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

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ABSTRACT

The quantification of moisture susceptibility has been a major concern for researchers as it adversely affects the performance of asphalt pavements. Several methods have been developed to assess bond strength using asphalt mixture in loose or compacted state. These tests lack in their ability to study fundamental properties that the bond between bitumen and aggregate. In this context, pull-off stub techniques have been developed such as pull-off stub based tests and direct tension type tests. First group only measures the maximum pull-off strength and second group has problems related to use of consistent binder film thickness and operational problems in test itself.

This paper presents a new test to evaluate bond strength in an attempt to solve problems associated with traditional pull-off techniques. This aim is achieved through review of existing techniques, development of a direct tension test assembly and its evaluation, development of a gap assembly and CPOT assembly. Key parameters for bitumen and mastics were evaluated. The results show promising potential for use of this technique to study cohesive as well as adhesive bond strength of binder.

Keywords: Pull-off test, asphalt, bitumen, adhesion, cohesion, bond strength tensile 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 energy (surface change), molecular orientation, and mechanical adhesion (mechanistic tenacity) (Shute et al. 1989; Bagampadde, Isacsson, and Kiggundu 2004). Kringos et al. (2008) have discussed three modes of moisture infiltration. The first being entry of water in connected macro-pores through rainfall. Secondly, due to stationary moisture in the form of liquid or vapour resides in the macro-pores. Third mode being the presence of water inside aggregate before laying of the wearing course and inadequate drying of aggregate.

One of the earliest test to evaluate properties of bitumen included chew test. In this test, builders tried to assess not only the consistency and but also stickiness of bitumen. Since then several methods have been devised for measuring fracture of interfaces and adhesive joints. In addition to these methods, several approaches has also been used to improve the bond strength of bitumen and aggregate. This includes selection of suitable combination of binder and aggregate, modification of bitumen (Baldi-Sevilla et al. 2017), improvement of mixing techniques, and reduction of dust powder on surface of aggregate. However, there are difficulties associated with these methods to improve bond between bitumen and aggregates using these methods (Peng et al. 2018). In addition to this different antistripping additives such as hydrated lime has been used to improve strength and reduce moisture susceptibility of asphalt mixtures (Huang et al. 2005; Kim, Pinto, and Park 2012; Ameri, Kouchaki, and Roshani 2013; Zaidi 2018).

In discussion of moisture damage in asphalt mixture the tests classified in two categories; tests conducted on loose and compacted mixtures, (Lottman et al. 1974). Several research has summarised these moisture sensitivity tests (Terrel and Shute 1989; Terrel and AI-Swailmi 1994; Airey and Choi 2002; Bagampadde, Isacsson, and Kiggundu 2004; Solaimanian et al. 2003). Test on loose mixture are empirical in nature and rely on visual inspection. On the other hand, test on compacted mixtures are more fundamental in nature (Airey and Choi 2002).

Additionally, several studies has been performed to quantify bond strength based on fracture parameters, surface energy, diffusion coefficients and adhesion (Kim, Pinto, and Park 2012). Hitherto, there is no unified and standard fundamental test method for evaluation of bond strength. (Moraes, Velasquez, and Bahia 2011) (Wang, Yi, and Feng 2014), (Rahim 2017) (Zhou et al. 2018). This is because the amount of research performed to study tensile properties of thin films of bitumen has been relatively little compared to other means of evaluating bitumen properties (Chang 1994).

2 Objectives

This research aims to evaluate pull-off bond strength through development of a better, simple and robust testing mechanism to solve problems associated with traditional pull-off testing techniques. The test method developed to achieve this aim is termed the 'Compression Pull-Off Test (CPOT)'. Following objectives were defined for this research;

- Review of existing bond strength techniques
- Evaluation of direct tension test approach
- Development of mechanism to achieve required binder film thickness
- Development of compression pull-off test (CPOT)
- Evaluation of the key parameters for binder testing and validation of results

3 Review of bond strength methods

There has been numerous research efforts to select most appropriate methods to study moisture sensitivity affected by loss of bond between bitumen and aggregate (Rice 1958; Lottman et al. 1974; Terrel and Shute 1989; Airey and Choi 2002). The traditional methods on loose and compacted mixtures such as Lottman's procedure and its advanced modification i.e. AASHTO T 283) are useful in comparative analysis of moisture suspectibilites of various HMA mixtures. These methods, however, meausres bulk properties of mixtures and lack in focus on fundamental material properties (Bhasin et al. 2006; Canestrari et al. 2010; Cho and Bahia 2010; Taylor, Hamedi, and Nejad 2014). This creates the necessasity to evalute bond strength using component charactersitics tests which measure the fundamental properties. Additionally, testing based on components characteristics is generally more economical (Kim, Pinto, and Park 2012). A fundamental test on binder and aggregate will give better understanding of moisture sensitivity and its effect on the cohesive and adhesive bond of bitumen and aggregateds (Moraes, Velasquez, and Bahia 2011).

In a study, Jakarni (2012) has summarized some of the common tests used in science and technology of adhesion to measure the adhesive bond strength of coatings of the composite materials. These tests included peel test, pull-off test, double cantilever beam (DCB) test, tapered double cantilever beam (TDCB) test, impact wedge peel (IWP) test and scratching of thin films test. The review of experience in adhesive technology lead Jakarni (2012) to formulate a new pull-off testing technique to evaluate the bond strength.

In binder research, peel test has been used to quantify the adhesive strength (Blackman et al. 2013; Zhang et al. 2017; Zhou et al. 2018). A flexible thin peel arm is adhered on a substrate with the use of adhesive material. A pull load is applied through peel arm at a constant speed and specific angle and force to initiate and propagate peel fracture is measured. This peel force is recorded as a function of the displacement to calculate fracture energy. In these studies, it has been demonstrated that that the peel test is a suitable method to determine the adhesive fracture energy. In addition to this, there are numerous studies which emphasis the importance of thin film binder to evaluate its response to pull-off loading (Kanitpong and Bahia 2005; Jakarni 2012; A Copeland et al. 2006; Audrey Copeland, Youtcheff, and Shenoy 2007; Poulikakos and Parti 2011; Alvarez, Ovalles, and Caro 2012; Harvey and Cebon 2003; Apeagyei, Grenfell, and Airey 2014; Sultana 2014; Al-Haddad and Al-Khalid 2015; Rahim 2017; Abd, Al-Khalid, and Akhtar 2018; Chaturabong and Bahia 2018)(Audrey Copeland, Youtcheff, and Shenoy 2007).

A commonly used pull-off stub type test is Pneumatic Adhesion Tensile Testing Instrument (PATTI) to assess the moisture damage based on bond strength. Copeland (2007)modified the procedure samples in PATTI to improve the control of the bitumen film thickness. (Santagata et al. 2009) has also modified PATTI and reported on reliability and practicality to evaluate the adhesion/cohesion properties of asphalt-aggregate system. One adaptability of PATTI is Bitumen Bond Strength test (BBS) to evaluate pull-off strength (Kringos, N., Scarpas, A., and de Bondt 2008; Moraes, Velasquez, and Bahia 2011; Zhou et al. 2018). BBS results are reported to be reliable, repeatable and reproducible (Canestrari et al. 2010; Moraes, Velasquez, and Bahia 2011; Chaturabong and Bahia 2018; Mohammed et al. 2018). However, pull-off stub based methods only measures the maximum pull-off strength and reports on description of coating fracture. This pull-off stub approach has its limitation in a sense that rate of loading (in terms of displacement) cannot be controlled (Zhang et al. 2017). In contrast another approach, Direct Tension Test (DTT) methods have their advantage in measuring the pull-off load and elongation (Rahim 2017; Abd, Al-Khalid, and Akhtar 2018).

There are several direct tension based studies to evaluate bond strength (Harvey and Cebon 2005; Jakarni 2012; Sultana 2014; Abd, Al-Khalid, and Akhtar 2018). In work with direct tension tests, researchers have reported difficulties in preparing aggregate samples, achieving consistent film thickness and perform tests itself (Abd, Al-Khalid, and Akhtar 2018). In DTT, epoxy resin adhesive has been used to fix the aggregate plates with testing fixture (Peng et al. 2018). The use of these adhesive materials result in a slow process and require removal of excessive adhesive from around the fixture. Another approach is to fix the aggregate plates in testing moulds using screws. This fastening mechanism has operational problems associated to it. The control of film thickness in specimens have been another concern, different approaches and gap assemblies have been used (Jakarni 2012) . The use of Dynamic Shear Rheometer for control of bitumen film at submicron level has also been reported (Zhang et al. 2017; Al-Haddad and Al-Khalid 2015).

Surface Free Energy (SFE) is another useful technique to evaluate adhesion between aggregate and binder. (Kringos, N., Scarpas, A., and de Bondt 2008) used surface energy approach and combined direct tension test approach with numerical moisture diffusion analyses to demonstrate that results consistent with expected field performance. The works highlights the importance of importance of mechanical tests to assess bond strength. In addition, there has been several attempts to correlate bond energy (from SFE) and total work of fracture (from pull-off tests). The problem with SFE is that it is a thermodynamics based approach that do not take into account energy dissipation during loading and unloading. Moreover, total work of fracture (pull-off strength) is dependent on test geometry and testing conditions (Howson 2011).

The review of pull off techniques concludes with an importance of direct tension based approach to evaluate the bond strength. There is, however, need to address the difficulties associated with this test approach. Any such attempt must be effective to measure maximum bond strength, rate of deformations corresponding to pull-off load and availability of fracture surface to examine.

4 Test materials

The conventional properties of bitumen for this research are listed in Table 1. The work included use of aluminium plates (35.1 mm diameter) as a control. This helped to ensure substrate plates were parallel to achieve a uniform film thickness. The control material was also convenient to use because of its high thermal conductivity to quickly cool down aluminium-binder system. This ensured ready availability of gap assemble to prepare multiple specimen in shorter interval of time. In addition, limestone (Ls) and granite (Gr) fillers (passing sieve 63 micron) were used to prepare mastics. The percentage of fillers was added as 40% by mass of bitumen. These mastics were prepared by gradually mixing fillers in an oven heated bitumen over a hot plate. In addition to these materials, limestone plates were used to validate the results of CPOT.

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

5 Test methods

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

4.1 Development of gap assembly for film thickness

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

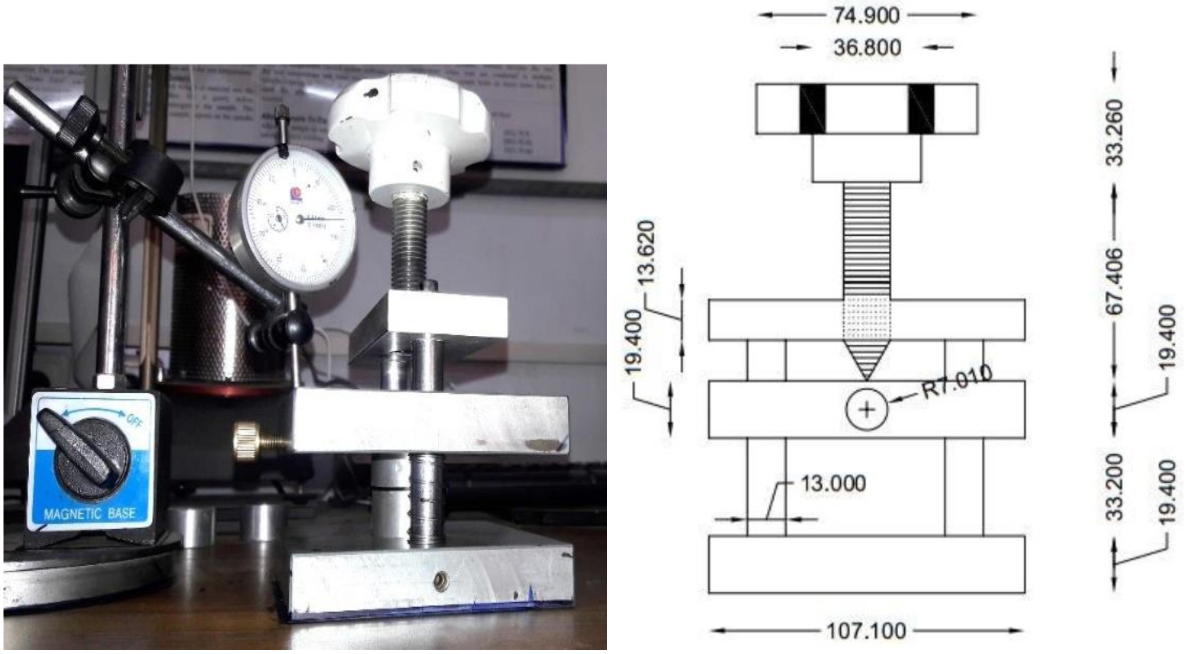


Figure 1: The compression gap assembly

In specimen preparation, hot bitumen was poured onto an aluminium plate. A second plate was placed onto first plate to create a sandwiched specimen. A preconditioning of these plates at 110 °C before pouring was identified to be a best practice. However, in case of plates conditioned at room a slight oven heating of sandwiched specimen was needed. The preheating before pouring helped to overcome hydrogen bond, p-p bonding and Van der Waals forces. The presences of these forces was expected to reduce the wetting of the substrate plates (Bagampadde et al., 2004). The sandwiched specimens were then compressed in gap assembly to achieve required thickness. After curing in gap assembly at room temperature, specimens were removed and left for additional curing/conditioning at room temperature for 24 hours. After conditioning period specimens were ready to test with direct tension test and CPOT.

4.2 Direct tension test assembly

In order to test contemporary direct tensile test (displacement controlled) approach, a direct tension assembly was fabricated. The modifications were made on the basis of joining mechanism to connect with a 10 kN capacity Universal Testing Machine (UTM). The test assembly is shown in Figure 2 (left). In tension testing, sandwiched aluminium plates were gripped in the upper and lower moulds with three under-head screws. The top and bottom extended arms of the moulds were fixed in the saw tooth grip of UTM as shown in Figure 2 (right). The testing was performed after adjusting the test parameters in software.

Figure 2: Direct pull-off testing assembly

In view of operational problems associated with DTT and variability of its result (section 5), a better mechanism for testing was needed. The need for following improvements were identified.

- The three-point grip (3 under-heads) was deemed insufficient in direct tension test. During testing, aluminium plates were getting damaged due to drag and slip of plates. A thorough gripping mechanism was needed.
- The surfaces of tested specimens were getting damaged, creating problem in analysing the cohesive failure. This also added problem in observing phenomenon of cavitation.

- A consideration of authors’ experience with pull off created need to conceptualise a better testing mechanism rather than adding further modifications to existing moulds.

4.3 Compression pull-off test assembly (CPOT)

The rationale of this test was derived from traditional compression tests performed on concrete and asphalt mixtures. The basic principle was thus formulated as “the load applied in compression of the UTM should generate a pull-off load on the specimen”. A conceptual diagram was prepared to translate this novel idea into reality (Figure 3, left). The idea was found to be feasible in terms of manufacturability. The assembly was designed and manufactured locally with ‘lean manufacturing’. This philosophy called for developing the product to a minimum functional level then improving the design according to needs (Shah and Ward 2003).

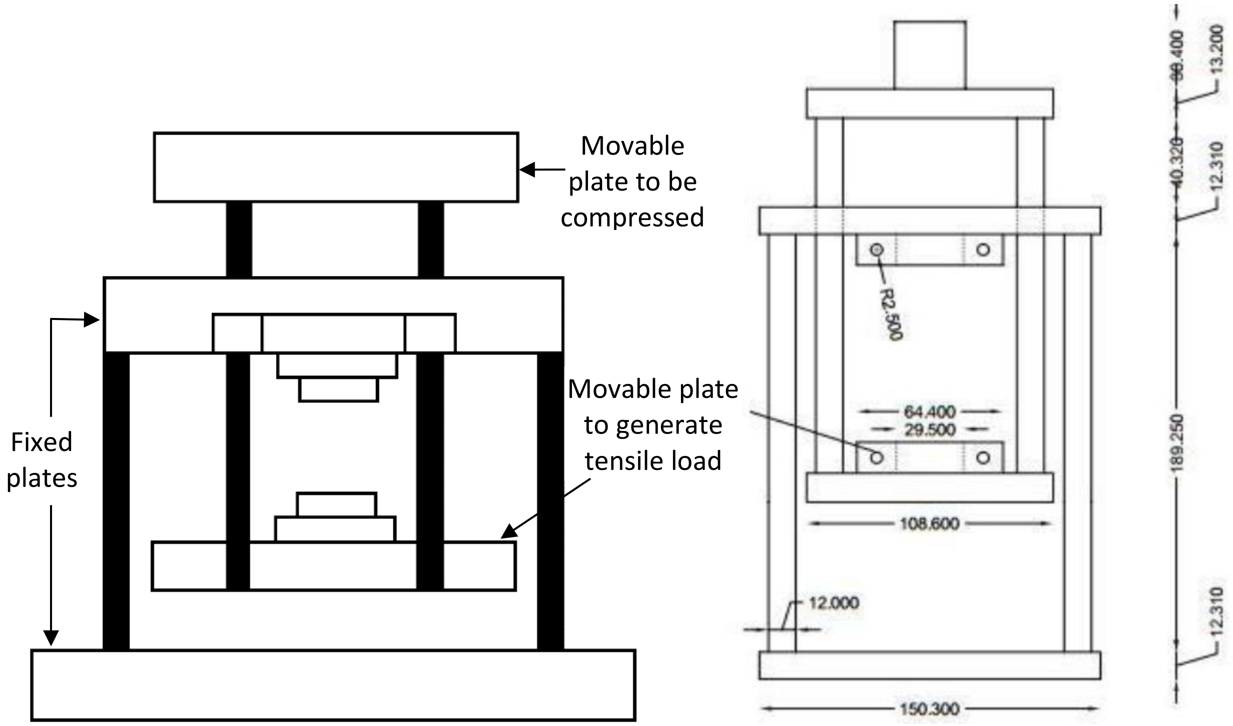


Figure 3: Conceptual diagram of compression device for pull-off testing

The Figure 4 (left) shows the first complete operational assembly. The sandwiched specimens were fixed by means of two horizontal sliding plates. Circular groves in these plates to thoroughly gripped the substrates. The screws in assembly were tighten to firmly squeeze the substrate. This approach added a simplicity to design and ease of operation. Figure 4 (right), shows the three further design improvements which were added as per need of trial testing. In first modification two springs were added around the bars in upper moveable part to prevent it from striking the fix part. In second modification, a shaft was introduced to movable part for ease of centring and alignment. Thirdly, a half spheroid was introduced on the loading shaft for the seating adjustments. A hole in the base of fix part was introduced for alignment purposes. This assembly was named as 'Compression Pull-Off Test (CPOT) assembly'. This signified that this assembly was able to take a compression load from an external source and translate it to a uniform co-axial pull-off load on a binder-substrate system.

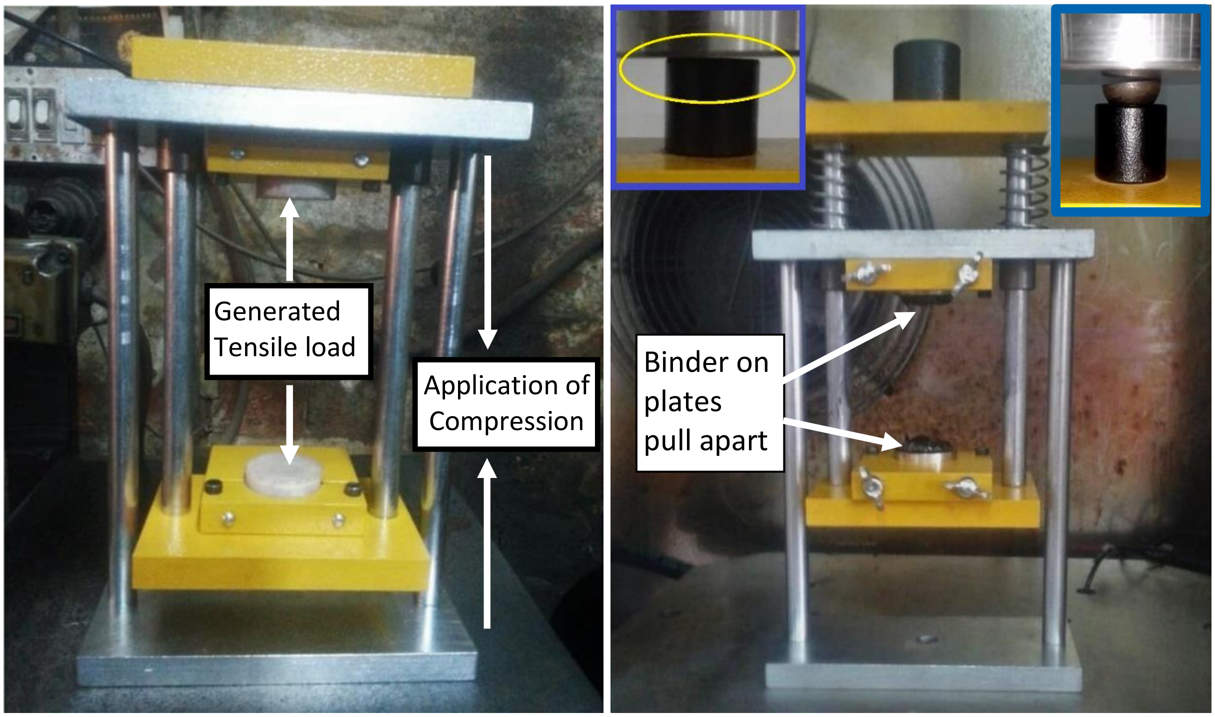


Figure 4: First prototype ad final design of CPOT assembly

The specimens prepared from gap assembly were inserted in the upper plate and tightened with hand pressure. Lower plate was raised to grip the bottom aluminium substrate. After fixing both ends of specimen, a slight pressure was applied with a small wrench to ensure firm grip. It is important to mention that in entire testing phase no specimen broke during installing, fixing, handling, and testing of assembly. This griping mechanism seemed to have better tolerance towards even slightly misaligned plates of sandwiched specimen.

In testing phase, the test parameters were defined in terms of loading direction, loading rate (mm/min), specimen dimension and elongation limits etc. After this, a small seating load (few Newtons) was applied to ensure proper contact. In some cases, specimens were discarded due to application of accidental load application (higher than 100N). The final load was applied in strain control mode and results were obtained in terms of load and elongation.

5 Results and discussion

The results were analysed in terms of mean pull-off strength (POS) and coefficient of various (COV) for at least five replicates. Maximum pull-off strength of a specimen, expressed in MPa, was calculated by dividing failure load in (F_n) by area of contact at surface i.e. $\mathrm{POS} = 4\frac{\mathrm{F_n}}{\pi \mathrm{D}^2}$, where F_n is expressed in Newtons and mean diameter of substrate 'D' is expressed in (mm). The acceptance of and rejection were based on observation as per some guidelines of ASTM: D4541-17. The pull-off strength parameters evaluated for materials, instrumentation and key test parameters.

5.1 Repeatability and operator variability of gap assembly

The accuracy of film thickness was checked with Vernier caliper for 36 specimens before and after pouring of binder. These specimens were grouped according to plate condition. In cold plate case, each specimen was prepared by pouring hot binder onto a clean control plate, which was preconditioned at room temperature. This plate condition was labelled as ‘cold’. The specimens in cold condition required slight heating in oven before introducing to gap assembly. In hot plate case, specimens were prepared by pouring hot bitumen onto preheated aluminium plates at 110 °C. This plate condition was labelled as ‘hot’. In comparison on method of pouring (Table 2), a good repeatability was achieved for pouring on hot aluminium plates. The operator 1 (experienced) achieved more repeatable results as compared to operator 2 with same method of pouring. This shows that the repeatability is dependent on method of specimen preparation and operator’s understanding of best laboratory practices.

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

In next stage, this effect of repeatability on these specimen was further evaluated with CPOT. Tests were performed with; 10mm/min rate of loading, 0.5mm film thickness, 15 hour dry/wet conditioning on untrimmed specimen. Two specimen were discarded for operator 1 in each case and six specimen for operator 2 were used for additional conditioning, the result of these specimens therefore are not included. The mean POS and COV values for these film thicknesses are shown in Table 3 to evaluate for repeatability.

Table 3: CPOT results for repeatability of gap assembly

Binder	POS	COV	Test condition	Replicates
Short term aged binder	1.17	13.54	Operator 1, dry 15 hours, cold	9
	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).

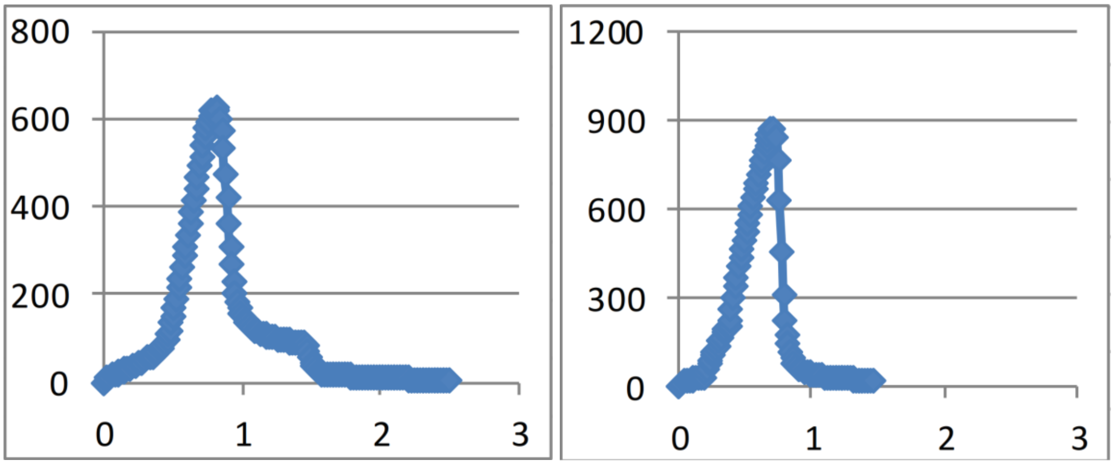


Figure 5: Brittle and ductile type failure using DTT

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 decreased by 20.6% only by changing the method of pouring. Silicon method of pouring gave lower POS with increased variability. The unaged binder showed only a 9.1% decrease of binder in bond strength. This difference of can be attributed to trimming and ageing. The aged binder shows increased bond strength with shift from ductile to brittle failure. The effects of silicon pouring and trimming seems to be contradictory with Dynamic Shear Rheometer testing in which former is a compulsory step and second is an alternative to direct pouring. This is attributed to different nature of two tests for quantification of tensile strength (pull-off strength) and shear strength respectively. In addition to this, different studies have used trimming of specimen in pull off testing (Sultana 2014; Al-Haddad and Al-Khalid 2015; Apeagyei, Grenfell, and Airey 2015; Abd, Al-Khalid, and Akhtar 2018). This study suggests effect of trimming on the results of pull-off strength. The coefficient of variability in all test groups indicate a good repeatability of CPOT results.

Table 5: Tests to check repeatability of the test method

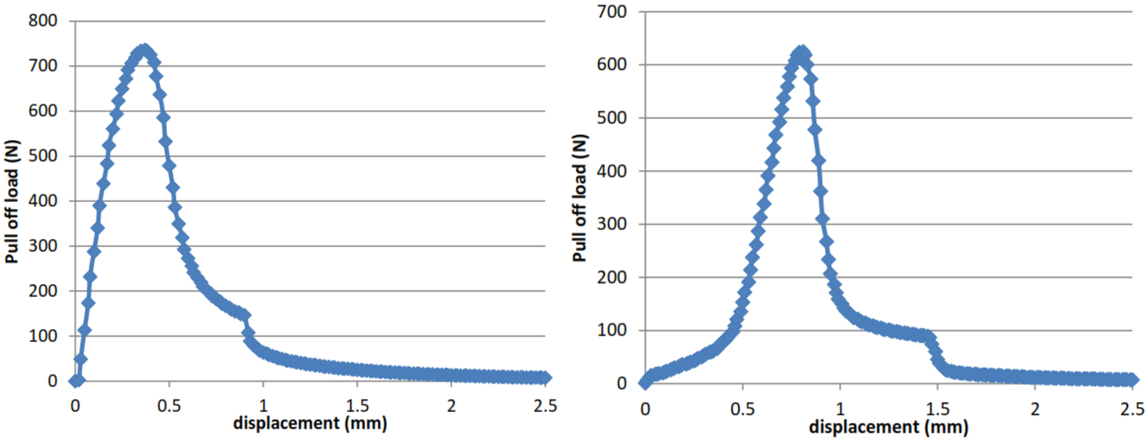
Round	Material	Pull-Off Strength (MPa)	COV (%)	Variable Testing conditions	Replicates
Round 1	Source 2 Pen 60/70	0.70	9.67	Silicon, tr	5
		0.77	6.82	Direct, un	8
Round 2	3-hour oven aged Source 1 Pen 40/50	1.4	13.35	Silicon, tr	9
		1.45	11.04	Direct, un	8
Round 3	RTFO aged Source 1 Pen 60/70	1.00	11.63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

In Table effect of decrease in film thickness have resulted in increase of pull-off bond strength. The mode of failure in case of 0.5mm film thickness was cohesive while for 0.3mm film thickness it changed from hybrid to adhesive. This is consistent with findings in literature on the effect of film thickness on pull-off bond strength (Marek and Herrin 1968; Chang 1994; Fond 2001; Harvey and Cebon 2003, 2005; Poulikakos and Parti 2011; Jakarni 2012; Sultana 2014; Abd, Al-Khalid, and Akhtar 2018).

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

The typical failure curves in ductile mode are presented in Figure 6 for comparison of DTT and CPOT. Firstly in both tests, a deviation from this is attributed to an anomaly in test specimens and test itself. These problem could arise due to issue in specimen preparation, curing/conditioning, non-uniform film thickness, non-homogenous mixing of filler particles and mainly slipping of plates during test. The load elongation curve is advantageous to both method in comparison to pull-off stub tests. In comparison to CPOT, direct tension test curve before peak load is different due to seating adjustments. The CPOT has already taken care of major seating adjustment during manual adjustment prior to start of test. The CPOT curve can be directly analysed while DTT curve need normalisation to study energy dissipation. Hitherto, authors have found no discussion in binder studies dealing with advantage due to compression approach. CPOT is useful in study of load elongation curves as compared to contemporary 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 40% in Source 1 Pen 40/50	1.38	11.53	Dry	New	6
	0.96	33.31	Dry	Used	5
	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

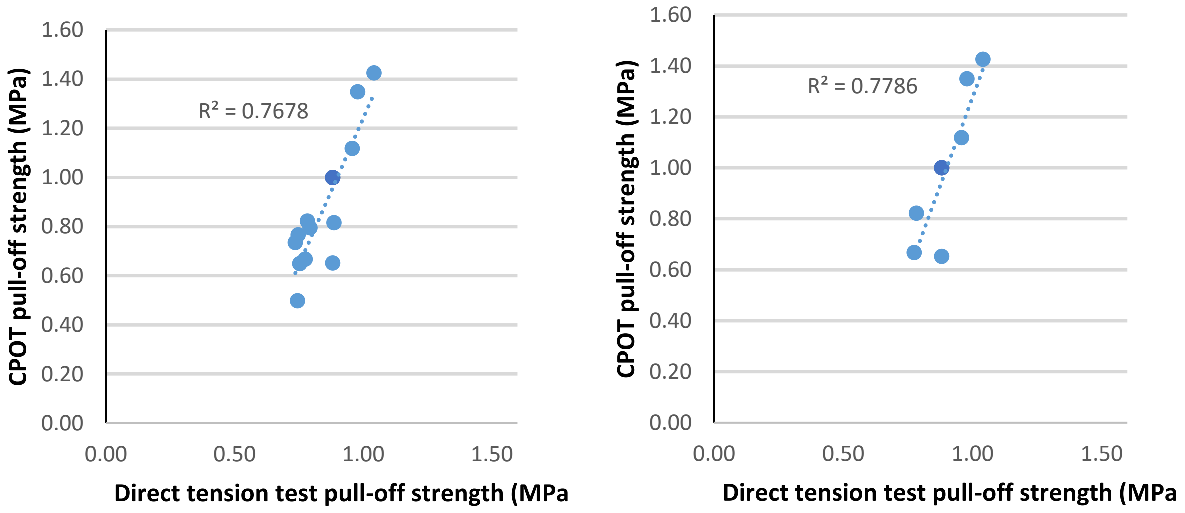


Figure 7: Correlational analysis of CPOT and DTT

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.

452 **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. ii. PATTI is reported to be reproducible and other methods require reproducibility. 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. ii. Reproducibility studies are required. iii. CPOT require further improved gap assembly and incorporation of other methods.

8 Conclusions

In this study, a novel test has been devised to evaluate cohesive and adhesive bond strength based on the principle of ‘tensile strength evaluation’. The research can be summarized in following conclusions:

- 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.

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