

Application of SCB Test and Surface Free Energy Method in Evaluating Crack Resistance of SBS Modified Asphalt Mixes

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ABSTRACT: Cohesion properties of the binder matrix within asphalt mixes and adhesion characteristics of the asphalt binder and aggregate particles are the two major mechanisms resisting against cracking in asphalt mixes. This study is focused on estimating crack resistance of asphalt mixes at intermediate temperatures through evaluation of cohesion and adhesion properties of binder-aggregate systems using Surface Free Energy (SFE) method. Semi-Circular Bending test (SCB) was used to support the SFE analysis. SFE measurements were performed applying Sessile Drop test method. A Granite aggregate type and two asphalt binders (PG64-16, PG58-22) containing various amounts of SBS polymer were used to produce six groups of asphalt mixes. Cohesion and adhesion energies obtained from SFE analysis and Flexibility Indexes and Fracture Energies determined in SCB test showed the positive effect of SBS on performance of asphalt mixes at intermediate temperatures, although the effectiveness of SBS modification was more pronounced with SCB parameters. A linear regression was performed and a strong correlation was observed between SFE results and SCB parameters.

Keywords: Asphalt Mixture, Crack Resistance, Semi-Circular Bending, Sessile Drop, Surface Free Energy.

INTRODUCTION

Fatigue cracking is one of the most common distresses in asphalt pavements which is caused by loss of cohesion in asphalt mastic or loss of adhesion between asphalt binders and aggregate particles or a combination of both mechanisms (Cong et al., 2017; Tan and Guo, 2013; Taherkhani, 2016).

Based on fundamental laws of thermodynamics, although chemical and physical properties of aggregates and asphalt

binders play a major role on bond strength of the mix, Surface Free Energy (SFE) and the balance of these energies at the interface of binder-aggregate affect the bond strength (Al-Qadi et al., 2014). Instruments such as Universal Sorption Device (USD), Wilhelmy Plate (WP), and Sessile Drop (SD) are used to measure SFE properties at the binder-aggregate interface (Hefer et al., 2006). Among these, SD method can be used as a simple approach to determine SFE components of a solid material (Moraes et al.,

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2017; Koc and Bulut, 2013). Although this method can determine the bonding properties of both asphalt binders and aggregates, it was observed in some studies that the mineral heterogeneity on the surface of aggregates and micro texture of the surface can lead to inaccurate results (Hedja et al., 2010; Ghabchi et al., 2014).

Many researchers analyzed SFE to evaluate the cohesion strength of binders and the adhesion properties between combinations of binders and aggregates in presence of water to assess moisture susceptibility of asphalt mixtures. The results were correlated well with protocols such as Tensile Strength Ratio (TSR) (Al-Qadi et al., 2014; Moraes et al., 2017). Similarly, it is possible to use SFE method to evaluate crack resistance of asphalt mixtures at intermediate temperatures.

Various tests were developed to investigate crack resistance of asphalt mixtures at intermediate to low temperatures (Taherkhani and Afroozi, 2017). Semi-Circular Bending test (SCB), due to the geometry of the specimen, the quick testing procedure, and repeatability has been widely used by many researchers (Artamendi and Khalid, 2006). Moreover, unlike beam fatigue specimens, the weight of the SCB specimens cause no sagging during the test (Artamendi and Khalid, 2006; Ozer et al., 2016a).

To quantify crack resistance of asphalt mixtures as a nonlinear viscoelasto-plastic material, the J-integral approach can be applied to SCB test to determine critical strain energy release rate (J_c) (Elseifi et al., 2012). SCB was also used at 25 °C to calculate crack growth rate of asphalt mixtures (Elseifi et al., 2012). Al Qadi and his team used SCB test at a constant displacement rate of 50 mm/min and at temperature of 25 °C to develop a new method in order to compare the crack resistance of various asphalt mixtures (Ozer

et al., 2016a). Their research was correlated strongly with the results of FHWA ALF full scale experiments, and consequently led to a new protocol based on fracture energy and Flexibility Index (FI) (Ozer et al., 2018; Howson et al., 2012).

The FI is a dimensionless parameter which indicates the susceptibility of asphalt mixtures to premature cracking. They found that FI values were decreased as the RAP content of mixtures were increased, indicating a more brittle behavior (Ozer et al., 2016b). Moreover, FI is sensitive to changes in materials including, various asphalt binders, use of modifiers at different concentrations, recycled materials, and volumetric properties of asphalt mixtures (Ozer et al., 2016b; Zhou et al., 2017). In a recent research, Al-Qadi and his colleagues compared various methods of estimating fatigue life of asphalt mixtures (Ozer et al., 2018). They concluded that the correlation of flexibility index, fracture energy, and Texas Overlay Test to field performance were more than 75% (Ozer et al., 2016a, 2018).

Provided that SFE results are consistent with the results of SCB test, SFE method can be used as a simple approach to find proper combinations of binders and aggregates in order to achieve better crack resistance of asphalt mixes at intermediate temperatures.

RESEARCH METHODOLOGY

The main objective of this study was to evaluate the crack resistance of asphalt mixtures at intermediate temperatures through measuring adhesion and cohesion forces within asphalt mixtures. This has been done by performing SFE analysis and making comparison between SFE and SCB test results. The approach used in the latter test was through fracture energy analysis.

Two types of asphalt binders containing different amounts of Styrene-Butadiene-

Styrene (SBS) were used. Given the significant role of aggregates in providing bond strength with asphalt binders, a Granite aggregate type was chosen as one of the critical aggregates that exhibits less adhesion with bitumen binders. Table 1 shows various combinations of mix compositions in this research. The tests were conducted in a randomized order to avoid systematic bias; three samples were tested for each type of mixture.

MATERIALS AND SAMPLE PREPARATION

A PG 64-16 asphalt binder and a PG 58-22 asphalt binder were used in this research since they are the most common binders in Iran. The modification of the bitumen with SBS results in physical cross-linking of

polystyrene blocks. The cross-linking process improves elastic behavior of the polymer-modified binder at intermediate temperatures (Aflaki and Tabatabaee, 2009; Kim et al., 2009). In order to achieve much more stable polymer network in the bitumen, the elemental Sulfur was used as a polymer cross linking agent. The SBS-modified binders were produced, using a high shear mixer with 5000 RPM speed and at 180 °C for 100 minutes (Aflaki and Tabatabaee, 2009; Kim et al., 2009).

SuperPave Performance Grade (PG) of the binders were determined in accordance with ASTM D7643. The results are reported in Table 2. Physical properties of the Granite aggregate used in this study are reported in Table 3. Aggregate gradation was selected so as to meet AASHTO MP 2 grading limits.

Table 1. Experimental design

Type	Composition	Additive
Binder	PG64-22, PG58-22	0%, 3%, 6% SBS
HMA	PG64-22, PG58-22 Granite aggregate	0%, 3%, 6% SBS

Table 2. Performance Grading (PG) data of the asphalt binders

Specimen	Continuous grade (°C)		Performance Grade (PG)
	High temperature	Low temperature	
PG64-16	66.2	-19.7	64-16
PG64-16+3%SBS	72	-19.5	70-16
PG64-16+6%SBS	79.5	-16.8	76-16
PG58-22	60.4	-23.1	58-22
PG58-22+3%SBS	66.1	-22.3	64-22
PG58-22+6%SBS	73.5	-20.1	70-16

Table 3. Physical properties of the Granite aggregate

Test	ASTM standard	Result	Specification
Bulk specific gravity (coarse aggregate)	C127-01	2.683	---
Bulk specific gravity (fine aggregate)	C128-01	2.689	---
Los Angeles abrasion	C131-01	12%	max 35%
Coarse aggregate angularity	D5821-95	96%	min 90%
Flat and elongated particles	D4791-99	5.5%	max 10%
Water absorption (%)	C127, C128	< 1%	---

The SD specimen preparation has a great impact on accuracy of determining the contact angles. When a droplet of probe liquid is going to touch the surface of specimen, the sample should be quite smooth and level (Kwok and Neumann, 1999). To this end, one side of a microscope slide was coated with heated asphalt binder and another slide was pressed on that so as to form a thin layer of the binder between the two slides. In this case, samples are protected against oxidation to avoid alteration in the results. Just before the test, the cover slide was heated and was removed slightly. Only one of the probe liquids can be tested on each slide. For each liquid, a minimum of five droplets should be tested. The reported contact angles are the average of five replicates.

Slabs of HMA mixtures were made and cylindrical specimens were cored from these slabs. Aggregates were heated to 10 °C above the mixing temperature for 3 hours prior to the slab fabrication. Asphalt mixtures were then produced in accordance with AASHTO T 245 Standard procedure. Furthermore, a 4-inch specimen was cored from each slab to control the air void of the slabs. SCB samples were cored from the slabs and three identical specimens were prepared for each test.

TEST METHOD

Sessile Drop method is based on the adhesion between a droplet of specified liquid and a solid surface (Koc and Bulut, 2013). SD method uses Eq. (1) (Young equation) for

quantifying the degree of adhesion (Koc and Bulut, 2013).

$$\gamma_s = \gamma_{sl} + \gamma_l \cos \theta \quad (1)$$

where γ_s : is the free energy of solid surface, γ_{sl} : is the free energy of solid-liquid interface, γ_l : is the surface tension of the probe liquid, and θ : is the contact angle as shown in Figure 1.

One of very common theories for measuring SFE is acid-base theory. It defines SFE as apolar (Lifshitz-van der Waals) and polar (Lewis acid-base) components (Hefer et al., 2006; Moraes et al., 2017). Work of cohesion is defined as the energy required making a separation inside a solid or liquid material, so the cohesion energy (ΔG^c) can be calculated from Eq. (2) below (Moraes et al., 2017):

$$\Delta G_s^c = -2\gamma_s = -2(\gamma_s^{LW} + \gamma_s^{AB}) \quad (2)$$

Work of adhesion between a pair of solid-liquid can be expressed as the energy needed for separating the liquid from the solid surface. It can be written based on Dupre Equation (Moraes et al., 2017; Koc and Bulut, 2013) as shown in Eq. (3):

$$\Delta G_{sl}^a = -2 \left[\sqrt{(\gamma_s^{LW} \times \gamma_l^{LW})} + \sqrt{(\gamma_s^{AB} \times \gamma_l^{AB})} \right] \quad (3)$$

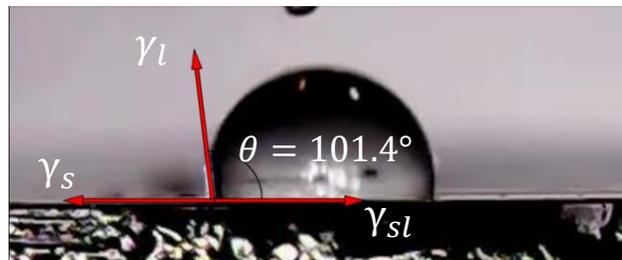


Fig. 1. Contact angle of the droplet of distilled water and solid surface of PG64-22 asphalt binder

where ΔG_{sl}^a : is the adhesion energy, γ_s : is the free energy of solid surface, γ_l : is the surface tension of the liquid.

The following equation can be applied to calculate SFE properties of a solid surface ($\gamma_s^{LW}, \gamma_s^{AB}$). Obviously, two probe liquids are needed to solve Eq. (4).

$$(1 + \cos\theta) \times \gamma_L = 2 \left[\sqrt{(\gamma_s^{LW} \times \gamma_L^{LW})} + \sqrt{(\gamma_s^{AB} \times \gamma_L^{AB})} \right] \quad (4)$$

In order to achieve higher accuracy, the above equation can be rewritten as Eq. (5) where more than two probe liquids will be used (Hedja et al., 2010; Kwok and Neumann, 1999). Regression method can be applied to solve Eq. (5).

$$(1 + \cos\theta) \times \gamma_L / 2 \times \sqrt{\gamma_L^{LW}} = \sqrt{\gamma_s^{AB}} \left(\sqrt{\gamma_L^{AB} / \gamma_L^{LW}} \right) + \sqrt{\gamma_s^{LW}} \quad \text{or} \quad (5)$$

{Y = aX + b}

There are several liquids of known surface tension components ($\gamma_L^{LW}, \gamma_L^+, \gamma_L^-$) which are used in SD method, such as distilled water, Formamide, Diiodomethane, Ethylene glycol, and Glycerol (Hedja et al., 2010). It is noteworthy that the liquid should not react chemically with bitumen during the test

(Kwok and Neumann, 1999). However, it is strongly recommended to use a pair of polar and non-polar liquids (Kwok and Neumann, 1999). After careful screening of liquids in literature, distilled water, Diiodomethane, and Formamide and Glycerin were recognized to be reliable probes. Properties of the probe liquids are reported in Table 4.

Moreover, Kwok and Neumann (1999) suggested a plot to assess the validity of SD results. They concluded that the plot of $(\cos\theta \cdot \gamma_L)$ versus (γ_L) for a solid surface should show a linear relationship with various probe liquids. Otherwise, if a point did not lie on the linear curve, the corresponding liquid should be excluded from SFE calculations, as there is a strong possibility that liquid had complex interactions with the solid surface (Moraes et al., 2017; Kwok and Neumann, 1999).

The standard SCB testing method for evaluating fracture potential of asphalt mixtures at intermediate temperatures is provided in AASHTO TP 124 (AASHTO, 2016). While the use of J-integral method to assess crack resistance of HMAs is very common (Ozer et al., 2018), AASHTO suggested the Illinois Flexibility Index method that was introduced in 2016 (AASHTO, 2016). This method is based on the vertical load-displacement curve of SCB test shown in Figure 2 (AASHTO, 2016). This test is primarily designed on a three point bending fixture. AASHTO TP 124 specified the geometry of SCB specimen as depicted in Figure 3.

Table 4. Properties of the probe liquids at 20 °C (mJ/m²) (Hefer et al., 2006)

Probe liquid	γ_L^{LW}	γ_L^+	γ_L^-	γ_L^{AB}	γ_L^{total}
Water	21.8	25.5	25.5	51.0	72.80
Glycerin	34.0	3.92	57.4	30.0	64.00
Formamide	39.0	2.28	39.6	19.0	58.00
Diiodomethane	50.8	0.0	0.0	0.0	50.80

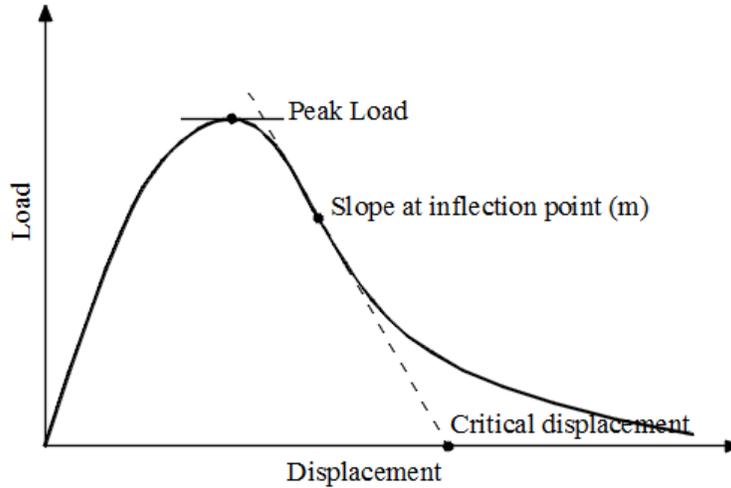


Fig. 2. Load-displacement curve of SCB

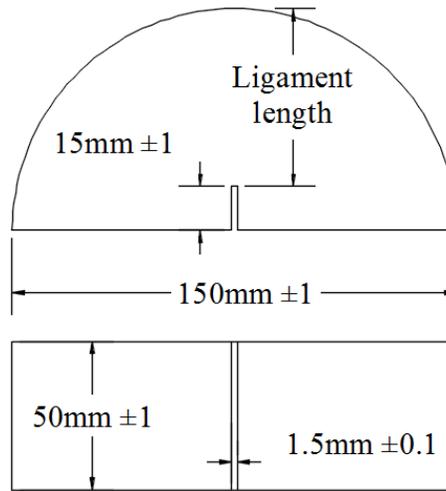


Fig. 3. Geometry of SCB specimen

The outcomes are fracture energy and Flexibility Index. The work of fracture is usually expressed as the area under the load-displacement curve till the failure point (Ozer et al., 2016a; Hakimelahi et al., 2013). The end of the test is the point where the load gets to a value of less than 0.1 kN. In case that the test is stopped prior to the failure point, an extrapolation should be fitted to the curve in order to calculate the closed area.

The fracture energy represents the overall capacity of asphalt mixtures to resist cracking related damage (AASHTO, 2016). It can be expressed as shown in Eq. (6):

$$G_{fa} = \frac{10^6}{b(D-a)} \int_0^{u_{final}} P(u) \cdot du \quad (6)$$

where G_{fa} : is the fracture energy reported in J/m^2 , u_{final} : is the total displacement up to the end of the test (mm); P : is the axial load (kN); b : is the thickness of the specimen (mm), D : is the height of the specimen (mm), a : is the notch depth (mm) and $(D - a)$: is the ligament length. G_{fa} can be used to calculate the main parameter of theoretical crack (cohesive zone) models. To that end, G_{fa} should be corrected to determine energy related to crack propagation only.

Regarding the testing conditions at intermediate temperatures, the thickness of 35 to 50 mm for the specimen, the notch length of 5 to 15 mm, and the loading rate of 1 to 5 mm/min are reported to provide consistent results (Ozer et al., 2016a). However, AASHTO TP 124 recommended loading rate of 5 mm/min.

Flexibility Index (FI), a dimensionless parameter, can be calculated from Eq. (7) below:

$$FI = \frac{G_{fa}}{|m|} \times A \quad (7)$$

where m : is the post-peak slope of the load-displacement curve at inflection point (kN/mm^2). Actually m indicates the average crack growth rate. The increase of this slope results in lower FI values, indicating more susceptible asphalt mixtures to premature

cracking (Hakimelahi et al., 2013). A : is a constant value equal to 0.01 for unit conversion and scaling of the results.

SANTAM UTM equipped with a temperature controlled chamber was used in this study. A laser leveler was used to align the loading line with the specimen notch as shown in Figure 4.

RESULTS

The contact angles for the binders obtained from Sessile Drop test and the results of SFE analysis are reported in Tables 5 and 6 respectively. It should be noted that regarding the aggregate SFE analysis, results of USD method were used. In fact, the results of SD test on aggregates were not consistent and were lower than those obtained from USD test.



Fig. 4. SCB specimen and the testing setup: a) Using laser leveler; b) Temperature controlled chamber

Table 5. Contact angles obtained from SD test

Sample	Contact angles ($^{\circ}$)				$(R^2)^a$	$(R^2)^b$
	Water	Glycerin	Formamide	Diiodomethane		
PG64-16	102.1	84.0	76.05	66.1	0.94	0.90
PG58-22	100.6	83.5	76.0	65.6	0.95	0.96
PG64-16+3%SBS	99.6	81.2	73.2	62.3	0.94	0.92
PG58-22+3%SBS	97.4	80.0	71.7	60.8	0.94	0.97
PG64-16+6%SBS	96.9	79.1	70.0	59.05	0.93	0.94
PG58-22+6%SBS	94.8	76.8	69.6	56.1	0.95	0.98

^a R^2 of Kwok-Neumann plot (the plot of $\cos\theta \cdot \gamma_L$ versus γ_L); ^b R^2 of regression analysis from Eq. (9)

Table 6. Results of SFE analysis in (mJ / m²)

Test	SFE parameters				
	γ_S^{LW}	γ_S^{AB}	γ_S^{total}	$\Delta G^{coh.}$	$\Delta G^{adh.}$
Granite Aggregate	59.60	57.20	116.80	-	-
PG64-16	26.32	0.61	26.93	53.86	91.03
PG58-22	26.21	0.81	27.02	54.04	92.66
PG64-16+3%SBS	28.52	0.74	29.26	58.52	95.47
PG58-22+3%SBS	28.94	0.98	29.92	59.84	98.04
PG64-16+6%SBS	30.36	0.95	31.31	62.62	99.82
PG58-22+6%SBS	31.21	1.14	32.35	64.70	102.41

* $\Delta G^{coh.}$ and $\Delta G^{adh.}$ were calculated from Eqs. (2) and (3), respectively.

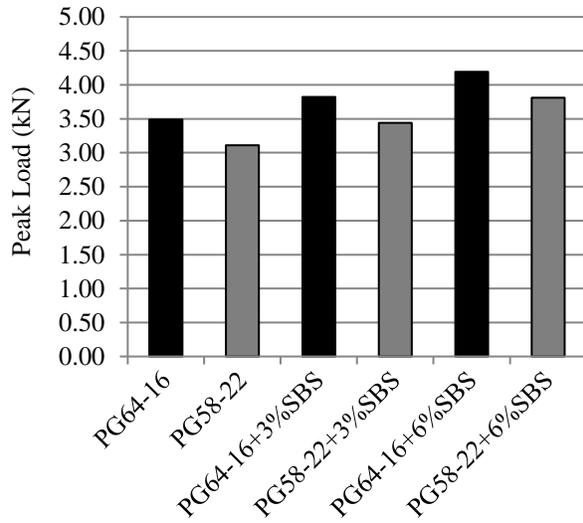
Although γ_S^{LW} of PG 64-16 is higher than PG 58-22, γ_S^{AB} or the polarity of PG 64-16 is lower than PG 58-22, and the overall SFE energy of PG 58-22 is higher than PG64-22 which resulted in better adhesion and cohesion of PG 58-22 in compare to former one.

Comparing the SFE results of PG 64-16 and PG 58-22 binders showed that, SBS modified binders had higher apolar and polar parts ($\gamma_S^{LW}, \gamma_S^{AB}$), compared with the neat binders. In other words SBS modification resulted in greater cohesion inside the modified binder and also better adhesion with Granite aggregates.

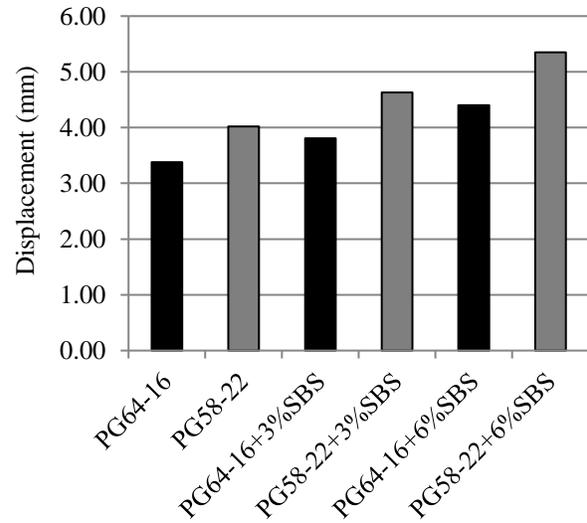
Making smooth curves for SCB load-displacement graphs, polynomial models were fitted to the graphs (Mohammad et al.,

2016). Coefficients of determination for all models used in this study were more than 0.95. The results of SCB tests are shown in Figure 5. It is clear that SBS had a positive effect on both the peak load and the displacement up to the failure point. The outcome was a bigger area under the load-displacement curve or higher fracture energy (G_{fa}). In addition, SBS modification resulted in lower post-peak slope of the load-displacement curve (m). This indicates that SBS, as an elastomer, reduces the average crack growth rate and results in greater FI values.

Coefficient of Variation (CV) for each group of SCB samples was calculated to evaluate the precision and repeatability of the results (Table 7).



(a) Peak load



(b) Displacement at failure point

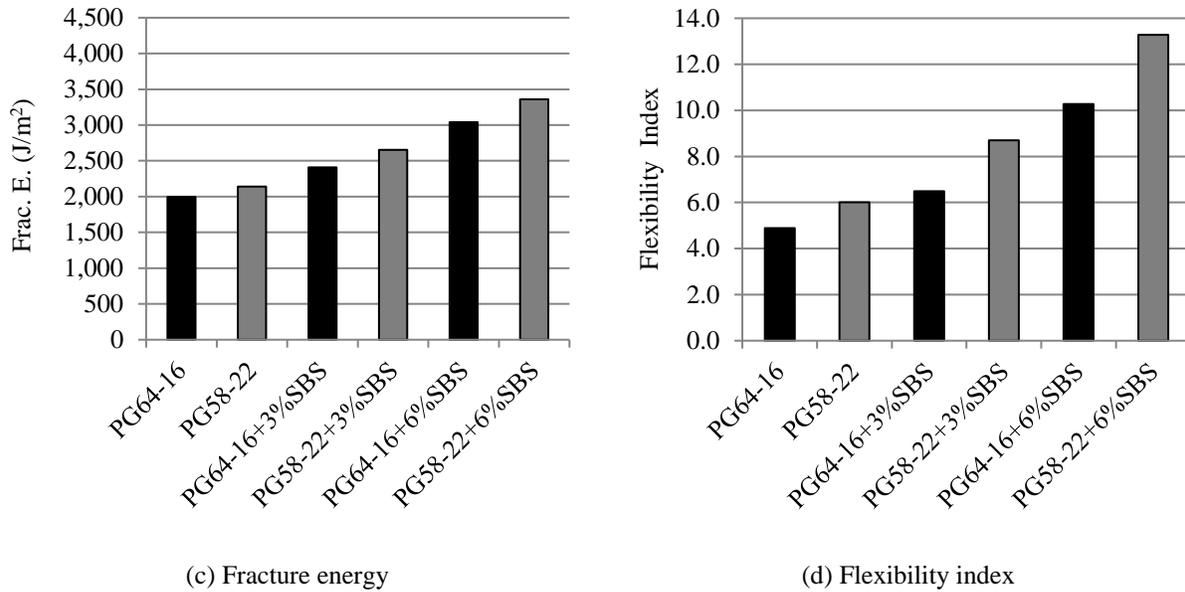


Fig. 5. Results of SCB test on mixes containing different amounts of SBS

Table 7. Coefficients of variation of the SCB test results

Sample	Fracture energy (G_{fa}) (J/m ²)	CV	Flexibility Index (FI)	CV
PG64-16	1996.33	5%	4.89	7%
PG58-22	2140.07	5%	6.01	6%
PG64-16+3%SBS	2406.51	7%	6.49	10%
PG58-22+3%SBS	2653.68	8%	8.70	10%
PG64-16+6%SBS	3042.00	9%	10.28	13%
PG58-22+6%SBS	3359.90	8%	13.28	12%

FI values in this study varied from 4.89 (for the mix with PG64-16) to 13.28 (for the mix with PG58-22+6%SBS). FI values above 6 are considered flexible and have acceptable performance at intermediate temperatures (Ozer et al., 2018).

DISCUSSIONS

Figure 6 illustrated SFE and SCB parameters for all of the mixes presented in this study. In order to make comparison between these parameters, the values were normalized. PG64-22 sample was assumed as the reference sample for normalization. To that end, SFE, G_{fa} , and FI values of the samples were divided by the corresponding value of the PG 64-22 sample.

Trends of all results were the same, approving the positive effectiveness of SBS-modified binder on crack resistance of asphalt mixtures at 25 °C. Regarding the degree of effectiveness of the SBS modification, SCB parameters showed a greater level of performance for the modified asphalt mixtures when it was compared with the SFE data. The difference can be attributed to the protocol of the latter test. SFE test evaluates adhesion and cohesion forces just at one point of the specimen; while, the SCB test imposes stresses on the specimen at a strip area with a wider contact points of cohesion or adhesion inside the specimen resisting against it. The stronger these contact points are, the greater stress level will be imposed before that the specimen is failed.

Regression analysis was performed between the results of SFE analysis and SCB data. Since the aggregate type was not a variable in this study, the adhesion energies were a function of cohesion energies obtained from SFE analysis. For this reason, the cohesion energies were taken into consideration, and the regression analysis was performed between them and the SCB parameters as shown in Figure 7.

The R-squared values of the linear regression in Figures 7a and 7b are 0.97 and 0.90, respectively. Although SFE method

does not fully represents how chemical and physical characteristics of binder-aggregate systems affect the bond strength of the asphalt mixture, SFE results showed an acceptable correlation with FI and fracture energies obtained from the SCB test. This can indicate that SFE analysis is a strong method for evaluating the role of additives, such as SBS on fracture resistance of modified asphalt mixtures at intermediate temperature. However, the degree of improvement cannot be predicted with absolute certainty.

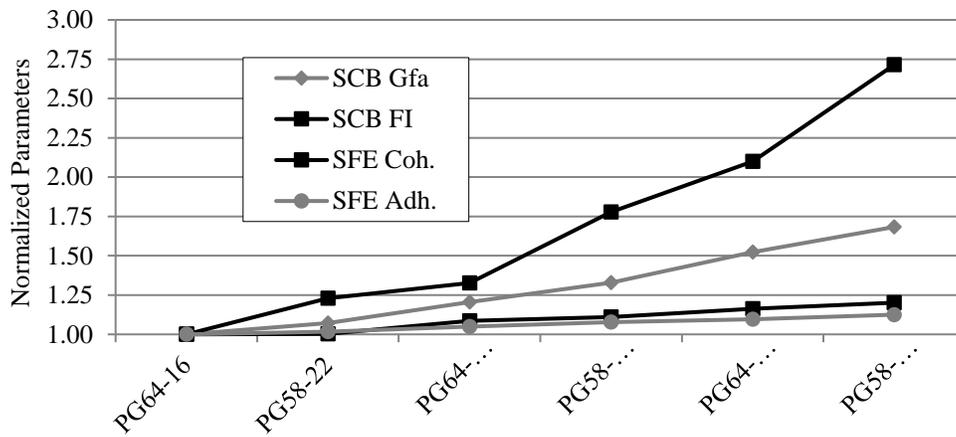
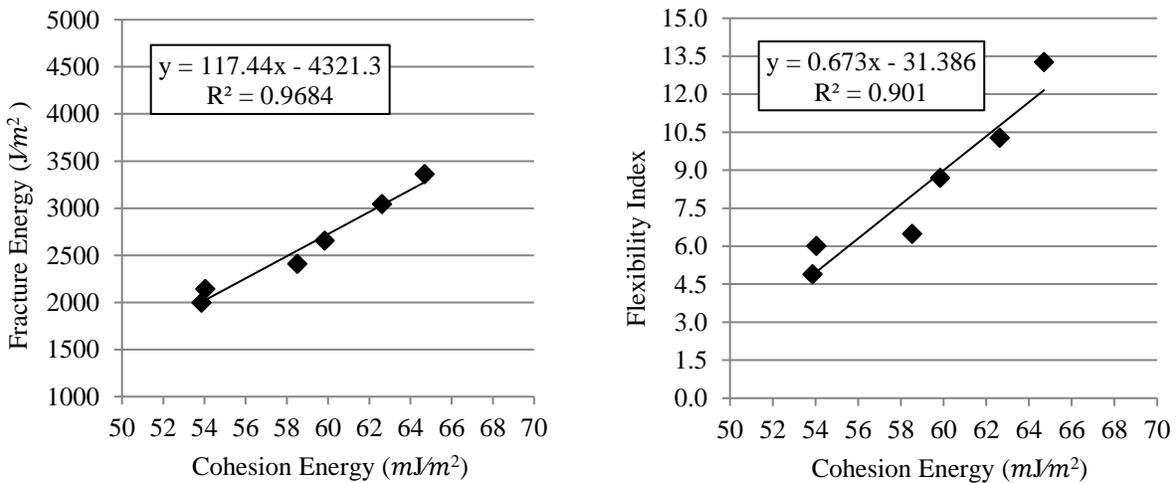


Fig. 6. Comparison of normalized results of SFE analysis and SCB test



(a) Correlation between $\Delta G^{coh.}$ and G_{fa} (b) Correlation between $\Delta G^{coh.}$ and FI

Fig. 7. Regression analysis between SFE and SCB test results

CONCLUSIONS

This study was focused on predicting crack resistance of SBS modified asphalt mixtures at intermediate temperatures through performing SFE analysis. The SCB test (AASHTO 124-16) was also applied to determine the fracture potential of the asphalt mixtures at intermediate temperatures and to evaluate the SFE results. The summary of the findings are as follows:

- It was determined that the probe liquid type has a significant effect on the results. Application of four probe liquids that one of them must be a non-polar liquid (i.e. Diiodomethane), and performing the regression analysis to calculate SFE properties were very reliable.
- SFE analysis indicated that SBS modification results in greater cohesion within the binder and also a better bond between the binder and aggregate particles.
- Performing regression analysis between SFE and SCB results showed that SFE analysis was able to predict crack resistance of asphalt mixtures at intermediate temperatures.
- Although SFE analysis of the data was in agreement with SCB data, SFE method is a localized analysis and consequently the positive effect of SBS modification based on SFE parameters was lower than those achieved in SCB test.
- SCB results indicated that the SBS modified mixes, compared with the control mix, showed greater tensile strength, higher fracture energies and resulted in greater FI values which indicate lower crack growth rates.

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