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Pull off Adhesive Bond Strength Assessments of some Corrosion-Protective Bitumen Coatings on Low Carbon Steel Plates

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Abstract: Premature adhesion failure of coatings in critical corrosion protection services can spell disaster or have significant costs. Adhesion testing is performed to check compliance of coatings to standards. Bitumen of satisfactory coating properties is used to protect structural steelwork in corrosion-predominant industries. The aim of this paper is to present the assessments with coatings of surface and underground natural bitumen samples gotten at Agbabu village and a synthetic bitumen sample from Kaduna refinery in Nigeria. The bitumen samples were separately used to produce 0.81mm to 1.46mm-thick coatings between similar pair surfaces of low carbon steel plates. The coatings were dry-conditioned in triplicate sets of each sample thickness for various curing durations of 12 to 36 hours. Bond strengths of the coatings to the plates were test-determined as the peak tensile stresses that detached the coatings and reported as the respective triplicate averages. Analysis of collated results showed that the entire bond strengths averaged 0.682N/mm^2 and increased from 0.27 to 0.978N/mm^2 with increase in conditioning time and decrease in coating thickness. Further analysis using relevant literature information on various coating grade bitumen indicated that the coatings had good adhesion to the steel with lots better results from the refinery bitumen coatings dry-conditioned for at least 24 hours. Corrosion inhibition capability levels of the steel by the coatings had previously been investigated and reported as a research effort to characterize bitumen from the sources for steelwork protection. The paper is follow-on supplementary information on bitumen from the sources for the protection intention.

Key words: Adhesion, Bitumen coatings, Corrosion inhibition, Evaluations, Standards, Steelwork

INTRODUCTION

The Carbon steel is the most commonly and widely used structural material, but it is a corrodible material of serious concern (Guma *et al*, 2019). Corrosion of carbon steel as the chief and most versatile structural material accounts to majority of all corrosion problems in the world. The most popular, economical, and widely used method of combating corrosion of carbon steel is by paints or organic coatings (Guma *et al*, 2015; Guma and Abu, 2018). Coating materials vary greatly; but they are all required among other things to be sustainably available at minimal cost, provide good adhesion, and completely inhibit corrosion of the substrate to be protected with minimal coating thickness (Guma *et al,* 2015). Maintained adhesion of coatings with adequate bond strengths is therefore crucial for long-lasting and reliable corrosion protection. However; coating systems are in practice subjected to mechanical stresses, elastic-plastic distortions, thermal stress and environment; so liable to fail eventually at different periods that depend on their adhesive bond levels to tolerate these (Internet-1, 2, 2016). Coating adhesion refers to the amount of energy or mechanical strength required to break the adhesive bond between the coating and its substrate. Inadequate adhesion means that the adhesive bond strength is not sufficient to maintain the coating to its substrate under the stress conditions that act upon the coating for the intended use in practice (Butt *et al*, 2007/2008; Jakarmi, 2012). Premature adhesion failure of coatings in critical corrosion protection services can spell disaster or have significant costs (Butt *et al*, 2007/2008; Internet-2, 2016). Adhesion testing is therefore performed to check compliance of coatings to industry standards or customer requirements for specific applications (Jakarmi, 2012). The level of adhesive bond strength of a coating is however affected by many complex variables that need meticulous control to make its test assessment result meaningful. Test results are influenced not only by the properties of the adhesive materials and the substrate; but also by the nature and preparation of the substrate, method of application of the adhesive material, thickness of the applied coating film, cure times, temperature of the coating, environmental humidity, and types of test equipment used (Marek and Henrrin, 1968; British Standard Institution, 2003; Kanitpong and Bahia, 2003, 2005; Baghdaci, 2014).

There are different developed adhesion test procedures; but all have some limitations in usage, reproducibility of coating thickness, flexibility to produce different coating thicknesses, and adhesive bond strength measurements. Pull off test is a commonly and widely used adhesion test procedure for measuring the bond strengths of paints or organic coating films, varnishes, mortars, concretes, and other coatings. It can be applied for coatings on wide range of substrates such as metals, plastics, woods, and aggregates. Generally, pull off test is conducted by measuring the minimum evenly distributed tensile stress necessary to detach or fracture coatings of adhesive material in a direction perpendicular to the substrate (Marek and Henrrin, 1968; Baglin, 1988; Arif Butt *et al,* 2007/2008; McKnight *et al*, 2014). Although there are various testing techniques and procedures used to conduct pull off test, the commonly used pull off test is conducted by inserting or casting thin films of uniform thickness of adhesive materials between two plates of rigid substrates as shown in Fig. 1.

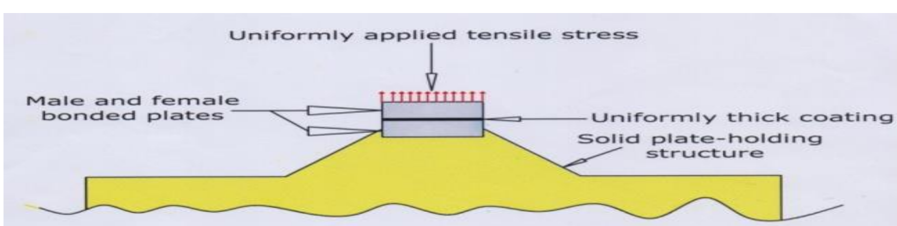

Fig. 1 Commonly used pull-off adhesive bond strength test process.

The bonded assemblies are then subjected to increasing tensile stress until fracture occurs. Based on the ASTM D4541 standard method for pull-off adhesive bond strength of coatings using portable adhesion testers, the applied tensile stress must be steadily increased within specified rate intervals perpendicular to the adhesive coating for even distribution of the applied stress throughout the coated surface. This is done to avoid fracture of the coating at the area where the stress is concentrated before the maximum tensile stress is reached elsewhere and not producing the best result. The fracture can occur due to either adhesive failure along the interface of the coating and the substrate (adhesion), or cohesive failure through the layer of the coating material, or combination of both. The combined force of adhesion and cohesion is what is referred to as the adhesive bond strength (Jakarmi, 2012). Bitumen has been an organic material of great importance in our technological annals. It is found naturally existent at various locations around the globe or synthesized from some petroleum, and supplied in a variety of grades or qualities that are not all good for coating and other specific engineering applications. Bitumen of satisfactory quality can be used in various coating forms to protect structural steelwork such as pipelines, surface and underground tanks in corrosion-predominant industries such as petroleum or other chemical and water industries (Guma *et al*, 2019).

Nigeria is blessed with vast reserves of natural bitumen. The country's bitumen deposit is the second largest in the world after the Canadian deposit. The compositional contents and quality of Nigerian bitumen are however not consistent but vary from location to location. Agbabu is a village in Ondo State of Nigeria with location coordinates E004^{o}48-49^{1} and N06^{o}34-36^{1}. It is a place where bitumen was first spotted in Nigeria in 1910. The bitumen deposit within the region of the village has been ranked as one of the first five major bitumen deposits in the world; but has essentially remained undeveloped, ungraded, and unexploited. On the other hand, Kaduna Refining and Petrochemical Company (KRPC) is the most important synthetic bitumen outfit in Nigeria (Adedemila, 2000; Adegoke, 2000; Guma *et al* 2010, 2011a, 2011b, 2013; KRPC-NNPC, 2017). The country's vast bitumen resources are seen among other engineering applications to be sustainably exploitable to economically coat-combat corrosion in the country's petroleum dependent economy that is bedeviled by effects and costly management of corrosion. Test-obtained information on corrosion inhibition capability levels of low carbon steel by 0.81, 0.93, 1.13, 1.29 and 1.46mm-thick coatings of two virgin natural bitumen samples named OndoS.A and OndoS.B harvested at Agbabu village in Ondo State, and a synthetic type named KPB harvested at KRPC have been reported by Guma *et al*; 2010, 2011a, 2011b, 2013, 2014. Their test information indicated that coat-treating the steel with the bitumen samples to the thicknesses can appreciably inhibit corrosion deterioration of the steel's mechanical properties to the tune of 63 to 91% tensile strength, 69.53 to 97.43% hardness, 57.26 to 90.47%impact strength and 53.36 to 86.89 fatigue strength (Guma *et al*; 2010, 2011a, 2011b, 2013, 2014). This paper is follow-on to provide supplementary information on suitability levels of the OndoS.A, OndoS.B, and KPB bitumen sample coatings for corrosion protection of structural steelwork with the main objectives;

i. To present a conducted pull off adhesive bond strength test of the 0.81, 0.93, 1.13, 1.29 and 1.46mm-thick coatings of the bitumen samples on low carbon steel.
ii. To analyze the test results and provide valuable insight into adhesive bond strength levels of Agbabu and Kaduna refinery bitumen coatings on the steel.
iii. To contribute to the multifarious research efforts towards engineering utilization of abundant bitumen resources in Nigeria for beneficially combating corrosion of structural steel works and other applications.

MATERIAL AND METHODS

MATERIALS

A. Bitumen Samples

Three bitumen samples, namely; OndoS.A, OndoS.B , and KPB were gotten for the research. Ondo S.A sample was harvested from outcrop bitumen in a waterlogged area on the outskirt of Agbabu village; Ondo S.B sample was gotten at about 10 kilometres from where OndoS.A was harvested, at about seven metres from underground through a standard extraction hole. KPB sample was harvested from synthesized bitumen at KRPC with the blend of Basra and Nigerian crudes as feedstock.

B. Low Carbon Steel

A solid low carbon steel bar of 60mm by 50mm in cross section and 15m length was procured from Dana Steel Company, Katsina, Nigeria for the study.

METHODS

Test Procedures

A. Ascertainment of the Procured Low Carbon Steel Bar

To ascertain the bar, its nominal composition was analyzed at R&D unit of Defence Industries Corporation of Nigeria using the Japanese-made Shimadzu PDA 7000 metal analyzer. The analysis confirmed the bar as low carbon steel with nominal compositions of; 0.084% Ca, 0.28% Mn, 0.20% Mo, 95.76% Fe, 1.51% Ag, 0.17% C, 0.59% Si, 0.05% P, 0.83% S, 0.32% Ni, 0.016% Cu, and 0.22% Re.

B. Production and Preparation of Plate Samples for Coating

450 plates were produced with dimensions of 60mm by 50mm cross section by 20mm thickness, by measurements with a steel ruler and mechanically sawing them out from the ascertained procured low carbon steel bar. For comparability of coating bond test results, the plates' surfaces were manually polished to similar smooth finishes using various grades of polishing paper starting with the 250 grade and finishing with the 400 grade. The plates were then cleaned with clean lint-free hand towels. The average thickness of the 450 cleaned plates was then determined to be 19.95mm from the measurements of each plate thickness with a venire caliper. The plates were then immersed in distilled water in an ultrasonic cleaner for 60 minutes at 60^{o}C ±2 to remove any residue on them and neutralize their surfaces. From there, the plates were degreased with acetone to remove any moisture and dust on them which could affect adhesion. The degreased plates were oven-kept at a temperature of 65^{o}C ±2 for one hour prior to coating; to remove any absorbed water on their surfaces to facilitate better bonds between them and the bitumen coatings, and to avoid significant drop in temperature from the bitumen coating temperature during the coating process. These were carried in principle with the ASTM D4541 procedures as used by Jakarmi, 2012. All the plate handlings were done with clean laboratory gloves throughout the test duration so as not to impart any surface undesirability to the prepared plate.

C. Coating the Plates

OndoS.A, OndoS.B, and KPB bitumen samples were separately used to produce 75 coatings of more or less 5mm thickness between pair surfaces of the prepared plates. 15 fabricated similar mild steel molds with cavities of 65mm by 55mm cross sections by 25mm depths, were used for the coating. OndoS.A bitumen sample was gas-heated outdoor in a fairly big steel container to a thermometer-monitored temperature of about 155^{o}C and ladled into a smaller steel container. The smaller container with its contents was put in the oven and the oven temperature adjusted to bitumen coating temperature of 150^{o}C ±2 (Jakarmi, 2012). This was to ensure that all heated bitumen samples were at the same temperature of 150^{o}C ±2 before coating. Before then, 15 prepared oven-kept plates were removed with steel tongs and separately placed in flat positions in the 15 mold cavities and grease applied to the cavities' surfaces to prevent or minimize bitumen sticking to the surfaces. The bitumen sample was then ladled from its container at its oven temperature and poured onto the prepared surfaces of the plates to the brims of the mold cavities. Matching male plates were then placed in position atop the poured bitumen and pressed slightly downwards by hand through suitable wooden slabs to make proper contact of the plates with the bitumen. The bonded plate assemblies were then removed from the molds with suitable steel tongs and placed in contact with ice blocks in a plastic bowl for 30minutes to allow the coatings set between the plates for better handling of the bonded assemblies. The bonded assemblies were removed from the bowl for further compression into the desired coating thickness. This was repeatedly carried out with the aid of 15 assistants until the required number of bonded assemblies was produced with each bitumen sample but in each case after cleaning off any attached bitumen to the mold cavities with kerosene and brush. Plate 1 shows one of the fabricated steel molds used for the coating, while Plates 2 and 3 show the bitumen heating facilities, and the front view of the oven respectively.

Plate 1. One of the 15 fabricated molds with 65mm by 55mm and 25mm-depth cavities for coating the plates

Plate 2. A bitumen sample as was being gas-heated in a steel container

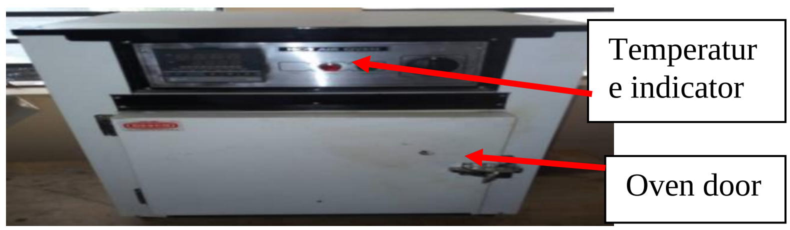

Plate 3. Front view of the oven used for heat-maintaining bitumen samples at150^{o}C ±2 in steel containers

D. Achievements of the desired coating thicknesses between the plate pairs

15 coatings of; 0.81mm, 0.93mm, 1.13mm, 1.29mm and 1.46mm-thickness were produced with each of OndoS.A, OndoS.B, and KPB bitumen samples. The coating thickness of 0.81mm was achieved by holding a given coat-bonded plate pair assembly between the jaws of a precision machine vice as shown in Plate 4 and gradually tightening the assembly by tightening the vice until the sum of hitherto measured average thickness of the uncoated plate pairs (39.9mm) and the required bitumen coating thickness of 0.81mm was 40.71mm. The 40.71mm perpendicular length across the bonded plate pair assembly was ensured by setting the jaws of an accurate Venire caliper across the assembly and slowly tightening the caliper jaws as shown in Plates IV through gradual tightening of the vice jaws until the micrometer reading was 40.71mm. Any bitumen that overflowed the edges of the assembly was trimmed off with a hot steel blade and wiped off by dipping a small bristle brush in kerosene and cleaning the edges. In that way, 15 coatings of 0.81mm thickness were achieved between 15 plate pairs with each bitumen sample. The whole procedure was repeated to achieve each of 0.93mm, 1.13mm, 1.29mm, and 1.46mm-thick coatings between the plate pairs but by ensuring that the micrometer readings across-the-bond assembly were 40.83mm, 41.03, 41.19, and 41.36mm for the respective thicknesses. Plate 5, for example, shows some plate pair assemblies with the 0.81mm-thick coating achieved between them by the process.

Plate 4. An instance of compressing plate pair assembly to achieve requisite coating thickness with a precision vice jaws and a micrometer

Plate 5. Some plate pair assemblies with 0.81mm-thick coating achieved between them

E. Conditioning the coatings

All the produced coatings between the plate pairs were dry-conditioned for curing with each bitumen sample thickness in triplicate sets each for; 12, 18, 24, 30 and 36 hours conditioning duration. The conditioning was carried out in the laboratory at average ambient room temperature of 23.8^{o}C and relative humidity of 32.5%. The prevailing ambient laboratory temperatures were determined three times daily at 08.00 hours, 13.00 hours, and 18.00 hours with a mercury-in-glass thermometer during the five-day test period and averaged to get the 23.8^{o}C value. The relative humidity of 32.5% was similarly determined but with a psychrometer.

F. Pull-off adhesive test of the coatings

At the expiration of conditioning durations, pull off adhesive bond strength of each coating to the plates was determined using the 1332-model INSTRON servo hydraulic frame. The coating bond strengths were all tested at a fixed deformation rate of 16mm/minute under ambient laboratory conditions immediately after the respective conditioning durations. The tests were conducted by gripping each given boned plate pair in the jaws of the INSTRON servo hydraulic frame and pulling the plates apart. The peak perpendicularly applied tensile force in Newton that detached the coating was noted. The collated forces were each divided by the plate coated area of 3000mm^2 to get the respective maximum tensile bond strengths of the coatings. The bond strengths were reported as the respective averages for the triplicate coated and conditioned samples. The failure mode for each bond was documented as cohesive (C) if the average area of cohesive failure of the triplicate samples was observed to be greater than 50%, adhesive (A) if the average area of adhesive failure was observed to be more than 50% or a combination of the two (AC) if the average area of cohesive and adhesive failure was more or less comparable.

RESULTS AND DISCUSSION

A. Results

Figs. 2-6 show results of coating thickness variation and bond curing conditioning durations of 12 to 36 hours at average ambient laboratory environmental temperature of 23.8^{o}C and relative humidity of 32.5% on adhesive bond strengths of; OndoS.A, Ondo S.B, and KPB bitumen coatings to the test low carbon steel. **Figs. 7-11** on the other hand depict effects of variation of bond strength of the 0.81to 1.46mm-thick bitumen sample coatings with the conditioning duration under the same ambient conditions. The evaluated mean bond strengths $(\bar{S_i})$, standard deviation (σ), and coefficients of variation $(\bar{V_i})$, of the 75 test-obtained data values of bond strengths (S_i) using Microsoft Excel statistical tools are shown in Tables 1 and 2. The determined pull off adhesive failure modes of the bitumen coatings for the various curing conditions is presented in Table 3 as adhesive (A), cohesive (C), and combined mode (AC).

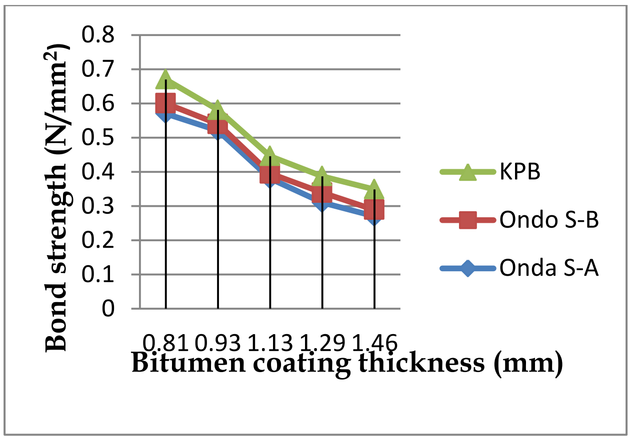

Fig 2. Effects of coating thickness variation on adhesive bond strength of coatings subjected to ambient condition of 23.8ºC and 32.5% relative humidity for 12 hours

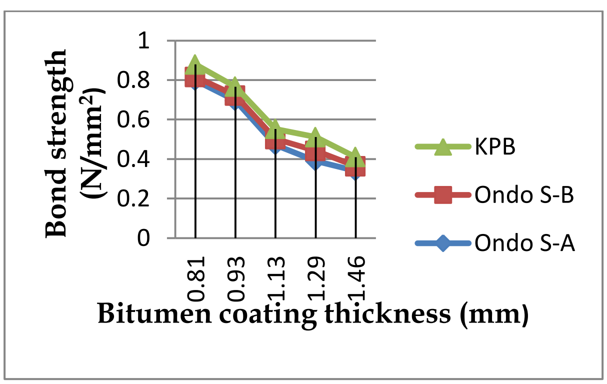

Fig 3. Effects of coating thickness variation on adhesive bond strength of coatings subjected to average ambient condition of 23.8ºC and 32.5% relative humidity for 18 hours

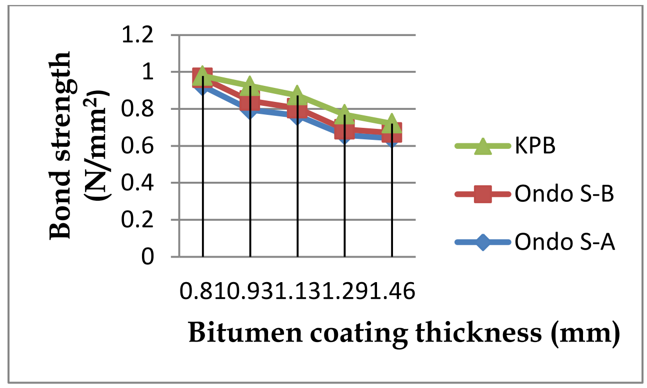

Fig 4. Effects of coating thickness variation on adhesive bond strength of coatings subjected to ambient condition of 23.8ºCand 32.5% relative humidity for 24 hours

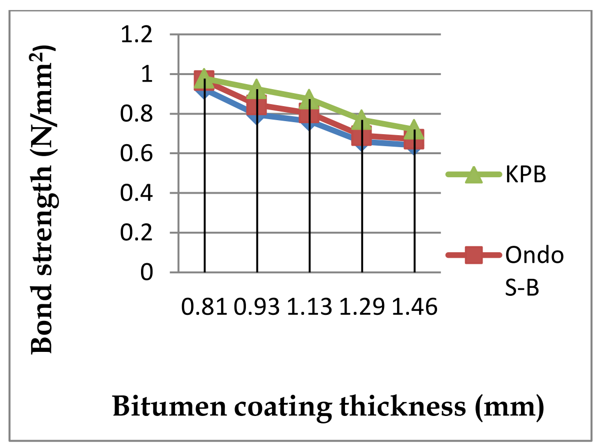

Fig. 5. Effects of coating thickness variation on adhesive bond strength of coatings subjected to ambient condition of23.8ºC and 32.5% relative humidity for 30 hour

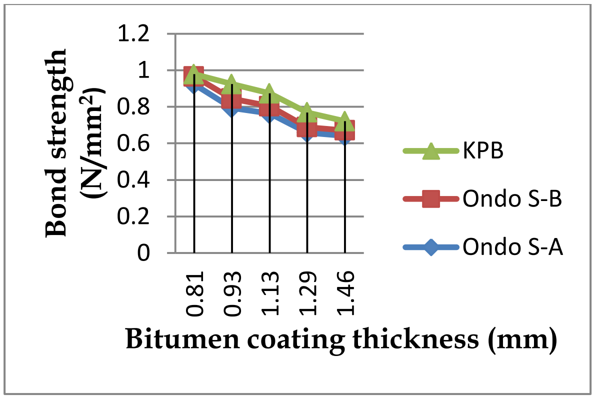

Fig 6. Effects of coating thickness variation on adhesive bond strength of coatings subjected to ambient condition of 23.8°Cand 32.5% relative humidity for 36 hours

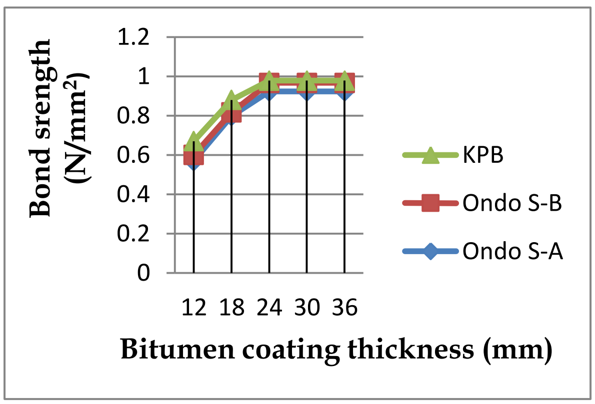

Fig 7. Effects of conditioning time at 23.8⁰C and32.5%relative humidity ambient condition on the adhesive bond strength of the 0.81mm-thick bitumen coatings

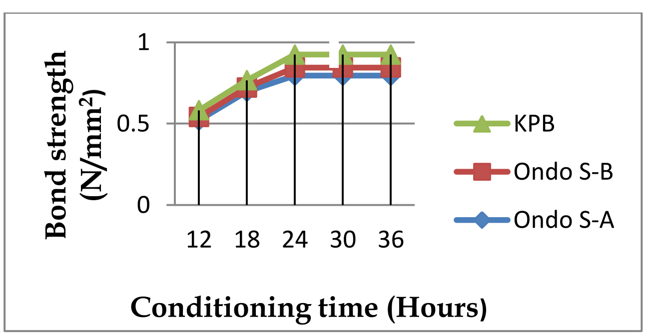

Fig 8. Effects of conditioning time at 23.8⁰C and 32.5% relative humidity ambient condition on the adhesive bond strength of 0.93mm-thickbitumen coatings

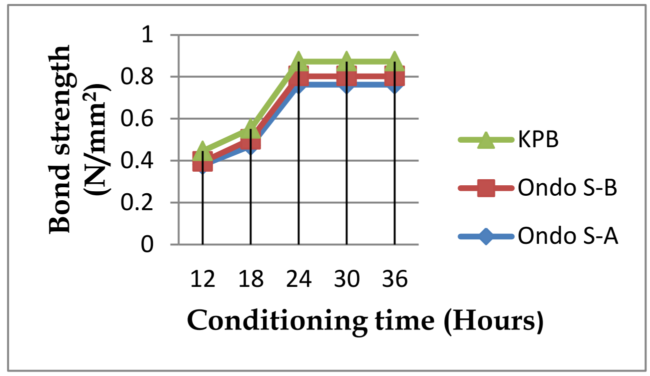

Fig 9. Effects of conditioning time at 23.8⁰C and 32.5% relative humidity ambient condition on the adhesive bond strength of 1.13mm-thick bitumen coating

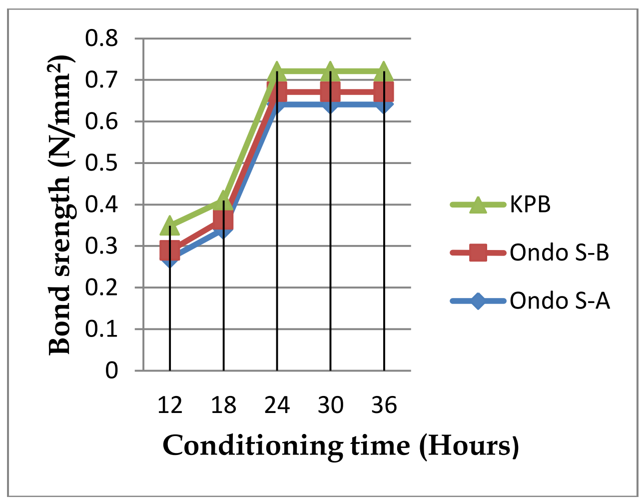

Fig 10. Effects of conditioning time at 25⁰C and 35% relative humidity ambient condition on the adhesive bond strength of the 1.29mm-thick bitumen coating

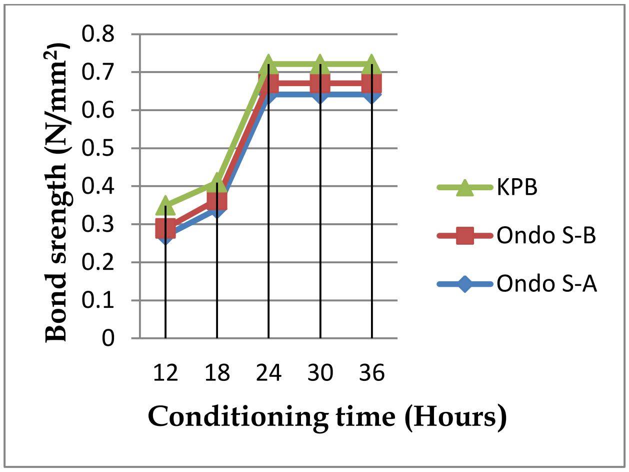

Fig 11. Effects of conditioning time at 25^{0}C and 35% relative humidity ambient condition on the adhesive bond strength of the 1.46mm-thick bitumen coatings

Table 1. The means, standard deviations, and overall range of adhesive bond strengths of the test coatings of the bitumen samples to low carbon steel plates

Bitumen Sample	Mean bond strength	Bond strength standard deviation	Minimum bond strength	Maximum bond strength	Bond strength Range
OndoSA	0.646427N/mm^2	0.19427N/mm^2			
OndoSB	0.6770427N/mm^2	0.197727N/mm^2			
KPB	0.72427N/mm^2	0.19127N/mm^2	0.27N/mm^2	0.97827N/mm^2	0.95127N/mm^2
Overall	0.68227N/mm^2	0.194927N/mm^2			

Table 2. Coefficients of variation of adhesive bond strengths of coatings of the tested bitumen samples to low carbon steel plate

Bitumen sample	OndoSA	OndoSB	KPB	Overall
Coefficient of bond strength variation [%]	0.3	29.5	26.389	28.58

Table 3. Failure modes of coatings of the test bitumen samples

Bitumen Sample	Conditioning time (Hours)	Coating thickness (mm) and average dominant failure mode				
		0.81mm	0.93mm	1.13mm	1.29mm	1.46mm
Ondo-S.A	12	C	C	C	C	A
	18	C	C	C	A	A
	24	C	C	A	C	C
	30	C	A	C	A	C
	36	C	C	A	A	A
Ondo-S.B	12	C	A	C	C	C
	18	C	C	C	AC	A
	24	C	C	A	A	C
	30	C	A	C	A	A
	36	C	C	A	A	AC
KPB	12	C	C	C	C	C
	18	A	C	C	C	A
	24	C	C	A	AC	C
	30	C	C	A	C	A
	36	A	C	C	A	A

B. Discussion of Results

From Figs. 2-6, it is evident that the adhesive bond strengths of the coatings to the steel plates decrease gradually with increase in coating thickness. The bond strengths of the bitumen samples deviate minimally from one another for the same coating thickness and test conditions as can be observed from Figs. 2-6. However, the adhesive bond strengths of KPB coatings had an edge over those of OndoS.B, and those of OndoS.B over those of OndoS.A. Coatings of OndoS.A and OndoS.B exhibit closer comparative bond-strength performances with one another for the same coating thickness and test condition than those of KPB as can also be observed from Figs.2-6. From Figs. 7-11, it apparent that the bond strengths of the bitumen coatings to the steel plates increase appreciably with the conditioning time of the coatings up to 24 hours; and from there, no noticeable increase up to the 36-hour conditioning time. The overall highest bond strength of 0.978N/mm^2 was got from the 0.81mm-thick KPB coating conditioned from 24 to 36 hours. The bond strength decreased from this value with increase in coating thickness and decrease in conditioning time to least value of 0.27 N/mm^2 from the 1.46mm-thick OndoS.A coating that was conditioned for 12 hours as can be observed in **Figs. 7- 11.** The differences in bond strengths of the bitumen coatings to the steel plates under the same conditions could be attributed to differences in the quality of bitumen from the samples' sources. This is because bitumen can exist naturally or be produced and supplied in various grades with appreciable differences in quality and service performances. Bitumen of different grades or qualities have been found to exhibit different levels of adhesion to a wide range of materials such as plastics, metals, and aggregates (Baglin, 1988; Jakarmi, 2012).

Tables 1 and 2 show that, the KPB coatings had the highest mean bond strength of 0.724N/mm^2 with standard deviation of 0.191N/mm^2 and coefficient of variation 26.389%. In overall, mean bond strength of 0.682N/mm^2, standard deviation of 0.1949N/mm^2, and coefficient of variation 28.5825% were obtained with the documented 75 triplicate coating average bond strength results with the bitumen samples as can be seen from Tables 1 and 2. From Table 3, it can be seen that the cohesive mode (C) of failure was the dominant failure mode of the bitumen coatings with 46 cases (61.33%) compared to adhesive mode (A) with 26 cases (34.67%), and combined mode (AC) with only three cases (4.0%). It can also be observed from Table 3 that the adhesive mode of failure tended to increase in number while the cohesive mode decreased in number with increase in coating thickness. From this, it was thinkable that the bond strengths between the interface of the coatings and steel plates were greater than the forces that held the molecular layers of the bitumen together. Marek and Henrrin, 1968; Kanitpong and Bahia, 2003; Lytton *et al*, 2005; Moraes *et al*, 2011; Jakarni. 2012; Ogundipe, 2013; and Twagirimana, 2014 test-obtained adhesive bond strengths of different bitumen films in different research works using various substrates, grades of bitumen and test procedures. Their obtained bond strengths generally fell within the range of 0.05 to 2.5N/mm^2. From this, it is apparent that the bond strength range of 0.27 to 0.978N/mm^2 from our tested bitumen coatings also fell within the range. Specifically; Jakarni, 2012 in a preliminary study for his work conducted adhesion test of 0.8mm-thick coatings of 70/100 conventional grade bitumen on carbon steel after subjecting the coatings to various dry-conditioning durations of 6 to 48 hours at 25^{o}C ambient temperature. He tested the pull off bond strengths of his conditioned coatings at a deformation rate of 20mm/minute using the INSTRON servo hydraulic frame. He reported from his results that the value of the maximum tensile bond strength increased as the total conditioning time was increased, and the increase in the bond strength tapered when the conditioning time of 24 hours was reached at average bond strength of about 0.850N/mm^2 but thereafter remained more or less constant up to conditioning time of 48 hours. He also found that bond strengths varied from 0.36N/mm^2 for coatings conditioned for six hours to more or less 0.850N/mm^2 for coatings conditioned within 24 to 48 hours. It is apparent from his results that the magnitudes and range of variation of 0.57 to 0.978N/mm^2 bond strengths from our tested 0.81mm-thick coatings are comparable with the results for his 0.8mm-thick bitumen coatings.

Marek and Henrrin, 1968 conducted tensile bond strength of asphalt cement bitumen films on different substrates such as steel, aluminum, aggregates, etc at 25^{o}C with deformation rate of 0.5mm/minute. They found on average that; with increase in bitumen film thickness, tensile strength first increased to a peak value of about 3.45N/mm^2 and then decreased and almost became a constant value of about 0.31N/mm^2. They also observed peak average bond strength at bitumen film thickness of 0.02mm and constant bond strength value for coating thicknesses greater than 0.2mm. They also found in parallel tests at deformation rates of 25.4, 2.5, 0.5 and 0.127mm/min with conditioning temperatures of 10, 20, 25, 30, 40, and 50^{o}C using various penetration grade bitumen that; tensile bond strength increased with increase in deformation rate, decrease in conditioning temperature, and increase in the hardness of the bitumen. The pattern of variation of their results was upheld by Jakarmi, 2012 with 24-hour dry-conditioned conventional 70/100 penetration grade bitumen film thickness of 0.5mm and aluminum substrate in similar test procedures with wide range of deformation rates and conditioning temperatures. Kanitpong and Bahia, 2005 separately conducted pull off adhesive bond strength tests with 0.2mm-thick films of 28-58 penetration grade bitumen on Silurian rock, galena, Platteville, prairie duchien and glass aggregates using the Pneumatic Adhesion Tensile Testing instrument (PATTI). After dry-conditioning the samples at room temperature for 24 hours, they tested them with a deformation rate of 65.7KPa/second, at the temperature and depicted their obtained results with tensile strengths that ranged from 0.873 to

1.982N/mm² for all the aggregates. Lytton *et al,* 2005 conducted pull off adhesive bond strength of coatings of various thicknesses with asphalt cement of penetration grade 52 on aluminum alloy substrate using dry conditioning procedure of 3 hours at room temperature. They tested the bonds with a deformation rate of 0.508mm/minute at 25^{o}C. They found that for bitumen coatings of thicknesses from 0 and 0.06mm, adhesive bond strength was less than the cohesive bond strength; hence adhesive failure was expected to occur. For bitumen coatings of thicknesses between 0.06 and 0.15mm; the cohesive bond strength was less than the adhesive bond strength, hence cohesive failure was expected to occur and for 0.8 to 1.46mm-thick coatings, bond strengths decreased with increase in thickness from 1.5 to 0.5N/mm². From these, it can be understood that our tested 0.81 to 1.46mm thick bitumen coatings which are much thicker than their tested coatings of less than 0.15mm also exhibit cohesive failure as the dominant mode of failure in a more or less similar pattern. The tensile bond strengths of 0.27 to 0.978N/mm² for our tested bitumen coatings are however less than their reported 0.5 to 1.5N/mm² bond strengths for their 0.8 to 1.46mm-thick coatings. Reasons attributed to these are due differences in the grades of bitumen used and test conditions. Our results are however comparable in values and pattern of variation with their own.

As discussed earlier in our introduction section to this work, adhesion testing to obtain meaningful and practicable quantitative information is a very difficult exercise because of the complexity and wide range of variables that affect it. For adhesive bond strength tests of bitumen coatings, the foregoing discussion on some results from previous relevant researches on the subject has illuminated that; differences in test results are expected due to the grade of bitumen, testing conditions, method, facilities and substrates used. However, in whichever case tests are properly conducted, results must follow acceptable pattern of variation with clear range of values. This is more evident according to Vickey, 2007 that, adhesion testing results present significant variability which is caused by inherent inconsistencies associated with specimen preparation as well as testing procedures. The ASTM D4551 standard for pull off testing even specifies allowable pull off adhesion testing results discrepancies of up to 41% for intra-laboratory testing and up to 58.7% for inter-laboratory testing (Vickey, 2007). It is thus demonstrable that the 0.27 to 0.978N/mm² bond strength range of our tested coatings of ungraded bitumen from the three critical Nigerian sources on low carbon steel plates follow typical pattern of variation and range of values for bitumen coatings of sound adhesion to commonly used bitumen coating substrates in engineering.

CONCLUSION

Nigeria is blessed with vast bitumen resources that can make her experience a quantum leap in her economic and technological development. The resources is however generally ungraded or uncharacterized, and unexploited for various engineering applications. Pull-off adhesive bond strength tests of differently dry-conditioned 0.81mm, 0.93mm, 1.13mm, 1.29mm and 1.46mm-thick coatings of outcrop and underground natural bitumen samples gotten at Agbabu village and a synthetic bitumen sample from Kaduna refinery in Nigeria to low carbon steel plates has been conducted and presented. Results from the tests indicate that bitumen from the sources have sound coating bond strengths to low carbon steel with a lot better results from thinner coatings of the refinery bitumen dry-conditioned at average ambient room environment for at least 24 hours. The information is hereby posited as follow-on from our previous research efforts to characterize bitumen from the sources for exploitation for structural steelwork corrosion protection and relevant research interests in bitumen developments.

RECOMMENDATION

The presented information is hereby recommended for consideration in utilizing the natural bitumen deposit at Agbabu village in Ondo state and synthetic bitumen from Kaduna refinery in Nigeria for corrosion coat-protection of structural steelwork and other research interests in developing the country's bitumen resources for various engineering applications.

CONFLICT OF INTEREST

The work is our original research work. It has not been published or under consideration for publication elsewhere.

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