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SCIENTIFIC FOUNDATIONS FOR DEVELOPING A NEW GENERATION OF INSULATING MASTICS USING GOSSYPOL RESIN

Yuldasheva Kh.Sh.
Bekberganova D.D.
Urgench State University, Urgench

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ABSTRACT

The origin of the problem. The processing of industrial waste and obtaining secondary products from them are crucial aspects of the national economy. Therefore, deriving bituminous mastics from gossypol tar, a byproduct of the oil industry, contributes to addressing waste-related issues. This approach underscores the significance of efficiently managing industrial waste to yield valuable secondary products, aligning with economic and environmental considerations.

Purpose of work. Title: Scientific Rationale for the Production of Next-Generation Insulating Mastics Utilizing Gossypol Resin.

Methodology. The research employed physical, physico-chemical, and analytical research methods to analyze the characteristics of raw materials and the resulting products.

Scientific Innovatio. Pioneering studies were conducted for the synthesis of oil-free mastics based on gossypol. To enhance the heat resistance and hardness of this novel mastic, as well as mitigate its brittleness, research investigated the impact of mineral fillers on the composition.

The results obtained. A series of studies aimed at determining the optimal composition of insulating mastics based on Gossypol resin were carried out. The incorporation of SaO into the composition revealed a reduction in the depth of needle penetration, accompanied by increased adhesion and elasticity of KMTs.

Characteristics. Performing Infrared (IR) spectroscopic investigations involved analyzing absorption spectra of primary components and examined compounds using a Shimadzu IRT Tracker100 from Japan. Wavelengths were determined within the range of 4000-400^{-1} sm, and samples were prepared in a presslab with KBr. The observed chemical impact of the mastic suggests that it can influence the chemical properties of its composition in an appropriate proportion.

Introduction. Mastics play a crucial role in the manufacturing of ruberoid, willow, pergamine, as well as in the insulation of building foundations, roofing, basements, and

swimming pools. The performance of waterproof mastics designed for roofing applications is influenced by various natural phenomena such as rain, snow, hail, wind, ultraviolet rays, and temperature fluctuations. Additionally, the mechanical and biological impacts of microorganisms contribute to the aging process of bituminous mastic compounds [1; b 159].

Cold bitumen and bituminous rubber mastics are typically produced centrally. The varnish is mixed with the filler for 8-10 minutes, and dewatered bitumen alloy heated to a temperature of 170-180 °C is gradually added to the mixer while continuously mixing. Subsequently, all components are mixed for 4-5 minutes until a homogeneous mass is achieved. The prepared mastic is then transferred to the collector using pumps. Quality control of raw materials for mastic preparation, as well as the finished mastic, is conducted either at the factory or in construction laboratories where work is in progress [2; b. 555., 3; b. 35., 4; b. 281-301].

In modern construction, mastics find extensive use for various purposes and compositions, including the coverage of building and structure roofs. Waterproof mastics (SHM) generally exhibit high levels of water, abrasion, and heat resistance. Their interaction with metal, concrete, and polymer materials demonstrates a high degree of adhesion (from Latin adhaesio — prilipanie = stickiness). Notably, they are characterized by ecological cleanliness, biostability, fire resistance, and long service life. Unique properties of mastics include the ability to apply them on wet and untreated surfaces and create coatings of different colors by incorporating dyes into their composition. In the author's previous research, conducted by Khozeev E.O., the interrelationship between the composition and properties of SHM, methods of obtaining bituminous compositions, and directions of their modification were explored [5; p. 39-42, 6; p. 159-162, 7; p.107-108].

Methods and materials. The main components of the theoretically and practically researched work include: Gossypol resin - a by-product of the cottonseed oil industry, calcium oxide, rubber crumbs, talc, basalt, curing agent, asbestos, and *carboxy methyl cellulose*.

Gossypol resin is inherently complex in nature, containing phenolic, hydroxyl group-containing aromatic compounds, and medium carboxyl groups. The composition of Gossypol resin includes phenol, aldehyde, carboxyl, carbonyl, and hydroxyl groups. However, these groups are not active under ordinary conditions. We have the ability to chemically activate Gossypol resin through thermal processing. Thermally activated Gossypol resin exhibits acidic properties and readily undergoes reactions with other reagents, forming simple and complex ethers, aliphatic amines, phosphatides, neutral salts, and other compounds.

The quantity of water not bound according to the requirements of GOST 18-114-73 in the composition of Gossypol resin is specified to be less than 1-2%. However, in the tar produced, the water content can be up to 5-10%. The presence of this water is crucial for the formation of the mastic, as it contributes to the polymerization process. Therefore, the removal of water from the composition before oxidation plays a significant role in the mastic production process.

Results and discussion. Materials for Ensuring the Stability of Constructions and Buildings in Various Natural and Climatic Zones: The use of constructions and materials in different natural and climatic zones requires the utilization of materials that respond to diverse conditions to ensure the stability of structures and constructions. Consequently, extensive research is conducted to obtain construction materials with enhanced characteristics. For

instance, traditional methods involving various sealants and mastics are employed in roofing and waterproofing works. The most widely used type of mastic, based on bitumen derived from oil, forms the fundamental component of polymer-bitumen compositions. The objective of this study is to consolidate scientific concepts related to the technology of obtaining bitumens, focusing on their basic composition and the interrelation of their properties.

In the operational lifespan of buildings and structures, roofing and waterproofing coatings are exposed to atmospheric phenomena such as rain, snow, frost, wind, ultraviolet radiation, and temperature fluctuations. Furthermore, the mechanical loads on coatings and the biological effects caused by various microorganisms also play a significant role in the aging process of bituminous mastic compounds, as emphasized by scholars like Yartsev V.P. and Erofeev A.V [1; p.80].

Researcher Khozeev E.O. has developed a conceptual framework for obtaining bitumen for construction mastics. Consequently, technologies for obtaining high-quality, oxidized, and compounded bitumens, as well as technological processes that contribute to the customization of bitumen properties, have been examined. These technologies take into account the physical-mechanical characteristics of bitumens, their resilience to climatic influences, and their durability. Thus, the development of technologies for obtaining bitumens is essential to ensure the required durability of construction mastics, as plain bitumens alone cannot provide the necessary properties. Therefore, further research is needed to create various modified and additive bitumen composites that meet the requirements of large-scale roofing and waterproofing coatings, to enhance their durability and adaptability to diverse climatic conditions [8].

Researchers T.L. Lazareva and N.I. Yarmolinskaya have developed new compositions used for repairing roads with bituminous mastics. For the preparation of polymer-bitumen mastics, carbonate crumbs dried at 105 °C and road bitumen of the BND 90\130 grade are heated to 160 °C in laboratory conditions. Carbonate crumbs are gradually introduced into the polymer-bitumen composition and mixed for 15-20 minutes at an estimated temperature of 169-180 °C. This mastic is then employed for road repairs [9; p. 38-39].

Scientist L.S. Sibgatullina, along with several other researchers, has addressed the challenges of creating long-lasting, environmentally friendly roofing and waterproofing materials. They proposed modifying bitumen and bitumen-polymer emulsions by reprocessing materials from the felt and roofing bitumen industry. The improvement of bitumen mastic properties involves suggesting a composition with a multifaceted impact mechanism. For this purpose, talcomagnesite has been introduced as a filler for bitumen mastics, and rubber has been utilized to optimize the distribution of particles. Moreover, dispersed emulsions with uniform distribution have been created. Bitumen mastics obtained from bitumen and bitumen-polymer emulsions have demonstrated optimal characteristics [10; p. 38-39].

Researcher V.S. Sokhadze has conducted scientific work on polymer-bitumen mastics. He has formulated guidelines for the preparation of polymer-bitumen mastics, which include the following components: butadiene-styrene thermoelastoplastic, talc as a filler, a plasticizer, and PN-6K petroleum. The mass ratios are as follows: bitumen 40.0-60.0%; butadiene-styrene thermoelastoplastic 2.0-14.0%; various dibutyl phthalate plasticizer 1.0-4.0%; PN-6K petroleum plasticizer 1.0-4.0%; and other components [11; Patent RU 2 345 107 C1].

The scientific sources mentioned above provide unique contributions and perspectives on various aspects. Nevertheless, their practical applications in industrial production have not been fully realized.

This article, based on gossypol resin and local resources, highlights the scientific foundations of obtaining insulation mastics that meet standard requirements.

In the initial stages of creating new types of oil-free mastics based on gossypol resin in the cottonseed oil industry, research was conducted to thermally oxidize and activate functional groups, followed by investigations to increase the density of the composition by introducing CaO. Experimental work was carried out by adding CaO to gossypol resin in the range of 0.5-2.5%. The temperature reached 220 °C, the reaction time was 180 minutes, and the stirring intensity was 120 times per minute, resulting in a mass loss of 5.6%. When treated with 2.5% CaO, the temperature reached 220 °C, the reaction time was 220 minutes, and the stirring intensity was 120 times per minute, resulting in a mass loss of 6.9%.

The research aimed at the creation of new types of oil-free mastics based on gossypol resin in the cottonseed oil industry proceeded through the initial stages of thermal oxidation and activation of functional groups, followed by experiments to increase the density of the composition by introducing CaO. Experimental work involved adding CaO in the range of 0.5-2.5% to gossypol resin. The temperature reached 220 °C, the reaction time was 180 minutes, and the stirring intensity was 120 times per minute, resulting in a mass loss of 5.6%. When treated with 2.5% CaO, the temperature reached 220 °C, the reaction time was 220 minutes, and the stirring intensity was 120 times per minute, resulting in a mass loss of 6.9%.

Table 1.

The results of studies aimed at determining the effect of CaO on the needle penetration depth of mastic based on gossypol resin are as follows

№	Amount of CaO, %	Gossypol resin %	Total mass, g	Temperature, °C	Mass loss,%	Reaction time, min	Mixing intensity, min	Needle penetration depth
1	0,5	99,5	250	220	5,6	180	120	145
2	**1,0**	**99,0**	**250**	**220**	**6,0**	**190**	**120**	**120**
3	1,5	98,5	250	220	6,4	200	120	110
4	2,0	98,0	250	220	6,8	210	120	105
5	2,5	97,5	250	220	6,9	220	120	90

In this process, 2% CaO was adopted as the optimal composition, the reaction time was 210 minutes, the temperature was 220 °C, and the mass loss was 6.8%. The needle penetration depth met GOST requirements when adding CaO 2%.

Further research is aimed at increasing the acid resistance of mastic based on gossypol resin. Here, the test results to determine the acid resistance of mastic in 20% sulfuric acid are presented in the table below (Table 2).

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Table 2.

Research aimed at increasing the acid resistance of mastic based on gossypol resin and the test results in sulfuric acid (20%).

№	Gossypol resin, %	Amount of urotro-pin, %	Total mass, g	Tempe-rature, °C	Mass loss, %	Reaction time, min	Mixing intensity min	Acid resistance, % (in the case of sulfuric acid)
1	**99,5**	**0,5**	**200**	**70**	**0,75**	**60**	**120**	**98,5**
2	99,0	1,0	200	70	1,0	65	120	98,6
3	98,5	1.5	200	70	1,5	70	120	98,7
4	98,0	2.0	200	70	1,5	75	120	98,7
5	97,5	2.5	200	70	1,75	80	120	98,7

Research aimed at increasing the acid resistance of mastic based on gossypol resin consists of the following. As can be seen from the results of the table, experiments were conducted by adding 0.5-2.5% $(CH_2)_6N_4$ to gossypol resin. When adding 0.5% $(CH_2)_6N_4$, the temperature was 70 °C, the reaction time was 60 minutes, the intensity of mixing was 120 cycles per minute, and the mass loss in this process was 0.75%. When exposed to 2.5% urotropin, the temperature was 70 °C, the reaction time was 80 minutes, and the intensity of mixing was 120 times per minute, and the mass loss was found to be 1.75%. In this process, 0.5% urotropin was obtained as the optimal content.

The results of studies aimed at determining the relative elongation coefficient of mastic obtained on the basis of gossypol resin are given in the table below. In this work, rubber powder was used as a plasticizer and its effect on gossypol resin was studied. Based on it, the table shows the amount of substances, temperature, intensity of mixing, reaction times and coefficients of relative elongation.

Table 3.

The results of studies aimed at increasing the coefficient of relative elongation of mastic.

№	Amount of rubber powder, %	Gossypol resin, %	Total mass, g	Tempe-rature, °C	Mixing intensity min	Mass loss, %	Reaction time, min	Stretcha-bility, mm
1	1,0	99,0	200	60	120	0,5	30	33

2	**2,0**	**98,0**	**200**	**60**	**120**	**1,0**	**35**	**37**
3	3,0	97,0	200	60	120	1,5	40	38
4	4,0	96,0	200	60	120	1,5	45	39
5	5,0	95,0	200	60	120	1,6	50	39

In this work, aimed at increasing the relative elongation coefficient of mastic, rubber powder was added to gossypol resin in proportions of 1-5%. In this case, 1% rubber powder was included at a temperature of 60°C. It was determined that the reaction time was 30 minutes, the mixing intensity was 120 revolutions per minute, and the mass loss was 0.5%. When adding 2% rubber powder to the composition, the temperature was 60°C, and the reaction time was 35 minutes. When adding 5% rubber powder, the temperature was 60°C, the reaction time was 50 minutes, and the mass loss was 1.6%. As a result of experimental studies, 2% rubber powder was considered optimal.

To enhance stickiness, gossypol resin was treated with carboxymethylcellulose. The influence of carboxy methyl cellulose on the composition is crucial for determining the adhesion of the mastic that meets the requirements of GOST (Table 4).

Table 4.

Results of studies aimed at increasing the adhesion of mastic based on gossypol resin

№	Amo-unt of CMC, %	Gossypol resin %	Total mass, g	Tempe-rature, °C	Mixing intensity min	Mass loss, %	Reaction time, min	Change in adhesion,%
1	1,0	99,0	200	220	120	0,25	60	98
2	**2,0**	**98,0**	**200**	**220**	**120**	**0,5**	**65**	98
3	3,0	97,0	200	220	120	0,75	70	98
4	4,0	96,0	200	220	120	1,0	75	98
5	5,0	95,0	200	220	120	2,25	80	98

To enhance the adhesion of gossypol resin-based mastic, 1.0-5.0% carboxymethylcellulose (CMC) was incorporated into the composition. The addition of 1% CMC occurred at a temperature of 220°C, with a reaction time of 60 minutes and a mass loss of 0.25%. This study determined that 2% CMC was the optimal content, meeting the adhesion requirements specified in GOST 15140-78.

In the pursuit of increasing the heat resistance, hardness, and reducing the brittleness of oil-free mastics, investigations were conducted to examine the influence of mineral fillers on gossypol resin. Over time, bituminous mastic experiences deterioration due to exposure to various atmospheric phenomena such as rain, snow, lightning, wind, and ultraviolet rays, along

with temperature changes. To mitigate this phenomenon, asbestos was introduced into the composition. The effects of asbestos, a mineral filler, on gossypol tar, are detailed in the table below.

Table 4.

Research on the Influence of Mineral Filler Asbestos on Gossypol Resin

№	Amount of asbestos %	Gossypol resin %	Total mass, g	Temperature, °C	Mixing intensity, min	Mass loss, %	Reaction time, min	Dielectric resistance, not less than kv/mm
1	1,0	99,0	200	220	180	0,5	70	5
2	**2,0**	**98,0**	**200**	**220**	**180**	**0,5**	**75**	**5**
3	3,0	97,0	200	220	180	0,75	80	5
4	4,0	96,0	200	220	180	0,75	85	5
5	5,0	95,0	200	220	180	1,0	90	5

According to the information provided above, the results table of the research indicates that experimental studies were conducted by incorporating 1.0-5.0% asbestos into gossypol resin. The addition of 1.0% asbestos was carried out at a temperature of 220°C, with a reaction time of 70 minutes, mixing intensity of 120 times per minute, and a determined mass loss of 0.5%. When 5% asbestos was introduced, the temperature was 220°C, and the reaction time extended to 90 minutes, with a mixing intensity of 120 times per minute, resulting in a mass loss of 1.0%. Throughout this process, 2% asbestos was identified as the optimal content, with a reaction time of 90 minutes, a temperature of 220°C, and a mass loss of 1.0%. The selection of 2% asbestos was based on the observation that there was almost no change in dielectric resistance.

In our subsequent research, we investigated the impact of kaolin, a mineral filler, on gossypol resin. Fillers are introduced into gossypol resin to enhance the mastic's heat resistance, and hardness, and reduce brittleness. In this case, kaolin was included in the composition to specifically address the reduction of mastic brittleness and the enhancement of its flexibility. The detailed results can be found in the table below (Table 6).

Table 6

The Results of the work on the Impact of Kaolin to Reduce the Brittleness of Mastic and Increase Its Elasticity

№	Amount of Kaolin, %	Gossypol resin, %	Total mass, g	Tempera-ture, °C	Mixing intensity, min	Mass loss, %	Reaction time, min	Flexibility mm. -15 °C
1	1,0	99,0	300	220	120	1,0	70	6,0
2	**2,0**	**98,0**	**300**	**220**	**120**	**1,5**	**75**	6,5
3	3,0	97,0	300	220	120	1,7	80	6,6
4	4,0	96,0	300	220	120	1,8	85	6,7
5	5,0	95,0	300	220	120	2,0	90	6,7

In the research, they conducted experiments by adding 1.0-5.0% kaolin into gossypol resin. They found that when 1.0% kaolin was added, the temperature reached 220°C, the reaction time was 70 minutes, and the mixing intensity was 120 times per minute, resulting in a mass loss of 1.0%. With 5.0% kaolin exposure, the temperature remained the same, but the reaction time extended to 90 minutes, and the stirring intensity remained at 120 times per minute, leading to a mass loss of 2.0%. They concluded that the optimal composition was 2% kaolin, with a reaction time of 75 minutes, a temperature of 220°C, and a mass loss of 1.5%.

They also explored further research to enhance the mastic's resistance to high temperatures, and Table 7 presented their findings. They introduced basalt to gossypol resin under various conditions, with the color being gray or dark. To evaluate the heat resistance of the gossypol resin-based mastic under the influence of basalt, they compared it against the standards outlined in GOST.

Table 7.

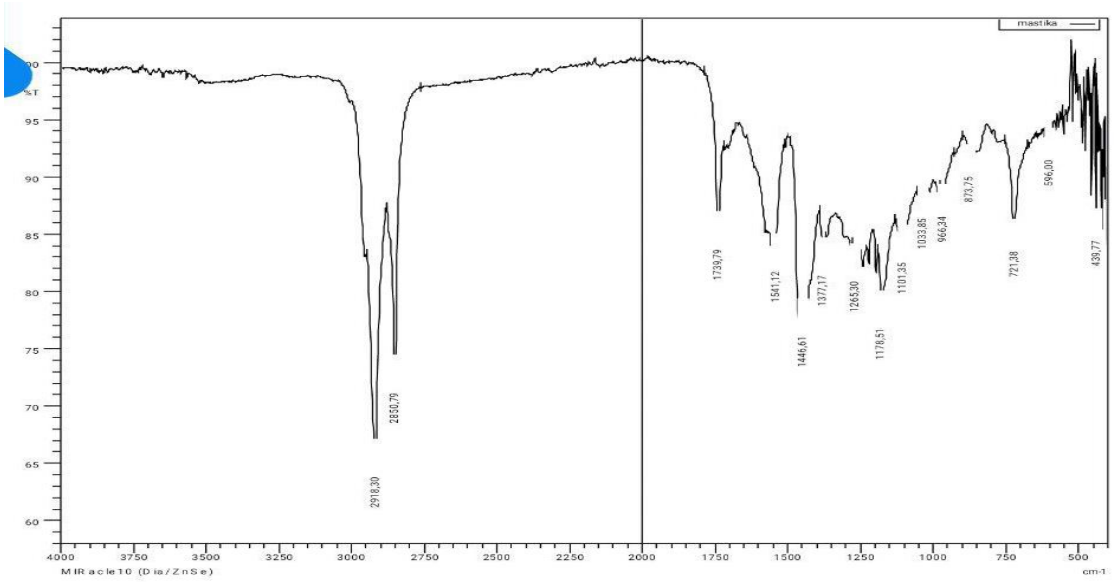
The outcomes of investigations focused on enhancing the mastic's resistance to high temperatures under the influence of basalt

№	Basalt amount, %	Gossypol resin, %	Total mass, g	Stirring intensity, min	Mass loss, %	Reaction time, min	Heat resistance, °C
1	1,0	99,0	200	120	0,5	60	85
2	**2,0**	**98,0**	**200**	**120**	**1,0**	**65**	88
3	3,0	97,0	200	120	1,0	70	89
4	4,0	96,0	200	120	1,25	75	89
5	5,0	95,0	200	120	1,5	80	90

As evident from the table results, experimentation involved incorporating 1.0-5.0% basalt into gossypol resin. With the addition of 1.0% basalt, the process operated at 220 °C, a reaction time of 60 minutes, a mixing intensity of 120 times per minute, and a mass loss of 0.5%. When exposed to 5.0% basalt, the temperature was 220 °C, the reaction time was 80 minutes, and the mixing intensity remained at 120 times per minute, resulting in a mass loss of 1.5%. The optimal

composition was determined as 2% basalt, with a reaction time of 65 minutes, a temperature of 220 °C, and a mass loss of 1.0%.

Several studies were conducted to perform IR analysis for the formulation of a novel composition of oil-free mastics, as outlined in the table below.



IR analysis of mastic substance

Table 8 displays the findings from IR spectroscopy, providing details on the positions of absorption bands in the vibrational spectrum of atom groups within the mastic substance.

Table 8.

IR analysis of mastic substance

Atomic group	The positions of the absorption bands in the vibrational spectrum of atomic groups V, sm^{-1}		Compound formula
	Valent vibrations	Deformation vibrations	
CO_3^{2-}	873,75	1739,79	Basalt
-OH	439,77	596,00	3MgO•2SiO2•2H2O
Ca=O	1377,17	-	CaO
SiO_2	966,34	721,38	SiO_2 glass
C-O-C	1033,85	-	Rubber
C=O	1265,30	-	Rubber
-C-H	1101,35	2918,30	Rubber
-C-H	2850,79	-	carboxycellulose
$-CH_2$	1446,61	-	carboxycellulose
-O-	1178,51	-	carboxycellulose
C=C	-	1541,12	$C_{30}H_{30}O_8$ gossypol

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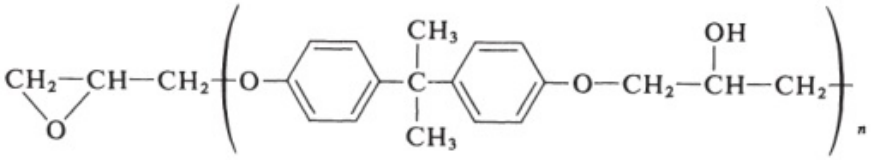
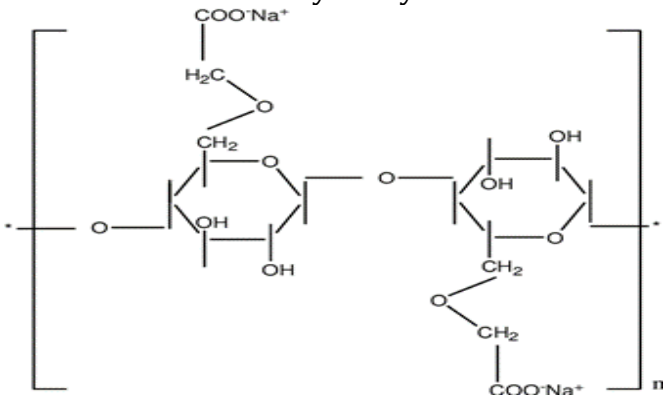
Based on the results of the FTIR spectroscopy analysis, specific vibrations related to the $CO3^{2-}$ ion, associated with the main component calcite $CaCO_3$ of basalt in the mastic composition, were identified at 873.75 and 1739.79 sm^{-1} regions. Additionally, vibrations corresponding to the -OH group in the asbestos component were observed at 439.77 and 596.00 sm^{-1} regions, representing valence and deformation oscillations. The Ca=O group of atoms indicated a stretching frequency at 1377.17 sm^{-1}. The SiO2 group, which constitutes the essential part of the mastic, demonstrated valence and deformation vibrations at 966.34 and 721.38 sm^{-1} regions.

The structural formula commonly used to represent rubber is expressed as follows.



The (C-O-C) atom group in the structure of rubber was observed at 1033.85 sm^{-1}, and the valence vibrations of the C=O atom group were identified at 1265.30 sm^{-1}. The -C-H atom group forming the phenyl group entering the rubber composition exhibited valence and deformation vibrations at 1101.35 and 2918.30 sm^{-1}, respectively.

The overall structural formula of carboxymethylcellulose is as follows:



The absorption frequencies associated with the -C-H group of atoms in carboxymethylcellulose were detected at 2850.79 sm^{-1}, the c group of atoms at 1446.61 sm^{-1}, and the ether bond -O- at 1178.51 sm^{-1}.

The deformation vibration of the S=S bond, part of the naphthyl group constituting gossypol in the mastic substance, was observed in the region of 1541.12 cm-1.

Conclusion. In conclusion, it can be summarized that the prominent peaks identified in the IR spectrum of the mastic correspond to the vibrational frequencies of crucial chemical groups. Notably, the intense peaks at 2918.30 sm^{-1} for the -C-H group of atoms in the rubber, 1446.61 sm^{-1} for the $-CH_2$ group in the carboxycellulose composition, and 2850.79 sm^{-1} for the -C-H group underscore the distinctive chemical characteristics of the mastic. This, in turn, signifies the potential of the chemical components' composition to interact proportionally and influence the chemical properties of the mastic.

Furthermore, this study sets the stage for further enrichment of the mastic's chemical composition. The detailed insights gained from the IR spectroscopy analysis provide a foundation for exploring the chemical nuances of the mastic substance. Future endeavors could

focus on fine-tuning the composition, considering the identified chemical groups and their corresponding vibrational frequencies. This nuanced approach holds promise for refining the chemical properties of the mastic, leading to potential advancements in its applications and performance characteristics.

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