MJFAS MALAYSIAN JOURNAL OF FUNDAMENTAL AND APPLIED SCIENCES
PRINT ISSN: 2289-5981 | ONLINE ISSN: 2289-599X

RESEARCH ARTICLE

Physical properties of bitumen containing diatomite and waste engine oil

Norhidayah Abdul Hassan [a,*], Nur Azni Ruzi [a], Nurul Athma Mohd Shukry [a], Ramadhansyah Putra Jaya [a], Mohd Rosli Hainin [a], Nurul Hidayah Mohd Kamaruddin [b], Mohd Ezree Abdullah [b]

[a] *Department of Geotechnics and Transportation, School of Civil Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Malaysia*
[b] *Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia*

**Corresponding author: hnorhidayah@utm.my*

Article history
Received 5 May 2018
Revised 1 June 2018
Accepted 2 July 2018
Published Online 25 August 2019

Abstract

The addition of modifier, either to replace bitumen or as an additive, could potentially improve the performance of conventional bitumen used in road construction. This study characterizes the physical properties of bitumen 80/100 penetration grade modified with diatomite powder and waste engine oil (WEO). Different percentages of WEO i.e. 1%, 2%, and 3%, were added with 1% diatomite to the bitumen. The conventional and modified bitumen samples were tested for penetration, softening point, viscosity, and loss on heating. Results showed that the increase of WEO content, particularly at 3% in the modified bitumen, has softened the bitumen with lower softening point and higher loss on heating than the unmodified sample. In contrast, the diatomite powder has shown potential in reinforcing the bitumen structure at high temperature based on higher viscosity obtained at 165°C compared to conventional bitumen.

Keywords**:** Diatomite, WEO, bitumen, penetration, physical

INTRODUCTION

Currently, the worldwide demand for bitumen accounts for more than 100 million tons per year, which is approximately 700 million barrels of bitumen consumed annually. Bitumen is a noncrystalline viscous black or dark brown material, which is substantially soluble in carbon disulfide and possesses adhesive and waterproofing qualities. Bitumen consists essentially of hydrocarbons and typically comprises of at least 80% carbon and 15% hydrogen, with the remainder being oxygen, sulfur, nitrogen, and traces of various metals (Kolo *et al*., 2016). Although bitumen is a widely used material for constructing and maintaining roads in most countries around the world, Malaysia is now burdened with issues related to the ability of conventional bitumen to sustain under the present traffic loading and climate change phenomenon. In addition, the increase of worldwide demand for petroleum-based product as well as the cost of road construction has encouraged the road industry to find alternatives over the conventional binder (Newman *et al*., 2012). Therefore, bitumen modification could be an alternative in solving the aforementioned issues. Various materials were studied previously including polymers and waste materials. Diatomite or diatomaceous earth is oftenly used as polymer filler because of its lightweight, high void content, low density, large surface area, chemical inertness, good insulation property, and strong absorption (Degirmenci and Yilmaz, 2009; Jiang and Liu, 2014). Diatomite originates from siliceous sedimentary rock formed by the remains of diatoms deposited in oceans or lakes. The skeletons of diatoms are mainly composed of amorphous silica content, which is a durable substance (Ibrahim, 2012). The unique physical characteristics of diatomite make the material useful for civil engineering applications.

Cong *et al*. (2010) evaluated the effect of diatomite on the viscosity of bitumen and found that the increase in diatomite content increases the viscosity and resistance against deformation of the modified bitumen. In other study, Yi-qiu *et al*. (2012) concluded that the diatomite-modified asphalt mixtures performed better at low temperatures compared to the neat asphalt mixtures. Zhu *et al*. (2013) evaluated the insulation property and pavement performance of diatomite-modified asphalt mixtures in the laboratory, and the results showed that diatomite not only improves the rutting resistance significantly but also reduces the thermal conductivity coefficient of asphalt mixtures. Guo *et al*. (2015) investigated the performance of diatomite and glass-fiber-compound-modified asphalt mixtures. Their results revealed that diatomite and glass fiber improved the rutting resistance and fatigue performance of asphalt mixtures. On the other hand, Cheng *et al*. (2015) studied the influence of diatomite and mineral powder on the thermal oxidative aging properties of asphalt. The group determined that the anti-aging effect of diatomite was better than that of mineral powder because of its porous structures. Diatomite was also used as anti-stripping additive in porous asphalt and resulted in a resilient modulus greater than that of hydrated lime and cement (Shukry *et al*., 2016). Abdullah *et al.* (2016) investigated the potential of diatomite as an anti-clogging agent in porous asphalt. The scholars observed that the samples with diatomite achieved a higher permeability rate than those without diatomite after a few clogging cycles.

Waste engine oil (WEO) is a waste product that often contaminated by impurities, such as lead, zinc, calcium, and magnesium. These heavy metals accumulate gradually during the physical and/or chemical processes in the engine oil. According to Jia *et al*. (2014), automobile repair shops collect waste motor oil residues from different vehicles and dispose them collectively. These contaminants originate from the wear of engine components and from the heating and oxidation of lubricating oil during engine operation. Thus, the chemical composite of WEO may differ from other sources. However, as a petroleum-based product, WEO has a molecular structure similar to that of bitumen. From the investigations conducted by other researchers, the results of rheological properties indicated that the inclusion of WEO significantly softens the bitumen with lower softening point and higher penetration value compared to the conventional bitumen (Kamaruddin *et al*., 2014; Villanueve *et al*., 2008). Therefore, this study investigated the potential of diatomite and WEO as modifiers in the bitumen. This is significant as the diatomite acts as a reinforcing agent and complements the WEO portion in the bitumen that acts as a rejuvenator to improve the properties of conventional bitumen under various conditions, and thus promote waste recycling in the asphalt pavement.

MATERIALS AND METHODOLOGY

Bitumen

In this study, bitumen with 80/100 penetration grade was supplied by ASA Infratech (M) Sdn. Bhd, Johor, Malaysia. The properties of bitumen are given in Table 1.

Table 1 Properties of conventional bitumen.

Property	Unit	Results	Test Method
Penetration at 25℃, 100gm, 5sec	0.1mm	80/100	ASTM D5-06e1
Softening Point	°C	45 - 52	ASTM D36/ D36M-14e1
Viscosity at 135 °C	cP	400 - 500	ASTM D2170
Viscosity at 165 °C	cP	100 - 200	ASTM D2170
Loss on Heating,	%wt	0.5 max	ASTM D-6

The physical and chemical properties of diatomite and WEO are shown in Tables 2 and 3, respectively. Diatomite was supplied by I-Chem Solution Sdn. Bhd., Selangor, Malaysia whereas WEO was collected from several automotive repair shops in Skudai, Johor. Diatomite primarily consists of silica content with minor percentage of other chemicals. According to Cong *et al*. (2012), the high silica content and porous structure of diatomite play important roles in providing hardness to the material and other useful characteristics, such as high absorptive capacity, chemical stability, and light weight. On the other hand, Table 2 shows that WEO has a high level of metals, such as zinc, phosphorus, calcium, and magnesium. These contaminants originate from normal wear of engine components and also from heating and oxidation of lubricating oil during the engine operation (Dominguez–Rosado and Pichtel, 2003).

Modified bitumen preparation

Bitumen was heated and mixed with WEO in the mixer at the speed of 1,000 rpm, and the temperature was kept between 150°C and 160°C. The diatomite was then slowly added, with a constant temperature and speed of 3,000 rpm. The content of diatomite was fixed at 1%, whereas the WEO percentage was varied at 1%, 2%, 3% by weight of the mixture: 400 g. The total weight of the control sample produced was maintained at 400 g, and the percentages of WEO and diatomite were calculated on the basis of the total weight of the control sample and used to replace the bitumen portion. Table 4 shows the specified weight for each of the sample prepared in the laboratory. The mixing process was continued for 30 minutes in order to produce a homogeneous mix.

Fundamental tests, i.e. penetration, softening point, viscosity, and loss on heating (LOH), were conducted on the bitumen samples.

Table 2 Chemical and physical properties of diatomite.

Chemical composition (%)		Physical properties	
Si	31.9	Specific gravity	2.2
Al	1.4	Colour	White
Au	8.9	Form	Solid
C	17.4	pH	10
F	6.7	Loss of ignition	0.5
Mg	0.3		
Na	1.7		
O	31.8		

Table 3 Chemical and physical properties of waste engine oil.

Chemical composition (ppm)		Physical properties	
Cu	10	Viscosity (cSt), 60°C	181
Cr	1	Specific gravity	0.89
Fe	9	Flash point, °C	193
Mo	31	Loss on heating, % of weight	2.61
P	668	Ash content, %	2.02
Pb	1		
Zn	749		
Ca	476		
Mg	188		

Penetration test

Penetration test was performed in order to evaluate the bitumen consistency in accordance to ASTM D5-06e1. Bitumen was heated and poured into a penetration cup prior to testing. The sample was then cooled and conditioned in the water bath for 1 hour at 25°C. The bitumen sample was tested with the applied total load of 100 g for 5 s at 25°C.

Penetration index (PI)

PI represents a quantitative measure of the bitumen responses at various temperatures (Ehinola *et al*., 2012). The PI value can be calculated using Eq. 1 by using the penetration and softening point values. Mallick and El-Korchi (2009) defined that normal bitumen has the PI value between −1 and +1, where a high PI corresponds to low temperature susceptibility and a low PI corresponds to high sensitivity to temperature changes.

$$PI = \frac{20-500A}{1+50A};\ A = \frac{Log\ (PEN\ at\ 25℃) - Log\ (800)}{25 - Softening\ point\ temp.} \quad (1)$$

Table 4 Total weight of samples.

Descriptions	Percentages (%)		Weight (g)			Total weight (g)
	Diatomite	WEO	Bitumen	Diatomite	WEO	
Conventional	0	0	400	0	0	400
ID: 1WEO	1	1	392	4	4	400
1D: 2WEO	1	2	388	4	8	400
1D: 3WEO	1	3	384	4	12	400

Softening point test

The test was performed in accordance with ASTM D36. Bitumen was heated, poured into two rings, and cooled for 30 minutes. The two rings and two ball centering guides were placed on the ring holder in a liquid bath. Subsequently, 3.5 g of steel balls were placed on each sample and heated. The temperature at which bitumen came in contact with the base plate was obtained, and the mean temperature of the two samples was calculated.

Viscosity test

The viscosity test was used to determine the flow resistance and internal friction of bitumen according to ASTM D2170. The sample with low friction recorded a low viscosity reading in units of centipoise. The viscosity value was measured at two temperatures i.e. 135℃ and 165℃.

Penetration-viscosity number (PVN)

The PVN was calculated using Eq. 2 with the penetration value measured at a standard temperature of 25°C and viscosity value at 135°C. Mallick and El-Korchi (2009) reported that the typical value of PVN lays within the range of −2 to 0.5.

$$PVN = -1.5 \times \frac{4.258 - 0.7967 Log\ P - Log\ V}{0.795 - 0.1858 Log\ P} \qquad (2)$$

Loss on heating, LOH test

This test was used to measure the effect of heat on bitumen by determining the mass loss of bitumen after it was heated. The finding can be attributed to the effect of heat that induces bitumen's volatility and hardness. Approximately 50 g of bitumen sample was heated at 163°C for 5 hours in a specified oven. The sample was weighed again after the heating period, and the loss in mass was expressed as the percentage by weight of the original sample. The percentage of LOH for the bitumen used in pavement should not exceed 1%. However, for bitumen with penetration values of 150 to 200, up to 2% weight loss is allowed. The test procedures were conducted as described in ASTM D-6.

RESULTS AND DISCUSSION

Penetration

Fig. 1 shows the penetration value gradually increases with WEO content. The finding shows that the increase in WEO content significantly affects the penetration value. Initially, the penetration value decreases sharply from 80 PEN (conventional) to 58 PEN with 1% diatomite added. The sample without WEO was prepared for comparison. However, the penetration value increases gradually after WEO was mixed in the bitumen. The addition of WEO more than 2% has further softened the bitumen and 1% diatomite seems ineffective as reinforcing agent within the mix as it is overpowered by WEO liquefied component.

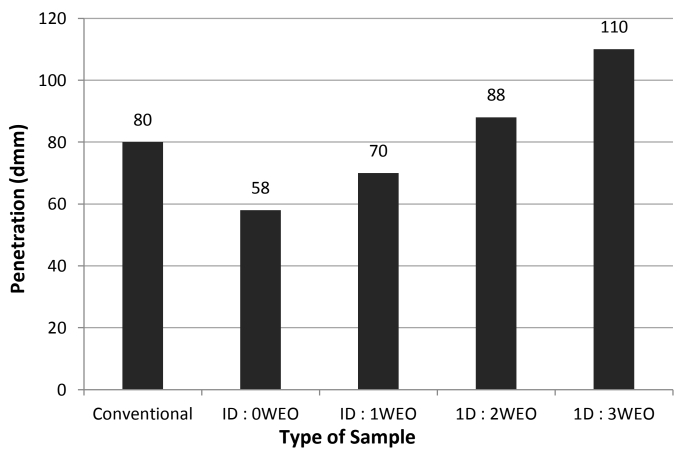

Fig. 1 Penetration test results.

In addition, at 1% WEO, both materials seem to complement each other within the mix, where WEO and diatomite exert opposite effects on bitumen. The hardness of bitumen is reduced with WEO addition and balanced with the addition of diatomite.

Softening point

Fig. 2 shows the softening point result for conventional 80/100 PEN and modified bitumen. The softening point value obtained for conventional bitumen is 40°C, which is quite low for typical value of standard 80/100 PEN bitumen. However, based on the comparison with the modified bitumen, generally, high WEO content in the bitumen has reduced the softening point from 40°C to 38°C. The finding reflects the penetration result, where the addition of WEO tends to soften the bitumen. However, with 1% diatomite in the mix, the bitumen sample with 1% WEO produced harder binder with high softening point, 41°C compared to conventional bitumen. In other words, 1% diatomite dominates the mix and stiffens the bitumen (with 1% WEO).

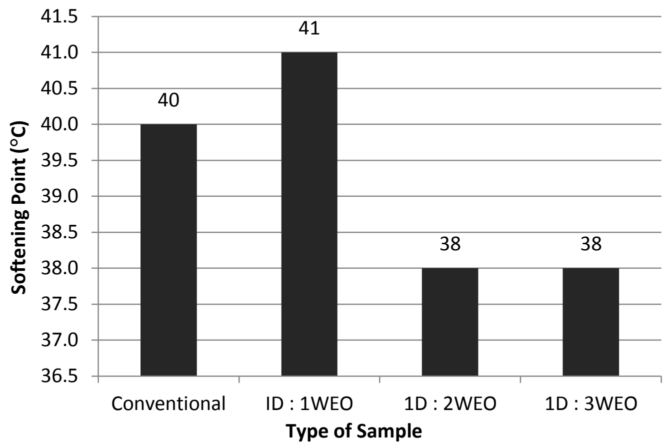

Fig. 2 Softening point results.

Viscosity

Fig. 3 shows the viscosity result of conventional and modified bitumen measured at 135°C and 165°C, respectively. Viscosity exhibits a different trend for both temperatures with WEO content. The modified bitumen with diatomite and WEO at 135°C achieves a lower viscosity than that of conventional bitumen grade 80/100 PEN. With the increase of WEO content, the viscosity value decreases. This trend indicates that the bitumen become decreasingly viscous. The figure shows that the viscosity of conventional bitumen is 500 cP at 135°C then decreases to 200 cP with 3% WEO. In contrast, at 165°C, the viscosity of conventional bitumen and 3% WEO is 100 cP, while the viscosity of the rest is slightly increases to 200 cP.

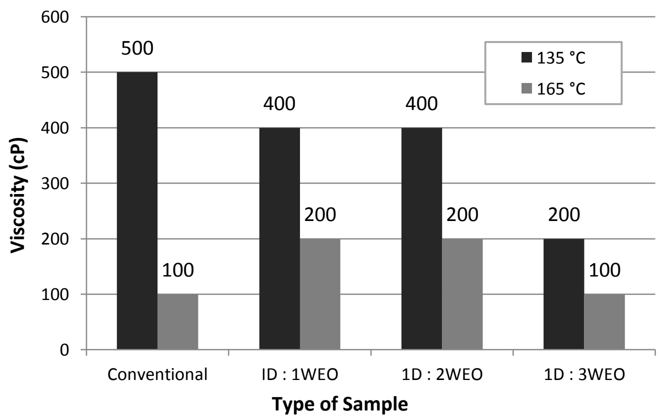

Fig. 3 Viscosity test results.

The finding indicates that at high temperature of 165°C, diatomite plays a more significant role in reinforcing the bitumen structure than that of 135°C, at which WEO significantly influences the reduction in viscosity. The porous structure or high surface area of diatomite powder could potentially encourage the particles to absorb light oil and increases the viscosity of the modified bitumen.

PI and PVN

The results of PI and PVN are shown in Table 5. The PI value decreases with WEO content. The result shows that the addition of WEO reduces the PI value and increases the temperature susceptibility relative to that of conventional bitumen. As reported by Ehinola *et al.* (2012), the PI value of approximately −3 indicates that the material is highly sensitive to temperature changes. In contrast, the value of PVN decreases as WEO increases. The finding suggests that the modified bitumen has slightly lower resistance against temperature susceptibility compared to the conventional bitumen. Normal bitumen was previously reported to possess PVN value between −2 and 0.5. The results confirm that all samples' PVNs lie within the typical range.

Table 5 PI and PVN results.

Descriptions	PI	PVN
Conventional	-3.07	-0.14
ID: 1WEO	-3.19	-0.61
1D: 2WEO	-3.59	-0.37
1D: 3WEO	-3.16	-1.20

Loss on heating

In general, bitumen used for pavement should not exhibit more than 0.5% loss in mass after heating. Table 6 shows the LOH value obtained for conventional and modified bitumen. Samples with 1% diatomite and 1% and 2%WEO exhibit the lowest reduction in mass of 0.2% compared to the conventional bitumen of 0.38%. This shows that 1% diatomite could potentially reduce the LOH of modified bitumen containing WEO. However, the replacement of bitumen with 3% WEO has caused the highest weight loss of 0.6% which exceeds the specification. This shows that at high WEO content, the oil dominates and determines the final properties of the bitumen sample.

Table 6 Loss on heating values.

Descriptions	Loss Properties		
	Weight Before (g)	Weight After (g)	% Loss
Conventional	52.0	51.8	0.38
1D:1WEO	49.8	49.7	0.20
1D:2WEO	50.1	50.0	0.20
1D:3WEO	50.1	49.8	0.60

CONCLUSION

From the study, the following conclusions can be drawn:

i. The results clearly illustrate that adding WEO at various contents significantly affect the temperature susceptibility of bitumen. As the WEO content increases from 1% to 3%, the penetration value increases gradually, whereas the softening point of the modified bitumen decreases.
ii. The addition of WEO decreases the viscosity of bitumen. At higher temperature of 165°C, diatomite plays a significant role in reinforcing the bitumen structure than those tested at temperature of 135°C, where WEO significantly influences the viscosity reduction.
iii. The LOH of conventional and modified bitumen is less than 0.5%, which complies with the specification except for the bitumen sample with 3% WEO.

ACKNOWLEDGEMENT

Universiti Teknologi Malaysia and Ministry of Higher Education, Malaysia are sincerely acknowledged for providing the research funds (Vote no. Q.J130000.2522.11H76 and R.J130000.7822.4F436) and laboratory facilities.

REFERENCES

Abdullah, N. A. M., Hassan, N. A., Shukry, N. A. M., Mahmud, M. Z. H., Putrajaya, R., Hainin, M. R., Yusoff, N. I. M. 2016. Evaluating potential of diatomite as anti-clogging agent for porous asphalt mixture. *Jurnal Teknologi (Sciences & Engineering)*, 78(7-2), 105-111.

ASTM D6/D6M-95(2011)e1, *Standard test method for loss on heating of oil and asphaltic compounds*, ASTM International, West Conshohocken.

ASTM D5-06e1, *Standard test method for penetration of bituminous material*, ASTM International, West Conshohocken.

ASTM D36/D36M-14e1, *Standard test method for softening point of bitumen (ring and ball apparatus)*, ASTM International, West Conshohocken.

ASTM D2170/D2170M-10, *Standard test method for kinematic viscosity of asphalts (bitumens)*, ASTM International, West Conshohocken.

Cheng, Y., Tao, J., Jiao, Y., Guo, Q., Li, C. 2015. Influence of diatomite and mineral powder on thermal oxidative aging properties of asphalt. *Advanced in Materials Science and Engineering*, 2015, 1-10.

Cong, P., Chen, S., Chen, H. 2012. Effects of diatomite on the properties of asphalt binder. *Construction and Building Materials*, 30, 495–499.

Degirmenci, N., Yilmaz, A. 2009. Use of diatomite as partial replacement for portland cement in cement mortars. *Construction and Building Materials*, 23(1), 284–288.

Dominguez-Rosado, E., Pichtel, J. 2003. Chemical characterization of fresh, used and weathered motor oil via GC/MS, NMR and FTIR techniques. *Proceedings of the Indiana Academy of Science*, 112(2), 109-116.

Ehinola, O. A., Falode, O. A., Jonathan, G., 2012. Softening point and penetration index of bitumen from parts of Southwestern Nigeria. *Nafta*, 63(9-10), 319-323.

Guo, Q., Li, L., Cheng, Y., Jiao, Y., Xu, C. 2015. Laboratory evaluation on performance of diatomite and glass fibre compound modified asphalt mixture. *Materials and Design*, 66(2015), 51–59.

Ibrahim, S. S. 2012. Diatomite Ores: Origin, characterization and applications. *Journal of International Environmental Application & Science*, 7(1), 191–199.

Jia, X., Huang, B., Bowers, B., Zhao, S. 2014. Infrared spectra and rheological properties of asphalt cement containing waste engine oil residues. *Construction and Building Materials*, 50 (1), 683-691.

Jiang, L., Liu, Q. 2014. Application of diatomite modified asphalt. *Applied Mechanics and Materials*, 477-478, 959-963.

Kamaruddin, N. H. M., Hainin, M. R., Hassan, N. A., Abdullah, M. E. 2014. Rutting evaluation of aged binder containing waste engine oil. *Advanced Materials Research*, 911, 405-409.

Kolo, S. S., Jimoh, Y. A., Adeleke, O. O., Adama, A. Y., Akinmade, O. D. 2016. Response of cold mix asphalt produced with straight run bitumen blended with polyethylene to static loading. *Jurnal Teknologi*, 79(1), 131-136.

Newman, P., Hargroves, C., Kumar, A., Whistler, L., Farr, A., Wilson, K., Beauson, J., Matan, A., Surawski, L. 2012. *Reducing the environmental impact of road construction*. Sustainable Built Environment National Research Centre, Brisbane, Queensland.

Shukry, N. A. M., Hassan, N. A., Hainin, M. R., Abdullah, M. E., Abdullah, N. A. M., Mahmud, M. Z. H., Putrajaya, R., Mashros, N. 2016. Experimental evaluation of anti-stripping additives on porous asphalt mixtures. *Jurnal Teknologi (Sciences & Engineering)*, 78(7-2), 113-119.

Villanueva, A., Ho, S., Zanzotto, L. 2008. Asphalt modification with used lubricating oil. *Can. Journal of Civil Engineering*, 35(2), 148–157.

Yi-qiu, T., Lei, Z., Xing-you, Z. 2012. Investigation of low-temperature properties of diatomite-modified asphalt mixtures. *Construction and Building Materials*, 36, 787-795.

Zhu, D. P., Zhang J. C., Chen J. B., Yuan, K., Cheng, C. 2013. Experiment on road performance of diatomite modified asphalt mixture in permafrost region. *China Journal of Highway and Transportation*, 26, 23-28.