MODIFICATION OF BITUMEN WITH SULFUR AND NATURAL RUBBER AND STUDY OF ITS CHEMICAL AND RHEOLOGICAL PROPERTIES

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Abstract. In this paper, the chemical, rheological and elasticity properties of sulfur and natural rubber modified bitumen (RSMB) were investigated. Sulfur (GOST 127-93), natural rubber and BND 60-90 brand bitumen were taken as raw materials in the process of bitumen modification. The percentages of these raw materials are as follows: sulfur -35%, natural rubber -1% to 6%, and bitumen - 59-64%. In studying the chemical properties of modified bitumen, its IR spectrum analysis, MIRA 2 LMU scanning electron microscope, and thermal gravimetric TGA and DTA analyzes were more widely covered. The rheological, wear and high elasticity properties of modified bitumen were analyzed depending on its application. From the results of the analysis, it can be seen that the amount of natural rubber in the modified bitumen content increased, which led to a decrease in the amount of sulfur in the bitumen content. The mechanical properties of bitumen modified with sulfur and natural rubber have reduced elasticity and hardening during cracking. In this research work, it was observed that modified bitumen is resistant to frost, compression, breaking, cracks, and does not lose its original properties and strength for a long time when used in road construction.

Key words: modified bitumen, natural rubber, n-hexane, sulfur, blue gum plant.

Introduction. Currently, the use of bitumen modified with sulfur and natural rubber in the production of waterproof membranes and emulsified asphalt products is one of the effective methods, as a strategic raw material that is in significant demand in the construction of roads and highways in many countries. is being used [1,2]. Over the past decade, research has been conducted on the benefits of natural rubber and sulfur-modified bitumen [3]. As a result of adding bitumen modified with natural rubber and sulfur to asphalt mixtures, it improves the asphalt composition and significantly increases the service life of the road surface, that is, it leads to an increase in the asphalt properties of elasticity, cohesion, hardness, viscosity and thermal properties [4-6]. As natural rubbers, sterol-isoprene-styrene, styrene-butadiene-styrene, polyisoprenes are obtained and are used as modifiers in bitumen modification. In fact, these modifiers can increase bitumen durability and improve the high stability, cracking strength and thermal resistance of asphalt [7,8]. Bitumen-asphalt binders are thermoplastic materials that exhibit elastic stiffness at low temperatures or rapid cracking, and viscous fluids at high temperatures or slow cracking. These features are a two-way process, in the first process, stress cracking occurs at low temperatures, and in the second process, permanent deformation occurs at high temperatures, which leads to an increase in the performance of bituminous binders [9,10]. Modified bituminous binders are added in order to increase the high performance quality and thermal properties of the road. However, as a mixed organic compound material, bitumen has the property of oxidation when exposed to the external environment, which makes the bitumen hard and brittle, resulting in pitting, cracking, service life, road durability and driving safety of the bitumen-asphalt pavemen has a great effect [11-14]. In order to overcome these shortcomings, it has become a trend to add modifiers to pure bitumen in order to increase the resistance of bitumen materials to wear reactions, reduce their wear rate, or improve the anti-wear properties of bitumen [15].

The use of modifiers improves the high and low temperature properties of bitumen, especially flexibility, and at the same time improves dust absorption properties. Modified bitumen is light and can improve the oxygen resistance property, but it is very difficult to control the aging process of oxygen bitumen in the thermal state [16]. It can effectively improve the storage and elastic properties of bitumen materials by adding natural rubber to improve the quality of bitumen, thereby improving the high-temperature decomposition resistance and anti-wear performance of bitumen [17]. Currently, natural and synthetic rubbers are one of the most widely used modifiers, which increase the properties of bitumen against wear, temperature, cold and cracking [18]. Addition of waste resins to bitumen can damage the composition of bitumen and cannot be preserved for a long time, because when processing waste resins, their chemical composition and viscosity properties decrease and affect the field of use of bitumen, which prevents the popularization of rubber-bitumen. In recent years, high-temperature desulfurization technology has been used as an effective tool to solve the compatibility problem between synthetic rubber and bitumen, and natural rubber rubbers have been used, because the rubberized bitumen in the form of a thermal mixture prepared by high-temperature desulfurization technology has its high storage stability and It is characterized by good construction [19,20]. The addition of synthetic rubber to bitumen affects its elasticity and thermal stability, because the degradation of high-temperature desulfurization can significantly reduce the mechanical properties of bitumen, which leads to a decrease in the anti-degradation properties of bitumen. Sulfur and a small amount of natural rubber are added to bitumen with synthetic rubber to improve the elastic properties of the binder to eliminate the defect of high temperature indicators. Natural rubber is one of the binders that helps to effectively increase the storage life of sulfur-modified bitumen and significantly improves the resistance to decomposition of bitumen [21,22]. Because on the one hand, sulfur molecules form polysulfide bonds between polymer molecules and some components of bitumen in order to improve the connection between natural rubber and bitumen and are interconnected through them. On the other hand, due to the chemical bonding of sulfur, the polymer structure helps to bind to the bitumen composition and significantly increases the resistance to decomposition of the modified bitumen [23]. In the implementation of modified bitumen preparation technology, the modification of bitumen by adding sulfur and natural rubber at high temperature is an important process, and the analysis of the reactions and results of the process plays an important role.

Currently, certain scientific and practical research is being conducted in our Republic on the selection of the most optimal method of mixing sulfur binders as modifiers and modified bitumen based on industrial waste, secondary products of the gas and oil processing industry [24]. The properties of modifiers when carried out by low temperature and fast attachment, their elastic property becomes a solid state substance, and at high temperature and carried out by slow attachment, they appear as a viscous liquid. So, in order to improve the adhesion properties of bitumen, it is necessary to keep its tension and cracking at high temperature in order to be in constant deformation at low temperature [25]. Currently, interest in modified bitumen in road construction is increasing, as many methods are used to increase the service life and durability of roads. As a result, the demand for natural rubber used in bitumen modification is increasing [26]. The most commonly used natural rubber polymers are styrene-butadiene-styrene and isoprene in the cis-, trans-position, which are usually added from 1% to 6% by weight of the bitumen phase. Economically, the only important source of industrial natural rubber (cis-1,4-polyisoprene) is currently the blue-gum, yellow-gum, and sedum plants that grow in countries with a tropical climate [27]. Blue-gum, yellow-gum and kokiot plants grow in natural conditions at an altitude of 1800-2100 meters above sea level, in more or less saline soils, gravel and thickets. These plants grow a lot in Central Asia, in the regions of Kazakhstan, Kyrgyzstan and Uzbekistan. The blue-gum and syrig-gum plants are perennial plants belonging to the family of sedges, and milky juice is present in all parts of the plant [28]. For the production of natural rubber on an industrial scale, the cultivation of wild-growing blue-gum, yellow-gum and sedum plants occupies the main place. scientific research is being carried out and is being tested. Natural rubbers obtained from the roots of the blue-gum and yellow-gum plants are not inferior in quality to the rubbers obtained from the heave tree, because they are better when used in tires of heavy vehicles [29,30]. We used the blue gum plant as a raw material, which was harvested in the wild in August-October, the root parts were washed until clean, dried at room temperature, and natural rubber was obtained and tested under laboratory conditions. . The general appearance of the blue gum plant (Fig. 1) is given in [31].

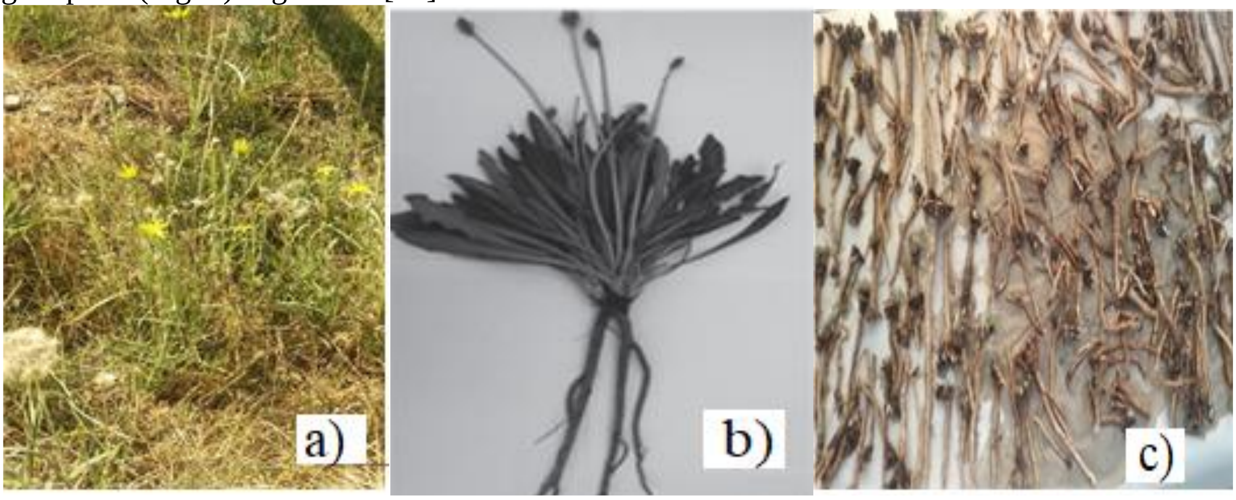


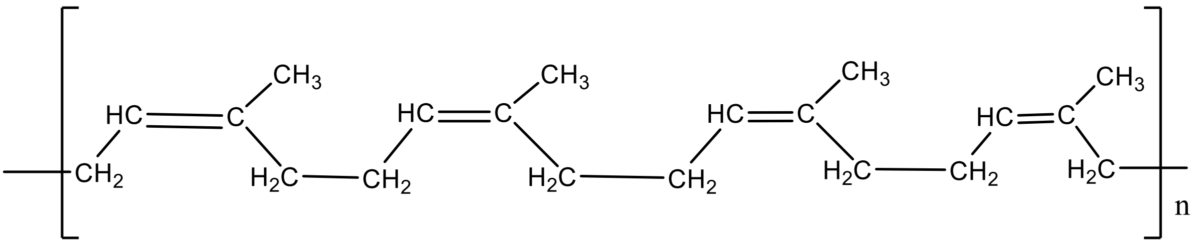
Figure 1. Blue gum plant. a) Surface view of the earth, b) Excavated and washed state, c) Root part construction state

Rubber content of blue-gum roots Focused on the convenient and modern method of growing rubber plants and increasing their rubber content, plants grown in different locations were taken and tested to determine the average yield of the plant. , resulting in the accumulation of high-quality rubber in the roots of the blue-gum plant, and its composition relative to the dry mass of the roots was studied. Natural rubber obtained from the blue gum plant improved the composition of bitumen when used in bitumen modification [32].

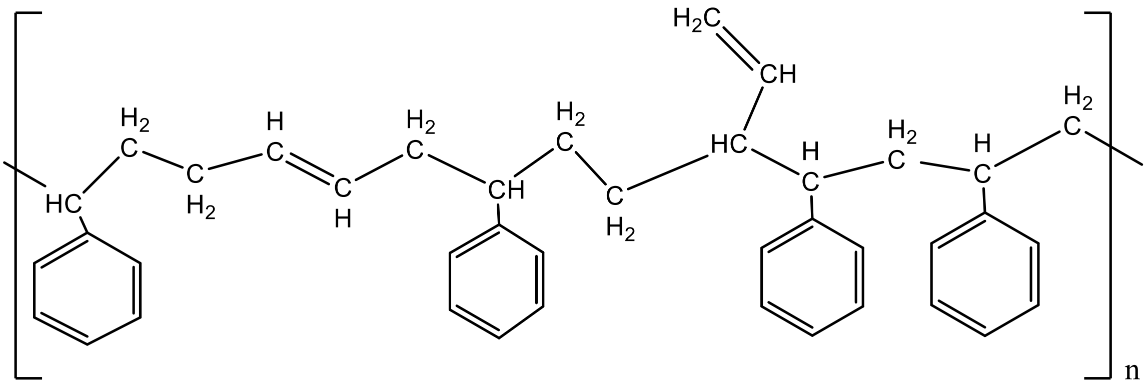
2. EXPERIMENTAL PART.

It was harvested with the roots of a yellow-gum plant that grows well in the wild, on rocky ground. It was thoroughly washed in water and dried at room temperature for two to three weeks. The dried root was ground in a mill until it became flour. 100 g of powdered root extract was placed in a soxhlet, 400 ml of n-hexane was poured over it and extracted for 10-12 hours at a temperature of 70-90 oC (Fig. 2). The extracted liquid was evaporated in a vacuum rotary evaporator until a pale yellow oily product was formed, after which the extracted light yellow oily product was dried in a drying oven at a temperature of 50 °C.

As the initial components, sulfur (GOST 127-93) and bitumen brand BND 60-90 were taken as raw materials, then 300 g of bitumen was placed in a container and heated until it liquefied at a temperature of 130-150 °C, 25 g of natural rubber was added to the liquefied bitumen. 1500-2000 rpm. mixed at high speed and while stirring, 175g of sulfur was added. During the bitumen modification process, it was heated for one hour at 180-200 oC with continuous stirring.



a)



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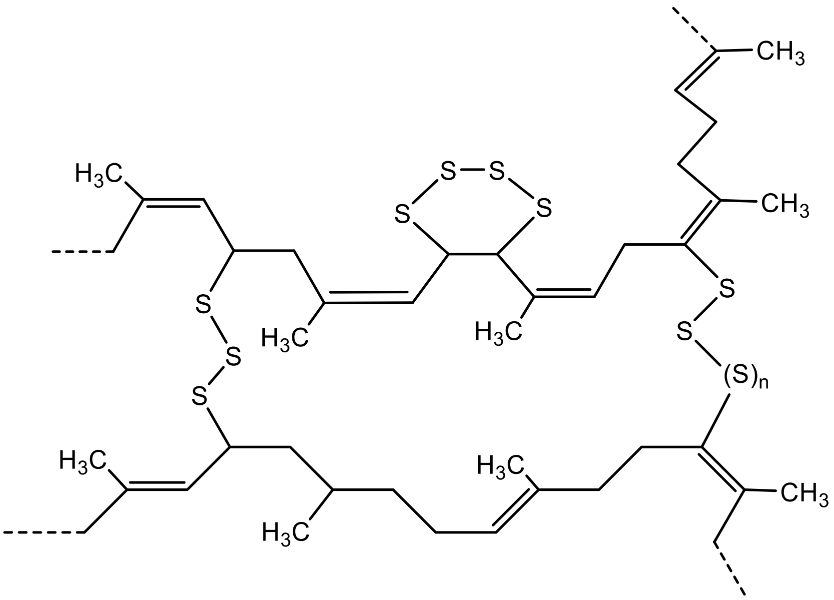

c)

The general formulas of the substances formed during the modification process are given: a) cis-polyisoprene, b) a mixture of bitumen and polyisoprene, c) the chemical structure of the interconnection between polyisoprene and bitumen after vulcanization.

The chemical composition of the obtained sample was studied using infrared spectroscopy, MIRA 2 LMU scanning electron microscope, thermal gravimetric analysis, and analysis of rheological properties.

3. RESULTS AND DISCUSSION

3.1. It can be seen from the IR spectrum analysis of modified bitumen that the vibration of the sulfoxide bond of the S=O group can be seen in the region of 1039.63 cm^{-1}. -C=N- valence vibrations were observed in 1653-1600.92 cm^{-1} areas, -C-S-C- valence vibrations were observed in 698.23 cm^{-1} areas (Fig. 3).

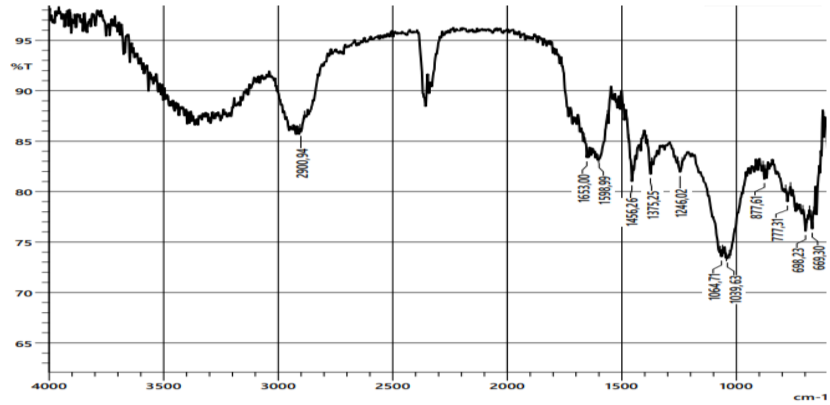

Figure 2. The IQ- Spectrature of Modified Bitum

Based on the IR-spectrum analysis of modified bitumen, we can see the following structure.

3.2. Surface part of the modified bitumon mira 2 lmm scanning electronic microscopic (SEM).

Scanning electron microscope analyzes were carried out under high vacuum, the completeness of the reaction and the elemental composition of the substances formed in parallel in the reaction were compared by taking 100 µm and 10 µm pictures (Fig. 3).

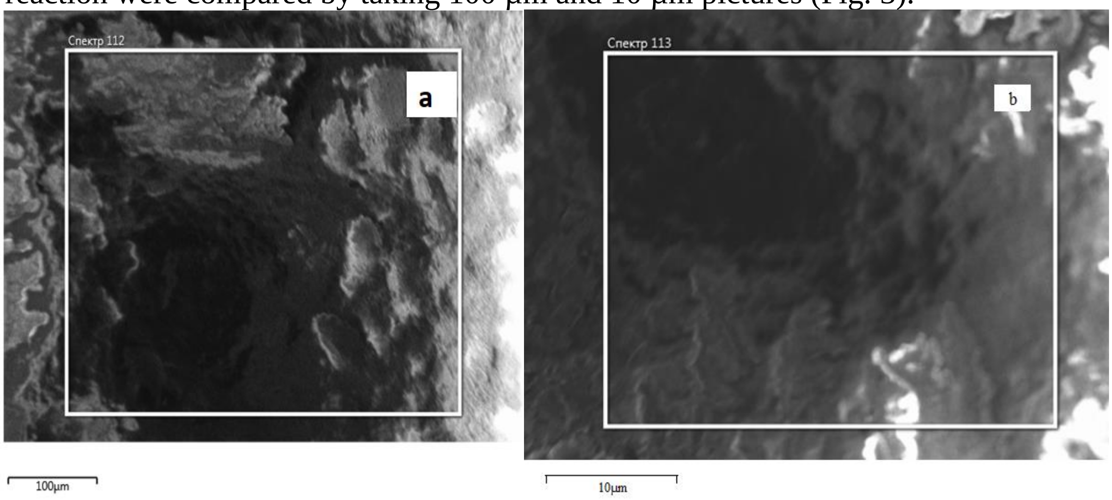

Figure 3. Appearance of modified bitumen taken by SEM (a-100, b-10 µm).

From the results of the analysis, it is clear that according to the images of modified bitumen, increased by 100 and 10 times, it is clear that the substances involved in reactions completely reacted and changed the composition of bitumen, the initial substances are completely combined with each other, and the substances show that the composition is as part there are no additives.

3.3. Elemental analysis of modified bitumen.

Elemental analysis of modified bitumen at 100 µm and 10 µm sizes was analyzed based on its SEM appearance (Fig. 4). From the results of the analysis, it can be seen that the percentages of carbon and sulfur, when comparing the elemental analysis of 100µm size with the elemental analysis of 10µm size, it can be seen that the amount of elements in 10µm size is higher than in 100µm size, so how small is the size of our sample. if there is, it can be seen that the amount of the elements in them increases.

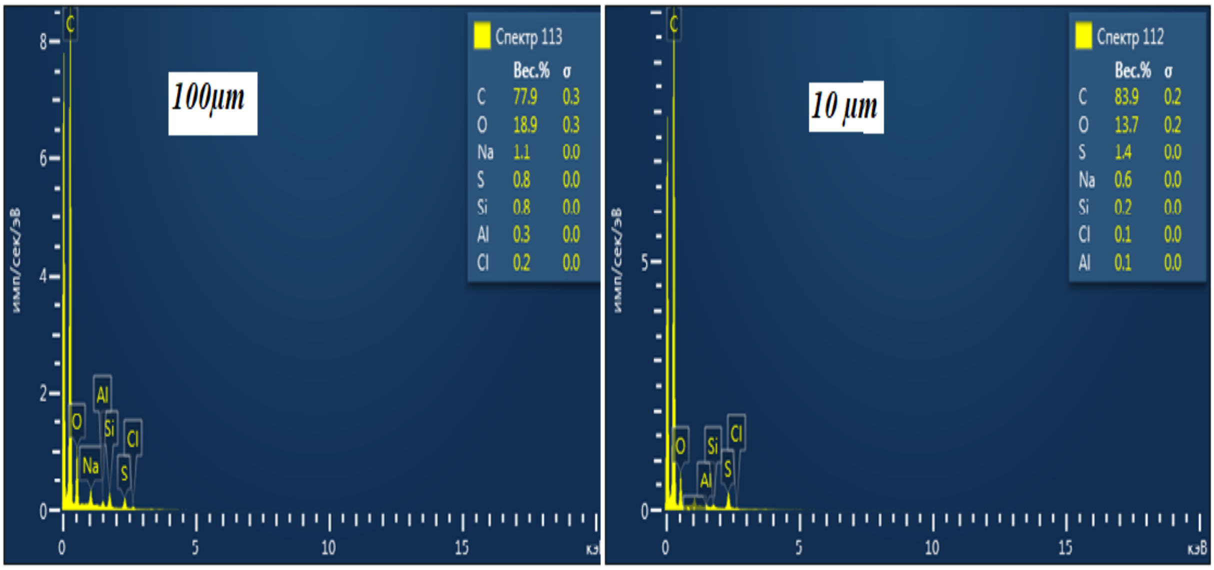

Figure 4. Elemental analysis of modified bitumen(a-100μm, b-10μm)

It can be seen in Table 1 that the percentages of the elements gave the result we expected

Table 1.

Elements and percentages of modified bitumen

Elements	***C***	***O***	***Na***	***Al***	***Si***	***S***	***Cl***
Mass. % 100μm	*83.9*	*13.70*	*0.64*	*0.11*	*0.17*	*1.38*	*0.12*
Sigma mass. % 100μm	*0.20*	*0.20*	*0.03*	*0.02*	*0.02*	*0.03*	*0.02*
Mass % 10μm	*77.95*	*18.86*	*1.05*	*0.32*	*0.79*	*0.80*	*0.23*
Sigma mass. % 10μm	*0.28*	*0.28*	*0.05*	*0.03*	*0.03*	*0.03*	*0.02*

3.4. Thermogrammetric analysis (TGA) and differential thermal analysis (DTA) of modified bitumen

Withdrew 16,136 mg from the modified bit, its TGA analysis was studied between 10-802 °C temperatures. Thermal oralism contains two endotheric effects, which was observed at the 60.87 and 722.83 temperatures. The curve line of the modified bitumon (TGA) curve was fulfilled between the temperature range where 3 intensive mass is lost. The first mass of the element of the curve to t to the tide line at a temperature of 20.39-191.54 °C, 75,54-458,35 °C, while the third mass loss interval is 458.33 K at 801.65 °C (Figure 5).

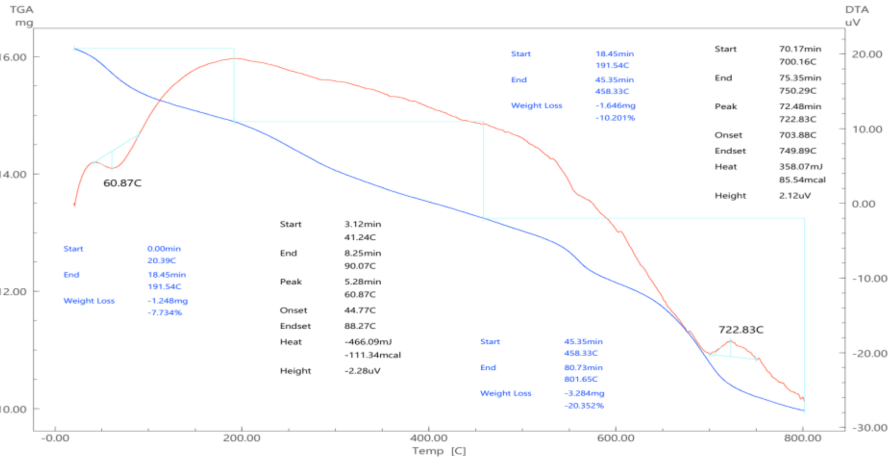

Figure 5. TGA and DTA Analysis of Modified Bitum

The results of thermogravimetric (TGA) and differential thermal analysis (DTA) of modified bitumen show that 7.734% mass loss can be observed in the 1st mass loss interval, 10.201% mass loss in the 2nd mass loss interval, and 20.352% mass loss in the 3rd mass loss interval. The results of differential thermal analysis of modified bitumen show that energy absorption occurs in the temperature range of 41.24-90.07 °C and 700.1-750.29 °C. Also, a more detailed analysis of the TGA and TDA curve results of this modified bitumen is presented in Table 2.

Table 2

The thermophravimmetrical (TGA) and differential thermal analysis of the modified bitumon (DTA)

№	*Temperature,°C*	*Lost mass, mg*	*Lost mass, %*	*The amount of energy consumed (µV*s/mg)*	*Residual mass, dw, (mg)*
1	100	15.3	0,836	5,18	11.49
2	200	14.8	1,336	8,27	19.28
3	300	14.05	2,086	12,9	16.3
4	400	13.5	2,636	16,3	12.8
5	500	13.02	3,116	19,3	8.7
6	600	12.14	3,996	24,7	-3.52
7	700	10.7	5,436	33,68	-20.2
8	800	9.9	6,236	38,6	-25,9

Study of the modified bitumen with the approval of rheological and depravity with JNR and R.

In studying the elasticity and viscosity properties of modified bitumen (SKMB), the loading frequency was analyzed as a function of temperature. The non-renewable part of bitumen modified with 1% to 6% rubber was determined by J_{nr} and R values, and its high elastic properties were studied. We obtained the temperatures as follows, i.e., the modification temperatures of fully mixed rubber and sulfur added to bitumen at 1350C, 155◦C, 180◦C and 200˚C and changes in J_{nr} and R under pressure from 0 to 90 kPa-1 Figures 6 and 7 show that modified bitumen in Figure 6 decreases to J_{nr} 0.1 and J_{nr} 3.8 kPa-1 at low temperature. As the temperature increases, both J_{nr} 0.1 and J_{nr}3,8 values increase, and Jnr3.2 is greater than Jnr0.1 for the same binder. The six Jnr0.1 ratings are 45SB-6K< 45SB-5K < 45SB-4K < 45SB-3K < 45SB-2K <45SB-1K and the order of J_{nr} 5.4 is the same as J_{nr} 0.1. The above results show that SKMB binders have high elasticity and 45SB-6K is the best. The switching rule R0.1 and R5.4 is opposite to Jnr0.1 and Jnr5.4 (Fig. 7). In addition, the ratings of R0.1 and R3.8 of the six binders vary in the order of 1RSMB < 2RSMB < 3RSMB < 4RSMB < 5RSMB < 6RSMB. From the above results, it can be seen that 6RSMB has a high elasticity, and then it decreases in sequence. The reason for this result is as follows: it can be assumed that the sulfur and natural rubber molecules are stabilized in the bitumen molecules by causing a certain degree of cross-linking reaction between the rubber and the pure bitumen. At the same time, it prevents the selfaggregation of rubber molecules, resulting in uniform distribution of natural rubber in the bitumen and improving the mechanical properties and elasticity of the RSMB binder [33].

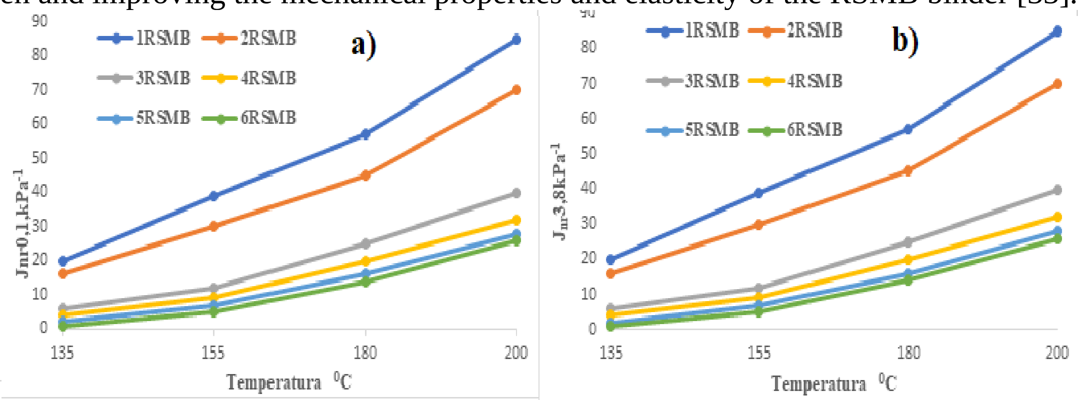

Figure 6. Jnr0.1 and Jnr3.8 of TBHB before aging: (a) Jnr0.1; (b) Figure 3.8. Jnr0.1 and Jnr3.8 of RSMB before aging: (a) Jnr0.1; (b) Jnr3.8

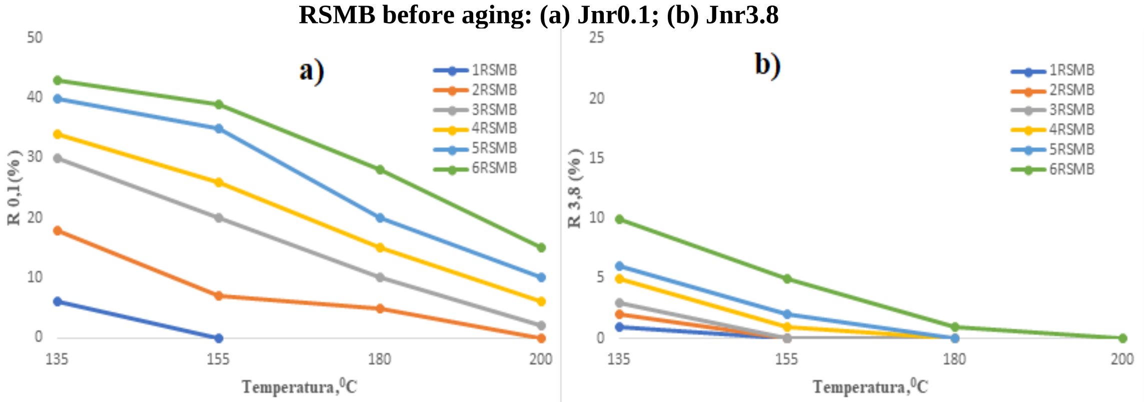

Figure 7. Jnr0.1 and Jnr3.8 of TBHB before aging: (a) Jnr0.1; (b) Jnr3.8. R0.1 and R3.8 of RSMB before aging: (a) R0.1; (b) R3.8

Overall, the effect of the modified bitumen binder on the complex modulus (G*) of the 1.5 Hz temperature versus the asphalt binder ratio is shown in Figure 6. In general, a high bond strength is desirable for high temperature coating applications. Figure 8 shows that the shear modulus of low-rubber bitumen is the lowest at low temperatures, while the G* effect of all binders decreases as temperature increases continuously (T>100°C) compared to other modified bitumen. In contrast to pure bitumen, isochronal plots of modified bitumen are more pronounced. G* reflects the strength and hardness of the material even at high temperatures. Figure 9 shows that the effect of modified bitumen on the phase angle depends on its elasticity at high temperature. The findings showed that the phase angle properties followed the same trend. The phase angle clearly shows that the addition of natural rubber improved the elastic behavior of the pure bitumen. The increase in elastic behavior at high temperatures can be related to the formation of a continuous rubber network structure that affects the properties of the binder when dispersed in the bitumen[34-36]. Greater flexibility at higher temperatures is beneficial to resist wear, cracking and damage. In general, when the phase angle approaches 90°C, the column is expected to be viscous and fully elastic when the phase angle value is 0°C.

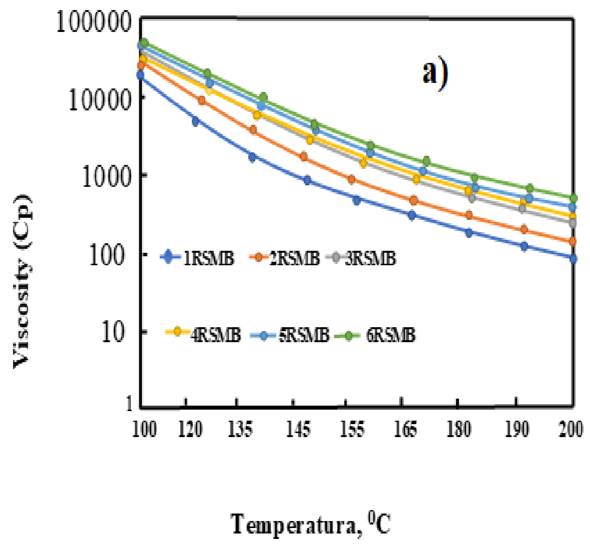

Figure 8. The complex module is as a function of temperature at 1.5 Gts

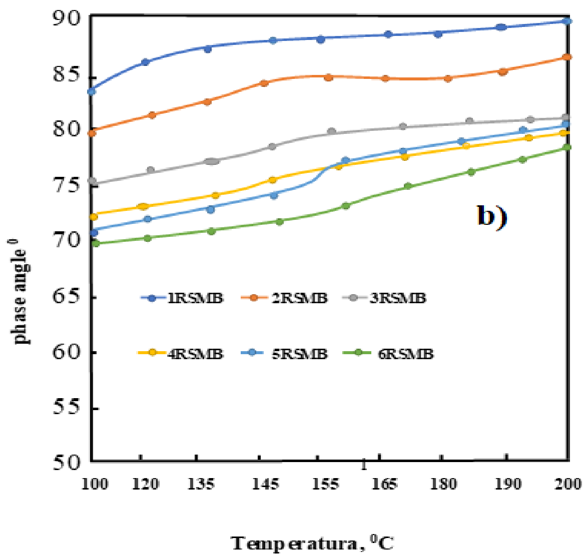

Figure 9. Phase angle as a function of temperature at 1.5 Hz

It was found that the addition of natural rubber to bitumen increased the wear resistance and elasticity properties. High values of shear modulus and low phase angle are important in determining the deformation characteristics [37-40].

4. Conclusion

The bitumen was synthesized with a new type of micropulation as a result of sulfur and natural rubber modification. Improves the composition and quality when we add the modified bitumen to the asphalt connector. The IQ-Spectrium, Scanning Electron microscopic tests, scanned electronic microskraphic analysis of the modified bitumen were analyzed and studied on the basis of thermomegrav nutrophy (TGA) and differential thermalmalm (DTA). The properties of rheological, obsolete bitumen and elasticity were studied. We observed the physical and chemical characteristics of the bituma when we add a natural rubber in the research work, and it was proved during analysis of the analysis of the fact that it is better to crack, cold, friction and heat. View the results of the analysis can be seen in the analysis of the physicochemical and mechanical properties of the modified bitumen, it was better to be better than the asphalt connector.

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