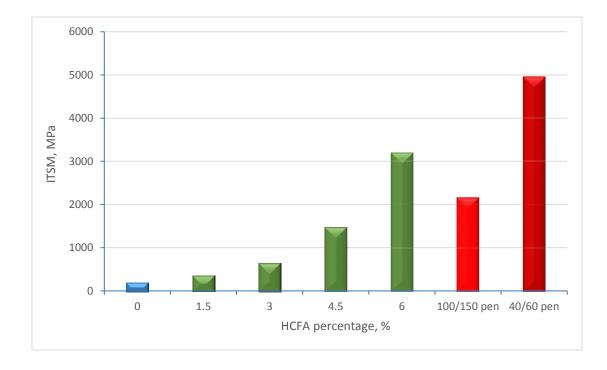


Figure 9. ITSM results for HCFA replacement after 3 days

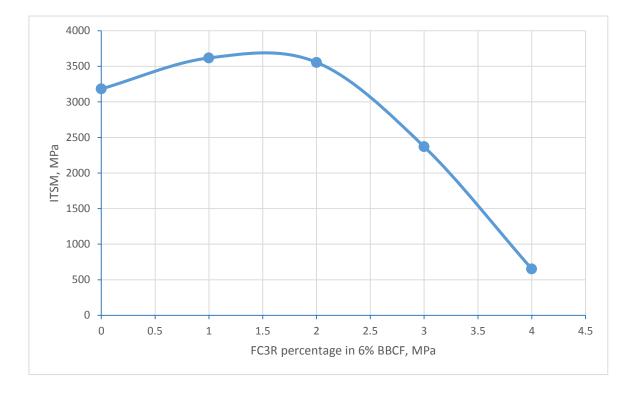


Figure 10. Influence of substitution of HCFH with FC3R on ITSM after 3 days



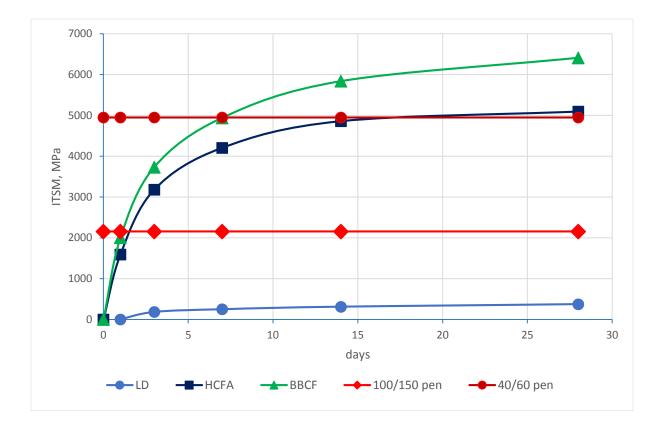


Figure 11. Effect of curing time on ITSM of BBCF mixture

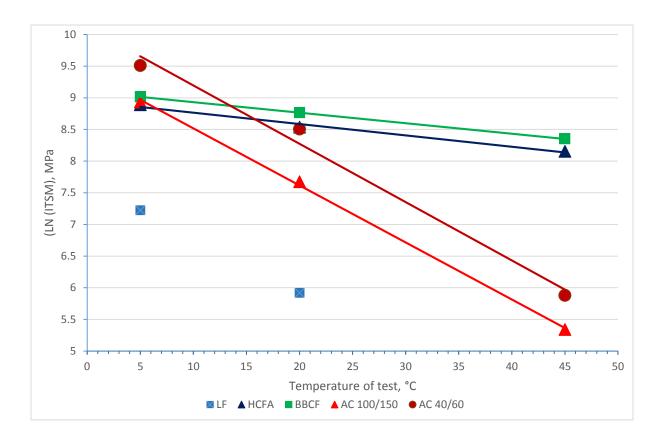


Figure 12. Temperature susceptibility results

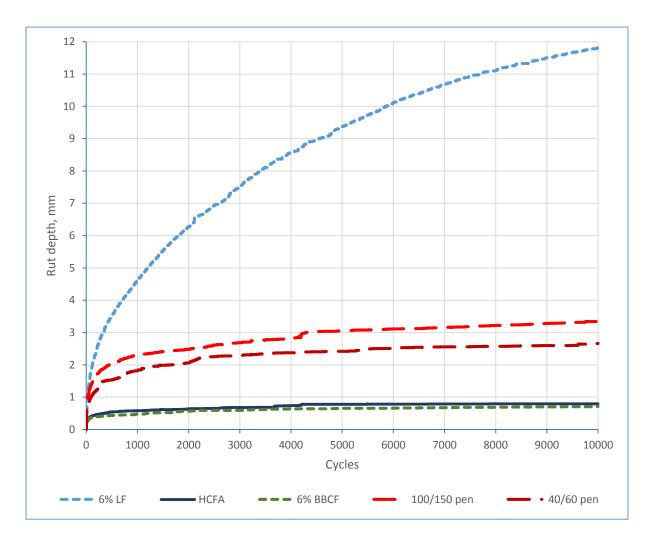


Figure 13. Wheel-tracking test results

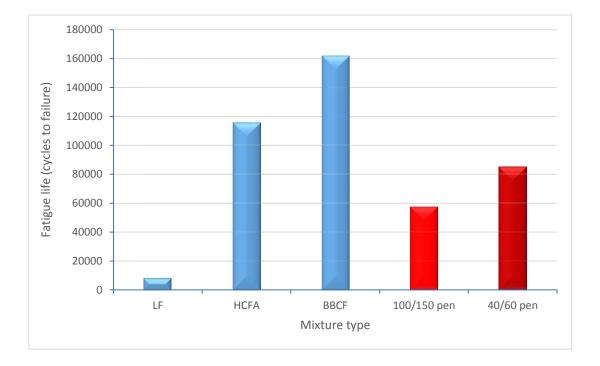


Figure 14. Four-point bending beam fatigue test results



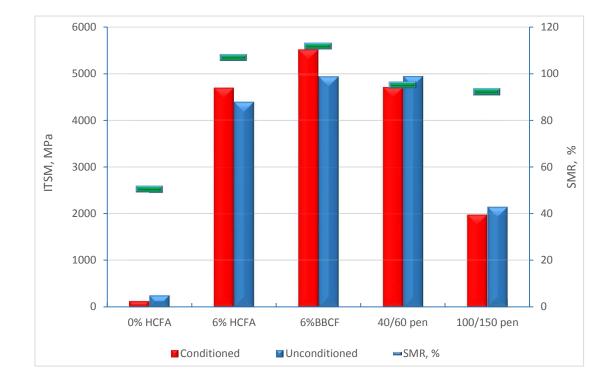


Figure 15. ITSM test results in saturated and dry conditions