



# Experimental evaluation of fatigue resistance of asphalt mixtures containing waste elastomeric polymers

Mahmoud Ameri <sup>\*</sup>, Sadeq Yeganeh, Peyman Erfani Valipour

School of Civil Engineering, Iran University of Science and Technology, Narmak, Tehran, Iran

## HIGHLIGHTS

- Study on effect of polymer production process waste on asphalt mixture fatigue life.
- Four-point bending test and indirect tensile fatigue test have been performed.
- Two analyses for fatigue: Ratio of Dissipated Energy Change and Energy Ratio Method.
- Comparison on fatigue life of modified mix by waste of polymer and unmodified mixes.
- Comparison on modified mixes by waste of polymer and mixes with original polymer.

## ARTICLE INFO

### Article history:

Received 8 May 2018

Received in revised form 1 October 2018

Accepted 1 November 2018

Available online 6 December 2018

### Keywords:

Polymer waste

Fatigue resistance

Hot-mix asphalt

Four-point bending test

Energy approaches

## ABSTRACT

This study concerning the asphalt mixture fatigue life, affected by the addition of waste elastomeric polymers obtained from polymer production process. Since the use of polymers is economically costly, and also there are some by-products in polymer production process, considering recycled polymers and waste of polymers is important in terms of environmental clean-up. Investigation of fatigue characteristics of HMA mixtures containing waste of elastomeric polymers and their comparison with unmodified mixtures are the main objectives of this research. In addition, different fatigue analysis approaches were evaluated in order to figure out the most precise results. Fatigue resistance of hot-mix asphalt (HMA) modified by elastomeric polymers such as SBR and PBR and the waste of their production process, was studied by performing four-point bending (4 PB) test and indirect tensile fatigue test (ITFT) based on conventional and energy approaches. As far as the technical aspects are concerned, the traditional method based on 50% reduction in initial stiffness is not suitable enough to be employed for calculating fatigue life, because this method underestimates the fatigue life of polymer modified mixtures and leads to high variations among the responses. Therefore, two alternative fatigue analyses were conducted: The Ratio of Dissipated Energy Change method and the Energy Ratio method. Both these methods provided a more detailed comparison between the fatigue life of these mixtures. As the results shows, fatigue life of mixes modified by the waste of polymers, is significantly higher than unmodified mixes and considerably close to mixes including original polymers.

© 2018 Published by Elsevier Ltd.

## 1. Introduction

Each pavement is subjected to numerous cyclic loadings of traffic which reduce its serviceability because of unstable propagation of cracks from the base layer to the surface. Material properties, axle loads, pattern of loading, environmental conditions and the compaction effort are factors which affect the fatigue resistance. Since fatigue cracks decrease bearing capacity of the road structure, it is necessary to investigate fatigue behavior, cracking

mechanism of the mixtures, the initiation threshold of cracks and resistance to crack extension [1,4].

Paving projects require a considerable amount of time and financial resources. Furthermore, the need for sustainable maintenance for pavement networks is widely recognized. The fact that sustainability is important in road construction, has led to utilizing environmentally friendly materials in asphalt mixtures production in order to reduce environmental footprint. In some of the petrochemical processes associated with polymer production, there are cheap and useless wastes whose decomposition in the environment last for many years. Hence, dumping them in the nature causes degradation of the environment and soil pollution and it

<sup>\*</sup> Corresponding author.

E-mail address: [ameri@iust.ac.ir](mailto:ameri@iust.ac.ir) (M. Ameri).

is necessary to find a proper solution to the reuse and recycle these wastes via other applications.

Over the last decades, many projects have been done and different materials have been successfully exploited to improve the fatigue properties of binder and Hot Mix Asphalt (HMA) as bitumen modifiers, while polymer modifiers had the most applications [2]. In a research by Haryati Yaacob et al. [3], the rheological properties of 60–70 bitumen modified Styrene Butadiene Rubber (SBR) was evaluated. By carrying out many tests on binder modified by a varying percentage of SBR up to 5% by the weight of bitumen binder, it was found that SBR increased viscosity and  $G^*/\sin\delta$  parameter, which means the better performance of modified bitumen against rutting at high temperature. But more than 3% SBR had a negative effect on the fatigue cracking resistance. In another study by Garcia-Morales et al. [6] on the mechanical and rheological properties of modified asphalt binders, four different waste polymers were added to petroleum bitumen. Ethylene-Vinyl Acetate (EVA), EVA/LDPE blend, crumb tire rubber and Acrylonitrile Butadiene Styrene (ABS) were used as bitumen modifiers. It is proved that enhanced rheological properties at both low and high in-service temperatures can be obtained by using a blend containing 3.5% EVA/LDPE and 3.5% crumb rubber by the weight of bitumen. As an elastomer, tire rubber endowed the pavement higher flexibility, which made it more resistant to thermal cracking at low in-service temperature. On the other hand, although thermoplastic elastomers such as styrene-butadienestyrene (SBS) and plastomers such as EVA copolymer are distinguished to be good modifiers [5,8], rubber is more preferable because of its lower prices.

The effect of SBR and SBR/Natural bitumen on classical and rheological properties of bitumen was investigated by Ji Zhuang et al. [40]. It was observed that 3% of SBR by the weight of bitumen is an appropriate content of SBR modified bitumen; otherwise the industrial production will be costly. It was also shown that addition of SBR resulted in improved recovery, low-temperature ductility, and rutting resistance, increased viscosity and complex modulus and decreased phase angle significantly. Furthermore, significant increase in complex modulus ( $G^*$ ) as a result of higher percentage of SBR at high temperature was obtained.

Since the use of polymers as bitumen modifier are economically costly, and also there are some cheap and useless by-products in polymer production process, mentioned material has been considered for this research. On the other hand, there is no consensus on the effect of waste polymers on fatigue behavior of hot mix asphalts, and because of environmental and economic aspects of using mentioned materials as bitumen modifier, the main objectives of this research are defined as follows:

- Study on effects of production process waste of two different elastomeric polymer (SBR and PBR) as bitumen modifier (in different percentage) on fatigue behavior of hot mix asphalt mixture.
- A comparison between effects of mentioned waste polymers and the original polymer as a bitumen modifier with respect to the fatigue characteristics of asphalt mixtures using two different fatigue testing methods.
- Comparing different energy approaches with the traditional approach ( $N_f/50$ ) for evaluating Fatigue life of hot mix asphalt and investigating the results.

## 2. Materials and samples

### 2.1. Materials

#### 2.1.1. Stone matrix

The crushed limestone aggregate used in this study was obtained from Asb-cheran quarries in the eastern part of Tehran.

Grading is done according to the Iran pavement standard. Fig. 1 shows particle size distribution of aggregates with nominal maximum aggregate size of 19 mm. The selected gradation falls within the upper and lower limits of No.4 gradation of Iran Highway asphalt paving code [7]. The physical properties of the aggregate are presented in Table 1.

#### 2.1.2. Base binder

The base binder with penetration grade of 60/70 was from Tehran's oil refinery. Properties of the base bitumen are presented in Table 2.

#### 2.1.3. Modifiers

According to the focus of this study on usage of waste elastomer polymers, the modifiers are included two elastomers called Styrene Butadiene Rubber (SBR) and Poly Butadiene Rubber (PBR) and the waste of them. SBR and PBR are mostly consumed in tire manufacturing, shoes production and glue industries. The waste of mentioned polymers are obtained from the production process and have some physical and minor chemical differences with the original ones. The picture of polymeric additives used in this study are illustrated in Fig. 2. Also, Properties of used polymers are presented in Tables 3 and 4.

### 2.2. Sample preparation

#### 2.2.1. Modified bitumen preparation

According to the introduced materials and goals of this study, seven different binders have been considered. One base binder has been used as the control sample. Six other are including polymer modified binders (PMB). Two different percentages of polymers' waste were used with one specific gradation of crushed limestone and one percentage of original polymer has been added to binder for comparison. These seven binder blends results in seven different hot mix asphalt (HMA) mixtures.

In order to prepare the PMBs, the bitumen heated up to 165 °C in the oven, and was poured into the container of the mixer. The mixing process was conducted by a high-shear mixer for different sets of bitumen. The container of mixer was heated up to 170 °C. First, rubbers were cut into small pieces with approximate dimensions of 5 mm (smaller pieces make the mixing process faster and easier) and added to the heated bitumen at the speed of 3000 rpm and mixed for first 10 min. Thereafter, the mixing was continued for the next 30 min at a speed of 6000 rpm. Each waste polymers were added to the neat bitumen at two different percentages (3% and 5% of bitumen weight) while the dosage of original polymers is 3% by the weight of bitumen.

In a research by Yousefi [39], the dispersion of same type of SBR and PBR polymers in binder (with proportion of 5% of the weight of bitumen) has been investigated. The results of this study shows that the PBR could forms a continuous phase, whereas, SBR disperse in bitumen in fine particles which is resulted in the stability of these PMBs.

#### 2.2.2. Asphalt mixtures

Optimum asphalt content for each mixture was calculated by using the Marshall Mix design procedure. Volumetric properties of the mixtures are summarized in Table 5.

Three specimens for each asphalt content (4.5%, 5.0%, 5.5%, and 6.0% of weight) were prepared for each control mixes and each polymer modified mixes. A total of 84 specimens were tested for unit weight, air voids, stability, flow, and voids in mineral aggregate (VMA), and the average values of them were calculated. The target air voids content of 4% is considered. The values of volumetric parameters were checked to meet the minimum requirements. According to the standard (Iran Highway asphalt paving code), all

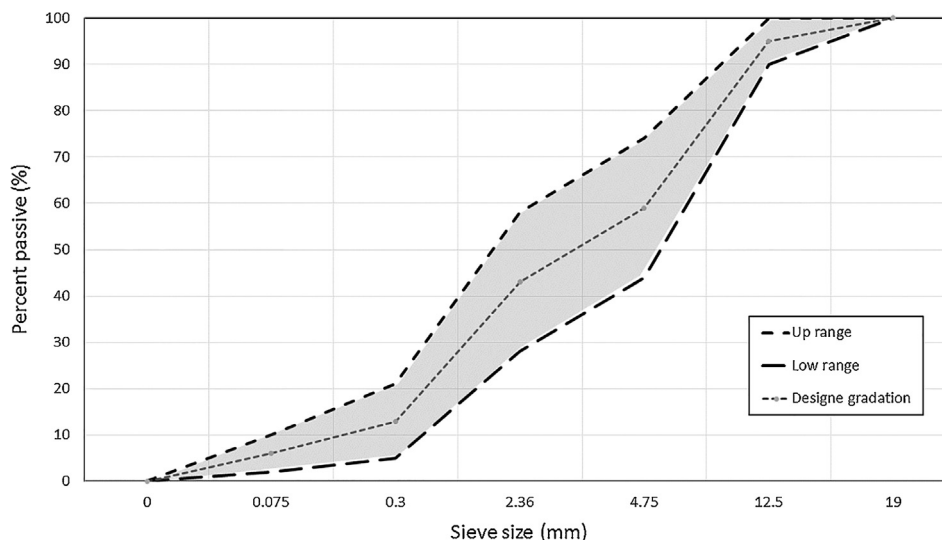


Fig. 1. Gradation.

**Table 1**  
Engineering properties of aggregates.

Property	Test method	Value
Bulk specific gravity of coarse aggregate	AASHTO T85	2.631
Bulk specific gravity of fine aggregate	AASHTO T84	2.592
Bulk specific gravity of mineral filler	AASHTO T84	2.702
Absorption coarse aggregate (%)	ASTM C127	1.4
Absorption fine aggregate (%)	ASTM C128	1.8
Los Angeles abrasion loss (%)	AASHTO T96	22.3
Tow fractured faces (%)	ASTM D5821	96
Flat and elongation (3-1)%	ASTM D4791	16

**Table 2**  
Engineering properties of base bitumen.

property	Test methods	Value
Specific gravity @ 25 °C (g/cm <sup>3</sup> )	ASTM D70	1.014
Penetration @ 25 °C (0.1 mm)	ASTM D5	65
Softening point (°C)	ASTM D36	48
Ductility @ 25 °C (cm)	ASTM D113	112
Flash point (°C)	ASTM D92	302
Fire point (°C)	ASTM D92	311
Solubility of asphalt materials in trichloroethylene	ASTM D2042	99.6
Kinematic viscosity @ 135 °C	ASTM D2170	351
Heating loss (%)	–	0.04
Penetration index (PI)	–	–1.098

samples passed the minimum Marshall Stability requirement which is 8.006KN [7]. The calculated optimum asphalt contents were 5.2% for A and B mixes and 5.3% for C, D, E and F mixes, prepared using waste of polymers, and 4.6% for control mix.

**Table 3**  
SBR1502 Typical properties.

Typical properties	Units	Values	Test Method
Raw Mooney viscosity	MU	46–58	ASTM D1664
Volatile Material	% wt	<0.75	ASTM D5668
Ash Content	% wt	<1.0	ASTM D5667
Organic acids	% wt	4.75–7	ASTM D5774
Soaps	% wt	<0.5	ASTM D5774
Bounded styrene	% wt	22.5–24.5	ASTM D5775
Compound Mooney viscosity	MU	<84	ASTM D1664
Tensile strength (35 min cured)	kg/cm <sup>2</sup>	>250	ASTM D 412
Ultimate elongation (35 min cured)	%	>350	ASTM D 412
300% Modulus (35 min cured)	kg/cm <sup>2</sup>	167–207	ASTM D 412

According to the Strategic Highway Research Program [10] a Gyrotory Shear Compactor (GSC) compacted the cylindrical specimens with 600 kPa vertical pressure, 30 rpm gyration speed, gyration angle of 1.25°, and 100 mm mold diameter. All the specific gravity (Unit Weight) of samples are calculated according to ASTM D1559 (Test Method for Resistance of Plastic Flow of Bituminous Mixtures Using Marshall Apparatus) to prepare samples with the similar characteristics to Marshall Samples considering the air void percentage and the revolution number.

Pursuant to the AASHTO T321-07 standard, the fatigue life of mixtures was investigated by the four-point beam fatigue test [11]. Slabs with dimensions of 400\*305\*80 mm were built to extract beam samples of flexural fatigue test. By using a compactor available in IUST laboratory the slabs were compacted to the target air voids content of 4% of total volume. Three beams were cut from each slab. The final dimensions after uniformly sawing of the

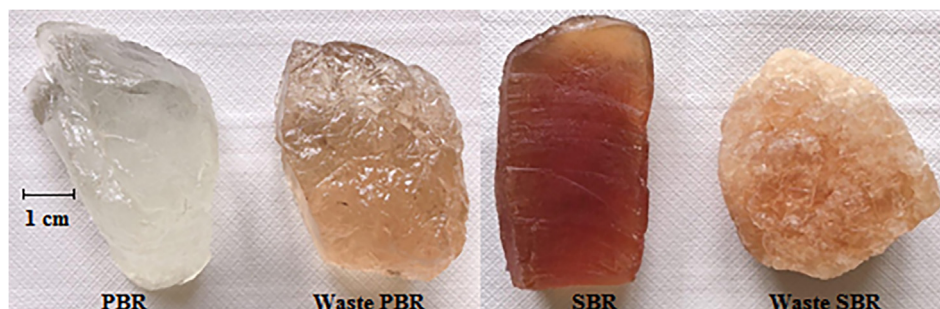


Fig. 2. Picture of original and waste of polymeric additives.

**Table 4**  
PBR1220 Typical properties.

Typical properties	Units	Values	Test Method
Mooney viscosity (ML 1 + 4 @ 100 °C)	MU	41–49	ASTM D1664
Cis Content	% wt	MIN 96	Internal Method
Volatile Material	% wt	MAX 0.75	ASTM D1416
Ash Content	% wt	MAX 0.3	ASTM D1416
<i>Typical properties-Compounds</i>			
Compound Mooney viscosity	MU	MAX 77	ASTM D1664
Tensile strength (35 Min)	kgf/cm <sup>2</sup>	MIN 150	ASTM D 412
Elongation at Break (35 Min)	%	MIN 440	ASTM D 412
<i>300% Modulus at 145 °C</i>			
25 Min	kgf/cm <sup>2</sup>	68–108	ASTM D 412
35 Min	kgf/cm <sup>2</sup>	74–114	ASTM D 412
50 Min	kgf/cm <sup>2</sup>	74–114	ASTM D 412

**Table 5**  
Marshall Properties of mixtures.

Marshall property	Results						
	Control	A	B	C	D	E	F
Modifier	–	3% SBR	3% PBR	3% Waste SBR	3% Waste PBR	5% Waste SBR	5% Waste PBR
Stability (KN)	8.33	11.91	12.28	10.62	9.78	13.26	12.36
Flow (mm)	3.24	3.87	4.04	4.18	3.51	4.29	3.84
Air voids (%)	3.63	3.29	3.88	4.08	3.86	3.97	3.88
Unit weight (kg/m <sup>3</sup> )	2.346	2.353	2.363	2.344	2.346	2.356	2.355
VMA (%)	15.887	15.726	15.817	16.489	16.379	16.066	16.086
V.F.B (%)	75.096	74.321	75.468	75.224	76.386	75.268	75.872
Optimum asphalt content (%)	4.6	5.2	5.2	5.3	5.3	5.3	5.3

specimens are  $50 \pm 6$  mm in height,  $380 \pm 6$  mm in length, and  $63 \pm 6$  mm in width. At least 6 mm from each side of beams was sawn to remove high air void and irregularity of the surface. Thereafter, specimens were kept at ambient temperatures of 20 °C. Although the air voids content of slabs was 7% of total volume, the mean air voids content of the individual beams was almost 5% for mixes because some of the voids were removed after sawing the edges.

### 3. Bitumen experiments

#### 3.1. Softening point

The softening point is the mean of the temperature values (°C) at which two 3.5 g steel balls pass through two disks of bitumen and fall through a height of 2.54 cm. This value was calculated for each type of bitumen according to ASTM D36 standard [12].

#### 3.2. Penetration

Penetration value is the vertical distance penetrated by a standard needle with a constant load (100 g) for 5 s at 25 °C. This is measured in units of 0.1 mm and is used for evaluating hardness of bitumen. This value was calculated for each type of bitumen according to ASTM D5 standard [13]. In addition, Eq. (1) is used for calculating the Penetration Index (PI) using the penetration values and the softening point.

$$PI = \frac{20(1 - 25A)}{1 + 50A} \quad (1)$$

$$A = \frac{\log V_p - \log 800}{25 - V_{sp}} \quad (2)$$

where  $PI$  is the penetration index,  $V_p$  is the penetration value at 25 °C and  $V_{sp}$  is the softening point of the bitumen.

#### 3.3. Ductility

In order to investigate the ductility of prepared bitumens, elongation before breaking is measured by this test when a specimen is pulled apart at a specified speed (5 cm/min) and temperature (25 °C). This value was calculated for each type of bitumen according to ASTM D113 standard [14].

#### 3.4. Fraass breaking point

This test is used to measure bitumen behavior at low temperatures (up to –30 °C) and determines the temperature at which bitumen reaches a critical condition and cracks. A steel plaque coated with 0.5 mm bitumen is slowly flexed and the temperature of the plaque reduces at the rate of 1 °C per minute until bitumen cracks. The point at which the bitumen cracks is the Fraass breaking point. This value was calculated for each type of bitumen according to BS EN 12593 (2015) standard [15].

#### 3.5. High-temperature storage stability

Phase separation usually happens during the pumping and storage process in modified bitumen, which is recommended to be checked. First, the binder was poured into an aluminum tube (32-mm diameter, 160-mm height). Then the tube was placed vertically in an oven at 163 °C for 48 h. Thereafter, the tube was cooled to ambient temperature and cut horizontally into three segments with the same size. The top and bottom segments were used to perform softening point test and viscosity tests at 135 °C. If the difference between the softening points is more than 2.5 °C, or if the ratio of the two samples' viscosities was below 0.9 or above 1.1, the modified binder is considered to be unstable. Otherwise, it is considered to have good stability storage in high-temperature [16,17].



## 4. Asphalt tests “fatigue”

### 4.1. Methods

Researchers have used different kinds of tests to investigate the fatigue resistance of asphalt mixtures. Among them, 4-point bending test (4 PB) is the most reliable and advantageous one and, in the second place, it is indirect tensile fatigue (ITFT), which is very common but underestimates the fatigue life of the materials because of taking the accumulation of permanent deformation into account [9,18].

In the phenomenological approach, the fatigue failure criteria is expressed as a 50 percent reduction in initial stiffness or complete fracture. It was originally defined by Van Dijk and Visser (1977), Pronk and Hopman (1990), Tayebali et al. (1992 and 1993), Abojareh et al. (2007), Shen and Lu (2011).

Flexural stiffness (S) in [Pa] is calculated using the following equations (AASHTO T321):

$$S = \frac{\sigma}{\varepsilon} = \frac{aP(3l^2 - 4a^2)}{4b\Delta h^3} \quad (3)$$

where  $\sigma$  is the tensile stress in [Pa];  $\varepsilon$ , the maximum tensile strain in [m/m]; P, the applied peak-to-peak load in [N]; a, the space between outside clamps in [m]; b, the average beam width, in [m]; h, the average beam height, in [m];  $\Delta$ , the beam deflection at neutral axis, in [m]; and l, the length of beam between outside clamps in [m] [19–23].

This model does not take the damage process into account and also cannot specify the fatigue characteristics of mixtures clearly, so it is difficult to investigate the healing process and fatigue endurance limit concept. In addition, mode of loading, materials, test temperature, and test method are the factors which affect the responses [24]. Dissipated energy is the energy loss per load cycle in any repeated or dynamic test [20,25,26]. This method can be employed when flexural beam fatigue tests are intended to be performed either with controlled stress or controlled strain mode. The stiffness, strain, phase angle and dissipated energy are the parameters which are calculated by software at each loading cycle. The dissipated energy can be determined by following equation:

Dissipated energy (DE) per cycle in [J/m<sup>3</sup>]

$$D = \pi \sigma \varepsilon \sin(360f\theta) \quad (4)$$

where f is the load frequency in [Hz];  $\theta$ , the time lag between P<sub>max</sub> and  $\delta_{max}$  in [s].

Cumulated dissipated energy (CDE) in [J/m<sup>3</sup>]:

$$W = \sum_{i=1}^{i=n} D_i \quad (5)$$

where  $D_i = D$  for the *i*th load cycle [11].

Based on previous studies (Van Dijk, 1975; Van Dijk and Vesser, 1977) [20,26], it was concluded that loading frequency, temperature, mode of loading and rest times do not have significant effects on the total dissipated energy. Within other projects by SHRP study (SHRP, 1994; Fakhri, 1999) [25,27], a unique relationship between the number of loading cycles to failure and the cumulative dissipated energy was found which was independent from loading mode and rest times; but was very dependent on mixture.

A strong correlation between fatigue life and initial dissipated energy has been shown by Baburamani and Porter (1996) [28]. Pronk and Hopman (1991) [19] suggested that the dissipated energy per cycle/period is inducing fatigue damage. Tayebali et al. [23] discovered a connection between the stiffness ratio at load cycle (i) to the initial stiffness and the dissipated energy ratio

independent of the mixture type, and also, it is recommended to conduct fatigue tests, because there are some factors which the cumulative dissipated energy versus number of cycles relationship is dependent on, such as the test condition and mixture type.

The energy – fatigue life relationship as one of the dissipated energy approaches, considers all the dissipated energy that represents the total damage being done to the specimen, while there should be a dissipated energy difference between two successive cycles, to make a crack start growing. Also, this relationship is dependent on the mode of loading and materials. Therefore, for each mode of loading or material, there should be an individual relationship [22]. In fact, cracks propagate by just a part of the dissipated energy and cumulative dissipated energy does not consider this fact completely. So, this method only can partially specify the amount of the damage done to the material [29].

The Dissipated Energy Ratio (DER) was taken one step forward by Ghuzlan (2001) [29] and Carpenter et al. (2000), (2003) [30,31]. They recommended and verified the Ratio of Dissipated Energy Change (RDEC) concept as a more precise approach for investigating mixture fatigue life. This ratio (RDEC) is calculating the dissipated energy change between two successive cycles and represents the new parameter called Plateau Value (PV). It also has a relationship with the number of loading cycles to failure (PV-Nf), which is independent of test condition, mode of loading (controlled strain or stress) and materials.

#### 4.1.1. Ratio of dissipated energy change

The RDEC determines the part of the dissipated energy which is responsible for crack producing and is attributable to damage from one cycle to another. This ratio can be obtained by using the following equation:

$$RDEC = \frac{(DE_{n+1} - DE_n)}{DE_n} \quad (6)$$

where:

- RDEC = the Ratio of Dissipated Energy Change
- $DE_n$  = the dissipated energy produced in load cyclen , and
- $DE_{n+1}$  = the dissipated energy produced in load cyclen + 1.

The damage curve represented by RDEC versus loading cycles, can be distinctively divided into three stages as shown in Fig. 3 [32]. In the first stage the level of damage is high, however during a long second stage, the damage level remains low, which the plateau value (PV) is usually considered at the point of 50% reduction in the initial stiffness modulus of the specimen, and finally in the third stage a high level of RDEC increase happens again until the fatigue failure. In this method, the point at which the specimen enters the third phase is considered as the failure point [31].

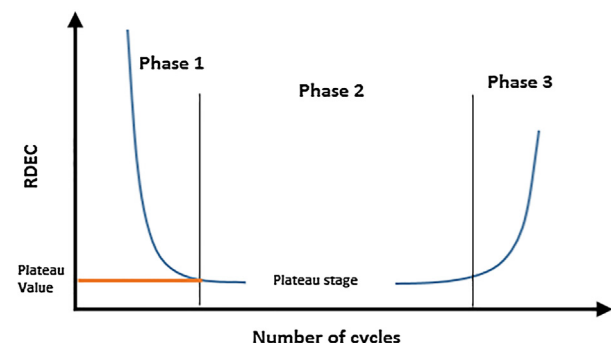


Fig. 3. Schematic diagram of RDEC versus load repetition [32].

#### 4.1.2. Energy ratio method

The energy ratio method was improved by proposing a new concept which is the same for both modes of loading (controlled strain or stress), represented by Rowe and Boulidin (2000) [33]. Based on this method, fatigue life can be determined by finding the peak value of the energy ratio of cycles versus the number of load cycles plot. It is found that this peak point is in a range between 35 and 65 percent of the initial stiffness modulus. The energy ratio is defined as the following equation:

$$ER = n_i S_i \quad (7)$$

where  $S_i$  is the stiffness at cycle (i) and  $n_i$  is the number of cycle (i) [34].

#### 4.2. Four point bending test

The Four Point Bending test (4 PB) has been investigated on all samples beams. The tests were run in the controlled strain mode and conducted in a test chamber at  $20 \pm 1^\circ\text{C}$ . Before each test, the beams were placed into the chamber for at least 4 h. All the specimens were exposed to sinusoidal loading pattern and at a frequency of loading of 10 Hz.

The criteria for ending the test was reaching 50% of the initial stiffness which is defined as the failure point in the standard [11]. On the other hand, to investigate the dissipated energy method parameters, the tests were continued to reach 35% of this value. The beam fatigue apparatus is shown in Fig. 4.

The length of the middle span was 118.5 mm. The setup records several parameters per cycle, such as beam deflection at the center (mm), peak load (N), dissipated energy (kPa) and its cumulative value (MPa), phase angle (Degree), stiffness (MPa), etc.

Automatically monitored by the software, the initial value is the 50th cycle. In order to ensure that the fatigue life was within the desired range ( $10^4$  –  $10^6$  cycles), different strain levels for each mixture was carried out. Each sample was subjected to three different strain levels of 600, 800 and 1000 microstrains, and for each strain level, three specimens were tested. The selected range is stipulated, considering the national standards for this test [35].

#### 4.3. Indirect tensile fatigue test

In a control stress mode, the cylindrical specimens were subjected to a cyclic constant load equal to 3.15 and 4.2 kN (300, 400 kPa) with a haversine loading shape and a frequency equal to 2 Hz (a 0.1 s loading followed by a 0.4 s rest period) through

Universal Testing Machine (UTM). The temperatures of the tests were 15, and  $25^\circ\text{C}$  and the specimens were placed in the chamber for at least 6 h before testing. The values of stress, force and strain rate were controlled and displacements were measured through two linear variable differential transducers (LVDTs) clamped on the specimen's diametrical sides. When the slope of accumulated strain curve significantly increases, the fatigue failure is occurred and the test is done. The device is shown in Fig. 5.

## 5. Results and discussion

### 5.1. Bitumen tests

The properties of the polymer modified bitumen samples are presented in Table 6. The results of high-temperature storage stability test of Modified bitumen presented in Table 7. According to AASHTO T-316 and AASHTO T-pp5, since the difference between the softening points is less than  $2.5^\circ\text{C}$  and the ratios of the two samples' viscosities are above 0.9 and below 1.1, produced PMBs have good storage stability in high-temperature and can be used efficiently in mixtures.

### 5.2. Four point bending test (4 PB)

#### 5.2.1. Fatigue analysis

In this type of analysis, by plotting the number of cycles to failure ( $N_{f50}$ ) versus the strain levels, the fatigue line of the mixtures can be obtained. The equation of the lines can be represented by the following form:

$$N_{f50} = k_1 \left( \frac{1}{\varepsilon} \right)^{k_2} \quad (8)$$

where  $N_{f50}$  is the number of cycles to failure,  $\varepsilon$  is the initial strain ( $\times 10^{-6}$ ),  $k_1$  and  $k_2$  are coefficients determined by the regression calculation of each mix.

In Fig. 6, the *Strain level* –  $N_{f50}$  graph for each mixture is plotted. The mixtures modified by 3% SBR, 3% PBR, 5% waste SBR, and 5% waste PBR indicate approximately the same  $N_{f50}$  values, while this value decreases in the mixtures modified by 3% waste SBR, 3% waste PBR, and the unmodified one.

Fig. 6 is illustrating each samples fatigue life in different applied strain levels. As this figure presents, the mixture A, modified by 3% SBR (by weight of the bitumen), shows the best fatigue resistance, followed by the mix B and after that the mixture (E) including 5% waste of SBR (by weight of the bitumen). Moreover, the trend of

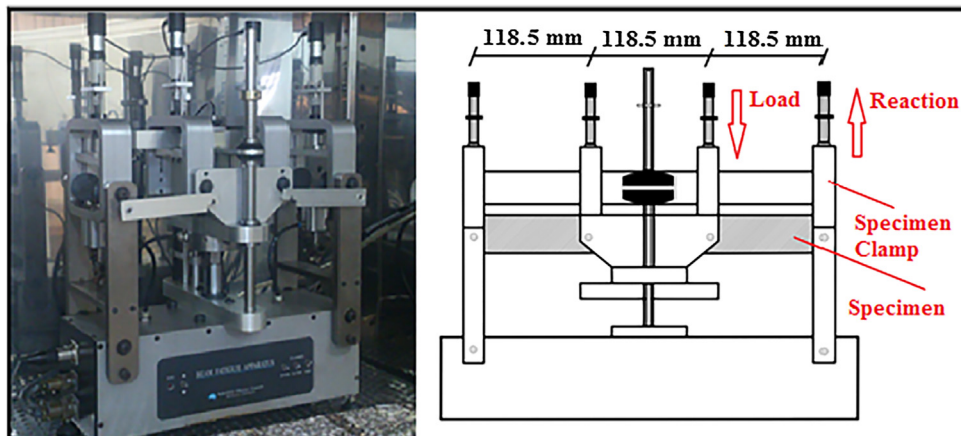


Fig. 4. Four point Beam fatigue test apparatus.

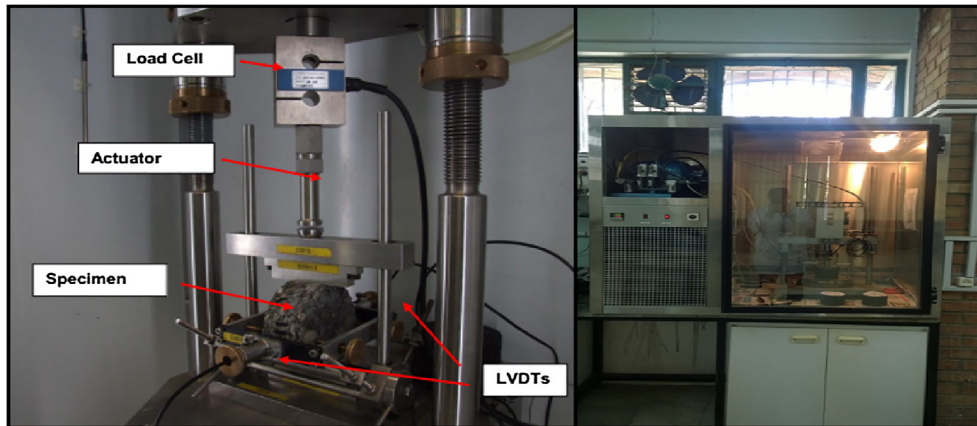


Fig. 5. Indirect tensile fatigue test setup with UTM.

Table 6

Properties of the polymer modified bitumen samples.

Sample name	Additive	Softening point (°C)	Penetration@ 25 °C (0.1 mm)	Penetration index (PI)	Ductility @25 °C (cm)	Fraass point (°C)	Specific Gravity (g/cm <sup>3</sup> )
Control	–	49	63	–0.908	112	–6	1.014
A	3% SBR1502	61	49	1.175	148	–12	0.998
B	3% PBR1220	59	53	0.975	160	–14	1.005
C	3% waste SBR1502	52	61	–0.228	121	–8	0.998
D	3% waste PBR1220	51.5	59	–0.435	130	–8	1.005
E	5% waste SBR1502	60.5	50	1.128	137	–11	0.991
F	5% waste PBR1220	58	54	0.816	142	–12	0.999

Table 7

Modified bitumen's storage stability test results.

sample	Softening point (°C)		S(t)-S(b)	Viscosity at 135 °C		V(t)/V(b)
	S(t)	S(b)		top	bottom	
A	62.2	60.8	1.4	0.895	0.866	1.0334873
B	59.5	58	1.5	0.886	0.838	1.057279
C	52.8	51.2	1.6	0.84	0.788	1.06599
D	52.5	51.3	1.2	0.834	0.816	1.022059
E	61	59.5	1.5	0.936	0.889	1.052868
F	59.5	58.1	1.4	0.914	0.851	1.074031

the graphs shows that the fatigue resistance is increasing as a result of polymers' waste addition up to 5% (by weight of the bitumen). Similarly, for the asphalt mix with 5% waste PBR (by weight of the bitumen), it was found that there was an increase in fatigue life.

Fig. 7 represents the mean initial stiffness for each mix, which was recorded at the 50th loading cycle.

In Fig. 7, it can be seen that at a strain level of 600 microstrain, the stiffness of the control mix is 21% lower than the E-WSBR-5 mix. This means that in constant strain mode, the stress applied to the control mix is 21% higher than the E-WSBR-5 mix. This concept is one of the difficulties which makes the comparison complicated. Also, a reduction in level of strain for example from 800 microstrain to 600 microstrain, resulted in an increase in stiffness modulus. The stiffness modulus of mixture E-WSBR5 interestingly decreased which shows a more flexible type of binder. Lower stiffness in the 4 PB test for A-SBR-3 mix were similarly obtained. However it is noted by J. Kent Newman that in a 14 mm stone mastic asphalt (SMA) mix including 5% SBR (by weight of the bitumen) there is no considerable effect on initial flexural modulus [36].

### 5.2.2. Ratio of dissipated energy change (RDEC)

The RDEC method was used for each sample of the 4 PB test. The results of A-800 sample is shown in Fig. 8. Since there is a significant variation in the RDEC responses, researchers have difficulties in determining the Plateau-Value at the conventional failure point, i.e.  $N_f50$  [37].

In control strain mode, the dissipated energy per cycle decreases during the test, while in control stress mode, this parameter increases. The RDEC-N graphs for each of the samples were plotted. It is found that the mix with 5% waste SBR has the best fatigue life and the mix with 5% waste PBR ranked fourth after the mixtures A-SBR3% and B-PBR3%, respectively, with acceptable values.

### 5.2.3. Plateau value

In this research, a loading cycle interval of 100 cycles was used to calculate PV for each specimen. It is found that a higher strain level results in an increase in the Plateau Value, while the mixtures with less fatigue life represent larger magnitudes of the PV. The power curves of PV versus  $N_f$  using a log-log coordinate system

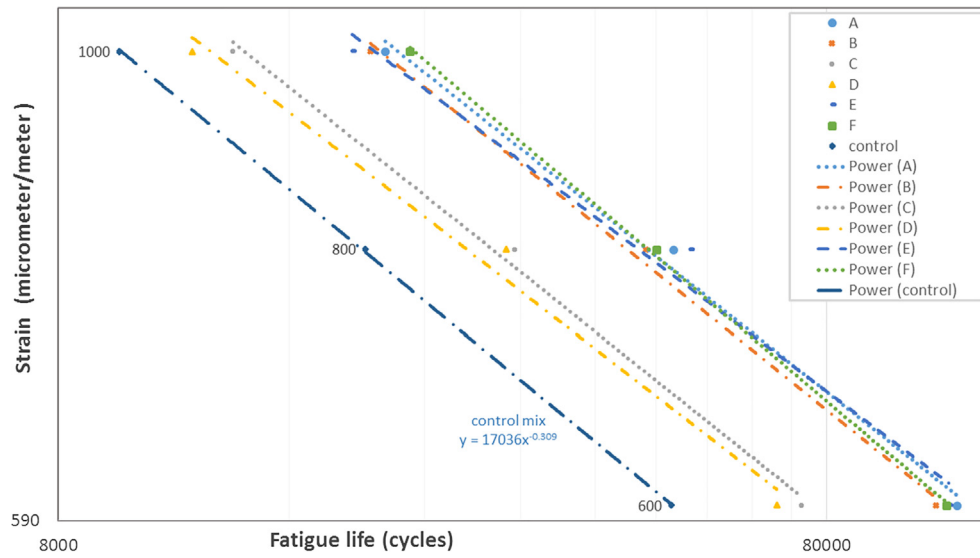


Fig. 6. Comparison of fatigue test results for A, B, C, D, E, F, and control mixtures.

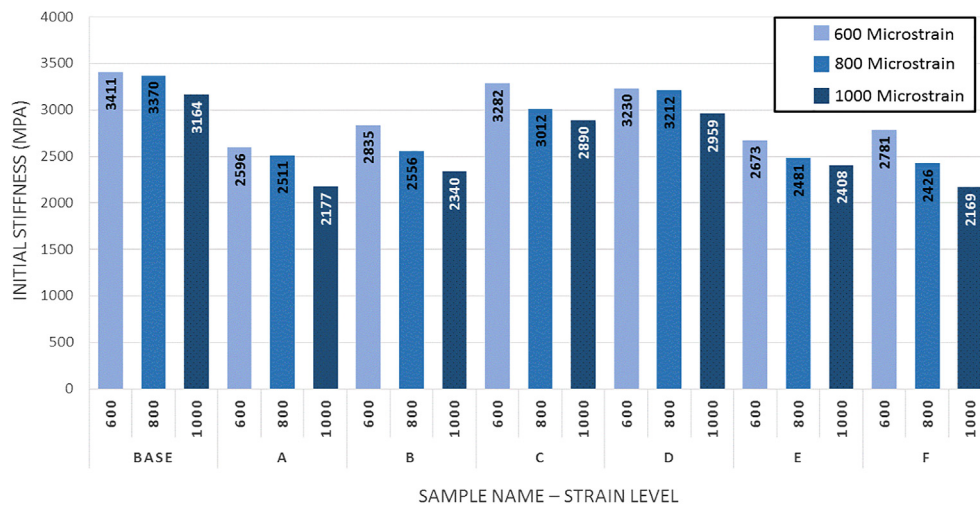


Fig. 7. Mean initial stiffness.

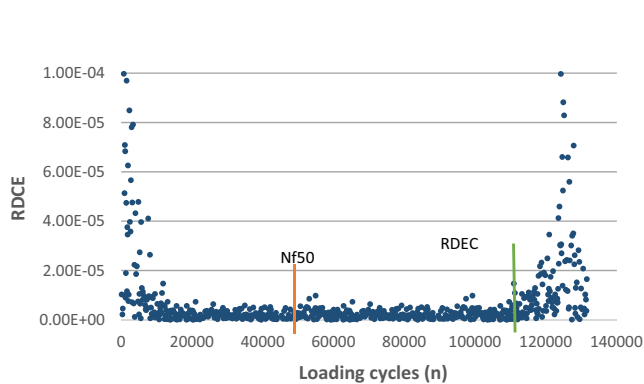


Fig. 8. RDEC versus the number of cycles for A-800 sample.

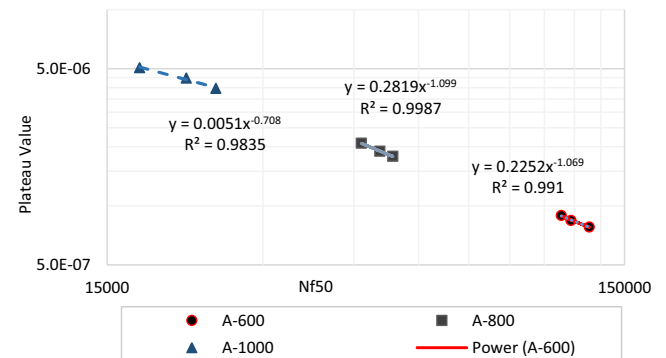


Fig. 9. Comparison of plateau value with  $N_{f50}$  for F-WPBR5 at 600, 800, and 1000 microstrains.



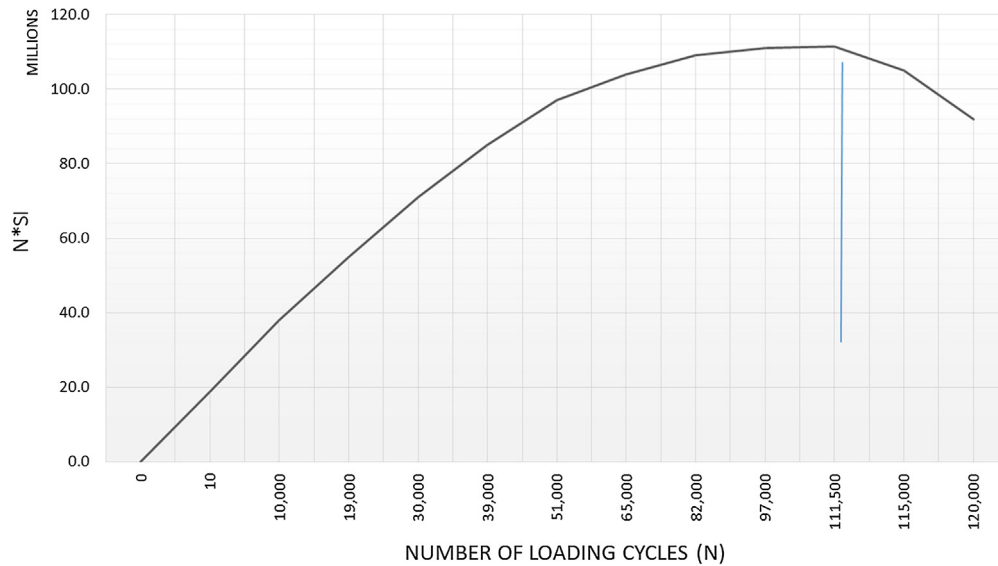


Fig. 10. Energy Ratio ( $N \times S_i$ ) versus Number of Load Cycles Using Rowe and Bouldin Method (Modified Sample A – 3% SBR at 800 microstrain).

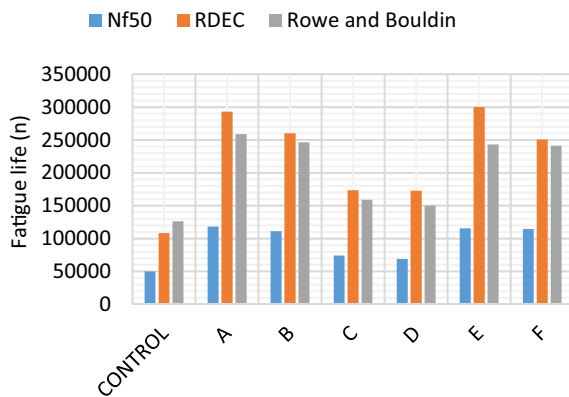


Fig. 11. The fatigue life of the mixtures at 600 ( $\mu$ s) strain level.

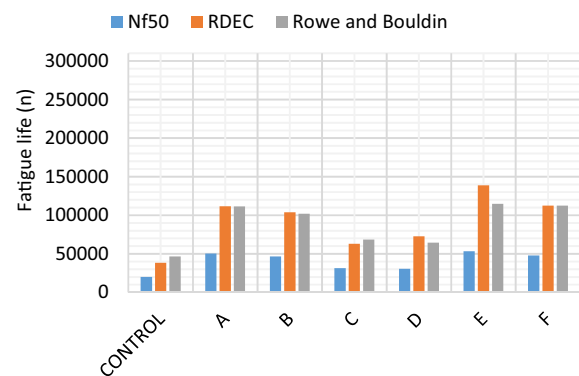


Fig. 12. The fatigue life of the mixtures at 800 ( $\mu$ s) strain level.

are depicted for F-WPBR5each mixture in Fig. 9.  $R^2$  value for each fitted line shows a very good correlation ( $>0.98$ ). The results for other mixtures are similar.

#### 5.2.4. Rowe and Bouldin energy ratio method

The  $NS_i - N$  graphs for A-3% SBR at the strain level of 800 microstrains is shown in Fig. 10.

#### 5.2.5. Statistical analysis

Statistical analysis including analysis of variance (ANOVA) with post-hoc Tukey HSD Test was performed using the SPSS software. The primary variables are the binder sources (A, B, C, D, E, F and Control), the strain level (600, 800 and 1000) and the analysis methods ( $N_{f50}$ , RDEC, and Rowe). ANOVA was first conducted to determine whether or not significant differences among the sample means exist. In this study, the level of significance was 0.05 ( $\alpha = 0.05$ ). Upon understanding that there were differences among sample means using the ANOVA, the HSD was performed. After that, all pairs of sample means were compared [38].

For comparison purposes, the fatigue life obtained from different approaches at different strain levels are presented in

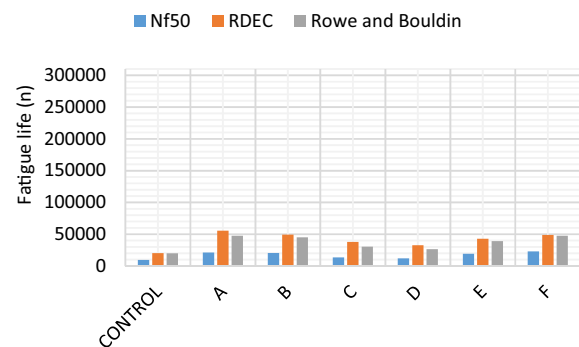


Fig. 13. The fatigue life of the mixtures at 1000 ( $\mu$ s) strain level.

Figs. 11–13. As shown in Figs. 11–13 and according to Tables 9 and 10 for RDEC method and Rowe's method, respectively, they result in significantly greater fatigue life for mixtures than  $N_{f50}$  method. The differences are bigger especially in lower strain levels.

**Table 8**

Anova Pairwise Tukey statistical analysis for fatigue life according to Rowe and Bouldin method.

ANOVA statistical analysis								
		Sum of Squares	Df	Mean Square	F	Sig		
600 Microstrain	Between Groups	5.608E+10	6	9347119286	666.988	0.000		
	Within Groups	196195000.0	14	14013928.57				
	Total	5.628E+10	20					
800 Microstrain	Between Groups	1.417E+10	6	2362427143	315.863	0.000		
	Within Groups	104710000.0	14	7479285.714				
	Total	1.428E+10	20					
1000 Microstrain	Between Groups	2205235714	6	367539285.7	64.404	0.000		
	Within Groups	79895000.0	14	5706785.714				
	Total	2285130714	20					
Pairwise Tukey statistical analysis								
		Control	A	B	C	D	E	F
600 Microstrain	Control	–	S	S	S	S	S	S
	A		–	S	S	S	S	S
	B			–	S	S	N	N
	C				–	N	S	S
	D					–	S	S
	E						–	N
	F							–
800 Microstrain	Control	–	S	S	S	S	S	S
	A		–	S	S	S	N	N
	B			–	S	S	S	S
	C				–	N	S	S
	D					–	S	S
	E						–	N
	F							–
1000 Microstrain	Control	–	S	S	S	N	S	S
	A		–	N	S	S	S	N
	B			–	S	S	N	N
	C				–	N	S	S
	D					–	S	S
	E						–	S
	F							–

Notes: a = 0.05, N: Not significance, S: Significance.

**Table 9**

Anova Pairwise Tukey statistical analysis for comparison between the three methods used for SBR modified samples.

ANOVA statistical analysis													
Pairwise Tukey statistical analysis													
		Control			A			C			E		
		Nf50	RDEC	Rowe	Nf50	RDEC	Rowe	Nf50	RDEC	Rowe	Nf50	RDEC	Rowe
Control	Nf50	–	S	S	S	S	S	S	S	S	S	S	S
	RDEC		–	S	S	S	S	S	S	S	N	S	S
	Rowe			–	N	S	S	S	S	S	S	S	S
A	Nf50				–	S	S	S	S	S	N	S	S
	RDEC					–	S	S	S	S	S	N	S
	Rowe						–	S	S	S	S	S	S
C	Nf50							–	S	S	S	S	S
	RDEC								–	S	S	S	S
	Rowe									–	S	S	S
E	Nf50										–	S	S
	RDEC											–	S
	Rowe												–

Notes: a = 0.05, N: Not significance, S: Significance.

As it has been shown in Figs. 11–13 and with respect to pairwise Tukey statistical analysis presented in Table 8 fatigue life of mixes modified by the waste of polymers, is significantly higher than unmodified mixes, considerably close to mixes including original polymers. Among all mixes, 5% waste SBR modified mix has the most fatigue life followed by 3% SBR modified mix.

### 5.3. Indirect tensile fatigue test (ITFT)

#### 5.3.1. Results of indirect tensile fatigue test (UTM)

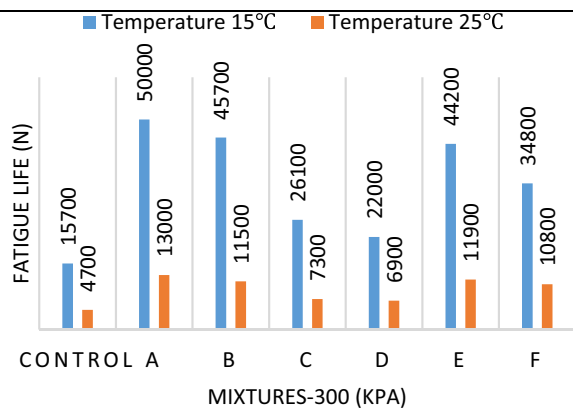
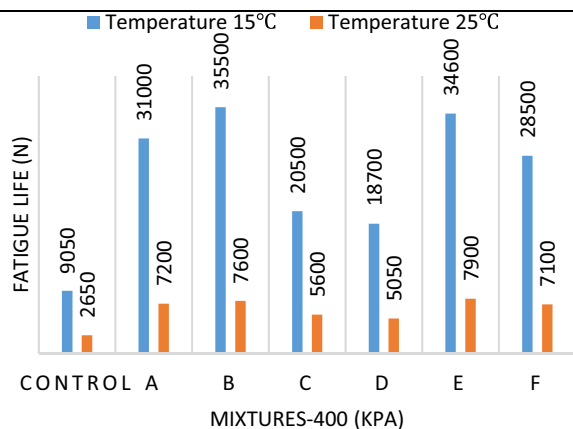
The indirect tensile fatigue test was performed using the universal testing machine (UTM) and the fatigue lives of samples were determined at different loading magnitudes (3.15, 4.2 KN) and dif-

**Table 10**

Anova Pairwise Tukey statistical analysis for comparison between the three methods used for PBR modified samples.

ANOVA statistical analysis Pairwise Tukey statistical analysis													
		Control			B			D			F		
		Nf50	RDEC	Rowe	Nf50	RDEC	Rowe	Nf50	RDEC	Rowe	Nf50	RDEC	Rowe
Control	Nf50	–	S	S	S	S	S	S	S	S	S	S	S
	RDEC		–	S	N	S	S	S	S	S	N	S	S
	Rowe			–	S	S	S	S	S	S	S	S	S
B	Nf50				–	S	S	S	S	S	N	S	S
	RDEC					–	S	S	S	S	S	N	S
	Rowe						–	S	S	S	S	N	N
D	Nf50							–	S	S	S	S	S
	RDEC								–	S	S	S	S
	Rowe									–	S	S	S
F	Nf50										–	S	S
	RDEC											–	N
	Rowe												–

Notes: a = 0.05, N: Not significance, S: Significance.

**Fig. 14.** The fatigue life of the mixtures at 300 (kPa).**Fig. 15.** The fatigue life of the mixtures at 400 (kPa).

ferent temperatures (15 °C and 25 °C). The test results are given in Figs. 14 and 15. At stress level of 300 (kPa), 3%SBR have the highest fatigue life followed by 3%PBR and 5%waste PBR, respectively, while, by increasing the stress level, 3%PBR and 5%waste PBR result in better fatigue performance.

### 5.3.2. Statistical analysis

Table 11 presents the statistical results of comparison between  $N_f$  values for ITFT in four different test conditions and 4 PB test in

three strain levels (Rowe and Boulidin method). It can be seen that the tests' results have a good correlation. The best correlation is between T25 °C-S300 kPa and strain level 600  $\mu$ s. Also, the ITFT's results can be used for fatigue resistance comparison of different mixtures in a high level of reliability.

## 6. Conclusion

The aim of this study was to evaluate the impact of introduction of polymer's waste into unmodified binder on fatigue life. Since the residual waste of polymer production process dump with no specific application and because of the high price of polymers as well, substituting polymers by waste of rubbers mitigates environmental damages and preserves natural resources at very low cost. Results of performing conventional bitumen tests, indirect tensile fatigue and four-pointed beam fatigue tests on modified asphalt binders and mixtures by combination of polymers and waste polymers and calculating P-values of them using different methods indicate:

- The waste of polymer production process grants the same benefits as the virgin polymers in terms of classical bitumen properties and fatigue characteristics. So, when they are used by 5% of bitumen content, they have the potential to be suitable alternatives at lower price.
- Among waste of elastomers used in the modification of bitumen, the waste SBR with the optimum ratio of 5% improved the performance the most. The Penetration index (PI) of binder E is 1.128 which is higher than the others. Also, it results in the least penetration grade (50) with the second highest softening point (60.5 °C) among the modified binders. In addition, binder E has acceptable ductility and Fraass Point in comparison with all other binders.
- In 4 PB test, at strains levels of 600 and 800 ( $\mu$ s) and based on RDEC approach, 5% waste PBR shows the most fatigue resistance, followed by 3%SBR. Moreover, at strain level of 1000 ( $\mu$ s), 3%SBR resulted in the best result followed by 3%PBR.
- The problem with conventional fatigue test that makes the comparison difficult is the mixture's fatigue life dependency on the stiffness, while the stiffness is highly under the influence of strain level. So, in order to have more precise comparison, two other alternative methods, energy methods and energy ratio were conducted. The energy ratio method ranked the mixtures with regard to fatigue performance as A, B, E, and F,

**Table 11**

Bivariate correlations analysis between the results of ITFT and 4 PB tests.

Correlations		strain 600	Strain 800	Strain 1000	T15-S300	T15-S400	T25-S300	T25-S400
Strain 600	Correlation Coefficient	1	0.981	0.966	0.959	0.943	0.987	0.925
	Sig.		0.000	0.000	0.001	0.001	0.000	0.003
Strain 800	Correlation Coefficient		1	0.944	0.916	0.937	0.972	0.945
	Sig.			0.001	0.004	0.002	0.000	0.001
Strain 1000	Correlation Coefficient			1	0.896	0.891	0.943	0.896
	Sig.				0.006	0.007	0.001	0.006
T15-S300	Correlation Coefficient				1	0.944	0.981	0.908
	Sig.					0.001	0.000	0.005
T15-S400	Correlation Coefficient					1	0.952	0.984
	Sig.						0.001	0.000
T25-S300	Correlation Coefficient						1	0.941
	Sig.							0.002
T25-S400	Correlation Coefficient							1
	Sig.							

Notes: T: Temperature, S: Stress.

respectively. However, in higher strain level (1000  $\mu$ s), the responses of all tests are in a same range, and the mixture F is as good as the mix A.

- Although ITFT underestimates the fatigue life, there is a good consistency between the results of ITFT and 4 PB test as proven by the bivariate correlation analysis.
- The traditional approach ( $N_{f50}$ ) for 4 PB and ITFT tests are reliable enough to be used for comparing different mixtures.

### Conflicts of interest statement

None.

### References

- [1] M.M.J. Jacobs, Crack Growth in Asphaltic Mixes (doctoral dissertation), Delft University of Technology, TU Delft, 1995.
- [2] Y. Yildirim, Polymer modified asphalt binders, *Constr. Build. Mater.* 21 (1) (2007) 66–72.
- [3] H. Yaacob, M. Ali Mughal, R.P. Jaya, M.R. Hainin, D.S. Jayanti, C.N. Che Wan, Rheological properties of styrene butadiene rubber modified bitumen binder, *J. Teknologi* 78 (2016) 121–126.
- [4] M. Ameri, M. Vamegh, H. Rooholamini, F. Haddadi, Investigating effects of nano/SBR polymer on rutting performance of binder and asphalt mixture, *Adv. Mater. Sci. Eng.* 2018 (2018).
- [5] Routes/Roads, Modified Binders, Binders with Additives and Special Bitumens, The World Road Association (PIARC), 1999.
- [6] M. Garcia-Morales, P. Partal, F.J. Navarro, C. Gallegos, Effect of waste polymer addition on the rheology of modified bitumen, *Fuel* 85 (7–8) (2006) 936–943.
- [7] Code-234, Iran Highway Asphalt Paving Code, Ministry of Road and Transportation Research and Education Center, Tehran, Iran, 2011.
- [8] M. Ameri, M. Vamegh, R. Imaninasab, H. Rooholamini, Effect of nanoclay on performance of neat and SBS-modified bitumen and HMA, *Pet. Sci. Technol.* 34 (11–12) (2016) 1091–1097.
- [9] M. Ameri, S. Nobakht, K. Bemana, H. Rooholamini, M. Vamegh, Effect of nanoclay on fatigue life of hot mix asphalt, *Pet. Sci. Technol.* 34 (11–12) (2016) 1021–1025.
- [10] E.T. Harrigan, R.B. Leahy, J.S. Youtcheff, The Superpave Mix Design System: Manual of Specifications, Test Methods and Practices, SHRP-A-379, Strategic Highway Research Program, National Research Council, Washington, D. C., 1994.
- [11] AASHTO T321-07, Determining the Fatigue Life of Compacted Hot Mix Asphalt (HMA) Subjected to Repeated Flexural Bending, American Association of State and Highway Transportation Officials, Washington, DC, USA, 2007.
- [12] ASTM D36, Standard Test Method for Softening Point of Bituminous Materials. Annual Book of ASTM Standards USA, 1992.
- [13] ASTM D5, Standard Test Method for Penetration of Bituminous Materials. Annual Book of ASTM Standards USA, 1992.
- [14] ASTM D113, Standard Test Method for Ductility of Bituminous Materials. Annual Book of ASTM Standards USA, 2007.
- [15] British Standard Institution BS EN 12593, Bitumen and Bituminous Binders. Determination of the Fraass Breaking Point, 2015.
- [16] M. Ameri, R. Mohammadi, M. Vamegh, M. Molayem, Evaluation the effects of nanoclay on permanent deformation behavior of stone mastic asphalt mixtures, *Constr. Build. Mater.* 156 (2017) 107–113.
- [17] P. Aashto, The Laboratory Evaluation of Modified Asphalt Systems, American Association of State Highway and Transportation Officials, Washington, 1993.
- [18] J.M. Matthews, C.L. Monismith, J. Craus, Investigation of laboratory fatigue testing procedures for asphalt aggregate mixtures, *J. Transp. Eng.* 119 (4) (1993) 634–654.
- [19] A.C. Pronk, P.C. Hopman, Energy Dissipation: The Leading Factor of Fatigue, 1991.
- [20] W. Van Dijk, W. Visser, Energy Approach to Fatigue for Pavement Design, in: Association of Asphalt Paving Technologists Proc, vol. 46, 1977.
- [21] M.A. Witczak, M.W. Mamlouk, M.S. Kaloush, K.E. Kaloush, Validation of initial and failure stiffness definitions in flexure fatigue test for hot mix asphalt, *J. Test. Eval.* 35 (1) (2006) 95–102.
- [22] S. Shen, X. Lu, Energy based laboratory fatigue failure criteria for asphalt materials, *J. Test. Eval.* 39 (3) (2010) 313–320.
- [23] A.A. Tayebali, G.M. Rowe, J.B. Sousa, Fatigue response of asphalt-aggregate mixtures (with discussion), *J. Assoc. Asphalt Paving Technol.* (1992) 61.
- [24] S. Shen, G.D. Airey, S.H. Carpenter, H. Huang, A dissipated energy approach to fatigue evaluation, *Road Mater. Pavement Des.* 7 (1) (2006) 47–69.
- [25] SHRP A404, Fatigue Response of Asphalt-aggregate Mixes. Strategic Highway Research Program, National Research Council, 1994.
- [26] W. Van Dijk, Practical fatigue characterization of bituminous mixes, *J. Assoc. Asphalt Paving Technol.* 44 (1975) 38–72.
- [27] M. Fakhri, Characterisation of Asphalt Pavement Materials, 1999.
- [28] P.S. Baburamani, D.W. Potter, Dissipated energy approach to fatigue characteristics of asphalt mixes, in: Combined 18th ARRB Transport Research Conference and Transit New Zealand Land Transport Symposium, 2–6 September 1996, Christchurch, New Zealand Part 2, 1996.
- [29] K.A. Ghuzlan, Fatigue Damage Analysis in Asphalt Concrete Mixtures Based Upon Dissipated Energy Concepts (Doctoral dissertation), University of Illinois at Urbana-Champaign, 2001.
- [30] K. Ghuzlan, S. Carpenter, Energy-derived, damage-based failure criterion for fatigue testing, *Transport. Res. Record J. Transport. Res. Board* 1723 (2000) 141–149.
- [31] S. Carpenter, K. Ghuzlan, S. Shen, Fatigue endurance limit for highway and airport pavements, *Transport. Res. Record J. Transport. Res. Board* 1832 (2003) 131–138.
- [32] S. Shen, S. Carpenter, Application of the dissipated energy concept in fatigue endurance limit testing, *v 1929 (2005)* 165–173.
- [33] G.M. Rowe, M.G. Bouldin, Improved Techniques to Evaluate the Fatigue Resistance of Asphaltic Mixtures, in: 2nd Eurasphalt & Eurobitume Congress Barcelona, vol. 2000, 2000.
- [34] Transportation Officials, Standard Specifications for Transportation Materials and Methods of Sampling and Testing, AASHTO, 2011.
- [35] Standard, B. (2012). Bituminous Mixtures-Test Methods for Hot Mix Asphalt. Part. 24, 12697–24.
- [36] J.K. Newman, Flexural beam fatigue properties of airfield asphalt mixtures containing styrene-butadiene based polymer modifiers, in: Sixth International RILEM Symposium on Performance Testing and Evaluation of Bituminous Materials, RILEM Publications SARL, 2003, pp. 357–363.
- [37] S. Carpenter, S. Shen, Dissipated energy approach to study hot-mix asphalt healing in fatigue, *Transport. Res. Record J. Transport. Res. Board* 1970 (2006) 178–185.
- [38] H. Rooholamini, R. Imaninasab, M. Vamegh, Experimental analysis of the influence of SBS/nanoclay addition on asphalt fatigue and thermal performance, *Int. J. Pavement Eng.* (2017) 1–10.
- [39] A.A. Yousefi, Rubber-modified bitumens, *Iran. Polym. J.* (2002) 303–309.
- [40] J. Zhang, J. Wang, Y. Wu, W. Sun, Y. Wang, Thermal behaviour and improved properties of SBR and SBR/natural bitumen modified bitumens, *Iran. Polym. J.* 18 (6) (2009) 465–478.