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Oil-Sludge Extended Asphalt Mastic Filled with Heavy Oil Fly Ash and Cement Waste for Waterproofing

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Abstract: Recycling as an economic disposal process for many hazardous waste materials has become a popular means of conserving our planet's scarce and diminishing natural resources. This paper is a study of the influence of oil sludge (OS) on the physical behavior and performance of asphalt filled with heavy oil fly ash (HOFA), cement kiln dust (CKD) and limestone dust (LMD). Conventional asphalt consistency tests in addition to a new bond strength (BS) test were conducted on the modified asphalt mastics. The results were statistically analyzed and assessed in accordance with American Society for Testing and Materials (ASTM) D 332 and ASTM D 449 specifications. Too much OS resulted in strength deterioration of the asphalt mastic, which can be compensated for by filling the mastic with HOFA. OS interacts constructively with the fillers to improve their effectiveness in raising the softening point (SP) and viscosity of the asphalt, and also in reducing its penetration and ductility. Even though sludge mastics hold promise as suitable composites for damp proofing and waterproofing, the resulting low flash point (FP) and SP of some of these mastics make their suitability for roofing applications questionable.

Keywords*:* Oil sludge, Oil fly ash, Asphalt mastic, Bond strength, Waterproofing, Roofing.

رواسب الزيت في ماستيك الأسفلت الممدد المملوء بالرماد المتطاير للزيت الثقيل لعزل المياه

حمد إبراهيم العبد الوهاب و محمد أبوبكر دلهات

الملخص: أصبحت إعادة التدوير عملية اقتصادية شائعة للتخلص من نفايات المواد الخطرة للحفاظ على قلة الموارد الطبيعية العالمية وندرتها. في هذه المقالة تمت دراسة تأثير رواسب الزيت على الخصائص الفيزيائية و الأداء للأسفلت المملوء بالرماد المتطاير للزيت الثقيل وغبار فرن الأسمنت وغبار الحجر الجير، بإجراء اختبارات الأسفلت التقليدية بالإضافة إلى اختبار جديد لقوة التماسك على أسفلت الماستيك المعدل. تم تحليل النتائج إحصائياً وتقييمها طبقاً للمواصفات ASTMD 332 و ASTM D 449. تؤدي كثير من رواسب الزيت إلى تدهور قوة أسفلت الماستيك الذي يمكن تعويضه بملء الماستيك بمادة HOFA. تتفاعل رواسب الزيت بالبناء مع الحشو لتحسين فعاليتها في رفع نقطة التليين و اللزوجة للأسفلت و أيضاً للحد من التغلغل و التلين. على الرغم من أن رواسب الماستيك مبشرة لمركبات مناسبة لعزل الرطوبة و المياه إلا أن نتائج نقطة الوميض والتليين لبعض أنواع هذا الماستيك تظهر أسئلة لمدى إمكانية استخدامها في تطبيقات التسقيف.

مفاتيح الكلمات: رواسب الزيت، رماد الزيت المتطاير، ماستيك الأسفلت وقوة الروابط ، عزل المياه، التسقيف.

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1. Introduction

The rapid growth in the asphalt waterproofing material market is creating more demand for asphalt. A 4.1% continuous annual increase in asphalt consumption is forecasted for the coming years (World Asphalt 2012). Nearly 15% of the global asphalt demand is generated from the roofing sector, and this demand rises annually (World Asphalt 2010). High demand, coupled with a limited supply of asphalt, has resulted in a continuous rise in cost. This fact, together with the need to conserve our scarce natural resources, calls for the exploration of a cheap local material supplement. In Kingdom of Saudi Arabia (KSA), more than 35,000 m^3 of tank bottom oil sludge (OS) is produced from crude oil storage tanks on a yearly basis. Additionally, about 340,000 m^3 of heavy oil fly ash (HOFA) waste is generated annually, along with more than 12 megatons of combined cement kiln dust (CKD) and limestone dust (LMD).

Air-blown asphalt is the main conventional bituminous component of most asphalt-based roofing and waterproofing products. But as roofing chemistry has become more sophisticated, various formulations with different viscosity ranges and physical and mechanical properties (for horizontal and vertical applications) have been developed for specific uses from regular asphalt/bitumen through chemical and mineralogical content modifications such as pourable sealers (pitch pocket mastics) and elastomeric sealants (mastics for high movement joints and terminations). This is due to the fact that the improvements achieved on the bituminous materials' durability and extensibility (especially at lower temperatures) by adding polymers and other additives will result in a modified material with superior physical properties, which surpasses alternative materials but costs the same amount (Paul 2002).

Many published works have explored roofing asphalt polymer modifications (Lucke 1989; Terry *et al*. 1999; Singh *et al*. 2002; Martin-Alfonso *et al*. 2008 a and b; Fang *et al*. 2009), and sufficient insight into the subject has been established. But besides polymers, mineral fillers are another major additive that can also dictate the behavior of waterproofing asphalt mastic. Studies have been carried out to investigate the effect of various mineral fillers on certain asphalt binders' properties (Johansson and Isacsson 1998; Recasens *et al*. 2005; Chen *et al*. 2008), but most of these works were directed towards the behavior and performance of asphalt concrete (AC) pavement and not roofing or waterproofing applications.

OS is waste that accumulates at the bottom of crude oil storage tanks or separation vessels. It was previously used as an asphalt extender by combining comminuted refinery OS with liquid asphalt that is either hot asphalt or an asphalt emulsion (Eric 2006). The resulting composition is similar in performance to Trinidad Lake asphalt and can be used as a binder for asphalt-aggregate compositions. Some of the mastic formulation was found to comply with ASTM D 1227 Type III roof coatings' specifications. The tank bottom sludge was also utilized as a sole binder for a moderate traffic paved and unpaved road mix design (Taha *et al*. 2007). Hot mixing yields a concrete with about 12 kN stability, satisfying the requirements for low (3.3 kN) or medium (5.3 kN) trafficked surfaces or base layers according to Asphalt Institute specifications.

Taha *et al*. (2001) examined the environmental and structural impact of OS as a substitute in asphalt paving mixtures. Experimental results indicated that mixtures containing up to 22% OS could meet the necessary criteria for a specific AC-wearing course or bituminous base course. Results from the chemical analysis of leachates from a 50% OS-containing mixture indicated an insignificant concentration level of toxic metals. In another environmentally-friendly disposal technique study of tank bottom OS, Al-Futaisi *et al*. found that the toxicity characteristics leaching procedure (TCLP) results show that no extracts exceeded the established TCLP maximum limits set by the US Environmental Protection Agency (EPA). The naturally occurring radioactive minerals (NORM) activity values obtained from sludge road mix and solidified sludge mixtures were found to be very low when compared to NORM standard values of 100 Bq/g; thus, it was concluded that the OS for this application was non-hazardous. In previous similar research by Veenstra *et al*. (2000), the findings were similar: none of the road base samples was determined to be hazardous according to TCLP analyses, but leachates from all the samples were toxic according to the 48-hour acute toxicity test. However, none of the samples had unacceptable levels of NORM. There was no disagreement whatsoever among these studies, but the characteristics of the sludge from different oil sources differed in their constituent proportions.

Heavy oil fly ash (HOFA) is a byproduct of the heavy fuel (diesel, cracked fuel, etc.) combustion process. HOFA is recycled for a wide variety of purposes, and its potential as a filler reinforcement in low density polyethylene (LDPE) polymer composites has been reported by Khan *et al*. (2011). Their results show enhancement in rheological properties of the modified LDPE. The use of HOFA to improve the performance of an asphalt binder and concrete was patented by Al-Methel *et al*. (2011). HOFA-asphalt mixes satisfy the asphalt performance grade limits with potential improvement in durability and strength.

Traditional asphalt physical tests such as the ductility test (ASTM D 113), penetration test (ASTM D 5), SP test (ASTM D 36), and flash and fire point test (ASTM D 92) still form the basis for general specifications in, among others, roofing asphalt cements, asphalt for waterproofing and damp proofing applications (ASTM D 312 2006; ASTM D 449 2008). There is a lack of adequate standard testing to assess and characterize appropriately the asphalt mastic on its own at the semi-finished product level for this application (Goikoetxeaundia *et al*. 2007). In this study, a new mastic bond strength (BS) test for assessing the performance of asphalt mastic was devised.

The objective of this research was to establish the effect of OS on the physical behavior and tensile bond strength (BS) of roofing asphalt filled with mineral fillers. Both the influence of OS on the SP, ductility, penetration, flash point and viscosity of asphalt filled with HOFA, CKD and LMD and the tensile BS of the resulting asphalt mastic were examined.

2. Materials and Methods

2.1 Materials

The asphalt used in this study, obtained from Riyadh's refinery, is the only locally available grade. It is a 60/70 penetration grade asphalt, with a 64-10 performance grade. The physical properties of the asphalt are shown in Table 1.

Table 1. Asphalt physical properties.

Property	Magnitude
Ductility (cm)	150 +
Penetration (dmm)	67.2
Softening point (°C)	52
Flash point (°C)	342
Viscosity (cP)	575

The OS was obtained from Rastanura crude storage tanks of the Saudi Arabian Oil Company (Aramco). The metal and sediment composition of the sludge is given in Table 2. The OS contained about 70% water, 22% sediments and 8% hydrocarbon oils. Water was separated by the centrifuge method (ASTM D 4007). The dehydrated sludge was subjected to analyses for sulfur (ASTM D 4294), metals (sulfur, nickel and vanadium; ASTM D 6481) and sediments (calcium oxide [CaO], iron rust [Fe_2O_3] and copper oxide [CuO]; ASTM D 6481). The dehydrated OS was also extracted with carbon disulfide (CS_2) for oil fraction and subjected to high temperature distillation (SIM; ASTM D 6352) to obtain the fuel oil fractions.

Table 2. Metal and sediment composition of oil-sludge.

Contents	Method	Unit	Amount
Sulfur	ASTM D-4294	Wt. %	1.4
Nickel	ASTM D-6481	ppm	5694
Vanadium	ASTM D-6481	ppm	38
CaO	ASTM D-6481	Wt. %	11
Fe_2O_3	ASTM D-6481	Wt. %	4.9
CuO	ASTM D-6481	Wt. %	5.73

Cement kiln dust (CKD) and limestone dust (LMD), which are by-products of limestone quarrying and cement manufacturing, were obtained from a local road construction company. HOFA was collected from Rabiq Thermal Power Station located on the western cost of the KSA, along the Red Sea.

Table 3. Oil-sludge-filler mastic experimental design.

Generalized mastic formulation[a]					
Binder Type	% filler (HOFA/CKD/LMD)				
	0 %	10%	15%	20%	25%
0% - Sludge	Plain Asphalt	200g _ asphalt + 20g _ filler	200g _ asphalt + 30g _ filler	200g _ asphalt + 40g _ filler	200g _ asphalt + 50g _ filler
20% - Sludge	160g _ asphalt + 40g _ sludge + 0g _ filler	160g _ asphalt + 40g _ sludge + 20g _ filler	160g _ asphalt + 40g _ sludge + 30g _ filler	160g _ asphalt + 40g _ sludge + 40g _ filler	160g _ asphalt + 40g _ sludge + 50g _ filler
40% - Sludge	120g _ asphalt + 80g _ sludge + 0g _ filler	120g _ asphalt + 80g _ sludge + 20g _ filler	120g _ asphalt + 80g _ sludge + 30g _ filler	120g _ asphalt + 80g _ sludge + 40g _ filler	120g _ asphalt + 80g _ sludge + 50g _ filler

[a] Note that, the sludge composition is by asphalt weight, and the filler composition is by asphalt weight in the case of the plain asphalt or by sludge modified asphalt weight in the case of sludge-filler-asphalt blends.

2.2 *Sample Preparation*

The raw OS was first dried in an oven at 70 °C for 48 hours. This temperature was selected to prevent the volatilization of the sludge's oily constituents; hence, the required slow water evaporation. The filler materials were also placed in an oven for 24 hours at 100–105 °C prior to mixing in order to eliminate the absorbed moisture. Neat asphalt (800 g) was poured into a 1000 ml mixing can, which was then placed in an oil bath at 140 °C. The asphalt was continuously stirred with the aid of a high speed shear mixer (1500 rpm) until thermal equilibrium between the bath and the container had been established. An appropriate amount of OS (20% and 40%), determined by the asphalt's weight, was introduced into the asphalt, and the stirring continued for 10 minutes. Afterwards, 200 g of the final mix was poured into four new mixing cans and stored temporarily inside the oven at 140 °C. The four blends were then mixed in the same manner as previously mentioned with appropriate filler content (10%, 15%, 20% and 25%) for five minutes. Test samples were cast immediately for each blend to avoid a prolonged storage additive settlement or separation. The generalized mastic composition matrix is shown in Table 3. The styrene butadiene styrene (SBS) polymer was mixed for 20 minutes at 200 °C, subsequent to the warm soaking of the SBS powder in the asphalt at 160 °C for two hours.

2.3 *Properties Measurement*

Conventional asphalt physical tests such as the ductility test (ASTM D 113), penetration test (ASTM D 5), SP test (ASTM D 36), flash and fire point test (ASTM D 92) and an additional viscosity test (ASTM D 4402) were first conducted on the samples. Then, selective blends were evaluated using the BS test, which was developed in this study to assess the tensile BS of asphalt mastics to smooth surfaces.

2.4 *Bond Strength Test*

The BS test was devised to measure the asphalt mastic ability to resist tensile force and to transfer a sizable amount of force between two bonded surfaces. It is also an indicator of the maximum BS the mastic can surmount when subjected to tension. The test involves loading a 30 mm x 20 mm x 6 mm prepared sample of the asphalt mastic in tension at a rate of 1.3 mm/minute at 25 °C (ASTM D 545-08, 2008). The load magnitude and its corresponding deformation are measured in the process. The tensile strength is reported as the maximum stress recorded, which is obtained by dividing the highest load carried by the sample before it fails by the plate area.

The current active specification test method for asphalt base expansion joint filler (ASTM D 545-08: Standard test methods for preformed expansion joint filler for concrete construction) prescribed the means of assessing the suitability of joint sealant performance and durability through various tests, including the failure due to compression test. However, it failed to include the tensile failure test. The fact is that joint openings are bound to widen during winter just as they are likely to narrow in the summer season, since concrete does not only expand but also contracts; therefore; an additional tensile test is relevant.

Another major common defect exhibited by an asphalt waterproofing membrane used in a flat roofing system is the formation of blisters (Finch et al. 2010). Blisters propagate more easily after the initial bond loss between the membrane and the roofing deck if the membrane is lacking in tensile strength. A membrane with low tensile strength that unsticks will certainly swell and bulge at the slightest opportunity and, as a result, facilitates failure of the whole process.

2.4.1 Apparatus

The apparatus consisted of two 30 mm x 20 mm x 6 mm plates. There was a mechanism that held and stretched the sample while the load was applied [Figure 1] and a hydraulic, or screwed-up, device. A sample grip having a wedge-like edge slot matching the size and shape of the plate was fixed to the load mechanism's main frame upper block with the aid of a short steel rod that was supplemented with a spring bearing to help eliminate any unnecessary compressive force while the sample was being inserted. The upper part of the mechanism rested on a bearing or spring suspension system which eliminated any additional load on the tested sample due to the self-weight of the upper frame.

2.4.2 Sample Casting

The two plates were preheated, spaced 6 mm apart and fixed in position with the aid of a holder; three sides of the arrangement were wrapped with non-sticking paper. The mastic was heated to a workable state capable of filling the 6 mm × 20 mm × 30 mm space without voids and also to promote sticking to the plate wall with full strength. The material was allowed to cool sufficiently before unwrapping it (≥15 minutes). When necessary, the sample was put in a freezer after 30 minutes for 5 minutes to enable the smooth removal of the non-stick paper without disturbing the sample. Then the sample was put in a 25 °C water bathe for at least 90 minutes before testing.

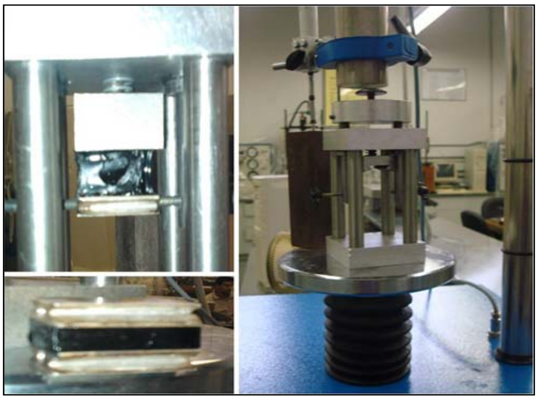

Figure 1. Bond Strength Test Setup.

3. Results and Discussion

3.1 Preliminaries

The OS increased the penetration value of the asphalt at doses above 10%. An average uniform increase of about 20 dmm for every 20% increase in OS could be observed (Fig. 2), while when the content was below 10%, the penetration tended to decline. This can be attributed to the critical filler/additive content that will reverse the original asphalt matrix, when the filler particles interlock within the asphalt network (asphalt continuous phase), to asphalt patches dispersed within the mesh of the OS additive.

Asphalt's SP drops with an increase in the amount of OS for quantities above 15% by weight of the asphalt, and rises below 10% content (Fig. 2). The OS acts as a filler when the content is below 10% and as a flux when it is

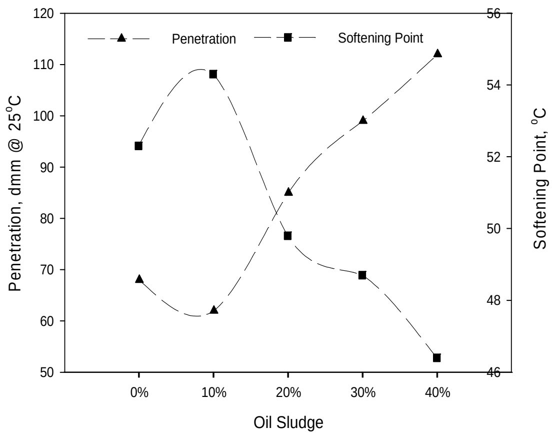

Figure 2. Penetration and Softening Point Vs. Oil-Sludge.

above 10%. This can also be confirmed from the viscosity plot of the asphalt (Fig. 3). Initially, the solid constituents of the OS, which function as fillers, dictate the OS's role at lower quantities, but as the percentage of the OS increases, its waxy component reaches significant levels and takes charge of the OS in a dominant role. At sufficient levels, the dissolved wax from OS lubricates the composite interlayer surfaces, causing a drop in viscosity. Thus, 20% and 40% OS contents were selected for its role as an asphalt-extending material.

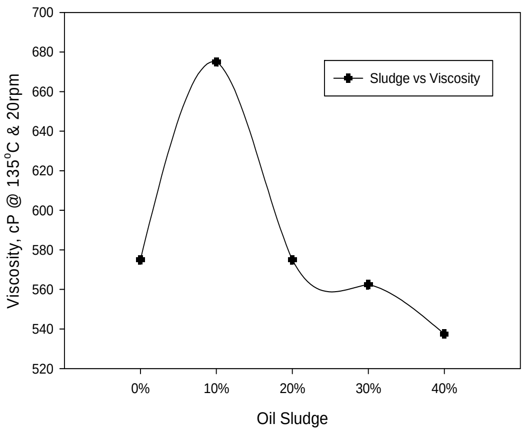

Figure 3. Viscosity Vs. Oil-Sludge.

3.2 *Softening Point (SP)*

A measure of the temperature at which the material will begin to flow (or soften), the SP gives an upper temperature range within which the asphalt mastic can function stably. HOFA raised the SP value appreciably but became less effective when combined with sludge when the HOFA content was 15% or below (Fig. 4). However, there was a constructive interaction between the two substances when the HOFA content was 20% or above, which yielded higher SP values than those of the HOFA-only blends. Each 20% increase in OS caused a decrease of about 3 °C in the SP value for sludge-only blends, but combining 25% HOFA with 40% OS yielded a material composite having about a 60% higher SP than the neat asphalt.

The CKD and OS demonstrated a similar constructive interaction when combined within the asphalt medium, with a positive effect on the SP-value, but it was not as significant as in the case of HOFA-OS blends. Figure 4 shows that there was just a 7 °C increase in SP for 25% CKD and 40% OS blend compared to pure asphalt.

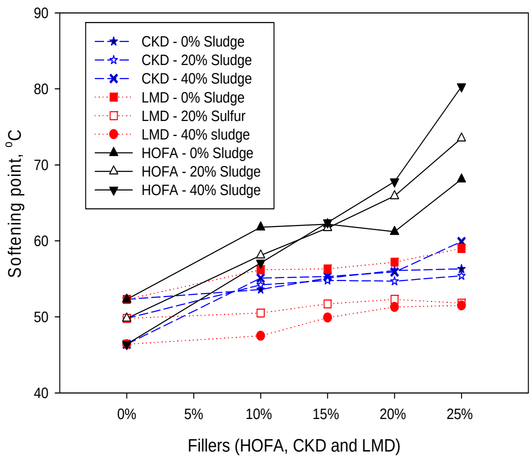

Figure 4. Softening Point of the Oil-Sludge Asphalt Mastics.

There seems to be little or no interaction between the LMD and OS for the given content ranges. Lower SP values were observed for higher OS-content blends containing the same amount of LMD than blends having no OS (Fig. 4). The 25% LMD and 40% OS blend had a lower SP relative to the neat asphalt.

3.3 *Viscosity*

HOFA has an exponential positive effect on the asphalt viscosity, which tended to increase with more OS for doses below 10%, after which it started declining until it fell a bit below the original asphalt viscosity (by 37.5 centi-poise) at a 40% OS content (Fig. 3). However, the resulting viscosities of the HOFA-OS blends were always higher than that of HOFA-only mastics (Fig. 5). This signifies a constructive interaction between the two additives.

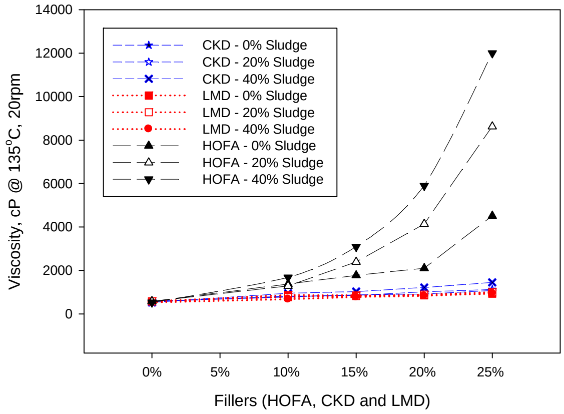

Figure 5. Softening Point of the Oil-Sludge Asphalt Mastics.

3.4 Ductility

Ductility is a measure of the ease with which the material can be deformed plastically at room temperature. OS had a negative effect on the ductility of the asphalt material. The initial level of OS (20%) caused a tremendous ductility loss (~75%) when compared to the fresh asphalt, after which the rate of decrease slowed considerably (Fig. 6). Blends containing both OS and HOFA exhibited a normal interaction between the two additives. The ductility decreased uniformly with the addition of OS and HOFA.

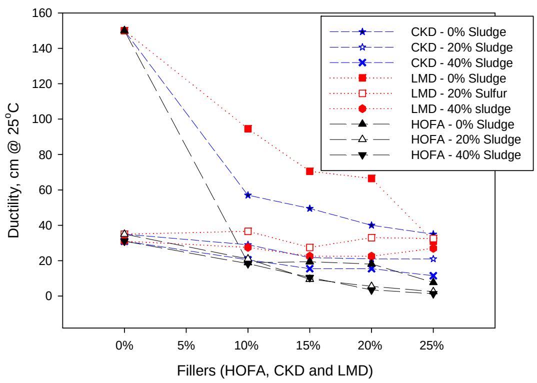

Figure 6. Ductility of the Oil-Sludge Asphalt.

Normal interaction could also be observed for blends containing both CKD and OS additives (Fig. 6). The ductility decreased with an increase in both CKD and OS when used individually and in combination. This trend was maintained for the hybrid blends, with 20% and 40% OS curves showing a uniform transition rate in ductility.

The ductility also decreased with more LMD for LMD-only blends, with an almost linear uniform declining trend. But when combined with OS, the LMD had little effect on ductility in the sense that the ductility value of LMD-OS blends was more or less equal to that of the OS-only composite. This can be seen from the almost horizontal 40% and 20% OS curves of Fig. 6.

3.5 Penetration

Penetration is a measure of the material hardness at room temperature. The individual trend of both OS and HOFA was maintained even in composites containing both additives, but the decrease in penetration with increasing HOFA was more significant in blends containing high OS (40%) than those containing 20% OS (Fig. 7). In (Fig. 7), an abnormal rise in penetration value with increasing CKD content can be observed for a 20% sludge curve. Instead of declining with more filler content as usual, it turns out that the penetration keeps rising with the addition of up to 20% CKD and then drops slightly at 25%. One possible explanation for this different trait could be the relatively higher particle size of CKD compared to the other fillers. This would result in uneven and sparsely distributed filler-grains which produce a weaker CKD-asphalt-sludge monolith with weaker asphalt-sludge three-dimensional (3D) spots at a lower CKD content. When the penetration needle was released, it passed through these weak spots and easily pushed down any CKD particle blocking its path. So, even when the CKD quantity increased, the result was a weaker adhesion of the asphalt-OS fluid to the more numerous CKD grains. However, as these fines increased further, their downward displacement by the needle tends to slow down, thus resulting in a relatively lesser penetration value (25% CKD). But in cases of higher sludge content (40% sludge), the 3D matrix is more stably compact, with large amounts of sludge solid fines. This resulted in a continuous decrease in downward and lateral displacement of the CKD grains as they became situated in a highly filled asphalt matrix. Similar but not obvious behavior can be observed in the LMD-OS blend (Fig. 7), but due to the finer LMD particles, the trend reversed immediately after the LMD content reached 10%. LMD tended to decline in the penetration reading when added to the sludge-asphalt composite, even though the effect was very slight.

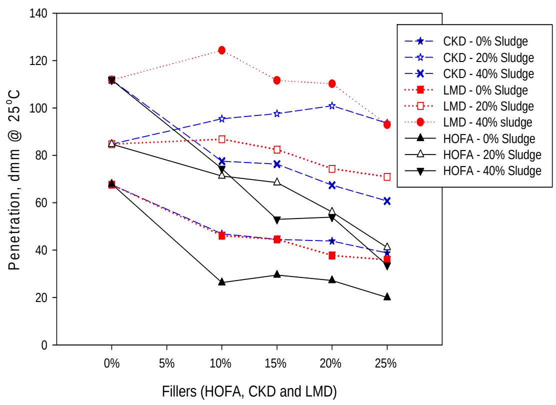

Figure 7. Penetration of the Oil-Sludge Asphalt Mastics.

3.6 *Flash Point (FP)*

Flash point (FP) is a measure of the minimum temperature at which material will ignite. It serves as an indicator for fire risk assessment in terms of safety when handling and in service. The OS had a decreasing effect on the FP of the asphalt which is due to the presence of some volatile flammable hydrocarbons in the waxy constituent of the OS. The 20% content resulted in a decline of about 80 °C, but this effect became less pronounced with the addition of OS (Fig. 8). As for the OS-HOFA composites, the FP remained almost unaffected with the addition of HOFA compared to the OS-only mastic.

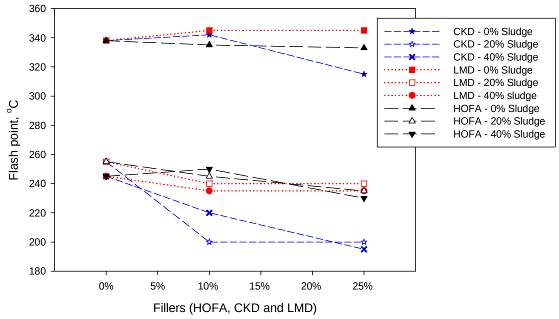

Figure 8. Flash Point of the Oil-Sludge Asphalt Mastics.

The CKD tended to affect the FP more significantly in the OS-asphalt medium as compared to HOFA. An additional decrease of about 30 °C was observed for 20% and 40% OS curves at 10% and 25% CKD contents, respectively (Fig. 8). All in all, there seemed to be little interaction between the substances. Adding LMD to an OS-asphalt blend did not seem to change its original FP value by more than 15 °C in a mixture with 25% content (Fig. 8). The 20% and 40% OS curves are almost straight lines.

3.7 *Tensile Bond Strength (BS)*

The tensile bond strength (BS) increased with OS initially, and then started to decline at higher content levels (Fig. 9). When the composition contained only a small amount, OS particles served as a filler reinforcement engulfed within the sufficiently thick asphalt network surrounding them. But as the sludge's solid particles increased in quantity along with its wax, the asphalt network responsible for binding them thinned out. This was also accompanied by reduced cohesion, which gradually led to phase inversion and a continuous decline of the asphalt's strength.

The neat asphalt's BS was slightly above 25 kN/m^2, and adding filler to the asphalt

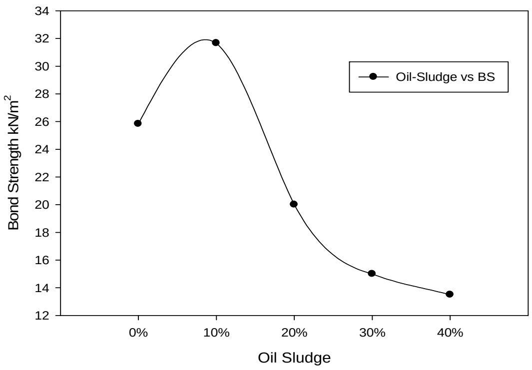

Figure 9. Bond Strength Vs. Oil-Sludge.

generally resulted in an increase in BS (Fig. 10). Both CKD and LMD produced composites with at least a 100% increase in BS compared to the original asphalt at 25% content. Mixtures with 25% HOFA yielded a material with a BS 12 times that of pure asphalt.

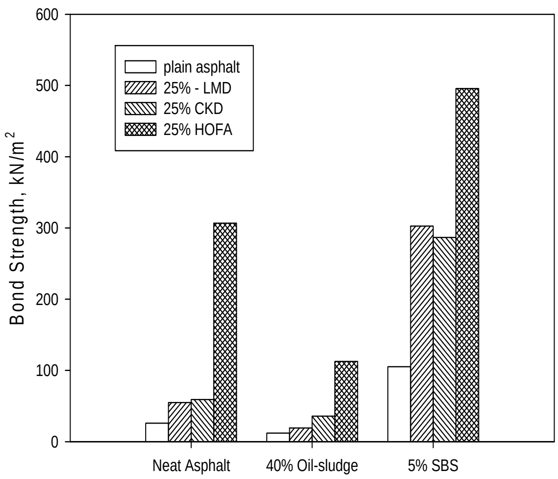

Figure 10. Bond Strength of Fillers, Oil-Sludge And SBS Asphalt Mastics.

In higher quantities, the OS yielded a composite with very little BS, and reviving its strength by adding 25% fillers seemed not to help improve the BS much (Fig. 10). But the HOFA-sludge mastic showed a considerable increase in BS when compared to neat asphalt, which had a BS above 100 kN/m^2.

For reference purposes, conventional SBS polymer mastics for asphalt roofing were also examined. The 5% SBS-modified asphalt exhibited almost twice the BS possessed by the 25% CKD and LMD containing asphalt. Adding CKD or LMD to the 5% SBS blend nearly tripled its BS, but the SBS-HOFA mastic resulted in a much higher BS (Fig. 10). The 40% OS mastic-containing HOFA possessed a BS higher than the 5% SBS of modified asphalt.

3.8 Results of Statistical Analysis of Variance

A two-way analysis of variance (ANOVA) was conducted on the physical test results obtained with the additives used as effects/factors, using Minitab Statistical Software (Minitab Ltd., Coventry, UK) Version16. This was to ascertain the relative effectiveness of the fillers within the sludge-modified asphalt and the OS's level of influence on the asphalt properties. However, before ANOVA was selected for the analysis, a model adequacy check was performed on the data. Tests such as the equality of variance test and normality check test were carried out to ensure the relevance and appropriateness of the selected test method.

Table 4 shows the results of the statistical analysis of the test results. OS did not have a significant influence on the SP when used along with HOFA and CKD fillers, unlike when it was used with LMD. But the OS filler's interaction was highly significant, and caused a more significant rise in the SP value when paired together than when the filler was utilized alone. In other words, the small loss in SP due to the use of sludge could be more than compensated for by the addition of CKD or HOFA. OS significantly affected the penetration when used along with the fillers, but CKD in particular showed an insignificant influence on the penetration compared to the rest of the fillers. Viscosity was slightly affected by OS, but all fillers significantly affected viscosity when paired with OS. Ductility was considerably influenced by OS and the fillers.

3.9 ASTM Specifications

According to ASTM D 449, which is the standard specification for asphalt used in damp proofing and waterproofing, all the mastics containing only HOFA are Type II and those containing CKD/LMD are Type I material. Most of the sludge-HOFA mixes are considered Type II class, but some containing higher sludge/HOFA levels failed to meet the minimum ductility limit. The majority of CKD-sludge blends that should have been classified as Type I material exhibited a slightly deficient

Table 4. Summary of statistical analysis result.

	Analysis of variance result obtained at 5% significance level				
	Factors/Additives	**Tabular F_{value}**	**Calculated F_{value}**	**P-value**	**Inference**
SOFTENING POINT (°c)	Oil Sludge	4.4590	0.32	0.736	Insignificant
	HOFA	3.8379	6.96	0.010	significant
	Oil Sludge	4.4590	0.24	0.795	Insignificant
	CKD	3.8379	15.76	0.001	Significant
	Oil Sludge	4.4590	61.99	0.000	Significant
	LMD	3.8379	10.07	0.003	Significant
DUCTILITY (cm)	Oil Sludge	4.4590	1.72	0.239	Insignificant
	HOFA	3.8379	2.82	0.099	Insignificant
	Oil Sludge	4.4590	5.84	0.027	Significant
	CKD	3.8379	2.17	0.163	Insignificant
	Oil Sludge	4.4590	7.96	0.013	Significant
	LMD	3.8379	1.30	0.349	Insignificant
PENETRATION (dmm)	Oil Sludge	4.4590	0.24	0.795	Insignificant
	HOFA	3.8379	15.76	0.001	Significant
	Oil Sludge	4.4590	16.99	0.001	Significant
	CKD	3.8379	1.40	0.316	Insignificant
	Oil Sludge	4.4590	98.57	0.000	Significant
	LMD	3.8379	4.44	0.035	Significant
VISCOSITY (cP)	Oil Sludge	4.4590	3.34	0.088	Insignificant
	HOFA	3.8379	11.54	0.002	Significant
	Oil Sludge	4.4590	6.56	0.021	Significant
	CKD	3.8379	22.92	0.000	Significant
	Oil Sludge	4.4590	3.66	0.074	Insignificant
	LMD	3.8379	37.03	0.000	Significant

Table 5. Recommended mastic additive content.

Roofing Application		
Sludge	HOFA	LMD/CKD
0–40%	10%–2 5%	15% –35%
Waterproofing and Damp proofing Application		
0–40%	0%–15%	0%–25%

FP value, which relegates them to a position outside the class range type. All the 20% sludge-LMD blends were Type I, but the 40% sludge-LMD materials which should also have been classified as Type I could not pass the ductility tests and some did not even pass the penetration standards.

The sludge-HOFA blends fall under various material classes according to ASTM D 312, which is the standard specification for asphalt used in roofing, but some have a FP slightly below the minimum set point. Most of the double-additive blends containing LMD or CKD in addition to sludge did not pass the SP criteria and all failed to satisfy the FP minimum set value.

4. Conclusions

All the fillers (HOFA, CKD and LMD) produced composites with at least a 100% increase in BS as compared to the original asphalt with 25% content. A large quantity of OS in a mixture resulted in deterioration of the asphalt mastic strength, but this loss in strength could be more than compensated for by filling the mastic with HOFA. Unlike the LMD and CKD sludge blends, the HOFA-sludge mastic showed a considerable increase in BS when compared to neat asphalt. OS interacted positively with the fillers (especially HOFA and CKD) to improve their effectiveness in raising the SP and viscosity of the asphalt, and also in reducing its penetration and ductility. The resulting low FP of some of the sludge mastics as per roofing asphalt specification ASTM D 312 put into question their suitability for this application, even though these mastics hold the promise of becoming suitable composites for damp proofing and waterproofing. Table 5 shows the recommended ranges of additive content for the two categories of waterproofing applications.

Finally, the BS test could encourage the bonding ability of the modified asphalt mastic which the other conventional asphalt tests such as the viscosity test could not do. The viscosity results show the ability of the sludge to thicken the asphalt mastic, a trend which is consistent with other fillers like HOFA, CKD and LMD. But while the viscosity increased as a result of the OS, yielding a lower BS, the increase in viscosity due to the fillers had a positive effect on the BS of the mastic.

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