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Storage Stability and Morphology of Latex Modified Bitumen

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Abstract: ***Bitumen modification is done to enhance the properties of bitumen related to elasticity, temperature susceptibility, softening point etc. This research intends to assess the effects of natural rubber latex (NRL) in liquid form as a bitumen modifier. Conventional tests, temperature susceptibility and phase separation due to hot storage were investigated using two separate mixing speeds of 1200 and 1300 revolution per minutes (rev/min). Morphology due to the addition of NRL has also been explored utilizing Atomic Force Microscopy (AFM). NRL was incorporated into the bitumen by weight of the binder for the modification at three different amounts (i.e. 3%, 5% and 6%). Based on the softening point, penetration value, temperature susceptibility and storage stability the latex-modified asphalt binder were analyzed. Results of the investigation showed that owing to enhancement in viscoelastic properties, the latex-modified asphalt binder will not undergo phase separation during hot temperature storage. Meanwhile, a uniform dispersed network was indicated by the morphological analysis with the presence of three phases of para, peri and cata. It can be deduced from the results obtained that the latex-modified asphalt binders will perform better in terms of softening point, penetration value and susceptibility due to temperature.***

Keywords: ***Natural rubber latex, modified bitumen, Atomic Force Microscopy, Temperature susceptibility, storage stability.***

I. INTRODUCTION

Bitumen consists of a highly complex hydrocarbon mixture and its nature depends heavily on temperature and time of processing [1, 2]. Bitumen as a colloidal structure is made up of oily light maltenes with lesser molecular weight and asphalt micelles with elevated molecular weight spread in maltenes [3].

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The rising volume of traffic and axle loads worldwide coupled with the underperformance of unmodified bitumen under varying conditions of weather has led to a greater emphasis on bitumen modification to mitigate the impacts of damage due to moisture, deformations, and fatigue cracking [4, 5]. Polymers such as thermoplastic elastomers and plastomers have been identified as one of the better materials used for the modification of bitumen after showing several enhancements on modified binders [6].

For many centuries, asphalt binders have been modified to fulfill optimum binder stiffness, decreasing asphalt pavement susceptibility to rutting and thermal cracking. The utilization of biomass and bio-waste products to boost asphalt binder properties has been investigated by various researchers [7-10]. Researchers have studied natural rubber latex (NRL) applications as a biopolymer [10-15] primarily to enhance the rheological characteristics of asphalt binders at varying temperatures and loading conditions. In this context [14] identified that a modified asphalt binder using NRL at different percentages resulted in enhanced resistance to both rutting and fatigue compared to the specimens fabricated utilizing base asphalt binder. The studies mostly recommended the optimal amount of NRL to be between 5%-7% by weight of the binder [12, 14, 15]. NRL was reported to be an effective modifier resulting in increased rutting resistance of asphalt binder at low and intermediate temperatures [12, 14, 16].

The primary advantages of adding polymers as modifiers includes reduced temperature sensitivity, improved resistant to permanent deformation, increased rigidity at high temperatures, better moisture resistance, cracking resistance at lower temperatures, and prolonged fatigue life [17]. The utilization of polymer materials such as plastics and rubber helps to reduce the issues related to pollution of the environment [18]. Rubber is among the polymers utilized to modify bitumen to improve its properties, specifically elasticity, stability and resistance to fatigue. Natural rubber is among the polymers most readily available and its thermal and binding abilities have resulted in its utilization in the modification of bitumen to increase the service life of pavements [19-21].

Natural rubber Latex is available worldwide and is used to manufacture a wide variety of life products such as boots, mattresses, balloons, gloves and swim caps in large amounts. These products should be recycled to avoid environmental pollution [19].

The concept of incorporating natural rubber latex (NRL) in bitumen modification started more than three decades ago when it was utilized to enhance bitumen performance in asphalt surface course [18]. The usage of natural rubber latex is regarded as a good way to improve bitumen properties as it includes natural rubber, and distinct rubber particles can quickly be mixed with bitumen. Natural rubber latex serves as an elastic band in cold weather, which aids to prevent cracks whilst preserving bitumen rigidity. As the temperature rises, natural rubber serves as a film that increases the shear resistance, thus preventing the flow of bitumen [14, 22]. The use of natural rubber latex has generally been shown to drastically enhance the long-term performance of pavement [16]. For bitumen modification liquid natural rubber (LNR) is preferred to latex as it combines better with binder and creates a more homogeneous mix. The abundance of liquid natural rubber gives it a unique benefit over synthetic rubbers which in turn increase the quality and advantages of utilizing natural rubber [22].

Given the enhancements made by modifying bitumen with NRL, there are still several issues. The main issues associated with polymer-modified bitumen include lack of morphological stability during hot storage (phase separation), poor compatibility with bitumen and higher modification costs among others [17, 23]. Therefore, the optimal quantity of liquid natural rubber and the rate of shearing needs to be investigated further to solve the major issues highlighted above in regions and countries that produced large amount of natural rubber such as Malaysia.

Numerous researches have been conducted on rheological properties of natural rubber latex modified bitumen. However, less emphasis was given to the use of natural rubber latex to boost the performance of asphalt binder and the effect of different mixing speed. The main objectives of this study is to characterize the properties of latex-modified bitumen prepared with two different mixing rates of 1200 rev/min and 1300 rev/min through conventional and empirical techniques as well as evaluation of the storage stability, temperature susceptibility and its morphology.

II. MATERIALS AND LATEX MODIFICATION PROCEDURE

A. Materials

The base bitumen used is 60/70 penetration grade in this research. Table 1 presents the physical properties of the base bitumen used. To prepare latex-modified blends, natural rubber latex (NRL) in liquid form obtained from Lembaga Getah Malaysia was used.

Table 1: Physical properties of the base binder

Physical Property	Results
Penetration (25ºC, 100g, 5s, 0.1mm)	64dmm
Softening Point temperature	49ºC
Ductility at 25ºC	>150cm
Specific Gravity	1.03

B. Preparation of latex-modified bitumen

The preparation of latex-modified asphalt binders is the first step of the process. The latex-modified asphalt binder was prepared by weight of binder using 3%, 5% and 6% of NRL. The asphalt binder was preheated in an oven at 160°C and the temperature of mixing was maintained during the blending process using a thermo-cell. NRL was then gently poured into the preheated base bitumen in liquid form to prevent excess bubbles and mixed for 2hours to produce a homogeneous modified blend using a propeller blade benchtop multi-mix high shear mixer at 1200 rev/min and 1300 rev/min. For basic conventional tests, temperature susceptibility, storage stability and morphology, the base and latex-modified asphalt binders were tested at two separate mixing speeds.

C. Experimental Methodology

i. Penetration and Softening point tests

Fundamental tests including penetration and softening point on the unmodified and latex-modified asphalt binders have been performed. The penetration test was performed to determine the level of penetration and to assess the asphalt binder consistency. While the softening point test was performed to assess the asphalt binder ability to maintain high temperatures until it began to soften. Based on ASTM D5 (ASTM 1997) and ASTMD36 (ASTM 1995), the penetration and softening point tests were performed respectively. Table 2 depicts the values of penetration and softening point for unmodified and latex-modified asphalt binders for the two mixing speed of 1200 rev/min and 1300 rev/min.

Table 2: Penetration and Softening point for control and latex-modified binders

NRL content (%)	1200 rev/min		1300 rev/min	
	Penetration	*Softening point*	*Penetration*	*Softening point*
0	63	52	61	50
3	44	51	44	52
5	40	54	46	53
6	34	56	39	56

ii. Temperature Susceptibility test

Temperature sensitivity test was measured in terms of penetration index (PI) for the base and latex-modified bitumen. As a function of temperature, bitumen temperature susceptibility is defined as the changes in its rheological parameters. Penetration and softening point temperature are utilized to determine the PI. The penetration index is estimated from Shell Bitumen Handbook [24] using Eq. 1.

$$PI_{TR\&B} = \frac{1952 - 500 log P_{25} - 20T_{R\&B}}{500 log P_{25} - T_{R\&B} - 120} \quad (1)$$

iii. Storage Stability and Phase separation

Hot storage stability test is applied to estimate high temperature stability of the base and latex-modified bitumen under storage conditions. Tube test (PN-EN 13399) specification was followed; aluminum foil tube was filled up with melted modified binder and stored vertically inside the oven for 72hours at a temperature of 163ºC. After the samples reach the predefined time, they are moved out and cooled at a temperature of -20ºC for 4 hours using a refrigerator. The samples were then cut into three equal sections. The samples stability under high temperature storage conditions was considered by evaluating the difference in softening point temperatures between the top and bottom section. For stable samples, the difference in softening point falls below 4ºC [5, 25]. Table 3 presents the hot temperature storage stability of the base and latex-modified bitumen.

Table 3: Hot storage temperature stability

Latex Content (%)	Softening Point (ºC)		
	Top	*Bottom*	*Discrepancy*
0	50.4	50.2	0.2
3	59.8	58.6	1.2
5	61.2	59.3	1.9
6	64.7	62.3	2.4

iv. Atomic Force Microscopy

The surface morphology of the samples was evaluated using Atomic force Microscopy (AFM) model SII NANO NAVI E-Sweep. AFM was used to study the internal morphology of the base and latex-modified bitumen. AFM images gives information in three dimensional topography and phase. The samples examined identified the degree of continuous process in the blend and the bitumen-latex compatibility within the blend. As higher molecular weight latex is incompatible with lower molecular weight bitumen, the compatibility of bitumen with latex depends on molecular weight. The AFM can work in several modes Contact Mode (CM), Dynamic Force Mode (DFM), and Non-Contact (AC) Mode. Non-contact mode is generally preferred due to the composition and topography of asphalt bitumen as it minimizes the normal and lateral forces (suitable for soft or viscous materials).

Morphological investigation was done to study the development of structure brought by chemical changes in the virgin bitumen after adjustment utilizing AFM. The analyzed samples depict the degree of the consistent stage in the mix and compatibility of the bitumen–latex. AFM test was conducted to examine the changes occurring due to the addition of various NRL contents to the virgin bitumen. The test provides information relating to the binders increase in stiffness, adhesion, and other features at nano scale.

The samples were prepared by preheating the sample of base and latex-modified binders in an oven at 160ºC and mixed for 2hours; later a drop of the bitumen was placed in the microscopic slide and kept at room temperature for 24hours as shown in Fig. 1. After that, the samples were tested for morphological examination utilizing the AFM as depicted in Fig. 2. Topographical and surface information were obtained.

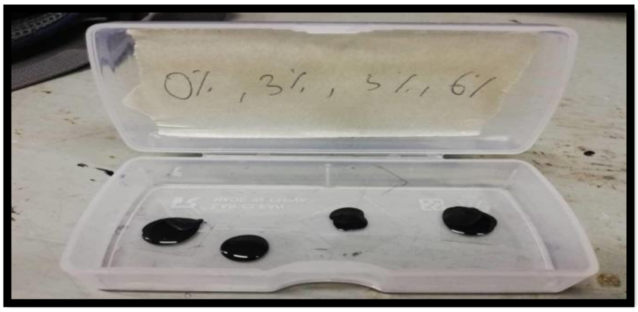


Fig. 1. Prepared samples for AFM test

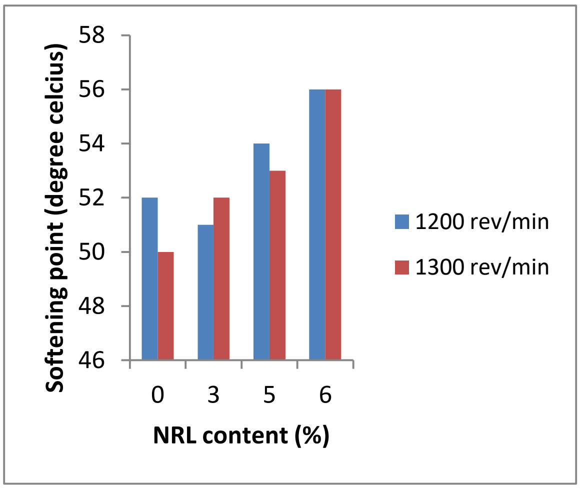
Fig. 2. AFM set up

III. RESULTS AND DISCUSSION

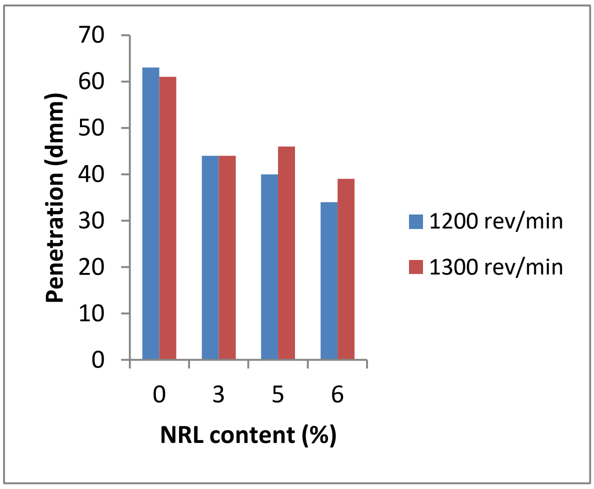
A. *Penetration and Softening point*

The outcomes of the penetration and softening point tests can be seen in Fig. 3 (a and b). The consistency of the base binder and latex-modified binder was determined through the penetration test while the softening point test was used to check the asphalt binder highest service temperatures. It can be seen that 3% NRL content in the latex-modified binder having a mixing rate of 1200 rev/min and 1300 rev/min were found to have little difference or equivalent softening point values to the base binder. By comparison, 5% and 6% latex-modified binders displayed higher softening point values than the base binder. This resulting softening point relies on the elastic nature of NRL and its concentration. For the two mixing speed, the 6% NRL content mixes have the highest softening point of 56°C, whereas the lowest softening point value was found to be 51°C for the 3% NRL content prepared with 1200 rev/min mixing rate. It can, therefore, be deduced that the values of the softening depends on the amount of NRL with the mixing rate having little or no effect. Fig. 3b also shows that the penetration value of the latex-modified binder decreased for the two mixing rate in comparison to the base asphalt binder with an increase in NRL content. Furthermore, it demonstrates that the penetration values of the latex-modified binder containing 3%, 5% and 6% NRL showed good performance relative to the base binder as the penetration values were decreased with increased NRL contents for the two mixing speeds.

It can, therefore, be concluded that the NRL contents has a more pronounced effect on the latex-modified binder penetration values than the mixing speed. The improvement in the stiffness of the latex-modified binder can be accredited to the hardening effect of adding NRL to the base binder. This, therefore, demonstrates that the introduction of NRL has a greater impact on the asphalt binder penetration and softening point than the mixing speed. Nonetheless, the mixing rate of 1200 rev/min has a better performance with the lowest penetration value of 34dmm at 6% NRL compared to 39dmm for 1300 rev/min at same NRL amount.



(a)



(b)

Fig. 3. Conventional tests for control and latex-modified asphalt binders (a) Softening point; (b) Penetration

B. *Storage Stability and Phase separation*

Storage stability was evaluated to confirm that, the effectiveness of additives used was not changed through modification processes. Softening point temperatures for the top, as well as bottom sections of base and latex-modified binder samples and their differences, are presented in Fig. 4. It can be observed that all latex modified binders have top and bottom sections difference of less than 4°C, when the difference between the top and bottom section of the samples treated for 72hours at 163°C is less than 4°C it indicates that the latex concentrations in the modified binder samples will be stable under high temperature storage. However, the control sample (unmodified) shows the lowest temperature difference of 0.2°C, and as the latex content is increased the temperature difference is also increase, based on the differences obtained it indicates that latex-modified binder will not exhibit phase separation during storage at high temperatures.

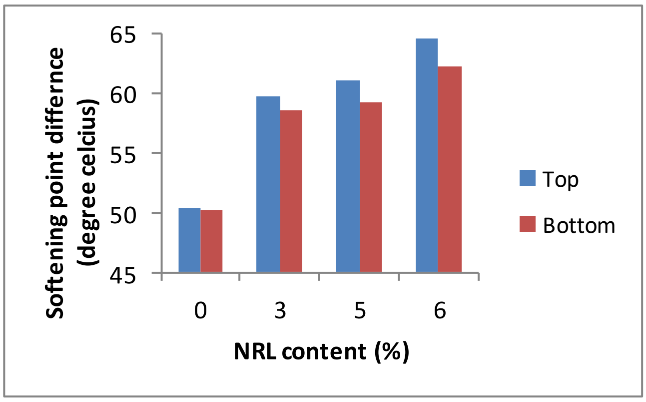


Fig. 4. Softening point gap for control and latex-modified asphalt binders

C. *Temperature Susceptibility*

The susceptibility to temperature effect is the change in rheological properties of binders with a change in temperature. The PI value generally ranges from -3 (highly temperature susceptibility bitumen) to +7 (highly blown low temperature susceptibility) [24, 26]. Fig. 5 shows the penetration indices of control and latex-modified binders, it can be observed that the latex-modified binders have higher PI values compared to control; this indicates that latex-modified binders are less susceptible to temperature compared to control. An increase in PI values up to 3% NRL content was observed after aging this shows that oxidative aging will not have an influence on latex-modified binders. This result confirms that NRL has a positive effect on enhancing temperature susceptibility of latex-modified binders.

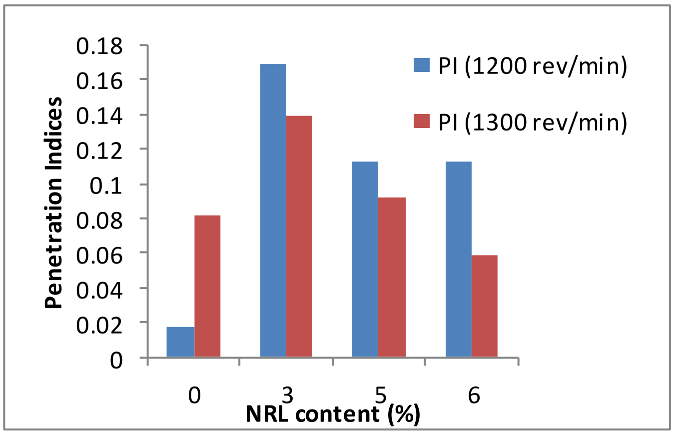


Fig. 5. Penetration indices for control and latex-modified asphalt binders

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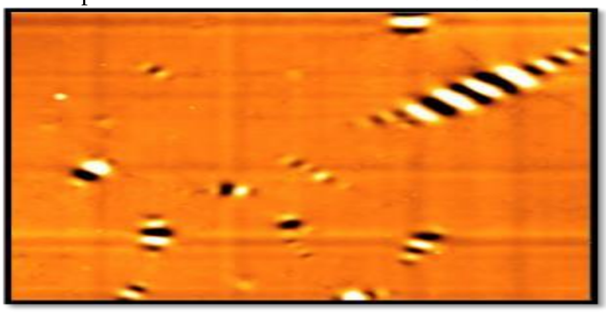
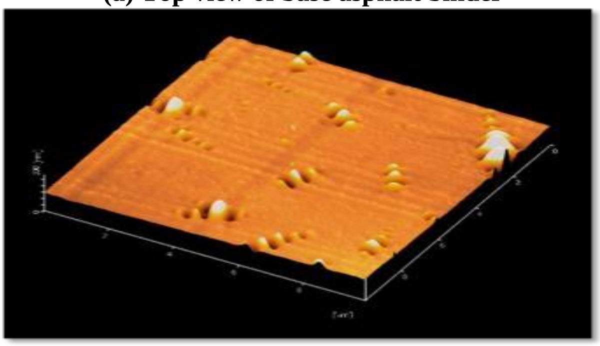
D. *Atomic Force Microscopy*

Sample sizes of (10x10) μm have been assessed in this study. The darker portion corresponds to the valleys and low portions of the surface profile in the topographic images shown in Fig. 6(a-h), whereas the lighter portions corresponds to peaks. Through examining the top view of the prepared samples, it reveals that the two dimensional images of the unmodified asphalt binder depicts a "bee" shape which is present only for the "GEL" bitumen, which was considered to be asphalt micelles. The AFM roughness is referred to as the mean height difference over a specific section of the chosen sample between the highest peaks and the lowest valleys relative to the centerline. The surface roughness of the latex-modified binder was decreased with increasing NRL contents.

AFM images shown in Fig. 6(a-h) indicated that bitumen is a multi-phase material with three identified phases present. These are the cata phase, the peri phase and the para phase. The catana phase displayed by the bee-shaped structures with a succession of alternating dark and light stripes, the peri phase are surrounding the catana domains and the para phase are in contact with the peri phase but not the catana domains. The AFM images only support the colloidal model standard of the bitumen structural characterization. For asphalt bitumen enriched with aromatics (which were originally reported to include the peri and catana phases), with improves frequency of the phases. Nonetheless, when the asphalt binder is enriched with resins, the total size of the "bee" structure (catana phase) stays identical for the three NRL content employed in this study to modify the base asphalt binder. The peri phase increased shows that the homogeneity of the latex-modified asphalt binder is good and the latex dissolved perfectly during the blending process while the catana phase increased after NRL addition was ascribed to the aging when the samples were heated for 2hours at 180°C.

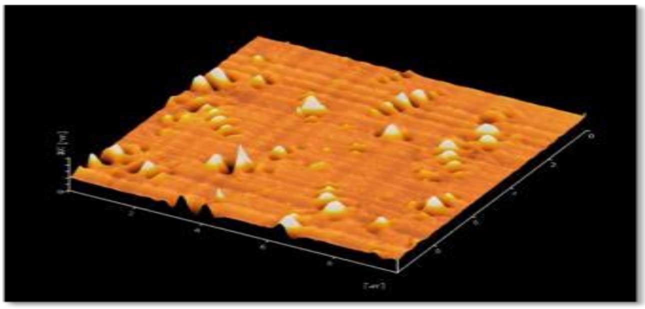


(a) Top view of base asphalt binder

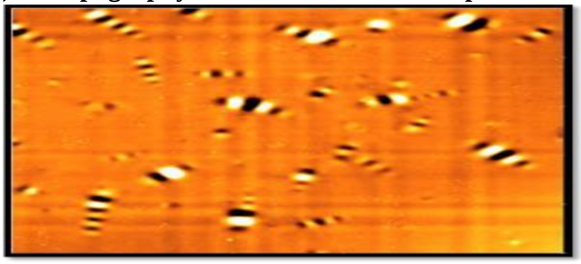


(b) 3D topography of base asphalt binder

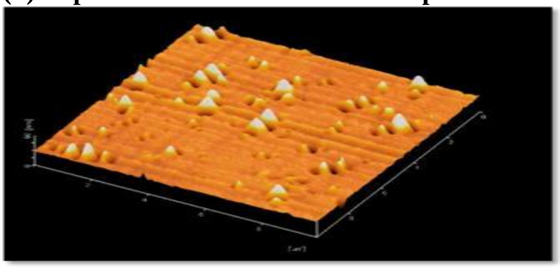
Fig. 6. Atomic Microscopy Scans



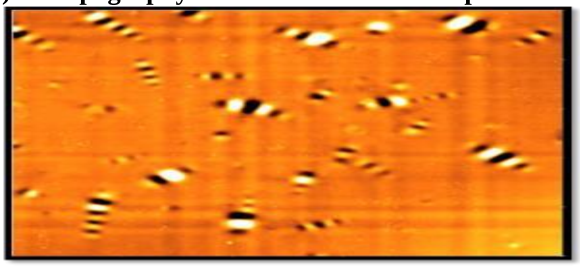
(c) 3D topography of 3% NRL modified asphalt binder



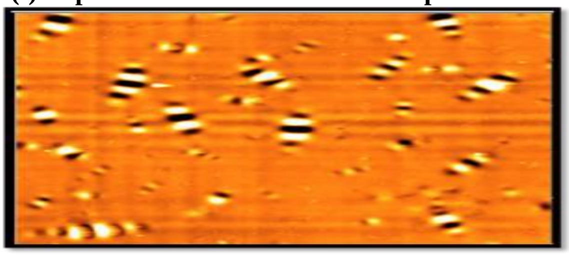
(d) Top view of 3% NRL modified asphalt binder



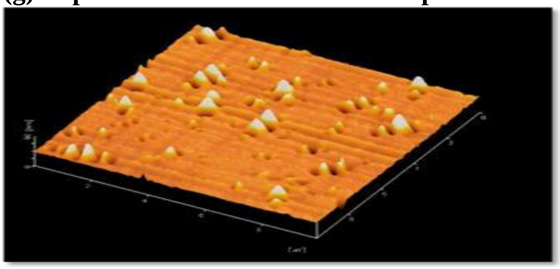
(e) 3D topography of 5% NRL modified asphalt binder



(f) Top view of 5% NRL modified asphalt binder



(g) Top view of 6% NRL modified asphalt binder



(h) 3D topography of 6% NRL modified asphalt binder

IV. CONCLUSIONS

In this research, the effect of different amounts of NRL at different mixing speeds on the basic properties, storage stability, and morphology of asphalt binders were evaluated. Depending on the outcomes of the physical properties (penetration and softening point), the incorporation of NRL, irrespective of the mixing speed used, stiffened the binder due to NRL elasticity. The improvement in elasticity showed improved performance at low temperatures. Also, the latex-modified binders with a mixing speed of 1200 rev/min showed superior improvement than both the base asphalt binder and latex-modified binders with 1300 rev/min mixing speed.

The latex-modified binder storage stability test indicates that all NRL contents used in this study to modify the base binder will be stable under high-temperature storage conditions due to NRL ability to improve binder cohesion. AFM analysis reveals a good dispersion due of NRL in the modified binder, indicating that NRL can greatly enhance bitumen-NRL compatibility.

Based on the results obtained in the course of this study, it is recommended to utilize liquid natural rubber latex for bitumen modification for an enhanced elasticity, penetration, softening point temperature, temperature susceptibility and compatibility.

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