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1 Investigation into the bond strength of bitumen-fibre mastic

2 Abstract

3 The loss of bond strength in road pavement surfacing due to high traffic loads or 4 moisture is a recurring problem, creating distresses such as ravelling, fatigue and 5 rutting. It is, therefore, important to find a way to prevent or at least delay the loss of 6 bond strength in asphalt mixtures. Such an improvement would lead to longer service 7 life and a more comfortable drive for road users. This study describes how the pneumatic 8 adhesion tensile testing instrument (PATTI) was used to examine the mechanism by 9 which fibres influence the pull-off tensile strength of asphalt mastic. This study assesses 10 the potential for chemical modification of the binder due to the presence of fibres, by 11 means of work of cohesion and work of adhesion calculations, based on surface energy 12 parameters and a binder drainage test. The study also evaluates the influence of 13 different filler-bitumen ratios and fibre percentages on pull-off tensile strength. The test 14 results indicate that the fibres enhance the pull-off tensile strength of the mastic, in 15 addition to changing the failure mode from cohesive to hybrid, implying an improvement

16 in the cohesive strength of the mastic.

17 1. Introduction

18 The bond strength is considered an important property affecting asphalt mixture 19 performance. In an asphalt mixture, the aggregates are much stiffer and stronger than 20 other components such as bituminous binder or mastic. Therefore, the possibility of 21 failure is expected to be very small through the aggregates themselves, while there is a 22 high possibility of failure in the material that bonds adjacent particles [1]. The loss of 23 strength of cohesive bonds within the bitumen itself is termed cohesive failure, and 24 adhesive failure is the breaking of the adhesive bonds between aggregate and bitumen 25 or mastic materials, mainly due to the effect of water or low temperatures [2]. Under the 26 effect of traffic loading forces, the aggregate particles tend to separate from each other. 27 As these forces increase, failure occurs at the interfaces between the aggregate and 28 mastic and/or inside the mastic film [3]. The loss of bond strength then compromises 29 pavement integrity, and this could lead to different pavement failure types such as 30 permanent deformation, ravelling and fatigue [4]. It is, therefore, important to 31 investigate this phenomenon and find a way to prevent or at least reduce such 32 deterioration in the bond strength.

- **33** The pull-off tensile strength test is commonly used to investigate the bond strength and
- **34** failure type of a bitumen-aggregate mix. Past studies found that dry samples showed a
- **35** cohesive failure, while moisture-conditioned samples showed a hybrid or adhesive failure
- **36** [1, 5, 6]. The physicochemical properties of bitumen and aggregate surfaces, their
- **37** surface energies, can be used to estimate the adhesive bond strength between the two
- **38** [7]. Also, adding modification (polymer, acid, anti-stripping additive) to bitumen will
- **39** influence the physicochemical properties and bond strength of bitumen [8].
- **40** Fibres have the potential to strengthen the bitumen phase, however studies have found
- **41** that different types and amounts of fibre strengthen the modified asphalt in different
- **42** ways [9, 10]. Past studies [11-13] have suggested that some fibres can absorb the light
- **43** fraction of bitumen, in addition to the fact that adding fibres such as those of polyester,
- **44** polyacrylonitrile, lignin and asbestos change the optimum bitumen content of an asphalt
- **45** mixture. It is important to note that the mechanism of possible chemical modification of
- **46** binder in contact with fibres remains in large part unknown. The binder drainage test can
- **47** provide results that may help in understanding such modification.
- **48** This paper explores the influence of different types and percentages of fibre on the pull-
- **49** off tensile strength of bituminous mastic. The effect of different filler-bitumen ratios on
- **50** the pull-off tensile strength of fibre reinforced mastic is also investigated. To gain a
- **51** better understanding of the chemical effect of fibre on the binding properties of bitumen,
- **52** the work of cohesion and work of adhesion of drained binder, before and after mixing
- **53** with fibres are calculated from surface energy components. The relationship between the
- **54** work of cohesion and the pull-off tensile strength results of fibre reinforced mastic are
- **55** investigated.

57 2. Materials and experimental methods

58 2.1 Materials

59 A combination of one penetration grade bitumen (40/60) [14], limestone filler with 60 maximum size less than 0.063 mm and four fibre types (including two types of glass, 61 cellulose and steel fibres recovered from tyres) were used. Table 1 lists the basic 62 properties of these fibres. The glass fibres had two different lengths, 13 and 6 mm, 63 classified as glass-I and glass-s respectively. Micrographs of these four fibre types and 64 the fibre reinforced mastic are shown in Figures 1 to 3 using a Cryo-scanning electron 65 microscope (Quanta 200 3D (FEI)). Samples were coated with platinum (Pt) at -160°C 66 for 60 seconds at current of 10 mA. Accelerating voltage used was 10 kV for glass and 67 cellulose and 15 kV for steel fibre. These show the needle-like structure of glass fibres; 68 the spongy and flexible nature of cellulose; and the thick steel fibres coated with mastic.

69

70 2.2 Binder drainage test basket method

71 In this work, the basket method [15] was used to measure the binder drainage of fibre-72 reinforced mastic; 2.0% by volume of fibres in 80 g of binder. The resulting composite 73 was placed into a steel mesh basket with sieve size 250 microns, which was then placed 74 on a pre-wrapped tray in the oven at 160°C for 3 hours. Binder flowed and drained 75 through the mesh into the pre-wrapped tray. After 3 hours the steel mesh basket and 76 pre-wrapped tray were removed from the oven and the mesh basket separated from the 77 pre-wrapped tray. Once cooled the weight of the tray and drained binder was measured 78 and the weight of drained material (D) calculated in percent.

79 2.3 Determination of asphaltene content

80 Asphaltene content in the drained binders was measured according to BS 2000 [16]. In

- 81 this method the drained binder was mixed with heptane and heated under reflux,
- **82** separating the binder into three components (asphaltenes, waxy substances and
- **83** inorganic material). The remaining material was extracted by chromatographic
- **84** separation.

85 2.4 Complex modulus test

86 A Bohlin Gemini 200 (DSR) was used in this study for measuring the rheological

87 properties of base and drained binder. Eleven testing frequencies ranging from 0.1 to

- **88** 10.0 (Hz) were used in the DSR tests with nine testing temperatures between 30 and
- **89** 70°C (at increments of 5°C).

90 2.5 Pneumatic Adhesion Tensile Testing Instrument (PATTI) test

91 The Pneumatic Adhesion Tensile Testing Instrument (PATTI) is used to measure the 92 cohesive and adhesive properties of a bitumen-aggregate system by measuring pull-off 93 strength. The PATTI consists of a portable pneumatic adhesion tester, piston and 94 reaction plate, air hose, camera and steel pull-out stub as shown in Figure 4. Aggregate 95 substrates and pull-stubs have to be cleaned and dried at room temperature for at least 96 24 hours. Then, in order to remove any dust, the aggregate substrates and pull-stubs 97 were wiped carefully using a damp paper towel. After that, all aggregate substrates and 98 pull-stubs were placed in an oven at 80°C for one hour. The fibre reinforced mastic was 99 placed in an oven at 160°C for 30 minutes, and by this time the mastic was fluid enough 100 to coat the aggregate substrate. Then fibre reinforced mastic was poured onto the 101 aggregate substrate, and a pull-stub was immediately pressed into it to achieve good 102 mastic-aggregate adhesion [17]. Washers and three raised edges on the pull-stub are 103 used to control the film thickness.

104 Samples were left for 24 hours at room temperature before testing in order to allow 105 enough time for the aggregate and mastic to adhere and for the sample to reach the test 106 temperature. After the mastic and substrate had cooled down the excess mastic at the 107 edge of the pull-stub was carefully removed using a heated pallet knife. The test then 108 consists of applying an upward pressure to the asphalt mastic by movement of the pull-109 out stub. Air pressure rate was fixed during the test in order to achieve repeatable 110 results. The pull-off tensile strength of mastic indicates when the applied pressure 111 exceeds the cohesive strength of the mastic or the adhesive bond strength between 112 mastic and aggregate.

113 2.6 Surface energy measurement

114 Thermodynamic (adsorption) theory is widely accepted as an approach to explain the 115 interfacial adhesion of a liquid-solid contact. This theory is based on the principle that an 116 adhesive (the liquid) will adhere to a solid depending on the physical forces established 117 at the interface, so long as contact is maintained. The interfacial forces are van der 118 Waals and Lewis acid-base interactions. These forces are generally related to 119 fundamental thermodynamic quantities, in particular the surface free energies of the 120 materials involved [18]. The contact angles of binders and limestone aggregate were 121 measured by using three selected probe liquids, distilled water, glycerol and

122 diiodomethane [7].

123 3. Surface energy

124 According to past studies the cohesive and adhesive strengths of bitumen-aggregate

- **125** systems have been successfully determined from the surface free energy (γ) [19, 20].
- **126** Previous research investigated the technical criteria of test methods, such as accuracy,
- **127** precision and the ability to provide all three surface energy parameters and
- **128** recommended the Whilhelmy plate method (dynamic contact angle) for routine use [19].
- **129** Another study focused on evaluation of the resistance of asphalt mixtures to moisture
- **130** damage, through understanding the mechanisms that influence the surface energy of
- **131** aggregates and binders in addition to fracture behaviour of asphalt mixture. They found
- **132** that the ratio of the adhesive bond energy under dry conditions to the adhesive bond
- **133** energy under wet conditions is related to the asphalt mixture resistance to moisture
- **134** damage [20]. The surface free energy is a measure of the amount of energy needed to
- **135** form a unit area of new surface at the interface of the material [7].
- **136** The surface free energy (γ) of a material comprises two components, a dispersive or
- **137** Lifshitz-van der Waals component (γ^{LW}) of electrodynamic origin, and a polar
- **138** component (γ^{AB}) caused by Lewis acid-base interactions [21] as shown in the following equation:

$$140 \quad \gamma^{total} = \gamma^{LW} + \gamma^{AB} \tag{1}$$

- **141** The acid-base components can be subdivided into a Lewis acid parameter of surface free
- **142** energy (γ^{+}) and a Lewis base parameter of surface free energy (γ^{-}) as shown in the following equation:

$$144 \quad \gamma^{AB} = 2(\sqrt{\gamma^{+}\gamma^{-}})$$
(2)

By combining equations (1) and (2) the surface free energy (mJ/m²) or (erg/cm²) of a
 material can be defined as:

$$147 \quad \gamma^{total} = \gamma^{LW} + 2(\sqrt{\gamma^+ \gamma^-}) \tag{3}$$

148 3.1 Work of cohesion

149 The work of cohesion (W_c) can be defined as the energy required to separate the liquid
150 or solid from itself and this depends on the attraction between molecules. The following
151 equation describes the work of cohesion [8]:

(4)

152 $W_c=2\gamma^{total}$

153 3.2 Work of adhesion

154 The work of adhesion (*W_a*) can be defined as the energy required to create new surfaces
155 between two materials (solid-liquid); therefore, work of adhesion represents the amount
156 of intermolecular interaction between two materials [22].

157 The work of adhesion is a dependent property of a solid-liquid pair. Accordingly the
158 interfacial surface free energy of a material (binder or aggregate) is the combination of
159 these non-polar (Lifshitz-van der Waals) and polar (Lewis acid/base) forces as shown in
160 the following equation:

$$161 \qquad \gamma_{BA} = \gamma_{BA}^{LW} + \gamma_{BA}^{AB} \tag{5}$$

162 Where B and A represent bitumen and aggregate, respectively. The adhesive Lifshitz-van

- **163** der Waals and Lewis acid/base bond strength can be determined by the following
- **164** equations, respectively [23].

$$165 \qquad W^{LW} = \gamma_B^{LW} + \gamma_A^{LW} - \gamma_{BA}^{LW} \tag{6}$$

$$\mathbf{166} \qquad \mathbf{W}^{AB} = \boldsymbol{\gamma}^{AB}_{B} + \boldsymbol{\gamma}^{AB}_{A} - \boldsymbol{\gamma}^{AB}_{BA} \tag{7}$$

167 The Lifshitz-van der Waals solid-liquid surface energy is described by the following168 equations [23]:

$$169 \qquad \gamma_{BA}^{LW} = (\sqrt{\gamma_B^{LW}} - \sqrt{\gamma_A^{LW}})^2 \tag{8}$$

$$\mathbf{170} \qquad \boldsymbol{\gamma}_{BA}^{AB} = 2(\sqrt{\boldsymbol{\gamma}_B^+} - \sqrt{\boldsymbol{\gamma}_A^+}) \left(\sqrt{\boldsymbol{\gamma}_B^-} - \sqrt{\boldsymbol{\gamma}_A^-}\right) \tag{9}$$

171 By substituting these two equations (8) and (9) into equations (6) and (7) the following172 equation is obtained:

$$173 \qquad W_a = 2\left(\sqrt{\gamma_B^{LW} \gamma_A^{LW}} + \sqrt{\gamma_B^+ \gamma_A^-} + \sqrt{\gamma_B^- \gamma_A^+}\right) \tag{10}$$

- Where W^{LW} is the Lifshitz-van der Waals work of adhesion and W^{AB} is the Lewis acid/base
 work of cohesion.
- 176 In this paper the work of cohesion and work of adhesion, computed in this way, will be177 used to interpret the pull-off test data.

178

179 4. Results and analysis

180 4.1 Binder drainage test results

- 181 The results of the binder drainage tests are the average of three values as shown in182 Table 2. The results have low coefficient of variation, although higher for cellulose fibres
- **183** due to having a similar level of variability and lower average drained binder percentage.
- **184** The percentage of drained material reflects bitumen stability, which may be a function of
- **185** the mechanical interaction of the fibres in the binder and absorption/adsorption of
- **186** certain bitumen fractions by the fibres.
- 187 Cellulose fibre showed by far the highest stabilisation and absorption/adsorption effect
 188 compared to the other fibre types, as expected due to the surface texture and large
 189 surface area of cellulose fibre (see Table 1), giving high absorption and retention of
 190 liquid media. Other fibre types (glass and steel) held lower amounts of bitumen. These
 191 results may reflect the amount of bitumen adhering to the fibre surfaces. Samples of
 192 base binder and drained binders were taken for analysis of asphaltene content and
- **193** surface energy components.

194 4.2 Asphaltene test results

195 Asphaltenes are the bitumen fraction that is insoluble in low molecular weight paraffin, 196 such as propane, n-butane, n-pentane, n-heptane and n-hexane [24]. They are 197 relatively high in heteroatoms such as nitrogen, oxygen, carbon and hydrogen with 198 sulphur. Asphaltenes are the largest fraction of bitumen, responsible for the increase in 199 thickening, strength and stiffness of the bitumen and non-Newtonian rheological 200 behaviour [24]. This paper measured the influence of different fibre types on the 201 asphaltene content of base binder to explore the effect on bitumen chemistry of adding 202 fibres. A previous study indicated that high asphaltene content might have led to 203 increased cohesive and adhesive strength of base binder [25].

204 Two tests were performed for each fibre type, and the average asphaltene content is 205 reported in Table 3. The drained binders showed higher asphaltene content compared to 206 the base binder. As noted in the introduction, previous studies showed that fibres can 207 adsorb /absorb the light fraction of bitumen [11-13], and it is important to note that the 208 cellulose fibres retained over 50% of the bitumen in the drainage test while the other 209 fibres retained about 25%, compared to 3% for the sample without fibres. Preferential 210 adhesion of the lighter binder fraction would lead to higher asphaltene content in the 211 drained binder. Also, steel fibre, as a thermal conductor, may influence the bitumen 212 heating process and lead to more ageing of the bitumen during the draining process and 213 hence, lead to higher asphaltene content. The results suggest that the presence of 214 fibres leads to higher asphaltene content, however, due to the variability in the 215 asphaltene test results, it is hard to draw conclusions about any differences. For this 216 reason the rheological properties of the drained binders were evaluated by creating 217 complex modulus master curves.

218 4.3 Complex modulus master curve of base and drained binder

219 Figure 5 shows the complex modulus master curves for base binder, drained base binder 220 and drained binder after mixing with fibres. A considerable difference in the complex 221 modulus values of drained binder after mixing with fibres, compared to both base and 222 drained base binder can be seen. This increase in complex modulus could be due to the 223 fibre affecting the chemical composition of the binder by adsorption/absorption of 224 different fractions, mainly lighter fractions. A previous study, using chromatography, 225 indicated that the fibre could influence the absorption of different fractions, particularly 226 lighter fractions [26]. This result reflects the asphaltene content results, as the 227 asphaltene content increased for drained binder after mixing with fibres, as shown in 228 Table 3.

- It is important to note that the complex modulus values of drained binder after mixing
- with glass and steel fibre were almost the same. Cellulose fibre showed higher complex
- modulus compared to other fibre types. This increase in complex modulus may be
- because cellulose fibre had lower drained binder percentage (Table 2), indicating
- retention of more of the bitumen.

4.4 Pneumatic adhesion tensile testing instrument (PATTI)

The PATTI test was used to measure the influence of fibres on mastic bond strength.

- Three filler-bitumen ratios (0.8, 1.0 and 1.2) and three fibre contents (0.5, 1.0 and
- 2.0% by bitumen volume) were used in this investigation. Failure strength results are
- shown in Figures 6 to 8 and the mode of failure in Table 4, as illustrated in Figures 9 and10.

4.4.1 Influence of fibre content and type

- Three tests were performed for each mastic type, and the average tensile strength was
- reported, as shown in Figures 6 to 8, where error bars represent plus and minus one
- standard error (standard deviation divided by the square root of number of samples).
- **244** The tensile strengths of different fibre types and filler-bitumen ratios were reasonably
- repeatable, indicating that the application of a constant rate of air pressure was
- successful for these tests. The specimens exhibited a linear response to the increase in
- **247** pressure until the pressure overcame the cohesive strength of the mastic or the
- adhesive strength of the mastic-aggregate interface and then rapidly decreased to zero.
- It can be seen that on average, all fibre types increased the pull-off tensile strength
- compared to the base mastic. Table 5 gives the average results along with t-test results,
- **251** comparing fibre mastics to the base binder, for the three filler-bitumen ratios. Most fibre
- **252** percentages and types show statistically significant increases in the pull-off strength.
- The glass-I fibre showed the highest increase in bond strength among all fibre types.
- This increase may be due to two reasons. Firstly, glass fibre contains silicon dioxide
- **255** (SiO₂) and aluminium oxide (Al₂O₃). This chemical combination can form strong chemical
- bonds with sulfoxides and carboxylic acids in bitumen [27, 28]. Secondly, glass fibre
- dimensions (length and width) may help the fibres to cross over each other to form three
- dimensional networks. These networks then provide support to the composite structure
- by holding the components together and spreading stresses [29].
- 260 Base mastics with different filler-bitumen ratios showed cohesive failure (failure within261 the mastic). The failure type changed from cohesive to hybrid (partly adhesive failure

- **262** between aggregate and mastic) with an increase in the pull-off strength when 1.0 and
- 263 2.0% cellulose and glass-s fibres were used as a modification for 1.0 and 1.2 filler-
- 264 bitumen ratios. This implies an improvement in the cohesive strength of the mastic with
- **265** the increase in fibre content, as shown in Figures 7 and 8. This behaviour may be
- **266** explained by the higher fibre content both mechanically stabilizing the material and/or
- absorbing/adsorbing the bituminous binder (see Table 2) [30]. The latter is likely with
- **268** cellulose fibre since the surface texture of cellulose fibres comprises many interweaved
- **269** branches with non-uniform sizes and rough surfaces, which will increase the specific
- **270** surface area and hence the absorption/adsorption capability (see Table 1), as shown in
- **271** SEM images (see Figure 2). These features of cellulose fibre might explain the hybrid
- 272 failure at 0.8 filler-bitumen ratio for mastic modified by cellulose fibre, whereas other
- **273** fibre types exhibited a cohesive failure, as shown in Table 4 and Figure 9 a.
- 274 On the other hand, the longer steel and glass-I fibre samples showed a hybrid failure for
- all fibre percentages at 1.0 and 1.2 filler-bitumen ratios. This could be due to the
- **276** dimensions of these fibres, leading to a more effective network and therefore enhanced
- **277** reinforcement effects.

278 4.4.2 Influence of filler-bitumen ratio

- 279 The results (Figures 6, 7 and 8) showed that the pull-off tensile strength increased with280 the increase in the filler-bitumen ratio. A possible explanation for this might be that
- **281** adding more filler enhanced the interaction between the bitumen and the filler particles
- **282** [31]. Bituminous binders are non-polar, while molecules of limestone are more polar.
- **283** This may help to satisfy the energy demand of the aggregate surface by improving the
- adhesion between the aggregate and the mastic [28, 32].
- 285 There is a decrease in the pull-off tensile strength of the 1.2 filler-bitumen ratios at 286 2.0% fibre content, particularly for cellulose, glass-I and steel fibres. This may indicate 287 that adding more fibre with a high absorption/adsorption and/or large surface area led to 288 an increase in the percentage of voids in the matrix [33, 34]. The stress applied to the 289 specimen may have been concentrated around these voids, and the fracture may have 290 begun at these points. Moreover, as the percentage of filler and fibre increased, the 291 composite became stiffer and was, therefore, more likely to show brittle behaviour. This 292 suggests that when adding more fibre and filler, there should be enough binder in the 293 mix to reduce the voids in the mastic.
- 294 It is important to note that the modulus and Poisson's ratio of steel and glass fibres are295 very large compared to the matrix, and therefore a concentration of stress will occur at
- **296** the interface. Also, steel fibre has large thickness and according to previous studies, the

stress concentration area depends on the fibre thickness and this area will increase asfibre thickness increases [35] (see Figure 11).

299 4.5 Surface energy

300 The dynamic contact angle (DCA) measurement of bitumen was used in this study. The 301 contact angles were measured for each drained binder for each probe liquid, and the 302 contact angle was taken as the average of four or five replicates and the variation 303 between results was low, as shown in Table 6. The results for the surface energy 304 components of the drained binders are shown in Table 7 in which γ_s^{AB} represents the 305 polar surface energy component of the solid and γ_s^{LW} represents the non-polar (Lifshitz-306 van der Waals) surface energy component of the solid. The results for the surface energy 307 components of the drained binders showed more variation in the γ_s^{AB} values than the γ_s^{LW} 308 values, ranging from 0.19 to 1.78 mJ/m^2 , as shown in Table 7. The results showed that 309 the bituminous binder molecular constituent forces are mainly Lifshitz-van der Waals 310 (non-polar) in character [36].

- **311** The surface energy components of limestone aggregate were determined by using a
- **312** dynamic vapour sorption (DVS) approach, and the results are shown in Table 8. This
- **313** method is usually used for high surface energy materials such as aggregate, and the
- **314** advantage of this method is that it takes into consideration the irregular shape and
- **315** surface texture of the aggregate [7]. The surface energy of limestone aggregate was
- **316** found to be 104.19 mJ/m².

317 4.6 Work of cohesion and adhesion

- 318 The results for work of cohesion (2 γ ^{total}) of the base drained binder and drained binder 319 after mixing with fibres are shown in Table 9. Table 4 shows the mode of failure in the 320 PATTI tests and reveals that the 0.8 filler-bitumen ratio mastics consistently failed by 321 fully cohesive failure. It is, therefore, interesting to compare the pull-off strength for 322 these mastics to the work of cohesion of the binders. The pull-off strengths are included 323 in Table 9 and Figure 12 shows a comparison between work of cohesion and pull-off 324 tensile strength. The work of cohesion results cover just a small range of values but it 325 was found that higher pull-off tensile strength corresponds to higher work of cohesion.
- **326** These results are consistent with data obtained in another recent study [8].
- **327** One consistent finding in this study is that all drained binders after mixing with fibres,
- **328** showed higher values for work of cohesion compared to that of the drained base binder.
- **329** Also, it is noted that all unmodified mastics showed a cohesive failure, while highly
- **330** modified mastics showed a hybrid failure (partially between aggregate and bitumen)

- **331** together with an increase in pull-off strength. These observations indicate that adding
- **332** fibre increases the cohesive strength of the mastic leading to hybrid failure.

333 The work of adhesion results are shown in Table 10. It is interesting to note that drained 334 binder after mixing with cellulose fibre had the lowest value of work of adhesion. This is 335 due to the low value of the Lewis base parameter of surface free energy of the drained 336 binder after mixing with cellulose fibre. This finding may explain the hybrid failure for 337 mastic modified with cellulose fibre at 0.8 filler-bitumen ratio and 1.0 and 2.0% fibre, 338 while other modified mastics showed cohesive failure at this filler-bitumen ratio (see 339 Table 4). These surface energy results support the ability of the (PATTI) test to detect 340 differences in bond strength. However, none of the pull-off tensile strength samples 341 showed adhesive failure because pull-off tensile strength tests were done at room 342 temperature (20°C) and in dry conditions. These conditions eliminate the possibility of 343 adhesive failure. Therefore, this study was not able to find a relationship between the

344 pull-off tensile strength and the work of adhesion.

345 4.7 Conclusions

346 This paper has examined the influence of fibres on bitumen mastic, based on pull-off
347 tensile strength, asphaltene content, surface energy measurement and scanning
348 electronic microscopy. The following conclusions are offered:

- 349 o Fibre asphalt mastics were observed using a scanning electron microscope (SEM).
 350 The formation of three-dimensional networks was observed for glass and steel
 351 fibre reinforced mastic.
- 352 o The drainage test results showed that cellulose fibre can retain more than 55% of353 binder. Other fibre types (glass and steel) can retain up to 30% of binder.
- 354 o All fibre types increased the asphaltene content and complex modulus of the
 355 drained binder compared to the base binder. However, the differences between
 356 asphaltene contents for different fibre types were too small to discriminate
 357 between them with confidence.
- 358 o Adding fibre led to an increased pull-off tensile strength of the mastics, at the
 359 same time changing the failure mode from cohesive to hybrid, implying an
 360 improvement in cohesive strength of the mastic.
- 361 o There was a general reduction in the pull-off tensile strength of the 1.2 filler362 bitumen ratio at high fibre content (2.0%).
- 363 o The surface energy of drained binder after mixing with fibres was estimated by364 dynamic contact angle (DCA) measurements. The contact angle results for each

- drained binder varied within a narrow range, making this a useful test for
- characterising these materials.
- 367 o In general, there is a good agreement between work of the cohesion and pull-off
 368 tensile strength for 0.8 filler bitumen ratio and higher work of cohesion results in
 369 higher pull-off tensile strength where the mode of failure is cohesive.

370 List of tables

Table 1 Basic properties of fibres

Fibre type	Specific density (g/cm ³)	Length (µm)	Width (µm)	Modulus of elasticity at 23 °C (GPa)	Specific surface area (m²/g)
Cellulose	1.50	20 to 2,500	25	-	138.53
Glass-s	2.58	6,000	12 to 20	80.3	8.85
Glass-l	2.58	13,000	12 to 20	80.3	14.36
Steel	7.85	4,000 to 12,000	180 to 300	210*	-

372 *Standard steel fibre modulus

Table 2 Results of binder drainage test (basket method)

Fibre type	Percent of drained material (average of three results) (%)	Coefficient of variation (%)	Standard error
Base binder	97.0	0.7	0.41
Cellulose	43.4	3.0	0.74
Glass-s	74.9	2.2	0.96
Glass-l	71.1	2.4	0.97
Steel	72.1	1.2	0.49

Table 3 Asphaltene content test results

Binder type	Asphaltene content (%)	Coefficient of variation (%)	Standard error
Base binder	12.7	1.1	0.10
Drained base binder	14.7	0.5	0.05
Drained cellulose	15.9	3.3	0.26
Drained glass-s	14.9	4.0	0.57
Drained glass-l	15.7	5.1	0.46
Drained steel	15.5	5.0	0.55

379	Table 4	Failure	mode i	n fibre	modified	asphalt	mastic

Mastic type	f/b*	Failure type				
		0.0% vol.	0.5% vol.	1.0% vol.	2.0% vol.	
Base mastic	0.8	Cohesion	-	-	-	
	1.0	Cohesion	-	-	-	
	1.2	Cohesion	-	-	-	
Cellulose fibre	0.8	-	Cohesion	Hybrid	Hybrid	
mastic	1.0	-	Cohesion	Hybrid	Hybrid	
	1.2	-	Cohesion	Hybrid	Hybrid	
Glass-s fibre	0.8	-	Cohesion	Cohesion	Cohesion	
mastic	1.0	-	Cohesion	Hybrid	Hybrid	
	1.2	-	Cohesion	Hybrid	Hybrid	
Glass-I fibre	0.8	-	Cohesion	Cohesion	Cohesion	
mastic	1.0	-	Hybrid	Hybrid	Hybrid	
	1.2	-	Hybrid	Hybrid	Hybrid	
Steel fibre mastic	0.8	-	Cohesion	Cohesion	Cohesion	
	1.0	-	Hybrid	Hybrid	Hybrid	
	1.2	-	Hybrid	Hybrid	Cohesion	

*f/b: filler-bitumen ratio

Table 5 Pull-off tensile strength t-test results

f/b ratio*	Fibre	Mean (MPa)	t-stat	p-value	Significant**
0.8	Base mortar	1.19	-	-	-
0.0	0 5% Glass-s	1 61	4 828	0 0202	Yes
	0.5% Glass-l	1.71	5.677	0.0054	Yes
	0.5% Cellulose	1.56	4.121	0.0271	Yes
	0.5% Steel	1.44	2.731	0.0359	Yes
	1% Glass-s	1.76	4.139	0.0072	Yes
	1% Glass-l	1.78	6.394	0.0038	Yes
	1% Cellulose	1.64	4.703	0.0091	Yes
	1% Steel	1.76	4.117	0.0073	Yes
	2% Glass-s	1.85	6.403	0.0038	Yes
	2% Glass-l	1.95	8.388	0.0069	Yes
	2% Cellulose	1.84	6.348	0.0039	Yes
	2% Steel	1.81	6.275	0.0041	Yes
1.0	Base mortar	1.25	-	-	-
	0.5% Glass-s	1.61	3.775	0.0317	Yes
	0.5% Glass-l	1.78	3.350	0.0393	Yes
	0.5% Cellulose	1.69	4.525	0.0101	Yes
	0.5% Steel	1.55	3.641	0.0339	Yes
	1% Glass-s	1.78	3.798	0.0160	Yes
	1% Glass-l	2.06	5.222	0.0068	Yes
	1% Cellulose	1.76	6.398	0.0038	Yes
	1% Steel	1.88	5.446	0.0060	Yes
	2% Glass-s	2.10	8.518	0.0017	Yes
	2% Glass-l	2.16	8.794	0.0015	Yes
	2% Cellulose	2.09	11.947	0.0034	Yes
	2% Steel	1904	10.204	0.0047	Yes
1.2	Base mortar	1.72	-	-	-
	0.5% Glass-s	1.93	2.174	0.0808	No
	0.5% Glass-I	2.08	4.087	0.0274	Yes
	0.5% Cellulose	1.99	3.410	0.0907	No
	0.5% Steel	2.00	2.617	0.0601	No
	1% Glass-s	2.03	2.372	0.0705	No
	1% Glass-l	2.47	7.338	0.0090	Yes
	1% Cellulose	2.10	3.353	0.0219	Yes
	1% Steel	2.12	3.242	0.0238	Yes
	2% Glass-s	2.06	2.368	0.0493	Yes
	2% Glass-l	2.17	4.786	0.0204	Yes
	2% Cellulose	2.06	4.316	0.0724	No
	2% Steel	2.05	3.405	0.0764	No

*f/b: filler-bitumen ratio.** indicates significant at the 95 percent confidence interval.

Table 5 Contact angle measurement of bitumen binder

	Contact Angle in Diiodomethane		Contact Angle in Glycerol		Contact angle in Water	
Sample	Average (°)	CV* (%)	Average (°)	CV (%)	Average (°)	CV (%)
Drained base binder	82.99	1.05	92.12	1.18	101.54	0.52
Drained cellulose	80.23	1.32	92.56	0.08	101.58	1.36
Drained glass-s	78.22	2.69	94.26	1.08	95.32	1.19
Drained glass-l	75.99	1.36	94.96	0.33	98.09	0.67
Drained steel	79.58	2.28	95.23	0.45	97.25	0.54

*CV: coefficient of variation

Table 6 Surface energy components of drained binder

Binder type	γ_s^{LW}	$\gamma^{AB}_{s}=2\sqrt{\gamma^{+}\gamma^{-}}$	γ ^{total} (mJ/m²)
Drained base binder	15.99	1.78	17.77
Drained cellulose	17.38	1.30	18.68
Drained glass-s	18.41	0.46	18.88
Drained glass-l	19.59	0.59	20.18
Drained steel	17.71	0.19	17.90

Table 7 Surface energy components of limestone aggregate

Aggregate	γ_s^{LW}	$\gamma^{AB}_{s}=2\sqrt{\gamma^{+}\gamma^{-}}$	γ^{total} (mJ/m ²)
Limestone	65.81	38.38	104.19

Table 8 Work of cohesion (2 γ $^{total})$ and pull-off tensile strength values (0.8 filler-bitumen ratio)

Sample	Pull-off tensile strength (MPa) 0.8 f/b ratio	Work of cohesion (mJ/m ²)
Drained base binder	1.19	35.53
Drained cellulose	1.84	37.35
Drained glass-s	1.85	37.76
Drained glass-l	1.95	40.36
Drained steel	1.81	35.80

Table 9 Work of adhesion results

Sample	Work of adhesion (mJ/m ²)
Drained base binder	122.69
Drained cellulose	125.18
Drained glass-s	172.88
Drained glass-l	158.64
Drained steel	163.21

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Figure 4 General representation of PATTI test [17]





Figure 5 Complex modulus master curves for drained binders (2.0% fibre by volume)









Figure 6 Pull-off tensile strength of base and modified mastics







Figure 7 Pull-off tensile strength of base and modified mastics







Figure 8 Pull-off tensile strength of base and modified mastics



462 Figure 9 Failure surfaces of 0.8 filler bitumen ratio asphalt mastic with limestone463 aggregate with different fibre types: (a) cellulose, (b) glass-s, (c) glass-l, (d) steel and

(e) base mastic.



Figure 11 Stress concentration due to fibre size [35]



485486Figure 12 Comparison between work of cohesion of drained binders and pull-off tensile486486 strength of 0.8 filler-bitumen ratio mastics (specimens undergoing cohesive failure)

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