

PRODUCTION OF BITUMEN MODIFIED WITH LOW-MOLECULAR ORGANIC COMPOUNDS FROM PETROLEUM RESIDUES.

1. EFFECT OF SOLVENT NATURE ON THE PROPERTIES OF PETROLEUM RESIDUES MODIFIED WITH FORMALDEHYDE

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Abstract. The possibility of petroleum residues (tar and oxidized bitumen) modification with formaldehyde (37 % aqueous solution) has been studied at the temperature of 393±3 K for 3 h in the presence of organic solvent and using hydrochloric acid as a catalyst. Toluene, *p*-xylene, naphtha solvent and *n*-octane were used as the solvents in the amount of 0–40 wt % relative to the initial material. By means of IR spectroscopy the resin-like compounds affecting the operational properties of petroleum residues have been detected. These compounds are formed as a result of formaldehyde reaction with residue components and solvent molecules.

Keywords: petroleum residues, formaldehyde, organic solvents, modified bitumen, IR spectroscopy.

1. Introduction

It is known [1–5] that introduction of polymers into the structure of products obtained from petroleum residues improves their operational properties. The amount of introduced polymer additives varies from 1 to 5 wt % [6]. The production volume of only polymer modified bitumen (PMB) in European countries is 10 % of the total quantity of used bitumen [6]. The following polymers are used to modify bitumen: elastomers (natural and synthetic rubber, rubber crumb); thermoplastics (polyethylene, polypropylene, polyvinyl chloride, *etc.*); thermoplastic elastomers (styrene-butadiene-styrene; ethylene vinyl acetate, ethylene-propylene-diene, *etc.*); thermosetting plastics (epoxy resins, polyurethane resins, *etc.*) [1]. Nowadays thermoplastics and thermoplastic elastomers are used most often [1, 6, 7].

Styrene-butadiene-styrene (SBS) is the most often used modifier for PMB production [8, 9]. The modified bitumens are divided into bitumen-polymer and polymer-bitumen binders depending on polymer content. At relatively low content of SBS (<3.5 wt %) bitumen are characterized by higher penetration and softening temperature in comparison with original bitumen; its elasticity is about 85–90 % [10, 11].

Last decades the scientific researchers of the Department of Chemical Technology of Oil and Gas Processing at Lviv Polytechnic National University have been conducting studies on possibility of using petroleum [12–14], phenol-formaldehyde [15], epoxy [16], coumarone-indene [17] and phenol-cresol-formaldehyde [18–22] resins as polymeric additives.

The above mentioned resins contain free epoxy, carboxy, hydroxyl and methacrylic groups in their structure. They are compatible with petroleum residues and improve operational characteristics of bitumen, namely its adhesion to mineral surfaces.

To continue the investigations regarding the improvement of operational characteristics of the materials based of petroleum residues, this work deals with the possibility of obtaining high-quality paving bitumen using formaldehyde. The idea to use formaldehyde for the bitumen production from petroleum residues, *e.g.* tar, is grounded on the fact that they contain the compounds with aromatic chains [23–24]. On the other hand it is known [20] that in the presence of acid catalysts the aromatic compounds can react with formaldehyde *via* condensation reaction and participate in the formation of carbon-formaldehyde resins. Naphthalene, anthracene, phenanthrene, fluorine and others serve as aromatic compounds in this case [25].

To our mind, during the process of bitumen production from tar and during bitumen modification, when aromatic compounds, which are contained in petroleum residues, react with formaldehyde, the formation of arene-

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formaldehyde resins will occur. These resins will modify the bitumen properties. However, petroleum residues are viscous systems under the modification conditions (temperature is 393K).

Thus, the aim of this work is to study the effect of solvent amount and nature on the modification of petroleum residues with formaldehyde, in particular the effect on softening temperature, penetration and adhesion to stone of the modified residues.

2. Experimental

2.1. Materials

The following materials were used for chemical modification of petroleum residues with formaldehyde:

- Tar produced at JSC Ukratnafta (Kremenchuk, Ukraine). Its characteristic is given in Table 1, designated as T1.

- Paving bitumen BND 70/100 (oxidized bitumen) produced at JSC Ukratnafta (Kremenchuk, Ukraine). Its characteristic is given in Table 2, designated as BO1.

- Formaldehyde (37% aqueous solution, formalin) was used as a modifier.

- Hydrochloric acid (37%, density 1.19 g/cm³) of P.A. grade was used as a catalyst.

- Solvents:

- toluene – colorless liquid with specific odor, density of 0.867 g/cm³ and boiling point of 383.6 K;

- *p*-xylene with density of 0.861 g/cm³ and boiling point of 411.3 K;

- petroleum solvent with density of ≤0.860 g/cm³ and boiling point of ≥403 K;

- *n*-octane with density of 0.703 g/cm³ and boiling point of 398.5 K.

To determine a value of adhesion to stone, a natural stone (fraction 5–20 mm) was purchased at JSC Novo-grad-Volynsky gravel factory (Ukraine). Its characteristic is represented in Figs. 1, 2 and Tables 3, 4.

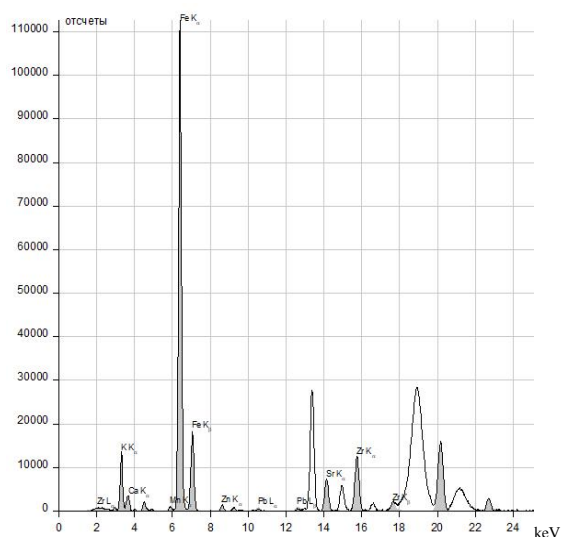


Fig. 1. XRF spectrum of stone heavy elements

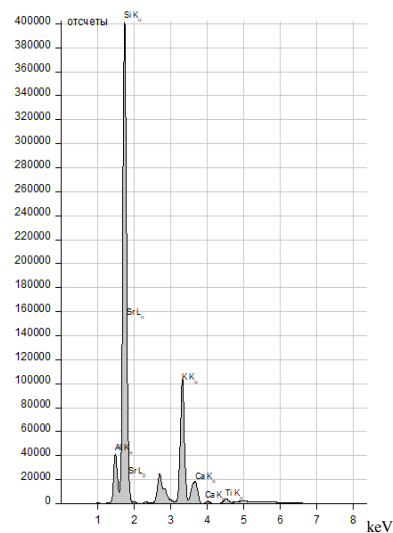


Fig. 2. XRF spectrum of stone light elements

Table 1

Characteristics of tar T1

Index	Value	Standard or Ref.
Flash point (open cup), K	555	ISO 2592:2000
Penetration at 298 K, m·10 ⁻⁴ (0.1mm)	247	EN 1426:2018
Softening temperature (R&B), K	312	EN 1427:2018
Ductility at 298 K, m·10 ⁻² (cm)	58.1	EN 13398:2018
Adhesion to crushed stone, points	3	[21]
Fraassbreaking point, K	255	EN 12593:2018
Ductile temperature range, K	337	[21, 22]
Penetration index	-1.5	[22]

Table 2

Characteristics of bitumen BO1

Index	Value	Standard or Ref.
Penetration at 298 K, $m \cdot 10^{-4}$ (0.1mm)	91	EN 1426:2018
Softening temperature (R&B), K	321	EN 1427:2018
Ductility at 298 K, $m \cdot 10^{-2}$ (cm)	>100	EN 13398:2018
Adhesion to crushed stone, points	3	[21]
Fraass breaking point, K	253	EN 12593:2018
Ductile temperature range, K	340	[21, 22]
Penetration index	-1.5	[22]

Table 3

Elemental composition of crushed stone

Element	Series	Intensity, rel.units	Concentration, wt %
Al	K	295851	6.07
Si	K	3000151	25.45
K	K	71229	15.40
Ca	K	14977	2.73
Ti	K	11603	0.77
Mn	K	6370	0.10
Fe	K	672667	7.08
Zn	K	8806	0.03
Ga	K	5401	0.01
Sr	K	59667	0.03
Zr	K	100842	0.05
Pb	L	7643	0.01

Table 4

Oxide composition of crushed stone

Compound	Series	Intensity, rel.units	Concentration, wt %
Al_2O_3	K	295851	11.47
SiO_2	K	3000151	54.45
K_2O	K	71229	18.55
CaO	K	14977	3.82
TiO_2	K	11603	1.28
MnO	K	6370	0.13
Fe_2O_3	K	672667	10.12
ZnO	K	8806	0.04
Ga_2O_3	K	5401	0.02
SrO	K	59667	0.04
ZrO_2	K	100842	0.06
PbO	L	7643	0.02

2.2. Experimental Procedure**2.2.1. Modification of petroleum residues with formaldehyde**

The modification was carried out in hermetic metal vessels capable to withstand an extreme pressure (Fig. 3).

The vessel is loaded with a raw material (tar or bitumen) preheated to the temperature providing its movable state and cooled to the temperature below boiling points of formalin and solvent. Then an organic solvent is added and the mixture is stirred till the homogeneous state. After adding formaldehyde and a catalyst the vessel is closed hermetically, placed into a thermostat, heated to

393±3 K and kept for 3 h. The mixture is shaken from time to time in order to intensify the process. After 3 h the vessel is cooled and volatile products are distilled at 363–368 K and residual pressure of 1.33–1.99 kPa. The resulting product is dried in a vacuum cabinet at 393 K and residual pressure of 2 kPa. The product yield, softening temperature, penetration, Fraass breaking point and adhesion to crushed stone are determined.

The modification conditions are shown in Table 5.

2.3. Methods of Analysis

2.3.1. Physico-technological characteristics

The following physico-technological characteristics of the initial materials and products were determined according to the standard procedures: softening temperature (EN 1427:2018); penetration [22]; Fraass breaking point (EN 12593:2018); adhesion to crushed stone [21].

2.3.2. Spectral investigations

IR spectra were recorded using Thermo scientific Nicolet iS10 Fourier-spectrophotometer. The results were processed using of the spectrophotometer software.

XRF analysis of crushed stone was carried out by the Laboratory of Advanced Technologies for the Creation and Physico-chemical Analysis of New Compounds and Functional Materials (Lviv Polytechnic National University, Ukraine) using ElvaX Light SDD spectrometer (Elvatech Company).

Fluorescence spectrum of light elements (from Na to Cl) was recorded with additional blowdown of the system by helium. For the elements with higher atomic number the spectrum was recorded without helium.

Fluorescence peaks of K and Ca are observed in both spectra due to the presence of high- and low-energy internal transitions in these elements.

The interpretation of the spectra was carried out taking into account the localization of the peaks that fall at the energy maximum of the electronic transition or the presence of all maxima of the characteristic transitions.

The quantitative composition was estimated using the device software by the method of deriving the ratios of the peak areas of each individual element to the total peak area of all elements according to the algorithm of fundamental parameters.



Fig. 3. Vessel for petroleum residues modification

Table 5

Conditions for T1 and BO1 modification with formaldehyde

Index	Value relative to the initial petroleum residue, wt %
Formalin amount (including formaldehyde)	10.0 (3.7)
Catalyst (HCl) amount	2.5
Solvent	0–40.0

Notes: values are calculated per 100 g of petroleum residue. Toluene, *p*-xylene, naphta solvent, and *n*-octane are the solvents.

3. Results and Discussion

The modification process was carried out in a hermetic vessel because formaldehyde is a high-volatile material. On the other hand, the initial petroleum residues are high-viscous components. Taking these facts into consideration it was necessary to examine the effect of

stirring on the modification process. The experimental results are represented in Table 6.

It is obvious that stirring actually does not affect the characteristics of the resulting products. Therefore, further investigations were carried out without stirring according to the procedure described in subsection 2.2.1. The results are represented in Tables 7 and 8.

Table 6

Effect of process conditions on the characteristics of the resulting products

Characteristic of the modified product	Process conditions	
	under constant stirring	without stirring
Based on T1		
Softening temperature (R&B), K	325	323
Penetration at 298 K, $m \cdot 10^{-4}$ (0.1mm)	41	53
Fraass breaking point, K	270	269
Adhesion to crushed stone, points	3	3
Based on BO1		
Softening temperature (R&B), K	333	331
Penetration at 298 K, $m \cdot 10^{-4}$ (0.1mm)	28	33
Adhesion to crushed stone, points	3	3.5

Notes: toluene is a solvent (40 wt % relative to the initial material); temperature is 393 ± 3 K, time is 3 h. The characteristics of the initial T1 and BO1 are given in Tables 1 and 2, respectively.

Table 7

Effect of solvent nature and amount on the characteristics of the resulting products based on T1

Solvent and its amount, wt%	Product yield relative to the initial T1, wt %	Softening temperature (R&B), K	Penetration at 298 K, 0.1 mm	Fraass breaking point, K	Adhesion to crushed stone, points
Without solvent	107	325	55	264	3.5
Toluene, 20	108	321	85	267	3.0
Toluene, 30	109	322	65	268	3.0
Toluene, 40	112	323	53	269	3.0
<i>p</i> -Xylene, 20	111	316	118	261	3.5
<i>p</i> -Xylene, 30	108	318	103	268	3.0
<i>p</i> -Xylene, 40	105	317	93	268	3.0
Naphta solvent, 20	108	320	73	–	3.5
Naphta solvent, 40	105	321	70	–	3.0
n-Octane, 20	105	324	78	267	4.0
n-Octane, 30	106	324	74	267	3.5
n-Octane, 40	101	326	62	–	3.0

Note: for the initial T1 a softening temperature is 312 K, penetration is 247, adhesion to stone is 2.5 points

Table 8

Effect of solvent nature and amount on the characteristics of the resulting products based on BO1

Solvent and its amount, wt%	Product yield relative to the initial T1, wt %	Softening temperature (R&B), K	Penetration at 298 K, 0.1 mm	Fraass breaking point, K	Adhesion to crushed stone, points
Without solvent	103	326	42	268	3.5
Toluene, 20	106	332	28	–	3.5
Toluene, 40	106	331	33	–	3.5
Naphta solvent, 20	107	326	46	–	4.0
Naphta solvent, 40	105	331	34	–	3.5
n-Octane, 20	108	332	26	269	3.0
n-Octane, 40	104	332	28	269	3.0

Note: for the initial BO1 a softening temperature is 319 K, penetration is 70, adhesion to stone is 2.5 points

One can see from Tables 7 and 8 that it is possible to obtain improved bitumen *via* modification of petroleum residues with formaldehyde. The resulting products have a softening temperature of 317 K and higher; penetration is more than 70. The value of adhesion is improved as well. At the same time, it is obvious that the solvent has more impact for BO1, which is more viscous material. For example, when T1 is modified without the solvent, the softening temperature increases by 13 K, whereas when toluene, *p*-xylene, naphta or *n*-octane is used as the solvent (40 wt %), this value increases by 11, 5, 9 or 14 K, respectively. When BO1 is modified without the solvent (Table 8), the softening temperature increases by 7 K, whereas when toluene, naphta or *n*-octane is used as the solvent (40 wt %), this value increases by 12, 12 or 13 K, respectively.

Apart from the improvement of some indices, the increase in product yield is observed (Tables 7, 8). This indicates that formaldehyde react with petroleum residue or the solvent. The reaction of formaldehyde molecules and those of petroleum residue is confirmed by the increase in yield after modification without the solvent.

The possible reaction of formaldehyde with the solvent is confirmed by IR spectroscopy. For this purpose the experiments were carried out according to the procedure described in subsection 2.2.1 but without petroleum residues. The ratio formaldehyde : catalyst (HCl) : solvent = 4 : 1 : 16 was chosen in such a way, that solvent amount would be 40 wt %. Resin-like products were obtained and analyzed (Figs. 4-7, Table 9). IR spectra of pure solvents were taken for the comparison.

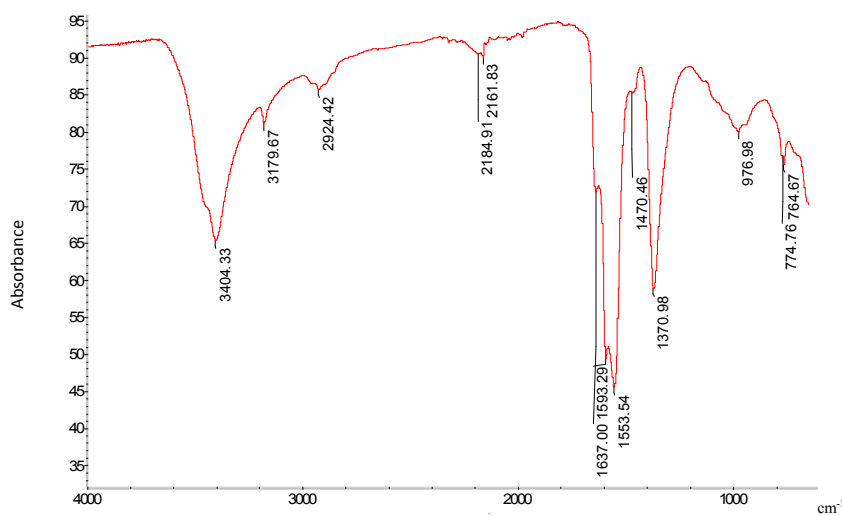


Fig. 4. IR spectrum of the product obtained *via* reaction of formaldehyde and toluene

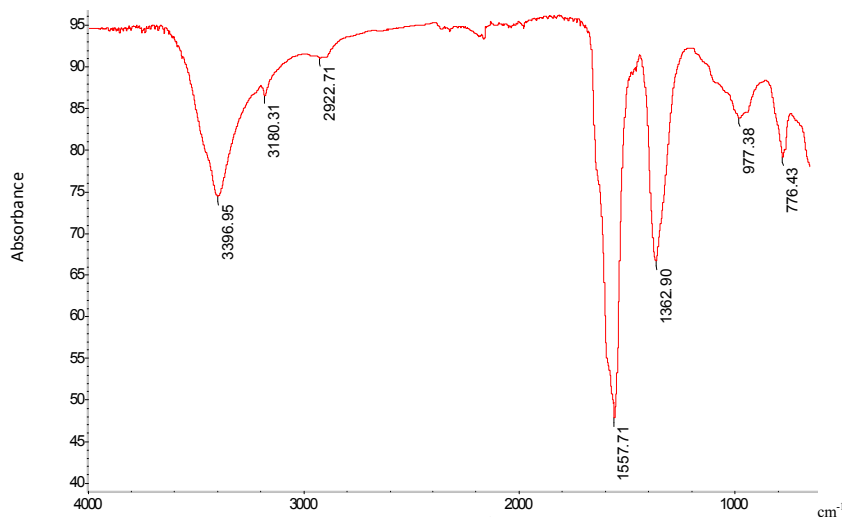


Fig. 5. IR spectrum of the product obtained *via* reaction of formaldehyde and *p*-xylene

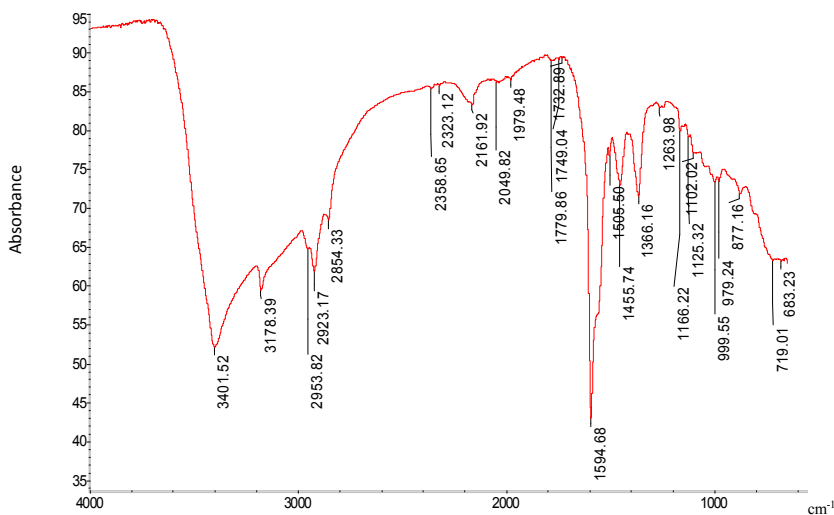


Fig. 6. IR spectrum of the product obtained *via* reaction of formaldehyde and naphta solvent

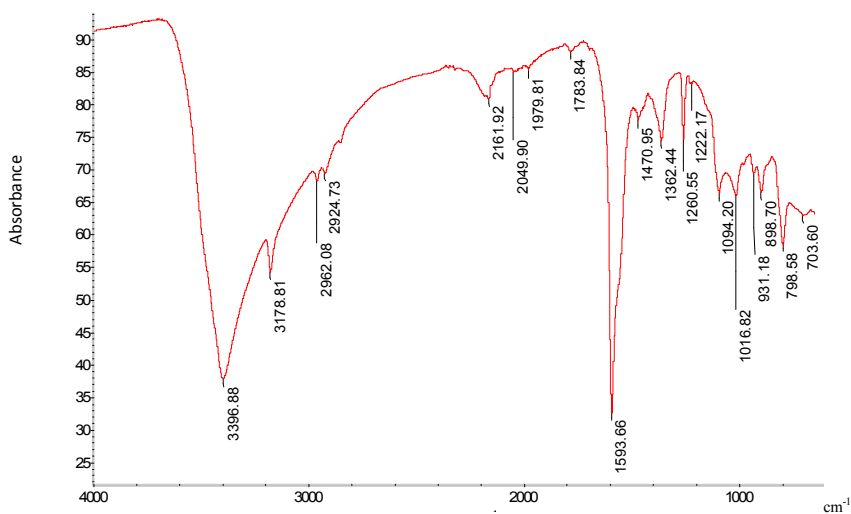



Fig. 7. IR spectrum of the product obtained *via* reaction of formaldehyde and *n*-octane

Table 9

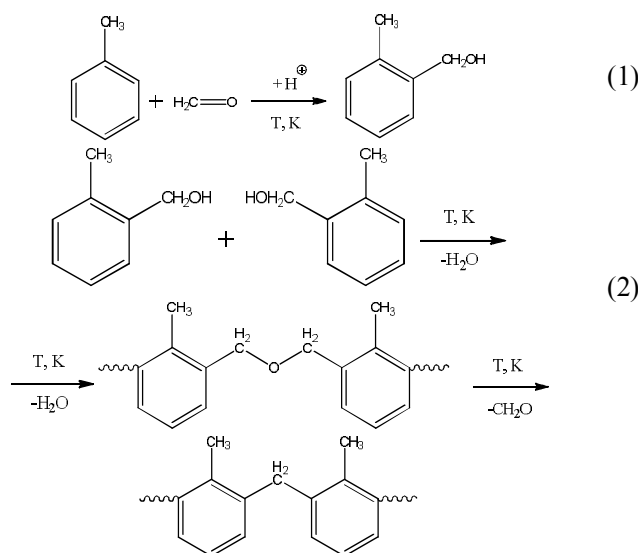
Yield of resin-like products (areno-formaldehyde resins) and their IR characteristics

Solvent	Yield of resin-like products, g/100 g of formaldehyde	Absorption bands, cm ⁻¹					
		$\begin{array}{c} \text{---OH} \\ \\ \text{---C---} \\ / \quad \backslash \end{array}$	---CH_3	$\text{---CH}_2\text{---}$		mono- and disubstituted arenes	di- and trisubstituted arenes
Toluene	80	3404 1371	3180	2924	1554	775	977
<i>p</i> -Xylene	92	3397 1363	3180	2923	1558	776	977
Naphta solvent	63	3402 1366	3178	2923	1595	719	877
<i>n</i> -Octane	35	3397 1362	3179	2924	1595	799	899

In the spectrum of the product obtained in the presence of toluene (Fig. 4, Table 9) we observe the absorption band at 1554 cm^{-1} corresponding to the stretching vibrations of benzene ring. Moreover, this spectrum shows absorption bands at 775 and 977 cm^{-1} indicating the presence of mono-, di- and trisubstituted benzene rings in the product. If the band at 775 cm^{-1} may be attributed to $-\text{CH}_3$ group of toluene, then di- or trisubstituted benzene rings indicate the reaction of toluene with formaldehyde molecules resulting in the formation of $-\text{CH}_2-$ groups. The presence of these groups is confirmed by absorption band at 2924 cm^{-1} .

The absorption bands at 3404 and 1370 cm^{-1} (Fig. 4) corresponding to $-\text{OH}$ and >C-OH groups, respectively, arouses the interest.

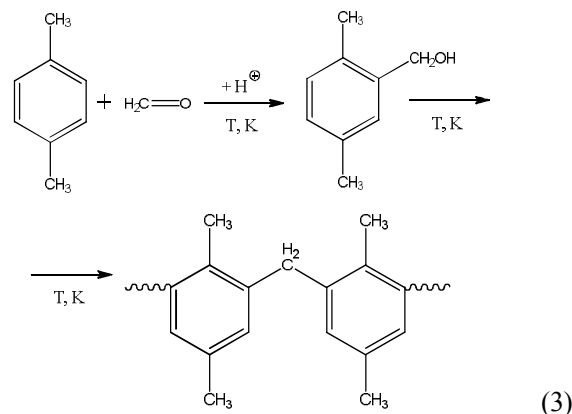
Thus we assume that during tar modification with formaldehyde in the presence of the catalyst (HCl) and toluene as the solvent, the following reactions occur:



The formation of products according to Eq. (1) is confirmed by the absorption bands at 3404 and 1370 cm^{-1} typical of stretching vibrations of $-\text{OH}$ and $-\text{CH}_2\text{OH}$ groups. The absorption bands at 1270 – 1020 cm^{-1} are absent in the spectrum. This indicates that formaldehyde is detached according to Eq. (2) with a high rate followed by the formation of toluene-formaldehyde resin. The absorption bands at 977 and 774 cm^{-1} confirm this.

The results for the product obtained *via* reaction of formaldehyde and *p*-xylene are similar (Fig. 5, Table 9).

The formation of xylene-formaldehyde oligomers based on *p*-xylene may be expressed by the following equation:



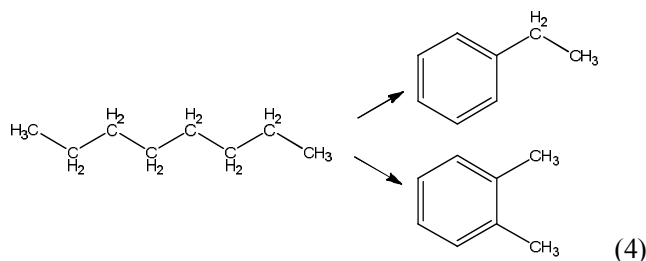
Similar to the previous case, the *p*-xylene based oligomer contains free $-\text{CH}_2\text{OH}$ group (3397 and 1363 cm^{-1} , Table 9), which indicates the presence in the resulting product of the mixture that consists of the resulting oligomer and methylol derivatives of *p*-xylene.

Naphta solvent, which is a mixture of xylene isomers, reacts with formaldehyde in a similar way (see Eqs. 1-3). The spectrum of the resulting product is given in Fig. 6 and its interpretation – in Table 9.

Unlike toluene, *p*-xylene and naphta solvent, *n*-octane does not contain aromatic compounds but it is an effective solvent for the modification of tar or bitumen. The product obtained in the presence of *n*-octane is a powdered compound, the spectrum of which is represented in Fig. 7 and its interpretation in Table 9. The absorption band at 1595 cm^{-1} corresponding to the stretching vibrations of benzene ring is observed. Moreover, there are also the absorption bands at 799 and 899 cm^{-1} typical of stretching vibrations of di- and trisubstituted aromatic rings. The bands at 3397 and 1362 cm^{-1} indicate the presence of free $-\text{OH}$ and $-\text{CH}_2\text{OH}$ groups, as well as the band at 1094 cm^{-1} typical of aliphatic ethers (>C-O-C<).

The absorption bands at 3397 , 1362 and 1595 cm^{-1} are absent in the IR spectrum of pure *n*-octane (Fig. 8) indicating the absence of free hydroxyl groups and benzene rings in its structure.

Thus, under the experimental conditions the dehydrocyclization of *n*-octane molecules takes place and benzene structures of the following structure are formed:



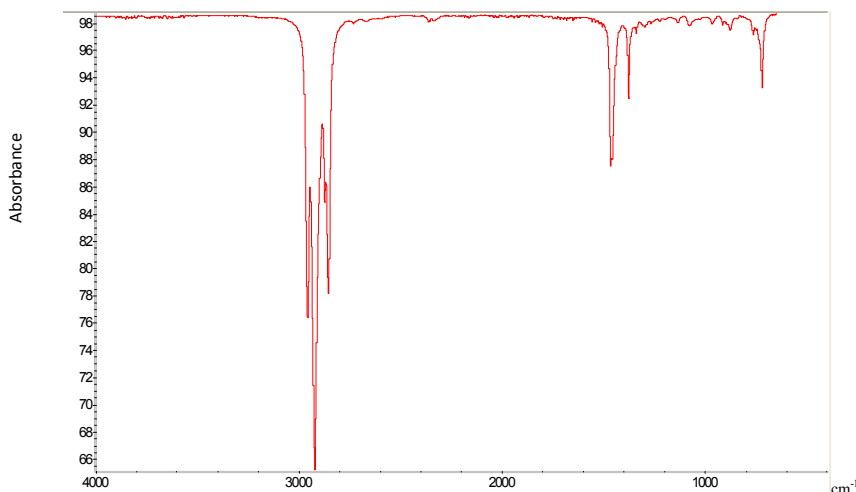
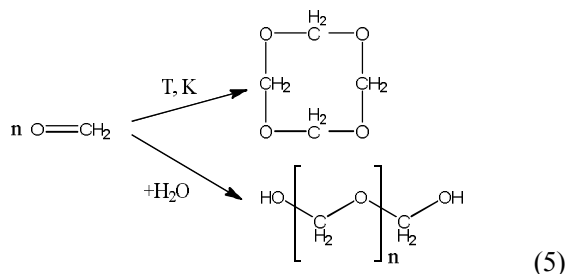


Fig. 8. IR spectrum of *n*-octane

The catalyst of this reaction may be FeCl_3 , which is formed due to the reaction of hydrochloric acid and metal surface of the vessel used for the synthesis. This is evidenced by the corrosion appeared on the internal surface of the vessel. Aromatic compounds which are formed according to Eq. (4) then react with formaldehyde in accordance with Eqs. (1-3), the same as in the case with toluene, *p*-xylene and naphtha solvent. In addition to the possible conversion of *n*-octane into aromatic compounds occurred under pressure in the presence of the catalyst, the formaldehyde condensation (Eq. 5) is possible too.



The possibility of reaction (5) proceeding is confirmed by the absorption bands at 3397 and 1362 cm^{-1} (Fig. 7) indicating the presence of $-\text{OH}$ and $-\text{CH}_2\text{OH}$ groups, as well the absorption band at 1094 cm^{-1} attributed to etheric bond ($\text{>C}-\text{O}-\text{C}<$).

All above mentioned results indicate the role of the solvent in the process of tar chemical modification due to the formation of oligomeric products. Under the reaction conditions (393 K , 3 h) formaldehyde, which was used for the modification, in the presence of the acid catalyst (HCl) is capable to react with the compounds presented in petroleum residues and with the solvent molecules. The reaction results in the formation of arene-formaldehyde resins, which play the role of polymeric modifiers in the resulting products and thus improve their operational characteristics.

4. Conclusions

The possibility of using formaldehyde as 37% aqueous solution to modify petroleum residues in the presence of organic solvents was established. It was determined that the solvents are most effective for the modification of high-viscous materials, bitumen in particular. *n*-Octane in the amount of 20 wt% relative to the initial residue was found to be the most effective among the solvents under study. The results of IR spectroscopic investigation confirm that arene-formaldehyde resins are not formed during modification process in the presence of toluene, *p*-xylene and naphtha solvent. When using *n*-octane as the solvent, dehydrocyclization of *n*-octane molecules followed by the formation of benzene compounds and condensation of formaldehyde molecules followed by the formation of linear and cyclic ethers can occur.

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ОДЕРЖАННЯ БІТУМУ, МОДИФІКОВАНОГО НИЗЬКОМОЛЕКУЛЯРНИМИ ОРГАНІЧНИМИ СПОЛУКАМИ ІЗ НАФТОВИХ ЗАЛИШКІВ. 1. ВПЛИВ ПРИРОДИ РОЗЧИННИКА НА ВЛАСТИВОСТІ НАФТОВИХ ЗАЛИШКІВ, МОДИФІКОВАНИХ ФОРМАЛЬДЕГІДОМ

Анотація. Вивчена можливість модифікування нафтових залишків (гудрону і окисненого бітуму) формальдегідом у вигляді 37% водного розчину за температури 393 ± 3 K впродовж 3 годин в присутності органічного розчинника з використанням як каталізатор хлорної кислоти. Як органічний розчинник використано толуен, п-ксилен, нафтовий сольвент і н-октан в кількості від 0 до 40 % мас. на вихідний нафтовий залишок. ІЧ-спектроскопічними дослідженнями встановлено утворення між молекулами формальдегіду та сполуками, що містяться у нафтовому залишку й молекулами розчинника смоло подібних продуктів, які впливають на експлуатаційні характеристики нафтових залишків.

Ключові слова: нафтові залишки, формальдегід, органічні розчинники, модифіковані бітуми, ІЧ-спектроскопія.