# Construction and Building Materials

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**DEVELOPMENT OF COMPRESSION PULL-OFF TEST (CPOT) TO ASSESS BOND STRENGTH OF BITUMEN**

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23    **STRENGTH OF BITUMEN**

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29

30    **ABSTRACT**

31    The quantification of moisture susceptibility has been a major concern for researchers as it

32    adversely affects the performance of asphalt pavements. Several methods have been

33    developed to assess bond strength using asphalt mixture in loose or compacted state. These

34    tests lack in their ability to study fundamental properties that the bond between bitumen and

35    aggregate.  In this context, pull-off stub techniques have been developed such as pull-off stub

36    based tests and direct tension type tests. First group only measures the maximum pull-off

37    strength and second group has problems related to use of consistent binder film thickness and

38    operational problems in test itself.

39    This paper presents a new test to evaluate bond strength in an attempt to solve problems

40    associated with traditional pull-off techniques. This aim is achieved through review of existing

41    techniques, development of a direct tension test assembly and its evaluation, development of a

42    gap assembly and CPOT assembly. Key parameters for bitumen and mastics were evaluated.

43    The results show promising potential for use of this technique to study cohesive as well as

44    adhesive bond strength of binder.

45    Keywords: Pull-off test, asphalt, bitumen, adhesion, cohesion, bond strength tensile

46    strength, moisture damage, Compression Pull-Off Test, test development

# 1 Introduction

Binding material 'bitumen' is used in road construction all across the globe because of its good adhesive, cohesive and waterproof characteristics. These bituminous pavements are experiencing an ever increase in traffic load and its complexity. In addition to traffic loading, there are environmental factors that adversely affect durability and integrity of asphalt mixture. These include ageing and moisture damage as primary factors affecting durability of pavements, provided that pavement is constructed according to specifications (Airey and Choi 2002). Ageing makes bitumen stiffer and brittle leading to its susceptibility of thermal cracking. On the other hand, moisture damage result in deterioration of adhesive and cohesive bond in asphalt mixtures.

Moisture damage is a complex phenomenon can be defined as "progressive functional deterioration of a pavement mixture by loss of the adhesive bond between the bitumen and aggregate surface (stripping) and/or loss of the cohesive resistance within the binder principally from the action of water"(Kiggundu and Roberts 1988). The loss of bond strength due to water damage lead to weaker pavement layer and makes it prone to deform under traffic loading leading to deterioration (Airey and Choi 2002; Moraes, Velasquez, and Bahia 2011). The cohesive failure occurs due to deformation under load at a distance from aggregate that is beyond the influence of mechanical interlocking and surface molecular orientation (Chaturabong and Bahia 2018). A cohesive failure mechanism can further to an adhesive failure when the emulsification effects reach the aggregate surface (Fromm, 1974).

The five mechanisms which produce moisture damage, have been reported as; detachment, displacement, spontaneous emulsification, pore pressure, and hydraulic scouring (Taylor A and Khosla Paul 1983; Bagampadde, Isacsson, and Kiggundu 2004) The later study has also discussed microbial activity and osmosis as additional factors. There are four common approaches/theories that explain the bond bitumen and aggregate; chemical reaction, surface

72    energy (surface change), molecular orientation, and mechanical adhesion (mechanistic

73    tenacity) (Shute et al. 1989; Bagampadde, Isacsson, and Kiggundu 2004). Kringos et al. (2008)

74    have discussed three modes of moisture infiltration. The first being entry of water in connected

75    macro-pores through rainfall. Secondly, due to stationary moisture in the form of liquid or

76    vapour resides in the macro-pores. Third mode being the presence of water inside aggregate

77    before laying of the wearing course and inadequate drying of aggregate.

78    One of the earliest test to evaluate properties of bitumen included chew test. In this test,

79    builders tried to assess not only the consistency and but also stickiness of bitumen. Since then

80    several methods have been devised for measuring fracture of interfaces and adhesive joints. In

81    addition to these methods, several approaches has also been used to improve the bond

82    strength of bitumen and aggregate. This includes selection of suitable combination of binder

83    and aggregate, modification of bitumen (Baldi-Sevilla et al. 2017), improvement of mixing

84    techniques, and reduction of dust powder on surface of aggregate. However, there are

85    difficulties associated with these methods to improve bond between bitumen and aggregates

86    using these methods (Peng et al. 2018). In addition to this different antistripping additives such

87    as hydrated lime has been used to improve strength and reduce moisture susceptibility of

88    asphalt mixtures (Huang et al. 2005; Kim, Pinto, and Park 2012; Ameri, Kouchaki, and Roshani

89    2013; Zaidi 2018).

90    In discussion of moisture damage in asphalt mixture the tests classified in two categories; tests

91    conducted on loose and compacted mixtures, (Lottman et al. 1974). Several research has

92    summarised these moisture sensitivity tests (Terrel and Shute 1989; Terrel and AI-Swailmi

93    1994; Airey and Choi 2002; Bagampadde, Isacsson, and Kiggundu 2004; Solaimanian et al.

94    2003). Test on loose mixture are empirical in nature and rely on visual inspection. On the other

95    hand, test on compacted mixtures are more fundamental in nature (Airey and Choi 2002).

96  Additionally, several studies has been performed to quantify bond strength based on fracture

97  parameters, surface energy, diffusion coefficients and adhesion (Kim, Pinto, and Park 2012).

98  Hitherto, there is no unified and standard fundamental test method for evaluation of bond

99  strength. (Moraes, Velasquez, and Bahia 2011) (Wang, Yi, and Feng 2014), (Rahim 2017) (Zhou

100 et al. 2018). This is because the amount of research performed to study tensile properties of

101 thin films of bitumen has been relatively little compared to other means of evaluating bitumen

102 properties (Chang 1994).

## 2    Objectives

104 This research aims to evaluate pull-off bond strength through development of a better, simple

105 and robust testing mechanism to solve problems associated with traditional pull-off testing

106 techniques. The test method developed to achieve this aim is termed the 'Compression Pull-Off

107 Test (CPOT)'. Following objectives were defined for this research;

108     • Review of existing bond strength techniques

109     • Evaluation of direct tension test approach

110     • Development of mechanism to achieve required binder film thickness

111     • Development of compression pull-off test (CPOT)

112     • Evaluation of the key parameters for binder testing and validation of results

## 3    Review of bond strength methods

114 There has been numerous research efforts to select most appropriate methods to study

115 moisture sensitivity affected by loss of bond between bitumen and aggregate (Rice 1958;

116 Lottman et al. 1974; Terrel and Shute 1989; Airey and Choi 2002). The traditional methods on

117 loose and compacted mixtures such as Lottman's procedure and its advanced modification i.e.

118 AASHTO T 283) are useful in comparative analysis of moisture suspectibilites of various HMA

119 mixtures. These methods, however, meausres bulk properties of mixtures and lack in focus on

120 fundamental material properties (Bhasin et al. 2006; Canestrari et al. 2010; Cho and Bahia

121 2010; Taylor, Hamedi, and Nejad 2014). This creates the necessasity to evalute bond strength

122 using component charactersitics tests which measure the fundamental properties. Additionally,

123 testing based on components characteristics is generally more economical (Kim, Pinto, and Park

124 2012). A fundamental test on binder and aggregate will give better understanding of moisture

125 sensitivity and its effect on the cohesive and adhesive bond of bitumen and aggregateds

126 (Moraes, Velasquez, and Bahia 2011).

127 In a study, Jakarni (2012) has summarized some of the common tests used in science and

128 technology of adhesion to measure the adhesive bond strength of coatings of the composite

129 materials. These tests included peel test, pull-off test, double cantilever beam (DCB) test,

130 tapered double cantilever beam (TDCB) test, impact wedge peel (IWP) test and scratching of

131 thin films test. The review of experience in adhesive technology lead Jakarni (2012) to

132 formulate a new pull-off testing technique to evaluate the bond strength.

133 In binder research, peel test has been used to quantify the adhesive strength (Blackman et al.

134 2013; Zhang et al. 2017; Zhou et al. 2018). A flexible thin peel arm is adhered on a substrate

135 with the use of adhesive material. A pull load is applied through peel arm at a constant speed

136 and specific angle and force to initiate and propagate peel fracture is measured. This peel force

137 is recorded as a function of the displacement to calculate fracture energy. In these studies, it

138 has been demonstrated that that the peel test is a suitable method to determine the adhesive

139 fracture energy. In addition to this, there are numerous studies which emphasis the importance

140 of thin film binder to evaluate its response to pull-off loading (Kanitpong and Bahia 2005;

141 Jakarni 2012; A Copeland et al. 2006; Audrey Copeland, Youtcheff, and Shenoy 2007; Poulikakos

142 and Parti 2011; Alvarez, Ovalles, and Caro 2012; Harvey and Cebon 2003; Apeagyei, Grenfell,

143 and Airey 2014; Sultana 2014; Al-Haddad and Al-Khalid 2015; Rahim 2017; Abd, Al-Khalid, and

144 Akhtar 2018; Chaturabong and Bahia 2018)(Audrey Copeland, Youtcheff, and Shenoy 2007).

145  A commonly used pull-off stub type test is Pneumatic Adhesion Tensile Testing Instrument

146  (PATTI) to assess the moisture damage based on bond strength. Copeland (2007)modified the

147  procedure samples in PATTI to improve the control of the bitumen film thickness. (Santagata et

148  al. 2009) has also modified PATTI and reported on reliability and practicality to evaluate the

149  adhesion/cohesion properties of asphalt-aggregate system. One adaptability of PATTI is

150  Bitumen Bond Strength test (BBS) to evaluate pull-off strength (Kringos, N., Scarpas, A., and de

151  Bondt 2008; Moraes, Velasquez, and Bahia 2011; Zhou et al. 2018). BBS results are reported to

152  be reliable, repeatable and reproducible (Canestrari et al. 2010; Moraes, Velasquez, and Bahia

153  2011; Chaturabong and Bahia 2018; Mohammed et al. 2018). However, pull-off stub based

154  methods only measures the maximum pull-off strength and reports on description of coating

155  fracture. This pull-off stub approach has its limitation in a sense that rate of loading (in terms of

156  displacement) cannot be controlled (Zhang et al. 2017). In contrast another approach, Direct

157  Tension Test (DTT) methods have their advantage in measuring the pull-off load and elongation

158  (Rahim 2017; Abd, Al-Khalid, and Akhtar 2018).

159  There are several direct tension based studies to evaluate bond strength (Harvey and Cebon

160  2005; Jakarni 2012; Sultana 2014; Abd, Al-Khalid, and Akhtar 2018). In work with direct tension

161  tests, researchers have reported difficulties in preparing aggregate samples, achieving

162  consistent film thickness and perform tests itself (Abd, Al-Khalid, and Akhtar 2018). In DTT,

163  epoxy resin adhesive has been used to fix the aggregate plates with testing fixture (Peng et al.

164  2018). The use of these adhesive materials result in a slow process and require removal of

165  excessive adhesive from around the fixture. Another approach is to fix the aggregate plates in

166  testing moulds using screws. This fastening mechanism has operational problems associated to

167  it. The control of film thickness in specimens have been another concern, different approaches

168  and gap assemblies have been used (Jakarni 2012) . The use of Dynamic Shear Rheometer for

169  control of bitumen film at submicron level has also been reported (Zhang et al. 2017; Al-Haddad

170  and Al-Khalid 2015).

171   Surface Free Energy (SFE) is another useful technique to evaluate adhesion between aggregate

172   and binder. (Kringos, N., Scarpas, A., and de Bondt 2008) used surface energy approach and

173   combined direct tension test approach with numerical moisture diffusion analyses to

174   demonstrate that results consistent with expected field performance. The works highlights the

175   importance of importance of mechanical tests to assess bond strength. In addition, there has

176   been several attempts to correlate bond energy (from SFE) and total work of fracture (from

177   pull-off tests). The problem with SFE is that it is a thermodynamics based approach that do not

178   take into account energy dissipation during loading and unloading. Moreover, total work of

179   fracture (pull-off strength) is dependent on test geometry and testing conditions (Howson

180   2011).

181   The review of pull off techniques concludes with an importance of direct tension based

182   approach to evaluate the bond strength. There is, however, need to address the difficulties

183   associated with this test approach. Any such attempt must be effective to measure maximum

184   bond strength, rate of deformations corresponding to pull-off load and availability of fracture

185   surface to examine.

186   **4   Test materials**

187   The conventional properties of bitumen for this research are listed in Table 1. The work

188   included use of aluminium plates (35.1 mm diameter) as a control. This helped to ensure

189   substrate plates were parallel to achieve a uniform film thickness. The control material was also

190   convenient to use because of its high thermal conductivity to quickly cool down aluminium-

191   binder system. This ensured ready availability of gap assemble to prepare multiple specimen in

192   shorter interval of time. In addition, limestone (Ls) and granite (Gr) fillers (passing sieve 63

193   micron) were used to prepare mastics. The percentage of fillers was added as 40% by mass of

194   bitumen. These mastics were prepared by gradually mixing fillers in an oven heated bitumen

195 over a hot plate. In addition to these materials, limestone plates were used to validate the
196 results of CPOT.

197 **Table 1: Bulk mass properties of bitumen**

| Bitumen | Source 1 Pen 60/70 | Source 2 Pen 40/50 | Source 2 Pen 60/70 |
|---|---|---|---|
| **Penetration Grade (dm)** | 64 | 47 | 69 |
| **Specific Gravity/Density** | 1.04 | 1 | 1.02 |
| **Softening Point (°C)** | 51 | 56 | 49.5 |

198 ## 5      Test methods

199 A literature survey helped to formulate the basic requirements for a pull-off strength
200 evaluation. This required a direct and fundamental method to measure adhesive/cohesive bond
201 strength (DTT and CPOT). These methods were selected/devised on the basis of; direct
202 measurement of practical work of fracture, approach in which displacement can be controlled,
203 simplicity, practicality and cost effective to test on binder level. A gap assembly was also
204 devised to achieve required binder film thickness. In addition to these tests, RTFO was used for
205 short term ageing of bitumen and mastics.

206 ### 4.1     Development of gap assembly for film thickness

207 A gap assembly was developed to achieve required film thickness of binder. In this assembly, a
208 compressive load was applied through a rotating disc. The resultant compression on binder was
209 measured with a deflection gauge (with precision 0.05mm and range 0-20mm). A 10° rotation
210 of the rotating disc compressed the bitumen layer to 50.56 micron. In order to achieve a
211 statistically consistent film thickness, the boundary of the whole assembly and position of the
212 needle was marked before testing. The compression device was able to apply a strong pressure
213 to uniformly compress even a penetration grade 40/60 at room temperature to layer of 0.5mm.
214 The lateral movement of substrate was prevented by providing grooves in the upper and
215 bottom plates of assembly.
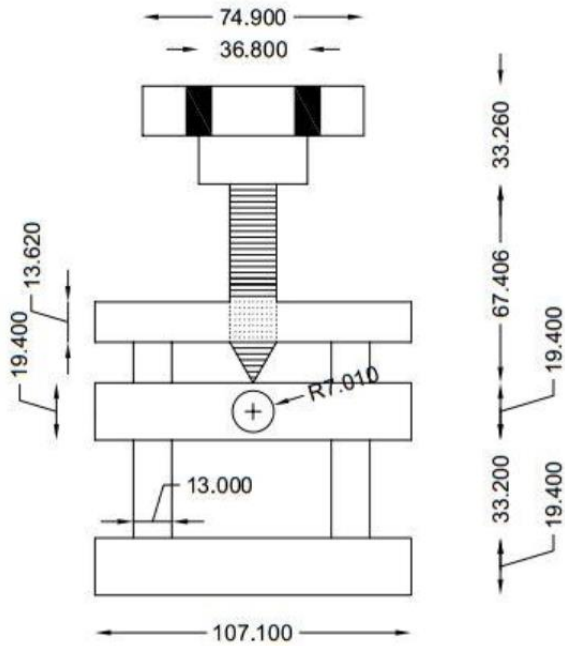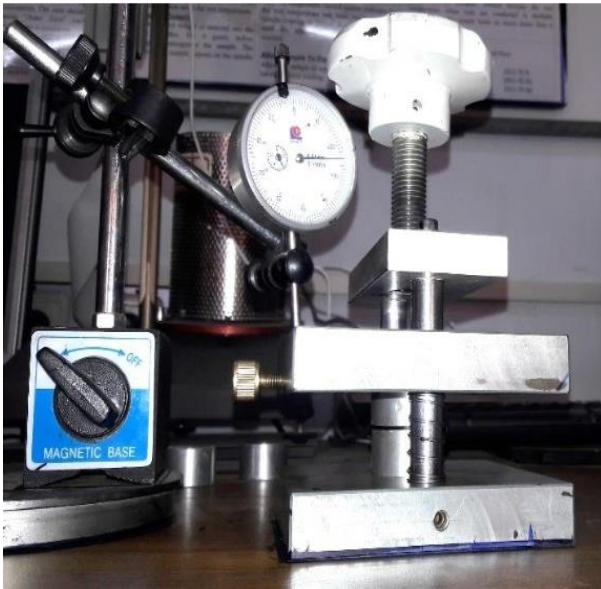
216



217 **Figure 1: The compression gap assembly**

218 In specimen preparation, hot bitumen was poured onto an aluminium plate. A second plate was

219 placed onto first plate to create a sandwiched specimen. A preconditioning of these plates at

220 110 °C before pouring was identified to be a best practice. However, in case of plates

221 conditioned at room a slight oven heating of sandwiched specimen was needed. The preheating

222 before pouring helped to overcome hydrogen bond, p-p bonding and Van der Waals forces. The

223 presences of these forces was expected to reduce the wetting of the substrate plates

224 (Bagampadde et al., 2004). The sandwiched specimens were then compressed in gap assembly

225 to achieve required thickness. After curing in gap assembly at room temperature, specimens

226 were removed and left for additional curing/conditioning at room temperature for 24 hours.

227 After conditioning period specimens were ready to test with direct tension test and CPOT.

228 **4.2   Direct tension test assembly**

229 In order to test contemporary direct tensile test (displacement controlled) approach, a direct

230 tension assembly was fabricated. The modifications were made on the basis of joining

10

231  mechanism to connect with a 10 kN capacity Universal Testing Machine (UTM). The test

232  assembly is shown in Figure 2 (left). In tension testing, sandwiched aluminium plates were

233  gripped in the upper and lower moulds with three under-head screws. The top and bottom

234  extended arms of the moulds were fixed in the saw tooth grip of UTM as shown in Figure 2

235  (right). The testing was performed after adjusting the test parameters in software.

236



237  **Figure 2: Direct pull-off testing assembly**

238  In view of operational problems associated with DTT and variability of its result (section 5), a

239  better mechanism for testing was needed. The need for following improvements were

240  identified.

241  • The three-point grip (3 under-heads) was deemed insufficient in direct tension test.

242   During testing, aluminium plates were getting damaged due to drag and slip of plates. A

243   thorough gripping mechanism was needed.

244  • The surfaces of tested specimens were getting damaged, creating problem in analysing

245   the cohesive failure. This also added problem in observing phenomenon of cavitation.

11

246    • A consideration of authors' experience with pull off created need to conceptualise a

247       better testing mechanism rather than adding further modifications to existing moulds.

248    **4.3    Compression pull-off test assembly (CPOT)**

249    The rationale of this test was derived from traditional compression tests performed on concrete

250    and asphalt mixtures. The basic principle was thus formulated as "the load applied in

251    compression of the UTM should generate a pull-off load on the specimen". A conceptual

252    diagram was prepared to translate this novel idea into reality (Figure 3, left). The idea was

253    found to be feasible in terms of manufacturability. The assembly was designed and

254    manufactured locally with 'lean manufacturing'. This philosophy called for developing the

255    product to a minimum functional level then improving the design according to needs (Shah and
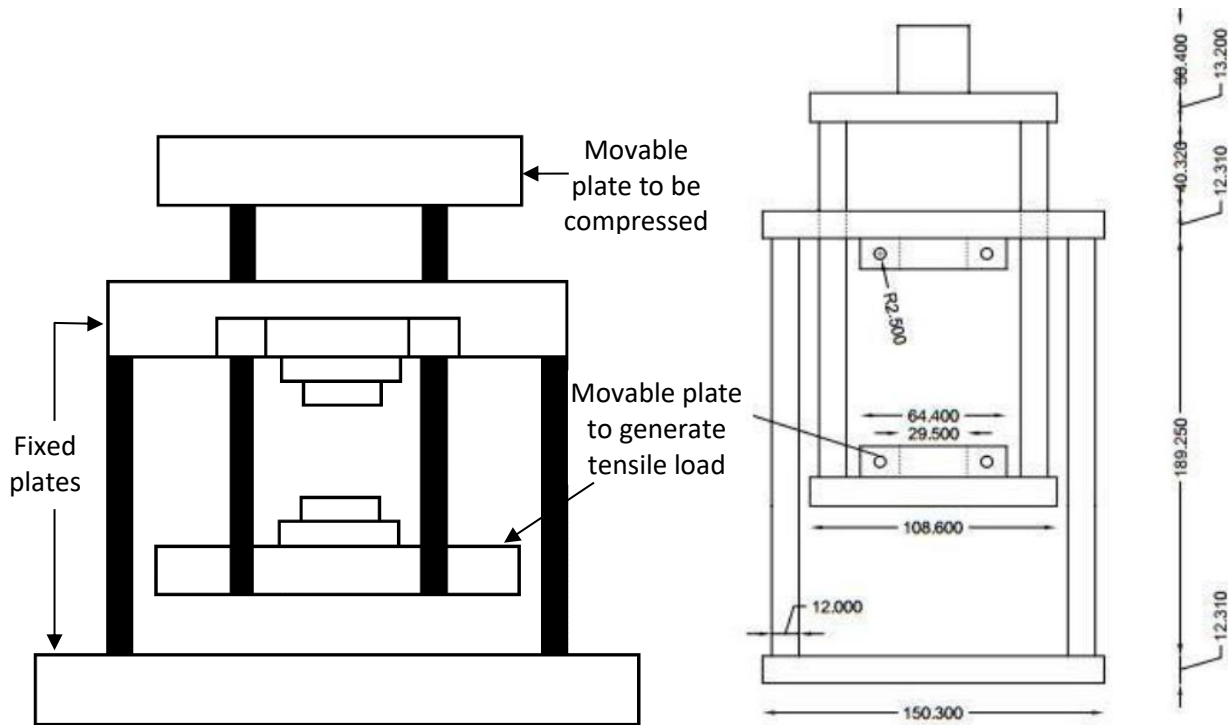
256    Ward 2003).



257
258    **Figure 3: Conceptual diagram of compression device for pull-off testing**

259  The Figure 4 (left) shows the first complete operational assembly. The sandwiched specimens

260  were fixed by means of two horizontal sliding plates. Circular groves in these plates to

261  thoroughly gripped the substrates. The screws in assembly were tighten to firmly squeeze the

262  substrate. This approach added a simplicity to design and ease of operation. Figure 4 (right),

263  shows the three further design improvements which were added as per need of trial testing. In

264  first modification two springs were added around the bars in upper moveable part to prevent it

265  from striking the fix part. In second modification, a shaft was introduced to movable part for

266  ease of centring and alignment. Thirdly, a half spheroid was introduced on the loading shaft for

267  the seating adjustments. A hole in the base of fix part was introduced for alignment purposes.

268  This assembly was named as 'Compression Pull-Off Test (CPOT) assembly'. This signified that

269  this assembly was able to take a compression load from an external source and translate it to a

270  uniform co-axial pull-off load on a binder-substrate system.
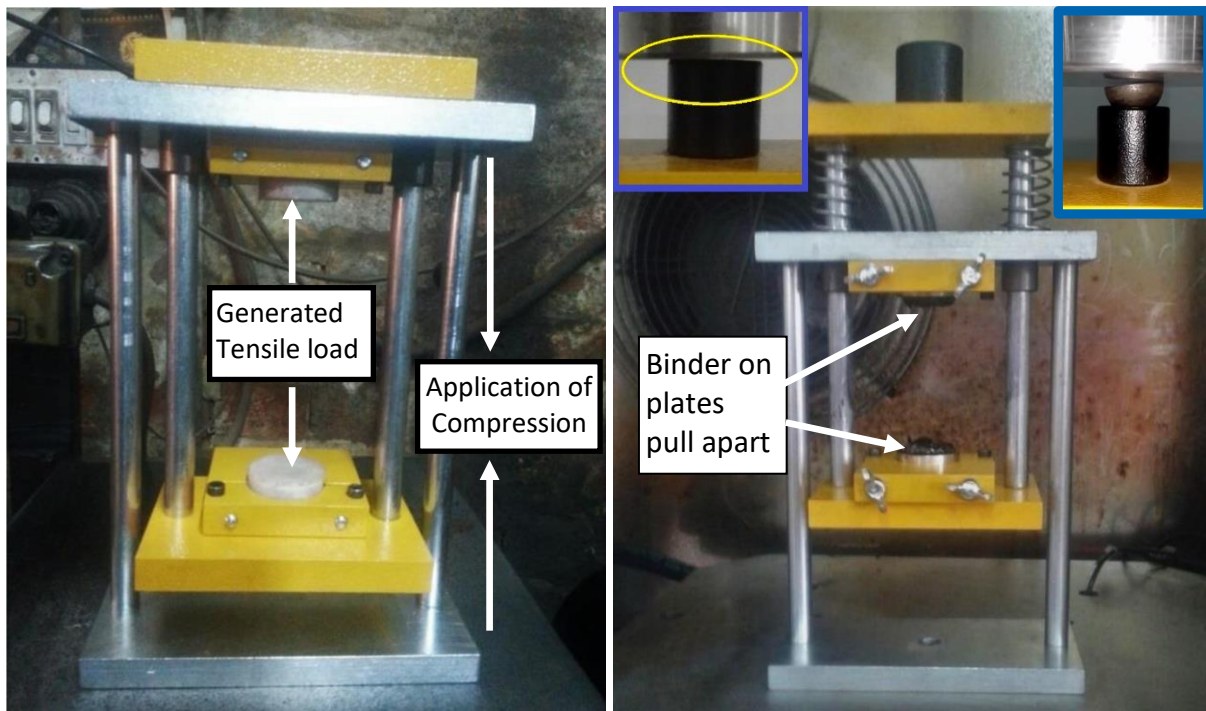


271

272  **Figure 4: First prototype ad final design of CPOT assembly**

273    The specimens prepared from gap assembly were inserted in the upper plate and tightened

274    with hand pressure. Lower plate was raised to grip the bottom aluminium substrate. After fixing

275    both ends of specimen, a slight pressure was applied with a small wrench to ensure firm grip. It

276    is important to mention that in entire testing phase no specimen broke during installing, fixing,

277    handling, and testing of assembly. This griping mechanism seemed to have better tolerance

278    towards even slightly misaligned plates of sandwiched specimen.

279

280    In testing phase, the test parameters were defined in terms of loading direction, loading rate

281    (mm/min), specimen dimension and elongation limits etc. After this, a small seating load (few

282    Newtons) was applied to ensure proper contact. In some cases, specimens were discarded due

283    to application of accidental load application (higher than 100N). The final load was applied in

284    strain control mode and results were obtained in terms of load and elongation.

285

286    **5    Results and discussion**

287    The results were analysed in terms of mean pull-off strength (POS) and coefficient of various

288    (COV) for at least five replicates. Maximum pull-off strength of a specimen, expressed in MPa,

289    was calculated by dividing failure load in ($F_n$) by area of contact at surface i.e. $\mathrm{POS} = 4\frac{F_n}{\pi D^2}$,

290    where $F_n$ is expressed in Newtons and mean diameter of substrate 'D' is expressed in (mm). The

291    acceptance of and rejection were based on observation as per some guidelines of ASTM:

292    D4541-17. The pull-off strength parameters evaluated for materials, instrumentation and key

293    test parameters.

294

295    **5.1    Repeatability and operator variability of gap assembly**

296

297    The accuracy of film thickness was checked with Vernier caliper for 36 specimens before and

298    after pouring of binder. These specimens were grouped according to plate condition. In cold

14

299     plate case, each specimen was prepared by pouring hot binder onto a clean control plate,

300     which was preconditioned at room temperature. This plate condition was labelled as 'cold'.

301     The specimens in cold condition required slight heating in oven before introducing to gap

302     assembly. In hot plate case, specimens were prepared by pouring hot bitumen onto preheated

303     aluminium plates at 110 °C. This plate condition was labelled as 'hot'. In comparison on method

304     of pouring (Table 2), a good repeatability was achieved for pouring on hot aluminium plates.

305     The operator 1 (experienced) achieved more repeatable results as compared to operator 2 with

306     same method of pouring. This shows that the repeatability is dependent on method of

307     specimen preparation and operator's understanding of best laboratory practices.

308

309     **Table 2: Film thickness in gap assembly**

| Operator | Plate condition | Film Thickness (mm) | | | | | | | Replicates |
|---|---|---|---|---|---|---|---|---|---|
| | | Max | Mean | Range | SD | COV (%) | Mean +2 SD | Mean -2 SD | |
| Operator 1 | Cold | 0.74 | 0.55 | 0.44 | 0.13 | 23.90 | 0.29 | 0.81 | 10 |
| | Hot | 0.63 | 0.50 | 0.21 | 0.06 | 12.09 | 0.38 | 0.63 | 10 |
| Operator 2 | Hot | 0.58 | 0.45 | 0.33 | 0.10 | 21.92 | 0.25 | 0.65 | 16 |

310

311     In next stage, this effect of repeatability on these specimen was further evaluated with CPOT.

312     Tests were performed with; 10mm/min rate of loading, 0.5mm film thickness, 15 hour dry/wet

313     conditioning on untrimmed specimen. Two specimen were discarded for operator 1 in each

314     case and six specimen for operator 2 were used for additional conditioning, the result of these

315     specimens therefore are not included. The mean POS and COV values for these film thicknesses

316     are shown in Table 3 to evaluate for repeatability.

317

Table 3: CPOT results for repeatability of gap assembly

| Binder | POS | COV | Test condition | Replicates |
|--------|-----|-----|----------------|------------|
| | 1.17 | 13.54 | Operator 1, dry 15 hours, cold | 9 |
| Short term aged binder | 1.25 | 8.40 | Operator 1, wet 15 hours, hot | 9 |
| | 1.53 | 14.80 | Operator 2, dry 15 hours, hot | 10 |

**Operator 1 and 2 for dry conditioning:** In Table 2 and Table 3, operator 1 achieved 100 µm more film thickness for than operator. This should have led to a slightly lower POS theoretically. However the actual difference is significantly less, possibly due to bad bonding because of cold plate condition and non-uniformity of binder inside the plates.

**Comparison of dry and wet for hot plate condition:** Operator 2 should have achieved slightly increased POS due to 50 µm decrease of film thickness. However, dry POS is significantly higher as compared to wet conditioning. This suggests main cause of adverse effect is moisture conditioning.

**Comparison for cold/hot and dry/wet for operator 1:** The wet conditioning should have resulted in decrease of POS and a 50 µm film thickness difference should result in slight increase of POS. The net effect should be a decrease, this is however not the case. The only possible explanation is a bad bond achieved with cold plate condition.

The quality of results for good and bad bonding was checked through load-elongation curves. The results of that gap assembly are repeatable for hot plate condition (already identified as a best laboratory practice in section 5.1). The film thickness and CPOT tests were performed on an aged (stiffer) binder. The results showed that repeatability was dependent on specimen preparation method and operator's understanding of best laboratory practices. In further tests, cold plate condition needed careful consideration for mastics and aged binder.

**5.2    Results and discussion on DTT results**

The sandwiched specimens prepared with gap assembly were tested with DTT moulds. In testing on bitumen specimens were by pouring hot bitumen on a hot aluminium plate and trimming the specimen after curing. For granite mastic, binder was prepared in cold plate condition and tested under untrimmed condition. The results show a variability of 16.8% (Table 4) for virgin bitumen and 33.9% in case of granite mastic. This significant variability is caused by the combined action of specimen preparation method as well as testing with DTT moulds itself.

**Table 4: Summary of test results using direct pull-off testing**

| Material | Pull-Off Strength (MPa) | COV (%) | Plate Condition | Replicates |
|:---:|:---:|:---:|:---:|:---:|
| Source 2 Pen 60/70 | 0.71 | 16.78 | Hot | 6 |
| Gr (40%) in Source 1 Pen 40/60 | 1.01 | 33.86 | Cold | 6 |

Figure 5 shows ductile (left) and brittle (right) mode of failures for bitumen and mastic respectively. This brittle failure indicates a sudden drop in load carrying capacity after failure load. This brittle failure may have been cause by increased stiffness and bonding due to cold conditions providing weakest plane of failure in specimen. The ductile mode was dominate for virgin bitumen and is identified from the ability of the material to take load after failure in cohesive mode. This indicate that bond failure starts within the molecules of the bitumen or bitumen mastic interface due to nucleation of micro voids. The negative slope in the softening portion is dependent on the degree of ductility of material (Poulikakos and Parti 2011).

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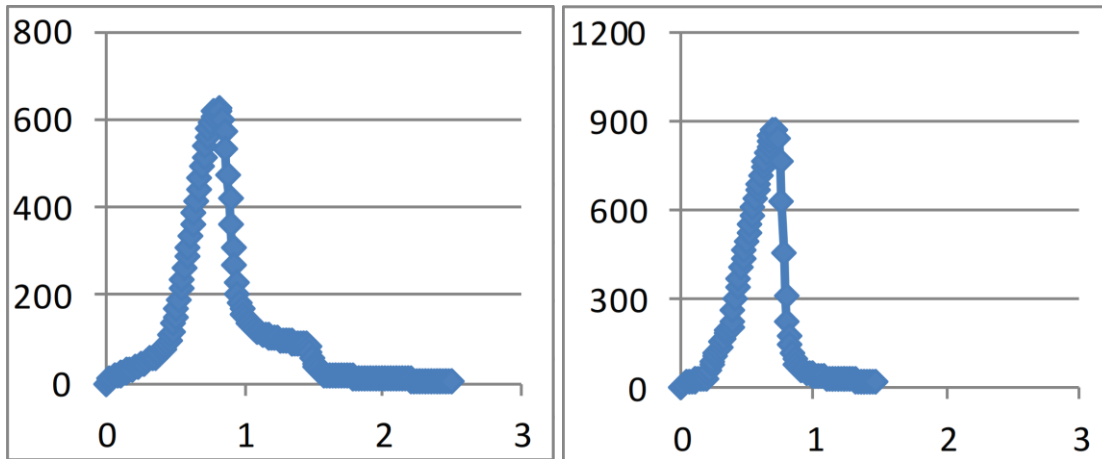


**Figure 5: Brittle and ductile type failure using DTT**

The direct tension assembly was provided with double universal joint on the upper and lower moulds for seating and self-aligning. This modification was created in an attempt to increase the repeatability of test results. However this resulted into damage of failure surfaces. The DTT of this research as well as other techniques in this approach require several procedural steps. This lead to excessive handling and breaking of specimens. Also some specimen slipped out of moulds during test, leading to misleading results. In order to solve these issues a new design and approach necessary.

**5.3    Discussion on Results of CPOT**

In this test, the main objectives was to evaluate parameters related to testing mechanism and material to establish its usefulness. The parameters related to specimen were evaluated using direct pouring and silicon method of pouring. The constant test parameters included; film thickness (0.5mm), rate of loading (10mm/min), cold plate condition, 24 hours dry conditioning, and room temperature testing.  Table 5 shows a decrease of pull-off strength and increased coefficient of variability with silicon pouring and trimming for bitumen in round 1. This was also found to be true for tests on 3 hours aged binder (round 2). However at this stage, POS include combined the effect of method of pouring and trimming of specimen. A third round of tests were performed on RTFO aged binder to exclude the effect of trimming. The bond strength

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376  decreased by 20.6% only by changing the method of pouring. Silicon method of pouring gave

377  lower POS with increased variability. The unaged binder showed only a 9.1% decrease of binder

378  in bond strength. This difference of can be attributed to trimming and ageing. The aged binder

379  shows increased bond strength with shift from ductile to brittle failure. The effects of silicon

380  pouring and trimming seems to be contradictory with Dynamic Shear Rheometer testing in

381  which former is a compulsory step and second is an alternative to direct pouring. This is

382  attributed to different nature of two tests for quantification of tensile strength (pull-off

383  strength) and shear strength respectively. In addition to this, different studies have used

384  trimming of specimen in pull off testing (Sultana 2014; Al-Haddad and Al-Khalid 2015; Apeagyei,

385  Grenfell, and Airey 2015; Abd, Al-Khalid, and Akhtar 2018). This study suggests effect of

386  trimming on the results of pull-off strength. The coefficient of variability in all test groups

387  indicate a good repeatability of CPOT results.

388                **Table 5: Tests to check repeatability of the test method**

| Round | Material | Pull-Off Strength (MPa) | COV (%) | Variable Testing conditions | Replicates |
|-------|----------|-------------------------|---------|-----------------------------|------------|
| **Round 1** | Source 2 Pen 60/70 | 0.70 | 9.67 | Silicon, tr | 5 |
|  |  | 0.77 | 6.82 | Direct, un | 8 |
| **Round 2** | 3-hour oven aged Source 1 Pen 40/50 | 1.4 | 13.35 | Silicon, tr | 9 |
|  |  | 1.45 | 11.04 | Direct, un | 8 |
| **Round 3** | RTFO aged Source 1 Pen 60/70 | 1.00 | 11.63 | Silicon, tr | 7 |
|  |  | 1.26 | 8.35 | Direct, tr | 9 |

389  In Table effect of decrease in film thickness have resulted in increase of pull-off bond strength.

390  The mode of failure in case of 0.5mm film thickness was cohesive while for 0.3mm film

391 thickness it changed from hybrid to adhesive. This is consistent with findings in literature on the

392 effect of film thickness on pull-off bond strength (Marek and Herrin 1968; Chang 1994; Fond

393 2001; Harvey and Cebon 2003, 2005; Poulikakos and Parti 2011; Jakarni 2012; Sultana 2014;

394 Abd, Al-Khalid, and Akhtar 2018).

395 **Table 6: Effect of film thickness on pull-off bond strength**

| Material | Mean POS (MPa) | COV (%) | Film Thickness (mm) | Failure Mechanism | Replicate |
|---|---|---|---|---|---|
| **Source 2 Pen 60/70** | 1.29 | 20.97 | 0.3 | Hybrid to adhesive | 7 |
| **Source 2 Pen 60/70** | 0.71 | 16.78 | 0.5 | Cohesive | 6 |

396 The typical failure curves in ductile mode are presented in Figure 6 for comparison of DTT and

397 CPOT. Firstly in both tests, a deviation from this is attributed to an anomaly in test specimens

398 and test itself. These problem could arise due to issue in specimen preparation,

399 curing/conditioning, non-uniform film thickness, non-homogenous mixing of filler particles and

400 mainly slipping of plates during test. The load elongation curve is advantageous to both method

401 in comparison to pull-off stub tests. In comparison to CPOT, direct tension test curve before

402 peak load is different due to seating adjustments. The CPOT has already taken care of major

403 seating adjustment during manual adjustment prior to start of test. The CPOT curve can be

404 directly analysed while DTT curve need normalisation to study energy dissipation. Hitherto,

405 authors have found no discussion in binder studies dealing with advantage due to compression

406 approach. CPOT is useful in study of load elongation curves as compared to contemporary
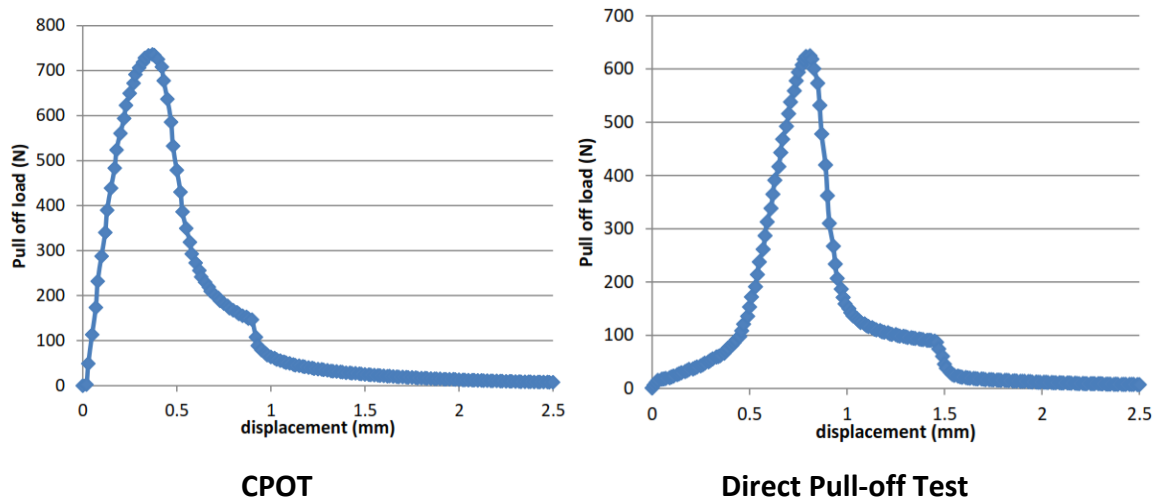
407 direct tension tests.

**CPOT**          **Direct Pull-off Test**

**Figure 6: Typical ductile failure with CPOT and direct pull-off testing**

### 5.4    Validation of CPOT results

The results were validate with RTFO aged limestone mastics and use of limestone aggregate
plates. The conditioning time was limited to 24 hours as main of equilibrium uptake occurs
during this conditioning period (Apeagyei, Grenfell, and Airey 2015). Table 5 shows strength of
aged mastic has significantly decreased tested with moisture conditioning. The dominate type
of failure in this case hybrid to adhesive. This is because mastics and aggregate plates allow
easier access of water within the mass of binder and to the interface. The dry conditioned
specimens have also shown tendency towards hybrid to adhesive failures in case of aggregate
plates. This may have been caused by residual dust on aggregate plates, in addition to less
control over plate surface as compared to control. This have increase chance of weak failure
plane near the bitumen aggregate interface. The cohesive strength is only completely available
if the interface bond between binder and aggregate is of good quality. The CPOT results have
shown a promising results to evaluate moisture damage.

**Table 5: CPOT results with aggregate plates and moisture conditioning**

| Material | Mean POS (MPa) | COV (%) | Conditioning | Plates | Replicate |
|---|---|---|---|---|---|
| Short term aged Ls | 1.38 | 11.53 | Dry | New | 6 |
| 40% in Source 1 | 0.96 | 33.31 | Dry | Used | 5 |
| Pen 40/50 | 0.81 | 16.15 | Wet | New | 6 |

A second evaluation was made with re-use of cleaned aggregate plates. The results indicate a significant reduction in bond strength also with increased coefficient of variance. This is because of decrease in presence of activate bond sites on re-used aggregate surface. This may have been caused by presence of water due to insufficient drying or accumulation of oily components due adsorption of binder in first use.

These findings in addition to discussion on previous results of CPOT have shown good repeatability and robustness of the method to test parameters related to material, ageing and moisture conditioning. This test is successful in quantifying the bond strength of bitumen involving fillers, effect of ageing, and moisture conditioning.

## 6   CPOT results comparison with developed DTT
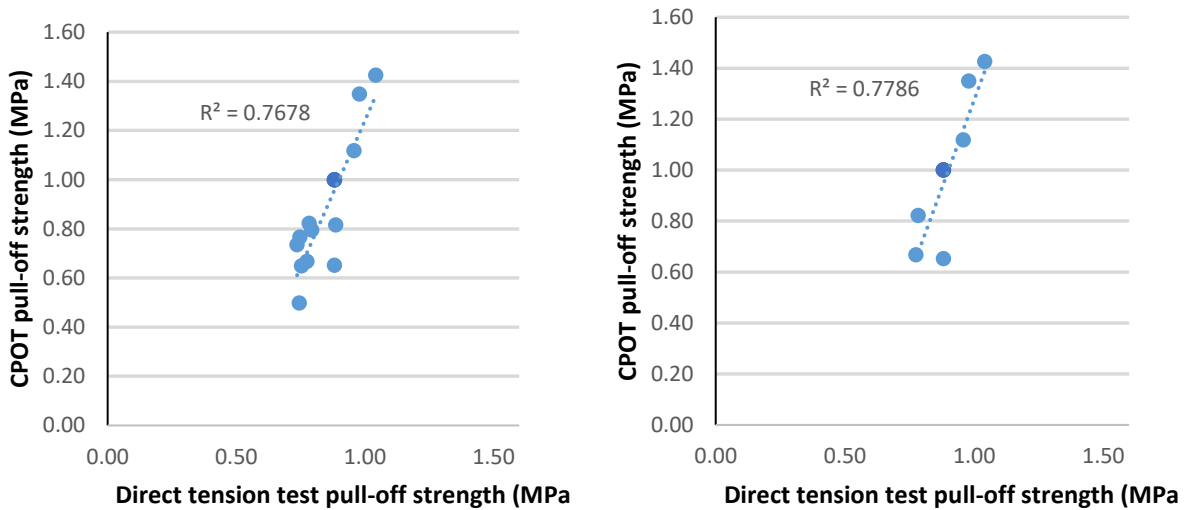
The results of the two test assemblies are presented in Table 6. It is evident from the variability of the CPOT results that this method is repeatable and effective in evaluating bond strengths. The variability of the Direct Pull-Off Test developed in this study is significantly higher than CPOT. This is in the case when CPOT specimens were poured onto a cold plate (condition selected to cause variability in results). A correlational analysis was performed between two tests as shown in Figure 7. The test shows a medium correlation between the two tests. CPOT seems to measure higher bond strength then DTT approach used in this research with limited tests results. This quantitative comparison needs further evaluation of DTT results.

**Table 6: CPOT Vs Direct Pull-Off Testing Assembly**

| Method | Material | POS (MPa) | COV (%) | Conditions | Replicates |
|---|---|---|---|---|---|
| Compression Pull-Off Test Assembly (CPOT) | Source 2 Pen 60/70 | 0.77 | 6.82 | Cold | 8 |
| | Granite (40%) in Source 1 Pen 40/50 | 0.89 | 12.2 | Cold | 6 |
| Direct Pull-Off Test Assembly | Source 2 Pen 60/70 | 0.71 | 16.78 | Hot | 6 |
| | Gr (40%) in Source 1 Pen 40/50 | 1.01 | 33.86 | Cold | 6 |



**Figure 7: Correlational analysis of CPOT and DTT**

# 7 Comparison with pull-off stub and DTT approach

A further qualitative comparison of CPOT with DTT approach of this study, pull-off stub as well as different direction tension approaches is presented in Table 7. The comparison ranging from specimen preparation and handling to examination of the failed surface. This comparison establishes the usefulness of this method for evaluating pull-off strength.

23

**Table 7: Comparison of CPOT with Pull-off stub and DTT approach**

| Parameter | Pull-off stub and DTT approaches | Compression Pull-Off Test Assembly |
|---|---|---|
| **Comparison with Pull Stub type tests** | Pull-off stub type tests such as PATTI only measure maximum pull-off strength and do not taken into account load elongation behaviour. | Captures complete range of data required for testing of adhesive and cohesive bond. |
| **Breakage of specimen during handling** | In DTT approaches, specimen breakage is a common problem due to handling especially during clamping in UTM. | Out of dozens of specimens tested no specimen broke during fixing and handling. |
| **Fitting into UTM** | DTT tests requires careful clamping to avoid misalignment and breakage of specimen. | Fixing of specimen is done separately with improved mechanism. It does not require any attachments, the assembly is placed under loading shaft of UTM and contact is made manually. |
| **Gripping of aggregate plates** | The 3 point clamping mechanism explored were not good enough to account for variation in plate diameter. The second problem was gripping of in variations of dia. | Easier to account for larger variation in diameter as well as ability to accommodate imperfect circular plates. |
| **Damage to Aluminium substrate** | Increase chance of plate slip with screwing mechanism, also three-point clamping damaged the aluminium plates. | A thorough clamping mechanism did not damage any plate. |

| Load elongation curve adjustment | Curve correction is needed by plotting a tangent to the initial curve. | No curve correction needed, once manual seating is complete. |
|---|---|---|
| Cavitation | In pull stub tests it is difficult to fully analyse cavitation phenomenon. DTT of this study damaged some of the failure surfaces. | Cavitation was clearly captured during CPOT test and in the result plots. |
| Failed surface | The fail surface can easily get damaged during the testing in DTT devised in this research. | The plates are separated effectively there is no such issue |
| Limitations and further improvement | i. PATTI is designed to test at room temperature. Direct tension test requires UTM temperature control environment.<br>ii. PATTI is reported to be reproducible and other methods require reproducibility.<br>iii. Further studies and improved procedures are required in establishing film thickness. | i. It is relatively easier to develop an integrated temperature control environment due to compression mechanism.<br>ii. Reproducibility studies are required.<br>iii. CPOT require further improved gap assembly and incorporation of other methods. |

453

## 8   Conclusions

455 In this study, a novel test has been devised to evaluate cohesive and adhesive bond strength

456 based on the principle of 'tensile strength evaluation'. The research can be summarized in

457 following conclusions:

458

- CPOT assembly is useful tool in evaluating bond strength (cohesive/adhesive). This method provides a panacea to many problems faced in historical test methods (as summarized in Table 9).

- The gap assembly is useful to achieve required film thickness under use in best laboratory practices. This assembly need further modifications for practicality and repeatability reasons.

- The test method devised in this research provides a useful insight into investigating material behaviour.

- The material response in CPOT is more elaborative in terms of load elongation curve than pull-off stub tests and direct tension test. Thus, this method makes it easier to further understand the phenomenon of fibrillary nucleation in cohesive bond of bitumen and mastics.

Finally, further research is required to evaluate special binders and adhesive properties. The cases of moisture conditioning needs further evaluation. It will be advantageous to carryout correlational analysis by use of other bond strength measurement techniques.

**Conflict of Interest**

None.

**Acknowledgment:**

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