CHARACTERIZATION OF THE PERFORMANCE OF ALUMINUM OXIDE NANOPARTICLES MODIFIED ASPHALT BINDER

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Graphical abstract

This study investigates the physical and rheological properties of asphalt binders modified by nano aluminum oxide (Al₂O₃). Several conventional tests were conducted, including penetration, softening point and ductility, rotational viscosity and dynamic shear rheometer (DSR). Based on the results of the tests, it was found that the hardness of modified asphalt binders increased with the addition of nano Al₂O₃ up to 5%. As a result of the increased hardness, the softening point of modified asphalt improved compared with base asphalt binders. The rheological property of modified binders was enhanced at low and high temperatures. The results of a DSR test revealed that the G* were improved, whereas the δ decreased slightly. The addition of a different percentage of Al₂O₃ to base binder had a remarkable influence on resistance to permanent deformation (high temperature rutting and low temperature fatigue). Results recognize 5 wt.% as the optimum content of the modifier. Therefore, nano Al₂O₃ can be considered as a proper alternative additive to modify the properties of asphalt cement.

Keywords: Nano aluminum oxide, physical and rheological properties, conventional tests, modified asphalt binder, dynamic shear rheometer and rutting and fatigue parameters.

1.0 INTRODUCTION

Asphalt paving roads show limitation based on temperature. They become cracked when the temperature is low, and soften when the temperature is high. Besides, high loading weight and hard traffic will destroy the roads earlier than predicted, which are expensive to repair and maintain [1-3]. Asphalt cement has the characteristics of low-temperature cracking and high temperature rutting. Moreover, fatigue, rutting and aging are some of the distresses of asphalt cement, which makes its manufacturing application limited. Moreover, asphalt roads undergo important distresses not only under temperature, but also with increasing heavy traffic loads [4-6]. Therefore, the modification of asphalt cement is essential to improve its properties, using methods and materials which can improve it (such as nanotechnology) in order to achieve the optimal asphalt performance. Nanotechnology has been discovered and used to address the problems in the design and construction of functional structures with at least one characteristic dimension measured in
nanometers [7]. Nanotechnology has recently become one of the interested areas in research and development worldwide, and has attracted numerous researchers in varied applications and domains. In general, the benefits of using nanotechnology to modify cement and asphalt mixtures include: improving the storage stability of polymer modified asphalt cement (PMAC), reducing the moisture susceptibility, and enhancing the properties of asphalt at low and high temperatures (resistant to the fatigue and rutting), improving the durability of the asphalt mixture, decreasing maintenance costs and saving energy [7-9].

In asphalt application, a limited number of studies have been carried out on nanotechnology modified asphalt cement, as well as on nanoclay, nanosilica and carbon nanotube [10-11]. In the study presented in this paper, the characterization of the performance of aluminum oxide nanoparticles (Al₂O₃) modified asphalt cement was evaluated using conventional and Dynamic Shear Rheometer (DSR) tests.

### 2.0 EXPERIMENTAL METHODS

#### 2.1 Materials

Asphalt cement 60/70 penetration grade bituminous was produced and supplied from a factory in Port Klang, Malaysia. The physical and chemical compositions of the asphalt cement are listed in Table 1. An aluminum oxide nanoparticle (Al₂O₃) was used in this study as a modifier of base asphalt cement, Al₂O₃, supplied by the Shijiazhuang Chanchiang Corporation in China. Its main properties are listed in Table 2.

#### 2.2 Preparation of Modified Asphalt Cements:

The modified asphalt cement was prepared at a temperature of 170 °C. The modifier quantities (3%, 5% and 7 wt. %) of Al₂O₃ were added into the base asphalt binder. The binders were mixed at a speed of 5000 rpm for about 90 min using a high shear mixer, in order to acquire the better dispersion of Al₂O₃ nanoparticles in the base binder.

| Table 1 The physical and chemical compositions of the base asphalt binder |
|-----------------------------|-----------------------------|
| Material                  | Properties                  | Value |
| Asphalt Cement 60/70      | Specific Gravity            | 1.03  |
|                           | Penetration @ 25 °C 0.1 mm  | 70    |
|                           | Softening point °C Pa.s     | 47.0  |
|                           | Viscosity @ 135 °C          | 0.5   |
|                           | Ductility (cm) @ 25 °C Cm   | ≥100  |
|                           | Asphaltenes (%)             | 12.2  |
|                           | Resins (%)                  | 30.8  |
|                           | Saturates (%)               | 8.1   |
|                           | Aromatic (%)                | 48.9  |

| Table 2 The physical and chemical compositions of the Al₂O₃ nanoparticles |
|-----------------------------|-----------------------------|
| Material                      | Properties                  | Value |
| Al₂O₃ Nanoparticle            | Formula                     | Al₂O₃, gamma |
|                              | Molecular Weight            | 101.96 |
|                              | Color                       | Milky white |
|                              | Form                        | Nanopowder |
|                              | Purity                      | > 99%   |
|                              | Average Nanoparticle Size   | 13-20 NM |
|                              | Specific Surface Area       | 100-200 m²/g |
|                              | Crystallographic Structure  | Cubic   |

The physical properties of the asphalt cement, including penetration, softening point, ductility at 25 °C and viscosity were tested according to ASTM D5, ASTM D 36, ASTM D 113 and ASTM D4402 respectively. The tests were performed three times, and the average values of the results were recorded. A DSR was applied for dynamic rheological measurements. A 25 mm diameter plate with 1 mm thickness and an 8mm diameter plate with 2 mm thickness were used for all binders, according to AASHTO TP5. A frequency sweeps test was conducted from 10 to 75 °C, at a fixed applied frequency of 1.159 Hz (10 rad/s). Figure 1 shows the flow chart of the experimental work of the study.
3.0 RESULTS AND DISCUSSION

3.1 The Physical Properties Test

The influences of adding AL₂O₃ on physical properties of nano AL₂O₃ modified asphalt binders samples were instigated. Figure 2 represents the relationship between the penetration values and the nano AL₂O₃ concentration. It was observed that there was a decrease in penetration values with an increasing AL₂O₃ concentration. This dramatic decreased was as a result of the base asphalt cement becoming stiffer with an increase in the concentration of the modifier AL₂O₃. Furthermore, it was noted based on Figure 3 that the addition of nano AL₂O₃ leads to enhance the softening point of the modified asphalt binders. This is due to the solid phase of nano AL₂O₃, which increases the Hardness of modified asphalt cement. In addition, it was recognized that 5% wt of the modifier was the best performance of penetration and softening point values.

The viscosity of the base binder becomes greater with the addition of nano AL₂O₃ at test temperatures (135, 155 and 165°C). However, the modified asphalt cement samples have shown a significant improvement compared with the base asphalt binder, as shown in figure 4. The increase in viscosity is a result of the hardening effect of nano AL₂O₃. The increased viscosity of the modified asphalt binders might be due to the better dispersion of the nano AL₂O₃ layers in the base binder, which leads to an increase in the bonding strength through restricting the flow of asphalt. This makes it harder and leads to enhance the asphalt’s physical properties.
3.2 Rheological Properties

The complex modulus ($G^*$) alongside temperature variations for the base asphalt binder and nano AL$_2$O$_3$ modified asphalt binders is represented in Figure 5. It is observed that there was an increase in $G^*$ with the addition of modifier to asphalt up to 5% of the base asphalt. Based on Figure 5, the modified asphalt with 5% of AL$_2$O$_3$ has a higher complex modulus among the binders, which means it has greater resistance to permanent deformation, while the base binder has the lowest $G^*$ value.

![Figure 5](image1.png)

Figure 5 The complex modulus ($G^*$) against temperature

The phase angle ($\delta$) against temperature variations for all asphalt binders are shown in Figure 5. The phase variance amongst stress and strain in an oscillatory test is defined as a phase angle. A smaller $\delta$ has a more elastic behaviour, while a greater $\delta$ value shows a more viscous behaviour. The $\delta$ of the modified asphalt binders decreases compared with the base binder. Hence, based on Figure 6, it is noted that the elastic behaviour of modified asphalt increases with the addition of nano AL$_2$O$_3$. Besides, the 5% of nano AL$_2$O$_3$ modified asphalt has the lowest $\delta$ among the modified asphalt binders, which means that it has a more elastic behaviour.

![Figure 6](image2.png)

Figure 6 The phase angle ($\delta$) against temperature

The resistance of the asphalt binder against rutting and fatigue cracking (permanent deformation) was measured by DSR tests. The DSR tests were conducted on asphalt binders for various temperatures, namely 45-75 °C with increments of 10°C, in order to measure the rutting resistance. Similarly, fatigue-cracking tests were also conducted at various temperatures of 10, 15, 25, and 35 °C. The rutting and fatigue-cracking tests utilized the constant loading frequency of 1.59 Hz (10 rad/s), as identified by the strategic highway research program (SHRP).

The value of $G^*/\sin \delta$ is regularly used to define the rutting resistance of the asphalt binder at high temperatures. The Superpave technique requires $G^*/\sin \delta = 1$ kPa as the minimum rutting parameter for an unaged sample [12-13]. Figure 7 shows that the lowest value of $G^*/\sin \delta$ was obtained by the unmodified asphalt binder, and that the 5% nano AL$_2$O$_3$ has the highest value of $G^*/\sin \delta$. A greater value of $G^*/\sin \delta$ indicates a pavement with better permanent deformation resistance [14-15]. However, the 7% sample shows a different behaviour as the value of $G^*/\sin \delta$ slightly decreased. Overall, it can observed that all asphalt binders have $G^*/\sin \delta$ values larger than 1.0 kPa at 65 °C.

![Figure 7](image3.png)

Figure 7 Effect of temperature on the rutting parameter

On the other hand, $G^*, \sin \delta$ is regularly used to define the fatigue resistance of the asphalt binder at a low temperature. The Superpave method requires a limitation at 5000 kPa for the maximum fatigue-cracking parameter [4]. Figure 8 shows that all asphalt
binders, including the base asphalt binder, have fatigue-cracking parameters greater than the required Superpave value at all test temperatures ranging from 10 to 35 °C. Moreover, it is observed that the decrease in the value of $G^* \sin \delta$ by increasing the nano content up to 5% of Al$_2$O$_3$ at 25 °C.

![Figure 8 Effect of temperature on the fatigue parameter](image_url)

### 4.0 CONCLUSION

Analysis of data obtained from the testing program yields the following outcomes:

- Conventional test results have shown that the addition of Al$_2$O$_3$ improved the stiffness of the modified binders, which declined their temperature susceptibility. However, the improvement was quite obvious at 5% of Al$_2$O$_3$.
- The softening point of modified binders improved compared with unmodified asphalt binder (base asphalt).
- The frequency sweep tests demonstrate that all modified binders had an increased complex modulus value and a decreased phase angle compared with the base asphalt binder, which leads to enhanced elastic behavior of asphalt.
- The rutting and fatigue parameters demonstrated that the Al$_2$O$_3$ nanoparticles able to increase the resistance high temperature rutting and improve intermediate temperature fatigue.

Overall, the study of characterization of nano Al$_2$O$_3$ demonstrates that modification using nano Al$_2$O$_3$ is able to improve the physical and rheological properties of asphalt binder significantly. Moreover, the best performance of modified asphalt binders was 5% of the modifier.

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### References


