

STUDYING OF METAL CONTAINING ACRYLIC COPOLYMERS AND SULFUR MODIFIED BITUMEN BH 90/30

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Abstract: In this article, BN 90/30 brand bitumen is modified by adding 5% to 15% acrylic copolymers containing Cu, Zn, Ni, and Sn metals, along with up to 24% sulfur. Copolymers based on acrylic and methyl methacrylate with metals (Ni, Sn, Cu, Zn) were used. Different masses were mixed in a heat-resistant 1000 ml container at various mixing speeds and temperatures, specifically 140, 160, 180, and 200°C. According to the obtained results, it was found that the reaction yield of bitumen modified at the optimal temperature of 200°C and a mass ratio of 15:24:61 is high 94.8%. The composition, physico-chemical properties, and mechanical properties of the modified bitumen were analyzed using IR-spectroscopy, thermogravimetric (TG) and differential thermal analysis (DTA), as well as X-ray phase and X-ray fluorescence analysis. Using X-ray phase analysis of the modified bitumen, it was determined that the initial substances underwent complete adhesion reactions with each other. The elasticity, viscosity curves, and rheological properties of the obtained modified bitumen were compared with those of unmodified bitumen and analyzed. Based on the above methods of analysis, modified bitumen can be used in asphalt roads, for covering the upper parts of roofs, and for producing waterproofing materials.

Keywords: bitumen, sulfur, nickel salts, tin salts, zinc salts, copper salts.

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Introduction

Currently, a wide range of high-quality bitumen products obtained from oil distillation are being developed for the modern oil refining industry [1-2]. Oil-based bitumen has good hydro-, electro-, and thermal insulation properties and is resistant to chemical substances, radiation, and atmospheric effects. It is widely used in electrochemistry, the chemical industry, nuclear power, and agriculture [3]. Recently, the demand for polymer-based additives in bitumen modification has been increasing. In particular, two main types of polymers, plastomers and elastomers, are widely used [4]. Plastomers are usually used to improve the elastic properties of binders and to increase the resistance of coatings to permanent deformation. Elastomers have also been used to

improve the insulation and high-temperature resistance of binders [5]. Bitumen modification is mainly carried out by the reaction of polar groups present in polymer molecules with polar components of bitumen [6]. This prevents phase separation and increases the strength of materials obtained from modified bitumen [7]. Acrylates, ethyl vinyl acetate (EVA), glycerin methacrylate (GMA) terpolymer, ethyl butyl acrylate (EBA) copolymer, etc., are commonly used in the production of waterproofing bitumens [8]. To study the effect on the physical and chemical properties of bitumen with a soft consistency, studies have been conducted on the use of secondary products, which are elastomeric polymers, and sulfur as bitumen additives [9]. Tripolymers, which are polymers

containing functional groups that can form chemical bonds with some bitumen components, are used as secondary polymers. These polymers form a network around asphaltenes contained in bitumen, creating an integral compound and exhibiting the ability to chemically react with bitumen [10-11]. However, when these chemical bonds occur, a polymer-bitumen mixture that lacks melting and solvent properties and is completely useless can be formed [12-13]. This limit has been taken as 5-5.5% by weight in some studies, and it has been found that this figure can be less than 1% [14]. Additionally, in some literature, much better results have been achieved with bitumen containing a smaller amount of tripolymer and sulfur. As a result, tri-polymers added in larger quantities are economically effective [15]. Currently, other types of copolymers used in bitumen modification include styrene-butadiene-styrene (SBS), styrene-isoprene-styrene, ethylene-vinyl acetate, and polyethylene [16]. In addition, attention is paid to the use of synthetic copolymers. When synthetic copolymers are added to bitumen, it

has been found that bitumen's temperature resistance, elastic properties, and rheological properties increase [17]. Alternatively, modified bitumens can be produced by reaction with bitumen based on the sequential polymerization of diblock precursor styrene and middle block monomers acrylonitrile and polyisoprene [18]. Therefore, not only linear copolymers but also multifaceted copolymers (star, radial, or chain copolymers) can be produced. The structure of the tripolymer consists of styrene polyacrylonitrile polyisoprene tri-block chains with a two-phase morphology of spherical polystyrene block domains within the polyisoprene matrix [19]. In many experiments conducted to determine the elasticity, mass loss, or insulating properties of bitumen modified with tripolymer, it has been found that bitumen modified with metal-containing copolymers has a positive effect on viscosity, elasticity, and insulating properties [20].

The *aim of this article* was to study the modification of bitumen grade BN 90/30 by adding acrylic copolymers containing the metals Cu, Zn, Ni and Sn, as well as sulfur.

Experimental part

Materials. BN 90/30 brand bitumen is produced in Uzbekistan "Bukhara Oil Refinery". Acrylic copolymers, sulfur, acrylic and methyl methacrylate-based copolymers containing metals (Ni, Sn, Cr, Cu, Zn) were used for this study also and they were purchased as "chemically pure" from "Merit Chemicals" company in Uzbekistan.

Methods.

IR analysis. The composition of the sulfur modified bitumen was investigated using IR-spectrum analysis with Shimadzu IR Tracer-100 (SHIMADZU (Japan)).

SEM-EDX- analysis. The images of the surface of the sulfur modified bitumen obtained in different sizes were taken on a JSM-IT200 device with elemental dispersion analysis (SEM - EDX) belonging to the Japanese company JEOL.

TGA and DTA analysis. Thermogravimetric studies of changes in the physicochemical properties of the obtained sulfur modified bitumen under the influence of temperature were studied on the DTG-60

device, SHIMADZU (Japan). Analytical conditions: in an argon atmosphere (80 ml/min), the rate of temperature increase was -10C/min.

X-ray fluorescence analysis. The analyzes were carried out on the EDX-8100 instrument. Rh element was used as a source of radioactive radiation in the analyses. In the course of research, it was carried out in the atmosphere of atmospheric air, in the time interval from 10 to 30 seconds.

Bitumen mixing machine. For mixing, Model- ELR401/3, Power (kW)- 1.1/1.5, Rotation speed- 3000(rpm), Flow-0.5 (m³/h), Inlet-DN40(mm) and outlet DN32(mm) were used.

Preparation of metal-containing acrylic copolymers and sulfur-modified bitumen.

BN 90/30 bitumen (GOST 127-93), along with acrylic and methyl methacrylate-based copolymers containing sulfur and metals (Ni, Sn, Cu, Zn), was used to prepare metal-containing acrylic copolymers and sulfur-modified bitumen. Different masses of bitumen (610g, 650g, 710g) were placed in a heat-

resistant 1000 ml container and mixed at five different speeds (300, 600, 900, 1200, and 1500 rpm) using an inline high ELR401/3 power 1.1/1.5 kW mixer. The bitumen was heated to liquefaction at four different temperatures (140, 160, 180, and 200°C). Sulfur (240g, 24%) and metal-containing acrylic copolymers (50 g, 110 g, and 150 g corresponding to 5%, 11%, and 15%) were added to the liquid bitumen and mixed for 2 hours at speeds of 1500-2000 rpm at 200°C. It was observed that the elasticity and

viscosity of the bitumen changed as the amount of modifiers increased during the bitumen modification process.

The effect of temperature and the ratio of starting materials on the product yield of acrylic and methyl methacrylate-based copolymers containing metals (Ni, Sn, Cu, Zn) and sulfur-modified bitumen was investigated. Table 1 shows a comparison of the yields of modified bitumen under different conditions [21].

Table 1. Effect of Temperature and Ratio of Starting Materials on Product Yield of Modified Bitumen

Raw material	Mass ratio (%)	T, °C	Reaction yield ω, %	Raw material	Mass ratio (%)	T, °C	Reaction yield ω, %
ZnAS+S+B	5:24:71	140	54,5	CuAS+S+B	5:24:71	140	51,4
		160	65,6			160	62,3
		180	68,7			180	69,1
		200	70,4			200	71,4
	11:24:65	140	61,2		11:24:65	140	60,3
		160	70,3			160	71,5
		180	76,7			180	74,6
		200	84,3			200	83,2
	15:24:61	140	65,6		15:24:61	140	62,3
		160	71,4			160	72,4
		180	78,4			180	79,3
		200	87,5			200	84,3
SnAS+S+B	5:24:71	140	55,4	NiAS+S+B	5:24:71	140	61,5
		160	61,3			160	74,3
		180	67,4			180	84,2
		200	71,3			200	86,4
	11:24:65	140	64,3		11:24:65	140	65,1
		160	70,3			160	74,8
		180	78,4			180	83,1
		200	86,4			200	91,4
	15:24:61	140	68,4		15:24:61	140	70,1
		160	72,4			160	78,4
		180	83,4			180	86,8
		200	94,3			200	94,8

Table 1 shows the effect of temperature and the ratio of starting materials on the product yield of copolymers based on acrylic and methyl methacrylate with metals (Ni, Sn, Cr, Cu, Zn)

and bitumen modified with sulfur. It can be seen from Table 1 that the reaction yield of bitumen modified with these copolymers at 200°C and a mass ratio of 15:24:61 is high, reaching 94.8%.

Results and discussion

Acrylic copolymers containing metals (Ni, Sn, Cr, Cu, Zn) and sulfur-modified bitumen have the highest yield. Since nickel-containing acrylic copolymers and sulfur-modified bitumen showed the best results, our research focused solely on the composition, structure,

physicochemical, and mechanical properties of nickel-containing acrylic copolymers and sulfur-modified bitumens [21].

IR analysis of nickel-containing acrylic copolymers and sulfur-modified bitumen (NiASB). The infrared spectra of the modified

bitumen were analyzed at the same intensity and scanning speed across the range of 500 to 4000 cm^{-1} . Vibrations of $-\text{CH}_3$ and $-\text{CH}_2$ bonds of acrylic monomers connected in a straight chain were observed in the 2920.23 cm^{-1} region with high intensity. The presence of double bonds in the copolymer was observed at 1620.21 cm^{-1} , where $\text{C}=\text{C}$ bonds are connected in the straight

chain and the aromatic ring. The valence vibrations of branched $-\text{S}-\text{S}-\text{S}-$ bonds in the aromatic ring were observed in the region of 1375.25 cm^{-1} , and $\text{C}=\text{O}$ carboxyl groups appeared in the region of 1120.64 cm^{-1} . The binding of nickel metal to carbocyclic groups in modified bitumen was observed in the region of 736.81 cm^{-1} (Fig. 1).

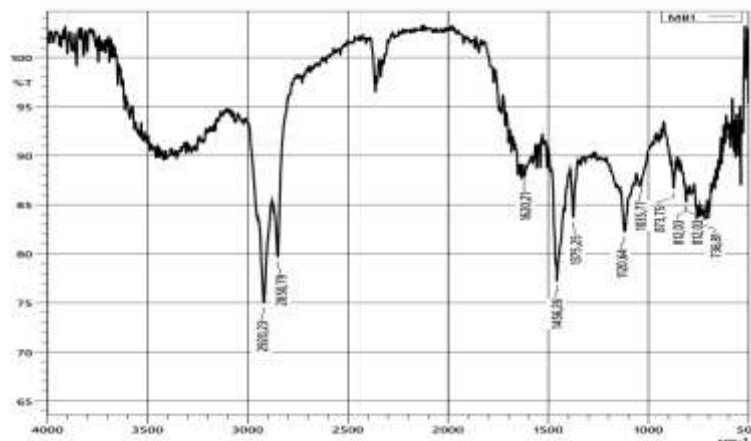


Fig. 1. IR-spectrum of bitumen modified with nickel-containing acrylic copolymers.

Raman spectrum analysis of modified bitumen (NiASB). The substances contained in modified bitumen are completely connected. The $\text{C}=\text{C}$ bonds in acrylic copolymers are connected in a straight chain and an aromatic ring in the area of 1617.84 cm^{-1} . The branching of sulfur atoms in an aromatic ring, indicated by

the $-\text{S}-\text{S}-\text{S}-$ bond, can be observed through its valence vibration in the 1198.96 cm^{-1} area. The $\text{C}=\text{O}$ carboxyl functional group is found in the 999.83 cm^{-1} area, and the nickel metal bond is formed by reaction in the 319.83 cm^{-1} area (Fig. 2).

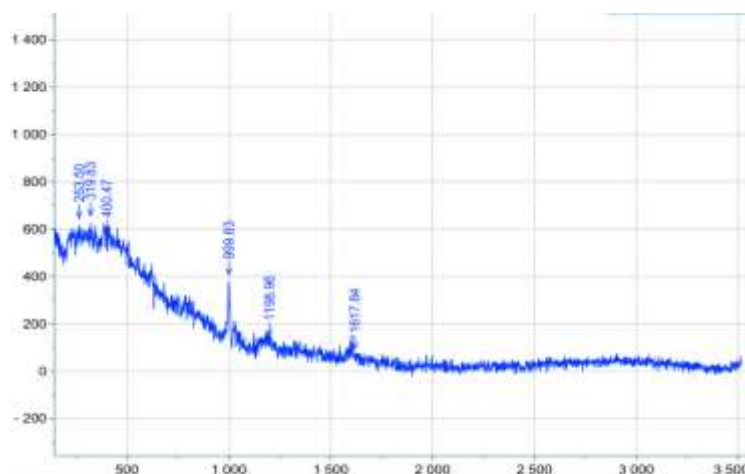


Fig. 2. Raman spectrum of bitumen modified with nickel-containing acrylic copolymers.

Based on the results of the above analysis, the structure of modified bitumen can be written as follows (Fig. 3).

It can be concluded from the results of IR-

spectrum and Raman spectrum analysis that it is possible to see that the initial substances have fully reacted in the modified bitumen [22].

Flash point, °C, not low	200	200	200	200	200	200	GOST 4333
Sticking to the guiding marble	Withstands according to control sample No. 2						GOST 11508
Adhesion applied to crushed stone and sand	Withstands according to control sample No. 2						GOST 11508
Uniformity	The same						GOST 11506-

It can be seen from Table 2 that the elasticity of modified bitumen is higher than that of unmodified bitumen. The resulting nickel-containing acrylic copolymer and modified bitumen were compared and studied based on their physico-mechanical properties, according to the requirements of GOST 11501-78 (Table 3).

It can be seen from Table 3 that the mechanical properties of bitumen modified with nickel-containing acrylic copolymers vary

depending on their application in various fields according to GOST requirements. The bitumen was classified as liquid (at cold temperature) and liquid (at hot temperature), and their relationship to opacity was studied [23]. To analyze these properties, modified bitumens were examined using the method of structural rheology. The rheological curves depicting the elastic and viscosity properties of the modified bitumen are shown in Fig. 4.

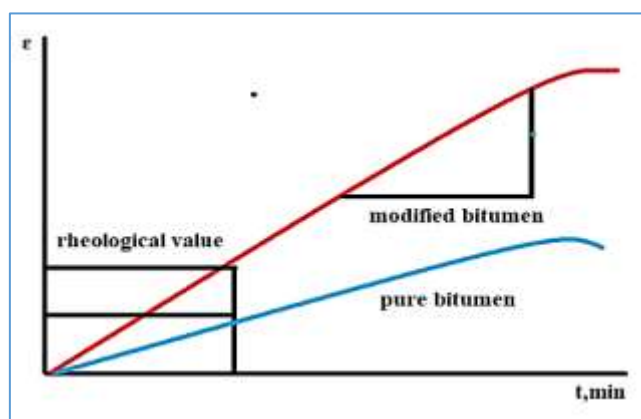


Fig. 4. Rheological characteristics of bitumen modified with nickel-containing acrylic copolymers.

Table 4. Comparison of temperature viscosity of bitumen modified with nickel-containing acrylic copolymers.

№	Substances taken for testing	Pressure, kPa, mPa.	The degree of stickiness
1	Solutions of nickel-containing acrylic copolymers ($T = 100^{\circ}\text{C}$ to 300°C and shear rate from 10 to 1000 s^{-1})	10 mPas dan 8 000 Pas gacha	1000-1400
2	Polymer solutions (zero shear rate, i.e. less than 1 s^{-1})	1 kPas to 1 MPas	800-1200
3	Unmodified bitumen $T = 80^{\circ}\text{C}$, 60°C , 40°C , 20°C , 0°C	200 Pas, 1kPas, 20 kPas, 0.5 MPas, 1 MPas	1600-2400
4	Bitumens modified with nickel-containing acrylic copolymers $T = 100^{\circ}\text{C}$, 200°C	450 Pas, 2kPas, 20 kPas, 0.5 MPas, 1 MPas	3400-4200

These parameters and their derivatives can be considered physically accurate because they do not depend on the deformation conditions, and Gmi, like their derivatives, are conditional characteristics and can only be used to compare materials under the same deformation conditions (Table 4).

From Table 4 above, it was determined that the viscosity of bitumen modified with nickel-containing acrylic copolymers is higher at a pressure of 1 MPa compared to unmodified bitumens. This conclusion was reached by examining the shearing speeds of polymer solutions and both unmodified and modified bitumens in the inline high device at various pressures.

TGA and DTA analysis.

Thermogravimetric (TG) and differential thermal analysis (DTA) were conducted on 16.136 mg of unmodified bitumen in the temperature range of 10-802°C. In the thermal analysis, two endothermic effects were observed at temperatures of 60.87°C and 722.83°C. The thermogravimetric (TG) curves of unmodified bitumen showed three significant mass loss temperature ranges. The first mass loss range of the TG curve occurred at 20.39-191.54°C, the second mass loss range at 191.54-458.35°C, and the third mass loss and hardening range was observed at 458.33-801.65°C (Fig. 5).

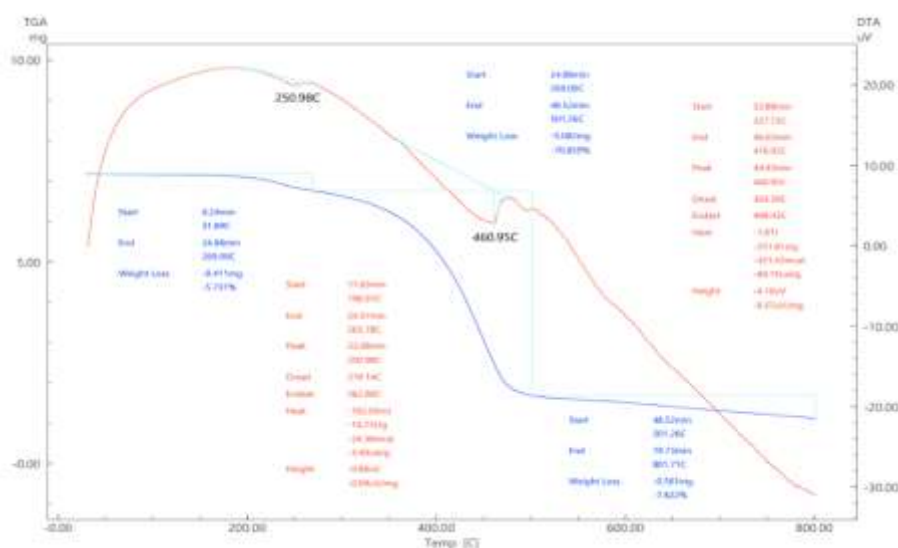


Fig. 5. Thermogravimetric (TG) and differential thermal (DTA) analysis of unmodified bitumen.

Table 5. Thermogravimetric and differential thermal analysis of unmodified bitumen

No	Temperature °C	Lost mass, mg	Mass loss, %	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

The results of thermogravimetric (TG) and differential thermal analysis (DTA) of unmodified bitumen show a 7.734% mass loss in interval 1, indicating the release of water in the form of crystalline hydrate. In mass loss interval 2, a 10.201% mass loss was observed,

corresponding to solidification and the release of gases from the aromatic ring. In the third mass loss range, a 20.352% mass loss was observed, indicating the release of coke and sulfur oxides. The differential thermal analysis results show energy absorption occurring at

temperatures of 41.24-90.07°C and 700.1-750.29°C. A more detailed analysis of the TG and DTA curves for this unmodified bitumen is presented in Table 5.

It can be seen in Table 5 that in the thermal analysis of unmodified bitumen, it was found that all substances were decomposed.

Thermal analysis of bitumen modified with nickel-containing acrylic copolymers. A sample of 7.172 mg of modified bitumen was taken, and its TG-DTA analysis was conducted

in the temperature range of 10-802°C. In the thermal analysis, two endothermic effects were observed at temperatures of 250.98°C and 460.95°C. The thermogravimetric (TG) curves of modified bitumen showed three intensive mass loss temperature ranges. The first mass loss range of the TG curve is 5.731% at 31.89-269°C, the second mass loss range is 70.859% at 269-501.26°C, and the third mass loss range is 7.822% at 501.26-801.71°C (Fig. 6).

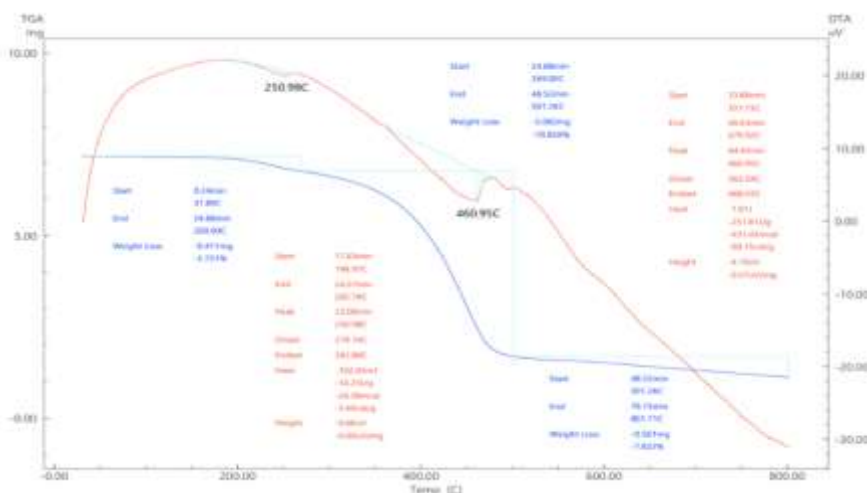


Fig. 6. TG and DTA analysis of bitumen modified with nickel-containing acrylic copolymers.

DTA analysis of bitumen modified with nickel-containing acrylic copolymers showed mass loss in three areas.

First Stage: At the first stage of decomposition, a weight loss of 0.02 mg or 5.731% was observed at a temperature of 31.89°C. At this stage, the release of water vapor bound in the form of crystalline hydrate was observed.

Second Stage: The main decomposition occurred at the second stage, with a mass loss of 5.587 mg or 70.859% at 269°C. Carbon (II) oxide was released as a result of the decomposition of coke gas in this temperature range.

Third Stage: Starting at 501.26°C and ending at 801.71°C, a mass loss of 1.648 mg or 7.822% was observed. It was observed that oxides from the carboxyl group were released from the decomposition of polymers, and metal oxides were released from the decomposition of metal carbonates.

In the DTA analysis of the synthesized product, it can be seen that 2.751 mg or

25.725% of residual substances remained at 460.95°C during heat absorption, indicating an endothermic process. At this stage, it can be observed that residues of metal salts remain. A more detailed analysis of the results of the TG and DTA curves for this modified bitumen is presented in Table 6.

From Table 6, it can be concluded that the substances contained in the modified bitumen are completely decomposed, and the initial substances are fully reacted. Therefore, it can be seen that modified bitumen is thermally stable compared to unmodified bitumen [24-26].

X-ray diffraction analysis. X-ray diffraction analysis of bitumen modified with nickel-containing acrylic copolymers was conducted under the following conditions: Cu voltage of 40.0 kV, current of 30 mA, no automatic crack slitting used, divergence slit of 1.00000°, propagation slit of 1.00000°, continuous scan speed of 2.0000°/min, sampling interval of 0.0500°, and set time of 1.50 s. The resulting graph shows three significant peaks: the first peak at $d=5.24$ at $2\theta=20^\circ$,

the second peak at $d=7.84d = 7.84d=7.84$ at 26° represents the width of the peak, which 2θ , and the third peak at $d=2.31d = 2.31d=2.31$ indicates the size of the substance. at $36^\circ 2\theta$ (Fig. 7). Typically, the value of "d"

Table 6. Thermogravimetric (TG) and differential thermal analysis (DTA) of bitumen modified with nickel-containing acrylic copolymers.

Nº	Temperature °C	Residual mass, mg	Mass lost (7.17 mg)	Mass loss, %	Amount of energy consumed ($\mu V \cdot s/mg$)
1	100	7,15	0,02	0,27	19,457
2	200	7,08	0,09	1,26	21,949
3	300	6,62	0,55	7,67	18,186
4	400	5,25	1,92	26,78	8,304
5	500	1,68	5,49	76,57	4,501
6	600	1,51	5,66	78,94	2,34
7	700	1,31	5,86	81,73	2,18
8	800	1,11	6,06	84,52	1,088

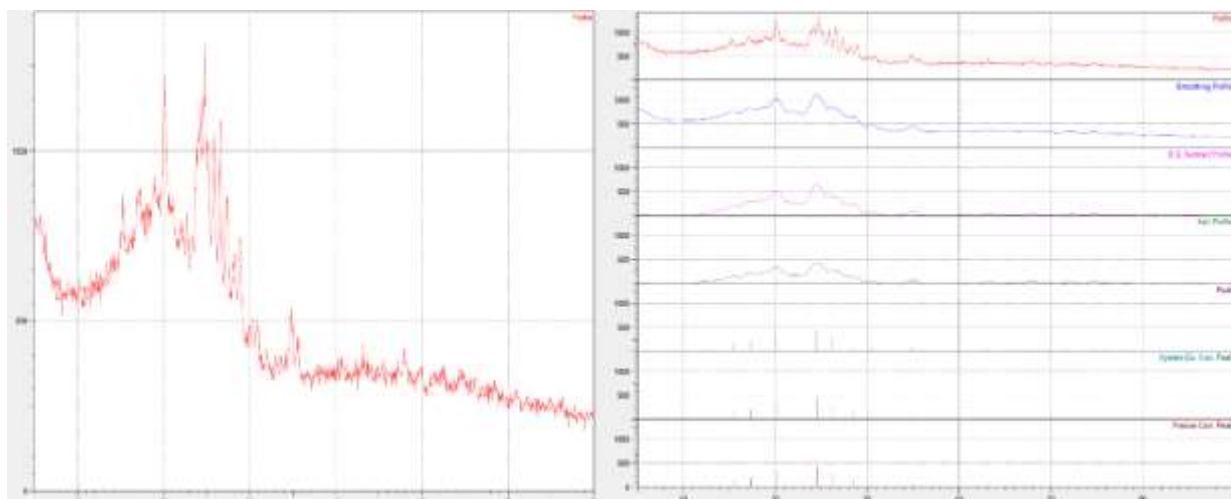


Fig. 7. X-ray phase analysis of bitumen modified with nickel-containing acrylic copolymers.

The presence of a high peak indicates that the atom is in a periodic mass, thus identifying it as a crystalline structure. According to the results of the X-ray phase diagram, it was found that the modified bitumen has complete adhesion reactions between the starting materials.

X-ray fluorescence analysis of bitumen modified with nickel-containing acrylic copolymers. The analyzes were carried out on the EDX-8100 instrument. Rh element was used as a source of radioactive radiation in the analyses. In the course of research, it was carried out in the atmosphere of atmospheric air, in the time interval from 10 to 30 seconds.

The first stage was carried out between Al-U metals, the second stage was carried out between Rh-Cd metals, the third stage was carried out between C-Sc elements <https://t.me/c/1424188715/72975> Analysis time of one sample 50 kV electric voltage and 321 A current to Al-U analytes for 10 minutes, Rh-Cd; 15 kV voltage and 1000 A current for analytes and 50 kV voltage and 453 A current were used for C-Sc analytes [25]. The amount of elements in the solid sample is given in percent. Al-U elements make up 30% of the sample, C-Sc elements -10%, Rh-Cd elements -4%, Zn-As elements and Ni-11%, Cr-Fe elements make up 30%, S-K elements make up 6% reached (Fig. 7).

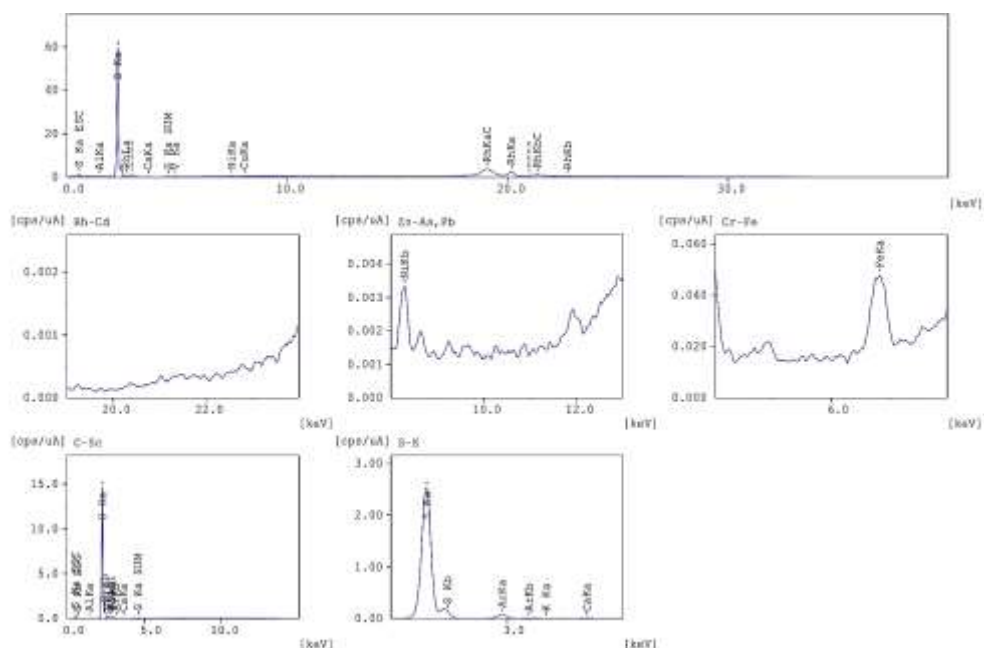


Fig. 7. X-ray fluorescence analysis of bitumen modified with nickel-containing acrylic copolymers.

X-ray fluorescence analysis results of copolymers show that the initial substances bitumen modified with nickel-containing acrylic have fully reacted.

Conclusion

The results of this study can be summarized as follows:

- (i). *Optimum Conditions:* The optimal conditions for modifying BN 90/30 bitumen with metals (Ni, Sn, Cu, Zn) based on acrylic and methyl methacrylate copolymers, such as temperature, mixing speed, duration, and mass ratio, were determined.
- (ii). *Composition of Modified Bitumen:* The bonding of double bonds in the copolymer was observed. At 1620.21 cm^{-1} , it was noted that C=C bonds are bonded in a straight chain and an aromatic ring. Valence vibrations of branched -S-S-S- bonds in the aromatic ring were observed in the 1375.25 cm^{-1} region, with C=O carboxyl groups appearing in the 1120.64 cm^{-1} region.
- (iii). *Physico-Chemical and Operational Properties:* The impact of nickel-containing acrylic copolymers on the physico-chemical and operational properties of bitumen was studied. Changes in structure due to temperature variations classified the bitumen as solid at cold temperatures and liquid at hot temperatures, with opacity also analyzed. Based on the results of the analysis, it was observed that when using modified bitumen as insulating coatings, the properties of elasticity and adhesion increased by 15%. Compared to unmodified bitumen, properties were observed to increase by 20%.
- (iv). *Thermal Stability:* Thermogravimetric (TG) and differential thermal analysis (DTA) of unmodified bitumen were conducted. Results indicated that the substances in the modified bitumen are completely decomposed and the initial substances fully reacted, proving that modified bitumen is more thermally stable than unmodified bitumen.
- (v). *X-ray phase and X-ray fluorescent:* X-ray phase analysis results showed that the modified bitumen had complete adhesion reactions between the starting materials. X-ray fluorescent analysis confirmed that the starting materials had fully reacted.

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