**Rheological Properties of Bitumen Containing Organic Foaming Agent for Warm Mix Asphalt**

**Abdul Muhaimin Memon**, Nur Hazwani, Muslich Hartadi Sutanto, and Nura Bala

1 Department of Civil & Environmental Engineering, Universiti Teknologi Petronas, 32610, Perak, MALAYSIA
2 Department of Civil Engineering, Bayero University, Kano, NIGERIA
*Corresponding author: abdul_18002747@utp.edu.my

**Abstract.** Pavement industry is placing more emphasis on incorporating the sustainability in road construction. The construction of road contributes to global warming in terms of greenhouse gas emissions. Generally, the construction of roads adopt hot mix asphalt (HMA) that requires high energy consumption and also lead to environmental pollution. Since last decade, warm mix asphalt (WMA) technology is an attempt to address the aforementioned energy and environmental problems by reducing the harmful emissions and energy consumption. This study investigates the effect of organic foaming agent zeolite on the rheological properties, mixing temperature and compaction temperature of bitumen used in WMA. Dynamic shear rheometer (DSR) characterization is performed to determine the effect of zeolite on complex modulus ($G^*$), phase angle ($\delta$) and Superpave rutting parameter ($G^*/\sin\delta$) of bitumen. In addition to that, viscosity characterization using Brookfield rotational viscometer is conducted to determine the mixing and compaction temperature. Rheological characterization of binder containing zeolite shows the improvement of elastic segment and short-term ageing resistance. Viscosity characterization reveals that the addition of zeolite in bitumen offers significant reduction in mixing and compaction temperature. Based on the findings of this study, 3% zeolite can be added to WMA technology as optimum additive content in order to reduce the energy consumption and improve the workability of road construction thereby promoting sustainability in pavement industry.

1. **Introduction**

In modern era, one of the primary targets of sustainable development is the friendly environment. The friendly environmental based sustainable development aims to reduce or minimize harm upon ecosystem or environment. The construction of roads contributes to emission of aerosols and fumes causing global warming. The construction of road normally uses HMA for mixing, laying and compaction at a temperature range of 140 - 160°C. This high temperature provides better workability for HMA, however, HMA demands more energy consumption and produces significant amount of greenhouse gases that can adversely affect the environment [1]. There are a number of technologies in pavement industry that have been developed to overcome the aforementioned consequences. WMA is one of the recently developed technology as an attempt to reduce harmful compound emissions and energy consumption by lowering the mixing and compaction temperature of asphalt mix [2].

The use of organic additive, chemical additives and water foaming additives to reduce the temperature of mixing and compaction of WMA is a result of recently developed technology [3]. The widespread benefits of this technology are to reduce harmful gases emission for better working
environment, reducing energy consumption in asphalt mixing and production, and providing longer distance transportation of the asphalt mix [4]. Foaming asphalt technology is a type of WMA technology that requires the addition of small amount of water to decrease the viscosity of bitumen at lower temperature. When the hot bitumen is mixed with water, it evaporates the entrapped steam that causes the mixture to become less viscous. In fact, when the water is added, a considerable amount of foam is generated which retards the viscosity by extending the binder volume. Moreover, the reduction in the viscosity to some extent is desirable to ensure the proper coating of aggregates and thus the workability of asphalt mix [1]. In order to produce the foaming effect, the quantity of water should be just enough so that it does not leads to stripping of aggregates. Adding of organic mineral into bitumen will produce all these effects, therefore, necessary precautions should be taken while adding this material into the bitumen [5].

The organic additive (zeolite) is used to reduce the temperature of WMA. Zeolite releases water when it is heated. In addition to that, the continuous release of water is observed from the zeolite structure without affecting its crystal volume. Because of the presence of water particles in their composition it is also termed as “zeolite water” [6]. Foaming asphalt technology uses zeolite to produce foaming effect in WMA. The heating of zeolite for 6 to 7 hours releases approximately 20% water of crystallization from its structure and results into micro foaming effects in the asphalt mix. Their nature to lose and absorb water without damaging its structure is the primary characteristic that emphasis on their use in foaming asphalt technology [2]. Generally, the viscosity of bitumen in HMA becomes higher at lower temperature such that it becomes difficult to mix, lay and compact. On the contrary, the mixing and compaction temperature used in WMA technology is lower than HMA without affecting its workability to mix, lay and compact. The addition of foaming agent like zeolite will decrease the viscosity of bitumen and thus improves its workability [5]. After adding zeolite to the bitumen, bitumen viscosity should be determined for mixing and compaction temperatures in addition to the rheological properties required for WMA technology. The rheological study offers the characteristics of bitumen that can indicate the performance of binder up to several years [7]. Hence, it is necessary to study viscosity and rheological properties of warm mix asphalt foamed bitumen.

The purpose of this study is to examine the effects of zeolite on bitumen using WMA technology. This study investigates the viscosity and rheological behaviour of virgin and modified bitumen at concentration of 3, 5, and 7% by mass of bitumen. These percentages are selected based on the past research [4]. The prepared samples were subjected to viscosity and rheological characterization. These laboratory evaluations were conducted to demonstrate the effects of zeolite on WMA.

2. Material and methods
The proper selection of material is necessary as it plays a vital part in the precision of results. The selection of materials is based on the availability and applied standards of the region. The preparation of samples was conducted using 80/100 penetration grade bitumen and zeolite (organic additive). The zeolite is a hydrothermally crystallized aluminosilicate mineral group of alkali metals. The preparation of samples was performed by adding 3, 5, and 7% of organic additive zeolite in bitumen as suggested in the past studies [4]. The prepared samples were subjected to viscosity and rheological characterization. These laboratory evaluations were conducted to demonstrate the effects of zeolite on WMA.

2.1. Viscosity characterization
The workability of bitumen required for mixing and compaction of asphalt mix is determined using Brookfield rotational viscometer. The viscosity measurement of virgin and modified binders was performed in accordance with ASTM D4402-06 [8]. This test uses a 30 g of binder specimen which is heated in an oven to become fluid enough to pour. This specimen is poured into the sample chamber and placed in a thermo container for 15 minutes to stabilize the temperature. When the temperature is stabilized at 135°C, the sample is tested. The WMA mixing and compaction temperatures are determined from the viscosity data using a log-log viscosity vs. temperature graph. Viscosity test result illustrates the effect of zeolite on mixing and compaction temperature of WMA.
2.2. Rheological characterization

The rheological characterization is carried out in accordance with AASHTO T-315 to determine the behaviour of bitumen at varying temperatures and loading times (frequency) [9]. Superpave binder system employs DSR to evaluate complex modulus (G*) and phase angle (δ) of bitumen samples to represent their viscoelastic characteristics. These viscoelastic characteristics of bitumen demonstrate the resistance to rutting deformation and fatigue cracking. In this study, the temperature for isochronal plots lies in the range of 20°C, 30°C, 40°C and 50°C with constant frequency of 10 rad/s. Besides, the effect of zeolite on rheological properties of bitumen are observed from the data imported from DSR.

3. Results and discussions

This section provides the results obtained from the aforementioned lab evaluations. The data analysis is divided into two parts; determining the bitumen viscosity to plot the mixing and compaction temperature of WMA and evaluating the DSR data to observe the effects of adding zeolite on rheological properties of bitumen.

3.1. Viscosity test results

The data obtained from the viscosity test was used to plot log-log viscosity vs temperature graph for determining the WMA mixing and compaction temperature as shown in Figure 1. The mixing and compaction temperatures are determined using temperature thresholds specified by Superpave. The mixing and compaction temperature thresholds lies in the viscosity range of 0.17 ± 0.02 Pa. s and 0.28 ± 0.03 Pa. s respectively.

Table 1 shows the data that was obtained from Brookfield rotational viscometer in accordance with ASTM D4402-06. Based on Table 1, bitumen containing 3% zeolite gives the lowest viscosity value among all the binders at temperatures of 120°C. In other words, a significant reduction of approximately 49% in the viscosity of bitumen is observed by the addition of 3% of zeolite at 120°C. Although this effect diminishes with increasing temperature. For instance, a 36% reduction in viscosity of bitumen is provided by the addition of 7% zeolite at 135°C. This value is almost identical to that of bitumen containing 3% zeolite. Finally, at 165°C the effect of zeolite is significantly reduced and the viscosity values are almost identical to that of control bitumen. This behaviour corresponds to the finding of previous studies [4]. Thus, these results ensure the lowering of mixing and compaction temperature by the addition of zeolite at low temperature. It indicates that using WMA for road construction would significantly reduce the energy consumption, providing better workability. In addition to this, all binders have viscosity value below 3 Pa. s as specified in Superpave binder protocols. This shows that organic additive zeolite improves the workability and depresses the mixing and compaction temperatures of WMA.

The mixing and compaction temperatures for each dosage of zeolite were determined as shown in Table 2. The lowest mixing and compaction temperatures of 130°C and 120°C for WMA are provided by bitumen containing 3% zeolite respectively. These values are 10°C and 12°C lower than the base bitumen. This trend is also observed in previous research [4]. The addition of zeolite reduces the mixing and compaction temperature, however, this effect minimizes after adding more than 3% zeolite content. This reduction in viscosity could be due to the temperature resistance property of material as reported in the literature [10]. It is concluded from the results that the addition of organic additive (zeolite) reduces the excessive viscosity of bitumen. Therefore, based on the viscosity data, 3% of zeolite content is proposed as an optimum zeolite content.
Figure 1. Rotational viscosity plots for virgin bitumen and bitumen containing zeolite.

Table 1. Viscosity test results

| Temperature (°C) | Viscosity (Pa.s) |
|-----------------|-----------------|
|                 | Control binder  | 3% zeolite | 5% zeolite | 7% zeolite |
| 120             | 0.59            | 0.29       | 0.37       | 0.42       |
| 135             | 0.19            | 0.13       | 0.15       | 0.12       |
| 165             | 0.08            | 0.09       | 0.10       | 0.08       |

Table 2. Mixing and compaction temperatures of WMA at different dosage of zeolite

| Type of Sample/ Properties | Mixing Temperature (°C) | Compaction Temperature (°C) |
|----------------------------|-------------------------|-----------------------------|
| Control Binder             | 140                     | 132                         |
| Bitumen + 3% Zeolite       | 130                     | 120                         |
| Bitumen + 5% Zeolite       | 138                     | 126                         |
| Bitumen + 7% Zeolite       | 137                     | 126                         |

3.2. Rheological test results
The rheological properties of virgin bitumen and bitumen containing 3, 5 and 7% of organic additives zeolite were evaluated using KINEXUS Pro+ rheometer. The temperature sweep for isochronal plot was performed on samples at four different temperature points i.e. 20°C, 30°C, 40°C and 50°C using constant frequency of 10 rad/s. This temperature was selected based on the available size of DSR parallel plates. Figure 2 shows the effects of adding zeolite on the complex modulus (G*) of bitumen.
Figure 2. Complex Shear Modulus (G*) against Temperature

Figure 2 shows that as the concentration of zeolite in bitumen is increased, an improvement in the complex modulus (G*) is observed compared to base bitumen. The complex modulus (G*) of the samples containing zeolite are greater (around 6%) than that of base bitumen. The increase in complex modulus (G*) demonstrates a higher resistance to plastic deformation at high temperature. In addition to that, the increment of complex modulus (G*) reflects a higher elastic segment thereby improving elastic behaviour of bitumen [4]. The complex modulus (G*) of zeolite binders at the temperature of 50 °C are slightly greater to that of control bitumen. This trend is also supported by previous studies [4].

Phase angle (δ) is the time lag between the applied shear stress and the resultant shear strain. Figure 3 shows the phase angle of virgin bitumen and bitumen with 3, 5 and 7% organic additives (zeolite) at four different temperature points, namely 20°C, 30°C, 40°C and 50°C. It is observed from the results that the phase angle (δ) varies from 55.20° to 79.67° for the given percentage of zeolite at various temperatures. The test result shows that the addition of 3% zeolite content produce a 5% increase in the phase angle at low temperature. This increased value of the phase angle after the addition of zeolite demonstrates the improved flexibility of the binder at low temperature. However, this effect depresses at high temperature and the values become identical. This could be due to the properties of material that improves the flexibility of bitumen at low temperature [1].

Figure 3. Phase angle versus temperature
The complex modulus ($G^*$) and phase angle ($\delta$) are used as predictors for resistance to rutting deformation and fatigue cracking. Early in the pavement life rutting is the main concern, while later in the pavement life fatigue cracking becomes the major concern [9-11]. According to Superpave specifications, the rutting parameter ($G^*/\sin\delta$) should be greater than 1 kPa for adequate stiffness of material to cope with the rutting deformation [12]. The rutting parameter values obtained from the test are in the range of 15.92 to 2860.05 kPa at the given temperature range. The 3% addition of zeolite content improves the rutting parameter of bitumen at 20°C as shown in Figure 4. It is observed from Figure 4 that zeolite modified bitumen has a slightly improved stiffness compared to control bitumen. This trend diminishes with the increasing temperature, however, the rutting parameter values of zeolite modified binders are even more than that of control bitumen. The increase in rutting parameter reduces the susceptibility to short-term ageing of bitumen caused during mixing of asphalt mix [7]. Literature also supports this fact that the production of WMA mix at lower temperature improves the rutting resistance [13].

Figure 4. Rutting parameter ($G^*/\sin\delta$) against temperature

Overall, the rutting parameter for the modified bitumen was more than the threshold value required in Superpave specifications. Based on the rheological findings, 3% zeolite is suggested as the optimum zeolite content.

4. Conclusion
The effect of adding organic additives (zeolite) into bituminous binder has been investigated and results show that the viscosity of bitumen significantly decline with the addition of zeolite. In other words, there is a remarkable reduction of 10°C and 12°C in mixing and compaction temperatures with the addition of zeolite respectively. Hence, this study shows that adding of organic additive zeolite in bitumen improves the workability of mix and reduces the energy consumption. The addition of zeolite slightly enhances the complex modulus of bitumen which in turns provides elasticity in bitumen at high temperature. The increase in rutting parameter after the addition of zeolite indicates the improvement in short-term ageing of bitumen. Furthermore, the addition of zeolite contents meets the rutting parameters as specified by Superpave binder system. Based on the above findings it can be stated that the 3% zeolite addition will be the optimum content that can be added to WMA to improve the performance aspects of pavement. Future studies are recommended on the morphological analysis of bitumen containing zeolite to demonstrate the mechanism behind its improved performance.
5. References

[1] Abdullah M E, Zamhari K A, Buhari R, Khatijah S, Bakar A, Hidayah N and Hassan S A 2014 Warm mix asphalt technology: a review Jurnal Teknologi 3 39-52

[2] Hasan M R M, You Z and Yang X 2017 A comprehensive review of theory, development, and implementation of warm mix asphalt using foaming techniques Construction and Building Materials 152 115-133

[3] Woszuk A and Franus W 2017 A review of the application of zeolite materials in warm mix asphalt technologies Applied Sciences 7(3) 293 A

[4] Sengoz B, Topal A and Gorkem C 2013 Evaluation of natural zeolite as warm mix asphalt additive and its comparison with other warm mix additives Construction and Building Materials 43 242-252

[5] Woszuk A, Zofka A, Bandura L and Franus W 2017 Effect of zeolite properties on asphalt foaming Construction and Building Materials 139 247-255

[6] Bish, D L and Ming D W 2018 Natural zeolites: occurrence, properties, applications (Vol. 45) (Walter de Gruyter GmbH & Co KG) 22-35

[7] Yaacob H, Mughal M A, Jaya R P, Hainin M R, Jayanti D S and Wan C N C 2016 Rheological properties of styrene butadiene rubber modified bitumen binder Jurnal Teknologi 78 (32) 40-48

[8] ASTM D4402-06 2015 Standard test method for viscosity determination of asphalt at elevated temperatures using a rotational viscometer: American Society for Testing and Materials

[9] AASHTO T 315 2012 Standard method of test for determining the rheological properties of asphalt binder using a dynamic shear rheometer (DSR): American Association of state and highway transportation officials

[10] Yao H, You Z, Li L, Lee C H, Wingard D, Yap Y K and Goh S W 2012 Rheological properties and chemical bonding of asphalt modified with nanosilica Journal of Materials in Civil Engineering 25(11) 1619-1630

[11] Sadeq M, Masad E, Al-Khalid H, Sirin O and Little D 2016 Rheological evaluation of short-and long-term performance for warm mix asphalt (WMA) binders. 8th RILEM international symposium on testing and characterization of sustainable and innovative bituminous materials (Ancona) (Italy:Springer) (pp. 129-139)

[12] Kataware A V and Singh D 2017 Evaluating effectiveness of WMA additives for SBS modified binder based on viscosity, Superpave PG, rutting and fatigue performance Construction and building materials 146 336-444

[13] Hurley G C and Prowell B D 2006 Evaluation of potential processes for use in warm mix asphalt, Journal of the Association of Asphalt Paving Technologists 75 41-90

Acknowledgments

Authors would like to appreciate the support provided from Universiti Teknologi Petronas for successfully conducting of this research.