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Study of Modification of Butadiene-Styrene Rubber with Biopolymer

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Abstract

This paper presents a detailed study aimed at the development of a new composite system based on butadiene-styrene rubber (BSR) and natural biopolymer wooden stone. The main goal of the work was the creation of a thermodynamically compatible material with improved ozone-resistant and physical-mechanical properties, able to function effectively in aggressive conditions, especially in the liberated areas of the Republic of Azerbaijan, where the requirements for the stability of materials in extreme climatic conditions are relevant. To achieve the goal, theoretical and experimental studies aimed at studying the interaction of components in the composition of the new composition were conducted. For the first time, ecologically clean natural biopolymer wooden stone was used as a modifier, which allowed the reduction of the level of use of synthetic additives and increased the environmental safety of the finished product. Recipes were developed, including from 4 to 8 mass parts of biopolymer, which ensured the production of materials with high strength characteristics. The work has a high degree of novelty, practical significance and relevance, since it is aimed at creating modern, environmentally friendly and technologically efficient solutions based on a combination of industrial and natural raw materials.

Keywords: Binary system, biopolymer, melt flow index of the composition, modifications, vulcanization

INTRODUCTION

Synthetic rubbers have contributed greatly to many industries; nearly all are flammable in air. BSR is one elastomer widely used in the tire industry and conveyors for underground coal mining, carpet backings, automobile industries, various pipes, and oil- and petrol-resistant rubber for transporting oil and gas. In summary, the scope of polymers and their materials is expanding daily;, therefore, every year many scientists devote research to the production and use of polymer materials based on multi-tonnage polymers [1–6]. Among the multi-tonnage rubbers, a special place is occupied by butadiene-styrene rubber, but it has very poor properties in the raw state and poor resistance to thermal-oxidative degradation [7-11]. Many authors have devoted their work to eliminating these shortcomings. Strength in the raw state, which is the cohesion in a stretched non-vulcanized rubber mixture, is of great importance, especially when processing tires [12-16]. To eliminate these shortcomings, many works have studied the modification of butadiene-styrene rubber, but the desired result of these works has not been achieved Therefore, work on the modification of butadiene-styrene rubber is very relevant today [17-21].

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The authors of the works showed that the use of biopolymers as modifiers of synthetic rubber is an ecologically promising direction in the polymer industry, and in their works they used biopolymers such as lignin, chitosan, cellulose, and humid acids and showed that modifications with biopolymers exhibit pronounced sorption and rheological properties and are also capable of forming hydrogen bonds with the polymer matrix, which contributes to an increase in its thermal stability and mechanical strength [22-25]. In these works, the mechanisms of interaction of bio modifiers with rubber matrices were studied using complex instrumental methods - IR spectroscopy, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and scanning electron microscopy (SEM). This allows us to establish structural transformations and phase distribution in modified systems Research. by R.R. Guseynov showed that the introduction of natural components into the structure of styrene-butadiene rubber (BSR) leads to an increase in strength characteristics, a decrease in residual deformation, and an increase in the material's resistance to aggressive environments [26-27]. Basically, to increase the efficiency of biomodification, it is necessary to take into account a number of factors, including heat resistance, moisture content, degree of dispensability, and compatibility of additives with the main matrix. Therefore, it is recommended to use preliminary processing stages for this purpose - plasticization, surface activation, and chemical modification [28]. In this work, polymer mixing was used to establish an effective means for constructively changing the mechanical and swelling properties of polymeric materials. However, some of these polymer mixtures are not miscible. Incompatibility between blended polymers results in weak interfacial interactions between the different polymer phases and, therefore, produces a polymer blend with poor mechanical properties [29]. Of particular interest are local natural materials, such as “wood stone” from Azerbaijan, which is highly sought after. A very interesting study, in our opinion, is the work of the authors who used a modifier that they called ACM for modification of SBR. The mechanical properties of different rubber compounds were studied as shown in Figure 1. It shows that there is a gradual increase in tensile strength with the increase of ACM content in the rubber compound. It reaches a maximum at 60 phr ACM with an increase in tensile strength by 550%. In addition, the elongation at break is improved by almost 350% with the addition of ACM, as shown in Figure 1. This shows a significant improvement in the mechanical properties of the SBR/ACM composite compared to the SBR matrix without ACM. This improvement can be attributed to the homogeneous mixing and crosslinking of both polymers, which contributes to a better reinforcement of the rubber matrix during the vulcanization process, and the high compatibility achieved using the vulcanization system described above. The tensile strength and elongation at break of the polymer blends are synchronously improved compared to unmodified. BSR and ACM, which is very unusual for polymer blends.

The hardness (left) and crosslink density (right) of various BSR/ACM blends are shown in Figure 1. They are resistant and suitable for use as a bio modifier in synthetic rubbers. Figure 1. Hardness and density of the composition BSR/ACM

After studying the literature review, we are particularly interested in local natural materials, such as wood stone from Azerbaijan, which is characterized by high heat resistance and is suitable for use as a modifier for modifying synthetic BSR rubber .Thus, the transition to environmentally friendly biopolymers for modifying rubbers opens up new horizons in increasing the sustainability of rubber products and reducing their impact on the environment [28-34].

A laboratory roller mill was used to obtain rubber mixtures based on butadiene-styrene rubber and biopolymer. To obtain a homogeneous composition based on BSR and biopolymer, the mixing mode, temperature, and mixing time were determined, which were 130°C and 12 minutes. At high temperatures, displacement leads to the destruction of the polymer macromolecule. After determining the optimal mode for obtaining a composition based on BSR + biopolymer, a recipe was selected. In this recipe, the optimal composition of the components of the composition was determined, and the data obtained are presented in Table 1.

Table 1. Compositions based on BSR + biopolymer (GUM).

S.No.	Password indicators	1	2	3	4	5	66	7	8
1	BSR	100	100	100	100	100	100	100	100
2	Kaptaks	3	3	2	3	3	3	3	3
3	Oil PN-6	2,5	2.5	2.5	2.5	25	2.5	2.5	2.5
4	Altax	1,9	1.9	1.9	1.9	1.9	1.9	1.9	1.9
5	Stearin	5	.5	5	5	5	5	5	5
6	zinc oxide	5	4.0	4.0	4.0	4.0	4.0	4.0	4.0
7	Neozone “D”	5	5	5	5	5	5	5	45
8	Carbon black	30	30	30	30	30	30	30	30
9	Sulphur	3	2.0	2.0	2.0	2.0	2.0	2.0	2.0
10	Wooden stone(Gum)	-	3	4	5	6	7	8	10

Figure 1. General view of gum.

In this work, the styrene-butadiene rubber obtained by emulsion polymerization with a low content of the regulator had the following properties: rigidity (rigidity according to Defoe 20–35 N), Mooney viscosity (above 100 conventional units), and elastic recovery (elastic recovery according to Defoe 4–5 mm). To reduce viscosity and improve processability, styrene-butadiene rubber was subjected to thermal-oxidative destruction in an air environment at 130–140 °C under a pressure of 0.30–0.33 MPa for 35–40 min. In this case, their rigidity dropped to 3.0–4.5 N.

As a biopolymer, we used wooden stone, called gum. General appearance is shown in Figure 1.

We took gum from trunks of cherry and plum trees. In Novkhani Baglar, resin flow appears on these trees, a phenomenon called gum flow. Gum swells in water; its composition includes complex carbohydrates secreted by the tree. Wood resin is also called gum. This glue heals trees: when cracks appear on the trunk, resin drops appear on damaged areas. At first, resin is liquid, then turns jelly-like and hardens. Wooden stone consists of swelling polymers including monosaccharides—glucose, galactose, arabinose, rhamnose, and uronic acids.

Test Methods

The Flame resistance is measured using a Stanton Redcroft FTA flammability tester per ASTM D 2863–77. Tensile strength of vulcanized specimens is determined per ASTM D 412–92 using dumbbell specimens. Test specimens are punched from sheets using a type C punch. Specimen thickness within gauge length is measured using a dial indicator with 0.001 mm accuracy. Specimens are tested on a Tinius Olsen H10K instrument at 25 ± 2 °C and a crosshead speed of 500 mm/min.

Determination of swelling of the compound and rubber The resistance of rubber to oil is one of the key characteristics when choosing its end use, especially in seals, hoses, and automotive applications.

The solubility parameter (δ) is used as a measure of the polarity of a polymer, where the polarity increases as the solubility parameter increases. ACM has a higher solubility parameter value (δ = 18.6 J1/2/cm3/2) and therefore has a higher polarity than BSR (δ = 17.3 J1/2/cm3/2 IR spectra).

IR spectra are recorded using a Jasco Model No. 4100 FTIR spectrophotometer. UV spectra are recorded using a UV-550 spectrophotometer. 1HNMR spectra are recorded on a Bruker FT-NMR 250 MHz spectrophotometer. Differential scanning calorimetry (DSC) is performed at 5 °C/min. TGA is performed using a Shimadzu-50 analyzer at 10 °C/min in dry nitrogen. atmosphere.

Determination of Melt Flow Index

Determination of melt flow index Binary mixtures of BSR + wooden stone (gum) were prepared in different ratios; rheological properties under different loads (11.75, 20.85, 26.10, and 32.60 kg) and temperatures were studied on the IIRT-5 device. (Figure 2)

RESULTS OF EXPERIMENTAL STUDIES

To determine rheological properties, melt flow indices were determined. This allows determination of main processing parameters. Melt flow indices were determined at 160–180 °C. To study the binary composition based on BSR + gum, a mixture was prepared. The optimal formulation is in Table 2.

To determine the rheological properties of the composition, melt flow indices were determined in this work, melt flow indices were determined for samples number 1, 2, and 3 at temperatures of 160-180°C. The calculation was carried out as follows: Sample No. 1

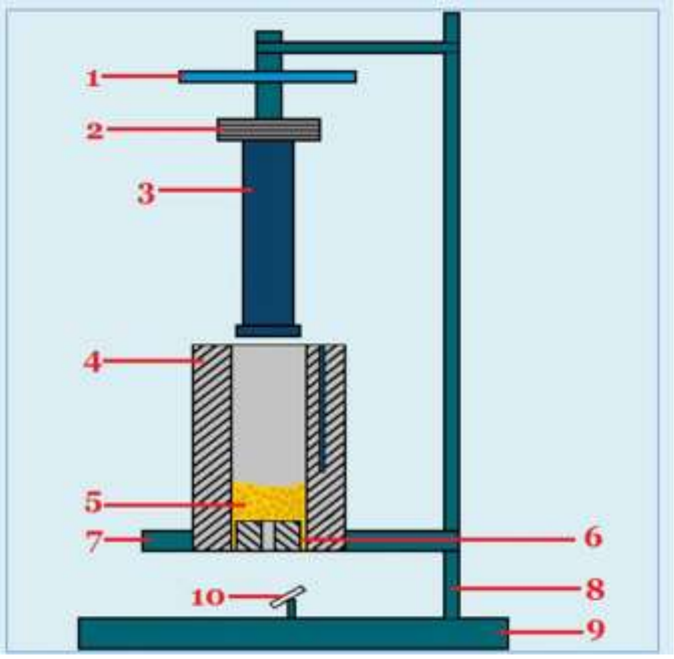

Figure 2. Scheme of the IIRT-5 device for determining the melt flow index.

1-Control lever, 2-Loads, 3-Piston, 4-Cylinder, 5-Polymer sample, 6-Capillary, 7-Support, 8-Shelf, 9- Container/Collector pan, 10- Cutting knife/Extrudate cutter

Table 2. Formulation of a composition based on styrene-butadiene rubber and wood-stone (gum).

S No.	Name of components	Contents gum sample 1	Contents gum sample 2	Contents gum sample 3	Contents gum sample 4	Contents gum sample 5
1	BSR	100	98	96	94	90
2	Gum	0	2	4	6	10

$\sqrt{\tau} = \sqrt{(2500.0)} = 50.00$
Q = V / t = (20001.000 мм^3) / (9.555 с) = 3363.291 мм^3/s

Simple No. 2
$\sqrt{\tau} = \sqrt{(2500.0)} = 50.00$
Q = V / t = (18370.000 мм^3) / (8.710 с) = 3689.581 мм^3/s

Simple No3
$\sqrt{\tau} = \sqrt{(2500.0)} = 50.00$
Q = V / t = (15150.000 мм^3) / (7.120 с) = 4513.518 мм^3/s

Simple No 4
$\sqrt{\tau} = \sqrt{(2500.0)} = 50.00$
Q = V / t = (11160.000 мм^3) / (4.928 с) = 6521.155 мм^3/s

In order to establish the regularities of processing the composition based on butadiene-styrene rubber modified with a natural biopolymer, preliminary laboratory tests were carried out. The tests included determining the dependence of the volumetric flow rate Q on the time of sample flow through a model nozzle at a fixed shear stress τ. To analyze the thermodynamic stability of the composition, the square root of the shear stress $\sqrt{\tau}$ was calculated, and the volumetric flow rate Q was calculated based on the mass of the material and the flow time. The calculations are presented for each sample separately; a graph of the dependence of Q on $\sqrt{\tau}$ was determined and shown in Figure 3. The dependence of Q on $\sqrt{\tau}$ results is shown in Figure 4. For the third sample, Q was also determined from t, and on the basis of the data obtained, we compiled schemes of the composition based on BSK and Gum. On the basis of the data, a figure was constructed, which is in Figure 4.

All the data obtained confirm that for the modification of styrene-butadiene rubber, it is sufficient to use no more than 4 mass parts of wood stone obtained from the spring tree Niuean bag depends Q on$\sqrt{\tau}$

To determine the modifying ability of wood stone, butadiene styrene rubber was investigated by infrared spectral analysis and pure polymer, and after modification of BSK, 4 mass parts of wood stone. The obtained X-ray analyses showed that by introducing into the composition 4 mass parts of wood, stone occurs modification of the polymer structure. Thus, it becomes possible to improve the physical and mechanical properties of butadiene styrene rubber, which must be obtained on the basis of the optimal composition of BSR + gum rubber materials. Because some butadiene styrene rubber does not allow us to obtain ozone-resistant and anticorrosive materials based on the composition of the presence of this rubber. Our studies have shown that it is possible by modifying this polymer with a biopolymer to improve its main disadvantages and get rubber that works in extreme conditions.

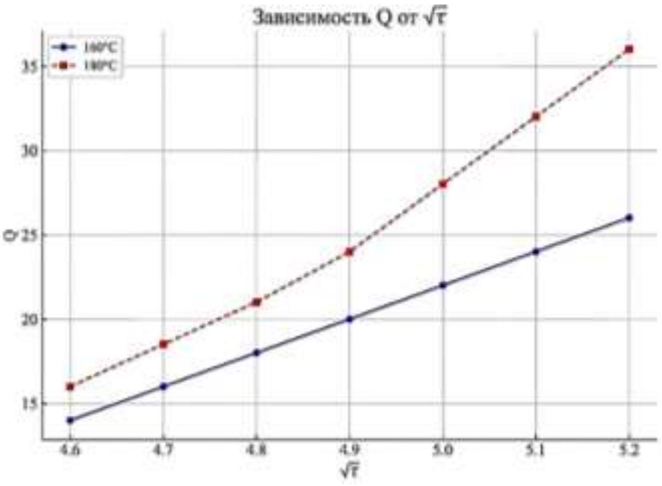

Figure 3. Dependence of volumetric flow rate (Q) on the root of shear stress ($\sqrt{\tau}$) at two temperatures.

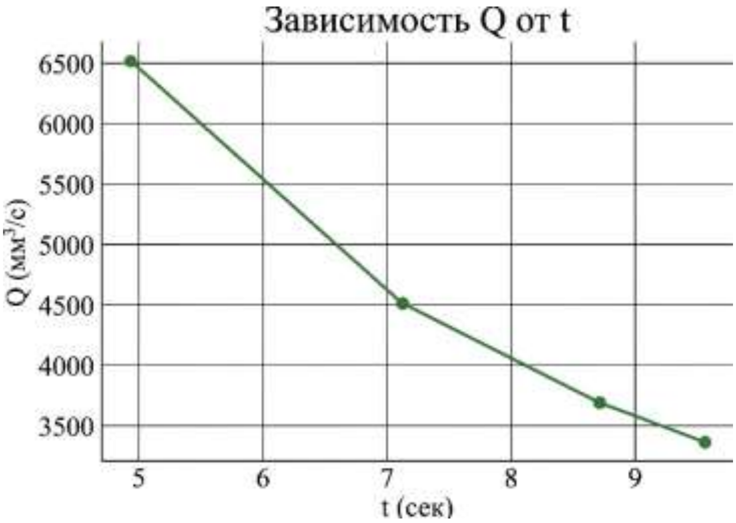

Figure 4. Dependence of volumetric flow rate (Q) on the flow time (t).

Based on the optimal composition of the BSR +Gum composite, their rheological properties and other moduli were investigated. The data obtained are shown in Figures 3 and 4. Accordingly, the loss modulus (E") is a sensitive indicator of molecular differences and is accepted as a very useful measure in failure studies. The peak in E" is observed at a different/alternative temperature compared to the peak observed in tan δ. Fluctuations in these values can be explained by their compatibility. Compatibility of the systems is achieved when BSK is 95% gum and 4-5%, while the change in the loss modulus (E") depends on the temperature of the mixtures and their mass parts. The data obtained as a result of calculations are shown in Figures 3 and 4. As can be seen from the figures, the peak of the loss modulus corresponds to the maximum heat dissipation per unit of deformation of the components. An important observation that can be made from the figure is that in the 4-6 parts by weight of gum mixtures, two distinct peaks can be observed, indicating the incompatibility and immiscibility of the gum and SBR phases at this mixing ratio above 6 parts by weight of gum. A broadening of the transition occurs in the case of partially compatible systems, which can be observed in all curves except for 0% Gum and 5% Gum. It is generally preferable to calculate Tg using the peak maximum from the tan δ curves shown in Figure 4 compared to the E' curves due to the highest uncertainty in the placement of the tangents compared to the exact determination of the tan delta peak apex.

Primary rheological structural analysis examined the behavior of the composition under the influence of temperature and time factors. The detected linear dependence of Q on $\sqrt{\tau}$ indicates the stability of the internal structure of the composition under various conditions, which indicates the compatibility of the components and the absence of phase separation. A uniform increase in Q with increasing temperature also confirms the stability of the structure with increased kinetic activity of macromolecules. Such data allow us to assert that the obtained composition can be used under conditions requiring stable flow behavior during processing. In this part of the study, a comprehensive analysis of changes in the physical, mechanical, and chemical properties of styrene-butadiene rubber with the introduction of various amounts of a natural biomodifier was carried out. Such parameters as tensile strength, elongation at break, Shore hardness, residual deformation, resistance to thermal aging, thermal stability, and microstructure of the samples were considered. The data obtained indicate that the biomodifier has a positive effect on a number of indicators, especially at a concentration of 6 mass parts. Improvements in thermal stability and ozone resistance were also confirmed, which allows us to recommend the developed compositions for use in aggressive external factors. Repeated tests confirmed the reproducibility of the results.

Study of the Vulcanization Process of the Composition Based on Wood Stone BSR+, the So-Called Resin

The vulcanization process was carried out at 158 °C for 24 minutes. Increasing wood resin amount affected strength, including ultimate strength and relative elongation (σ and ε) (Table 3).

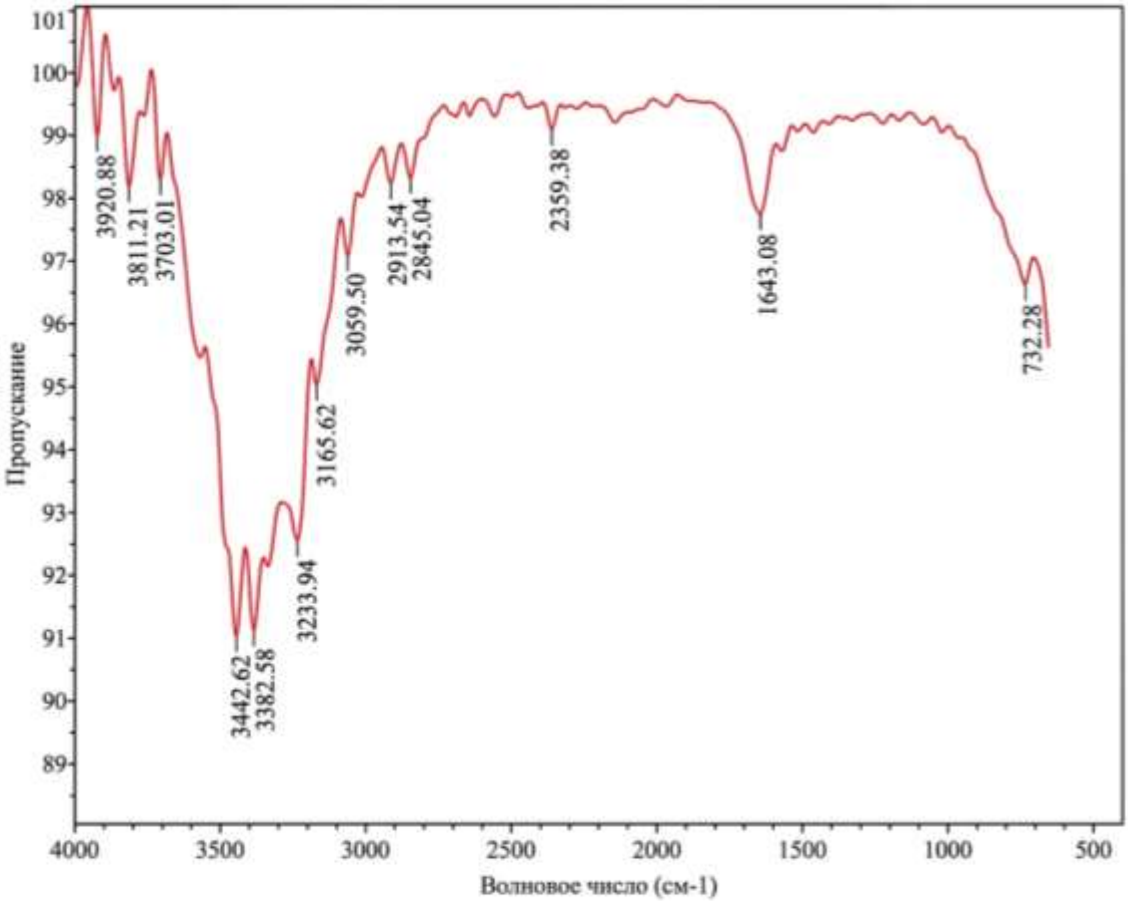

Figure 5. IR spectra of pure polymer.

It has been established that with an increase in the content of gum in the composition from 4% to 6%, the highest strength value is recorded in the gum ratio of 4.0:6.0% (12.9 MPa). At this time, a decrease in relative elongation (ε) is observed from 580% to 26%, which is characterized by a decrease in the modulus of elasticity. The study of the effect of dispersion on the nature of the hardness of tensile samples leads to a decrease of 10 times, and the introduction of 6.0% DK leads to a decrease of 18-20 times. The results obtained are presented in Table 4. The physical and mechanical properties of the composition based on BSR + Gum showed that heat resistance, fluidity, and other strength properties of the obtained rubber increased in comparison with the standard by 1.5 times. It determines a number of effects arising in connection with the dressing on the surface of the molecules of the studied fillers. Thus, the observed changes in the properties of polymer compositions are due to the slowing down of the movement of particles of the solid filler of macromolecules between phases. They were recorded during the study of the chemical resistance of their materials. Some results obtained during the studies are given in Table 4.

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Table4The influence of the nature of wood stone on the physical and mechanical properties of BSR compositions

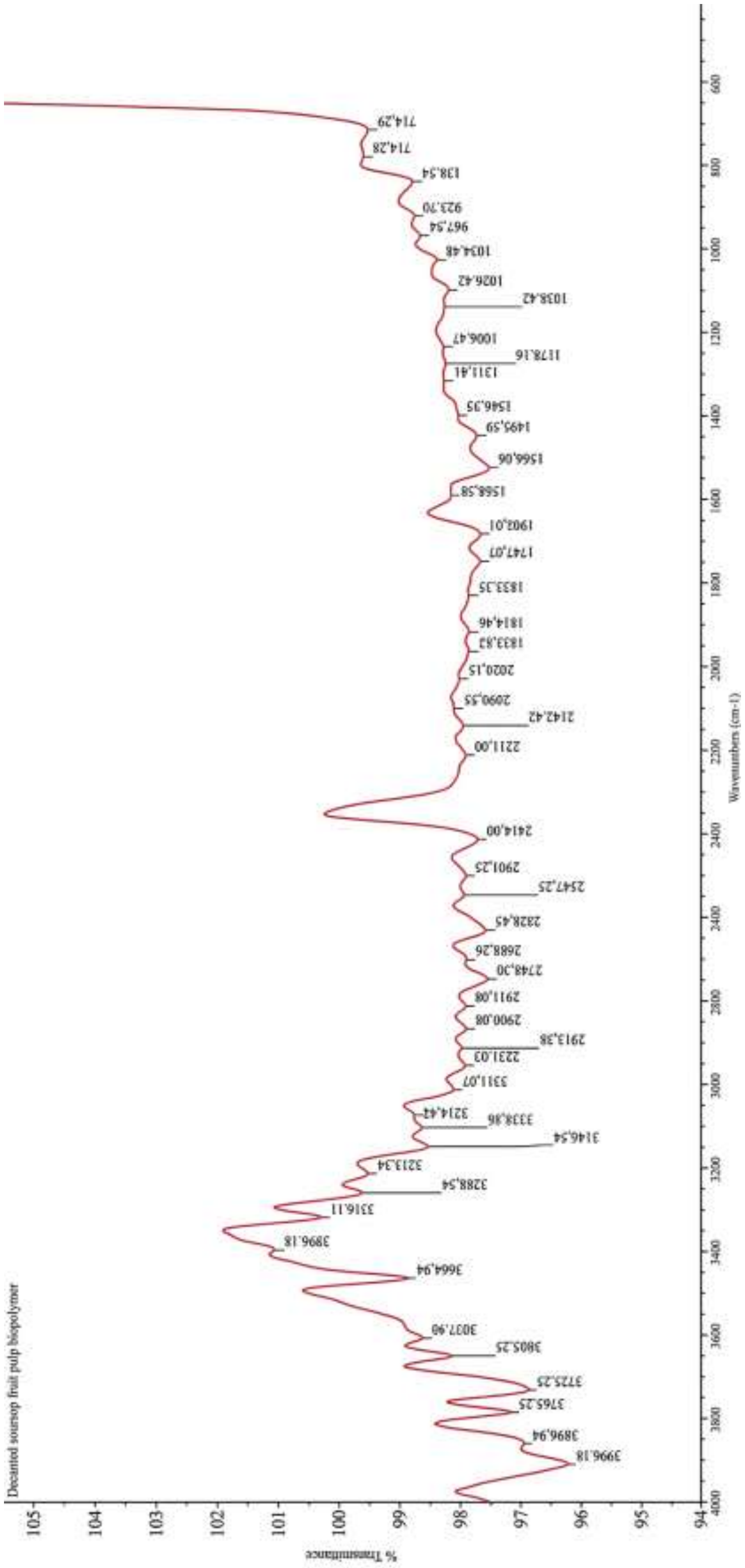

Figure 6. IR spectra of modified 5 mass parts of BSR + Gum.

Table 3. Physical and mechanical properties of compositions based on BSR-GUM %.

Row number	Indicators	Resin content, mass %					
		10	20	30	40	50	60
1	Tensile strength, σ, MPa	8.9	9.5	10.3	10.8	11.5	13.9
	Relative elongation, ε,%	40.0	33.0	32.0	30.0	28.0	26.0
2	Tensile strength, σ, MPa	8.2	9.2	9.5	10.2	10.8	12.3
	Relative elongation, ε,%	45.0	55.0	50.0	42.0	38.0	30.0
3	Tensile strength, σ, MPa	8.0	8.2	8.5	8.9	9.3	12.9
4	Relative elongation, ε,%	48.0	43.0	40.0	38.0	35.0	32.0
5	Tensile strength, σ, MPa	8.0	8.2	8.5	8.8	9.0	12.5
6	Relative elongation, ε,%	55.0	52.0	50.0	45.0	40.0	35.0
7	Tensile strength, σ, MPa	7.9	9.2	9.2	10.5	12.7	13.3
8	Relative elongation, ε,%	52.0	45.0	46.0	40.0	43.0	38.0
9	Tensile strength, σ, MPa	10.9	-	9.4	11.4	12.2	14.8
10	Relative elongation, ε,%	35.0	-	21.0	25.0	20.0	21.0

Table 4. Effect of GUM on the physical and mechanical properties of BSR. GUM on the physical and mechanical properties of BSK.

Amount of wood resin, mass %						
0	**10**	**20**	**30**	**40**	**50**	**60**
0.85	1.12	1.45	1.34	1.30	1.51	1.85
	0.91	1.1	1.13	1.20	1.25	1.33
4.62	4.45	3.35	2.85	1.55	Нет	Нет
	1.40	31.45	1.60	0.45	1.16	0.48
105	108	120	126	132	136	140
	106	110	112	123	125	128
9.8	10.2	11.8	13.5	15.3	16.2	17.5
	9.5	10.6	11.5	12.2	13.1	14.6
	205	95	85	48	35	11.2
	225	105	92	54	41	16

Table5. The influence of the nature of wood stone on the physical and mechanical properties of BSR compositions.

Row number	Particle size, μm	50-63	100-160	160-200	200-315
	Amount of GUM, %	3,0	4,0	5,0	6,0
1	Tensile strength, σ, MPa	9.5	10.8	11.2	12.0
2	Modulus of elasticity, εeff 10-2	12.1	14.0	16.1	18.0
3	Relative elongation, ε, %	95	55	40	38
4	Crystallization rate, %	45	46	52	52
5	Tensile strength, σ, MPa	10.5	10.8	11.2	12.0
6	Modulus of elasticity, εeff 10-2	10.2	13.2	14.5	16
7	Relative elongation, ε, %	45	39	35	30
8	Crystallization rate, %	46	45	47	53
9	Tensile strength, σ, MPa	12.8	13.5	14.2	16.0
10	Modulus of elasticity, εeff 10-2	10.7	11.5	12.8	14
11	Relative elongation, ε, %	97	42	41	38
12	Crystallization rate, %	51	52	48	53

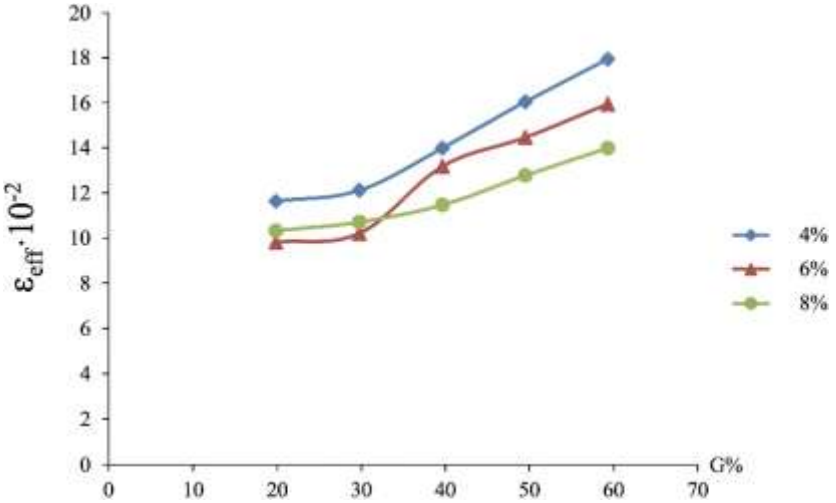

Figure 7. Dependence of the tensile strength on the amount of gum.

We studied the dependence of the rubber tensile strength on the amount of wood stone (GUM), and the results obtained are shown in Figure 7.

In the composition in all three cases with the highest content of GUM (6.0%), the amorphousness of the composition increases, and the rate of crystallization of BSR decreases. It turned out that one of the main factors increasing a number of physical and mechanical properties of the compositions is that the GUM particles have a spherical shape. This shape also increases the effect of interphase mutual adhesion in the composition and reduces the crystallization rate. The obtained parameters show that with an increase in the amount of GUM to 6.0%, the strength increases, with a decrease in relative elongation, and elasticity decreases.

RESULT

1. In this work, the main objective of the study was achieved - a new composite system based on butadiene-styrene rubber modified with natural biopolymer GUM was developed and characterized.
2. The experimental work confirmed that the inclusion of 4-8 mass parts of biopolymer in the rubber structure leads to a significant improvement in the physical and mechanical properties of the resulting material.
3. As a result of serial tests, the optimal manufacturing parameters were determined: a temperature of 130-160°C and a vulcanization duration of 15-20 minutes. .
4. The work has both theoretical significance - Thus, the obtained results can serve as a basis for further scientific and industrial developments in the field of modifying elastomers with natural components.

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