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Research Paper

Evaluation of the Effect of Cellulose Base Mixture on the Behavior of Rheology and Performance of Bitumen

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Abstract

Bitumen and asphalt mixture, cellulose fibers have attracted more attention of researchers due to their lower price and availability; the purpose of this research is to investigate the possibility of using cheap waste materials from paper factories to replace expensive modifiers, which can increase the life of asphalt pavement and reduce the need for its frequent repairs . In this research, the physical properties of the bitumen modified with cellulose fibers and black liquor have been investigated. The results showed that the increase in stiffness of the bitumen containing cellulose micro fibers and liquor. The increase in bitumen stiffness has decreased the degree of penetration and increased the softening point in the modified bitumen. The viscosity of the modified bitumen also increased with increasing stiffness. The results also showed the improvement of the high-temperature performance of all the samples modified with cellulose micro fibers and liquor. However, the improvement of the low temperature of the modified bitumen is limited to the use of 4 to 6% cellulose microfibers. Increasing the resistance to rutting of the modified bitumen showed the positive effect of using cellulose micro-fibers in order to modify the properties of the base bitumen; Therefore, by using the wastes of paper factories, it is possible to achieve favorable results in reducing the common failures of asphalt pavements, including rutting at high temperature and cracking at low temperature, and reducing the costs of modifying bitumen and asphalt mixtures.

Keywords: cellulose fibers, black liquor, rheological properties, dynamic shear rheometer test, bending beam rheometer test

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1. Introduction

As one of the most widely used human and goods transportation infrastructures around the world, roads play an essential role in the economic development of any region (Banerjee et al., 2020). Today, the good quality of road construction and implementation can play an effective role in reducing travel time, vehicle maintenance costs, and also road repair and maintenance costs (Small et al., 2012).

At present, asphalt pavements constitute a significant part of road pavements. This type of pavement consists of a combination of aggregates and bitumen. Despite the share of 4 to 7% of bitumen in the asphalt mixture, this substance plays an essential role in the physical and chemical properties of the asphalt mixture as well as its durability and stability. Therefore, the quality of asphalt pavement depends on the used bitumen and the use of appropriate bitumen can have a significant effect on the resistance and reduction of asphalt pavement damages such as cracking, rutting and etc. For this, a lot of research has been done to improve the properties of bitumen in order to reduce the destructive effect of atmospheric factors and also the effect of successive loadings caused by the traffic of vehicles on asphalt pavements (Mashaan et al., 2021).

The purpose of adding fibers to bitumen is to increase the tensile strength of bitumen and, as a result, to increase the ability of bitumen to absorb strain energy during the fatigue and failure process. This action makes the bitumen able to withstand more tensile forces and, as a result, show better performance in the face of fatigue and failure conditions (Mahrez et al., 2003).

Both natural and synthetic fibers have been used in the research conducted to modify asphalt mixtures. The use of fibers in granular mastic asphalt strengthens and hardens the asphalt mixture, and chemical reaction does not occur by adding the fibers. Among the advantages of using fibers in asphalt pavement, we can mention the reduction of fatigue and thermal cracks, reduction of reflective cracks and economic benefits due to the increase of the useful life of asphalt pavement modified with fibers (Mahrez & Karim, 2007).

In a study, Wu and his colleagues (2007) studied the asphalt mixtures modified with three types of cellulose, polyester and mineral fibers (with values of 0.3%, 0.3% and 0.4%, respectively) by dynamic modulus test. By examining the dynamic modulus test results of the asphalt including the dynamic modulus and phase angle, they concluded that the asphalt mixture modified with fibers has a higher dynamic modulus compared to the unmodified asphalt mixture. Also, the resistance to fatigue and rutting is improved in asphalt mixture modified with fibers (S. Wu et al., 2007).

In a research conducted by Jenq and his colleagues, the fracture mechanics method was used to investigate the effect of fibers on the resistance to crack growth in asphalt mixture (Jenq et al., 1993). For this purpose, asphalt mixtures modified with two types of polyester and polypropylene fibers were evaluated in terms of modulus of elasticity, fracture energy and tensile strength. The results of this study show an increase in stiffness with a 50-100% increase in fracture energy, while the increase in fracture energy has little effect on elasticity and tensile strength. An increase in the rate of fracture stiffness parameter can indicate a higher resistance of the modified asphalt mixture to the applied loads and a higher resistance to crack growth (Cleven, 2000).

In a study, Simpson and Mahboob (1994) investigated asphalt mixtures modified with fibers and polypropylene and polyester polymers by conducting Marshall tests, indirect tensile strength, moisture and freezing sensitivity, modulus of elasticity and rutting. In their research, asphalt mixtures modified with polypropylene fibers showed higher tensile strength and cracking resistance. However, polypropylene fibers did not have an effect on improving resistance to moisture and damage caused by freezing and thawing. Also, unlike the asphalt mixtures modified with polymers and polyester fibers, the asphalt mixture containing polypropylene fibers did not have a problem in terms of thermal cracking. The results of the modulus of elasticity test showed that the asphalt mixture modified with polypropylene fibers had more stiffness and the reduction of rutting was observed only for the sample modified with polypropylene fibers (Cleven, 2000; Simpson & Mahboub, 1994).

In past researches, the use of cellulose fibers to modify the properties of bitumen has reduced the degree of penetration and increased the softening point of bitumen (Mohammed et al., 2018; M.-M. M. Wu et al., 2015). In the research conducted by Mohammad and his colleagues, the penetration degree of bitumen modified with glass fibers and cellulose fibers has decreased; however, glass fibers have a greater effect on reducing bitumen penetration. They found the shorter length of cellulose fibers than glass fibers to be effective in their less effect on reducing the degree of penetration (Mohammed et al., 2018).). Reduction of the penetration degree of bitumen modified with cellulose fibers has increased its softening point; therefore, the use of cellulose fibers can improve the properties of bitumen at high temperature. As the fibers are dispersed in the bitumen and the bitumen is absorbed by the fibers, due to the creation of a three-dimensional network in the bitumen, a strong binding force is created between the surface of the fibers and the bitumen; Therefore, by maintaining this structure at high temperature, the properties of bitumen containing fibers are also improved at high temperature (Mohammed et al., 2018; M.-M. M. Wu et al., 2015); However, the effect of fibers on the properties of bitumen depends on the characteristics of the fibers including the length and type of fibers (Chen & Lin, 2005).

In the research by Wu et al., to determine the minimum amount of fibers, the results of softening point test of the bitumen modified with fibers have been used. So, considering the maximum temperature of 60°C for asphalt pavement in summer, the minimum amount of cellulose fibers has been determined as one percent based on the softening point temperature. The minimum amount of fibers used depends on the properties of pure bitumen and the type of fibers, and therefore it is not possible to determine the minimum amount of fibers necessary to modify the properties of bitumen based on the results of past researches. One of the reasons for the effect of the type of fibers on the obtained results can be the entanglement and different involvement of various types of fibers with bitumen (M.-M. M. Wu et al., 2015).

The results of past researches indicate an increase in the viscosity of bitumen modified with fibers. Consequently, increasing the viscosity and stiffness of bitumen increases resistance to rutting of the modified bitumen (Chen & Lin, 2005; Mohammed et al., 2018); Therefore, regardless of the test temperature, the use of fibers including cellulose fibers can increase the rutting parameter compared to the bitumen without additives (Mohammed et al., 2018; Shaopeng et al., 2006; M.-M. M. Wu et al., 2015); However, the increase in resistance to rutting of modified bitumen has depended on the amount of the fibers used. For example, in a research, by comparing the bitumen modified with different amount of fibers, Shaopeng and his colleagues concluded that if the fibers in the bitumen are reduced, they do not have enough interaction with each other, and the fibers work as fillers in the bitumen. Therefore, the use of a small amount of fibers has not had much effect on increasing the amount of the mixed modulus (Shaopeng et al., 2006).

In the research conducted by Wu and his colleagues, by increasing the amount of cellulose fibers used from 4 to 6%, the rutting resistance parameter of the modified bitumen shows a greater increase. Also, compared to carbon fibers and mineral fibers, cellulose fibers have had a greater effect on improving the rutting parameter. In their research, they pointed out that cellulose fibers absorb more resin and oils in bitumen, which can be the reason for increasing the hardness and thus increasing the rutting parameter in the bitumen containing cellulose fibers (M.-M. M. Wu et al., 2015). Among the other reasons for the improvement of high functional temperature of the bitumen modified with fibers, we can mention the reduction of the phase angle. According to the results of the research conducted by Mohammad and his colleagues, cellulose fibers have has a greater effect on reducing the angle than glass fibers. Among the reasons for this is the uneven surface of cellulose fibers compared to glass fibers and the formation of cellulose fibers from smaller bundles of fibers (Mohammed et al., 2018).

Liquor can be mentioned as another used additive to improve bitumen properties. Liquor contains significant amounts of lignin, which can be used as a partial replacement of bitumen (up to 25%) or as a modifier additive in asphalt pavement. At this time, lignin can be considered as one of the most abundant biopolymers on earth with the production of about 50 to 70 million tons per year, a significant part of which is burned or discarded without being reused (Boerjan & Ralph, 2003; Mandlekar et al., 2018).

According to the results of past researches, the use of lignin can improve the properties of bitumen. Bitumen containing lignin has a higher viscosity than pure bitumen. This increase is dependent on the amount of the lignin consumed, and the viscosity of bitumen increases in proportion to the amount of lignin (Sundstrom et al., 1983; Terrel & Rimsritong, 1979). As the viscosity increases, the rutting resistance of the bitumen modified by lignin also increases (McCready & Williams, 2008).

In the research conducted by Zareii et al., black liquor with different weight percentages (2, 4, 6, and 8%) was used to modify the properties of 60/70 bitumen of Isfahan Refinery, and the effect of this additive on various properties of bitumen was investigated. The results of their research indicate the improvement of the characteristics and performance of the modified bitumen at high temperature. The addition of black liquor to pure bitumen has decreased the degree of penetration, increased kinematic viscosity, increased softening point and decreased thermal sensitivity of bitumen (Zarei et al., 2017). In past researches, the improvement of bitumen properties was dependent on the amount of additive, and the use of lignin in an amount less than 3% did not affect the functional properties of bitumen. Also, an increase of more than 6% of lignin caused a significant increase in bitumen hardness and had a negative effect on the low-temperature performance of bitumen (Zahedi et al., 2020).

In another study, Wang and his colleagues investigated the rheological properties of bitumen modified with lignin in vitro. In this research, two types of bitumen with performance grade of PG 64-22 and PG 76-22 were combined with two different percentages of lignin (5 and 10% of weight) and the rheological properties of the samples were evaluated. The results of the dynamic shear rheometer test showed that increasing the amount of lignin improved the resistance to rutting and increased the high-temperature performance of bitumen. They stated that the lignin in the liquor does not just act as filler but chemically reacts with the bitumen (Wang & Derewecki, 2013).

Su et al. (2017), investigated in a study the rheological properties and anti-aging performance of the bitumen modified with lignin. They concluded that the use of lignin has increased the viscosity and the resistance to rutting of bitumen; however, the appropriate viscosity amount for mixing and densification of bitumen has been met according to the Superpave Mix Design for hot asphalt production. The results obtained from the bending beam rheometer test and the linear range sweep test showed that lignin had a small negative effect on the bitumen's low-temperature performance and fatigue resistance. So that the fatigue life of bitumen modified with lignin has been reduced especially at lower strain level (Xu et al., 2017).

Among the other reasons for using lignin as a bitumen modifier, we can mention the antioxidant property of lignin due to its polyphenolic structure and control of free radicals, which has led researchers to use this additive to improve the aging or brittleness of bitumen due to oxidation and or loss of volatile substances (Bishara et al., 2005).

According to the results of Su and his colleagues' research, lignin can help resist the formation of carbonyl functional groups in the modified bitumen after short-term and long-term aging; therefore, lignin can be used as an antioxidant to reduce the negative effects of aging in bitumen and asphalt mixture (Xu et al., 2017).

According to the important role of economic parameters and environmental issues in the evaluation of engineering projects, it is necessary to consider the economic and environmental aspects in selecting additives in addition to their effect on improving pavement performance and durability. Compared to polymer additives, the use of cellulose fibers as an additive in improving the properties of bitumen and asphalt mixture has advantages such as a more reasonable price and a lower temperature required for mixing and compaction; Therefore, in this research, the waste of paper factories, including cellulose micro-fibers obtained from pulp and liquor mills, along with their combination, has been used to modify the properties of base bitumen. The use of waste materials can be cost-effective and improve the performance characteristics of bitumen. Considering the need to build new roads and the increase in fuel costs and the pollution caused by the production and implementation of asphalt mixture, the use of waste from paper production factories can be a low-cost and suitable solution to reduce the problems and failures of asphalt pavement.

2. Methodology

2.1. Materials

In this research, in addition to the effect of additives on the modification of bitumen properties, special attention has been paid to economic aspects and environmental issues. Due to the significant volume of paper factory wastes and aiming at reusing these wastes, in this research paper pulp and liquor have been used as additives to modify the properties of base bitumen. Thus in addition to improve the properties of bitumen, it has been tried to sustainably use the production wastes of paper factories.

2.2. Bitumen

In this research, for the base bitumen, the bitumen produced by Jey oil refining company with a performance grade of PG 64-16 according to the specifications presented in Table 1 was used.

2.3. Additives

The liquor used in this research was obtained from the waste of Mazandaran wood and paper factory with the specifications presented in Table 2. In this research, the black liquor was heated indirectly (with a maximum temperature of 110 degrees Celsius) and the powder that remained after passing through the sieve 30 was used as an additive to the bitumen to avoid a SHRP increase in the volume of bitumen during mixing due to the liquid state of the black liquor and reduce its impurity. Compared to the liquid state, this powder contains significant amounts of lignin, which may have a greater effect on improving the performance properties of bitumen and asphalt mixture.

Paper pulp has been prepared from the waste of Barghdaran Sanat company, with an average fiber length of 0.2 mm. Like liquor, this additive has been used in a oven for 24 hours at a temperature of 110 degrees Celsius, after complete drying and reducing its dimensions by an industrial mill for mixing with bitumen.

Table 1. Properties of the base binder

Humidity: 20 % Pressure: 630 mmHg Temperature: 25 ºC

No	Test	Standard Range		Result	Test Meted
		Max	Min		
1	Viscosity @ 135C	3 Pa.s	--	0.326	ASTM D4402
2	Flash Point Temp.	--	230ºC	334	ASTM D92
3	Orig. Dynamic Shear G*/sinδ ,Test Temp at 10 rad/s (ºC)	--	1.00 kPa	1.57	AASHTO T315
4	RTFO Percent Change	1.0%	--	0.048	ASTM D2872
5	RTFO Dynamic Shear G*/sinδ ,Test Temp at 10rad/s(ºC)	--	2.20kPa	3.36	AASHTO T315
6	PAV@100(ºC) Dynamic Shear G*.sinδ .Test Temp at10 rad/s(ºC)	5000 kPa	--	4920	AASHTO T315
7	PAV Creep Stiffness S. Test Temp (ºC) @ 60 s	300 Mpa	--	114.33	ASTM D6648
8	m-value, (slope)	--	0.300	0.3179	ASTM D6648
9	PAV Direct Tension,Failure Strain ,Test Temp@ 1.0 mm (ºC)	--	1.0%	--	AASHTO T314
Performance Grade Bitumen: PG 64-16					

Table 2. black liquor analysis

Total Solids	%	3.66
Ash		24.8
Organics		75.2
Lignin		20.2
Residual Soap	mg/kg	0.29
Total Oxalate		<50
chloride		37
Calcium		98
Sodium		5070
Sulphur		3230
Potassium		270
Mgnesium		36
Phosphorus		22
Nickle		5.9
Silicon		10
Manganese		3.6
Iron		1.6
Zinc		0.8
Aluminium		0.7
Stronsium		0.7
Brium		0.9

2.4. Mixing Bitumen and Additives

The appropriate amount of each additive has been selected based on past studies and the results of preliminary tests. Considering that the use of liquor and paper pulp with an amount of less than 3% cannot have much effect on improving the properties of bitumen and asphalt mixture, Therefore, according to Figure 1, choosing the optimal percentage of cellulose fibers, 4, 6, and 8 percent of cellulose microfibers have been used to modify the properties of bitumen. Choosing 8% of fibers is to determine the optimal percentage of cellulose fibers in order to show clearly the effect of a large amount of fibers in bitumen (Andrés-Valeri et al., 2018; Gupta et al., 2019; Zahedi et al., 2020).

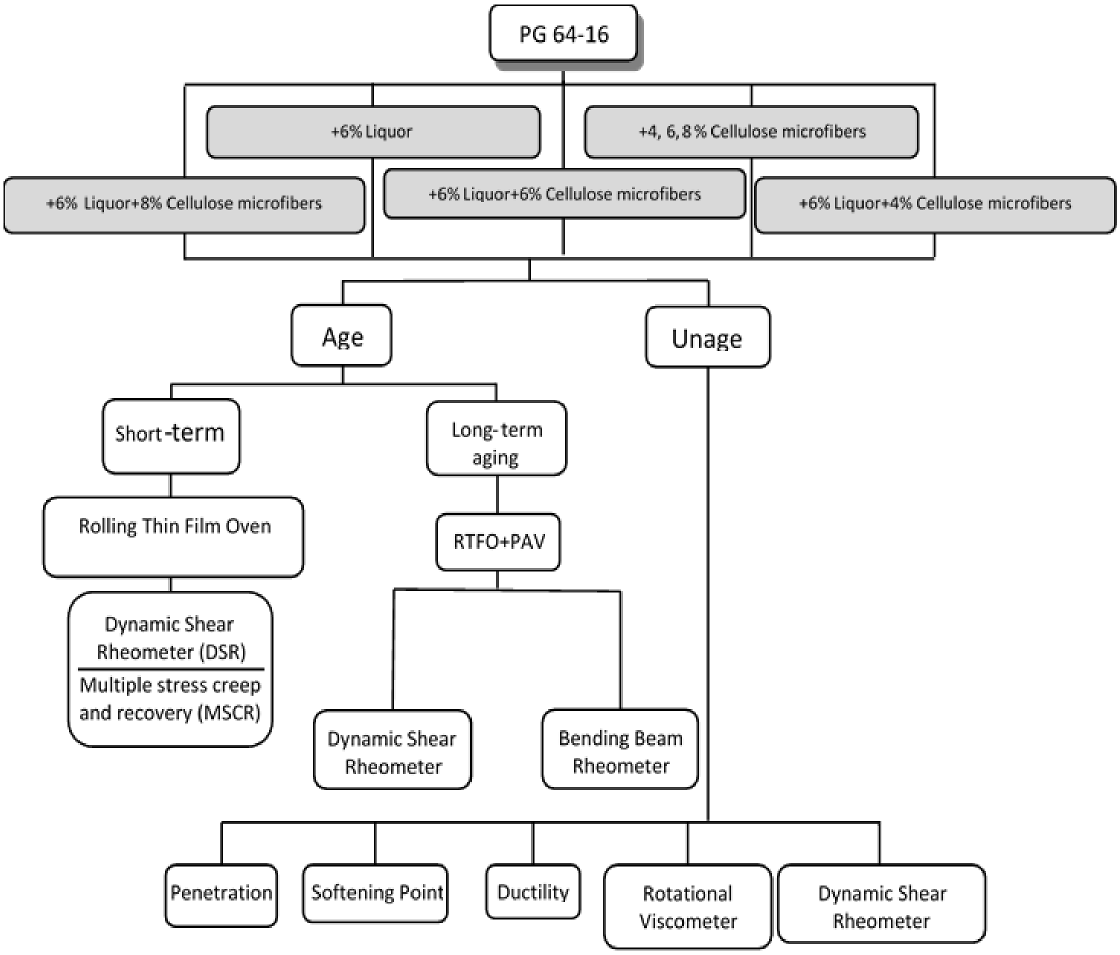


figure 1. Experimental flow chart

Due to the limitation of the particle size of modifiers in the dynamic shear rheometer test, grinding was used to reduce the size of additives (liquor and paper pulp) to less than 250 microns so that this test could be performed on the modified bitumen (Read & Whiteoak, 2003). For this purpose, the additives were first placed at 110°C for 24 hours and after complete drying, the mill has been used to reduce the dimensions of the additives. Due to the solid and insoluble nature of the additives, high shear mixer has been used for homogeneous mixing of lignin and bitumen in past researches (Lynam et al., 2018); Therefore, similar to the research conducted in this field (Arafat et al., 2019; J. Wu et al., 2021), in this research, for mixing bitumen with additives, the high shear mixer according to Figure 1, with the speed of 2500 cycles per minute has been used. Bitumen and additives were mixed at 160°C for 60 minutes.

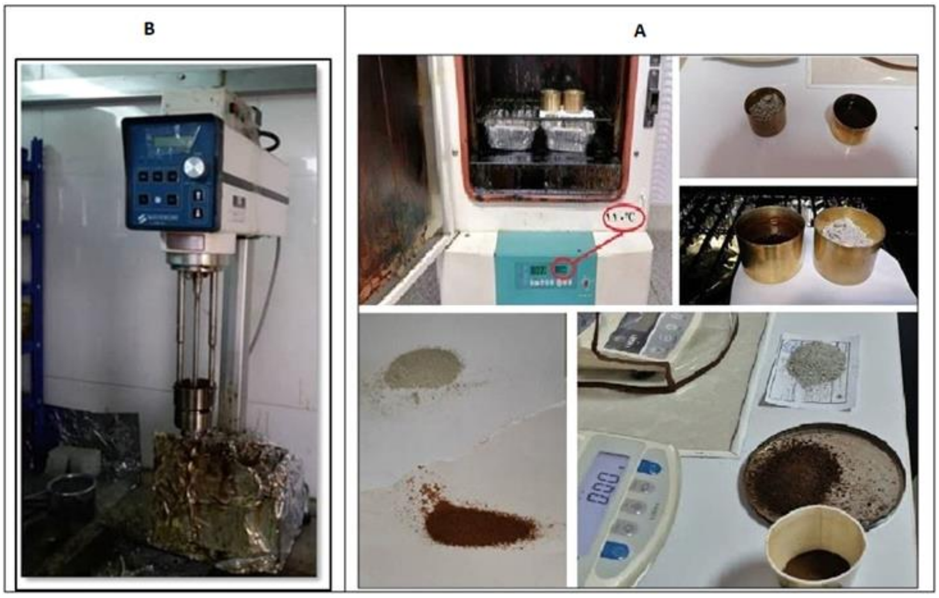


Figure 2. a: preparation of additives b: high shear mixer

2.5. Penetration Degree and Softening Point Tests

Common tests of bitumen, including penetration degree and softening point according to ASTM-D5 and ASTM-D36 standards were performed to determine the physical properties of modified bitumen; In addition, by comparing the penetration index value for base bitumen and modified samples, the effect of additives on the thermal sensitivity of bitumen was investigated. To determine the temperature sensitivity of bitumen, the equation developed by Pfeiffer and Van Doormaal in 1936 was used (Pfeiffer & Van Doormaal, 1936).

in which:

A: temperature sensitivity, PI: penetration index and $T_{R\&B}$: softening point temperature.

$$A = \frac{\log 800 - \log(\text{pen at } 25℃)}{T_{R\&B} - 25℃} \quad (1)$$

$$PI = \frac{20 - 500\,A}{1 + 50A} \quad (2)$$

The results of penetration degree and softening point tests according to Figure 2 show an increase in bitumen hardness in all modified samples.

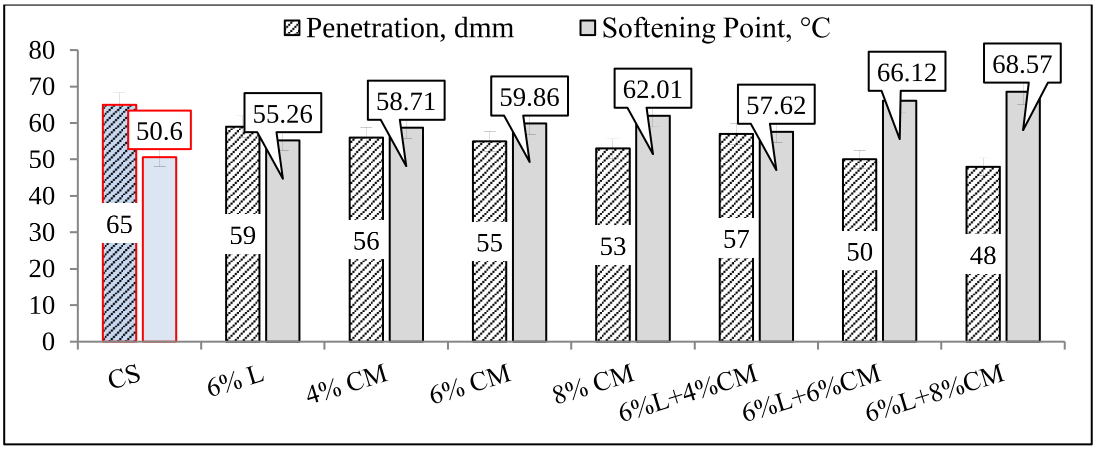


Figure 3. Penetration degree and softening point

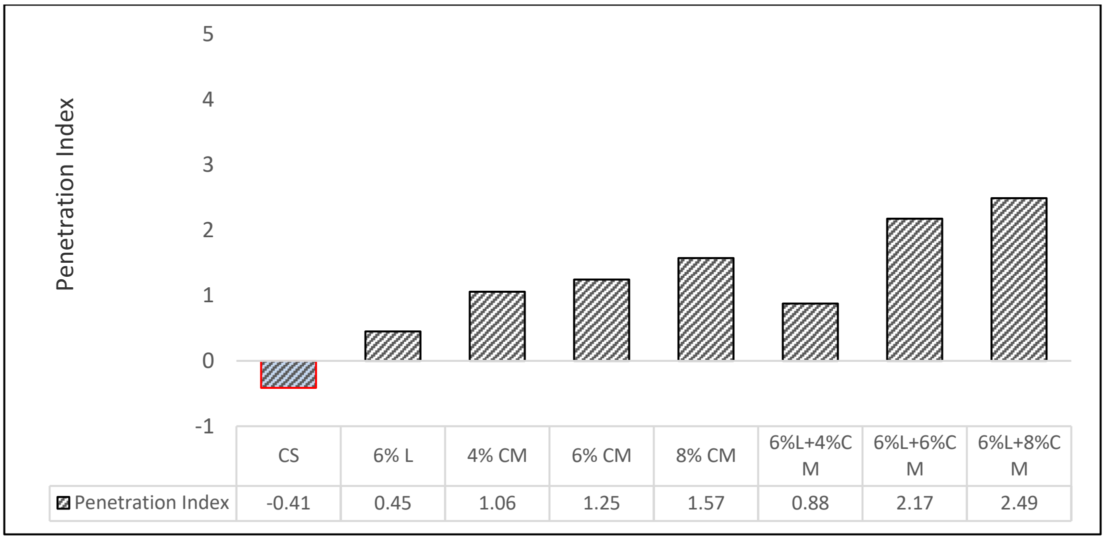


Figure 4. Penetration index

Choosing the proper degree of penetration for bitumen based on the life and performance of the pavement is very important. If the area faces a hotter climate or heavier and more frequent traffic conditions, it is necessary to use bitumen with a lower degree of penetration. The results of the tests according to Figure 3 have shown that among the investigated additives, the combination containing 6% liquor with 8% cellulose micro-fibers had the greatest effect on the softening point (about 35% increase) compared to the base bitumen. The increase in hardness of bitumen has a direct relationship with the amount of the additive, and with the increase in the amount of cellulose fibers from 6 to 8% of weight, the hardness of bitumen has also increased.

By reducing the penetration degree of the bitumen modified with cellulose micro fibers, its softening point has increased; therefore, the use of cellulose fibers can improve the properties of the modified bitumen at high temperature. As the fibers are dispersed in the bitumen and the bitumen is absorbed by the fibers, due to the creation of a three-dimensional network in the bitumen, a strong binding force is created between the surface of the fibers and the bitumen; Therefore, by maintaining this network at high temperature, the properties of the bitumen containing fibers are also improved at high temperature (Pamplona et al., 2012; M.-M. M. Wu et al., 2015).

Softening point is used in determining bitumen permeability (PI). The temperature sensitivity of bitumen depends on the sign and magnitude of penetration index, which for the bitumen used in road construction, the appropriate value of penetration index is in the range of 2 to 2-. The increase in penetration index indicates the improvement of temperature sensitivity of bitumen; in all amounts of additives, the value of penetration index has increased compared to the base bitumen. As shown in Figure 4, the penetration index of the modified bitumen increased by about 86 to 290% compared to the base bitumen; therefore, it can be concluded that the thermal sensitivity of the modified bitumen and their performance in different temperature conditions have been improved.

2.6. Ductility Test

To check the adhesion of bitumen, ductility test has been used according to ASTM-D113 standard. In this test, the bitumen sample is stretched at a temperature of 25°C at a constant speed of 50 mm/min, and the amount of bitumen elasticity is determined by measuring the elongation of the bitumen sample before tearing (in centimeters). As it can be seen in Figure 5, the ductility property of all samples modified with cellulose fibers and liquor has decreased. According to the direct relationship between bitumen ductility and its adhesive property, it can be concluded that the adhesive property of the modified bitumen has decreased.

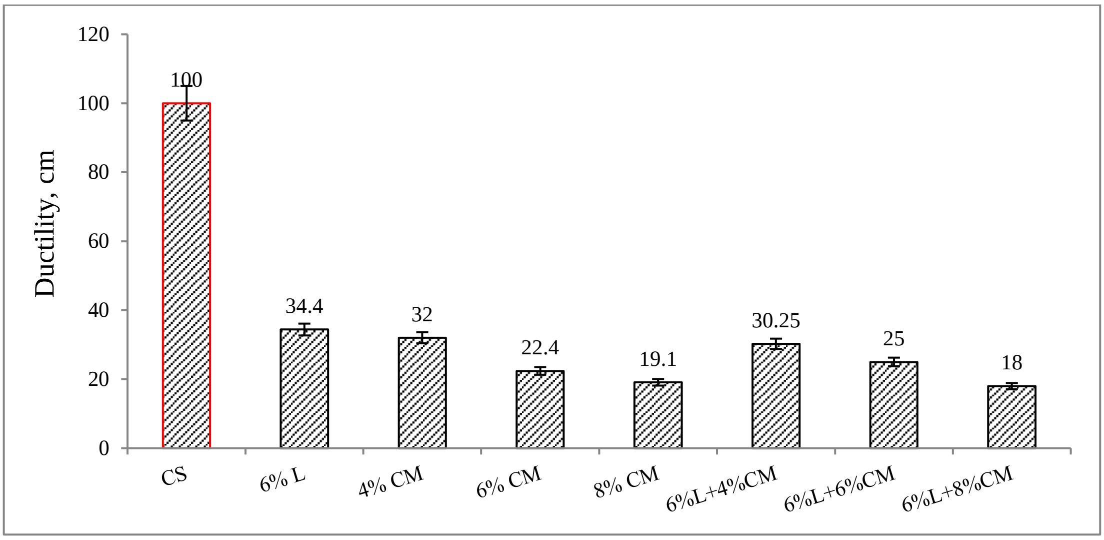


Figure 5. Ductility

2.7. Rotational Viscosity Test

In order to evaluate the efficiency and determine the proper mixing temperature and density of bitumen, rotational viscosity test was used according to AASHTO T316-04 standard. Determining bitumen viscosity is very important because of its role in bitumen pumping and moving, as well as in determining the mixing temperature and density of asphalt mixture. This test was conducted at four temperatures of 100, 135, 160 and 180 degrees Celsius to investigate the effect of each additive on bitumen viscosity at different temperatures.

As shown in Figure 6, and as it was expected, the viscosity of all the modified samples increased. Meanwhile, cellulose micro-fibers have had a greater effect on increasing the viscosity of bitumen. The increase in viscosity has a direct relationship with the additive percentage, and with the increase in the additive percentage, the bitumen viscosity has increased too. Among the investigated additives, the combination containing 6% of liquor with 8% of cellulose micro fibers has had the greatest effect on increasing the viscosity of bitumen with a 197% increase in viscosity at 135°C compared to the base bitumen.

According to Table 3, based on ASTM D6926-04 standard and according to the values of **170±20** and **280±30** centistokes, for the proper mixing and compaction temperature of bitumen, it can be concluded that the mixing and compaction temperature of all the modified samples has increased compared to the base bitumen, so that the mixing temperature of the modified bitumen increased 2.2 to 10.3 °C and their compaction temperature increased 4.3 to 17.1 °C compared to the base bitumen. However, this increase was higher in the bitumen modified with the combined addition of liquor and cellulose fibers.

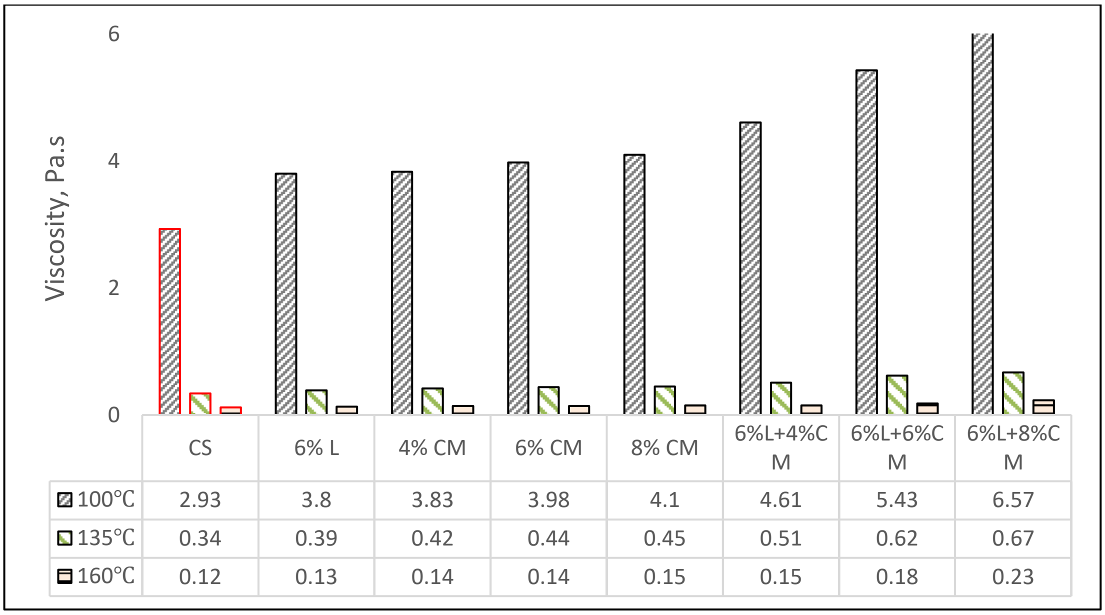


Figure 6. The results of rotational viscosity test

Table 3. Mixing and compaction temperatures for unmodified and modified binders

Components	Mixing Range (°C)	Mixing Temperature changes (°C)	Compaction Range (°C)	Compaction Temperature changes (°C)
CS	156.6-152	-	145.2-138.4	-
6% L	158.1-154.2	+2.2	148.5-142.7	+4.3
4% CM	159.1-155.5	+3.5	150.2-144.8	+6.4
6% CM	159.2-155.8	+3.8	150.8-145.8	+7.4
8% CM	160-156.7	+4.7	151.7-146.7	+8.3
6%L+4%CM	160-157.2	+5.2	153.1-148.9	+10.5
6%L+6%CM	161.7-159.4	+7.4	156-152.6	+14.2
6%L+8%CM	164.5-162.3	+10.3	158.9-155.5	+17.1

2.8. High-Temperature Performance

To describe the viscoelastic behavior of bitumen in the temperature range of 3 to 88 degrees Celsius (middle and high temperature), a dynamic shear rheometer device is used according to the AASHTO M-320 standard. In this experiment, two parameters of total shear stress modulus (G*) and phase angle (δ) which are determined based on shear force applied by the machine are used to describe both viscous and elastic behavior of bitumen. The high and medium temperature performance of bitumen will be determined based on the rutting parameter G*/sinδ (as a measure against bitumen deformation and stiffness) and comparing its value with the standardized limits. For high-temperature performance, the value of this parameter should not be less than 1000 pascals for un-aged bitumen and 2200 pascals for aged bitumen in the rotary thin glaze test. It should be noted that conducting this test and its acceptable results depend on compliance with the size limit of bitumen particles; Therefore, the investigated bitumen should not contain particles with dimensions greater than 250 microns (250 m = ¼ mm).

According to Figures 7 to 11, the rutting parameter has been increased in all modified samples in both un-aged and short-term aged states; however, only in the modified samples with the combined addition of liquor and cellulose fibers, the increase in the Rutting parameter has led to an increase in the high-temperature performance of the bitumen sample. One of the reasons for the increase in the rutting parameter in the combined samples can be the increase in hardness in these samples, which has finally led to the improvement of the high-temperature performance in the modified samples by affecting the phase angle.

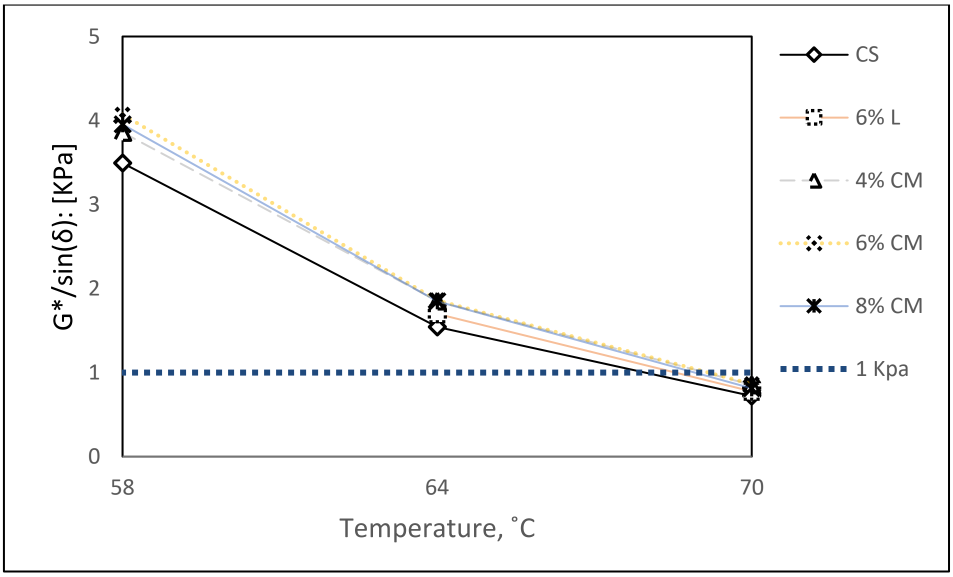


Figure 7. Rutting parameter of the samples modified with liquor and cellulose microfibers in the un-aged state

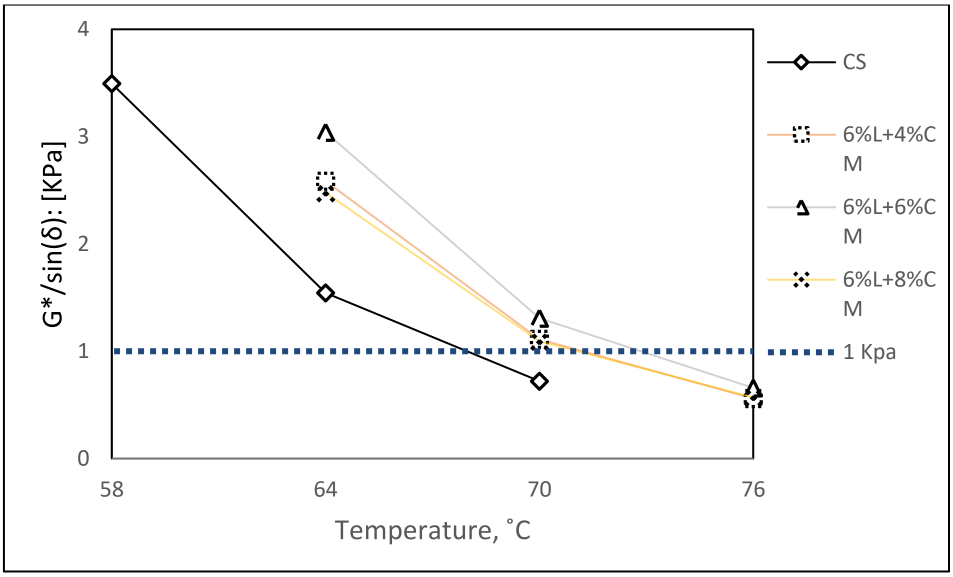


Figure 8. Rutting parameter of the samples modified with the combined additive of liquor and cellulose microfibers in the un-aged state

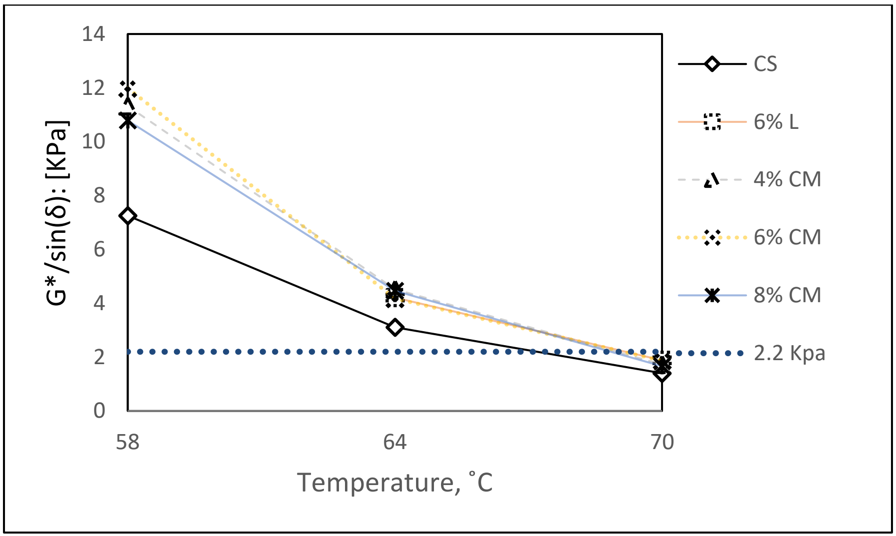


Figure 9. Rutting parameter of the samples modified with liquor and cellulose micro-fibers in a short-term aged state

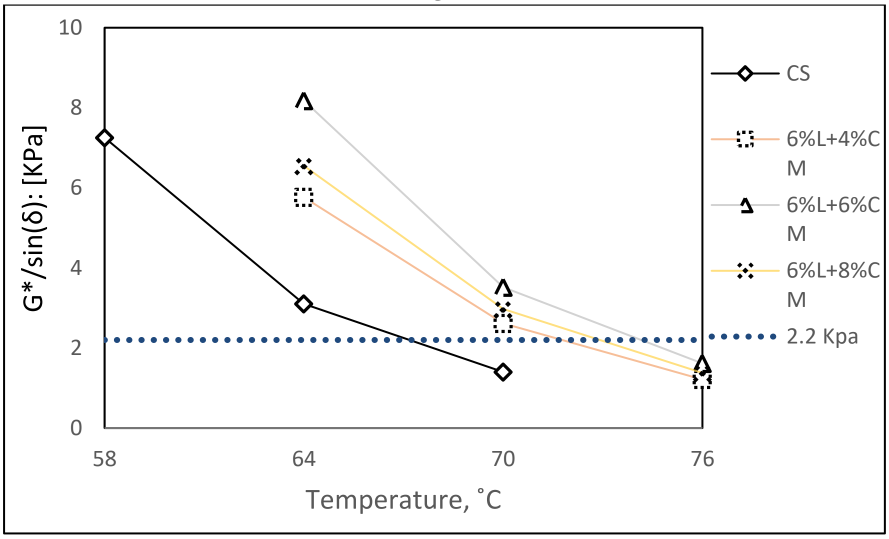


Figure 10. Rutting parameter of the samples modified with the combined additive of liquor and cellulose micro fibers in the short-term aged state

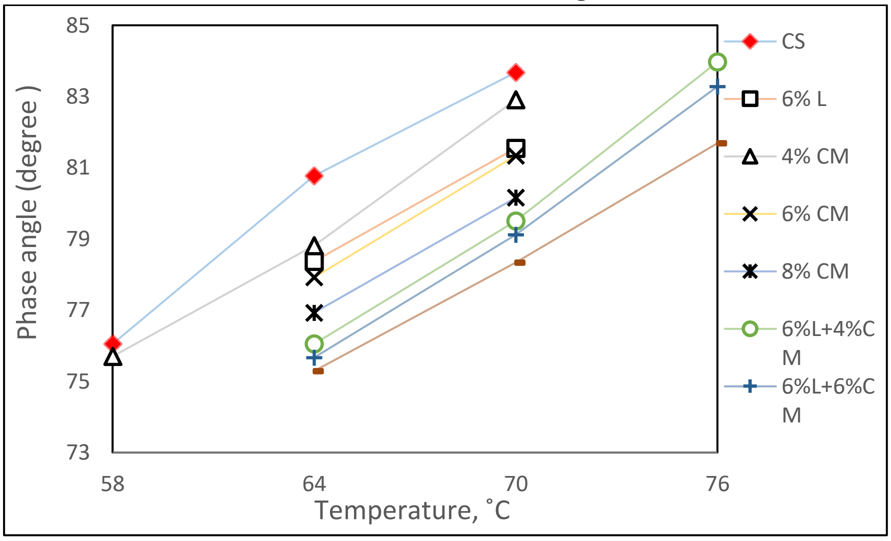


Figure 11. Phase angle changes for the base bitumen and the modified bitumen from 58 to 76°C (un-aged)

2.9. Multi Stress Creep and Recovery

The inability of conventional bitumen tests to fully describe the properties of the modified bitumen has led researchers to use SHRP Plus tests, including Multi Stress Creep and Recovery tests at several stress levels in accordance with the AASHTO T350 standard, in order to investigate and describe the properties of the modified bitumen. In the dynamic shear rheometer test, the parameters of mixed modulus and phase angle are measured in the linear range (viscosity-elasticity) and bitumen flow is considered linear and does not depend on the stress level; however, this assumption is not correct for the modified bitumen and the amount of stress applied during the experiment influences on the obtained results. The creep and return test at several stress levels is performed similar to the dynamic shear rheometer test, with a difference in how the loading is applied. This test includes 1 second of loading and 9 seconds of unloading at the stress levels of **1/0** and **2/3** kPa in 10 cycles for each stress level. The results of this test, including the irreversible softening (Jnr) as well as the return percentage (R) in each cycle, are used to check the high performance temperature of the asphalt mixture. As shown in Figures 12 and 15, cellulosic fibers have been able to improve the bitumen return percentage at both stress levels and also reduce the irreversible softness in the modified bitumen compared to the base bitumen. The decrease in Jnr parameter indicates an increase in the resistance to rutting in the samples modified with cellulose fibers, and its amount depends on the type and percentage of the added fibers; however, Jnr parameter in the bitumen modified with the combined additive of cellulose micro-fibers and liquor has increased at the stress level of 2/3 kPa compared to the sample containing only cellulose micro-fibers, as well as an increase of more than 6% of cellulose micro-fibers in the combined samples due to an extreme increase of the stiffness of bitumen has caused the sensitivity of the sample to increase the stress level; So that in these samples, the changes of the irreversible softening parameter, i.e. $J_{nr\ difr}$, are more than 75%.

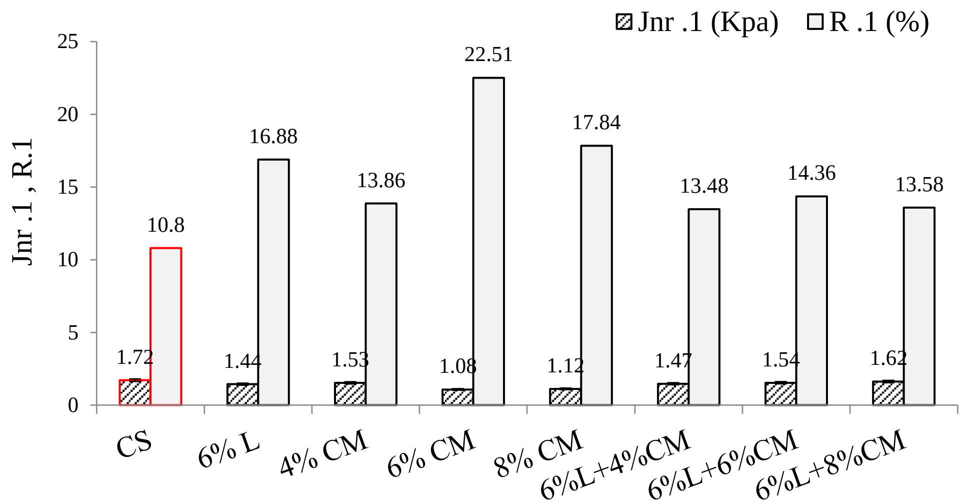


Figure 12. Irreversible softening parameter and return percentage (stress level 1/0 kPa and temperature 64 degrees Celsius)

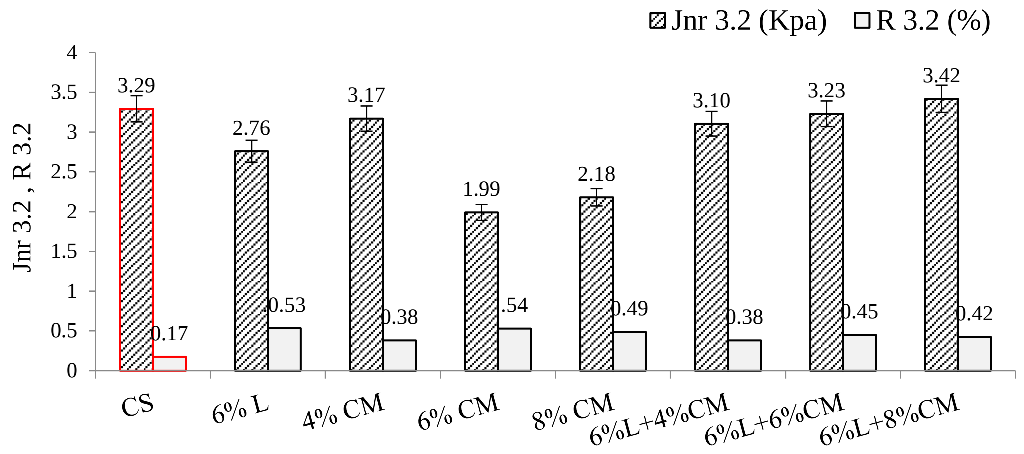


Figure 13. Irreversible softening parameter and return percentage (stress level 2.3 kPa and temperature 64°C)

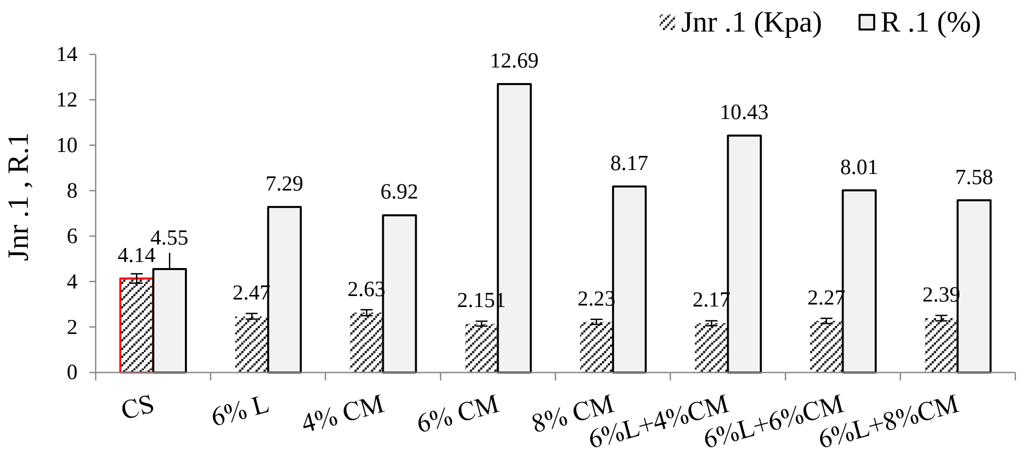


Figure 14. Irreversible softening parameter and return percentage (stress level 1/0 kPa and temperature 70°C)

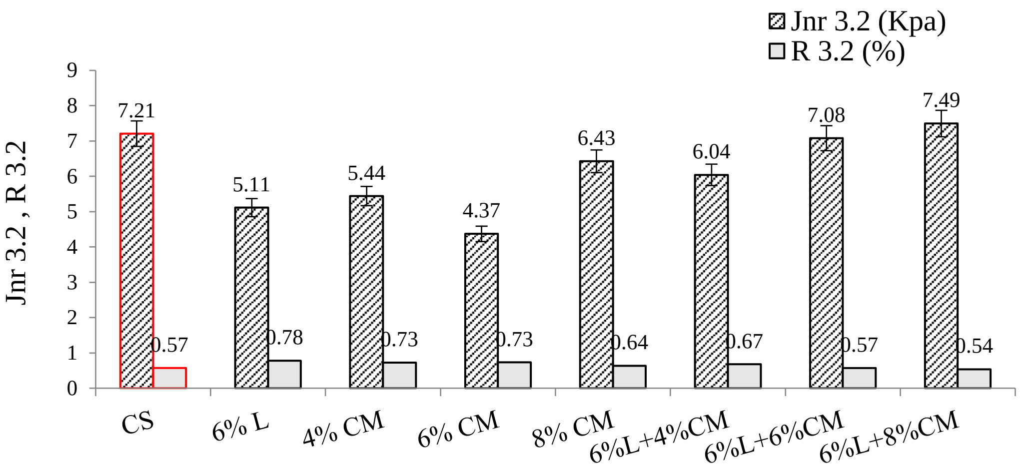


Figure 15. Irreversible softening parameter and return percentage (stress level 2.3 kPa and temperature 70°C)

2.10. Linear Amplitude Sweep Test

To evaluate the bitumen fatigue behavior, the linear amplitude sweep test according to AASHTOTP-101 standard has been used. This test was performed with a dynamic shear rheometer device and with a spindle with a diameter of 8 mm on short-term aged bitumen. The linear amplitude sweep test consists of two stages. The first step, the frequency sweep, is designed to obtain information about the bitumen's rheological properties. In this stage, loading is done with constant strain and with a wide range of frequencies from 1/0 to 30 Hz. By determining the shear complex modulus and the phase angle at each frequency, the storage modulus and finally the failure analysis parameter (α) will be obtained. In the second stage, i.e. strain sweep, loading is performed with a constant frequency of 10 Hz and with a linear change of the strain amplitude from 1/0 to 30%.

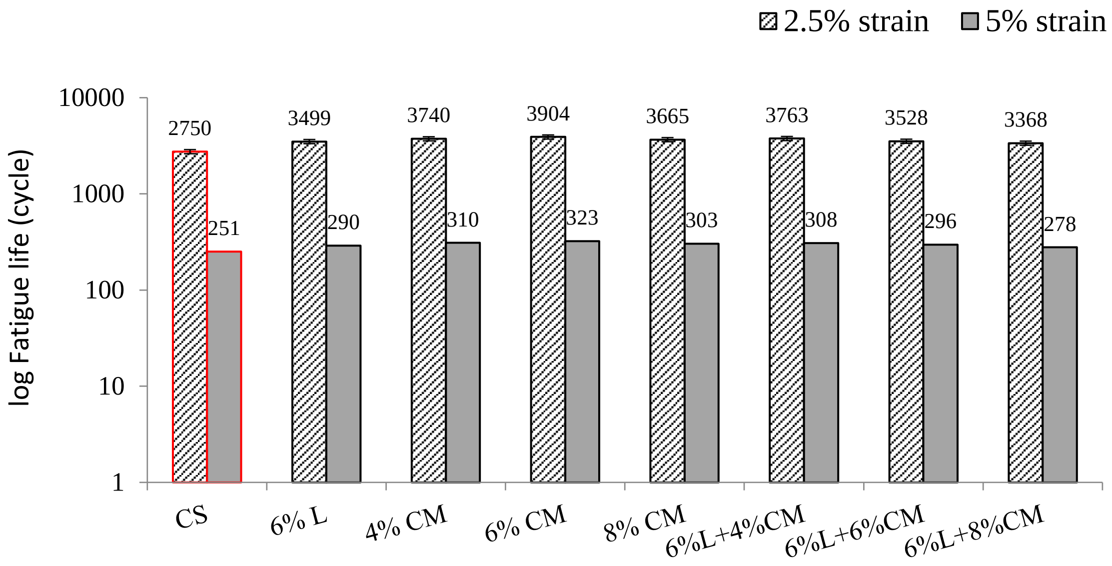


Figure 16. Fatigue life of RTFO-aged samples at 2.5% and 5% strain amplitude

By analyzing the outputs of linear amplitude sweep test, the fatigue life of bitumen and the modified samples was determined based on viscoelastic cumulative failure method. According to Figure 16, the results show improved fatigue life in the modified samples; however, the upward trend of fatigue life has been stopped with an increase of more than 6% of cellulose micro fibers. The data obtained from this experiment confirm the results of SHRP tests with the same trends but at different levels.

2.11. Low-Temperature Performance

Bending beam rheometer test is performed in order to measure the amount of fluctuation or creep of bitumen under constant load and constant temperature, on the aged bitumen under short-term aging and long-term aging. In this test, the values of two parameters, creep hardness and m-value, which respectively indicate bitumen resistance against creep loading and changes in bitumen hardness during loading, are measured and reported at the times of 8, 15, 30, 60, 120, and 240 seconds. According to AASHTO T313 standard, m-value in 60 seconds should be greater than or equal to 3/0 and the value of creep hardness should not exceed 300 MPa in 60 seconds. According to the obtained results, the value of creep hardness in all samples has been lower than the standard limit of 300 MPa; therefore, in this research, only the results of m-value parameter have been used.

As it can be seen in Figure 17, except for liquor, which had a negative effect on the low-temperature performance, m-value has increased in the samples modified with other additives. Among the studied samples, the use of 6% cellulose micro fibers has had the most positive effect on the low-temperature performance of the bitumen, so that the low performance temperature of this combination has improved one level higher than the basic bitumen (from 16-°C to 22-°C). The use of cellulose microfibers in the sample mixed with liquor has been able to reduce the negative effect of liquor on the low-temperature performance to some extent. The creep stiffness of the samples modified with cellulose microfibers has increased corresponding to the results obtained for the penetration degree and the softening point; however, despite the increase in the hardness of the bitumen modified with fibers, due to the increase of the m-value parameter, the Low-temperature performance of the bitumen modified with cellulose microfibers has been improved.

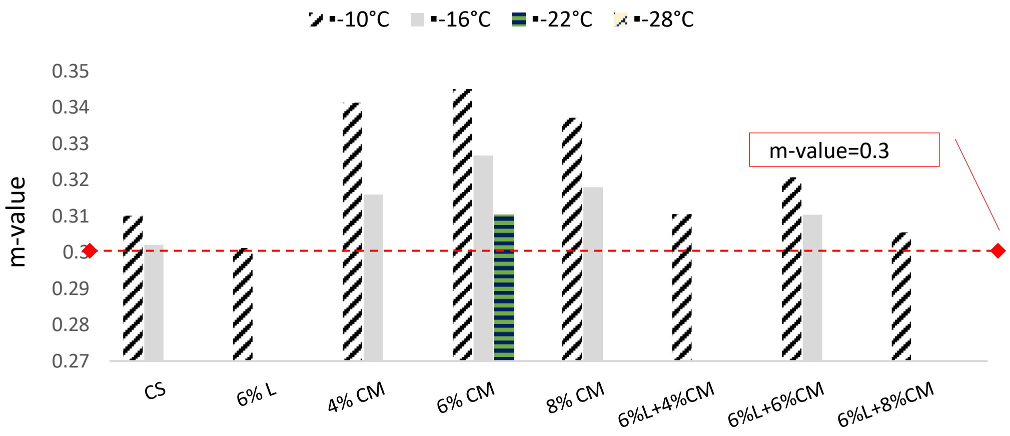


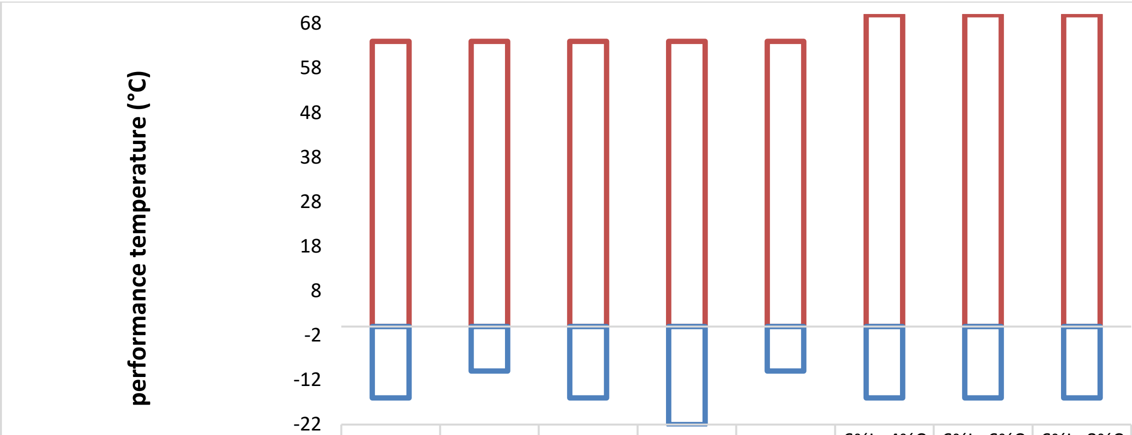
Figure 17. Changes in the m-value parameter for each combination

2.12. Performance Grades of Samples

According to the obtained results, the properties of bitumen modified with cellulose fibers have been improved; however, it is not recommended to add more than 6% of cellulose micro-fibers in order to modify the properties of bitumen. In this research, the use of 6% cellulose fibers compared to 8% cellulose microfibers has had a more positive effect on the properties of the modified bitumen.

According to Figure 19, the use of liquor in combination with cellulose microfibers has improved the high-temperature performance to one-step from 64 to 70 degrees Celsius. One of the reasons for the improvement of the high-temperature performance of the modified samples can be the increase of the stiffness of these samples. The results of the past researches also support the increase in stiffness of bitumen modified with cellulose fibers (Li et al., 2021; Mohammed et al., 2018; M.-M. M. Wu et al., 2015), so that in the research of Mohammed and his colleagues, the use of two percent of cellulose fibers has increased the softening point temperature of bitumen from 52 to 59 degrees Celsius (Mohammed et al., 2018). In this research, the use of 6% of pure cellulose microfibers has increased the softening point by 9.26 degrees Celsius compared to the base bitumen.

The difference in the obtained results can be due to the difference in the type of cellulose fibers used in the research of Mohammad and his colleagues, as well as the size of the fibers used (20 to 2500 micrometers long and 25 micrometers wide) compared to the present research.



	CS	6% L	4% CM	6% CM	8% CM	6%L+4%CM	6%L+6%CM	6%L+8%CM
high-temperature performance	64	64	64	64	64	70	70	70
low-temperature performance	-16	-10	-16	-22	-10	-16	-16	-16

Figure 18. performance grades of samples

In Table 4, the zoning of the suitable bitumen based on performance (PG) for the provincial centers of the country has been given. The results of this research showed that by using liquor and cellulose fibers, it is possible to obtain several types of modified bitumen (several performance categories) at a low cost, which can provide several choices for selecting modified bitumen according to the temperature conditions of each region for the officials and contractors of bitumen and asphalt industry of the country. According to the average air temperature of the centers of Iran's provinces during summer and winter, it is possible to provide suitable bitumen zoning based on the following temperatures:

1. Northern provinces (such as Gilan, Mazandaran, Golestan, etc.), which have cold winters and mild summers; for these areas, the bitumen with normal winter temperature (-10 degree Celsius) and normal summer temperature (58-64 degrees Celsius) (i.e. samples of 6%L and 8%M) will be suitable.

2. Eastern provinces (such as Razavi Khorasan, North Khorasan, Sistan and Baluchistan, etc.) which have cold winters and hot and dry summers; for these regions, high temperature bitumen (64 degrees Celsius) is used in summer and low temperature (-16 degrees Celsius) is used in winter. Samples of 4%CM can be used in these areas.

3. Western provinces (such as Kurdistan, West Azarbaijan, Kermanshah, etc.), which have cold winters and mild summers; for these regions, the bitumen with winter temperature (-16 degrees Celsius) and summer normal temperature (64 degree Celsius) will be suitable.

4. Southern and southwestern provinces (such as Fars, Kerman, Bushehr, etc.), which have mild winters and very hot and dry summers; for these areas, the bitumen with high temperature (70 degrees Celsius) and normal temperature (-16 degrees Celsius), i.e. samples of 6%L+6%CM, 6%L+8%CM and 6%L+4%CM can be used in summer and winter respectively.

Other factors such as asphalt characteristics (including traffic, vehicle weight, etc.) as well as the specific needs of each area can play a role in choosing the proper bitumen based on environmental conditions.

Table 4. The minimum and maximum suitable pavement temperature for the centers of the provinces of Iran based on air temperature (https://pgi.bhrc.ac.ir/)

	Minimum air temperature, at 98% confidence level (°C)	maximum air temperature, at 98% confidence level (°C)	Minimum pavement temperature, at 98% confidence level (°C)	maximum pavement temperature, at 98% confidence level (°C)
Eastern Azarbaijan	64	-16	59.82	-15.38
Western Azerbaijan	58	-16	57.21	-15.2
Ardabil	58	-22	55.18	-20.77
Esfahan	64	-10	62.5	-9.49
Alborz	64	-16	61.25	-12.14
Ilam	64	-16	62.71	-10.14
Bushehr	64	-10	63.56	4.72
Tehran	64	-10	62.6	-4.96
Chaharmahal and Bakhtiari	64	-28	59.02	-24.25
southern Khorasan	64	-16	63.43	-12.86
Khorasan Razavi	64	-16	61.15	-15.3
North Khorasan	64	-16	59.6	-14.93
Khuzestan	76	-10	71.17	0.31
Zanjan	64	-22	59.19	-19.83
Semnan	64	-10	63.43	-6.49
Sistan and Baluchestan	64	-16	63.29	-10.44
Fars	64	-10	63.33	-6.08
Qazvin	64	-16	60.8	-13.99
Qom	70	-16	65.32	-11.66
Kurdistan	64	-16	62.53	-15.19
Kerman	64	-16	62.58	-13.32
Kermanshah	64	-16	63.57	-12.92
Kohgiloyeh and Boyerahmad	64	-10	61.08	-9.76
Golestan	64	-10	61.37	-5.27
Guilan	58	-10	57.81	-6.76
Lorestan	70	-10	64.54	-8.84
Mazandaran	64	-10	60.14	-3.16
Central	58	-16	56.4	-12.33
Hormozgan	70	-10	67.59	4.43
Hamedan	64	-28	59.68	-22.69
Yazd	70	-10	66.07	-7.95

3. Statistical Analysis

In this study, Pearson's correlation analysis was conducted aiming at investigating the effect of cellulose fibers on the characteristics of the modified bitumen. SPSS22 software was used to perform this analysis. According to the results of the tests, only the effect of cellulose fibers was investigated in the tests of penetration degree, softening point and rotational viscosity. The different effect of 8% fibers caused a limitation in performing Pearson analysis for the results of other tests, including high functional temperature and low functional temperature. According to Table 5, the results show a significant relationship (Sig < 0.05) of the amount of the added fibers with changes of the degree of penetration, softening point and viscosity of the modified bitumen.

Also, the amount of Pearson Correlation indicates a strong correlation among the variables.

Table 5. Pearson analysis and investigation of the influence of cellulose fibers on bitumen properties

Factor	Characteristics	Sig.	Pearson Correlation
cellulose fibers	Penetration degree	0.004	-0.879
	softening point	0.005	0.873
	viscosity (135˚C)	0.053	0.70

4. Discussion

All the additives examined in this research have increased the hardness of the modified samples; this increase is evident in the samples modified with a high percentage of cellulose fibers. One of the reasons for improving the performance temperature of the bitumen modified with cellulose microfibers is the role of fibers in transferring and dispersing stress and preventing excessive stress concentration. Also, with the absorption of bitumen by the fibers, due to the creation of a three-dimensional network in the bitumen, a strong connection force is created between the surface of the fibers and the bitumen; therefore, by maintaining this network at high temperature, the properties of the bitumen containing fibers are also improved at high temperature (Mohammed et al., 2018; M.-M. M. Wu et al., 2015).

Nevertheless, the modified bitumen must maintain its elastic property to some extent so that it does not suffer premature failure during successive loading (Bessa et al., 2019). Excessive increase of fiber amount can have a negative effect on the properties of the modified bitumen, so that the functional temperature of the samples containing 8% cellulose microfibers has been reduced compared to the sample containing 6% cellulose fibers. The results of the linear amplitude sweep test also show a decrease in fatigue life in the samples containing 8% cellulose microfibers. The negative effect of the large amount of fibers on the properties of the modified bitumen can be due to an excessive increase in bitumen roughness; in addition to cause premature cracks in bitumen and asphalt mixture, this increase can also reduce the integrity of the modified asphalt mixture (Andrés-Valeri et al., 2018; Bessa et al., 2019; Gupta et al., 2019; Zahedi et al., 2020). However, based on the results of this research and other researches, the use of the appropriate amount of cellulose fibers can improve the properties of the modified bitumen according to the method of preparation and the source of fibers. Various methods of producing cellulose microfibers and nanofibers, like mechanical, physical, and chemical methods, including acidic and alkaline hydrolysis, as well as the availability of different sources for preparing cellulose fibers have caused the characteristics and cost of modifying bitumen and asphalt mixture with these types of fibers to be very different. For example, in the mechanical method, high shearing force and prolongation of the production process can cause damage to the cellulose crystals (Moon et al., 2011), or in the chemical method, due to dissolution in acid, the desired properties of the fibers such as the surface roughness of the fibers decreases. Also, the physical method requires using a lot of energy to produce microfibers (Frone et al., 2011); Therefore, in addition to reduce the production time of cellulose micro fibers and its lower cost compared to other researches, using the waste materials of paper factories has a less negative effect on the properties of the fibers including the surface roughness of the fibers.

Also, unlike polymer bitumen, the samples containing paper pulp have storage stability and therefore there is no need to use other additives to solve the problem of storage stability. According to the results of past researches, the use of lignin can help resist the formation of carbonyl functional groups in the modified bitumen after short-term and long-term aging; therefore, lignin can be used as an antioxidant to reduce the negative effects of aging in bitumen and asphalt mixture.

5. Conclusion

The main purpose of this research was to investigate the possibility of using cheap waste materials from paper factories to improve the mechanical and rheological properties of bitumen. Based on the tests and analysis and evaluations performed, both additives investigated in this research, i.e. cellulose micro fibers and liquor have improved the properties of bitumen at high temperature; however, some limited weaknesses, such as an excessive increase of bitumen stiffness when an increase in the additive percentage, can limit the use of this type of additive to improve the properties of bitumen.

According to the results of the past researches, the use of cellulose fibers has increased the hardness of bitumen in this research too, so that the penetration degree of the modified bitumen has decreased by 10-26%.

Also by reducing the degree of penetration, the softening point of the modified bitumen shows an increase of about 9 to 35% compared to the base bitumen; therefore, with the increase of the penetration index due to the increase of bitumen hardness, the thermal sensitivity of all the modified bitumen has decreased.

Despite the increase in the mixing temperature and compactness of the modified samples, they still did not have much effect on the increase in the mixing temperature and compactness of the bitumen compared to the polymer bitumen of cellulose micro fibers and liquor.

Due to the increase in hardness and as a result of the increase in the rutting parameter in the modified bitumen, all the additives examined in this research have improved the high working temperature of the bitumen; however, despite the improvement of high-temperature performance, the use of large amounts of additives can have a negative effect on other bitumen properties. Also, the data obtained from the creep and return tests at several stress levels and the linear amplitude sweep confirms the results of the SHRP tests with the same trends but at different levels. According to the results of SHRP Plus tests, the use of cellulose micro fibers with an amount of 6% or less can have a significant effect on improving the properties of bitumen. Also, in the combined sample of 6% liquor and 4% cellulose micro fibers, the properties of bitumen, including its fatigue life, have been significantly improved.

Regarding the low-temperature performance of bitumen, due to the increase of the m-value parameter, all the modified bitumen, except the bitumen containing liquor, have performed better at low temperature; however, it has only improved to one grade.

According to the obtained results, liquor has had a negative effect on the low-temperature performance of bitumen due to the increase of bitumen hardness; however, the use of cellulose microfibers has been able to improve the high-temperature performance of bitumen and reduce the negative effect of liquor on the low-temperature performance of bitumen.

In this research, only the effect of cellulose micro fiber additives and liquor on the physical and rheological properties of bitumen has been investigated, so the results of this study cannot be generalized to asphalt mixtures and it is necessary to investigate the effect of using the mentioned additives in asphalt mixtures.

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