

Research Article/Araştırma Makalesi

**Investigating Aluminum Oxide and Silicon Dioxide
Modified Bitumen Stiffness Modulus with Empirical
Method**

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Abstract

In this study, bitumen was modified with aluminum oxide and silicon dioxide using high shear mixer. As a result of this modification, bitumen performance was investigated with standard test methods. Besides, stiffness modulus and plastic stiffness of bitumen was calculated using empirical methods. Nowadays, nanotubes were used for modification of bitumen, commonly. It is known that, nanotubes can improve the bitumen performance at nano level because of having high specific surface area. Bitumen modification was conducted with aluminum oxide at 3%, 5% and silicon dioxide at 0.3%, 0.5% by weight of bitumen because silicon dioxide has high specific surface area and specific gravity. Bitumen characteristics were determined by bitumen tests (rotational viscometer, penetration, softening point, ductility, elastic recovery and penetration index). In this study, bitumen performance grading (PG) system was used and bitumen was used as PG 64-22. In addition, stiffness modulus and plastic stiffness of bitumen was calculated with empirical methods. As a result, 3% Al₂O₃ and 0.3% SiO₂ modified bitumen has the best performance according to the modification and base bitumen.

Keywords: Empirical modeling, Stiffness modulus, Plastic stiffness, Bitumen performance, Nanotube modification.

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Alüminyum Oksit ve Silikon Dioksit ile Modifiye Edilmiş Bitümün Rijitlik Modülünün Ampirik Yöntemle İncelenmesi

Öz

Bu çalışmada, bitüm ile alüminyum oksit ve silikon dioksit yüksek devirli karıştırıcı yardımıyla karıştırılarak modifikasyon sağlanmıştır. Bu modifikasyonun sonucunda bitüm performansı standart bitüm deneyleri ile incelenmiştir. Ayrıca ampirik yöntemler kullanılarak rijitlik modülü hesaplanmıştır. Günümüzde nanotüp modifikasyonu yaygın olarak kullanılan bir metot haline gelmiştir. Nanotüplerin yüksek yüzey alanına sahip olmasından dolayı nano boyutta bitüm performansının iyileştirildiği bilinmektedir. Bitüm modifikasyonu ağırlıkça %3, %5 alüminyum oksit ve %0.3 ve %0.5 oranlarında silikon dioksit ile modifiye edilmiştir çünkü silikon dioksitin yüzey alanı ve özgül ağırlığı fazladır. Bitüm karakteristikleri bitüm testleri (dönel viskozimetre, penetrasyon, yumuşama noktası, duktilite, elastik geri dönme ve penetrasyon indeksi) ile belirlenmiştir. Çalışmada bitüm performans derecelendirme (Performance Grade – PG) sistemine göre sınıflandırılmış ve PG 64-22 olarak kullanılmıştır. Ayrıca ampirik yöntemle rijitlik modülü hesaplanmıştır. Sonuçta, 3% Al₂O₃ ve 0.3% SiO₂ modifiye edilmiş bitüm diğer modifikasyonlara göre daha iyi sonuç vermiştir.

Anahtar kelimeler: Ampirik modelleme, Rijitlik modülü, Plastik rijitlik, Bitüm performansı, Nanotüp modifikasyonu.

1. Introduction

Nowadays, nanotubes are popular modifiers to improve the bitumen properties because of having high specific surface area and specific properties at nano level. Too many studies have been performed nanotube modification of bitumen. Bitumen properties are improved by nanotube modification. Nano TiO₂, SiO₂, ZnO, CNTs are well-known materials used to modify bitumen in the literature (Hongliang et al., 2016; Shafabakhsh and Ani, 2015; Nejad et al., 2017). Sadeghnejad and Shafabakhsh (2017) investigated nano TiO₂ and SiO₂ together in different contents (0, 0.3, 0.6, 0.9 and 1.2%) and the results show that different percentages of nano materials is capable to improve the mechanical behavior of stone mastic asphalt, significantly (Sadeghnejad M and Shafabakhsh G., 2017). Zhu, Zhang, Shi and Li (2017) investigated rheological properties of nano ZnO and vermiculite modified bitumen before and after aging. As a result, modified bitumens containing nano modifier showed the lower complex modulus and the higher phase angle (Zhu et al., 2017). Also, carbon nanotubes are usually used nano materials which are comparatively cheap nano material in bitumen modification. Carbon nanotubes can be effectively used to enhance the bitumen rutting performance (Santagata et al., 2012; Arabani and Faramarzi, 2015; Galooyak et al., 2015).

As seen from the literature review there are few study about aluminum oxide modified bitumen and its performance. Also, nano aluminum dioxide was investigated in the literature only by Ali,

Ismail, Yusoff, Hassan and Ibrahim (2016) as a modifier of bitumen (Ali et al., 2016). The study indicated that nano AL₂O₃ can be considered as a proper alternative additive to modify the properties of bitumen. This study aims to address the deficiency in the literature. In this study, stiffness modulus and plastic stiffness was calculated with empirical model different from the literature. To this aim, a model was calculated based on Ullidtz and Larsen's mathematical model (Ullidtz and Larsen, 1984). Stiffness modulus and plastic stiffness of bitumen was determined by mathematical model developed by Ullidtz and Larsen (1984). The parameters used are acting time, penetration index and softening point for stiffness modulus. Plastic stiffness was effected by viscosity and the acting time.

In this study, data were taken from previous study (Karahancer et al., 2019). Thus, modification rates were chosen in accordance with the literature. Aluminum oxide and silicon dioxide were added to the bitumen in the rate of 3%, 5% and 0.3%, 0.5%, respectively. High shear mixer was used to modify bitumen with nano materials. Modification effort was conducted at 3000 rpm and at 160 °C degree. After that, rheological tests were conducted on all modified bitumens.

2. Materials and Method

2.1 Materials

60/70 penetration grade bitumen was produced and supplied from Tupras,

İzmir, Turkey. The physical and chemical compositions of the bitumen are listed in Table 1. An aluminum oxide nanoparticle (Al_2O_3) and silicon dioxide

nanoparticle (SiO_2) were used in this study as a modifier of base bitumen. Their main properties are listed in Table 2 and Table 3.

Table 1. Bitumen properties

Test		Unit	Base Bitumen
Specific Gravity		gr/cm ³	1.02
Penetration @25 °C		0.1 mm	62.2
Softening Point (Ring & Ball)		°C	49.9
Ductility @25°C, 5 cm/min		cm	>100
RV @135 °C, ≤3Pa.s		Pa.s	0.475
RV @165 °C		Pa.s	0.15
RV @185 °C		Pa.s	0.075
DSR G*/sinδ>1 kPa @10 rad/s	Fail Temperature	°C	67.9
	Grade	°C	64
Mass Loss		%	0
Permanent Penetration		%	70.4
Change in Softening Point		°C	+3.2
DSR G*/sinδ>2.2 kPa @10 rad/s	Fail Temperature	°C	67
	Grade	°C	64
DSR G*.sinδ<5.000 kPa @10 rad/s	Fail Temperature	°C	28.6
	Grade	°C	22
		°C	-12
BBR S≤300 MPa, m≥0.300 @60 s	m-value		0.325
	Stiffness	MPa	213
		Performance Grade	PG 64-22

Table 2. Nano Al_2O_3 properties

Specific Surface Area	≥550 m ² /g
Color and Form	White
Molecular Weight	101.96
True Density	2.9 g/cc
Bulk Density	0.20 g/cc
Al Content (Based on Metal)	>99.1%

Table 3. Nano SiO_2 properties

Specific Surface Area	≥500 m ² /g
Molar mass	60.08 g mol ⁻¹
Appearance	Transparent
Density	2.648 g/cm ⁻³
Melting point	1.713 °C
Boiling point	2.950 °C
Purity	99.9%

2.2 Preparation of Modified Bitumen

The modified bitumen was prepared at a temperature of 160 °C. The modifier rates (3% and 5%) of Al_2O_3 and (0.3% and 0.5%) of SiO_2 were added into the base bitumen. The bitumen was mixed at a rate of 3000 rpm for about 90 min using a high shear mixer, in order to acquire the better dispersion of nanoparticles in the base bitumen. Modified bitumen properties were given in Table 4.

Table 4. Nano modified bitumen properties

	0.3% SiO ₂ 3% Al ₂ O ₃	0.5% SiO ₂ 5% Al ₂ O ₃
Penetration	60.2	65.6
Softening Point	50.9	50.8
Specific gravity	1.025	1.027
Viscosity @135 C	0.654	0.497
Viscosity @165 C	0.13	0.13
Ductility	>100	>100

2.3 Empirical model

Stiffness modulus and plastic stiffness of bitumen was determined by mathematical model developed by Ulliditz and Larsen (Ulliditz and Larsen, 1984). According to Ulliditz and Larsen’s stiffness modulus empirical formula, in this study, stiffness modulus of bitumen was calculated. The motivation was to estimate stiffness modulus empirically was to show effect of penetration index on modification.

Ulliditz and Larsen developed a model to determine the stiffness of bitumen given below:

$$S(t) = 1.157 \times 10^{-7} t^{-0.368} e^{-PI} x (T_{rb} - T)^5 \quad (1)$$

where S(t) is stiffness modulus (MPa), t is time for load duration (s), PI is penetration index, T_{rb} is softening point (ring and ball method) (°C) and T is bitumen temperature (°C).

In this model, the time for load duration was calculated from 0.01 s to 0.1 s and the other parameters was calculated from test results (Kuloglu, 2001).

For the plastic stiffness modulus of bitumen was determined as follows (Ulliditz and Larsen, 1984):

$$S(t)_p = 3v/t_a \quad (2)$$

where, S(t)_p is plastic stiffness modulus (MPa), v is viscosity of bitumen (MPa.s), t_a is time for load duration (s).

In this model, the time for load duration was calculated from 0.01 s to 0.1 s and 135 °C viscosity was chosen because the 165 °C and 185 °C viscosity trend was different for modifications (Ulliditz and Larsen, 1984).

3. Results and Discussion

3.1 Penetration Index Results

Penetration Index (PI) was determined to find out the temperature susceptibility of bitumen. Penetration index should be within -1 <PI <+1. As shown in Figure 1, all bitumen has low penetration index value. Best result was obtained with 5% Al₂O₃ and 0.5% SiO₂ modified bitumen.

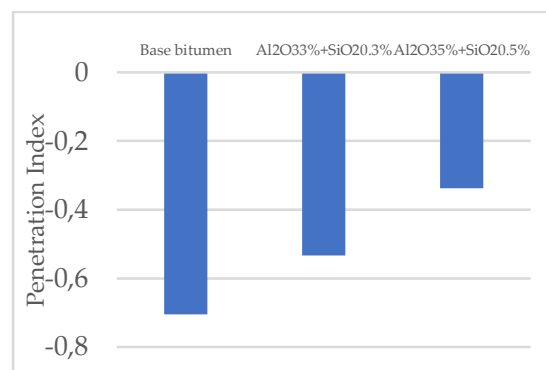


Figure 1. Penetration index values of bitumen

3.2 Viscosity results

The viscosity of the base bitumen becomes greater with the addition of nano Al₂O₃ and SiO₂ at test temperatures (135 and 165°C). However, the 3% and 0.3% modified bitumen samples have shown a significant improvement compared with the base bitumen, as shown in Figure 2. The increase in viscosity is a result of the hardening effect of nano Al₂O₃.

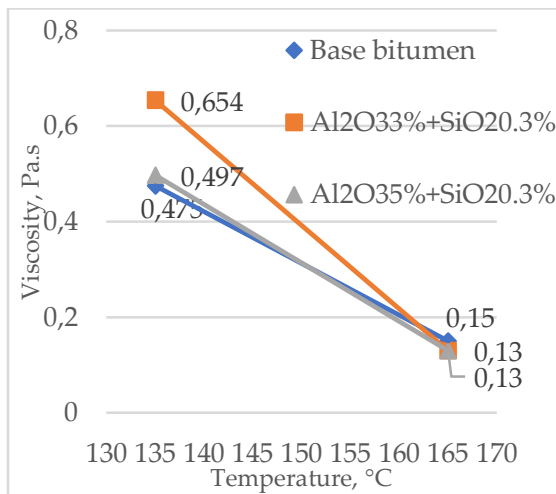


Figure 2. Viscosity values of bitumen

3.3 Empirical Model

Stiffness modulus of Al₂O₃ and SiO₂ modified bitumen was calculated and compared to base bitumen’s stiffness modulus. The graph was drawn based on the stiffness modulus of base bitumen (Figure 3). The results showed that 3% Al₂O₃ and 0.3% SiO₂ modified bitumen has the best stiffness modulus better than the base bitumen. All modified samples showed better results when compared to base bitumen.

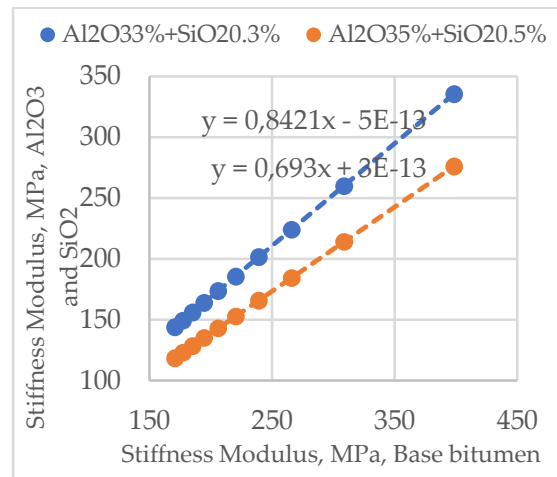


Figure 3. Stiffness modulus of base vs. modified bitumen

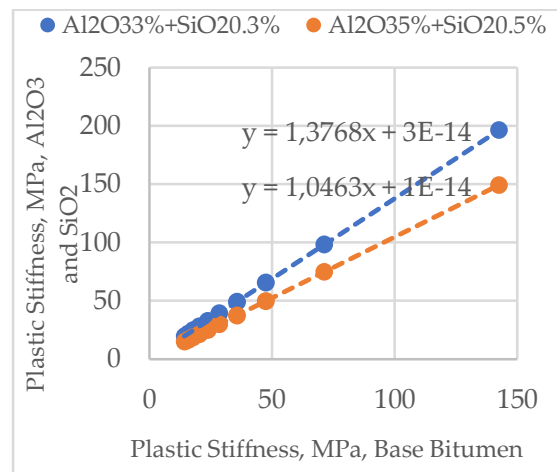


Figure 4. Plastic stiffness of base vs. modified bitumen

Plastic stiffness of Al₂O₃ and SiO₂ modified bitumen was calculated and compared to base bitumen’s plastic stiffness. The graph was drawn based on the plastic stiffness of base bitumen (Figure 4). The results showed that 3% Al₂O₃ and 0.3% SiO₂ modified bitumen has the best stiffness modulus better than the base bitumen. Base bitumen has the less plastic stiffness and all modified samples showed better results when compared to base bitumen.

4. Conclusion

In this study, stiffness modulus of Al₂O₃ and SiO₂ modified bitumen was investigated. Bitumen properties and stiffness modulus was determined. Conclusions can be drawn as follows:

According to penetration index, all modifications were in the limit and means that they are less susceptible to temperature than base bitumen. The 3% and 0.3% modified bitumen samples have shown a significant improvement compared with the base bitumen. 3% Al₂O₃ and 0.3% SiO₂ modified bitumen has the best stiffness modulus better than the base bitumen. 3% Al₂O₃ and 0.3% SiO₂ modified bitumen has the best stiffness modulus better than the base bitumen. As a conclusion, 3% Al₂O₃ and 0.3% SiO₂ modified bitumen has the best performance.

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