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Original Article

Studying Synthesis of Modifier Based on Croton Aldehyde and Phthalimine and Sulfur Modified Bitumen BH 90/30

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Abstract - *In this paper, the optimal conditions for synthesising a modifier with a new composition for bitumen modification were studied. For the synthesis of this modifier, the initial croton aldehyde was obtained from substances such as aldehyde and phthalimine in a 1:1 mol ratio, and the temperature was kept below 5ºC. To determine the chemical structure of this synthesized compound, the chemical formula of the new structural modifier was proposed using modern methods such as IR-spectroscopy, NMR and PMR. First, this modifier interacted with sulfur, and then BN 90/30 brand bitumen was used to prepare modified bitumens. Different masses were mixed in a heat-resistant 1000 ml container at various mixing speeds and temperatures, specifically 135, 140, 145, and 160°C. According to the results obtained, the reaction yield of bitumen was modified at the optimal temperature of 160°C and a high 94.8%. Modified bitumen's composition and physicochemical and mechanical properties have been thoroughly studied using modern analytical methods. Infrared spectroscopy analysis methods determined its structure and composition. The surface morphology and element content were analyzed in tabular form using an MIRA 2 LMU scanning electron microscope. Heat resistance and destruction of modified bitumen were studied using thermogravimetric (TG) and differential thermal analysis (DTA) methods. Based on the results of this analysis, it is recommended that modified bitumen be used in asphalt roads, roofing and waterproofing materials.*

Keywords - *Modifier, Croton aldehyde, Phthalimine, BN 90/30 brand bitumen, NMR and PMR.*

1. Introduction

Currently, the oil refining industry is developing a wide range of high-quality bitumen products obtained as a result of the distillation process[1,2]. These bitumen products are widely used in producing inexpensive and effective waterproofing and building materials and are effectively used in repair and moisture insulation works[3]. These petroleum-based bitumens formed during the distillation process are used as the main moisture barrier raw material [4].

Bitumen obtained based on oil has high hydro-, electrical- and thermal insulation properties and is a material resistant to chemical substances, radiation and atmospheric effects[5]. Therefore, they are widely used in electrochemistry, chemical industry, nuclear power and agriculture. In recent years, the demand for the use of polymer-based additives to improve the quality of bitumen has been increasing. In the modification of bitumen, polar groups present in polymer molecules are mainly carried out by reacting with polar components of bitumen [6]. It prevents the separation of phases and increases the strength of materials obtained based on modified bitumen [7]. The amount of tripolymers and sulfur used in bitumen modification must have clearly defined high-quality indicators so that the chemical composition of the main network does not reach the gel state. According to research, this amount can usually be 5-5.5% by weight, but in some cases, less than 1% has been found to be effective[8]. In addition, in some sources, higher results were recorded for bitumen samples with lower amounts of tripolymer and sulfur. Therefore, adding an excess amount of tripolymer is considered a cost-effectiveness factor[9].

2. Materials

BN 90/30 brand bitumen is produced in Uzbekistan “Karshi Oil Refinery”. Sulfur, were croton aldehyde, and phthalimine were used for this study also, and they were purchased as "chemically pure" from the "Merit Chemicals" company in Uzbekistan.

2.1. Methods

2.1.1. IR Analysis

The SHIMADZU IR-Fure spectrophotometer, made in Japan, was used to investigate the infrared spectra of the sulfur-modified bitumen at 400 and 4500 cm^{-1}. The analysis based on the IR spectrum.

2.1.2. 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.

2.1.3. 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 mi/min), the rate of temperature increase was -10C/min.

2.1.4. Quantum Chemical Analysis

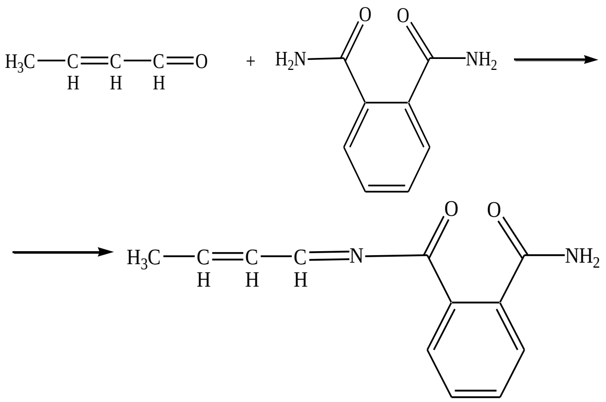
Quantum-chemical calculation of the reactivity of the corrosion inhibitor molecule using Avogadro, Hyper Chem 8.01, Asselrys MS Modeling 3.0.1 using the constrained Semi-empirical (DFT) method, semi-empirical AM1, MNDO, PM3, RM1 and MINDO3 using SCF-MO calculations were carried out using the Intel Pro Pentium 1.40 GHz computer.

2.1.5. Bitumen Mixing Machine

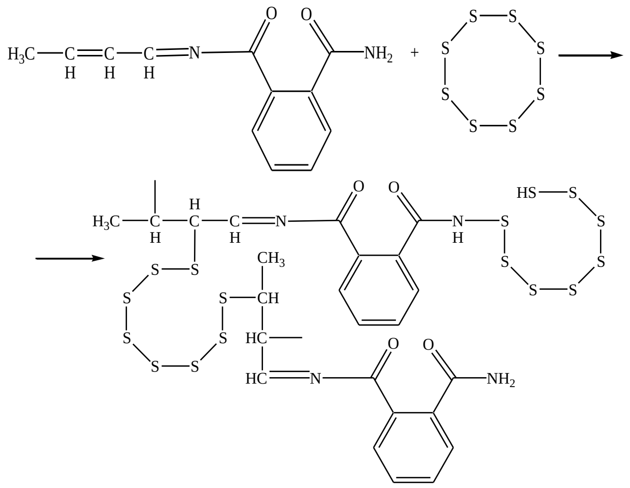
For mixing, Model- ELR401/3, Power (kW)- 1.1/1.5, Rotation speed- 3000(rpm), Flow-0.5 (m^3/h), Inlet-DN40(mm) and outlet DN32(mm) were used.

2.1.6. Synthesis of the Modifier

0.5 mol of croton aldehyde and 1 mol of phthalamine are added to a 250 ml round-bottomed flask filled with ice water and mixed for two hours after cooling. The solution is evaporated in a porcelain container, and the volume is brought up to 100 ml. Then, the resulting modifier is filtered (Scheme 1).



1-Scheme. Synthesis of the modifier



2-Scheme. Modification of sulfur with a modifier

2.2. IR Analysis

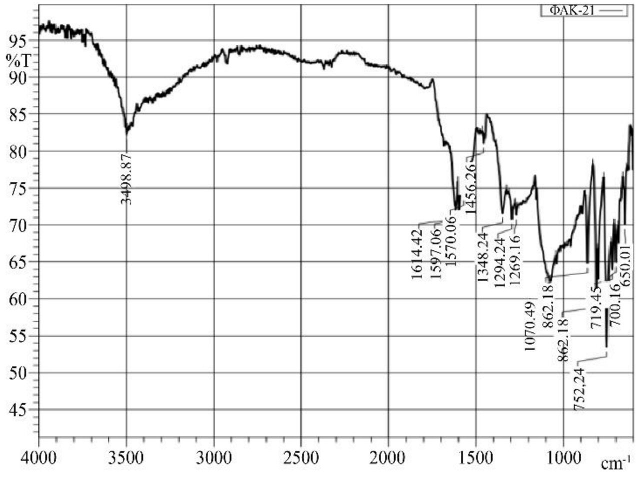


Fig. 1 IR-spectroscopy of the modifier

To determine the structure of the modifier, IR-spectroscopy analysis was carried out. The IR spectrum of the modifier is depicted in Figure 1, in which the CO-NH$_2$ bonds characteristic of amide groups are clearly observed in the bands of 3498, 700 and 650 cm^{-1}.

Absorptions corresponding to –C–N bonds in amide groups were recorded at 1614 and 1269 cm^{-1}. Bonds specific to the benzene ring were detected at 1597 and 1570 cm^{-1}, while C–H bonds in the benzene ring were recorded at 1070, 862, 810, 752 and 719 cm^{-1}. Also, absorption wavelengths corresponding to C–O bonds were observed in the 1348 and 1294 cm^{-1} bands[10,11].

2.3. NMR and PMR

In addition, the study and analysis of the structure of these synthesized modifiers were also studied with the help of NMR and PMR spectra.

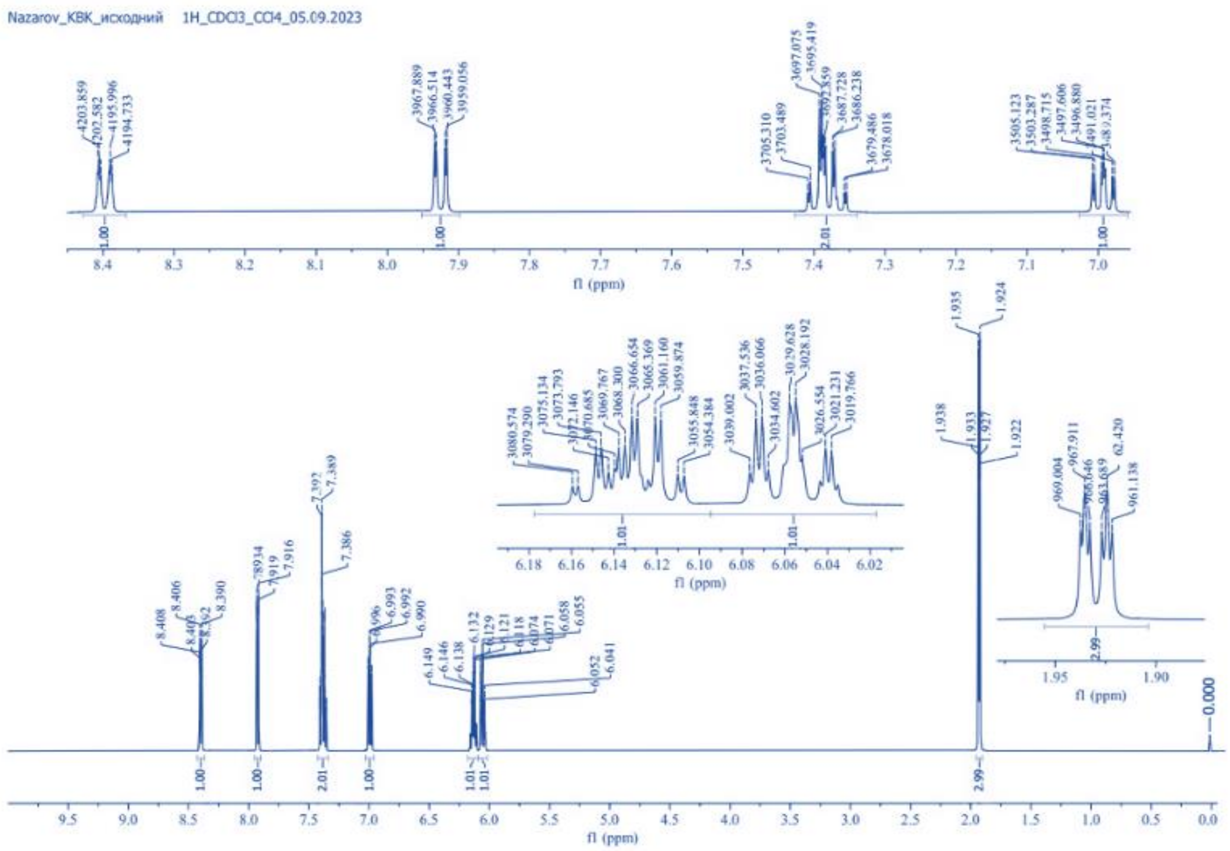


Fig. 2 ^{1}H NMR spectrum analysis of the modifier

In the NMR-spectrum of the modifier was analyzed, the proton signals in the benzene ring (H -3 and H-4) were quintet resonance at 7.392-7.386 m.u., (H -5) was triplet resonance at 6.996-6.990 m.u., (H -6) was 7.9 m.u. Doublet resonance at (N-9) 8,408-8,390 m.u. Doublet resonance at (H -10) 6,129-6,041 m.u. Triplet resonance at (H -11), multiplet resonance at 6,149-6,132 m.u., and (H -12) doublet resonance signals at 1,938-1,922 m.u.

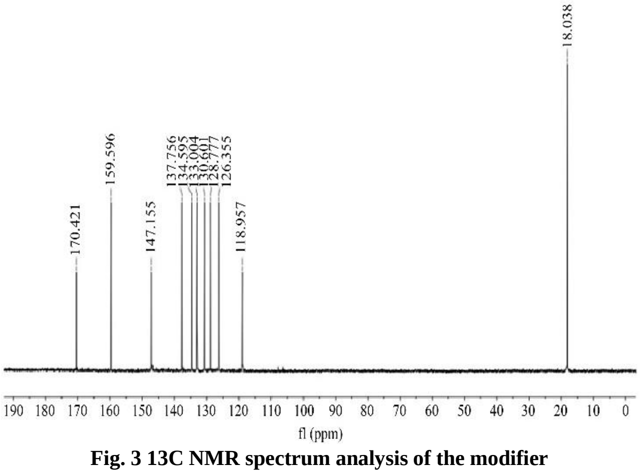


Fig. 3 13C NMR spectrum analysis of the modifier

In the ^{13}C NMR spectrum of the modifier, the 1st carbon in the carboxyl group is 118.957 m.u., the 2nd carbon is 147.155 m.u., the 3rd carbon is 128.777 m.u., the 4th carbon is 134.595 m.u., the 5th carbon is 126.355 m.u glerod 133.004 m.u., carboxyl carbon in group 170.421 m.u., carbon in -N=CH- (C-9) 159.596 m.u., carbons C-10 and C-11 connected with a double bond are 130.601 and 137.756 m.u., and carbon in methyl group is 18.038 m.u. showed the relevant signals. The resulting spectrum confirms the structure of the synthesized substance[12, 13].

2.3.1. Analysis of Modifier Reactivity with Sulfur using Quantum Chemical Method

The process of studying the "composition-structure-property" system in chemical compounds allows us to theoretically estimate the properties, composition and molecular structure of compounds. This information helps in the synthesis of compounds with selected properties.

In order to determine the reaction properties of the modifier molecule, quantum chemical calculations were performed using semi-empirical AM1, MNDO, PM3, RM1 and MNDO3 methods in Avogadro, Hyper Chem 8.01 and Asselrys MS Modeling 3.0.1 programs. Molecular geometry optimization was performed using the Polak-Ribiere (Conjugate Gradient) algorithm.

Table 1. Effective charge distribution on donor atoms of modifier molecules

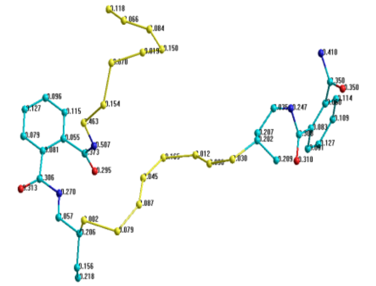
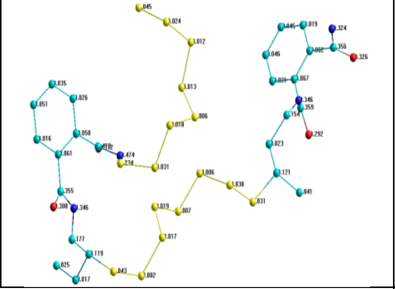
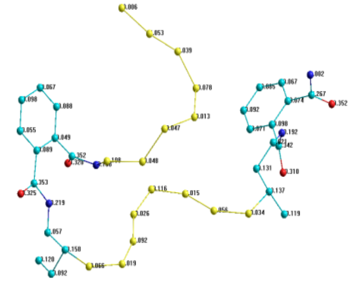
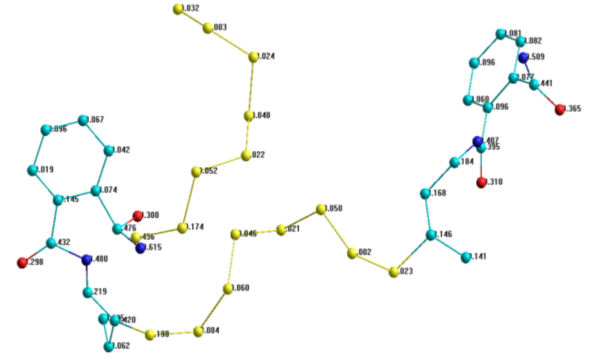
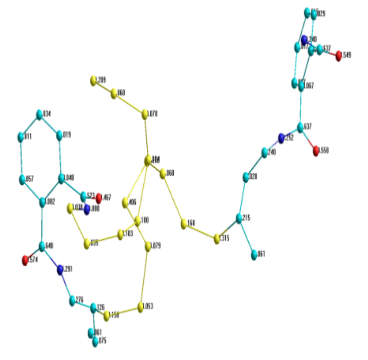
<table>
<tr><th>Methods of Calculation</th><th>AM1</th><th>MNDO</th><th>PM3</th></tr>
<tr><td rowspan="3"></td><td></td><td></td><td></td></tr>
<tr><td colspan="2">RM1</td><td>MNDO3</td></tr>
<tr><td colspan="2"></td><td></td></tr>
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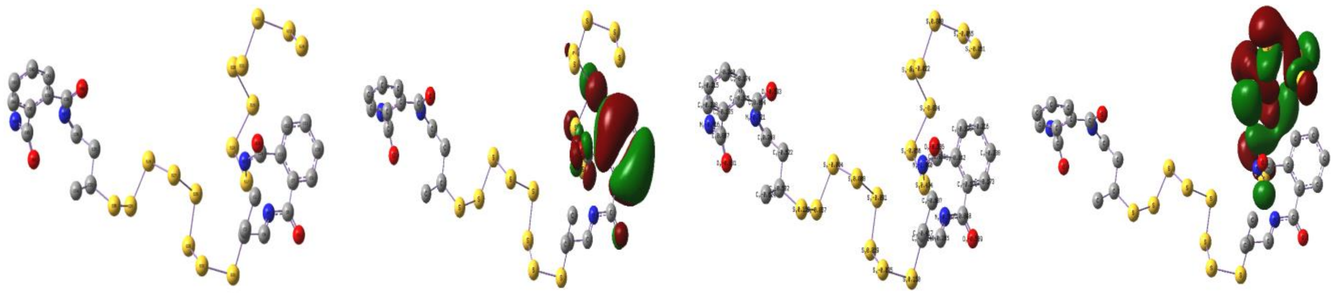
Table 2. Effective charge values of donor atoms in modifier molecules

Atoms	AM1, eV	MNDO, eV	PM3, eV	RM1, eV	MNDO3, eV
XXX					
δ_q S^1(-S-)	-0,118	0,045	-0,006	-0,032	-0,209
δ_q S^2(-S-)	0,066	-0,024	0,053	0,003	0,060
δ_q S^3(-S-)	0,084	-0,012	0,039	-0,024	-0,078
δ_q S^4(-S-)	-0,150	-0,013	-0,078	-0,048	0,004
δ_q S^5(-S-)	-0,019	0,006	-0,013	0,022	0,100
δ_q S^6(-S-)	-0,070	-0,018	-0,047	-0,052	-0,103
δ_q S^7(-S-)	-0,154	-0,031	-0,048	-0,174	0,035
δ_q S^8(-S-)	0,463	0,234	0,108	0,436	-0,038
δ_q N^1(NH)	-0,507	-0,474	-0,168	-0,615	-0,080
δ_q O^1(C=O)	-0,295	-0,312	-0,320	-0,300	-0,467
δ_q O^1(C=O)	-0,313	-0,308	-0,325	-0,298	-0,574
δ_q N^1(-N=)	-0,270	-0,346	-0,219	-0,480	-0,291
δ_q S^9(-S-)	0,002	0,043	-0,066	0,198	-0,150
δ_q S^{10}(-S-)	-0,079	-0,002	0,019	-0,084	-0,053
δ_q S^{11}(-S-)	0,087	-0,017	0,092	-0,060	-0,079
δ_q S^{12}(-S-)	0,045	-0,019	-0,026	-0,046	0,406
δ_q S^{13}(-S-)	-0,165	-0,007	-0,116	0,021	-0,230

$\delta_q S^{14}$(-S-)	0,012	-0,006	0,015	-0,050	0,060
$\delta_q S^{15}$(-S-)	0,096	-0,030	0,056	0,002	0,160
$\delta_q S^{16}$(-S-)	0,030	0,031	-0,034	0.023	-0,315
$\delta_q N^2$(NH)	-0,247	-0,346	-0,192	-0,407	-0,252
$\delta_q O^1$(C=O)	-0,310	-0,292	-0,310	-0,310	-0,558
$\delta_q O^1$(C=O)	-0,350	-0,326	-0,352	-0,365	-0,546
$\delta_q N^2$(-N=)	-0,410	-0,324	0,002	-0,509	-0,240
E	-7236,2244 (kcal/mol)	-7224,9711 (kcal/mol)	-7264,9073 (kcal/mol)	-3939,1605 (kcal/mol)	-7428,9709 (kcal/mol)

According to Mulliken's method, the obtained results show high values of negative effective charge in modification molecules shown in oxygen atoms of C=O and C-O-C groups and nitrogen atoms of N-H groups[14, 15]. The geometric optimization of the modifier was performed using the Avogadro program and was performed using the RHF/6-311G(d,p) basis set in GaussView 6.0.16. Charges were calculated using the DFT (B3LYP) method and the Mulliken method.

Charge distribution is important in determining a molecule's electrophilic and nucleophilic centers. A high negative charge is localized on nitrogen and oxygen atoms in the modifier molecule (Figure 4b). Electron density in frontier molecular orbitals (ChegMOs) plays an important role in evaluating the reactivity of organic compounds. The higher occupied molecular orbital (LUMO) is recognized as an electron donor, and the Lower Unoccupied Molecular Orbital (LUMO) as an electron acceptor[16].



HOMO = -7,02 *eV* LUMO = 3,85 *eV*

Fig. 4 Effective charge distribution on donor atoms in modifier molecules

According to the charge density of the higher occupied molecular orbital (HOMO) and lower unoccupied molecular orbital (LUMO), the largest distribution of charge density belongs to the -O-, C=O and N-H groups (Figure 4c,d). HOMO and LUMO orbital energies and the difference between them (DE=E_{LUMO}-E_{HOMO}, eV) are also important. An increase in the HOMO energy (increasing the occupied level towards the LUMO) in a series of compounds indicates an increase in the electron-donating property of the compound. The LUMO energy determines the electron acceptor ability (propensity for electrons) of compounds. Ionization potential, I=-E_{HOMO}, (eV), Electron propensity, A=-E_{LUMO}, (eV), Electronegativity, χ= (I + A)/2 (eV), Chemical hardness, η = (I - A) /2 (eV), Chemical potential, μ_p=-(I + A)/2 (eV), Chemical softness, σ = 1/(2η) (eV-1), Electrophilicity index, $\omega=\mu_p^2/2\eta$ (eV), dipole moment, μ (Debay) was calculated. The results are presented in Table 3.

Table 3. Calculated quantum chemical parameters for the modifier

Quantum-Chemical Parameters	**Modification**
E_{HOMO}, eV	-9,74
E_{QBMO}, eV	-0,32
|ΔE|= E_{QBMO} – E_{HOMO} (eV)	9,43
Ionlanish potensiali, I=- E_{HOMO}, (eV)	9,74
Elektronga moyillik, A=-E_{QBMO}, (eV)	0,32
Elektromanfiylik, χ= (I + A)/2 (eV)	5,03
Kimyoviy qattiqlik, η = (I - A)/2 (eV)	4,71

Kimyoviy potensial, $\mu_p=-(I + A)/2$ (eV)	-5,03
Kimyoviy yumshoqlik, $\sigma = 1/(2\eta)$ (eV-1)	0,11
Elektrofillik indeksi, $\omega=\mu_p^2/2\eta$ (eV)	2,68
Dipol moment, μ (Debay)	6,67

It is known that the level of electrostatic potential is used to determine the electron donor and electron acceptor parts of a molecule. MEP (molecular electrostatic potential) plots in red, blue, and green are negative, positive, and neutral electrostatic potentials, respectively (Figure 5). Due to the high splitting energy in the orbital of this modifier, it forms compounds with sulfur.

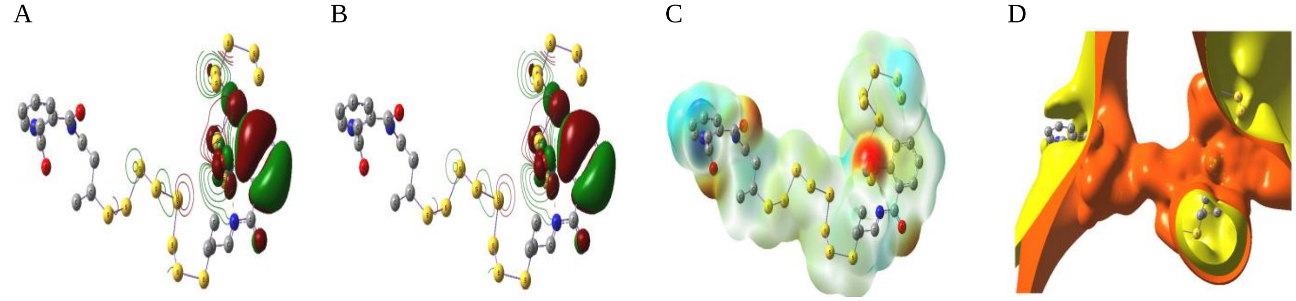


Fig. 5 ESP-HOMO (a), ESP-LUMO (b) and ESP(c,d) of the modifier

3. Results and Discussion

3.1. Modification of Bitumen with a Modifier

Sulfur was modified with the originally synthesized modifier. In this case, the modifier was added in the amount of 2% by weight of sulfur. This process helps improve sulfur's properties and obtain a modified material. BND 60/90 bitumen must be heated until it becomes anhydrous at a temperature not exceeding 160 °C. If the temperature exceeds 160 °C, the binding properties of bitumen may be lost. After the bitumen is fully heated, it is mixed with modified sulfur. In this process, bitumen should be poured over modified sulfur, and the mixture should be stirred for 30-40 minutes. Modified sulfur and bitumen enter into a chemical reaction and the physicochemical properties of bitumen change. The optimal ratio is to mix sulfur and bitumen in a 20-80% ratio. If the sulfur content of cerobitum exceeds or decreases by 10%, the viscosity and plasticity decrease, and the cerobitum produced does not meet standard requirements[17]. Considering the above, 600 g of bitumen was placed in a container and heated until it liquefied at a temperature of 130-160 °C. 160 g of modified sulfur (3.2 g of modifier) should not be added to diluted bitumen at a speed of 150-200 ylan/minute because if this norm is exceeded, the quality indicators of sulfur-bitumen may change negatively. The modification process is carried out at a temperature of 200°C for 2 hours. The most optimal conditions for the production of modified bitumen were studied, and the physico-chemical properties of the bitumen were analyzed. According to the research results, the composition of bitumen with the highest efficiency was determined. This optimal composition has properties that help to increase the performance of modified bitumen, improve the workability of bitumen and make it suitable for various construction works[18-22]. Modified bitumen was tested under different conditions and ratios. The modified bitumen obtained as a result of the research was calculated depending on the temperature and mass ratio of the components. The highest reaction temperature was 160 °C, and the highest yield was obtained from the modified bitumen with a ratio of 80:19.6:0.4 (Figure 6).

Table 4. Effect of modification on product yield, temperature and ratio of starting materials (bitumen (B), sulfur (S), modifier in %)

No	B:S:M	T,°C	Reaction yield ω,%	No	B:S:M	T,°C	Reaction yield ω,%
1	**80:19:0.3**	125	38.2	**1**	**80:19,5:0.5**	125	25.3
2		135	43.6	**2**		135	48.4
3		145	69.8	**3**		145	68.7
4		**160**	76.4	**4**		**160**	76.8
No	**B:S:M**	**T,°C**	**Reaction yield ω,%**	**No**	**B:S:M**	**T,°C**	**Reaction yield ω,%**
1	**80:19,7:0.3**	125	25.3		**80:19,6:0,4**	125	25.3
2		135	61.2			135	68.4
3		145	75.8			145	84.8
4		**160**	88.3			**160**	**93.4**

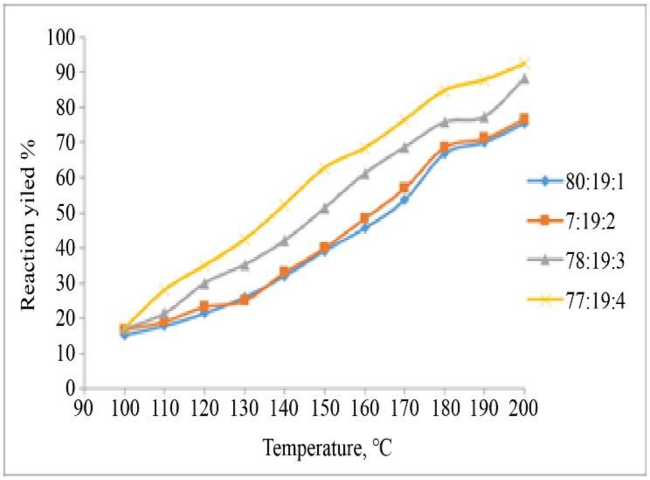


Fig. 6 The effect of the amount of starting materials and temperature on the reaction yield of bitumen

3.2. IR Analysis

Based on the IR spectrum analysis, we can see the structure of bitumen modified with sulfur, which has the following structure (Figure 7). In the area of 2902.87 cm^{-1}, groups caused by asymmetric and symmetric valence vibrations of methyl (-CH_3) and methylene (-CH_2) groups are located. In the region of 1039.63 cm^{-1}, the vibration of the sulfo bond of the S-S group can be seen. -C=C- bonds in 1653 cm^{-1} area, -C-S-C- valence vibrations in 1064.71 cm^{-1} area, as well as NH-S bonds belonging to amide groups in 777.31 cm^{-1} and 877.61 cm^{-1} areas we can see from Figure 7.

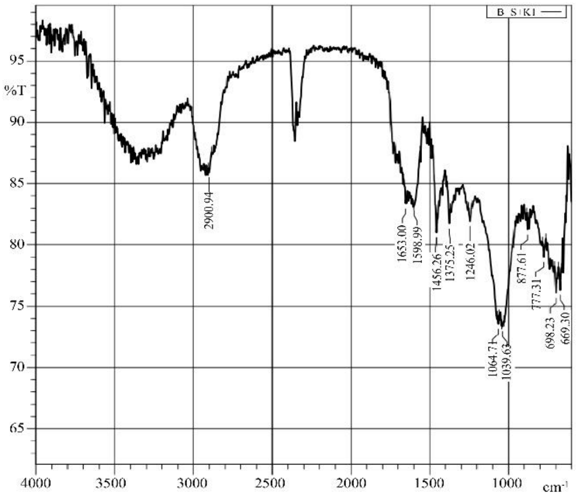


Fig. 7 The IR spectrum of modified bitumen

3.3. SEM and Elemental Analysis of Modified Bitumen

The surface morphology and elemental composition of the modified bitumen were studied by scanning electron microscope and elemental analysis, in which the completeness of the reaction and the elemental composition of the substances formed in parallel in the reaction are presented in Figure 8.

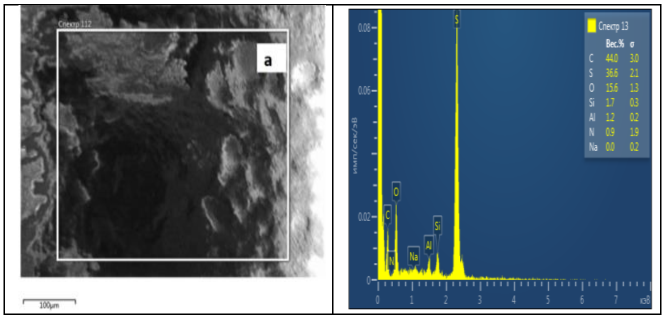


Fig. 8 SEM and elemental analysis of modified bitumen

100 magnified images of the modified bitumen were taken, and it was proved that the substances involved in the reaction had fully reacted and changed the composition of the bitumen, that the initial substances were completely combined with each other, and that there were no additives in the composition of the substances.

3.4. Thermogravimetric (TG) and Differential Thermal Analysis (DTA)

To study the thermal properties of modified bitumen, 16,136 mg of samples were taken, and TG-DTA analysis was performed at temperatures ranging from 10°C to 802°C. In the thermal analysis results, two main endothermic effects were observed, which were recorded at temperatures of 60.87°C and 722.83°C, respectively.

The thermogravimetric (TG) curve of modified bitumen has three intensive mass loss stages. In the first stage, mass loss occurs in the temperature range from 20.39°C to 191.54°C. The second stage of mass loss is observed from 191.54°C to 458.35°C. The third and highest temperature mass loss occurs in the interval from 458.33°C to 801.65°C. These three stages of mass loss reflect different thermal stability and degradation processes of bitumen components.

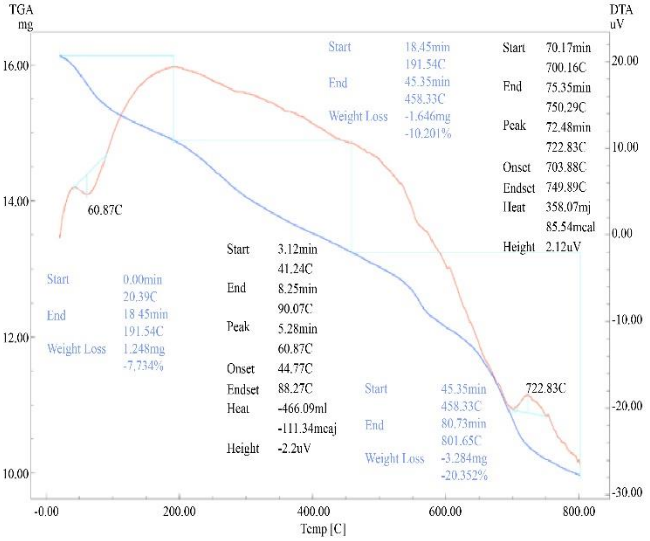


Fig. 9 Thermogravimetric (TG) and Differential Thermal (DTA) analysis of modified bitumen

The thermogravimetric (TG) and differential thermal analysis (DTA) results of the modified bitumen show that 7.734% mass loss was observed in the first mass loss stage, 10.201% mass loss in the second stage and 20.352% mass loss in the third stage. DTA results of modified bitumen show energy absorption in temperature ranges from 41.24°C to 90.07°C and from 700.1°C to 750.29°C[18-21]. For a more comprehensive analysis of the TG and DTA results of this modified bitumen, detailed information is provided in Table 5. The information presented in the table helps to better understand the thermal properties of bitumen and the thermal stability of its components.

Table 5. Thermogravimetric and differential thermal analysis of modified bitumen

No	Temperature (°C)	Lost mass (mg)	Mass loss (%)	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

4. Conclusion

As a result of this study, a new type of modifier was synthesized and modified with sulfur to modify BN 90/30 bitumen. The following can be concluded from the research results.

- When the composition of the modifier obtained based on croton aldehyde and phthalimine is studied by IR spectra, the resulting substance contains CO-NH_2 bonds characteristic of amide groups are clearly observed in the bands of 3498, 700 and 650 cm^{-1}, –C–N bonds in amide groups were recorded at 1614 and 1269 cm^{-1}, 1597 and 1570 cm^{-1}, while C–H bonds in the benzene ring were recorded at 1070, 862, 810, 752 and 719 cm^{-1};

- The structure of the synthesized modifier was also studied using NMR and PMR analysis methods. When the NMR spectrum of the modifier was analyzed, the proton signals in the benzene ring were observed as follows:
  - H-3 and H-4 at 7.392–7.386 m.u., H-5 at 6.996–6.990 m.u., and H-6 at 7.9 m.u. Additionally, a doublet resonance was observed at H-9 (8.408–8.390 m.u.), another doublet resonance at H-10 (6.129–6.041 m.u.), and a triplet resonance at H-11. A multiplet resonance appeared at 6.149–6.132 m.u., and H-12 exhibited doublet resonance signals at 1.938–1.922 m.u.

- In the ^{13}C NMR spectrum of the modifier, the chemical shifts were as follows:
  - The 1st carbon in the carboxyl group at 118.957 m.u., the 2nd carbon at 147.155 m.u., the 3rd carbon at 128.777 m.u., the 4th carbon at 134.595 m.u., and the 5th carbon at 126.355 m.u. Additionally, carbon at 133.004 m.u., the carboxyl carbon at 170.421 m.u., and the carbon in the -N=CH- group (C-9) at 159.596 m.u. were observed. The carbons C-10 and C-11, connected by a double bond, appeared at 130.601 and 137.756 m.u., respectively, while the carbon in the methyl group was 18.038 m.u. The resulting spectrum confirms the structure of the synthesized substance.

- The degree of bitumen modification of the compound obtained as a result of the interaction of sulfur with the modifier and determine the reaction properties of the modifier molecule, quantum chemical calculations were performed using semi-empirical AM1, MNDO, PM3, RM1 and MNDO3 methods in Avogadro, Hyper Chem 8.01 and Asselrys MS Modeling 3.0.1 programs. Molecular geometry optimization was performed using the Polak-Ribiere (Conjugate Gradient) algorithm. According to Mulliken's method, the obtained results show high values of negative effective charge in modifier molecules shown in oxygen atoms of C=O and C-O-C groups and nitrogen atoms of N-H groups;

- Optimum conditions for modifying bitumen with the synthesized modifier, such as the proportions of substances, temperature and mixing speed, were studied;

- The structure of modified bitumen was studied and analyzed using IR-spectra. In the region around 2902.87 cm^{-1}, absorption bands corresponding to the asymmetric and symmetric stretching vibrations of methyl (-CH_3) and methylene (-CH_2) groups are observed. The 1039.63 cm^{-1} region exhibits vibrations associated with the sulfo bond (S–S group). Stretching vibrations of C=C bonds appear near 1653 cm^{-1}, while C–S–C bond vibrations are detected around 1064.71 cm^{-1}. Additionally, bands at 777.31 cm^{-1} and 877.61 cm^{-1} are attributed to NH–S bonds related to amide groups.

- Morphology and composition of modified bitumen were studied and analyzed using SEM and elemental analysis. Thermogravimetric (TG) and differential thermal analysis (DTA) of modified bitumen were also studied.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Authors' Contribution Statement

M.Rosulov: data curation and formal analysis. Kh.Beknazarov: investigation, methodology, and original draft. J.R Chuliev: review and editing. Ashurov J. M.: review and editing. Sh. U. Babadjanova: conceptualization, writing (original draft), and supervision. All authors have read and agreed to the published version of the manuscript.

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