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**Effect of prolonged heating on the Asphalt-Aggregate Bond Strength of HMA
containing Liquid Anti-strip Additives**

by

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16. Abstract <p>In this study, an attempt was made to determine the effect of prolonged heating on the bond strength between aggregate and asphalt that contained anti-strip additives (LOF 6500 and Morelife 2200). On account of the substantial decrease of anti-strip additive contents for both asphalt binders and mixes when subjected to prolonged heating, whether the loss of additive content due to storage and transport at elevated temperatures affects mix performance in terms of moisture sensitivity or not, was evaluated in this study. A series of tests, namely Tensile Strength Ratio Test (TSR), Contact Angle test, Pneumatic Adhesion test and Atomic Force Microscopy (AFM) test were performed, and the results obtained from each of these tests were compared to come to a reliable conclusion regarding effectiveness of the various tests for assessing the effect of prolonged heating on the adhesive bond strength. Results obtained from the Tensile Strength Ratio test clearly show that as the prolonged heating duration increased the TSR values failed the limiting value of 85% as followed by NCDOT specification in as little as six hours and continued to further decrease with heating duration.</p> <p>Tests were also conducted on asphalt cement containing LOF 6500 antistrip additive using the contact angle goniometer, PATTI device and atomic force microscopy (AFM). The results obtained in this study were inconclusive for the above mentioned three devices.</p>			
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Executive Summary

In this study, an attempt was made to determine the effect of prolonged heating on the bond strength between aggregate and asphalt that contained anti-strip additives (LOF 6500 and Morelife 2200). Previous studies have shown that the anti-strip additive content decreased when asphalt cement and mixtures were subjected to prolonged heating. Therefore, the question arose that, would the asphalt-aggregate bond strength decrease increasing the moisture susceptibility of mixtures when subjected to prolonged heating?

A series of tests, namely Tensile Strength Ratio test (TSR), Contact Angle Goniometer test, Pneumatic Adhesion test (PATTI device) and Atomic Force Microscopy (AFM) test were performed, and the results obtained from each of these tests were compared to come to a reliable conclusion regarding effectiveness of the various tests for assessing the effect of prolonged heating on the adhesive bond strength.

Results obtained from the Tensile Strength Ratio test clearly show that as the prolonged heating duration increased the TSR values failed the limiting value of 85% as followed by NCDOT specification, in as little as six hours and continued to further decrease with heating duration.

Tests conducted on asphalt cement containing LOF 6500 antistrip additive using the contact angle goniometer, PATTI device and atomic force microscopy (AFM) show that the results obtained in this study were inconclusive for the above mentioned three devices.

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1. Introduction

NCDOT requires liquid organic antistripping additives in all asphalt mixtures to mitigate moisture damage. Findings of a recent NCDOT research study (Tayebali et al., 2005) indicate that liquid antistripping additives LOF 6500 and Morelife 2200, commonly used by NCDOT, volatilize and their presence in binder or mixture cannot be detected after as little as 12-24 hours of prolonged heating of the binder and 6-12 hours of prolonged heating of the asphalt mixture at nominal compaction and storage temperatures.

It can be argued that only lighter amine fractions from the antistripping additive are volatilized and that the heavier fraction (residue) remaining after volatilization may still represent an effective antistripping additive capable of mitigating moisture damage in mixtures. Results of the NCDOT research project HWY-2004-05 (Tayebali et al. 2005) indicate that mass losses after 24 hours of heating pure LOF6500 and Morelife2200 additive were 35 and 50%, respectively, as shown in Figure 1.1. The mass loss did not change appreciably between 24 and 48 hours. However, it was noted that the residue not only emitted a strong odor but was also smoky and changed color to brown-black as shown in Figure 1.2, which suggests decomposition (breakdown) in chemical composition. This raises a serious question regarding the effectiveness of the antistripping additive, that is, whether the loss of antistripping additive through volatilization and the eventual breakdown of the residue results in degradation of the asphalt-aggregate bond, which in turn could lead to moisture-sensitive mixtures.

A definitive answer regarding the integrity of asphalt-aggregate bond strength will ensure that NCDOT mixtures are not prone to become moisture-susceptible due to loss of organic antistripping additive through volatilization or breakdown in chemical composition. If the asphalt-aggregate bond is compromised due to extended heating, NCDOT will be provided with a clear basis for pursuing alternative solutions to the currently used organic antistripping additives.

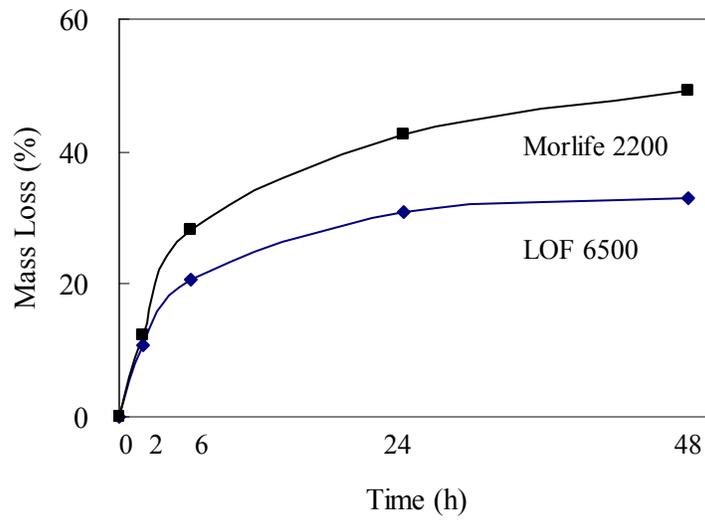


Figure 1.1 Mass Loss of Pure Antistrip Additives as Function of Heating Time $T = 150^{\circ}\text{C}$

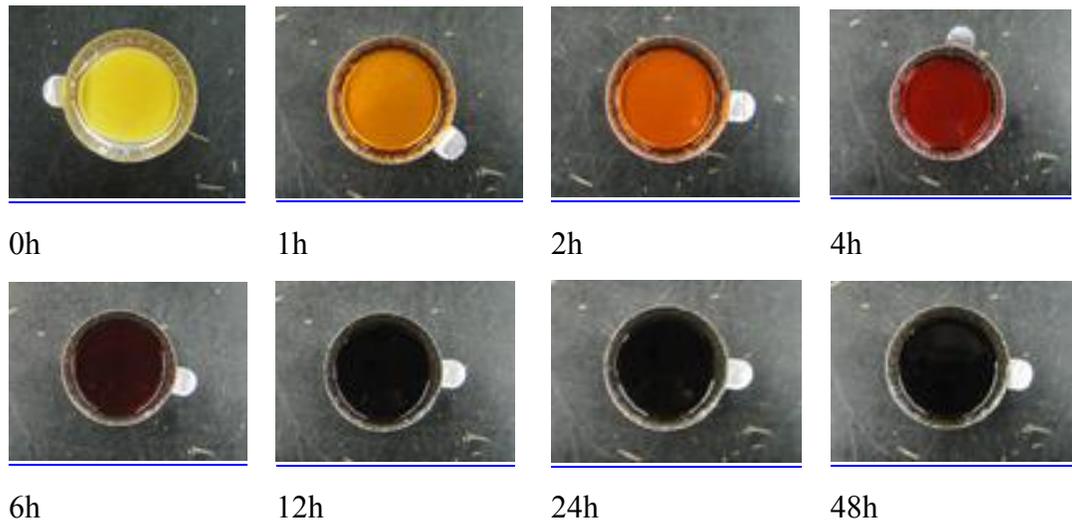


Figure 1.2 Effect of Prolonged Heating on LOF 6500 Antistrip Additive.
 $T = 150^{\circ}\text{C}$

2. Research Objectives

The objectives of this research are to determine the effect of prolonged heating on (1) asphalt-aggregate bond strength and (2) the moisture sensitivity of asphalt mixtures. In particular, this study will first evaluate the performance of mixtures using the tensile strength ratio (TSR) test, and second, study the effect of additive content and prolonged heating on the surface interaction between asphalt binder and aggregate. Figure 2.1 shows the work plan with individual tasks. The specific objectives of this study are:

- Provide a literature review to determine the current state of knowledge regarding the nature and strength of the adhesive bond between asphalt binder and aggregate as well as the loss of adhesion due to moisture in the asphalt pavement.
- Consult with NCDOT personnel in to identify required materials.
- Using LOF 6500 and Morelife 2200 antistrip additives prepare mixes and subject them to TSR testing using NCDOT procedure (modified AASHTO T283).
- Determine the asphalt-aggregate bond strength through measurement of contact angle between asphalt samples containing LOF 6500 and glass/quartz plate (i.e., a model aggregate surface). Determine the effect of prolonged heating and moisture on contact angle.
- Repeat objective 4 with a simple pull-test device.
- Explore the use of atomic force microscopy (AFM) to measure asphalt-aggregate bond strength.
- Correlate the results between tasks 2 to 5.
- Prepare interim and final reports. The final report will provide recommendations to NCDOT regarding the use of organic anti-strip additives and the effect of prolonged heating on moisture sensitivity of NCDOT mixes.

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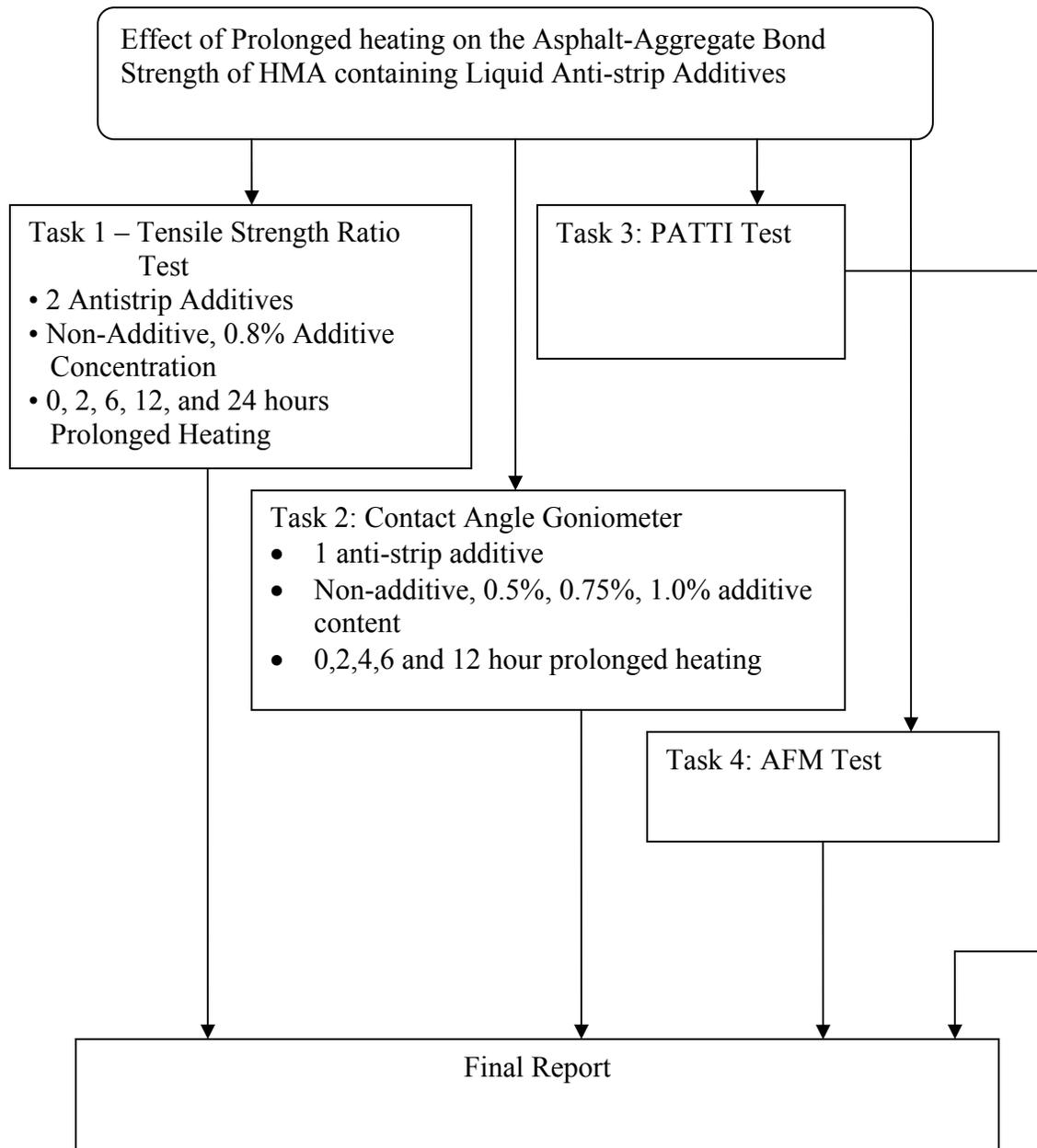


Figure 2.1 Summary of research approach and methodology

3. Literature review for asphalt-aggregate Adhesion

3.1 Background: The presence of the moisture normally causes the problem of debonding of asphalt film from the aggregate surface, which results in the premature failure of the pavement [1, 2]. Mittal [3] has classified adhesion into three categories, namely basic or fundamental adhesion, thermodynamic or reversible adhesion and practical adhesion. According to Mittal, adhesion deals with the basic bond between the particles (like the ionic, covalent, etc) as it is impossible to measure the adhesive forces using this theory, this theory is considered impractical. The reversible adhesion is also considered as impractical as it deals with the equation that involves surface energies and the equation holds good only if there is at least one liquid in the substances considered. The practical adhesion is defined as the force required per unit area to separate the asphalt layer from aggregate [3]. As per the experiments that were performed by researchers, the failure of the asphalt aggregate bond during the adhesive strength determination, might be an adhesive failure or a cohesive failure. According to Mittal [3], adherence is defined as the failure of the bond between the surfaces irrespective; that includes both the adhesion and cohesion failures. Cohesion failure is defined as the loss of bond within the particles of the bitumen and adhesion is defined as the loss of bond between the aggregate and the bitumen. As per the experiments done by the researchers, it can be inferred that, the cohesive failure occurs in the presence of dry aggregates (that is in the absence of water) and adhesive failure between asphalt and aggregate occurs in the presence of water [4]. In order to develop a moisture resistant pavement it is important to determine the effect of moisture over the adhesive strength between asphalt and aggregate. The cohesive and adhesive force of asphalt and asphalt-aggregate system is a function of moisture content. So quantification of the adhesive strength is important to determine the effect of the moisture over the bond strength between asphalt and aggregate [5]. Extensive research has been done over decades to quantify the adhesive strength between asphalt and aggregate. Formerly, qualitative measurements were used to determine the effect of moisture over the asphalt-aggregate bond strength. Due to continuous efforts of the researchers semi-quantitative and quantitative methods have been developed to determine the effect of moisture over the bond strength.

3.2 Adhesion: The measurement of the adhesion is of primary importance in many fields [3]. There are over 300 techniques for the determination of the adhesive strength between the coat and the substrate. This shows the extent of research that is being carried out in various fields in the act of determining the adhesive strength. The techniques are either qualitative or quantitative in nature. Most of these techniques are destructive in nature. As mentioned above, the kind of adhesion that is of primary importance for asphalt-aggregate is the practical adhesion. This adhesive measurement depends over various factors like the stress in the coating, thickness and their mechanical properties of the coatings, mechanical properties of the aggregate (substrate), work consumed by the plastic deformation or viscous dissipation, mode of failure, mode and rate of applying force or energy to detach the film, i.e. the technique used to determine the adhesive strength and the parameters of the technique [3]. The historical development for determination of the adhesive strength between asphalt-aggregate is summarized in the Table 3.1.

Table 3.1 Summary of the historical development in the field of determination of the adhesive strength between the asphalt and aggregate [6].

Author	Year	Type of test used	Remarks
Engineers	1800's	chew test[13]	The engineers used to chew the asphalt to determine the adhesion of the asphalt. If it adhered to their teeth, the asphalt passed the test and is good at adhesion.
Nicholson	1932		The adhesive force between the asphalt and the aggregate reduces due to the presence of water. This fact has been known to the researchers as early as 1920's. Ground work for the determination of the moisture sensitivity was done by these scientists.
Riedel and Weiber	1934	Boiling water test	
Mc Leod	1937		
Hubbard	1938		
Powers	1938		
Winterkorn et al.	1937	Boiling Water test	

Table 3.1 (continued).

Author	Year	Type of test used	Remarks
Saville and Axon	1937	boiling and soaking test	These researchers have performed the tests and compared the stripping performance of various aggregates.
Winterkorn	1937, 1938 and 1939		
Krchma and Nevit	1942		
Krchma and Loomis	1943	wash test, swell test, wet-dry test	
Hveem	1943		
ASTM standard	1950	Immersion-Compression test	First moisture damage test on compacted specimens as per ASTM standard.
Thelen	1958		The researcher tried to determine the relationship between the surface energy of asphalt and aggregate to that of their bonding properties.
Andersland and Goetz	1956	sonic test	This researcher tried to evaluate the stripping resistance in compacted bituminous mixtures.
Johnson	1969		These four researchers tried to develop tests that could be used for the determination of the asphalt moisture mixture sensitivity.
Schmidt and Graf	1972		
Jimenez	1974		
Lottman	1978		Lottman procedure was a breakthrough in the determination of the moisture damage. This method was later standardized as the AASHTO test procedure T283.
Kennedy, Roberts, Anagnos and Lee (UTA)	1982	Freeze thaw pedestal test	This boiling test is almost similar to that of the test used by Saville and Axon (1937).
	1984	Texas boiling test	
Western Research Institute Center			It determined that the displacement of the asphalt polar from aggregate by water varies by the asphalt source.
Ensley et al.	1984	asphalt-aggregate system	This researcher worked towards developing the techniques for measuring the bonding energy of asphalt aggregate system.

Table 3.1 (continued).

Al-Awailmi and Terrel (SHRP)	1992		developed environmental conditioning system
	1993		The Hamburg wheel-tracking device was introduced in Aschenberner and Currier.

3.3 Asphalt-Aggregate adhesion mechanisms: A number of theories have been developed by many scientists and researchers to explain the asphalt-aggregate bond formation. These mechanisms have been summarized in Table 3.2:

Table 3.2 Mechanisms explaining the asphalt-aggregate adhesion [7, 9 and 18]

Theory	General Principle	Supporting Research Source
Mechanical Theory	Asphalt is forced into the pores and the irregularities of the aggregate surface, providing the mechanical interlock.	Knight 1938 (7), Lee and Nicolas 1954 (8) and Rice 1958 (6)
Chemical Reaction Theory	Chemical Reaction occurs between the absorbed asphalt and the constituents of the aggregate phase.	Rice 1958 (6) and Maupin 1982 (9)
Molecular orientation Theory	Asphalt molecules orient themselves so as to satisfy the energy demands of the aggregate surface to the maximum of their capacity	McBain and Lee 1932 (10) and Mack 1957 (11)
Interfacial Energy Theory	Adhesion is a thermodynamic phenomenon related to the surface energy of the materials involved (asphalt, water, air and aggregate)	Thelen 1958 (12), Ishai and Craus 1977 (13)
Weak bond layer theory	Adhesion bond fails due to the presence of the interfacial region of low cohesive strength	J.Schultz and M.Nardin (1994)

3.4 Stripping: The phenomenon of detachment of the asphalt layer from the aggregate is termed as “Stripping”. This stripping of the asphalt layer from the aggregate surface is mainly due to the presence of moisture. The various sources of the moisture can be classified as from the external source (due to the poor drainage), internal source (from the ground water or the moisture present internally within the pavement due to poorly dried aggregates). Researchers have conducted various tests on the adhesion-tension at the asphalt-aggregate interface and applied the principles of surface chemistry and physics to understand the stripping phenomena. Several laboratory tests have been developed to quantify the susceptibility of the asphalt mixes to moisture damage. These theories generally indicate that the moisture damage occurs due to the presence of water and the pore pressure, and is influenced by the properties of the asphalt and aggregates [8]. As

per the researchers, the performance of the asphalt due to the addition of the anti-strip additive is better in adhesion failure and is neutral in the cohesion failure. Review shows that the theories (summarized in Table 3.2) could not singly explain the phenomena of the field moisture damage due to the variability in the highway materials, environment, construction practices and the evaluation methods; and the complex interaction among them [8]. The adhesion of asphalt to the aggregate is also governed by the presence of the clay over the aggregate. Clay is present in the form of aggregate or a thin layer around the aggregate. As the clay expands in the presence of the moisture, it forms a barrier between the asphalt and the aggregate; hence a poor adhesive strength is obtained [1]. Figure 3.1 shows the stripping of the asphalt from the aggregate due to lack of bond between them. Table 3.3 summarizes the theories explaining the stripping phenomena.

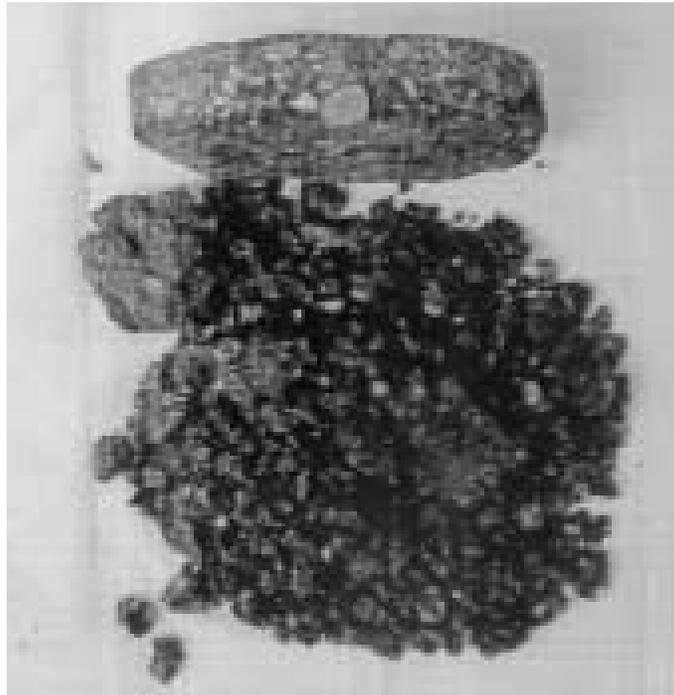


Figure 3.1 Stripping of asphalt due to lack of bond between the asphalt and aggregate [1]

Table 3.3 Summary of Theories explaining the stripping phenomena [7, 18]

Theory	General Principle	Supporting Research Source
Contact Angle Theory or Mechanical Adhesion Theory	Asphalt is displaced because the contact angle of water is less than that of asphalt	Taylor and Khosla 1983(14), Stuart 1990(15), and Hicks et al. 1991(16)
Theory of interfacial energy or molecular orientation theory	Asphalt molecules are displaced from the aggregate surface because the surface energy of water is less than that of asphalt	Taylor and Khosla 1983(14), Stuart 1990(15), and Hicks et al. 1991(16)
Chemical Reaction Theory	Change in the pH value of the water around the aggregates affect the microscopic water at the mineral surface leading to the buildup of the opposing, negatively-charged electrical double layers on the aggregate and the asphalt surfaces.	Taylor and Khosla 1983(14), Stuart 1990(15), and Hicks et al. 1991(16)
Pore Pressure or Hydraulic Scouring Theory	Pore pressure of water entrapped due to the mix densification under traffic results in the increased pore pressure on the asphalt films, leading to the rupture of the asphalt films.	Taylor and Khosla 1983(14), Stuart 1990(15), and Hicks et al. 1991(16)
Theory of Spontaneous Emulsification	Adhesion between the asphalt and aggregates is lost due to the formation of the inverted emulsion	Taylor and Khosla 1983(14), Stuart 1990(15), and Hicks et al. 1991(16)

4. Evaluation of the Effect of Prolonged Heating on the Asphalt-Aggregate Adhesion Using Tensile Strength Ratio Test

4.1 Introduction: The adhesive bond strength between asphalt binder containing antistrip additive and aggregate was evaluated in this study using Tensile Strength Ratio (TSR) test method. On account of the substantial decrease of antistrip additive contents for both asphalt binders and mixes when subjected to prolonged heating, whether the loss of additive content due to storage and transport at elevated temperatures affects mix performance in terms of moisture sensitivity or not was evaluated in this study.

In this study, specimens were divided into two equal batches containing 0.8% LOF 6500 antistrip additive and Morelife 2200 antistrip additive in the asphalt respectively. Eight mixes of each batch were heated for 0, 2, 6, 12, and 24 hours. Samples without antistrip additive were also produced to determine the effectiveness of the antistrip additive in preventing moisture damage.

4.2 Job-Mix-Formula Evaluation and Revision: The asphalt binders and mixes used in this study were obtained from NCDOT. A PG 76-22 asphalt binder was used for all mixes, which is different from previous mixes used for litmus and colorimetric tests that used PG 64-22 [7]. A copy of the original JMF provided by NCDOT is attached as Appendix A.

The JMF had batching percentages for the three aggregate constituents, baghouse fines, asphalt and antistrip additive. The aggregate fractions were 34 percent 78-M stone, 18 percent #67 stone, and 48 percent washed screenings. For these three aggregate constituents, the material passing the #200 sieve was removed. The proportions of aggregate constituents and baghouse fines were adjusted a little to increase the #200 sieve percent passing from 4.8% to 6.5%, which would increase the moisture susceptibility of mixtures. The gradation data are shown in Table 4.1 and gradation curves are plotted in Figure 4.1.

4.3 Evaluation of volumetric properties: The volumetric properties of the laboratory mix were evaluated once the gradation was determined. The asphalt used in

this JMF was a PG 76-22 produced by Conoco in Knoxville, TN. The design asphalt content was determined to be 5.6 percent by weight of the mix. The LOF 6500 antistrip additive was added to the asphalt cement at 0.8 percent by weight of the asphalt. The asphalt concrete was mixed in the laboratory at 163°C and the maximum specific gravity was determined. Using AASHTO T 209 (Maximum Specific Gravity of Bituminous Paving Mixtures), the maximum specific gravity, G_{mm} , was found to be 2.530 compared to the G_{mm} of 2.520 for the JMF.

$$G_{mm} = \frac{A}{A - C}$$

Where:

A = mass of dry sample in air in grams

C = mass of water displaced by sample at 25°C in grams

The quantity of each Superpave gyratory compactor (SGC) samples was calculated by use of experimental G_{mm} value. The target air voids of samples were 7% and compacted at 143°C for testing.

4.4 Tensile Strength Ratio Testing Sample preparation: The moisture susceptibility testing performed in this study followed the NCDOT modified AASHTO T 283 standard. This standard calls for a set of 8 specimens with a 150 mm diameter and a height of 95 mm. These specimens were compacted to a 7 ± 1 percent air-void level; otherwise the sample would be discarded. The specimens were then divided into subsets with half being dry and the other half being moisture conditioned. The samples were conditioned in a 60°C water bath until saturated between 50 and 80 percent. Once saturated, a Marshall indirect tensile test was performed on each specimen. The average tensile strength for each subset was then used to calculate the TSR value as shown below:

$$TSR = \frac{S_2}{S_1}$$

Where:

TSR=tensile strength ratio

S_1 =average dry sample tensile strength

S_2 =average conditioned sample tensile strength

After the TSR is calculated, it is compared to a minimum value to determine the level of moisture damage. The NCDOT acceptable minimum retained strength is 85 percent. Any mix that falls below this value is unsatisfactory and action must be taken to inhibit moisture damage. Two notable differences between the AASHTO T-283 standard and the test performed by NCDOT is the number of specimens and the freeze-thaw cycle. NCDOT uses eight specimens per subset while T-283 requires six. The freeze-thaw cycle, which is optional in T-283, is not used by NCDOT.

Each specimen was mixed at 163°C and subsequently aged for four hours at 65°C following the NCDOT specifications. The mixes were then heated for two hours at 143°C, after which they were compacted using a Superpave Gyratory Compactor. The compaction of each specimen was controlled to a height of 95 mm. After the samples fully cool down to room temperature, the bulk specific gravity of specimen was measured by AASHTO T 166 Saturated Surface Dry (SSD) method (Bulk Specific Gravity of Compacted Bituminous Mixtures Using Saturated Surface-Dry Specimens).

$$G_{mb} = \frac{A}{B - C}$$

Where:

A = dry weight

B = SSD weight

C = submerged weight

Then air voids of samples were calculated.

$$\text{Percent Air Voids} = 100 (G_{mm} - G_{mb}) / G_{mm}$$

Specimen Nomenclature: In order to keep track of the large number of samples produced and tested throughout this study, the following specimen designation system was developed. The names of the subsets had 4 characters describing the type of antistripping additive, percentage of antistripping additive content, prolonged heating time and quantity of specimens. A list of the terms and meanings follows:

First Character – Testing type

L – LOF 6500 additive

M – Morelife 2200 additive

N – None additive

Second set of characters – Percentage of antistripping additive content

00 – none antistripping additive added to asphalt binder

08 – 0.8% antistripping additive added to asphalt binder

Third set of characters – Prolonged heating time

12 – 12 hours prolonged heating

Fourth character – Specimen number.

4.5 Test results and analysis: A total of 88 specimens were prepared and delivered to NCDOT for conditioning and testing. Out of the 88 specimens, 8 specimens were prepared with pure asphalt (without adding antistripping additive). Following the NCDOT specifications the samples were mixed at 163°C and subsequently aged for four hours at 65°C. These mixes were then heated for two hours at 143°C, after which they were compacted using a Superpave Gyratory Compactor. The air voids data of these eight samples are shown in Table 4.2. The remaining 80 specimens were divided into two equal batches containing 0.8% LOF 6500 and Morelife 2200 antistripping additives in the asphalt binders respectively. Eight specimens of each batch were heated for 0, 2, 6, 12, and 24 hours at 143°C respectively. The air voids data for samples containing LOF 6500 and Morelife 2200 are shown in Table 4.3 and Table 4.4.

The TSR test results for these 88 specimens are tabulated in Tables 4.5 through Table 4.15. Table 4.5 shows the TSR test results for specimens prepared with asphalt without any antistripping additive, using NCDOT modified T283 method. The TSR value is obtained to be 71.1%. Table 4.6 to Table 4.10 show TSR test results for asphalt mixes containing 0.8% of LOF 6500 (8 samples each), heated for the duration of 0, 2, 6, 12, 24 hours respectively. The TSR values are 84.1%, 90.9%, 83.1%, 77.2%, 76.3% respectively. Table 4.11 to Table 4.15 show TSR test results for asphalt mixes containing 0.8% of Morelife 2200 (8 samples each), heated for the duration of 0, 2, 6, 12, and 24 hours respectively. The TSR values are 88.0%, 85.4%, 79.8%, 79.3%, 67.4% respectively. The TSR values of asphalt mixes increased from 71.1% to 90.9 and 85.4% for LOF 6500 and Morelife 2200 antistripping additives, respectively, and are higher than NCDOT required value of 85%. As can be noted from the results the TSR values reduced with prolonged heating. Based on the previous work performed by Chun and Tayebali [7], no antistripping additive content was detectable in asphalt mix after 6 to 12 hours of

extended heating. In the TSR test, the TSR values decreased below 85% after 12 hours of prolonged heating, and the TSR values of mixes with LOF 6500 and Morelife 2200 antistripping additives are 77.2% and 79.3%, respectively. These values are slightly higher than TSR value of specimens with no additive, which is 71.1% but clearly they fail the NCDOT standard value. After 24-hour prolonged heating, the TSR values of mixes with LOF 6500 and Morelife 2200 antistripping additives are 76.3% and 67.4%, which are similar to or lower than TSR value obtained without additive.

The declining trend of the curve plotted between TSR test results vs. prolonged heating are shown in Figure 4.2 to Figure 4.4. The raw data for TSR test are shown in Appendix A.

4.6 Discussion of Test Results: From the TSR test results, the improvement of moisture sensitivity using amine based antistripping additive was lost to a great extent after prolonged heating. It is seen that after only 6-hour heating, TSR values drop to about 80% for both LOF 6500 and Morelife 2200 antistripping additives, which are below the NCDOT standard value. Considering storage and transport as well as the duration between production and its ultimate paving in the field, this situation is very serious and needs due attention by NCDOT.

Based on the previous work done by Tayebali et. al. [7], Morelife 2200 antistripping additive is more volatile as compared to LOF 6500 antistripping additive. After 24 hours, the mass loss for antistripping additives LOF 6500 and Morelife 2200 is roughly 25 and 45 percent, respectively. TSR test results show that the residual higher molecular weight fraction of anti-strip additive that remain in the mix after prolonged heating is not effective in lowering the moisture susceptibility of the mixtures. The chemical change that occurs during the heating process of amine based anti-strip additive is a very important factor that affects the improvement of asphalt-aggregate adhesion. It should be noted that two types of amine based anti-strip additives; one level of anti-strip additive content and one gradation of asphalt mix were used in this study. For further confirmation, more mixes, different organic anti-strip additives and dosages are recommended for testing.

Table 4.1 Gradations for Original JMF and Revised Lab Test

Sieves (mm)	Percent Passing		Control Points
	JMF Batching	Revision	
37.5	100.0	100.0	
25	99.8	99.9	
19	98.2	98.5	100.0
12.5	90.1	91.7	90.0-100.0
9.5	82.3	85.0	<90.0
4.75	59.3	63.3	
2.36	44.9	49.1	28.0-58.0
1.18	31.9	35.5	
0.6	22.4	25.4	
0.3	13.7	16.0	
0.15	7.6	9.7	
0.075	4.8	6.5	4.0-8.0

Table 4.2 Air Voids Data for samples containing no Anti-strip additive

Sample ID*	Weight in air (g)	Weight in water (g)	SSD (g)	BSG	Air Voids (%)	Water Absorbed (%)
N00-2-1	3932.4	2277.6	3952.7	2.348	7.2	1.2
N00-2-2	3941.0	2276.5	3955.4	2.347	7.2	0.9
N00-2-3	3941.2	2277.6	3956.4	2.348	7.2	0.9
N00-2-4	3942.3	2282.0	3958.3	2.352	7.0	1.0
N00-2-5	3942.0	2284.0	3959.6	2.353	7.0	1.1
N00-2-6	3929.6	2280.6	3950.8	2.353	7.0	1.3
N00-2-7	3943.4	2275.2	3953.8	2.349	7.1	0.6
N00-2-8	3941.2	2279.2	3957.2	2.349	7.2	1.0

Table 4.3 Air Voids Data of LOF 6500 Additive Samples

Sample ID	Weight in air (g)	Weight in water (g)	SSD (g)	BSG	Air Voids (%)	Water Absorbed (%)
L08-0-1	3911.5	2257.9	3928.2	2.342	7.4	1.0
L08-0-2	3929.5	2267.8	3949.0	2.337	7.6	1.2
L08-0-3	3924.7	2274.6	3946.8	2.347	7.2	1.3
L08-0-4	3938.9	2281.7	3955.7	2.353	7.0	1.0
L08-0-5	3940.5	2277.1	3956.5	2.346	7.3	1.0
L08-0-6	3942.9	2276.4	3962.1	2.339	7.5	1.1
L08-0-7	3939.5	2286.1	3958.5	2.356	6.9	1.1
L08-0-8	3934.4	2284.2	3961.2	2.346	7.3	1.6
L08-2-1	3928.3	2263.1	3945.7	2.335	7.7	1.0
L08-2-2	3942.9	2290.2	3954.3	2.369	6.3	0.7
L08-2-3	3942.7	2293.0	3955.7	2.371	6.3	0.8
L08-2-4	3913.2	2260.2	3927.4	2.347	7.2	0.9
L08-2-5	3905.1	2238.3	3913.3	2.331	7.8	0.5
L08-2-6	3933.0	2272.4	3949.4	2.345	7.3	1.0
L08-2-7	3930.5	2269.9	3940.2	2.353	7.0	0.6
L08-2-8	3919.0	2272.9	3944.5	2.344	7.3	1.5
L08-6-1	3933.7	2267.9	3945.9	2.344	7.3	0.7
L08-6-2	3928.9	2266.2	3942.1	2.344	7.3	0.8
L08-6-3	3943.6	2275.7	3955.6	2.348	7.2	0.7
L08-6-4	3939.1	2273.9	3954.1	2.344	7.3	0.9
L08-6-5	3941.7	2277.1	3954.8	2.349	7.1	0.8
L08-6-6	3936.8	2269.3	3946.8	2.347	7.2	0.6
L08-6-7	3934.9	2274.2	3952.5	2.345	7.3	1.0
L08-6-8	3933.1	2267.5	3946.1	2.343	7.4	0.8
L08-12-1	3908.4	2255.9	3919.8	2.349	7.2	0.7
L08-12-2	3913.0	2265.8	3930.2	2.351	7.1	1.0
L08-12-3	3929.0	2277.2	3936.5	2.368	6.4	0.5
L08-12-4	3949.8	2297.1	3962.1	2.372	6.2	0.7
L08-12-5	3927.7	2275.9	3936.3	2.366	6.5	0.5
L08-12-6	3935.9	2267.0	3946.0	2.344	7.3	0.6
L08-12-7	3934.5	2268.2	3941.7	2.351	7.1	0.4
L08-12-8	3941.6	2291.3	3965.7	2.354	7.0	1.4
L08-24-1	3934.7	2263.5	3946.3	2.338	7.6	0.7
L08-24-2	3934.5	2264.8	3947.6	2.338	7.6	0.8
L08-24-3	3935.1	2267.3	3949.2	2.340	7.5	0.8
L08-24-4	3944.9	2264.9	3952.5	2.338	7.6	0.5
L08-24-5	3940.3	2267.3	3953.4	2.337	7.6	0.8
L08-24-6	3946.3	2273.6	3957.3	2.344	7.4	0.7
L08-24-7	3939.6	2268.9	3952.1	2.341	7.5	0.7
L08-24-8	3938.3	2267.5	3950.8	2.340	7.5	0.7

Table 4.4 Air Voids Data of Morelife 2200 Additive Samples

Sample ID	Weight in air (g)	Weight in water (g)	SSD (g)	BSG	Air Voids (%)	Water Absorbed (%)
M08-0-1	3945.2	2277.7	3956.0	2.351	7.1	0.6
M08-0-2	3936.9	2269.4	3947.1	2.347	7.2	0.6
M08-0-3	3941.2	2267.5	3949.6	2.343	7.4	0.5
M08-0-4	3937.5	2266.7	3947.6	2.342	7.4	0.6
M08-0-5	3911.1	2244.5	3921.4	2.332	7.8	0.6
M08-0-6	3941.7	2275.1	3952.4	2.350	7.1	0.6
M08-0-7	3911.0	2249.4	3924.4	2.335	7.7	0.8
M08-0-8	3943.5	2273.8	3952.9	2.349	7.2	0.6
M08-2-1	3936.1	2273.4	3945.4	2.354	7.0	0.6
M08-2-2	3920.5	2259.1	3934.8	2.340	7.5	0.9
M08-2-3	3921.4	2260.9	3935.6	2.342	7.4	0.8
M08-2-4	3940.5	2281.3	3955.2	2.354	7.0	0.9
M08-2-5	3917.7	2252.2	3928.0	2.338	7.6	0.6
M08-2-6	3935.8	2282.4	3951.6	2.358	6.8	0.9
M08-2-7	3928.0	2274.3	3944.8	2.351	7.1	1.0
M08-2-8	3943.1	2278.3	3954.7	2.352	7.0	0.7
M08-6-1	3940.3	2270.6	3950.7	2.345	7.3	0.6
M08-6-2	3942.4	2266.5	3953.0	2.338	7.6	0.6
M08-6-3	3939.6	2267.4	3950.7	2.340	7.5	0.7
M08-6-4	3936.9	2266.6	3950.3	2.338	7.6	0.8
M08-6-5	3938.3	2266.5	3951.3	2.338	7.6	0.8
M08-6-6	3916.8	2249.4	3930.7	2.330	7.9	0.8
M08-6-7	3928.8	2258.5	3941.0	2.335	7.7	0.7
M08-6-8	3936.4	2268.9	3950.1	2.341	7.5	0.8
M08-12-1	3937.1	2274.1	3946.1	2.355	6.9	0.5
M08-12-2	3932.5	2262.3	3939.3	2.345	7.3	0.4
M08-12-3	3939.5	2273.8	3948.7	2.352	7.0	0.5
M08-12-4	3937.3	2271.2	3945.5	2.352	7.1	0.5
M08-12-5	3939.8	2272.8	3946.3	2.354	6.9	0.4
M08-12-6	3928.9	2269.7	3942.4	2.349	7.2	0.8
M08-12-7	3934.6	2277.8	3947.2	2.357	6.8	0.8
M08-12-8	3934.7	2269.0	3940.1	2.355	6.9	0.3
M08-24-1	3937.4	2258.7	3946.4	2.333	7.8	0.5
M08-24-2	3945.2	2262.5	3956.2	2.329	7.9	0.6
M08-24-3	3917.9	2247.6	3926.3	2.334	7.8	0.5
M08-24-4	3936.5	2264.6	3947.2	2.340	7.5	0.6
M08-24-5	3948.2	2268.7	3963.5	2.330	7.9	0.9
M08-24-6	3940.6	2256.3	3948.6	2.329	8.0	0.5
M08-24-7	3924.6	2252.3	3935.7	2.331	7.9	0.7
M08-24-8	3934.9	2257.9	3942.8	2.335	7.7	0.5

Table 4.5 TSR Test Results: No Additive in Asphalt with 2-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
N00-2-4	7.0	1397.0	1509.3	N00-2-1	7.2	1052.2	1072.6
N00-2-5	7.0	1522.4		N00-2-2	7.2	1069.9	
N00-2-6	7.0	1516.7		N00-2-3	7.2	1075.4	
N00-2-7	7.1	1501.8		N00-2-8	7.2	1131.8	
Tensile Strength Ratio (%)				71.1			

Table 4.6 TSR Test Results: 0.8% LOF Additive in Asphalt without Prolonged Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
L08-0-4	7.0	1023.5	991.8	L08-0-1	7.4	791.4	833.8
L08-0-5	7.3	1008.0		L08-0-2	7.6	819.6	
L08-0-6	7.5	937.8		L08-0-3	7.2	936.3	
L08-0-8	7.3	975.7		L08-0-7	6.9	847.9	
Tensile Strength Ratio (%)				84.1			

Table 4.7 TSR Test Results: 0.8% LOF Additive in Asphalt with 2-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
L08-2-4	7.2	1370.4	1289.3	L08-2-1	7.7	1078.7	1171.4
L08-2-5	7.8	1208.2		L08-2-2	6.3	1210.1	
L08-2-7	7.0	1386.6		L08-2-3	6.3	1191.0	
L08-2-8	7.3	1204.4		L08-2-6	7.3	1151.8	
Tensile Strength Ratio (%)				90.9			

Table 4.8 TSR Test Results: 0.8% LOF Additive in Asphalt with 6-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
L08-6-4	7.2	1541.3	1544.3	L08-6-1	7.3	1285.2	1283.8
L08-6-5	7.1	1547.3		L08-6-2	7.3	1252.9	
L08-6-7	7.2	1551.3		L08-6-3	7.3	1323.2	
L08-6-8	7.4	1450.1		L08-6-6	7.3	1282.5	
Tensile Strength Ratio (%)				83.1			

Table 4.9 TSR Test Results: 0.8% LOF Additive in Asphalt with 12-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
L08-12-2	7.1	1975.3	1925.8	L08-12-1	7.2	1457.7	1487.7
L08-12-5	6.5	1981.5		L08-12-3	6.4	1478.5	
L08-12-7	7.1	1876.4		L08-12-4	6.2	1705.7	
L08-12-8	7.0	1818.9		L08-12-6	7.3	1496.9	
Tensile Strength Ratio (%)				77.2			

Table 4.10 TSR Test Results: 0.8% LOF Additive in Asphalt with 24-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
L08-24-5	7.6	1189.2	1212.7	L08-24-1	7.6	955.7	925.7
L08-24-6	7.4	1078.4		L08-24-2	7.6	899.5	
L08-24-7	7.5	1298.5		L08-24-3	7.5	839.3	
L08-24-8	7.5	1236.2		L08-24-4	7.6	915.9	
Tensile Strength Ratio (%)				76.3			

Table 4.11 TSR Test Results: 0.8% Morelife Additive in Asphalt without Prolonged Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
M08-0-2	7.2	884.2	882.4	M08-0-1	7.1	816.9	776.6
M08-0-3	7.4	880.5		M08-0-2	7.4	755.7	
M08-0-7	7.7	844.4		M08-0-5	7.8	739.1	
M08-0-8	7.2	884.2		M08-0-6	7.1	797.4	
Tensile Strength Ratio (%)				88.0			

Table 4.12 TSR Test Results: 0.8% Morelife Additive in Asphalt with 2-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
M08-2-5	7.6	1196.2	1255.7	M08-2-1	7.0	1083.1	1072.6
M08-2-6	6.8	1283.8		M08-2-2	7.5	1026.7	
M08-2-7	7.1	1265.2		M08-2-3	7.4	1062.1	
M08-2-8	7.0	1246.2		M08-2-4	7.0	1154.3	
Tensile Strength Ratio (%)				85.4			

Table 4.13 TSR Test Results: 0.8% Morelife Additive in Asphalt with 6-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
M08-6-5	7.6	1444.1	1461.1	M08-6-1	7.3	1232.7	1166.3
M08-6-6	7.9	1429.4		M08-6-2	7.6	1119.9	
M08-6-7	7.7	1521.1		M08-6-3	7.5	1136.4	
M08-6-8	7.5	1478.1		M08-6-4	7.6	1196.2	
Tensile Strength Ratio (%)				79.8			

Table 4.14 TSR Test Results: 0.8% Morelife Additive in Asphalt with 12-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
M08-12-4	7.1	1829.1	1813.2	M08-12-1	6.9	1366.1	1438.4
M08-12-6	7.2	1778.4		M08-12-2	7.3	1397.0	
M08-12-7	6.8	1826.5		M08-12-3	7.0	1479.7	
M08-12-8	6.9	1799.9		M08-12-5	6.9	1531.6	
Tensile Strength Ratio (%)				79.3			

Table 4.15 TSR Test Results: 0.8% Morelife Additive in Asphalt with 24-Hour Heating

Unconditioned Specimens				Conditioned Specimens			
Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)	Sample ID	Air Voids (%)	Tensile Strength	Mean Tensile Strength (lb)
M08-24-5	7.9	1346.7	884.6	M08-24-1	7.8	412.0	596.2
M08-24-6	8.0	938.4		M08-24-2	7.9	745.4	
M08-24-7	7.9	830.8		M08-24-3	7.8	447.0	
M08-24-8	7.7	714.4		M08-24-4	7.5	1021.4	
Tensile Strength Ratio (%)				67.4			

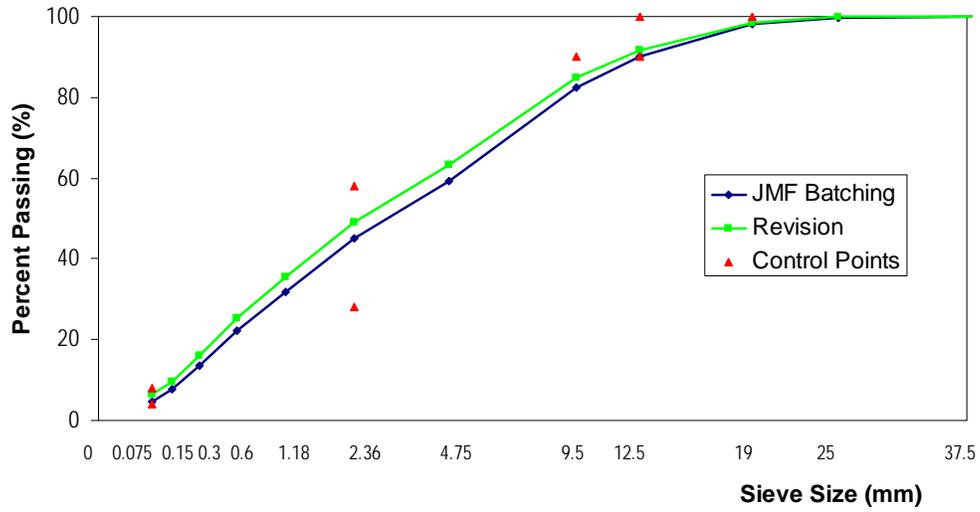


Figure 4.1 Gradation Curves for Original JMF and Revised Lab Test

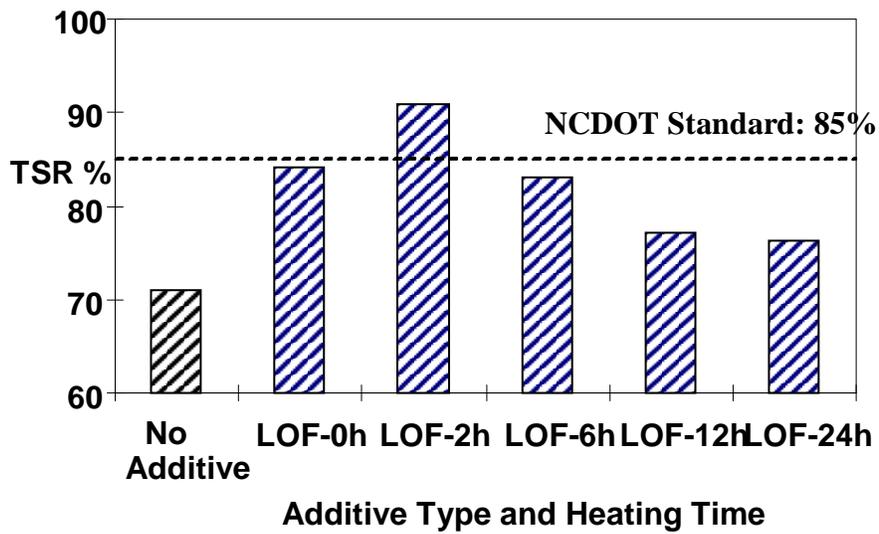


Figure 4.2 TSR Values of Non-Additive and 0.8% LOF Additive

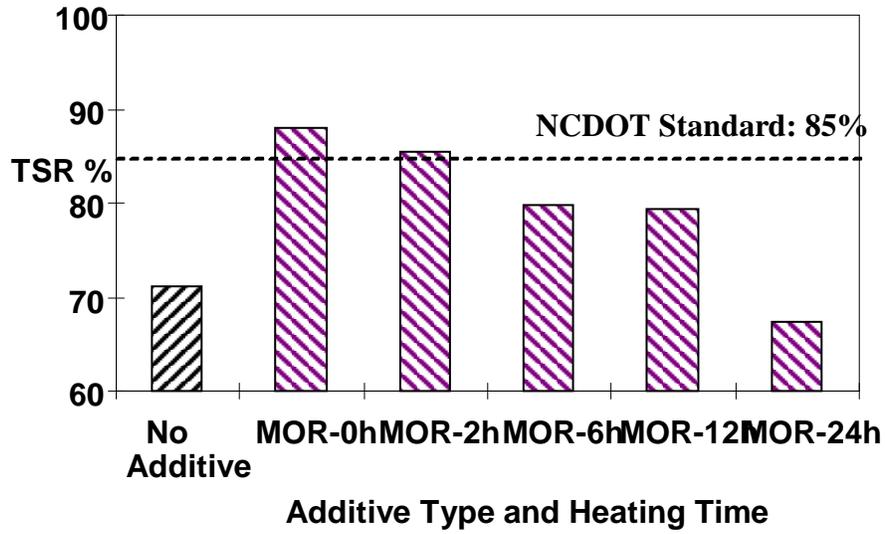


Figure 4.3 TSR Values of Non-Additive and 0.8% Morelife Additive

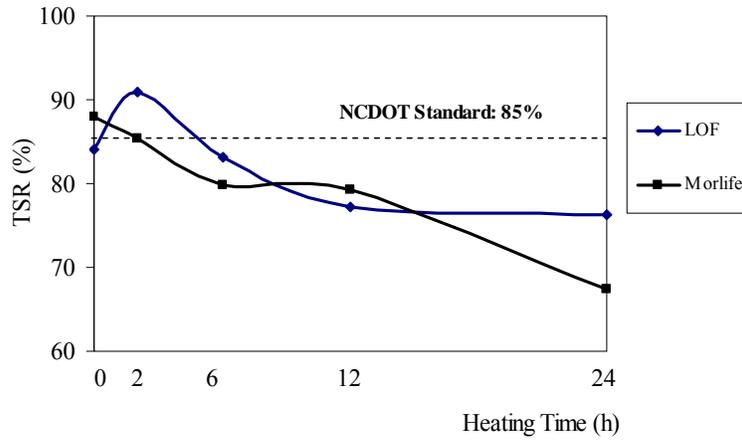


Figure 4.4 Loss in TSR with Prolonged Heating

5. Contact Angle Goniometer Test

5.1 Theoretical background for Contact Angle Test: “Contact Angle” can be defined as the angle formed by a liquid over a solid at the intersection of the three phases (solid, liquid and gas). Contact angle test was developed to quantify the adhesive bond between a liquid and a substrate based on the surface free energy between them (in our case the liquid and the substrate are asphalt and aggregate respectively). The application of the contact angle test is not only confined in the determination of adhesive properties but also in the determination of colloid, lubrication and is also extensively used in the paint industry [9]. The two techniques that are most popularly used to determine the contact angle are the Wilhelmy Plate technique and the Contact Angle Goniometer [10]. Contact Angle Goniometer was used in this study to determine the contact angle between asphalt and quartz/glass slide. The two methods in Goniometry are the Sessile Drop Method and the Pendant Drop Method. The best suited method to determine the contact angle between asphalt-aggregate is the Sessile Drop Method, because it allows us to measure the contact angle coincidentally with the determination of the surface tension and moreover it also allows measurement of contact angle at high temperatures [10]. The principle of the Sessile Drop Method is the measurement of a contact angle between a known liquid over a solid surface [10, 11]. The size of the droplet should be considerably small, to avoid the effect of the gravity over the droplet. Hence, only the surface tension will act over the droplet [12, 13].

When the size of the droplet is small, the contact angle is controlled by the surface tension between the media [13] (solid-liquid, liquid-gas and gas-solid). The droplet comes to an equilibrium state exhibiting a constant contact angle when equilibrium is achieved between the surface tension (or surface energy) between the various media (as shown in Figure 5.1). Equation 5.1 shows the balanced equation among the surface free energy between the media [13, 14].

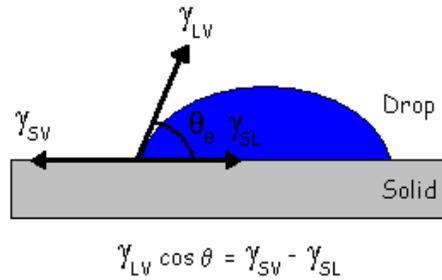


Figure 5.1 Shows the direction of the surface energies working between various media

$$\gamma_{LV} \cos \theta = \gamma_{SV} - \gamma_{SL} \quad \text{Equation 5.1}$$

Where,

γ_{LV} = surface tension between the liquid and vapor,

γ_{SV} = surface tension between the solid and vapor,

γ_{SL} = surface tension between the solid and liquid,

θ = contact angle measured between the solid and liquid.

The other concept that is commonly used by the researchers to define the adhesive property is Work of Adhesion. Work of Adhesion can be defined as the work done on the system; when two condensed phases forming an interface, are separated to form unit areas of each with the interface.

As the Work of Adhesion is high, it implies that the bond strength between the solid and the liquid is high, hence showing that there is a good bond between them. Based on the Young-Dupree equation (Equation 5.2), the contact angle that is obtain from the direct measurement using a Contact Angle Goniometer is inversely proportional to the work of adhesion. Hence, as the contact angle reduces, the work of adhesion increases which in turn shows that the adhesive bond between the solid and liquid is high.

$$W_a = \sigma (1 + \cos \theta) \quad \text{Equation 5.2}$$

Where,

W_a = Work of Adhesion

σ = surface energy (Depending on the size of the droplet)

θ = Contact angle between the liquid and the solid surface.

5.2 Goniometer overview: The Contact Angle Goniometer consists of a light source, camera, microscope, environmental chamber; etc. The environmental chamber

was maintained at high temperatures to simulate the field conditions. The Goniometer is connected to the computer and the image of the contact angle between the asphalt and the substrate captured by the camera is processed by computer. The DROP image software automatically calculates the contact angle of the captured image. Figure 5.2 & 5.3 shows the Contact Angle Goniometer.

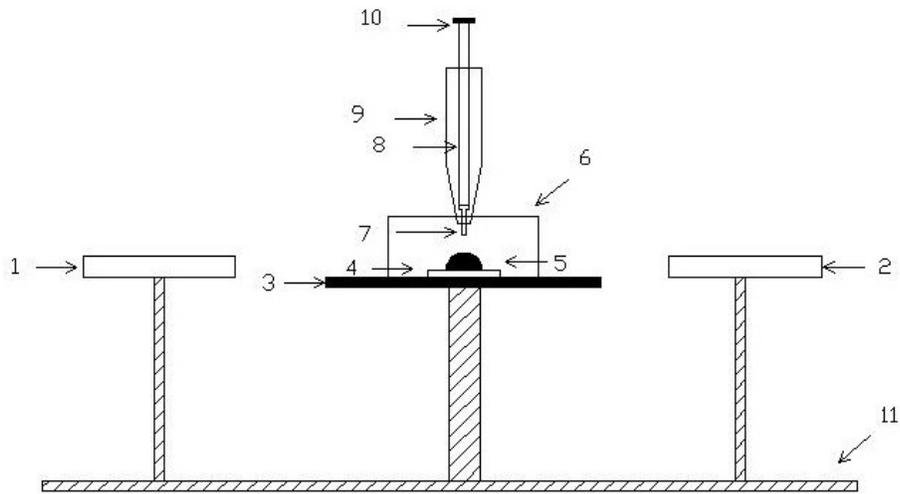


Figure 5.2 The sketch of contact angle goniometer

1. Light Source 2. Microscope Camera 3. Platform 4. Solid Plate 5. Liquid Droplet
6. Environmental Chamber 7. Metal Needle 8. Micro syringe (1cc) 9. Elevated Temperature Syringe Chamber 10. Piston 11. Base of Instrument

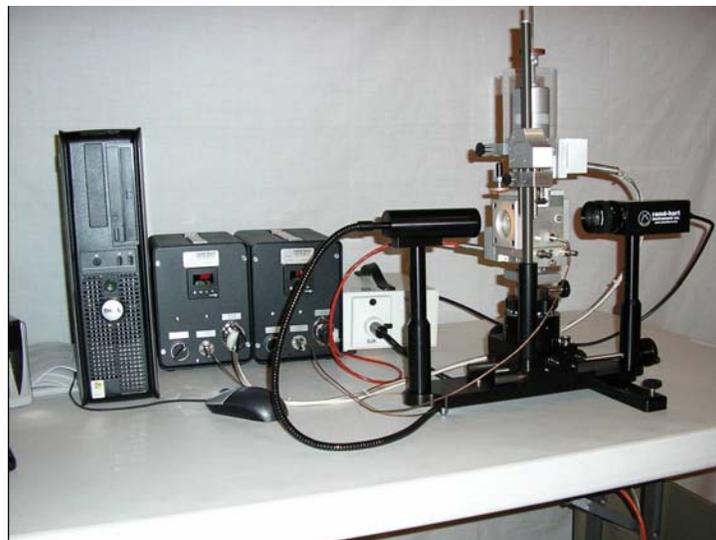


Figure 5.3 Contact angle goniometer

5.3 Objectives and Task: The main objective of this work was to determine the effect of prolonged heating and the anti-strip additive over the adhesive strength between asphalt and quartz/glass slide. This was attempted by observing the change in the contact angle between the asphalt and quartz/glass slide with different quantities of anti-strip additive (added to asphalt) and at different durations of prolonged heating.

5.4 Materials: The materials that were supplied by the North Carolina Department of Transportation were PG 76-22 asphalt and LOF 6500 (Anti-Strip Additive). Quartz slides and glass slides were obtained from Chemglass (Glass Supply Company). The chemical composition of the microscopic glass slides that were obtained from the Chemglass is borosilicate. The dimension of the quartz slide and glass slide that were used in the experiments is 1-inch by 1-inch with thicknesses of 1/8 of an inch and 1/16 of an inch respectively.

5.5 Preliminary tests performed with Goniometer: Various tests have been conducted to determine the contact angle of water, anti-strip additive (LOF 6500 and Morelife 2200) and asphalt with respect to the glass slide and quartz slide. A brief summary of the procedure followed in order to determine the contact angle is described as follows. It should be noted that the contact angle of water was determined at 25° C and for asphalt and anti-strip additive the temperature of the environmental chamber was maintained at 135° C.

1. The designated temperature (as mentioned above) in the environmental chamber and the micro-syringe chamber are controlled during the test process.
2. The micro-syringe was used to place a drop of water/anti strip additive over the slide (quartz/glass). The quantity of the droplet was controlled by the micro-syringe. The quantity of the asphalt droplet was 0.01 ml and the water and anti-strip additive droplet was 0.02 ml.
3. The slide was placed inside the environmental chamber. It was left for 15 minutes to reach temperature equilibrium.
4. The platform was checked for the horizontality. To obtain this condition, the tilt of the platform was set to zero.

- The contact angle of the liquid with respect to the slide was measured automatically with the help of the DROPimage software.

The captured pictures of water droplet on quartz and glass slides measured at 25°C are shown in Figure 5.4 and 5.5. The contact angle values are 36.2° and 61.1° respectively. The contact angle value for water-glass is in good agreement with the values observed in the published research [13, 15]. The captured pictures of asphalt droplet on quartz and glass slides at 135°C are shown in Figure 5.6 and 5.7. The contact angle value is 8.2° for asphalt droplet on glass slide, but it is too small to be measured for asphalt droplet on quartz slide.

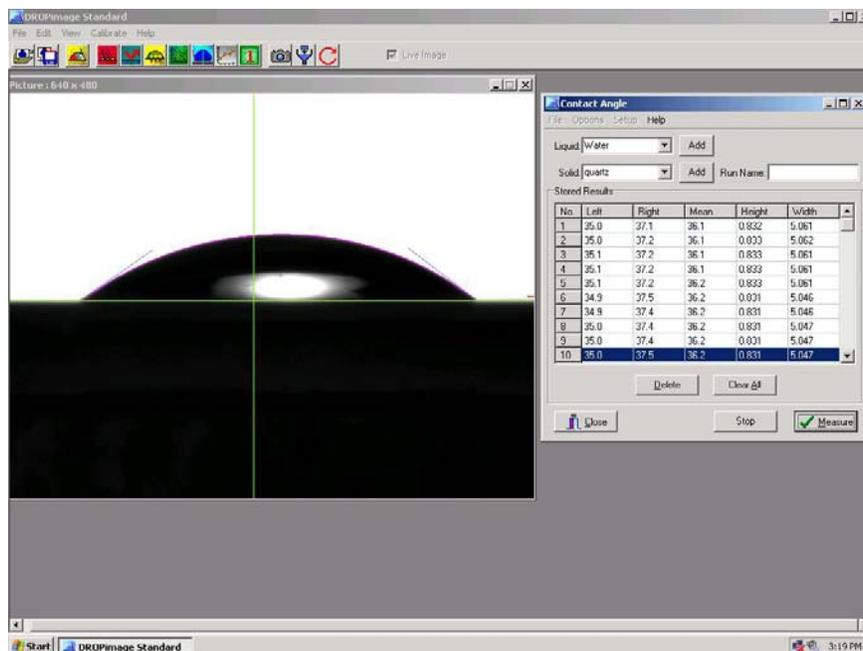


Figure 5.4 Captured Picture of Water Droplet on the Microscope, Quartz Slide at 25°C

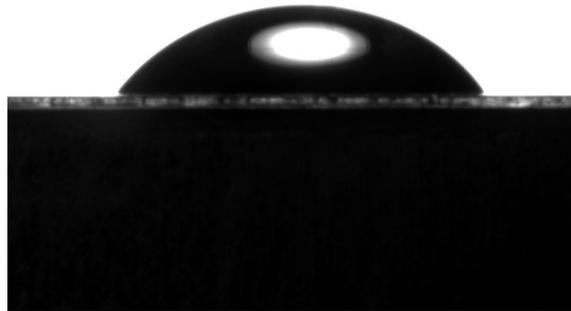


Figure 5.5 Captured Picture of Water Droplet on the Microscope, Glass Slide at 25°C

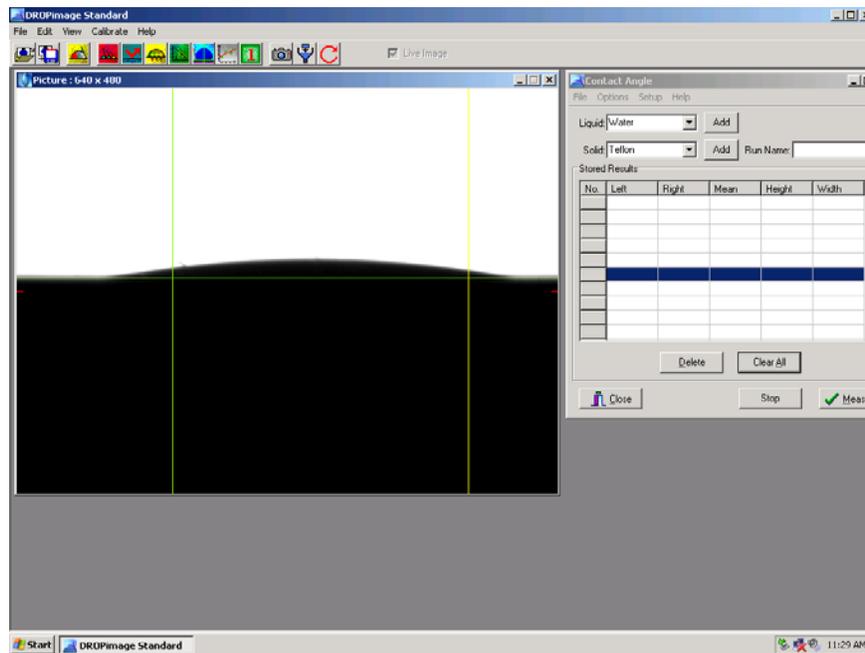


Figure 5.6 Captured Picture of Asphalt Droplet on the Microscope, Quartz Slide at 135°C

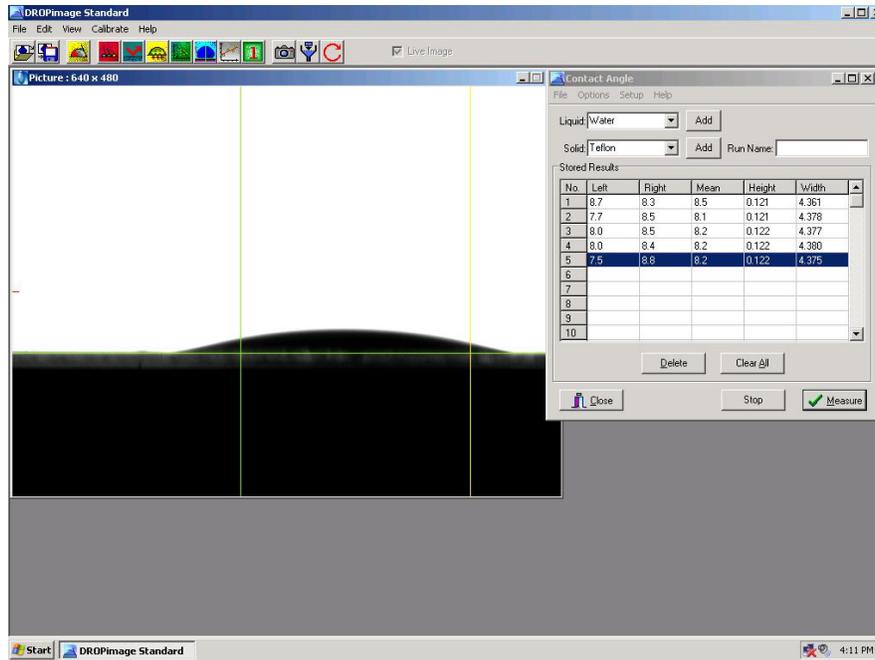


Figure 5.7 Captured Picture of Asphalt Droplet on the Microscope, Glass Slide at 135°C

5.6 Standardization of the size of Asphalt droplet: The primary problem that was encountered was the volume of the droplet of asphalt that was placed over the glass slide or quartz slide. The variation of the quantity of asphalt for each test led to large variations in the contact angle measurements. Hence, to control the volume of the droplet a Teflon mold similar to that used for DSR test was used (Figure 5.8) that could control the volume of the droplet.

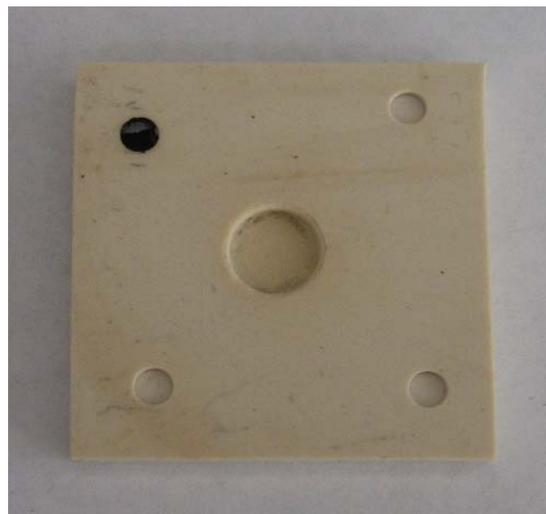


Figure 5.8 Mold designed to obtain reproducible quantities of asphalt droplet

5.7 Test Procedure: A procedure was developed to determine the contact angle of the asphalt with an aggregate slide as detailed in the following section.

Preparation of samples: A summary of the various steps taken in the preparation of the sample and the performance of the test is described as follows.

Apparatus required: Goniometer setup, steel scale, torch, cleaning cloth, citra-solv spray, methanol, and de-ionized water.

A) Preparation of the sample:

1) Preparation of the glass/quartz slide:

- a. The quartz/glass slide was taken and first cleaned with soap.
- b. The quartz/glass slide was cleaned with de-ionized water (distilled water) followed by methanol.
- c. The cleaned glass/quartz slides were carefully placed on a clean aluminum foil (one per each piece of foil) and were neatly wrapped.
- d. The neatly wrapped aluminum foils with the glass/quartz slides were put in an oven that was pre-set at a temperature of 105° C.
- e. The glass/quartz slides were removed from the oven after one hour.

Note: The glass/quartz slides were un-wrapped from the aluminum foil only at the time of testing.

2) Preparation of the asphalt with 0.5% anti-strip additive:

- a. The pure asphalt was placed in an oven at a temperature of 150° C.
- b. The anti-strip additive was measured for a quantity of 0.5 gm and is added to a clean container.
- c. The pure asphalt that was placed in the oven is allowed to be in the oven until it was workable (30-45 minutes).
- d. The liquid asphalt was taken out of the oven and a 100 Gms was added to the container containing the anti-strip additive and was stirred vigorously.
- e. Hence the asphalt with 0.5% anti-strip additive is prepared for the testing purpose.

3) Preparation of the Mold:

- a. The mold is made of silicon rubber in which asphalt doesn't stick to it.

- b. The mold is thoroughly wiped with a tissue paper to avoid any dust in the cavity.
- c. The mold has to be stored in a clean place to avoid the intrusion of dust in the sample.

4) Preparation of the specimen:

- a. The glass slide that was prepared was unwrapped from the aluminum foil.
- b. The asphalt sample that was prepared was placed in the oven at temperature of 150⁰ C for a period of 30-45 minutes before the sample was extracted.
- c. The asphalt was thoroughly mixed for good dispersion of the LOF-6500 (anti-strip additive).
- d. The asphalt was extracted from the container with the help of a syringe and needle and it is placed into the mold. Slightly additional asphalt is added to the mold as shown in Figure 5.8.
- e. A steel scale was heated with the help of a torch for a period of 10-15 seconds.
- f. The stainless steel scale was used to wipe the excessive asphalt over the mold.
- g. Hence the asphalt present inside the cavity of the mold solidifies and forms in the form of a pellet.
- h. The asphalt pellet was extracted with a sharp needle and it is placed over the glass /quartz slide.

B) Preparation of the instrument:

- a. The instrument has to be calibrated prior to the usage.
- b. The tilt of the instrument has to be checked at every measurement. (The level of the base tilt should not exceed 0.2 (constant)).
- c. The environmental chamber is set to the initial required temperature.

5.8 Procedure for Goniometer test: The following procedure has been followed to perform the Goniometer test.

1. The prepared sample was placed in the environmental chamber that was pre-set for the initial temperature.
2. The sample was left at the initial temperature for 15 minutes for the stabilization of the temperature.

3. Then the contact angle of the asphalt with the glass/quartz slide was measured.
4. The temperature was increased at the rate of 5⁰ C and measurements were taken for every 15 minutes with the help DROPimage software.

Following tests were performed in succession to the preliminary tests to obtain a better understanding over some parameters that affect the contact angle.

5.9 Tests Results and discussion: The following tests have been done to determine the effect of prolonged heating over adhesive strength between asphalt and glass/quartz slide.

Test 1: To determine the importance of cleaning the quartz/glass slide with methanol: The results are summarized as below in Figure 5.9 and Table 5.1

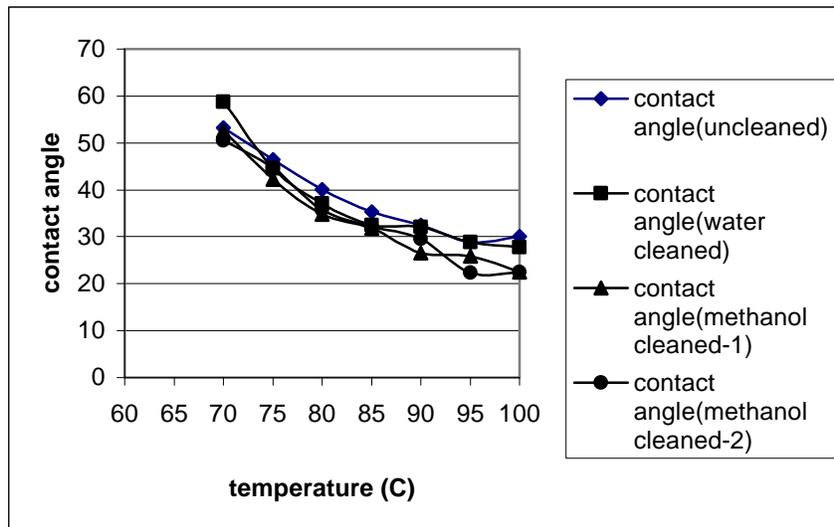


Figure 5.9 Results showing the importance of cleaning of Quartz slide with methanol

Table 5.1 Results showing the importance of cleaning of Quartz slide with methanol

Temperature (C)	contact angle (uncleaned)	contact angle(water cleaned)	contact angle(methanol cleaned-1)	contact angle(methanol cleaned-2)
70	53.2	58.7	52.6	50.5
75	46.5	44.5	42.2	44.7
80	40.1	37.1	34.8	35.7
85	35.3	32.5	31.8	32.2
90	32.5	32.1	26.6	29.5
95	28.8	28.9	25.9	22.3
100	30	27.7	22.5	22.5

Observations: As per the analysis of the results obtained, the contact angles obtained over the samples where the slides were cleaned by methanol were much closer to each other than the contact angles obtained from the samples for which the slides were uncleaned and cleaned by water. This showed the importance of cleaning of slides with methanol (glass/quartz/aggregate).

Test 2: This test was conducted to determine the validity of the result that was obtained from the TSR test. As per the results that were obtained from the TSR with respect to the prolonged heating, the adhesive strength (as measured by the tensile strength ratio) reduces when the asphalt is subjected to prolonged heating. This phenomenon has been tested by the application of Contact Angle Goniometer over asphalt (with 0.5% LOF) and a quartz slide. The results are summarized as below in Figure 5.10 and Table 5.2.

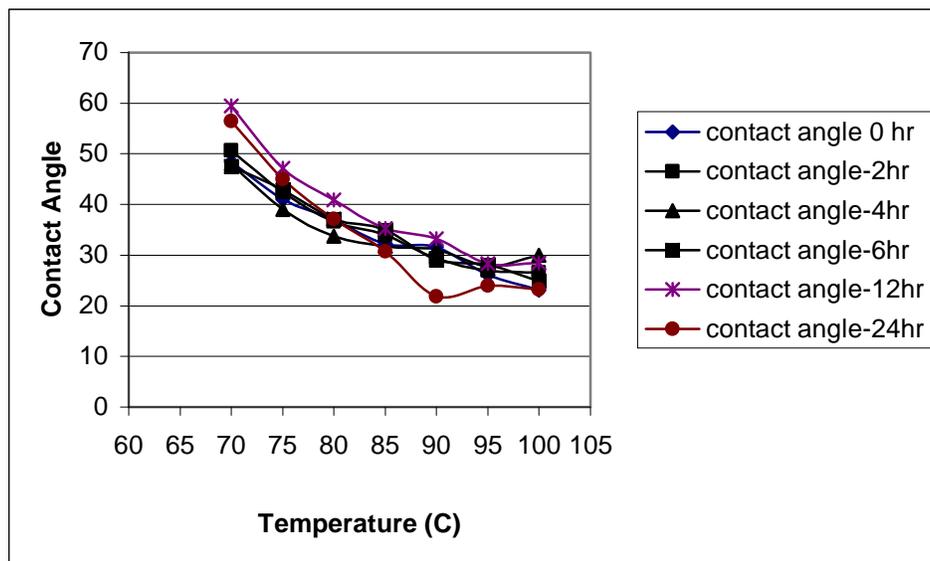


Figure 5.10 Results obtained that validate TSR test results

Table 5.2 Results obtained in the test that was performed to validate TSR test (asphalt with 0.5% LOF 6500)

Temperature (C)	contact angle-0 hr	contact angle-2 hr	contact angle-4 hr	contact angle-6 hr	contact angle-12 hr	contact angle-24 hr
70	48.4	50.7	48	47.6	59.5	56.4
75	41	42.4	39	42.9	47.2	45
80	37	36.6	33.7	37.1	40.9	37.1
85	32.3	34	31.7	35	35.2	30.7
90	31.6	29.4	31.1	29.1	33.3	21.8
95	26.1	27	27.9	28.1	28.3	24
100	23.1	26.6	30	24.9	28.5	23.2

Observations: Based on the principle of Work of Adhesion, as the contact angle between the liquid and the substrate is less, the adhesive bond strength between them is more. As per the analysis of the above results, as the heating time increased at the temperature of 70⁰ C, the contact angle also increased. This result obtained at 70⁰ C is in agreement with the TSR result that is as the prolonged heating duration increased the adhesive bond strength between asphalt-aggregate reduced. This agreement of results with the TSR could not be observed at other temperatures. However, it may be noted that there are some anomalies in preliminary results.

Test 3: Importance of Anti-Strip Additive in Asphalt: The determination of contact angle at various temperatures ranging from 95-150⁰ C was conducted between asphalt (with and without anti-strip additive) and glass slide. The following results have been obtained. The results obtained in this test are summarized below in Figure 5.11 and Table 5.3.

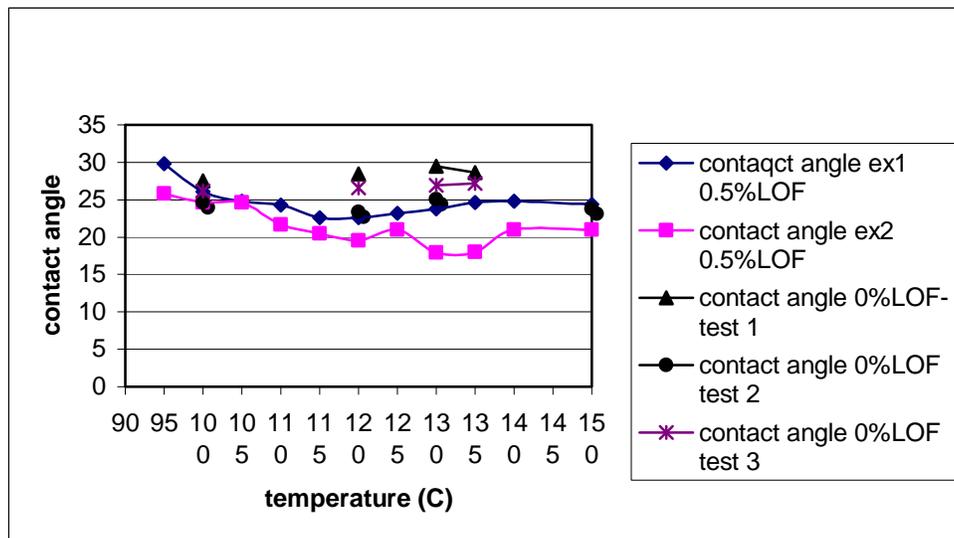


Figure 5.11 Results showing the importance of Anti-Strip Additive in asphalt

Table 5.3 Results showing the importance of Anti-Strip Additive in asphalt

Temperature (C)	contact angle test example1 0.5%LOF	contact angle test example 2 0.5%LOF	contact angle 0%LOF-test 1	contact angle 0%LOF test 2	contact angle 0%LOF test 3
95	29.8	25.8			
100	26.1	24.6	27.5	24.6	26.2
105	24.8	24.6			
110	24.3	21.7			
115	22.6	20.5			
120	22.6	19.5	28.5	23.4	26.6
125	23.2	21			
130	23.8	17.9	29.5	25.1	26.9
135	24.6	18	28.6		27.2
140	24.8	21			
150	24.4	21		23.8	

Observations: As per the analysis of the above obtained results, it can be observed that the presence of anti-strip additive increases the adhesive strength (as the observed contact angles are in general, less than the contact angle obtained between asphalt and glass without additive).

Test 4: Effect of Volume of the droplet: This test was performed to determine the importance of the control of the volume of asphalt droplet. The test was performed with asphalt and quartz slide. The results obtained in this test have been summarized as below in Figure 5.12 and Table 5.4.

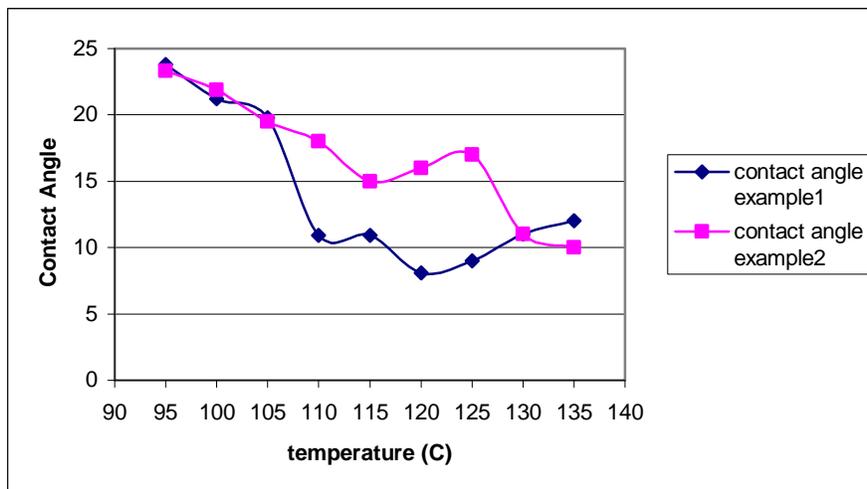


Figure 5.12 Results to show the importance of volume control of droplet.

Table 5.4 Results to show the importance of volume control of droplet.

Temperature (C)	contact angle example 1	contact angle example 2
95	23.8	23.3
100	21.2	21.9
105	19.8	19.5
110	10.9	18
115	10.9	15
120	8.1	16
125	9	17
130	11	11
135	12	10

Observations: As per the analysis of the above results, the contact angles obtained at 95⁰ C, 100⁰ C, 105⁰ C, 130⁰ C and 135⁰ C for the examples1 and 2 are close to each other. Such observation could not be obtained at the temperatures ranging from 110⁰ C to 125⁰ C. Based on the contact angle that was obtained at the initial temperature, it can be concluded that the control of volume helps to obtain reproducible results. As per the primary purpose of this test, the asphalt should be subjected to prolonged heating for specified durations. The asphalt is heating to the desired duration prior to the preparation of the sample. The results obtained at the higher temperatures could be discarded, as the duration of prolonged heating condition is not satisfied as the sample is subjected to different temperatures for longer time.

Analysis over the performed preliminary tests:

Tests 1 to 4 have been performed over a range of temperatures. The asphalt samples were subjected to prolonged heating prior to the preparation of the sample. It was noticed that the samples that were already subjected to the required prolonged heating duration at the required temperature, should not be further heated in the Contact Angle Goniometer during the determination of the contact angle. Hence, test 5 and 6 were performed to determine the test time (the duration required for the stabilization of the sample that is placed in the environmental chamber). Test 7 was an attempt to replicate the results that were obtained in test 6. The importance of the volume of the asphalt droplet was determined from the results obtained from the preliminary tests. Hence, the mold that could produce replicates was designed. To reconfirm the performance of the mold and the effect of the volume of the droplet over the change in the contact angle, test 8 and test 9

were performed with asphalt (with and without additive) against quartz slide. Furthermore, the captured pictures of asphalt with quartz/glass slides at various temperatures that are obtained from the above tests were observed and 100°C was selected as the test temperature, as the curvature obtained at that temperature was smooth and also the temperature was sufficient for the activation of the asphalt. Figure 5.13 & 5.14 shows the captured pictures of asphalt droplet after testing at 135°C and 100°C respectively. Further tests performed are summarized in the Figure 5.15 as shown below.

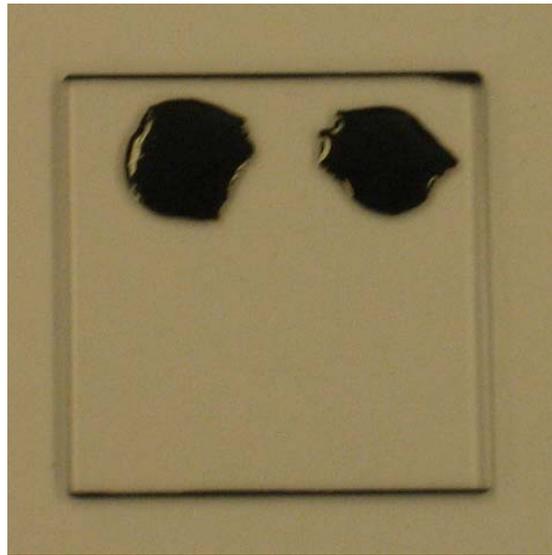


Figure 5.13 The sample after testing for contact angle at 135°C temperatures



Figure 5.14 The sample after testing for contact angle at 100°C temperatures

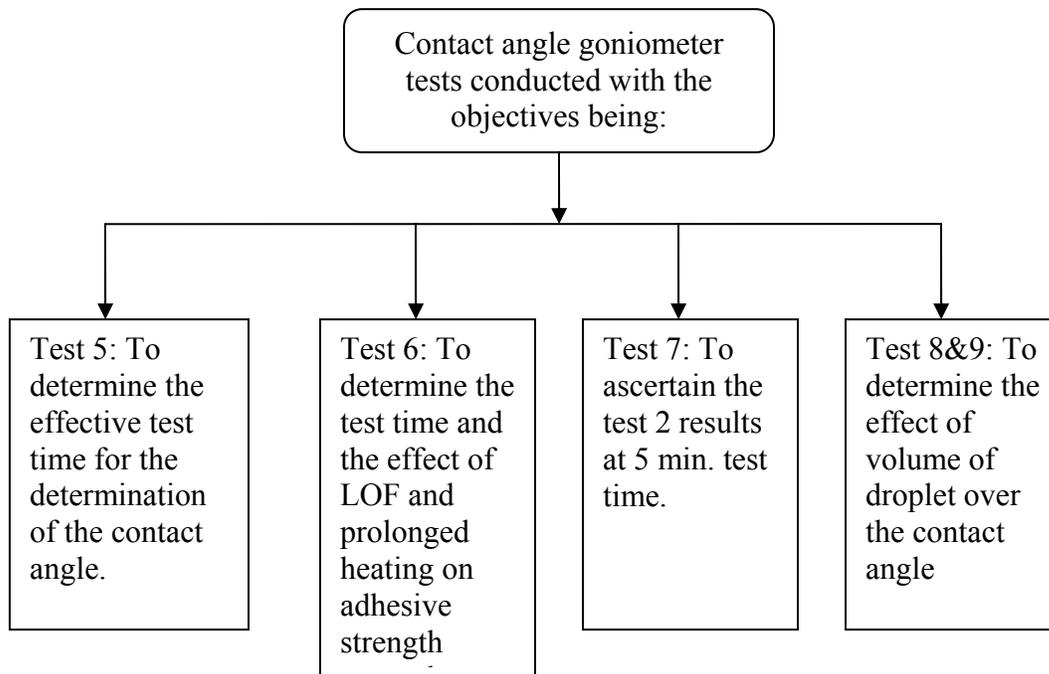


Figure 5.15 Summary of the further tests conducted

Test 5: This test was conducted to determine the contact angle between asphalt (with 0.5%LOF), and glass slide at different durations of heating (from 0 to 12 hours) for a test time of 5 minutes, 10 minutes and 15 minutes. The test was conducted to observe the change in contact angle at various test times to determine desirable test time. The data are summarized as below in Table 5.5 and Figure 5.16.

Table 5.5 Results of measured contact angles at 5, 10 and 15 minute test times at 100⁰ C

Duration of heating(hrs)	contact angle@ 0.5%LOF @5 min	contact angle@ 0.5%LOF @10 min	contact angle@ 0.5%LOF @15 min
0	30.8	25	23.8
2	31.2	26.4	25.8
4	32.8	28.2	26.3
6	35	28.2	27.1
12	47.2	36.8	34.4

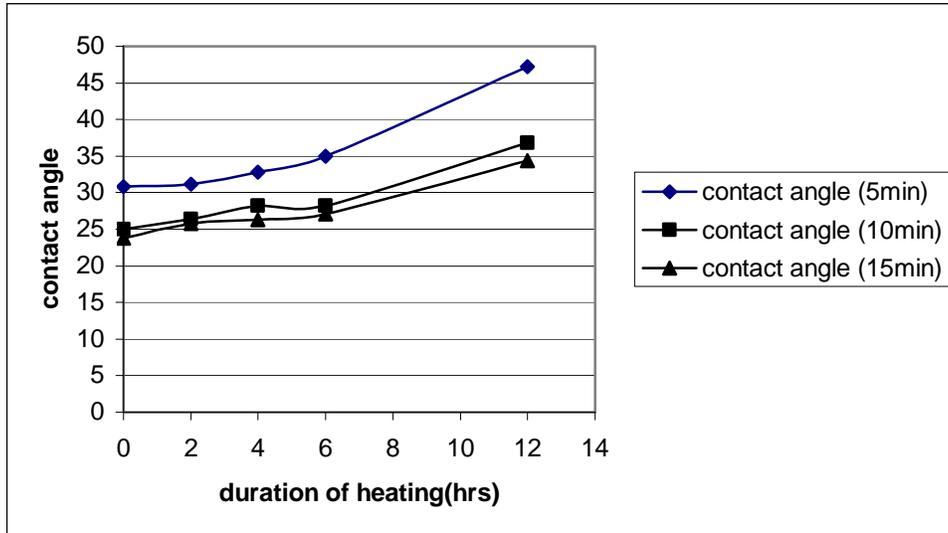


Figure 5.16 Results showing Contact angle determination at various test times

Discussion: The data show that the 5 minute test time duration gives consistently higher values than the 10 and 15 minute test time durations. No significant difference can be observed between the 10 and 15 minute test time duration. Moreover the trend in the data is very similar for all testing time duration.

The angles were observed to increase with the prolonged heating duration, and this result is in conformance with the TSR result where declining TSR values were observed. Note that larger contact angle corresponds to lower adhesive strength. In this particular test it was observed that the TSR values reduce as the value of the contact angle increases. Although, this trend was observed during this test, it could not be repeated in the following tests that were done. To the contrary, it was observed that no correlation could be ascertained between the contact angle and the results of the TSR test. In order to minimize the excessive heating of the asphalt sample, 5 minute test duration was chosen for further testing.

Test 6: This test was conducted to determine the contact angle between asphalt (with 0.5%LOF, 0.75%LOF, and 1.0% LOF) and quartz slide at different durations of heating (0, 2, 4, 6 and 12 hours) for a test time of 5 minutes and 10 minutes in the environmental chamber which was setup at 100⁰ C. The 15 minute test time duration was dropped from the test as it was found that there was no significant difference in the contact angle

between the 10 and 15 minutes test time durations (as per the results obtained from test 5).

This test was done to observe the change in contact angle at various test times and also to determine the combined effect of anti-strip additive and prolonged heating over the adhesive strength between asphalt (with anti-strip additive) and quartz slide. The data are summarized in Table 5.6 and Figure 5.17.

Table 5.6 Contact angles at 5 and 10 minute test times with various quantities of LOF

Duration of heating(hrs)	contact angle 5 min (0.5%L)	contact angle10 min (0.5%L)	contact angle 5 min (0.75%L)	contact angle10 min (0.75%L)	contact angle 5 min (1.0%L)	contact angle10 min (1.0%L)
0	33.1	32.4	30.4	27.8	29.3	25.7
2	38.6	35	31.7	27.2	29	26.9
4	32.5	29.3	35.4	32.3	27	25.7
6	27.9	27	31	25.9	29.8	25.6
12			27.2	23.3	27.2	29.3

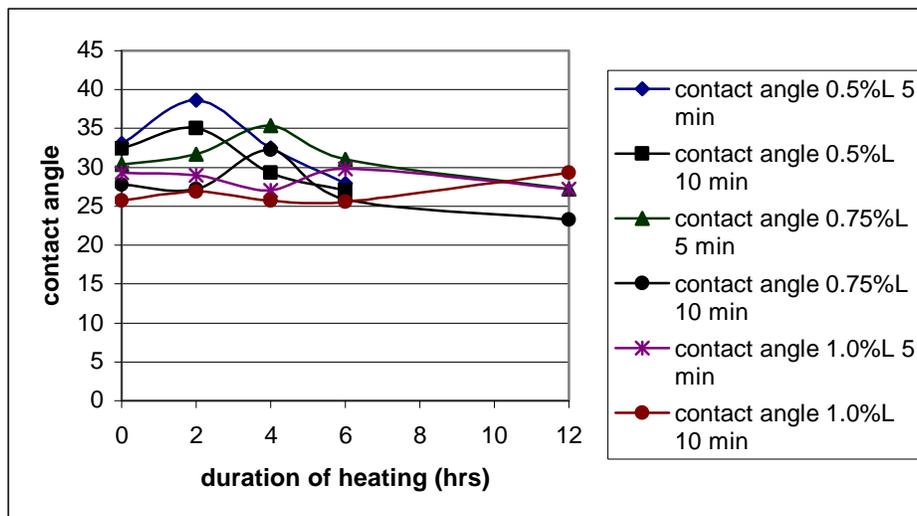


Figure 5.17 A plot between contact angle and duration of heating for various samples

Discussion: The data show that there seems to be a variation in the trend observed between the results obtained at 5 minute, 10 minute, similar to the trend observed in test 5.

When a sample is considered (say 1.0%LOF for test duration 5 min), the contact angles did not seem to change for different durations of heating. A non uniform pattern in the measured contact angles at different durations of heating for 0.5%LOF and 0.75%LOF specimens can be observed from the results obtained. This shows that the effect of prolonged heating over asphalt cannot be measured using a Contact Angle Goniometer.

When the contact angles are observed at a particular duration of heating for different amounts of LOF, a variation can be observed in the trends. E.g., the contact angles for the durations 0 and 2 hours are in a descending order with respect to increasing LOF content; whereas when we observe the 4 and 6 hour heating duration, the contact angle at 0.75%LOF is higher than at 0.5%LOF and 1%LOF specimens. Hence based on the results obtained it can be concluded that Goniometer could not differentiate the LOF amount with any certainty nor could it differentiate effect of prolonged heating. This will be further shown through the test results obtained in test 7.

Test 7: This test was conducted to ascertain the results (replicate test) obtained in test 6 at test time of 5 minutes where the temperature of the environmental chamber was set at 100⁰ C.

Three samples of asphalt containing 0.5%, 0.75%, and 1.0% of anti-strip additive (LOF) respectively were prepared and subjected to prolonged heating for duration of 0, 2, 4, 6 and 12 hours prior to the preparation of the specimens.

The results are summarized as shown below in Table 5.7 and shown in Figure 5.18:

Table 5.7 Contact angle at 5 minute test time at various quantities of LOF 6500

Duration of heating(hrs)	contact angle 0.5%LOF@100C-5min	contact angle0.75%LOF@100C-5min	contact angle 1.0%LOF@100C-5min
0	18.8	16.1	18.4
2	25	21.4	21
4	22	23.4	21.9
6	18	26.2	22
12	17.6	13.8	17.3

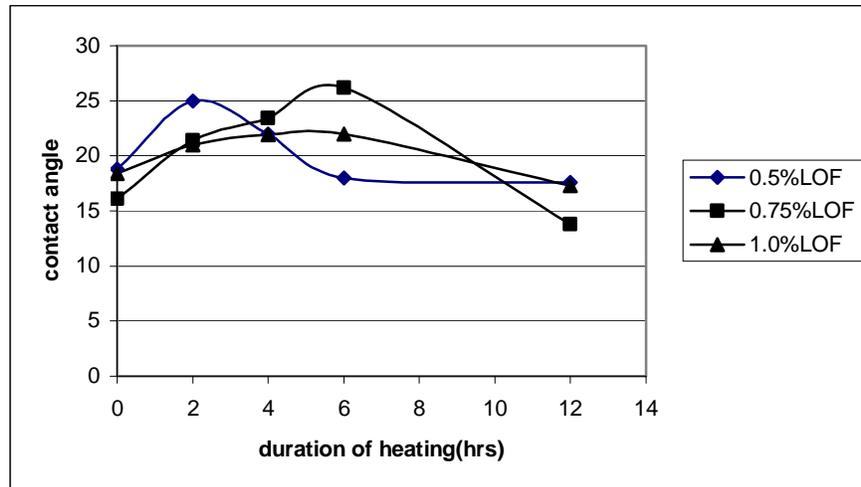


Figure 5.18 A plot between contact angle and duration of heating for various samples

Discussion: The objective of this test was to replicate the results of test 6. However, it can be seen that test results are different from those obtained in test 6. Therefore, there was no replicability in the test results. This could be due to many factors associated with the testing protocol or due to the complex interaction of various parameters like the mixing of the anti-strip additive with asphalt, quantity of the anti-strip additive, the size of the droplet, the procedure followed to clean the glass/quartz slide, duration of the test time and the prolonged heating time. It would therefore appear that our testing protocol was not completely standardized as the results obtained are highly variable. Therefore, test number 8 was devised to further refine the analysis protocol including looking at the size of the droplet.

Test 8: This test was conducted to determine the effect of prolonged heating over adhesive strength of virgin asphalt. Replicate tests were performed over asphalt with quartz slide in this test. The treatment procedure that was followed in the preparation of the sample (asphalt with a particular percentage of anti-strip additive) is exactly implemented to the pure asphalt in order to obtain a standard procedure. The results obtained were also observed for the volume of the droplet, and found that there were variations in the volume of the droplet when compared to the volume of the mold. Observing the variations in the volume of the droplets, we have attempted to correlate the volume of droplet and the contact angle.

The calculations of the volume of the asphalt from the results obtained from the goniometer are based on the mathematical calculations for the determination of the volume of the segment of a sphere, where the height of the segment is h and the radius of the sphere segment is r_1 .

The volume of the segment is given by $V = (\pi/6)*h*(3* r_1^2 + h^2)$ Equation 5.3

The actual volume of the mold is 0.00796 cc.

The results that were obtained are as summarized below in Table 5.8.

Table 5.8 Results showing the contact angles and volume adjustments of droplet

Sample Number	Duration of heating (hrs)	Contact angle (pure asphalt)@5 min	Height (h) cm	Total Width(2*r1) (cm)	Width (r1)	Volume (cc)	Volume % Difference
1	0	30.5	0.0615	0.5003	0.25015	0.00616	22.61637
2	0	31.7	0.0678	0.5478	0.27390	0.008144	-2.30681
3	0	31.8	0.0669	0.5343	0.26715	0.007648	3.920424
4	0	33.4	0.0652	0.5288	0.26440	0.007296	8.336711
1	2	31.4	0.0656	0.5484	0.27420	0.007886	0.926754
2	2	33.4	0.0634	0.4718	0.23590	0.005669	28.78236
3	2	30.3	0.0641	0.5234	0.26170	0.007026	11.73781
1	4	32.5	0.0598	0.4529	0.22645	0.004923	38.1506
2	4	32.4	0.0676	0.5391	0.26955	0.007868	1.156966
3	4	32.9	0.0619	0.4709	0.23545	0.005508	30.80255
4	4	38.4	0.0796	0.5351	0.26755	0.009204	-15.6275
1	6	31.5	0.0645	0.5095	0.25475	0.006708	15.72857
2	6	33.8	0.0679	0.5074	0.25370	0.007021	11.80005
3	6	32.5	0.0656	0.5378	0.26890	0.00759	4.648694
4	6	32.9	0.0706	0.5441	0.27205	0.008382	-5.30611
1	12	31.4	0.063	0.506	0.25300	0.006458	18.87095
2	12	31	0.066	0.5236	0.26180	0.007248	8.946321
3	12	31.2	0.0683	0.5363	0.26815	0.007872	1.104194
4	12	32.5	0.0694	0.5324	0.26620	0.007891	0.867746

Discussion: When the replicate sets of test results for prolonged heating are compared, significant differences in the angle were not observed. That is, differences in volume (as used in this study) did not significantly affect the contact angle. As per the data, when we consider a particular duration of heating say , 0 hour (from Table 5.8) though the volume of the droplet varied in the range of -2.3% to 22.6% from the original volume of the

mold, the angle did not vary significantly. Similarly this trend can be observed at all the durations of heating in Table 5.8.

Another observation that can be made is that as the asphalt hardens (due to prolonged heating) the contact angle does not change appreciably.

Test 9: As per Test 8 result a significant change in the contact angle was not observed with the change in the volume. An attempt was made to determine if there is any effect of volume of the droplet in the presence of anti-strip additive over the measured contact angle. Contact angle test was performed for asphalt with 0.75 % LOF. This test was conducted to determine the effect of prolonged heating over adhesive strength of asphalt with 0.75% LOF. Replicate tests were prepared over asphalt containing 0.75%LOF with quartz slide in this test. The calculations of the volume of the asphalt from the results obtained from the goniometer are based on the mathematical calculations as given in Equation 5.3. The results obtained are as summarized below in Table 5.9.

Table 5.9 Results showing the contact angles and volume adjustments of droplet

Sample number	Time of exposure	Contact angle (asphalt with 0.75%lof)	Height(h) in cm	Total width (2*r1) cm	Width (r1) in cm	Volume in cc	% Volume Difference
1	0	32.7	0.063	0.537	0.268	0.0072	9.17
2	0	31.2	0.064	0.559	0.279	0.008	0.07
3	0	38	0.057	0.453	0.226	0.0046	41.7
4	0	34.9	0.057	0.456	0.228	0.0047	40.92
1	2	26.2	0.059	0.554	0.277	0.0071	10.21
2	2	28.6	0.058	0.531	0.265	0.0065	17.93
3	2	29	0.06	0.547	0.273	0.0071	10.33
1	4	29.1	0.06	0.567	0.283	0.0077	3.13
2	4	28.9	0.063	0.511	0.256	0.0066	16.99
3	4	30.6	0.061	0.546	0.273	0.0072	9.17
4	4	29.8	0.062	0.574	0.287	0.0081	-1.48
1	6	39.2	0.062	0.517	0.258	0.0066	17.18
2	6	35.3	0.09	0.53	0.265	0.0102	-28.56
3	6	34.3	0.069	0.483	0.241	0.0065	18.71
4	6	35.5	0.059	0.539	0.269	0.0069	13.74
1	12	38.6	0.077	0.558	0.279	0.0096	-20.48
2	12	33.3	0.061	0.58	0.29	0.0082	-2.51
3	12	39.3	0.063	0.54	0.27	0.0073	8.13
4	12	31.8	0.051	0.492	0.246	0.0049	38.1

Discussion: When we observe the 4 hour duration though the variation of the volume of the droplets varied from -1.48% to 16.99% from the original volume, a significant change in the measured contact angle is not observed. But when we observe the results that are obtained in the 12 hour duration, as the volume of the droplet varied from -20.48% to 38.10% from the original volume, the angle varied from 31.8 to 39.3. A pattern could not be observed in this variation of the contact angle with the change in the volume of droplet (either increase of contact angle with increase in volume or decrease in contact angle with increase of volume of droplet). Similar results were obtained with the virgin asphalt that were presented earlier. No correlation between the volumes of the droplet to that of the corresponding measured contact angle was seen.

5.10: Summary and conclusion: The following conclusions can be drawn from the results obtained:

- 1) The size of the asphalt droplet should be as small as possible to avoid the effect of gravitational force over the droplet that might affect the contact angle.
- 2) The glass/quartz slides should be cleaned carefully to avoid dust over it, which could affect the contact angle.
- 3) As per the results obtained from the Test 3, the contact angle values were less for the combination of asphalt containing additive with glass slide than the virgin asphalt-glass slide. This shows that the presence of anti-strip additive increases the adhesive strength.
- 4) Test 4 shows the importance of the control of volume. Accuracy in the control of volume of the asphalt droplet could not be achieved to the desired level, as the stiffness of the asphalt increased due to prolonged heating. The increased stiffness did not allow the asphalt to exactly occupy the cavity in the mold.
- 5) At the end of the test 4, the importance of the standardization of the test time and the test temperature was noticed. Tests 5 & 6 were performed and the test time was determined to be 5 minutes and the test temperature was determined to be 100⁰ C

Note: Test time: The duration required for the stabilization of the sample that is placed in the environmental chamber before measuring the contact angle. Test temperature is the temperature in the environmental chamber while testing.

6) As per the results obtained from Test 5, as the prolonged heating duration increases the contact angle between the asphalt and quartz slide increases showing that there is a reduction in the adhesive strength.

7) As per the results obtained from Test 5, the results are correlate well with the results that were obtained for the TSR test. Due to prolonged heating, loss of the anti-strip additive occurs resulting in the reduction of the adhesive strength between the asphalt-aggregate slides. This is showed in the contact angle test, as there is an increase in the contact angle with the increase in the prolonged heating duration. On the contrary, these results could not be replicated in the following tests, showing the degree of complexity of the performance of the contact angle test over asphalt.

8) Based on the results obtained in Test 6, it can be concluded that Goniometer could not differentiate the LOF amount with any certainty nor could it differentiate effect of prolonged heating.

9) Tests 8&9 show that no correlation was observed between the volume of the droplet and the change in the contact angle between the asphalt-quartz/glass slides.

10) A high variability in the results was obtained from the various tests performed. This could be due to many factors associated with the testing protocol or due to the complex interaction of various parameters like the mixing of the anti-strip additive with asphalt, quantity of the anti-strip additive, the size of the droplet, the procedure followed to clean the glass/quartz slide, duration of the test time and the prolonged heating time.

Hence, based on the current conditions and the control we have over the various parameters Goniometer is not a good instrument to determine the effect of prolonged heating and the anti-strip additive over the adhesive strength between asphalt-aggregate.

5.11 Recommendations: The following recommendations were made based on the test results obtained:

1. Further standardization of the testing protocols like the mixing of the anti-strip additive to asphalt, cleaning of the quartz slide, control of the volume of the droplet, etc have to be achieved. The volume of the droplet should be small to avoid the effect of gravitational force over the contact angle. The only governing force over the contact angle should be the surface tension between the various media. The complex interactions between them have to be understood to obtain better results.

2. Hence based on the control that we have currently over the contact angle test, we would not recommend contact angle test as a good test to determine the effect of prolonged heating and anti-strip additive over the adhesive strength between asphalt-aggregate.

6. Pull-Off Test Using PATTI Device

6.1 PATTI Background: Adhesion testers are used, to determine the bond strength between the adhesive and the surface (for e.g. asphalt and aggregate). Formerly, there existed hand operated mechanical devices that were used to determine the adhesive bond strength of the adhesive to the aggregate/glass plate. The disadvantage with the hand operated mechanical device is that the force applied to pull the adhesive from the surface is non-uniform. So the reliability over the result was not considerable [16]. Pneumatic Adhesion Tensile Testing Instrument (PATTI) was initially developed by the National Institute of Standards and Technology (NIST). Pneumatic Adhesion testers have the advantage over the mechanical devices that the force applied to cause the failure in the bond between the adhesive and the surface is uniform [8]. The fundamental dimensions for the pull off tests is M/LT^2 , where M, L, T represent mass, length and time respectively [8]. The applied stress at the time of failure is taken as the measure of adherence [3]. The adhesive strength that is obtained using one instrument may not be comparable to the measurement made using another one. So it is prudent to mention the kind of instrument that has been used for the experiment in the final report. Even in case of using a particular instrument, the accuracy and precision are not obtained. As per the ASTM D4541 the variability of the results are presented in three forms , namely coefficient of variation , t-distribution of the sample and the allowable percentage difference of the obtained strength values. As per the ASTM specifications the range of the allowable deviations depends upon the kind of instrument used. The values could range from 25% to 58% [17].

Pneumatic adhesion testing instruments used in the field: ASTM D 4541-02 is designated to “Standard test method for the Pull-off Strength of coatings using Portable Adhesion Testers.” There are five instrument types that are designated as Method A-E that could be used to determine the adhesive strength of coatings over metal, concrete and wood. The test methods that are available for the determination of the adhesive strength of various coatings as determined by the ASTM Method are listed below:

Test Method A – Fixed Alignment Adhesion Tester Type -1.

Test Method B – Fixed Alignment Adhesion Tester Type -2.

Test Method C – Self Alignment Adhesion Tester Type -3.

Test Method D – Self Alignment Adhesion Tester Type -4

Test Method E – Self Alignment Adhesion Tester Type -5

Among these five available instruments, the method that is most suitable for the determination of adhesive strength between asphalt and aggregate is Type-D “Self-Alignment Adhesion Tester Type 4.” The pull off test that is determined, depends upon the material and the instrumental parameters. Figure 6.1 shows the Pneumatic Adhesion Tensile Testing Instrument (PATTI 110) [18].



Figure 6.1 Pneumatic Adhesion Tensile Testing Instrument

Parts of the Instrument: The main parts of the Pneumatic Adhesion testing instruments are portable adhesion tester, pull stub, pressure hose, meter and rubber mold. A brief explanation of each part of the instrument is summarized below.

Pneumatic Adhesion tester: The portable pneumatic adhesion tester is used to apply the perpendicular force over the adhesive. The adhesion tester is to be selected such that the expected value of the force should be intermediate of the range of the load values that can be applied by the tester. The range of the instrument that is suitable for the determination

of the adhesive strength between asphalt and glass/quartz slide is 3.5 MPa to 70 MPa. The load that is applied should be done smoothly and as continuous as possible at the rate less than 1 MPa/s (150 psi/s) so that the failure occurs within 100 seconds.

Pull Stub [19]: It is also termed as dolly. The pull stub is normally manufactured using a wide variety of metals including aluminum, carbon steel and stainless steel. The preparation of this part of the instrument for the test plays a vital role in obtaining the repeatability of the test. The preparation of the stub for the test includes three main activities namely, degreasing, abrasion and cleaning. Degreasing refers to the removal of any trace of oils and grease that might be due to the oils from the skin of the person handling the dolly. Abrasion refers to the alteration of the dolly surface, where the primary purpose is to increase the surface area for bonding. This increase of the area of the pull-stub surface increases the adhesive strength between the adhesive and the pull-stub. Cleaning is the removal of the loose particles that might be present on the surface. Twisting of the pull stub needs to be avoided, since it can cause tiny bubbles that might cause discontinuities in the testing. An aluminum dolly can be used for the purpose of testing and reuse of the dolly is generally not recommended.

Pressure hose: The pressure hose is connected to the pressure source and is connected to the piston. The pressurizing gas enters the pressure hose and is transferred to the detaching assembly. A vertical force is created perpendicular to the specimen till failure. The hose is also connected to a pressure rate controller that allows control over the pressure that is applied over the adhesive-substrate system. The pressure control should be opened at the rate of $\frac{1}{4}$ turn. The rate of pressure that has been used in this research is 0.005.

Rubber Mold: A rubber mold (8 mm diameter and 1mm depth) that is normally used in the DSR test is used in this test to control the thickness of the asphalt film over the quartz slide. The procedure to control the thickness of the asphalt film is explained in the section 6.4.

Meter: The load that would be applied over the specimen with the help of the pressure source is displayed over the meter screen. This value can be noted down and can be converted to the force (with units: MPa) using the standard charts or by using the formula that has been presented in section 6.5 (Equation 6.1).

6.2 Objectives and Task: The main objective of this task was to determine the effect of prolonged heating and the anti-strip additive over the adhesive strength between asphalt and quartz/glass slide (water conditioned for 24 hours at room temperature, 25⁰ C) using PATTI 110 device.

6.3 Materials: The materials that were supplied by the North Carolina Department of Transportation are PG 76-22 asphalt which was manufactured at Knoxville, TN and LOF 6500 (Anti-Strip Additive). Quartz slides were obtained from Chemglass (Glass Supply Company). The dimension of a quartz slide that were used in the experiments is 1-inch by 1-inch with thicknesses of 1/8 of an inch.

6.4 Use of DSR to control the Thickness of the Asphalt film: The primary problem that was encountered was the control of thickness of the asphalt film between the pull stub and the quartz slide. Due to the variation in the thickness of the asphalt film, the tensile strength required to de-bond the pull stub from the slide also varied. So to obtain the results that are comparable, between different specimens an attempt was made to control the thickness of the asphalt film. Dynamic Shear Rheometer (DSR) was used to prepare the specimens and the procedure followed to prepare replicate samples with identical thickness is summarized below. An adaptor was designed as shown in Figure 6.2, such that it fitted into the DSR in place of a standard plunger and the free end was mounted with the pull-stub (as shown in Figure 6.3) that could be lowered onto the quartz slide which was placed on the platform and had an asphalt pallet on it.



Figure 6.2 The adaptor designed to control the thickness of the sample

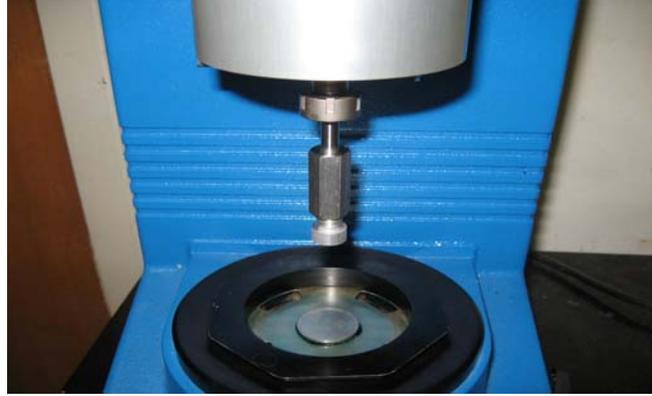


Figure 6.3 The adaptor fixed to the DSR with pull-stub at the free end

6.5 Preparation of specimens:

Various steps taken to prepare a specimen for testing is summarized as follows:

A) Preparation of the sample:

1) Preparation of the glass/quartz slide:

- a. The Quartz slide was taken and first cleaned with soap.
- b. Then the glass slide was cleaned with methanol followed by de-ionized water (distilled water).
- c. Then the cleaned aggregate slides were carefully placed on a clean aluminum foil (one per each piece of foil) and were neatly wrapped.
- d. The neatly wrapped aluminum foils with the slides were put in an oven that was pre-set at a temperature of 105° C.
- e. The quartz slides were removed from the oven after one hour.

Note: The quartz slides were un-wrapped from the aluminum foil only at the time of testing.

2) Preparation of the asphalt with 0.75% anti-strip additive LOF 6500:

- a. The pure asphalt (PG 76-22) was placed in an oven at a temperature of 150° C.
- b. The anti-strip additive (LOF 6500) was measured for a quantity of 7.5 gm/1000gm of asphalt and was added to a clean container.
- c. The virgin asphalt that was placed in the oven and was allowed to be in the oven until it was workable (30-45 minutes).

- d. The liquid asphalt was taken out of the oven and a 1000 gm of it was added to the container containing the anti-strip additive and was stirred vigorously.
- e. Hence the asphalt with 0.75% anti-strip additive was prepared for the testing purpose.
- f. The 1000gm asphalt sample prepared was distributed into small containers each of 100gm.

3) Preparation of the pull stub: The preparation of the pull stub was done similar to the preparation of the quartz slide that is mentioned above.

B) Procedure followed to control the thickness of the asphalt film and the determination of the adhesive strength between asphalt and quartz slide:

1. Dynamic Shear Rheometer was maintained at a temperature of 64⁰ C.
2. The asphalt was heated in an oven at a temperature of 150⁰ C and the sample was collected in the DSR mold (8mm mold was used).
3. The asphalt was allowed to cool to form a pellet and was transferred onto the quartz slide.
4. The Dynamic Shear Rheometer scale was calibrated to zero and the height pre-set to 0.1 mm (this reading determines the thickness of the asphalt film between the pull stub and the quartz slide).
5. The temperature of the DSR platform was maintained at 64⁰ C to make the asphalt pellet warm, for better adhesion between the asphalt and quartz slide.
6. The adapter that was fabricated (as shown in Figure 6.2) worked as a replacement for the spindle that is used in the DSR test, where the adapter allows holding a stub at the free end where it is lowered to the quartz slide.
7. The stub was heated with a torch for about 15 seconds before it was lowered onto the asphalt that is on the quartz slide.
8. The prior heating of the stub was to obtain good bonding between the asphalt and the pull-stub. This would avoid the debonding at the stub surface.
9. After the stub is lowered onto the asphalt, the whole set-up is un-screwed and placed onto a table platform.
10. The excess asphalt that surrounded the stub was scraped out using a heated needle. (As shown in Figure 6.4)

11. The prepared sample was left out in the air until it cooled down to room temperature and then was kept in the refrigerator for storage purpose.
12. Prior to testing, the sample was kept in the water bath for about 24 hours at room temperature and the sample was tested using PATTI device. Schematic of the device is shown in Figure 6.6 [18].
13. Steps followed for the preliminary set-up of the PATTI device prior to testing:
 - a. The battery was inserted in the slot of the PATTI device.
 - b. The CO₂ cartridge was inserted into a slot in the PATTI device to create a source to apply pressure over the specimen through a piston.
 - c. The pressure hose was connected to the F-4 piston.
 - d. The Burst Pressure reading over the scale was set to zero prior to testing.
14. The specimen that was prepared is fixed in the piston as shown in Figure 6.5.
15. The Burst Pressure was applied at the rate of 0.005 to the specimen.
16. The run button was pushed and sustained until de-bonding of specimen occurred and the burst pressure on the scale is noted at that point
17. The Burst Pressure value that is obtained from the PATTI device is in PSIG. This obtained value is converted into psi by using the Equation 6.1 that was provided by the manufacturer.

Calculations involved in the determination of the POTS (Pull off Tensile Strength, in pounds per square inch (psi)):

$$\text{POTS} = ((\text{BP} * \text{AG}) - \text{C}) / \text{APS} \quad \text{Equation 6.1}$$

(Note: This formula has been obtained from the Elcometer Inc)

Where,

POTS = Pull off Tensile Strength, psi

BP = Burst Pressure of Piston (PSIG-Pounds per square inch gauge)

AG= Area of Gauge (4sq in for an F-4 piston)

Gauge area: Contact area of the gasket with the reaction plate

APS = Area of the Pull Stub = 0.196 sq in

C= F-4 piston constant = 0.286 lbs (± 1.5%)

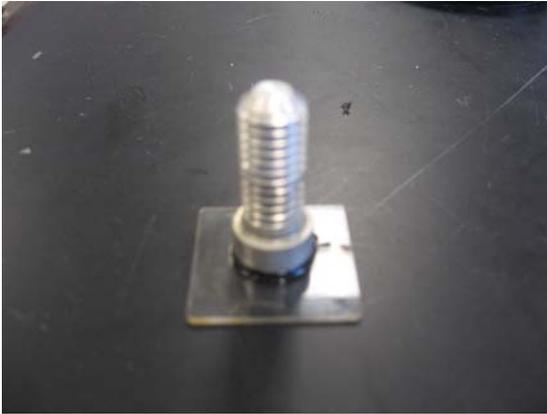


Figure 6.4 The specimen prepared with the thickness of 0.1mm asphalt film



Figure 6.5 The specimen placed in the piston prior to fixing the reaction plate over it

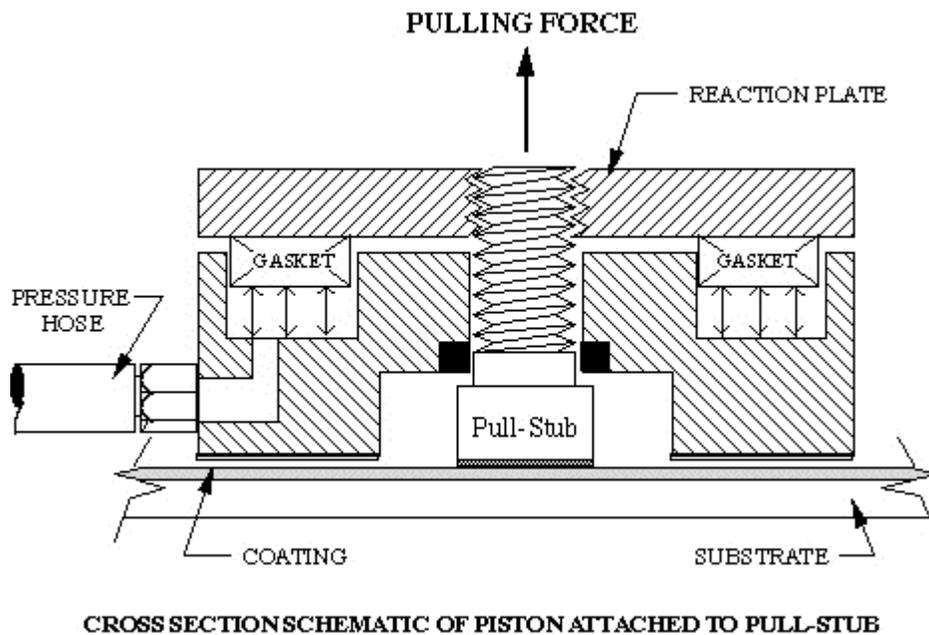


Figure 6.6 shows schematic of the test set up for the adhesion pull-off test [18]

6.6 Test results and Discussion: The following tests have been performed using the PATTI instrument to determine the effect of prolonged heating over adhesive strength between asphalt and aggregate.

Test 1: This test was conducted to determine the effect of prolonged heating for water conditioned specimen over the adhesive strength between virgin asphalt and quartz slide. Three replicates were prepared for each prolonged heating duration and 0, 2, 6 and 12

hours were considered as the prolonged heating durations for this test. Table 6.1 and Figure 6.7 shows the results obtained from the performed test:

Table 6.1 Measured adhesive strength between virgin asphalt and quartz slide

serial number	duration of heating (hrs)	anti-strip additive %	thickness of the asphalt film (mm)	type of piston	rate of loading	PSIG	POTS	average POTS for each duration of heating
1	0	0	0.1	F-4	0.005	5	101	
2	0	0	0.1	F-4	0.005	4.6	92	
3	0	0	0.1	F-4	0.005	4.5	90	94
1	2	0	0.1	F-4	0.005	5	101	
2	2	0	0.1	F-4	0.005	4.4	88	
3	2	0	0.1	F-4	0.005	5	101	97
1	6	0	0.1	F-4	0.005	4.4	88	
2	6	0	0.1	F-4	0.005	4	80	
3	6	0	0.1	F-4	0.005	3.8	76	105
1	12	0	0.1	F-4	0.005	5.3	107	
2	12	0	0.1	F-4	0.005	4.1	82	
3	12	0	0.1	F-4	0.005	4.7	94	94

POTS: Pull of Tensile Strength, psi

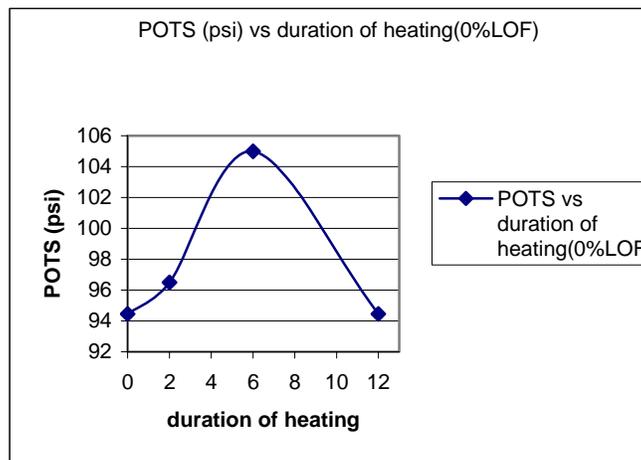


Figure 6.7 Graph showing variation of POTS with duration of heating (for pure asphalt and quartz slide)

Test 2: This test was conducted to determine the effect of prolonged heating and conditioning over the adhesive strength between asphalt with 0.75%LOF and quartz slide. It could be determined by observing the change in the adhesive strength between asphalt-quartz slide due to the effect of prolonged heating and the conditioning of the specimen in water for 24 hours. Three replicates were prepared for each prolonged

heating duration and 0, 2, 6 and 12 hours were considered as the prolonged heating durations for this test. Table 6.2 and Figure 6.8 shows the results obtained from the performed test:

Table 6.2 Measured adhesive strength between asphalt with 0.75%LOF and quartz slide

Serial number	Duration of heating (hrs)	Anti-strip additive %	Thickness of the asphalt film (mm)	Type of piston	Rate of loading	Psig	Pots	Average pots for each duration of heating
1	0	0.75	0.1	F-4	0.005	4.5	90	
2	0	0.75	0.1	F-4	0.005	5.3	107	
3	0	0.75	0.1	F-4	0.005	4.1	82	93
1	2	0.75	0.1	F-4	0.005	5.1	103	
2	2	0.75	0.1	F-4	0.005	5	101	
3	2	0.75	0.1	F-4	0.005	4	80	94
1	6	0.75	0.1	F-4	0.005	5.4	109	
2	6	0.75	0.1	F-4	0.005	4.6	92	
3	6	0.75	0.1	F-4	0.005	4.7	94	99
1	12	0.75	0.1	F-4	0.005	4.4	88	
2	12	0.75	0.1	F-4	0.005	4.5	90	
3	12	0.75	0.1	F-4	0.005	4.4	88	89

POTS: Pull of Tensile Strength, psi

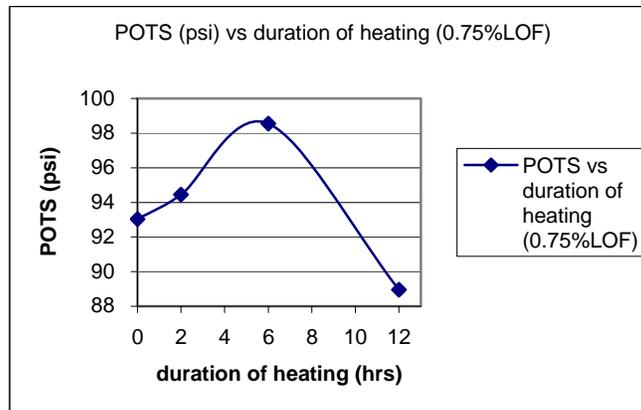


Figure 6.8 Graph showing variation of POTS with duration of heating (for 0.75%LOF in asphalt and quartz slide)

Results: Table 6.3 and Figure 6.9 shows a comparison of Pull-off Tensile Strength obtained between the virgin asphalt–quartz slide and asphalt with 0.75%LOF- quartz slide.

Table 6.3 Comparison between the average POTS obtained for asphalt with additive (0.75%LOF 6500) and virgin asphalt against quartz slide.

duration of heating (hrs)	average POTS without additive	average POTS with additive
0	94	93
2	97	94
6	105	99
12	94	89

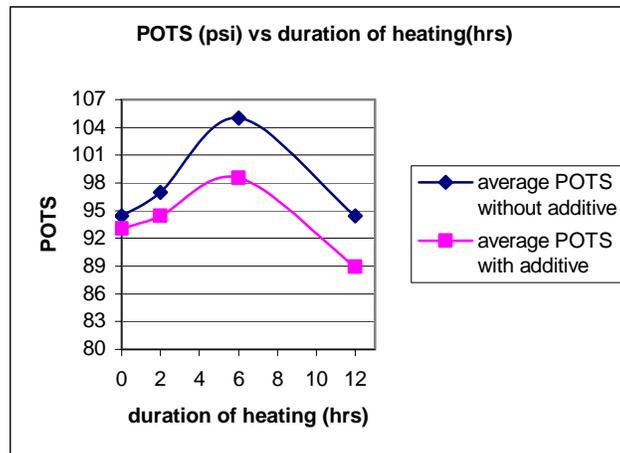


Figure 6.9 Graph showing variation of POTS with duration of heating (for pure asphalt and 0.75%LOF in asphalt and quartz slide)

Discussion: As per the mechanism involved with the action of the anti-strip additive, as we add anti-strip additive to the asphalt, it reduces the surface tension between the asphalt and aggregate and hence, promotes a better bonding between the asphalt and aggregate [20].

Normally due to prolonged heating of asphalt containing additive, the anti-strip additive gradually escapes from the asphalt. As per literature review, when a specimen is conditioned in water, the water enters between the asphalt and the aggregate through the pores of the aggregate, thus, weakening the bond between them. As the conditioning time increases the bond strength decreases.

As per the results obtained above, the Pull off Tensile Strength (POTS) between asphalt containing additive and pure asphalt with the quartz slide obtained at various durations (0 hour, 2 hour, 6 hour and 12 hour) of heating are observed to be in agreement with the results that were obtained by Kanitpong and Bahia [18]. The pull off tensile strength between asphalt containing additive-quartz slide is observed to be lower than the tensile strength obtained for quartz slide-pure asphalt at all prolonged heating durations.

As per the results obtained from the test, we can observe in general that the pull-off tensile strength with pure asphalt is higher than the asphalt with 0.75%LOF though the variation is not very much.

It can be inferred from the results that the effect of the prolonged heating and the water time conditioning is not seen in this experiment due to the complex interaction of various parameters like the mixing of the anti-strip additive, procedure followed in cleaning of the aggregate slide, roughness of the surface of the slide, temperature of the DSR instrument during the preparation of the sample, temperature of the pull stub during the application over the asphalt and the conditioning time of the specimen. As the quartz slide is non-porous, it may not have allowed the water to enter between the asphalt and the quartz slide. Therefore, it can be expected that the mode of failure to be cohesive rather than adhesive.

Figure 6.10 & 6.11 shows the pull stub and the quartz slide after the test.

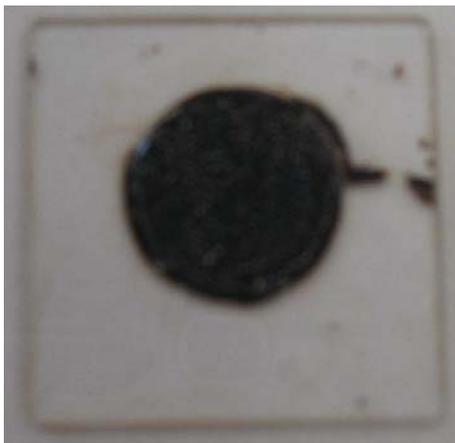


Figure 6.10 Quartz slide with asphalt film after the test

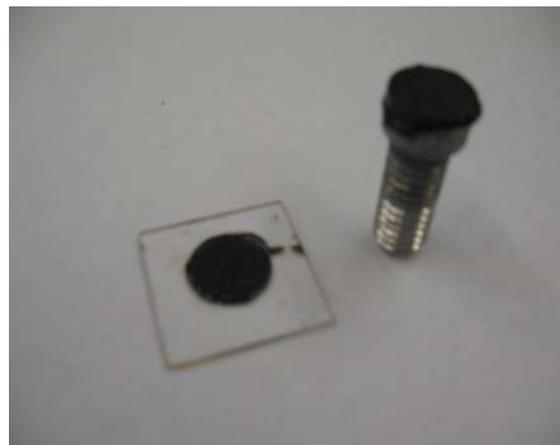


Figure 6.11 Quartz slide and pull stub after the test (Cohesive failure)

6.7 Summary and conclusion:

- 1) As per the results obtained from the pull-off test , the effect of the anti-strip additive and prolonged heating could not be observed between asphalt and quartz slide. This might be due to the complex interaction of various parameters like the mixing of the anti-strip additive, procedure followed in cleaning of the aggregate slide, roughness of the surface of the slide, temperature of the DSR instrument during the preparation of the sample, temperature of the pull stub during the application over the asphalt and the conditioning time of the specimen.
- 2) As the quartz slides possess a non-porous structure, it may not allow the water to enter between the asphalt and aggregate system. Hence the mode of failure can be expected to be a cohesive failure. The obtained result, regarding the mode of failure is in concurrence with the results that were obtained by Kanitpong and Bahia [18].
- 3) As we prepare the aggregate slides, we polish them to get a smooth surface. This smooth surface would be similar to the quartz slide that has been used in the test. As per the analysis of the results that were obtained, similar results are expected due to the occurrence of similar conditions (resemblance of the surface of the polished aggregate to that of the quartz slide: non-porous).
- 4) Test over the determination of the adhesive strength between the asphalt and aggregate slide could not be performed as the preparation of the aggregate slide was \$75/piece, which was beyond the budget of the project.

7. Atomic Force Microscopy Test

7.1 AFM overview: Atomic Force Microscopy was invented in 1986 by Gerd Binnig, Cal Quate and Christof Gerber when they recognized that the force exerted by a small physical probe (tip) on surface could be used to map the topography of the sample [21]. Atomic Force Microscopy was employed to measure the force curves, obtained as a function of contact loading and sampling frequency. It was hypothesized that the work of adhesion between asphalt binders and aggregate particles directly relates to the pavement tendency to micro crack and heal [22, 23]. The interpretation of the work of adhesion was based on Johnson-Kendall-Roberts (JKR) contact theory that was applied in the measurement of force curves. The application of the contact theory was due to the exhibition of polymer like characteristics by the asphalt [23]. The force between tip and sample can be measured by determining the deflection of the sample [21]. The force commonly associated with AFM is the van der Waals' forces [23]. The force curves were not only useful in the determination of the adhesive property but can also be used to determine properties like elasticity, hardness, Hamaker constant, and surface charge density. The Western Research Institute (WRI) has reported use of Atomic Force Microscopy to develop quantifiable images of asphalts and asphalt with additives [25] and their adhesive properties with aggregate at interfacial region. Atomic Force Microscopy is also extensively used in the fields of surface science, materials engineering and biology [26].

7.2 Objectives and task: The main objective of this test was to obtain the effect of the anti-strip additive over the adhesive strength between asphalt and aggregate (glass bead). This is obtained by observing the difference in the adhesive strength between the glass bead-pure asphalt and glass bead-asphalt with 0.75%LOF at various temperatures, based on the force curves that would be obtained from the Atomic Force Microscopy test.

7.3 Materials: The materials that were supplied by the North Carolina Department of Transportation are PG 76-22 asphalt and LOF 6500 (Anti-Strip Additive). Silicon slides

which were obtained from the Atomic Force Microscopy laboratory at NCSU were used as the plate over which the asphalt was coated and tested for adhesive strength.

7.4 Preliminary test to investigate the uniformity of the probes using Scanning Electron Microscopy:

Investigation for the probe: The probe was investigated for the uniformity of the chemical composition of the probe. Scanning Electron Microscopy was used to image the probe at the micro level to investigate the uniformity of the chemical composition of the probes. (Figure 7.1 shows a schematic of an image of Scanning Electron Microscopy).

Mechanism of Scanning Electron Microscopy:

1. As the current flow through the tungsten filament, the electrons are boiled off the surface of the filament.
2. The emitted electrons were accelerated by the anode that is present below the tungsten filament.
3. To concentrate the electron beam at a particular point, condenser lenses were used.
4. Then the accelerated beam passes through the objective lens where the lens focuses the beam on the sample.
5. As the electrons strike the probe, the electrons are reflected and are detected by the Backscatter detector.
6. The number of the electrons that are backscattered (same as Rutherford Backscattering) [26] is a function of the atomic number of the material of the sample.
7. Hence, based on the number of electrons that are backscattered the elemental composition of the sample is determined.

Note: If there was presence of asphalt over the glass bead, a black spot would be seen due to the detection of the carbon over the surface of the bead by the SEM.

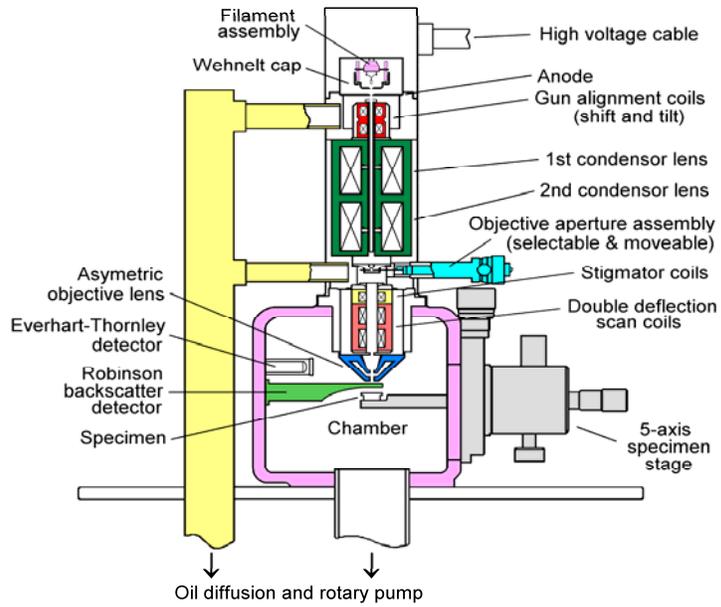


Figure 7.1 Scanning Electron Microscopy

Based on the investigation that has been done over two probe beads that were randomly selected, the images are displayed below as shown in Figure 7.2.

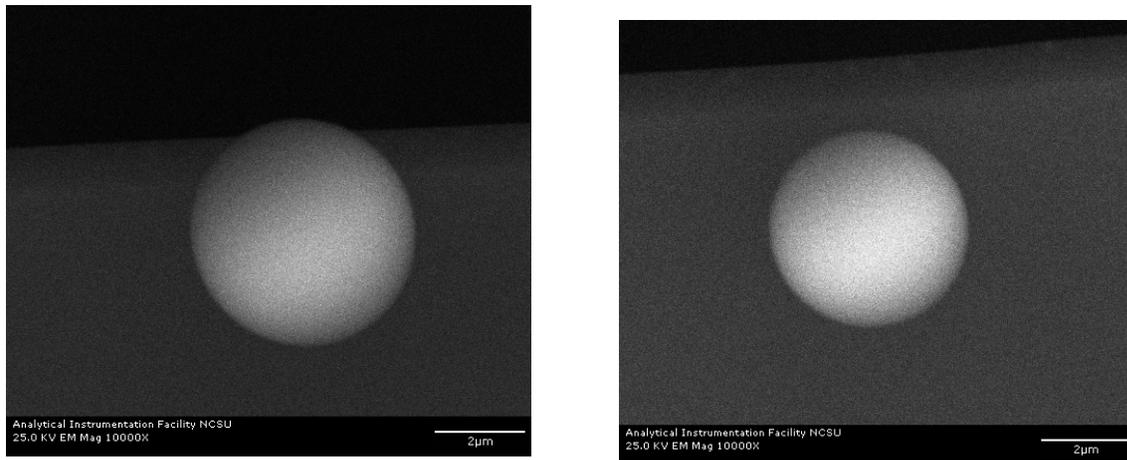


Figure 7.2 A comparison between the beads of two different probes selected at random

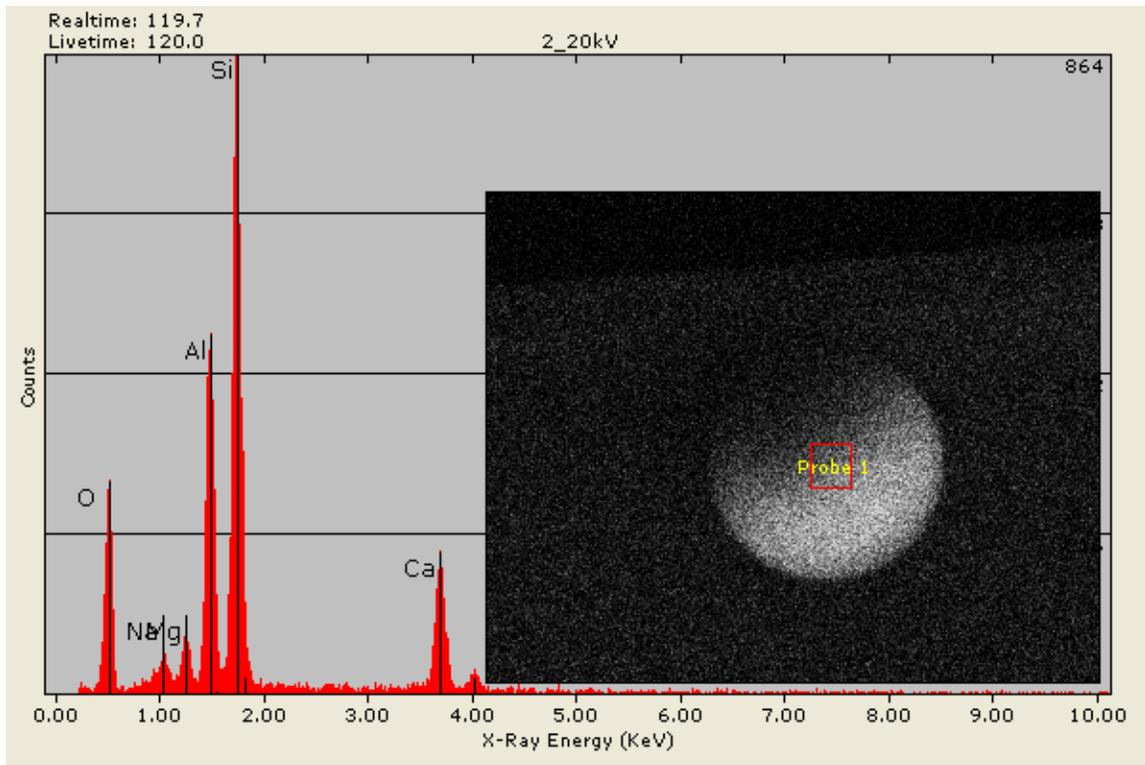


Figure 7.3 Chemical Composition of the Probes that were investigated for uniformity

1. As per the image (as shown in Figure 7.3) that is obtained from the SEM, the presence of the Si and O₂ is due to the presence of silicon bead that is present as the tip of the probe.
2. Traces of calcium, sodium and magnesium might be due to the detection of the glue that has been used to stick the bead to the probe.
3. The probe is coated with a thin layer of aluminum to obtain better bonding between glass bead and the cantilever.

Both the glass beads were found to be 5µm and the colors of the images were found to be identical. It shows that the composition of the probe was identical.

7.5 Preparation of specimens:

The following procedure was used in the preparation of the specimen:

1. The asphalt was heated at a temperature of 150⁰ C for 30 minutes.
2. A silicon plate of thickness 1mm was taken and cut into pieces of 10mm by 3mm.

3. A stainless steel, disposable needle was taken and heated with a heating gun.
4. The hot needle was dipped into the hot asphalt and is swiped over the silicon slide.
5. A thin layer of asphalt was obtained over the silicon slide.
6. The silicon slide with asphalt layer on it was carefully stored in a box prior to testing.

7.6 Procedure for AFM test: The following procedure has been followed using the AFM instrument.

- 1) The prepared sample was mounted in the Atomic Force Microscopy instrument.
- 2) The temperature in the environmental chamber was set to desired temperature (say 100° C). The specimen was left for 10 minutes after reaching the temperature in the environmental chamber to obtain equilibrium in temperature between the platform and the specimen.
- 3) Then the cantilever probe was carefully fixed in position.
- 4) After the temperature reached equilibrium, the platform is raised towards the cantilever probe that is in static position.
- 5) The deflection of the sample is registered by the instrument and the cantilever is deflected by the raised specimen.
- 6) After rising to the desired level, the platform is lowered back towards the original position.
- 7) As the platform is lowered, the glass bead that is stuck to the asphalt during the upward movement of the platform is detached from the asphalt and a force curve is obtained in this process.
- 8) The various segments of a force curve and the calculation of the adhesive strength from the obtained force curve are presented in the following sections

7.6.1 A brief explanation of general force curve:

1. The force curve that is obtained using AFM is plotted over a graph, with x-axis as tip sample displacement and y-axis as the force (in Volts). Figure 7.4 shows various segments of the Force Curve.
2. Segment a-c signifies the rise of the platform towards the cantilever that is at rest.
3. The segment c-d-e (v-notch) is obtained just as the specimen touches the cantilever probe (v-notch in Figure 7.4: termed as snap-in point).

4. Segment e-f is the result which is the sum of the spring constant of the cantilever probe and the young's modulus of asphalt. This segment signifies deflection of the cantilever in the upward direction.
5. Segment f-g signifies the retrieval of the sample towards the original position.
6. The deflection of the force curve towards the original position at point g signifies the detachment of the asphalt (point g in Figure 7.4: termed as pull-off point) from the cantilever and the deflection of the cantilever to the initial position.
7. This segment g-b is used to determine the adhesive strength between the asphalt and glass bead.

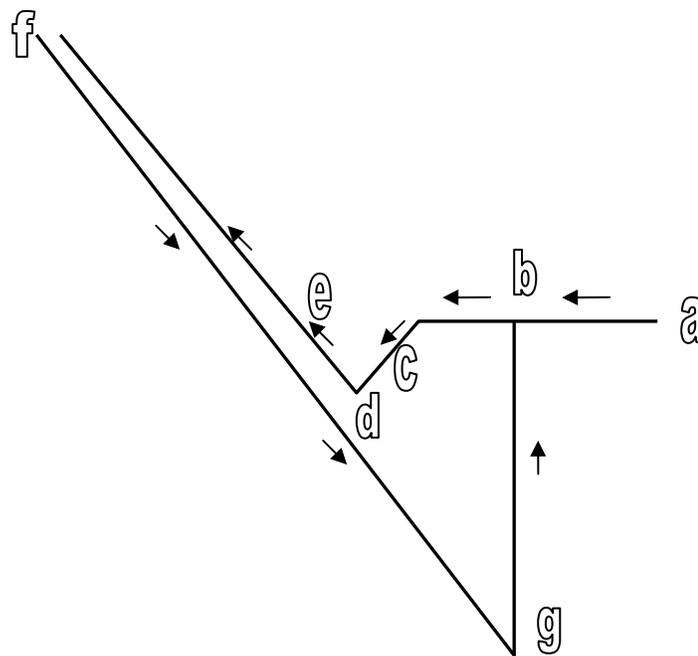


Figure 7.4 Schematic of force curve obtained from the Atomic Force Microscopy

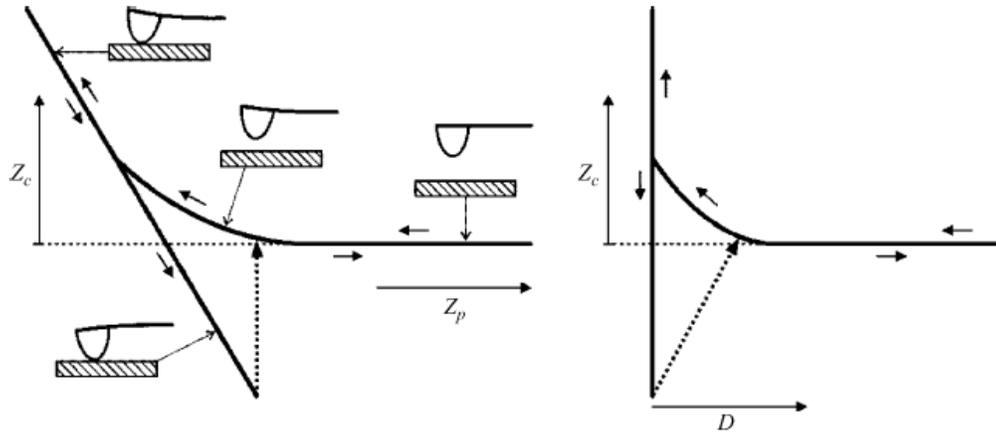


Figure 7.5 Schematic of a typical cantilever deflection-vs.-piezo height (Z_c -vs.- Z_p) curve (left) and corresponding Z_c -vs.- D plot, with $D = Z_c + Z_p$ (Butt et al. 2005)

Adhesive strength between asphalt and aggregate = [tip deflection f_g (in nm)] * [spring constant of the cantilever (in N/m)]. The adhesive strength is obtained in nano-newtons.

Determination of pull-off point and the adhesive strength from the force curve: For some of the force curves that were obtained in this experiment, the pull-off point could not be obtained due to the limitation of the sensor. Assuming the profile of the curve to be uniform, the curve lines are extended and the pull-off point is determined.

To determine the adhesive strength between the asphalt-glass bead from the force curve:
 (Adhesive Strength) nN = slope of the curve (nN/V) * deflection of the cantilever (V)

Where,

Slope of the curve (nN/V) = Spring Constant (N/m) * Sensitivity

Sensitivity = [1 nanometer/V] (default value)

V is in volts.

7.7 Test results and discussion: Various tests have been performed to obtain a desired temperature at which the atomic force microscopy test can be conducted to obtain the force curves. The tests are discussed in the following sections.

Test 1: The temperature in the environmental chamber was maintained at 100⁰ C.

A test was performed over virgin asphalt-glass bead at 100⁰ C. Figure 7.6 shows the asphalt stuck to the cantilever probe and formed a neck during the retrieval of the asphalt platform away from the cantilever. Figure 7.7 shows the force curve obtained in this test.



Figure 7.6 The retrieval of the asphalt platform from the cantilever probe (100⁰ C).

Discussion: As per the limitations of the Atomic Force Microscope, the sensor can detect the deflection of the cantilever probe only within a certain range. When the test was performed at 100⁰ C, as the asphalt was raised towards the cantilever probe, the asphalt did not show any resistance to the cantilever probe for it to deflect. When the platform was lowered, the asphalt could not be detached from the glass bead within the range of the sensor. Hence, the force curve segment g-b (as explained in section 7.6.1) could not be obtained. This result led to the failure of the test. Figure 7.7 shows the force curve that was obtained during this test. Therefore, further investigation was carried out to determine a suitable temperature, at which decent force curves could be obtained.

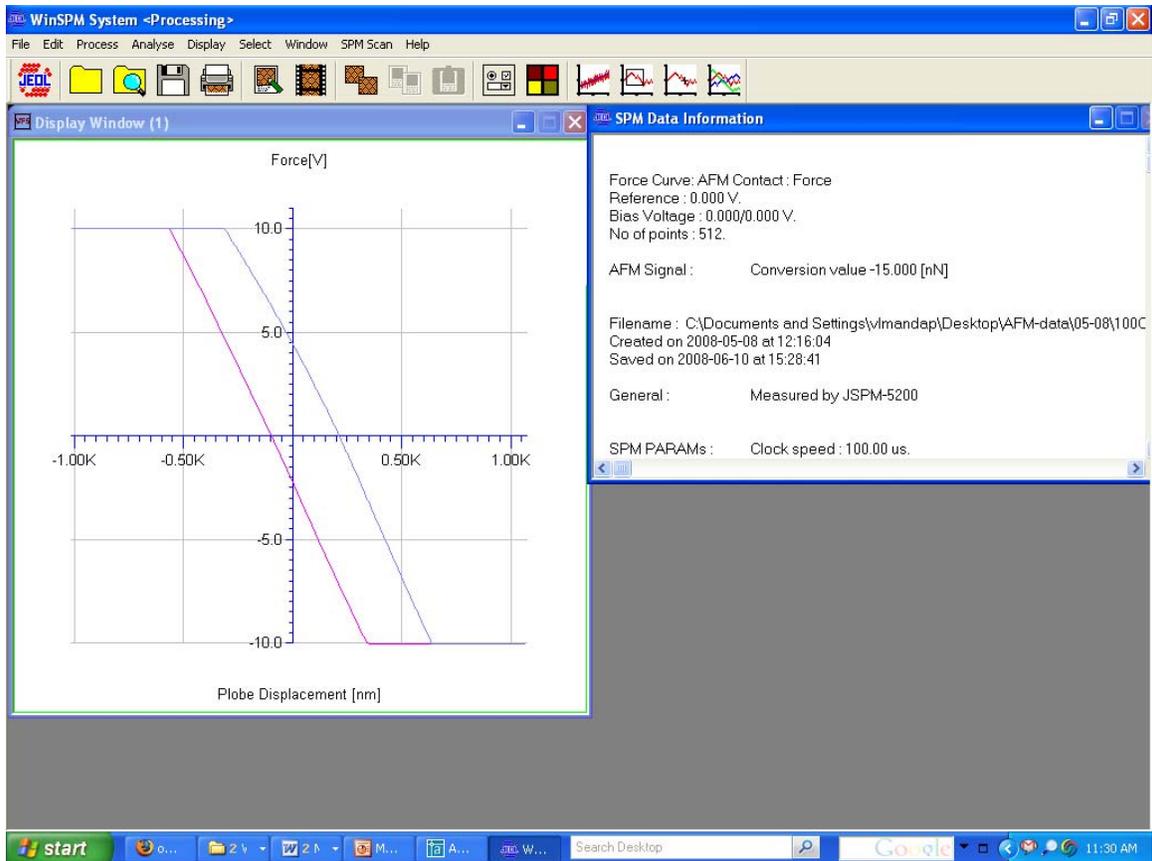


Figure 7.7 The force curve obtained between virgin asphalt-glass bead (100°C)

Test 2: The temperature in the environmental chamber was maintained at 67°C .

A test was performed over virgin asphalt-glass bead at 67°C . Figure 7.8 shows the asphalt that's stuck to the cantilever probe and formed a neck during the retrieval of the asphalt platform away from the cantilever. Fig 8 shows the force curve obtained in this test.

Discussion: The ductility of the asphalt was observed to be lesser at 67°C than at 100°C . Hence, the detachment of the asphalt from the glass bead was observed to be earlier at 67°C than at 100°C , though again not in sensor range. Figure 7.8 shows the force curve that is obtained at 67°C (The curve obtained was similar to the result that was obtained at 100°C).

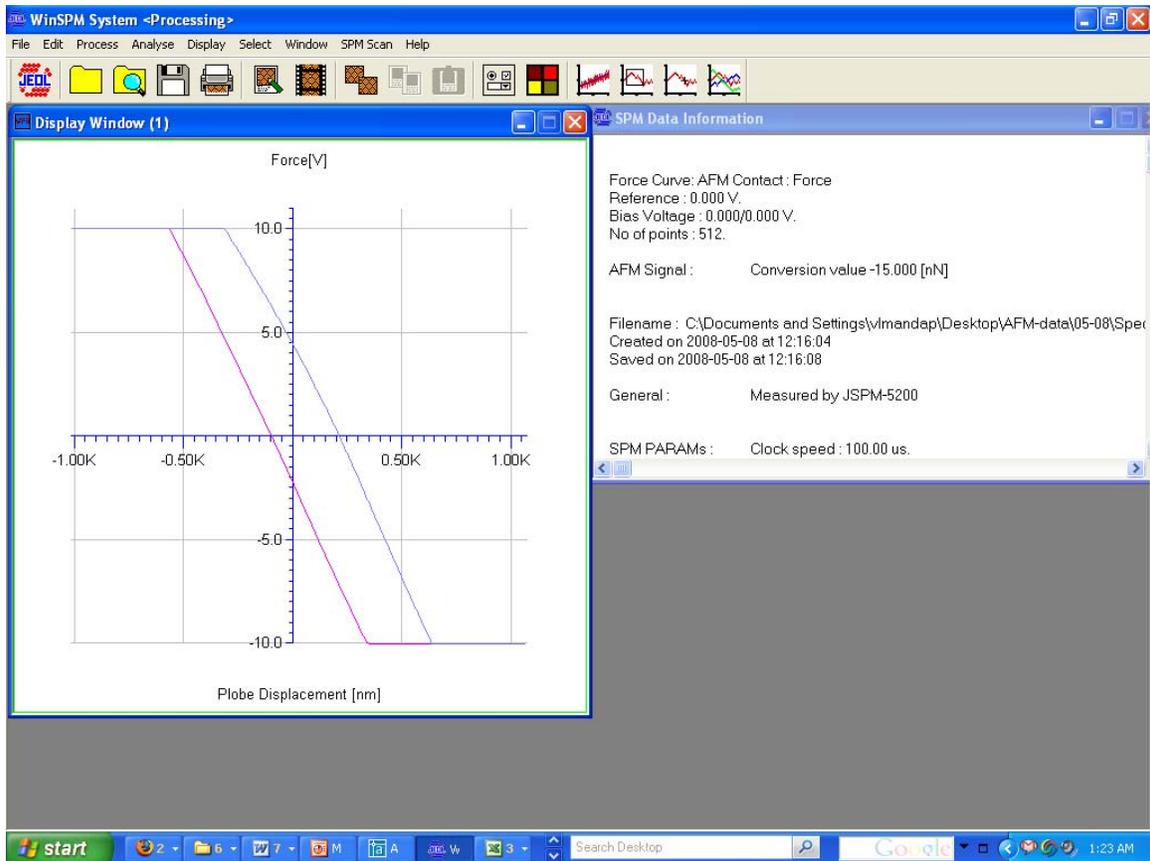


Figure 7.8 The force curve obtained between virgin asphalt-glass beads (67⁰ C)

Based on the results obtained at 100⁰ C and 67⁰ C, it was necessary to further reduce the temperature in the environmental chamber. Next, after some experimentation the temperature was lowered to 43⁰ C at which point a force curve could be obtained for asphalt without anti-strip additive.

Test 3: The temperature in the environmental chamber was maintained at 43⁰ C.

Based on the procedure specified above in section 7.5, the Force curve determination between the virgin asphalt-glass bead and asphalt with 0.75%LOF-glass bead was performed. The results obtained are summarized below in Table 7.1 and Figure 7.9 & 7.10.

Table 7.1 Average adhesive strength between virgin asphalt-glass beads (43⁰ C)

Serial No.	Temperature of Chamber in C	%LOF	Probe ID	Spring Constant (N/m)	Displacement (Volts)	adhesive strength (nN)
1	43	0	1	5.35	33.10	177.09
2	43	0	1	5.35	33.00	176.55
3	43	0	1	5.35	35.40	189.39
4	43	0	1	5.35	22.50	120.38
5	43	0	1	5.35	36.57	195.65
6	43	0	1	5.35	36.57	195.65
7	43	0	1	5.35	33.00	176.55
8	43	0	1	5.35	37.75	201.96
9	43	0	1	5.35	36.25	193.94
10	43	0	1	5.35	36.25	193.94
11	43	0	1	5.35	42.03	224.86
12	43	0	1	5.35	41.78	223.52
13	43	0	1	5.35	35.00	187.25
14	43	0	1	5.35	35.00	187.25
15	43	0	1	5.35	40.30	215.61
16	43	0	1	5.35	40.55	216.94
17	43	0	1	5.35	40.30	215.61
18	43	0	1	5.35	30.80	164.78
19	43	0	1	5.35	35.00	187.25
20	43	0	1	5.35	31.76	169.92
Average Adhesive Strength (nN)						190.70

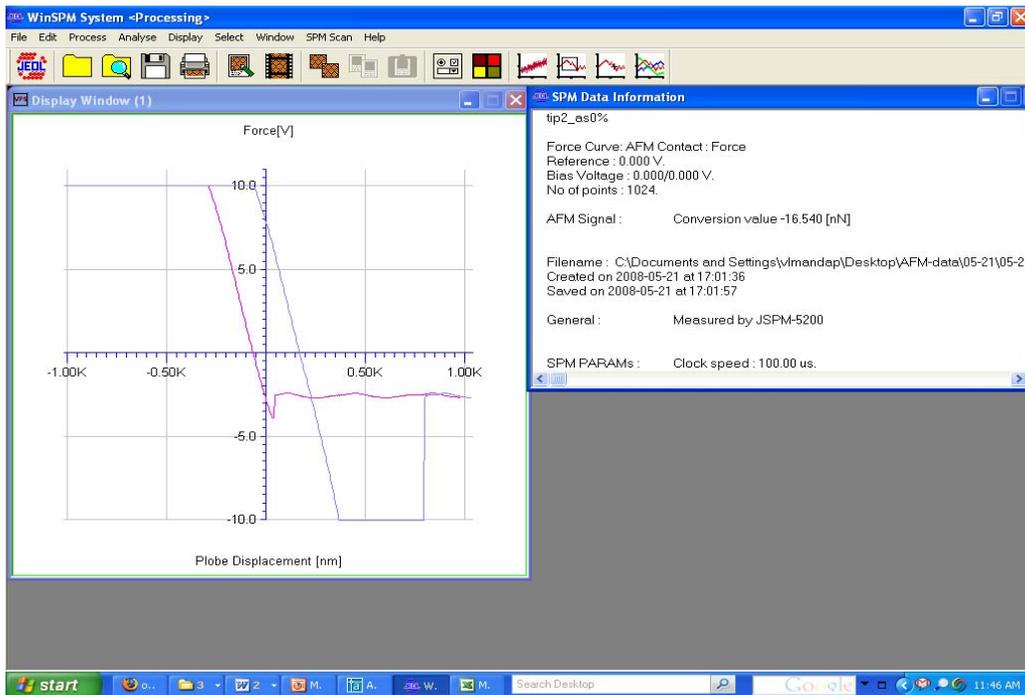


Figure 7.9 Force curve obtained @43⁰ C between virgin asphalt-glass beads

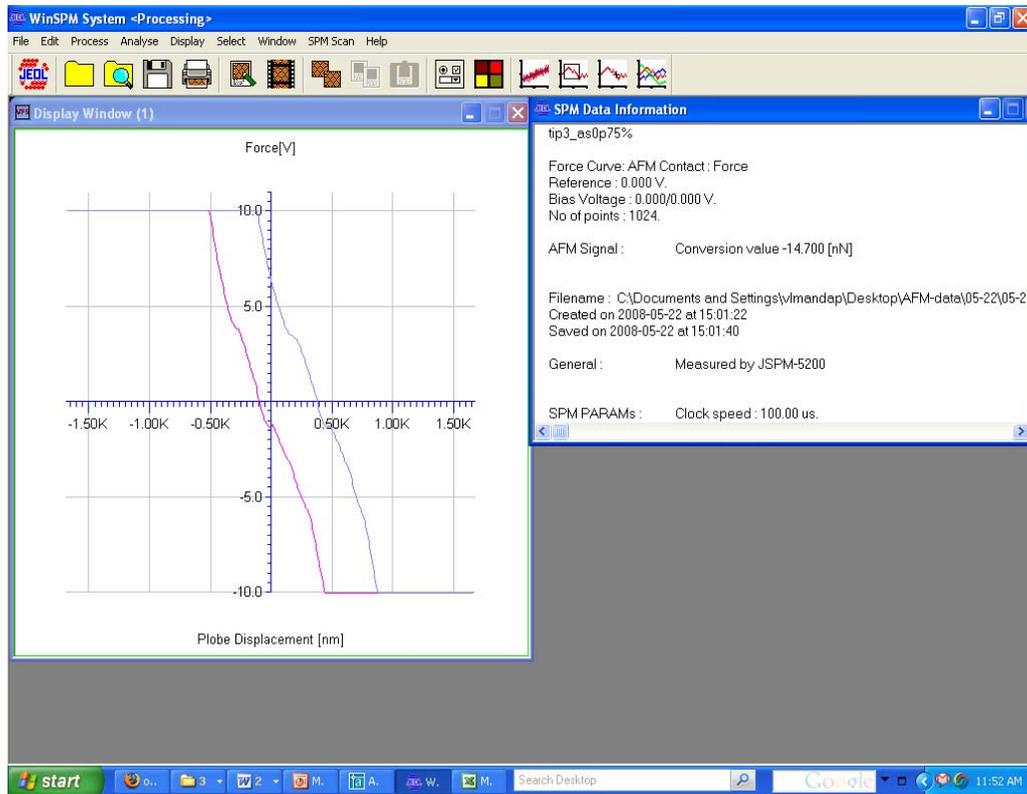


Figure 7.10 Force curve obtained at 43⁰ C between asphalt (with 0.75%LOF)-glass bead

Discussion: The average adhesive strength between the pure asphalt-glass beads observed at 43⁰ C is 190.7 nN. As we observe the force curves that are obtained for asphalt with anti-strip additive-glass bead the adhesive strength couldn't be measured as the adhesive strength of the asphalt with anti-strip additive was so high that the glass bead was plucked out from the cantilever. The asphalt containing the glass bead after the test is shown in figure 7.11. Hence, based on the result, adhesive strength of asphalt has increased due to the presence of anti-strip additive. Although the force could not be measured due to the limitation of the sensor it maybe concluded that anti-strip additive improves adhesive strength of asphalt over asphalt without anti-strip additive.

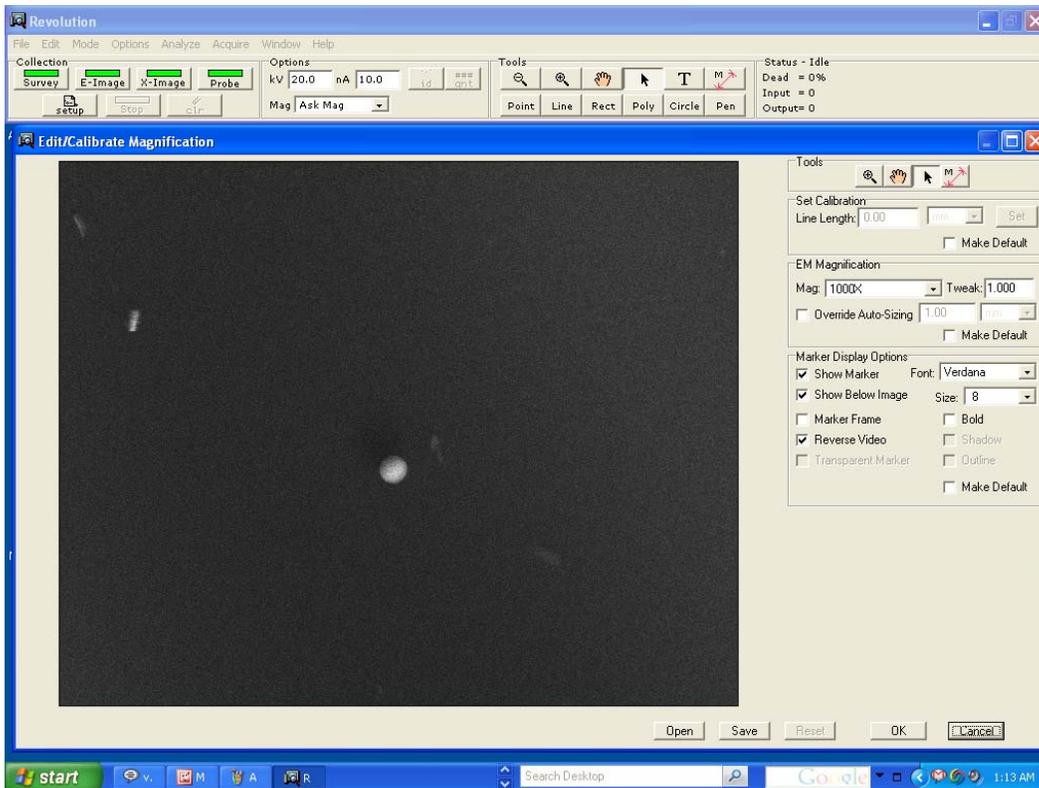


Figure 7.11 The pulled out bead stuck to the asphalt (containing anti-strip additive 0.75%LOF)

Test 4: As the force curve for asphalt with anti-strip additive could not be generated, the temperature of the testing was reduced further. The temperature in the environmental chamber was maintained at 20⁰ C.

Based on the procedure specified above in section 7.5, the Force curve determination between the virgin asphalt-glass bead and asphalt with 0.75%LOF-glass bead was performed. The results obtained are summarized below in Table 7.2 and Figure 7.12 & 7.13.

Table 7.2 Average adhesive strength between virgin asphalt-glass beads (@20⁰ C)

Serial No.	Temperature of Chamber in C	%LOF	Probe ID	Spring Constant (N/m)	Displacement (Volts)	adhesive strength (nN)
1	20	0	2	8.27	9.6	79.392
2	20	0	2	8.27	7.2	59.544
3	20	0	2	8.27		
4	20	0	2	8.27	6	49.62
5	20	0	2	8.27	5.8	47.966
6	20	0	2	8.27	7	57.89
7	20	0	2	8.27	7	57.89
8	20	0	2	8.27	6.6	54.582
9	20	0	2	8.27	10	82.7
10	20	0	2	8.27	10	82.7
11	20	0	2	8.27	8	66.16
12	20	0	2	8.27	7.6	62.852
13	20	0	2	8.27	7.6	62.852
14	20	0	2	8.27	7.5	62.025
15	20	0	2	8.27	---'	
Average Adhesive Strength(nN)						63.55177

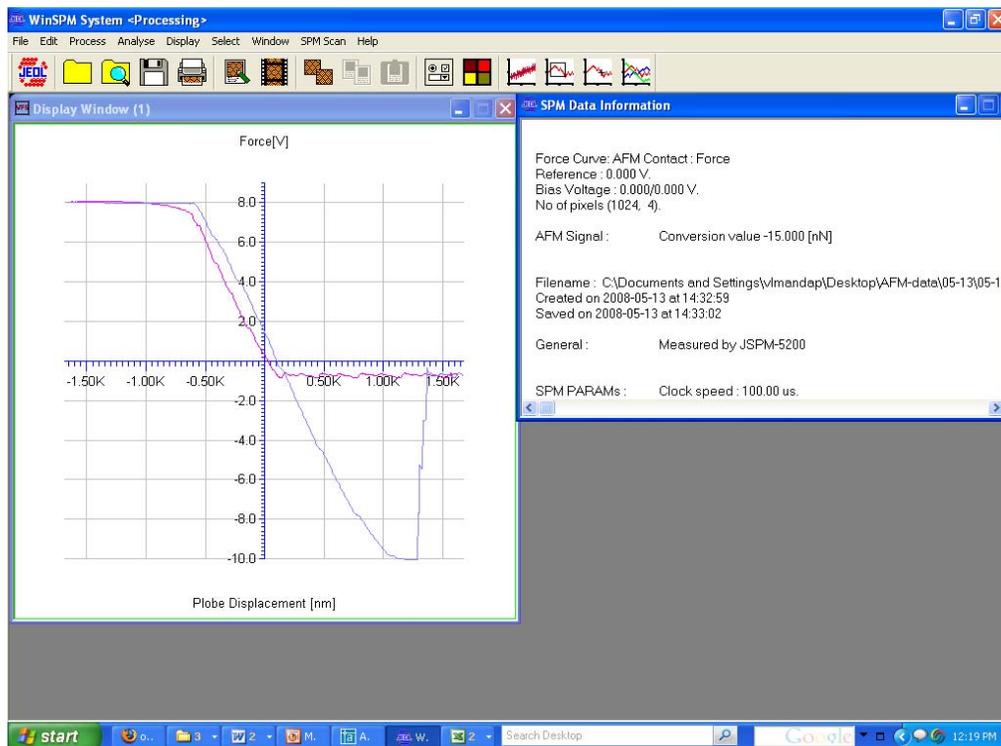


Figure 7.12 Force curve obtained at 20⁰ C between virgin asphalt -glass bead

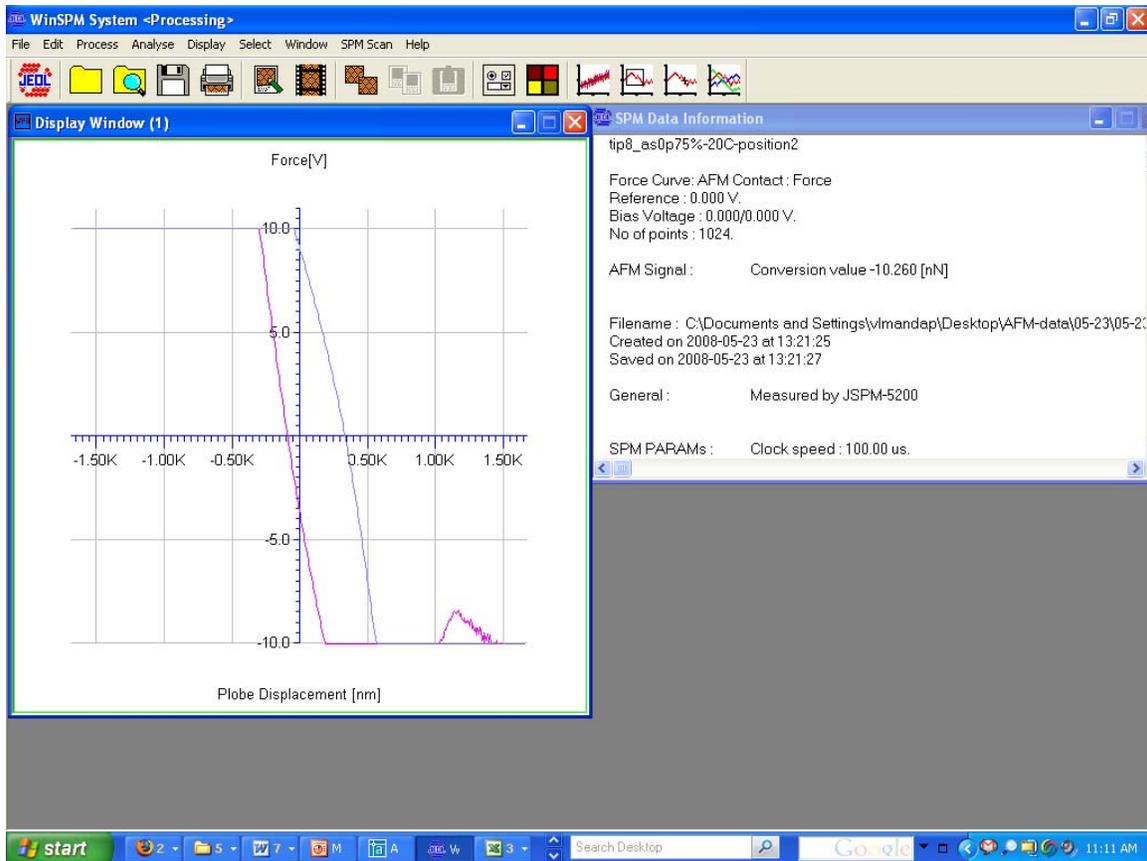


Figure 7.13 Force curve obtained at 20⁰ C between asphalt (with 0.75%LOF)-glass bead

Discussion: The average adhesive strength between the pure asphalt-glass beads observed at 20⁰ C is 63.55 nN. As we observe the force curves that are obtained for asphalt with anti-strip additive-glass bead the adhesive strength couldn't be measured as we could not obtain a pattern in the force curve.

Test 5: The temperature in the environmental chamber was maintained at 7.5⁰ C. Based on the procedure specified above in section 7.5, the force curve determination between the virgin asphalt-glass bead and asphalt with 0.75%LOF-glass bead was performed. The results obtained are summarized below in figures 7.14 & 7.15.

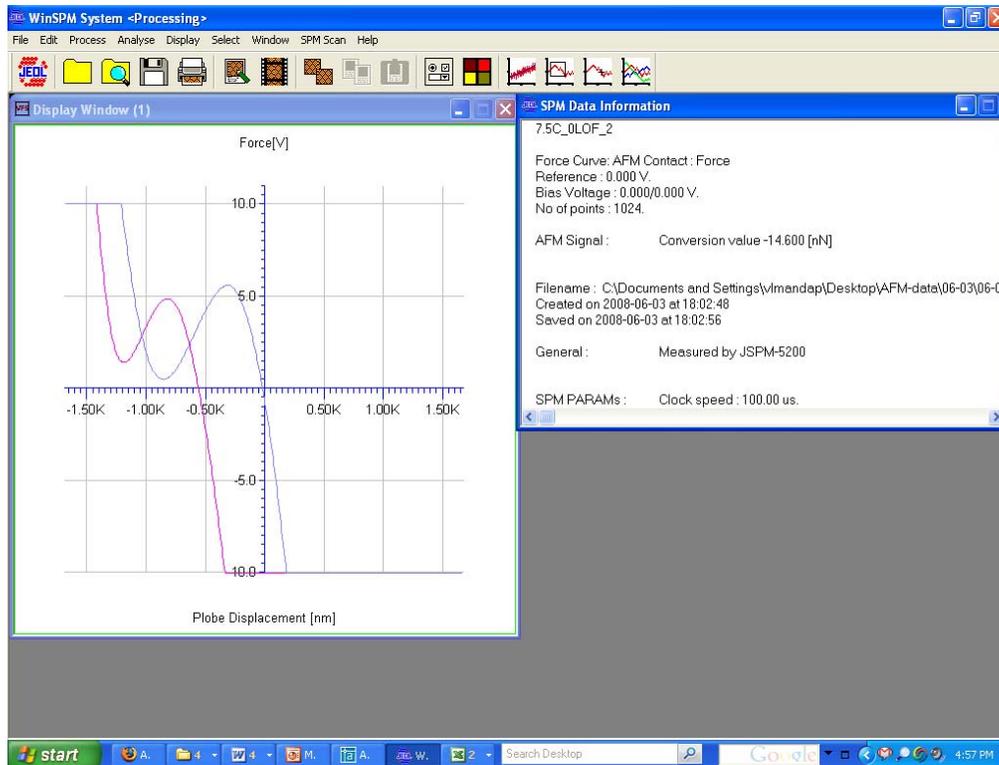


Figure 7.14 Force curve obtained at 7.5°C between virgin asphalt -glass bead

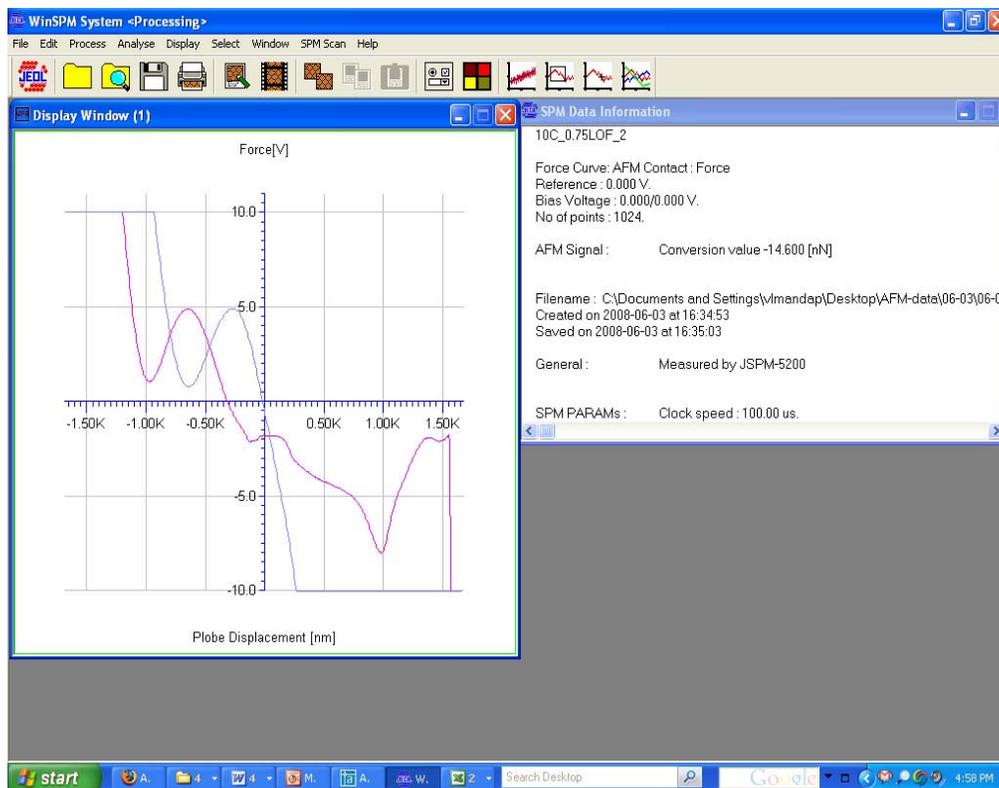


Figure 7.15 Force curve obtained at 7.5°C between asphalt (with 0.75%LOF)-glass bead

Discussion: Based on the force curves obtained at 7.5⁰ C, for both virgin asphalt and asphalt with anti-strip additive, the adhesive strength could not be measured. This could mainly be due to the fact that the asphalt is so hard at this temperature that it essentially behaves as a solid substance, in which case the force between the glass bead and solid asphalt surface is negligible.

7.8 Summary and conclusion:

1. The ductility of the asphalt at 100⁰ C and 67⁰ C was so high that the force curve couldn't be obtained to determine the adhesive strength between virgin asphalt and glass bead.
2. The adhesive strength between asphalt (with 0.75%LOF) and glass bead at 43⁰ C was not measurable due to the limitation of the sensor.
3. The adhesive strength between virgin asphalt-glass bead at 43⁰ C and 20⁰ C was measured to be 190.7nN and 63.55 nN respectively. Adhesive strength of asphalt (with 0.75%LOF)-glass bead was not measurable. At 43° C the adhesive strength between asphalt containing 0.75%LOF and glass bead was relatively high enough to tear out the bead from the cantilever. Based on this observation, it may be concluded that there certainly is an improvement in adhesive strength with addition of 0.75%LOF anti-strip additive.

8. Summary, Conclusions and Recommendations

Off the tests conducted in this study, the most consistent results obtained were from the TSR test. The AFM (Atomic Force Microscopy) test that was conducted on cursory basis relatively indicate that the adhesive strength increased when LOF 6500 anti-strip additive was used in asphalt. However, the force (adhesive strength) could not be measured because 1) at high temperature, the asphalt was too soft and the AFM cantilever bead stuck to the asphalt; 2) at intermediate temperature the force was so high that it could not be measured for asphalt with LOF 6500 anti-strip additive due to the limitations of the instrument; and 3) at lower temperatures the force could not be measured probably because the asphalt was hard enough to act as a solid substance. It may be noted that a PG76-22 asphalt was used in this study.

On a qualitative basis, the AFM results that the adhesive strength increases with addition of anti-strip additive is in agreement with the TSR test results that show that mixtures containing anti-strip additive reduces the moisture susceptibility of mixtures that are not subjected to prolonged heating. However, when subjected to prolonged heating, especially beyond 6-hours, the results show that the moisture susceptibility of these same mixtures containing anti-strip additive increases and the TSR values decrease to a point where they fail the NCDOT standard requirement of 85% retained strength.

Considering storage and transport, as well as the duration between mixture production and the ultimate paving in the field, it is foreseeable that the mixture may be unacceptable with regards to moisture susceptibility by the time it is placed in the field. This situation needs due attention by NCDOT when using amine based liquid anti-strip additive in their paving mixtures.

The results using contact angle goniometer were found to be highly variable and not consistent. Results from the pull-off test using PATTI device were also not very useful as it essentially measured the cohesive strength as opposed to the adhesive

strength, at least in this study. Therefore, both these devices are not a useful tool in measuring adhesive strength for asphalt.

Previous NCDOT studies have clearly shown that there is a loss of organic liquid anti-strip additive content in both asphalt binders and mixtures when subjected to prolonged heating. Results of this study clearly show that this loss of additive is reflected in increased moisture sensitivity of mixtures. It is therefore recommended that NCDOT take a careful look at the practices that are being followed currently, and take necessary steps to insure that the mixtures being used in paving meet the minimum NCDOT standard with regards to moisture sensitivity at the time the mixture is placed in field rather than during the process of formulating the job mix formula (JMF). If minimum standard cannot be assured with regards to storage, transport and the ultimate use of the mix in the field, NCDOT may need to look at alternatives to the use of organic amine based liquid anti-strip additives.

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**Appendix A Job-Mix-Formula and Tensile Strength Ratio
Test Data for Asphalt Mixes with 0.8% LOF 6500 and Morelife
2200 Antistrip Additives in Asphalt Binders after Prolonged
Heating**

Table A - 1 Original JMF Provided by NCDOT

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REVISED November 2003

MAT FORM 001 (3P)

REPORT ON SUPERPAVE MIX DESIGN

MD#06-222

DATE SUBMITTED:	DATE APPROVED: 04-20-06
PROJECT NO.:	BINDER: CONOCO Knoxville AT29 PG 78-22
COUNTY:	ADDITIVE: ARR-MAZ Ad-Here 6500 LOF (.8%)
CONTRACTOR:	Vulcan Materials Enka CA69 Washed Scrps.
PLANT & NO.:	Vulcan Materials Enka CA69 78-M Stone
DESIGNED BY:	Vulcan Materials Enka CA69 # 67 Stone
SPECIFICATION: S 12.5 D Surface Mix	Baghouse Fines
GRAVATIONS: 9/125/ 150 mm 157 °C	
TRAFFIC LEVEL: >30.0 Million ESALs	
BINDER SPECIFIC GRAVITY: 1.030	
COMPACTOR TYPE: Trolder	

GRADATION OF MATERIALS USED

MATERIAL	Washed Scrps	78-M Stone	# 67 Stone			BqHsFines	Rap	BLEND	CONTROL
PERCENT (MD)	45.8	34.0	18.0			2.2		100.0	POINTS
PERCENT (JMF)	45.0	34.0	18.0					100.0	
Sieves(mm) 50.0	100.0	100.0	100.0			100.0		100	
37.5	100.0	100.0	100.0			100.0		100	
25.0	100.0	100.0	99.0			100.0		100	
19.0	100.0	100.0	90.0			100.0		98	100.0
12.5	100.0	99.0	47.0			100.0		90	90.0 - 100.0
9.5	100.0	92.0	17.0			100.0		82	< 90.0
4.75	97.0	33.0	8.0			100.0		59	
2.36	85.0	9.0	4.0			100.0		45	28.0 - 58.0
1.18	60.0	5.0	3.0			100.0		32	
0.600	41.0	3.0	2.0			100.0		22	
0.300	22.0	3.0	2.0			100.0		14	
0.150	10.0	2.0	1.0			100.0		8	
0.075	4.7	1.4	0.9			90.0		4.8	4.0 - 8.0
Ign.Fum. Corr.Factor									
Agg. Bulk Dry S.G.	2.734	2.690	2.697			2.734		2.712	
								Agg. Effective S.G.:	2.757
Agg. Apparent S.G.	2.774	2.752	2.743			2.774		2.761	

Mix Properties at N design

Opt. Pb	Mix Properties at N design				
% Asphalt Binder-Total Mix	5.0	5.5	6.0	6.5	% RAP / % Virgin: 0/100
Gmb @ Ndes (or Nmax)	2.398	2.415	2.433	2.447	Pb in RAP: —
Max. Specific Gravity(Gmm)	2.544	2.524	2.505	2.486	Pb from RAP: —
% Voids-Total Mix (VTM)	5.7	4.3	2.9	1.6	Pb Absorption: 0.6
% Solids-Total Mix	94.3	95.7	97.1	98.4	% ASH: —
% Effective Binder Content (Pbe)	-0.6	4.4	4.9	5.4	TSR % Retained : 108.5
Dust to Pbe Ratio (Fp.0.075/Pbe)	-8.00	1.09	0.96	0.89	Ignition Fum. Calibr.: —
By Volume of Effective Pb	10.2	11.5	12.8	14.0	Pb (Design): 5.6
% Solids by Vol. of Agg. Only	84.1	84.2	84.3	84.4	Rice Specific Gravity: 2.520
% Voids in Mineral Agg. (VMA)	16.0	15.8	15.7	15.6	Lab Specific Gravity: 2.419
% Voids Filled w/Binder (VFA)	63.8	72.8	81.5	89.7	Percent Air Voids: 4.0
% Gmm @ Nini	87.9	89.2	90.4	91.6	Percent VMA: 15.8
% Gmm @ Ndes	94.3	95.7	97.2	98.4	Percent VFA: 74.7
% Gmm @ Nmax					DUST/AC Ratio: 0.95
COMMENTS: Rut depth = 1.6 mm	Uncomp. Void Cont.*	NA			% Gmm @ Nini: 89.4
DESIGNED BY: Sandra Clayton 507 50670	Sand Equivalent:	78.5			% Gmm @ Nmax: —
APPROVAL: C.A. Clippard, Jr.	C. Agg. Angularity	100/100			Pb ADDED: 5.6
	F. Agg. Angularity:	51.3			Pb from RAP: —
	Flat & Elongated:	8.7			Pb TOTAL: 5.6

Table A-2 TSR Test Data for None Antistrip Additives in Asphalt Mixes with 2-Hour Heating

Revised 11-29-99

NCDOT

M&T 612 (QMS-2)

TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Marshall Compactive Method

Date Mix Produced:	Mix Type: N00-2	JMF No.:
Contractor: NCSU	Plant Location:	Plant Cert. No.:
Additive Supplier:	Additive Grade:	Additive Dosage:
Date Compacted:	No. Gyration: to height	Date Test Completed: 2/27/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(in) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(in.) (b)	96.000	96.000	95.900	96.000	95.900	95.700	95.800	96.000
DRY MASS IN AIR (c)	3932.4	3941.0	3941.2	3942.3	3942.0	3929.6	3943.4	3941.2
SSD MASS IN AIR (d)	3952.7	3955.4	3956.4	3958.3	3959.6	3950.8	3953.8	3957.2
MASS IN WATER (e)	2277.6	2276.5	2277.6	2282.0	2284.0	2280.6	2275.2	2279.2
VOLUME (d-e) (f)	1675.1	1678.9	1678.8	1676.3	1675.6	1670.2	1678.6	1678.0
BULK SP. GR. (c-f) (g)	2.348	2.347	2.348	2.352	2.353	2.353	2.349	2.349
MAX. SP.GR. (From Actual Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h-g) / h) (i)	7.2	7.2	7.2	7.0	7.0	7.0	7.1	7.2
VOLUME AIR VOIDS (i X f) / 100 (j)	120.6	120.9	120.9	117.3	117.3	116.9	119.2	120.8
PEAK LOAD (psi) (k)				31600	34400	34200	33900	
DRY TS (2000 X k) + (a X b X 3.1416) (l)				1397.0	1522.4	1516.7	1501.8	
CALC. SSD AT 70% SAT. (0.70 X j) + c	4016.8	4025.6	4025.8	4024.4	4024.1	4011.4	4026.8	4025.8
CALC. SSD AT 80% SAT. (0.80 X j) + c	4028.9	4037.7	4037.9	4036.2	4035.8	4023.1	4038.7	4037.9

SATURATED	MINUTES @	"Hg							
Date and Time in:	01/16/02 9:15AM		Date and Time out:						
SSD MASS (m)		4018.4	4030.3	4033.0					4028.9
MASS IN WATER (n)		2347.0	2354.4	2358.0					2354.1
VOLUME (m-n) (o)		1671.4	1675.9	1675.0					1674.8
VOL. ABS. H2O (m-c) (p)		86.0	89.3	91.8					87.7
% SATURATION 100 X (p-j) (r)		71.3	73.9	75.9					72.6

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4019.0	4033.0	4037.5					4033.5
MASS IN WATER (r)	2347.7	2352.0	2355.6					2352.9
VOLUME (q-r) (s)	1671.3	1681.0	1681.9					1680.6
VOLUME ABS. H2O (q-c) (t)	86.6	92.0	96.3					92.3
% SATURATION 100 X (t-j) (u)	71.8	76.1	79.7					76.4
PEAK LOAD (psi) (v)	23800	24200	24300					25600
WET TS (2 X u) + (a X b X 3.1416) (w)	1052.2	1069.9	1075.4					1131.8
INTERNAL SPECIMEN TEMPERATURE (°F.) (x)	77.00	76.00	76.50	76.00	76.50	76.50	76.50	76.50

	Aver. VTM	Aver. Saturation	Aver. Temp	Median TS	QA/QC Joint Test?	TESTED BY:
Dry Subset	7.1		76.4	1509.3		Clippard/Massey
Wet Subset	7.2	73.4	76.5	1072.6	Circle One	
TENSILE STRENGTH RATIO				71.1	Yes No	CERT. NO.:
QA/QC COMPARATIVE TSR						TESTED BY:
						CERT. NO.:
						LAB LOCATION:
						LAB CERT NO.:

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Visual Stripping:(Circle one)			
None	Minor	Moderate	Severe

Table A-3 TSR Test Data for 0.8% LOF 6500 Antistrip Additives without Prolonged Heating

Revised August 2008

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)

Date Mix Produced:	Mix Type:	JMF No.:	LO8-0-1-8
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: LOF 6500	Additive Dosage:	(%)
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/18/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	96.000	95.800	96.100	96.200	96.000	96.400	96.100	95.700
DRY MASS IN AIR (c)	3911.5	3929.5	3924.7	3938.9	3940.5	3942.9	3939.5	3934.4
SSD MASS IN AIR (d)	3928.2	3949.0	3946.8	3955.7	3956.5	3962.1	3958.5	3961.2
MASS IN WATER (e)	2257.9	2267.8	2274.6	2281.7	2277.1	2276.4	2286.1	2284.2
VOLUME (d-e) (f)	1670.3	1681.2	1672.2	1674.0	1679.4	1685.7	1672.4	1677.0
BULK SP. GR. (c + f) (g)	2.342	2.337	2.347	2.353	2.346	2.339	2.356	2.346
MAX. SP. GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) ÷ h) (i)	7.4	7.6	7.2	7.0	7.3	7.5	6.9	7.3
VOLUME AIR VOIDS (i X f) ÷ 100 (j)	124.3	128.0	120.9	117.1	121.9	127.2	115.3	121.9
PEAK LOAD (Newton's) (k)				23200	22800	21300		22000
DRY TS(kPa) (2000 X k) ÷ (a X b X 3.1416) (l)	*****	*****	*****	1023.5	1008.0	937.8	*****	975.7
CALC. SSD AT 70% SAT. (0.70 X j) ÷ c	3998.5	4019.1	4009.4	4020.9	4025.8	4032.0	4020.2	4019.7
CALC. SSD AT 80% SAT. (0.80 X j) ÷ c	4010.9	4031.9	4021.4	4032.6	4038.0	4044.7	4031.7	4031.9

SATURATED	MINUTES @	"Hg	Date and Time out: 11/17/99 1:00 PM					
Date and Time in:	11/16/99 1:00 PM							
SSD MASS (m)	4010.9	4020.6	4013.1				4030.9	
MASS IN WATER (n)	2331.1	2342.9	2343.3				2351.0	
VOLUME (m - n) (o)	1679.8	1677.7	1669.8	*****	*****	*****	1679.9	*****
VOL. ABS. H2O (m - c) (p)	99.4	91.1	88.4	*****	*****	*****	91.4	*****
% SATURATION 100 X (p ÷ j) (q)	80.0	71.2	73.1	*****	*****	*****	79.3	*****

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4025.3	4032.7	4019.6				4041.4	
MASS IN WATER (r)	2348.4	2356.1	2354.0				2363.5	
VOLUME (q - r) (s)	1676.9	1676.6	1665.6	*****	*****	*****	1677.9	*****
VOLUME ABS. H2O (q - c) (t)	113.8	103.2	94.9	*****	*****	*****	101.9	*****
% SATURATION 100 X (t ÷ j) (u)	91.6	80.6	78.5	*****	*****	*****	88.4	*****
PEAK LOAD (Newton's) (v)	17900	18500	21200				19200	
WET TS(kPa) (2000 X u) ÷ (a X b X 3.1416) (w)	791.4	819.6	936.3	*****	*****	*****	847.9	*****
INTERNAL SPECIMEN TEMPERATURE ("F.) (x)	78.00	78.00	78.00	79.00	78.00	79.00	77.00	78.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint Test?	TESTED BY:
Dry Subset	7.3		78.5	991.8		JASON THOMPSON
Wet Subset	7.3	74.7	77.8	833.8	Circle One	51972
TENSILE STRENGTH RATIO				84.1	(Yes) No	
QA/QC COMPARATIVE TSR						
Visual Stripping: place x in appropriate box					LAB LOCATION:	
					LAB CERT NO.:	
					Comments:	

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Table A-4 TSR Test Data for 0.8% LOF 6500 Antistrip Additives with 2-Hour Prolonged Heating

Revised 11-29-99

NCDOT

M&T 612 (QMS-2)

TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Marshall Compactive Method

Date Mix Produced:	Mix Type:	L08-2	JMF No.:
Contractor:	NCSU	Plant Location:	Plant Cert. No.:
Additive Supplier:	Additive Grade:	Additive Dosage:	
Date Compacted:	No. Gyration:	to height	Date Test Completed: 2/20/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(in) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(in.) (b)	96.000	95.400	95.500	95.700	95.900	95.800	95.500	96.200
DRY MASS IN AIR (c)	3928.3	3942.9	3942.7	3913.2	3905.1	3933.0	3930.5	3919.0
SSD MASS IN AIR (d)	3945.7	3954.3	3955.7	3927.4	3913.3	3949.4	3940.2	3944.5
MASS IN WATER (e)	2263.1	2290.2	2293.0	2260.2	2238.3	2272.4	2269.9	2272.9
VOLUME (d-e) (f)	1682.6	1664.1	1662.7	1667.2	1675.0	1677.0	1670.3	1671.6
BULK SP. GR. (c-f) (g)	2.335	2.369	2.371	2.347	2.331	2.345	2.353	2.344
MAX. SP.GR. (From Actual Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h-g) = h) (i)	7.7	6.3	6.3	7.2	7.8	7.3	7.0	7.3
VOLUME AIR VOIDS (1 X i) = 100 (j)	129.6	104.8	104.8	120.0	130.7	122.4	116.9	122.0
PEAK LOAD (psi) (k)				30900	27300		31200	27300
DRY TS (2000 X k) = (a X b X 3.1416) (l)	*****	*****	*****	1370.4	1208.2	*****	1386.6	1204.4
CALC. SSD AT 70% SAT. (0.70 X j) + c	4019.0	4016.3	4016.0	3997.2	3996.6	4018.7	4012.3	4004.4
CALC. SSD AT 80% SAT. (0.80 X j) + c	4031.9	4026.8	4026.5	4009.2	4009.6	4030.9	4024.0	4016.6

SATURATED	MINUTES @	"Hg	Date and Time out:					
Date and Time in:	01/16/02 9.15AM							
SSD MASS (m)		4026.9	4020.3	4017.2			4019.2	
MASS IN WATER (n)		2348.5	2358.7	2357.1			2346.6	
VOLUME (m-n) (o)		1678.4	1661.6	1660.1	*****	*****	1672.6	*****
VOL. ABS. H2O (m-c) (p)		98.6	77.4	74.5	*****	*****	86.2	*****
% SATURATION 100 X (p + j)		76.1	73.8	71.1	*****	*****	70.4	*****

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4034.9	4029.0	4026.0				4029.9	
MASS IN WATER (r)	2355.7	2366.0	2364.3				2355.6	
VOLUME (q-r) (s)	1679.2	1663.0	1661.7	*****	*****	*****	1674.3	*****
VOLUME ABS. H2O (q-c) (t)	106.6	86.1	83.3	*****	*****	*****	96.9	*****
% SATURATION 100 X (t + j)	82.3	82.1	79.5	*****	*****	*****	79.2	*****
PEAK LOAD (psi) (u)	24400	27200	26800	*****	*****	*****	26000	*****
WET TS (2 X u) = (a X b X 3.1416) (v)	1078.7	1210.1	1191.0	*****	*****	*****	1151.8	*****
INTERNAL SPECIMEN TEMPERATURE ("F.")	77.50	77.50	77.00	76.50	76.00	77.00	77.00	77.00

	Aver. VTM	Aver. Saturation	Aver. Temp	Median TS	QA/QC Joint Test?	TESTED BY:
Dry Subset	7.4		76.6	1289.3		Clippard/Massey
Wet Subset	6.9	72.9	77.3	1171.4	Circle One	
TENSILE STRENGTH RATIO				90.9	Yes No	CERT. NO.:
QA/QC COMPARATIVE TSR						LAB LOCATION:
						LAB CERT NO.:

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Visual Stripping: (Circle one)			
None	Minor	Moderate	Severe

Table A - 5 TSR Test Data for 0.8% LOF 6500 Antistrip Additives with 6-Hour Prolonged Heating

Revised August 2008

NCDOT

M&T 612 (GMS-2)

TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

Date Mix Produced:	Mix Type:	JMF No.:	LO8-6-1-8
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: LOF6500	Additive Dosage: (%)	
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/24/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	96.100	96.200	96.100	95.900	96.000	96.300	96.300	96.000
DRY MASS IN AIR (c)	3933.7	3928.9	3943.6	3939.1	3941.7	3936.8	3934.9	3933.1
SSD MASS IN AIR (d)	3945.9	3942.1	3955.6	3954.1	3954.8	3946.8	3952.5	3946.1
MASS IN WATER (e)	2267.9	2266.2	2275.7	2273.9	2277.1	2269.3	2274.2	2267.5
VOLUME (d-e) (f)	1678.0	1675.9	1679.9	1680.2	1677.7	1677.5	1678.3	1678.6
BULK SP. GR. (c + f) (g)	2.344	2.344	2.348	2.344	2.349	2.347	2.345	2.343
MAX. SP. GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) + h) (i)	7.3	7.3	7.2	7.3	7.1	7.2	7.3	7.4
VOLUME AIR VOIDS (i X f) + 100 (j)	123.2	123.0	121.2	123.2	119.7	121.5	123.0	124.0
PEAK LOAD (Newton's) (k)			34900			35000		32800
DRY TS(kPa) (2000 X k) + (a X b X 3.1416) (l)	*****	*****	1541.3	*****	1547.3	1551.3	*****	1450.1
CALC. SSD AT 70% SAT. (0.70 X j) + c	4019.9	4015.0	4028.4	4025.4	4025.5	4021.8	4021.0	4019.9
CALC. SSD AT 80% SAT. (0.80 X j) + c	4032.2	4027.3	4040.5	4037.7	4037.5	4034.0	4033.3	4032.3

SATURATED	MINUTES @	"Hg	Date and Time out: 11/17/99 1:00 PM					
Date and Time in:	11/16/99 1:00 PM							
SSD MASS (m)		4028.8	4025.6		4026.2			4029.0
MASS IN WATER (n)		2348.5	2344.7		2348.6			2352.0
VOLUME (m - n) (o)		1680.3	1680.9	*****	1677.6	*****	*****	1677.0
VOL. ABS. H2O (m - c) (p)		95.1	96.7	*****	87.1	*****	*****	94.1
% SATURATION 100 X (p + j) (q)		77.2	78.6	*****	70.7	*****	*****	76.5

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4044.6	4042.4		4039.9			4043.1	
MASS IN WATER (r)	2361.5	2359.7		2360.9			2363.1	
VOLUME (q - r) (s)	1683.1	1682.7	*****	1679.0	*****	*****	1680.0	*****
VOLUME ABS. H2O (q - c) (t)	110.9	113.5	*****	100.8	*****	*****	108.2	*****
% SATURATION 100 X (t + j) (u)	90.0	92.3	*****	81.8	*****	*****	88.0	*****
PEAK LOAD (Newton's) (v)	29100	28400		29900			29100	
WET TS(kPa) (2000 X u) + (a X b X 3.1416) (w)	1285.2	1252.9	*****	1323.2	*****	*****	1282.5	*****
INTERNAL SPECIMEN TEMPERATURE ("F.") (x)	79.00	79.00	79.00	79.00	80.00	80.00	79.00	80.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint	TESTED BY:
Dry Subset	7.2		79.8	1544.3	Test?	JASON THOMPSON
Wet Subset	7.3	75.5	79.0	1283.8	Circle One	CERT. NO.: 51972
TENSILE STRENGTH RATIO				83.1	(Yes) No	TESTED BY:
QA/QC COMPARATIVE TSR						CERT. NO.:
						LAB LOCATION:
						LAB CERT NO.:
						Comments:

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Visual Stripping: place x in appropriate box

None Minor Moderate Severe

Table A - 6 TSR Test Data for 0.8% LOF 6500 Antistrip Additives with 12-Hour Prolonged Heating

Revised 11-29-99

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Marshall Compactive Method

M&T 612 (QMS-2)

Date Mix Produced:	Mix Type:	L08-12	JMF No.:
Contractor:	NCSU	Plant Location:	Plant Cert. No.:
Additive Supplier:	Additive Grade:	Additive Dosage:	
Date Compacted:	No. Gyration:	to height	Date Test Completed: 2/22/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(in) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(in) (b)	95.500	95.400	95.300	95.300	95.100	96.400	95.900	95.900
DRY MASS IN AIR (c)	3908.4	3913.0	3929.0	3949.8	3927.7	3935.9	3934.5	3941.6
SSD MASS IN AIR (d)	3919.8	3930.2	3936.5	3962.1	3936.3	3946.0	3941.7	3965.7
MASS IN WATER (e)	2255.9	2265.8	2277.2	2297.1	2275.9	2267.0	2268.2	2291.3
VOLUME (d-e) (f)	1663.9	1664.4	1659.3	1665.0	1660.4	1679.0	1673.5	1674.4
BULK SP. GR. (c + f) (g)	2.349	2.351	2.368	2.372	2.366	2.344	2.351	2.354
MAX. SP. GR. (From Actual Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) ÷ h) (i)	7.2	7.1	6.4	6.2	6.5	7.3	7.1	7.0
VOLUME AIR VOIDS (i X f) ÷ 100 (j)	119.8	118.2	106.2	103.2	107.9	122.6	118.8	117.2
PEAK LOAD (psi) (k)		44400			44400		42400	41100
DRY TS (2000 X k) ÷ (a X b X 3.1416) (l)	*****	1975.3	*****	*****	1981.5	*****	1876.4	1818.9
CALC. SSD AT 70% SAT. (0.70 X j) ÷ c		3992.3	3995.7	4003.3	4022.1	4003.2	4021.7	4017.7
CALC. SSD AT 80% SAT. (0.80 X j) ÷ c		4004.2	4007.5	4014.0	4032.4	4014.0	4034.0	4029.6

SATURATED	MINUTES @	"Hg								
Date and Time in:	01/16/02 9.15AM		Date and Time out:							
SSD MASS (m)	3994.5		4003.4	4023.2		4025.9				
MASS IN WATER (n)	2332.3		2345.5	2361.5		2352.3				
VOLUME (m - n) (o)	1662.2		*****	1657.9	1661.7	*****	1673.6	*****	*****	
VOL. ABS. H2O (m - c) (p)	86.1		*****	74.4	73.4	*****	90.0	*****	*****	
% SATURATION 100 X (p ÷ j) (q)	71.9		*****	70.1	71.1	*****	73.4	*****	*****	

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4013.2		4020.3	4036.4		4043.8		
MASS IN WATER (r)	2342.9		2357.6	2369.2		2362.3		
VOLUME (q - r) (s)	1670.3		*****	1662.7	1667.2	*****	1681.5	*****
VOLUME ABS. H2O (q - c) (t)	104.8		*****	91.3	86.6	*****	107.9	*****
% SATURATION 100 X (t ÷ j) (u)	87.5		*****	86.0	83.9	*****	88.0	*****
PEAK LOAD (psi) (v)	32800		33200	38300		34000		
WET TS (2 X u) ÷ (a X b X 3.1416) (w)	1457.7		*****	1478.5	1705.7	*****	1496.9	*****
INTERNAL SPECIMEN TEMPERATURE ("F.) (x)	78.00		77.50	78.00	77.50	77.00	77.50	77.00

	Aver. VTM	Aver. Saturation	Aver. Temp	Median TS	QA/QC Joint	TESTED BY:
Dry Subset	6.9		77.3	1925.8	Test?	Clippard/Massey
Wet Subset	6.8	71.6	77.8	1487.7	Circle One	
TENSILE STRENGTH RATIO				77.2	Yes No	
QA/QC COMPARATIVE TSR						
Visual Stripping: (Circle one)						
None Minor Moderate Severe						

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Table A - 7 TSR Test Data for 0.8% LOF 6500 Antistrip Additives with 24-Hour Prolonged Heating

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)
L08-24-1THRU-8

Date Mix Produced:	Mix Type:	JMF No.:	Pending
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier: MORELIFE	Additive Grade:	Additive Dosage:	(%)
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/10/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	97.700	97.200	97.600	97.200	98.500	97.600	97.400	97.500
DRY MASS IN AIR (c)	3934.7	3934.5	3935.1	3944.9	3940.3	3946.3	3939.6	3938.3
SSD MASS IN AIR (d)	3946.3	3947.6	3949.2	3952.5	3953.4	3957.3	3952.1	3950.8
MASS IN WATER (e)	2263.5	2264.8	2267.3	2264.9	2267.3	2273.6	2268.9	2267.5
VOLUME (d-e) (f)	1682.8	1682.8	1681.9	1687.6	1686.1	1683.7	1683.2	1683.3
BULK SP. GR. (c + f) (g)	2.338	2.338	2.340	2.338	2.337	2.344	2.341	2.340
MAX. SP.GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) + h) (i)	7.6	7.6	7.5	7.6	7.6	7.4	7.5	7.5
VOLUME AIR VOIDS (i X f) + 100 (j)	127.6	127.7	126.5	128.4	128.7	123.9	126.0	126.7
PEAK LOAD (Newton's) (k)					27600	24800	29800	28400
DRY TS(kPa) (2000 X k) + (a X b X 3.1416) (l)	*****	*****	*****	*****	1189.2	1078.4	1298.5	1236.2
CALC. SSD AT 70% SAT. (0.70 X j) + c	4024.0	4023.9	4023.7	4034.7	4030.4	4033.0	4027.8	4027.0
CALC. SSD AT 80% SAT. (0.80 X j) + c	4036.8	4036.6	4036.3	4047.6	4043.2	4045.4	4040.4	4039.6

SATURATED	MINUTES @	"Hg								
Date and Time in:	11/16/99 1:00 PM		Date and Time out:	11/17/99 1:00 PM						
SSD MASS (m)	4040.0	4039.4	4013.9	4029.9						
MASS IN WATER (n)	2342.9	2347.5	2317.2	2337.9						
VOLUME (m - n) (o)	1697.1	1691.9	1696.7	1692.0	*****	*****	*****	*****		
VOL. ABS. H2O (m - c) (p)	105.3	104.9	78.8	85.0	*****	*****	*****	*****		
% SATURATION 100 X (p + j)	82.5	82.2	62.3	66.2	*****	*****	*****	*****		

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4065.8	4067.7	4067.2	4069.0				
MASS IN WATER (r)	2357.6	2363.8	2355.2	2364.2				
VOLUME (q - r) (s)	1708.2	1703.9	1712.0	1704.8	*****	*****	*****	*****
VOLUME ABS. H2O (q - c) (t)	131.1	133.2	132.1	124.1	*****	*****	*****	*****
% SATURATION 100 X (t + j)	102.8	104.3	104.4	96.7	*****	*****	*****	*****
PEAK LOAD (Newton's) (u)	22000	20600	19300	21800				
WET TS(kPa) (2000 X u) + (a X b X 3.1416) (v)	955.7	899.5	839.3	951.9	*****	*****	*****	*****
INTERNAL SPECIMEN TEMPERATURE (°F.)	78.00	78.00	78.00	78.00	79.00	79.00	79.00	79.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint	TESTED BY:
Dry Subset	7.5	79.0	79.0	1212.7	Test?	JASON THOMPSON
Wet Subset	7.6	73.3	78.0	925.7	Circle One	51972
TENSILE STRENGTH RATIO				76.3	(Yes) No	
QA/QC COMPARATIVE TSR						

Visual Stripping: place x in appropriate box

None	Minor	Moderate	Severe

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Table A - 8 TSR Test Data for 0.8% Morelife 2200 Antistrip Additives without Prolonged Heating

Revised August 2008

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)

Date Mix Produced:	Mix Type:	JMF No.:	MO6-00-1-8
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: MORLIFE	Additive Dosage:	(%)
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/17/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	95.600	96.000	96.400	96.600	95.900	95.800	96.000	96.000
DRY MASS IN AIR (c)	3945.2	3936.9	3941.2	3937.5	3911.1	3941.7	3911.0	3943.5
SSD MASS IN AIR (d)	3956.0	3947.1	3949.6	3947.6	3921.4	3952.4	3924.4	3952.9
MASS IN WATER (e)	2277.7	2269.4	2267.5	2266.7	2244.5	2275.1	2249.4	2273.8
VOLUME (d-e) (f)	1678.3	1677.7	1682.1	1680.9	1676.9	1677.3	1675.0	1679.1
BULK SP. GR. (c + f) (g)	2.351	2.347	2.343	2.342	2.332	2.350	2.335	2.349
MAX. SP. GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) + h) (i)	7.1	7.2	7.4	7.4	7.8	7.1	7.7	7.2
VOLUME AIR VOIDS (i X f) + 100 (j)	118.9	121.6	124.3	124.6	131.0	119.3	129.2	120.4
PEAK LOAD (Newton's) (k)		20000	20000				19100	20000
DRY TS(kPa) (2000 X k) + (a X b X 3.1416) (l)		884.2	880.5				844.4	884.2
CALC. SSD AT 70% SAT. (0.70 X j) + c	4028.5	4022.0	4028.2	4024.7	4002.8	4025.2	4001.4	4027.8
CALC. SSD AT 80% SAT. (0.80 X j) + c	4040.3	4034.2	4040.7	4037.2	4015.9	4037.2	4014.3	4039.8

SATURATED	MINUTES @	"Hg	Date and Time out: 11/17/99 1:00 PM					
Date and Time in:	11/16/99 1:00 PM							
SSD MASS (m)	4028.6		4035.3	4014.3	4035.1			
MASS IN WATER (n)	2351.0		2353.8	2334.9	2357.1			
VOLUME (m - n) (o)	1677.6		1681.5	1679.4	1678.0			
VOL. ABS. H2O (m - c) (p)	83.4		97.8	103.2	93.4			
% SATURATION 100 X (p - j)	70.1		78.5	78.8	78.3			

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4043.3		4050.8	4031.2	4048.3			
MASS IN WATER (r)	2363.4		2365.0	2348.5	2367.9			
VOLUME (q - r) (s)	1679.9		1685.8	1682.7	1680.4			
VOLUME ABS. H2O (q - c) (t)	98.1		113.3	120.1	106.6			
% SATURATION 100 X (t - j)	82.5		90.9	91.7	89.3			
PEAK LOAD (Newton's) (u)	18400		17200	16700	18000			
WET TS(kPa) (2000 X u) + (a X b X 3.1416) (v)	816.9		755.7	739.1	797.4			
INTERNAL SPECIMEN TEMPERATURE ("F.)	78.00	79.00	79.00	78.00	78.00	78.00	79.00	79.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint Test?	TESTED BY:
Dry Subset	7.4		79.0	882.4		JASON THOMPSON
Wet Subset	7.4	76.4	78.0	776.6	Circle One	51972
TENSILE STRENGTH RATIO				88.0	(Yes) No	
QA/QC COMPARATIVE TSR						
Visual Stripping: place x in appropriate box					LAB LOCATION:	
None Minor Moderate Severe					LAB CERT NO.:	
Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA					Comments:	

Table A- 9 TSR Test Data for 0.8% Morelife 2200 Antistrip Additives with 2-Hour Prolonged Heating

Revised August 2008

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)

Date Mix Produced:	Mix Type:	JMF No.:	MO6-02-1-8
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: MORELIFE	Additive Dosage:	(%)
Date Compacted:	No. Gyration:	To height:	Date Test Completed:

SPECIMEN NUMBER		1	2	3	4	5	6	7	8
DIAMETER(mm)	(a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm)	(b)	96.000	95.900	95.900	95.600	95.800	96.200	95.600	95.700
DRY MASS IN AIR	(c)	3936.1	3920.5	3921.4	3940.5	3917.7	3935.8	3928.0	3943.1
SSD MASS IN AIR	(d)	3945.4	3934.8	3935.6	3955.2	3928.0	3951.6	3944.8	3954.7
MASS IN WATER	(e)	2273.4	2259.1	2260.9	2281.3	2252.2	2282.4	2274.3	2278.3
VOLUME	(d-e) (f)	1672.0	1675.7	1674.7	1673.9	1675.8	1669.2	1670.5	1676.4
BULK SP. GR.	(c + f) (g)	2.354	2.340	2.342	2.354	2.338	2.358	2.351	2.352
MAX. SP.GR. (Optimum Binder Content Rice Test)	(h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) / h)	(i)	7.0	7.5	7.4	7.0	7.6	6.8	7.1	7.0
VOLUME AIR VOIDS (i X f) / 100	(j)	116.2	126.1	124.7	116.4	127.3	113.5	117.9	117.9
PEAK LOAD (Newton's)	(k)					27000	29100	28500	28100
DRY TS(kPa) (2000 X k) - (a X b X 3.1416)	(l)	*****	*****	*****	*****	1196.2	1283.8	1265.2	1246.2
CALC. SSD AT 70% SAT. (0.70 X j) + c		4017.5	4008.8	4008.7	4022.0	4006.8	4015.3	4010.6	4025.6
CALC. SSD AT 80% SAT. (0.80 X j) + c		4029.1	4021.4	4021.2	4033.6	4019.5	4026.6	4022.3	4037.4

SATURATED	MINUTES @	"Hg							
Date and Time in:	11/16/99 1:00 PM		Date and Time out:	11/17/99 1:00 PM					
SSD MASS	(m)	4025.6	4015.1	4019.3	4024.3				
MASS IN WATER	(n)	2353.0	2336.7	2341.3	2349.3				
VOLUME	(m - n) (o)	1672.6	1678.4	1678.0	1675.0	*****	*****	*****	*****
VOL. ABS. H2O	(m - c) (p)	89.5	94.6	97.9	83.8	*****	*****	*****	*****
% SATURATION	100 X (p - j)	77.0	75.0	78.5	72.0	*****	*****	*****	*****

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS	(q)	4038.1	4031.7	4030.9	4035.8				
MASS IN WATER	(r)	2363.1	2351.0	2353.6	2361.0				
VOLUME	(q - r) (s)	1675.0	1680.7	1677.3	1674.8	*****	*****	*****	*****
VOLUME ABS. H2O	(q - c) (t)	102.0	111.2	109.5	95.3	*****	*****	*****	*****
% SATURATION	100 X (t - j)	87.8	88.2	87.8	81.9	*****	*****	*****	*****
PEAK LOAD (Newton's)	(u)	24500	23200	24000	26000				
WET TS(kPa) (2000 X u) - (a X b X 3.1416)	(v)	1083.1	1026.7	1062.1	1154.3	*****	*****	*****	*****
INTERNAL SPECIMEN TEMPERATURE ("F .)		77.00	77.00	77.00	77.00	78.00	78.00	78.00	78.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint	TESTED BY:
Dry Subset	7.1		78.0	1255.7	Test?	JASON THOMPSON
Wet Subset	7.2	75.6	77.0	1072.6	Circle One	CERT. NO.: 51972
TENSILE STRENGTH RATIO				85.4	(Yes) No	TESTED BY:
QA/QC COMPARATIVE TSR						CERT. NO.:
						LAB LOCATION:
						LAB CERT NO.:
						Comments:

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Visual Stripping: place x in appropriate box

None	Minor	Moderate	Severe
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Table A- 10 TSR Test Data for 0.8% Morelife 2200 Antistrip Additives with 6-Hour Prolonged Heating

Revised August 2006

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)

MO8-6-1-THRI

Date Mix Produced:	Mix Type:	JMF No.:	Pending
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: MORELIFE	Additive Dosage:	(%)
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/11/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	96.400	97.400	97.100	95.800	96.400	96.200	95.700	95.900
DRY MASS IN AIR (c)	3940.3	3942.4	3939.6	3936.9	3938.3	3916.8	3928.8	3936.4
SSD MASS IN AIR (d)	3950.7	3953.0	3950.7	3950.3	3951.3	3930.7	3941.0	3950.1
MASS IN WATER (e)	2270.6	2266.5	2267.4	2266.6	2266.5	2249.4	2258.5	2268.9
VOLUME (d-e) (f)	1680.1	1686.5	1683.3	1683.7	1684.8	1681.3	1682.5	1681.2
BULK SP. GR. (c + f) (g)	2.345	2.338	2.340	2.338	2.338	2.330	2.335	2.341
MAX. SP. GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) - h) (i)	7.3	7.6	7.5	7.6	7.6	7.9	7.7	7.5
VOLUME AIR VOIDS (i X f) + 100 (j)	122.7	128.2	126.1	127.6	128.2	133.2	129.6	125.3
PEAK LOAD (Newton's) (k)					32800	32400	34300	33400
DRY TS(kPa) (2000 X k) + (a X b X 3.1416) (l)	*****	*****	*****	*****	1444.1	1429.4	1521.1	1478.1
CALC. SSD AT 70% SAT. (0.70 X j) + c	4026.2	4032.2	4027.9	4026.2	4028.0	4010.0	4019.5	4024.1
CALC. SSD AT 80% SAT. (0.80 X j) + c	4038.4	4045.0	4040.5	4039.0	4040.8	4023.3	4032.5	4036.6

SATURATED	MINUTES @	"Hg	Date and Time out: 11/17/99 1:00 PM						
Date and Time in:	11/16/99 1:00 PM								
SSD MASS (m)	4026.6	4041.9	4036.8	4030.4					
MASS IN WATER (n)	2350.1	2359.2	2356.7	2352.1					
VOLUME (m - n) (o)	1676.5	1682.7	1680.1	1678.3	*****	*****	*****	*****	*****
VOL. ABS. H2O (m - c) (p)	86.3	99.5	97.2	93.5	*****	*****	*****	*****	*****
% SATURATION 100 X (p + j) (r)	70.4	77.6	77.1	73.3	*****	*****	*****	*****	*****

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4042.2	4058.3	4051.7	4049.5				
MASS IN WATER (r)	2360.2	2369.1	2365.5	2364.9				
VOLUME (q - r) (s)	1682.0	1689.2	1686.2	1684.6	*****	*****	*****	*****
VOLUME ABS. H2O (q - c) (t)	101.9	115.9	112.1	112.6	*****	*****	*****	*****
% SATURATION 100 X (t + j) (u)	83.1	90.4	88.9	88.2	*****	*****	*****	*****
PEAK LOAD (Newton's) (v)	28000	25700	26000	27000				
WET TS(kPa) (2000 X u) + (a X b X 3.1416) (w)	1232.7	1119.9	1136.4	1196.2	*****	*****	*****	*****
INTERNAL SPECIMEN TEMPERATURE (°F) (x)	79.00	79.00	79.00	79.00	79.00	79.00	79.00	79.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint Test?	TESTED BY:
Dry Subset	7.7		79.0	1461.1		JASON THOMPSON 51972
Wet Subset	7.5	74.6	79.0	1166.3	Circle One	TESTED BY:
TENSILE STRENGTH RATIO				79.8	(Yes) No	CERT. NO.:
QA/QC COMPARATIVE TSR						LAB LOCATION:
						LAB CERT NO.:
						Comments:

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA

Visual Stripping: place x in appropriate box

None	Minor	Moderate	Severe
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Table A- 11 TSR Test Data for 0.8% Morelife 2200 Antistrip Additives with 12-Hour Prolonged Heating

Revised August 2006

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)

MO6-12-1-E

Date Mix Produced:	Mix Type:	JMF No.:	Pending
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: MORELIFE	Additive Dosage:	(%)
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/15/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	96.000	96.000	95.800	95.600	95.600	95.700	96.200	95.500
DRY MASS IN AIR (c)	3937.1	3932.5	3939.5	3937.3	3939.8	3928.9	3934.6	3934.7
SSD MASS IN AIR (d)	3946.1	3939.3	3948.7	3945.5	3946.3	3942.4	3947.2	3940.1
MASS IN WATER (e)	2274.1	2262.3	2273.8	2271.2	2272.8	2269.7	2277.8	2269.0
VOLUME (d-e) (f)	1672.0	1677.0	1674.9	1674.3	1673.5	1672.7	1669.4	1671.1
BULK SP. GR. (c + f) (g)	2.355	2.345	2.352	2.352	2.354	2.349	2.357	2.355
MAX. SP. GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) + h) (i)	6.9	7.3	7.0	7.1	6.9	7.2	6.8	6.9
VOLUME AIR VOIDS (i X f) + 100 (j)	115.8	122.7	117.8	118.1	116.3	119.8	114.2	115.9
PEAK LOAD (Newton's) (k)				41200		40100	41400	40500
DRY TS(kPa) (2000 X k) - (a X b X 3.1416) (l)	*****	*****	*****	1829.1	*****	1778.4	1826.5	1799.9
CALC. SSD AT 70% SAT. (0.70 X j) + c	4018.2	4018.4	4021.9	4019.9	4021.2	4012.7	4014.6	4015.8
CALC. SSD AT 80% SAT. (0.80 X j) + c	4029.8	4030.6	4033.7	4031.7	4032.8	4024.7	4026.0	4027.4

SATURATED	MINUTES @	"Hg	Date and Time out: 11/17/99 1:00 PM					
Date and Time in:	11/16/99 1:00 PM							
SSD MASS (m)	4024.8	4022.9	4026.7		4024.6			
MASS IN WATER (n)	2352.6	2345.7	2353.1		2351.9			
VOLUME (m - n) (o)	1672.2	1677.2	1673.6	*****	1672.7	*****	*****	*****
VOL. ABS. H2O (m - c) (p)	87.7	90.4	87.2	*****	84.8	*****	*****	*****
% SATURATION 100 X (p + j) (r)	75.7	73.7	74.0	*****	72.9	*****	*****	*****

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4042.8	4042.6	4044.3		4044.1			
MASS IN WATER (r)	2364.4	2358.7	2364.2		2364.4			
VOLUME (q - r) (s)	1678.4	1683.9	1680.1	*****	1679.7	*****	*****	*****
VOLUME ABS. H2O (q - c) (t)	105.7	110.1	104.8	*****	104.3	*****	*****	*****
% SATURATION 100 X (t + j) (u)	91.3	89.8	89.0	*****	89.7	*****	*****	*****
PEAK LOAD (Newton's) (v)	30900	31600	33400		34500			
WET TS(kPa) (2000 X u) - (a X b X 3.1416) (v)	1366.1	1397.0	1479.7	*****	1531.6	*****	*****	*****
INTERNAL SPECIMEN TEMPERATURE ("F.)	79.00	79.00	79.00	78.00	79.00	78.00	79.00	79.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint Test?	TESTED BY:
Dry Subset	7.0		78.5	1813.2		JASON THOMPSON
Wet Subset	7.1	74.1	79.0	1438.4	Circle One	51972
TENSILE STRENGTH RATIO				79.3	(Yes) No	
QA/QC COMPARATIVE TSR						
Visual Stripping: place x in appropriate box					LAB LOCATION:	
None Minor Moderate Severe					LAB CERT NO.:	
Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA					Comments:	

Table A- 12 TSR Test Data for 0.8% Morelife 2200 Antistrip Additives with 24-Hour Prolonged Heating

NCDOT
TENSILE STRENGTH RATIO (TSR) TEST WORKSHEET
Gyratory Compactive Method

M&T 612 (QMS-2)
MO8-24-1-THR

Date Mix Produced:	Mix Type:	JMF No.:	Pending
Contractor: NC STATE	Plant Location:	Plant Cert. No.:	
Additive Supplier:	Additive Grade: MORELIFE	Additive Dosage:	(%)
Date Compacted:	No. Gyration: To height	Date Test Completed:	5/11/2007

SPECIMEN NUMBER	1	2	3	4	5	6	7	8
DIAMETER(mm) (a)	150.000	150.000	150.000	150.000	150.000	150.000	150.000	150.000
THICKNESS(mm) (b)	98.900	98.500	97.800	96.400	97.700	98.600	99.100	99.800
DRY MASS IN AIR (c)	3937.4	3945.2	3917.9	3936.5	3948.2	3940.6	3924.6	3934.9
SSD MASS IN AIR (d)	3946.4	3956.2	3926.3	3947.2	3963.5	3948.6	3935.7	3942.8
MASS IN WATER (e)	2258.7	2262.5	2247.6	2264.6	2268.7	2256.3	2252.3	2257.9
VOLUME (d-e) (f)	1687.7	1693.7	1678.7	1682.6	1694.8	1692.3	1683.4	1684.9
BULK SP. GR. (c + f) (g)	2.333	2.329	2.334	2.340	2.330	2.329	2.331	2.335
MAX. SP.GR. (Optimum Binder Content Rice Test) (h)	2.530	2.530	2.530	2.530	2.530	2.530	2.530	2.530
% AIR VOIDS (100 X (h - g) + h) (i)	7.8	7.9	7.8	7.5	7.9	8.0	7.9	7.7
VOLUME AIR VOIDS (i X f) + 100 (j)	131.4	134.3	130.1	126.7	134.2	134.8	132.2	129.6
PEAK LOAD (Newton's) (k)					31000	21800	19400	16800
DRY TS(kPa) (2000 X k) + (a X b X 3.1416) (l)	*****	*****	*****	*****	1346.7	938.4	830.8	714.4
CALC. SSD AT 70% SAT. (0.70 X j) + c	4029.4	4039.2	4009.0	4025.2	4042.2	4034.9	4017.1	4025.6
CALC. SSD AT 80% SAT. (0.80 X j) + c	4042.5	4052.7	4022.0	4037.8	4055.6	4048.4	4030.3	4038.6

SATURATED	MINUTES @	"Hg							
Date and Time in:	11/16/99 1:00 PM		Date and Time out: 11/17/99 1:00 PM						
SSD MASS (m)	4040.6	4051.0	4021.0	4025.4					
MASS IN WATER (n)	2329.8	2344.1	2323.6	2346.7					
VOLUME (m - n) (o)	1710.8	1706.9	1697.4	1678.7	*****	*****	*****	*****	
VOL. ABS. H2O (m - o) (p)	103.2	105.8	103.1	88.9	*****	*****	*****	*****	
% SATURATION 100 X (p - j)	78.5	78.8	79.2	70.2	*****	*****	*****	*****	

CONDITIONED 24 HOURS IN 140 DEGREE WATER

SSD MASS (q)	4091.9	4082.6	4062.5	4051.7				
MASS IN WATER (r)	2360.9	2361.6	2347.6	2358.7				
VOLUME (q - r) (s)	1731.0	1721.0	1714.9	1693.0	*****	*****	*****	*****
VOLUME ABS. H2O (q - s) (t)	154.5	137.4	144.6	115.2	*****	*****	*****	*****
% SATURATION 100 X (t + j)	117.6	102.3	111.1	90.9	*****	*****	*****	*****
PEAK LOAD (Newton's) (u)	9600	17300	10300	23200				
WET TS(kPa) (2000 X u) + (a X b X 3.1416) (v)	412.0	745.4	447.0	1021.4	*****	*****	*****	*****
INTERNAL SPECIMEN TEMPERATURE ("F .)	79.00	79.00	79.00	79.00	79.00	79.00	79.00	79.00

	Aver. VTM	Aver. Saturation	Aver. Temp.	Median TS	QA/QC Joint	TESTED BY:
Dry Subset	7.9	79.0	884.6	Test?		JASON THOMPSON
Wet Subset	7.7	76.7	79.0	596.2	Circle One	51972
TENSILE STRENGTH RATIO				67.4	(Yes) No	
QA/QC COMPARATIVE TSR						
Visual Stripping: place x in appropriate box						LAB LOCATION:
None Minor Moderate Severe						LAB CERT NO.:
						Comments:

Note: Attach proposed M&T 601 form when TSR specimens are being submitted to QA