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Chemistry

EFFECT OF BISPHENOL-FORMALDEHYDE RESIN ON PHYSICO-MECHANICAL PROPERTIES OF ROAD BITUMEN

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Abstract. A bisphenol-formaldehyde resin was synthesized using the polycondensation method of bisphenol A with formaldehyde. Road bitumen has been modified with this resin. The possibility of its use as a road petroleum bitumen modifier has been established for different contents of the synthesized resin. It has been established that the introduction of synthesized bisphenol-formaldehyde resin into the composition of bitumen significantly increases its heat resistance. The synthesized resin and modified bitumens were characterized using IR spectroscopy. The change in the composition and properties of the bitumen modified with bisphenol-formaldehyde resin has been described.

Keywords: bisphenol A, bitumen modification, IR spectroscopy

1. Introduction

It is known that traditional asphalt concretes based on unmodified bitumen cannot provide the necessary physical and mechanical properties of road surfaces and their durability. Therefore, it is necessary to improve the quality of materials and operational reliability of layers of road clothing. An effective way to solve this problem is to improve the structure and properties of bitumen by using inexpensive modifiers of various types.[1-4]

The processes of modification of oil residues are divided into physical and chemical. Physical modification of oil residues involves the introduction of various types of applications into their composition, which ensure improvement of operational characteristics. At the same time, the chemical structure of the residue remains unchanged.[5,6] The use of physical action modifiers has certain disadvantages, namely low thermal stability and problems with the homogeneity of the obtained bitumen-polymer mixtures, so it is more appropriate to use chemical modification. Chemical modification of oil residues involves changing their chemical structure as a result of the action of the modifier, usually in the presence of a catalyst/initiator. As a result, products with improved operational characteristics are obtained (compared to the original oil residues). Chemical modification of oil residues, in general, and bitumen in particular, has been studied and used on a much smaller scale, compared to physical modification. At the same time, quite often chemical modification manages to obtain no worse, or even better, results from the point of view of the operational characteristics of the modification products.[7-10]

Among the chemical modifiers that are actively used today to improve and preserve the initial properties of petroleum bitumens, the class of reactive plastics occupies a significant place. In particular, the most widely used: reactive terpolymers (RET),[11,12] epoxy resins,[13-15] phenol-formaldehyde [16,17] and polyester resins.[18,19]

It is also known that one of the effective modifiers of petroleum bitumen are phenol-aldehyde resins.[20-22]

Phenol-aldehyde resins are liquid or solid oligomeric products obtained as a result of the polycondensation reaction of phenol, its homologues (cresols, xylenols) or polyatomic phenols (resorcinol) with aldehydes (formaldehyde or furfural). However, the most widely used resins obtained as a result of polycondensation of phenol and formaldehyde.[23-25]

The article[20] describes a number of studies devoted to the production and use of phenol-formaldehyde resins (FFS) as a modifier of petroleum road bitumens. It was established that the introduction of 2 wt.% of phenol-formaldehyde resin into bitumen leads to an increase in the softening temperature of bitumen from 322.2 to 324 K, that is, its heat resistance increases, bitumen penetration at a temperature of 298 K slightly decreases from 62 to 60 dmm. The penetration index of the original bitumen is –0.902, and that of the modified bitumen is –0.523. This can be explained by the fact that the introduction of phenol-formaldehyde resin into the composition of bitumen reduces the sensitivity of the binder to heating. Also,

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the addition of resin increases the kinematic viscosity at 408 K from 0.35 to 0.44 Pa·s. In the studies described in the article,[21] it was established that the optimal content of Bakelite (phenol-formaldehyde resin of the resol type) in BMP is 1.75 wt.%. When adding this amount of bakelite, there is an increase in Marshall stability from 20 to 24.2 kN, an increase in the softening temperature from 325 to 334 K, and a decrease in bitumen penetration at a temperature of 298 K from 49 to 39 dmm. In the article[22], it was established that the introduction of 2 wt.% Bakelite into bitumen leads to an increase in the softening temperature of bitumen from 331 to 339 K, *i.e.*, its heat resistance increases, the penetration of bitumen at a temperature of 298 K decreases from 62 to 20 dmm, as well as an increase in the kinematic in viscosity at 408 K from 0.4 to 0.98 Pa·s, which will allow the operation of roads in hot climates. Research related to the use of coal tar as a raw material for obtaining phenols from it, with the subsequent synthesis of phenol-cresol-formaldehyde resins based on them, deserves special attention. The obtained phenol-cresol-formaldehyde resins can serve as polymer additives to improve the properties of road petroleum bitumens, primarily adhesion with mineral materials. The use of such a modifier contributes to ensuring a complete, irreversible and water-resistant connection between the bituminous binder and the stone material.[26-28]

Observing the positive effect of the use of phenol-formaldehyde resins on the physical and mechanical properties of road petroleum bitumens, the idea arose to conduct research on the effect of bisphenol-formaldehyde resins on the corresponding properties of bituminous binders.

2. Experimental

2.1. Materials

Bitumen produced by JSC Ukrtatnaphta (Kremenchuk, Ukraine) was used as a raw material. Its characteristic is presented in Table 1.

The following materials were used for bisphenol-formaldehyde resin synthesis:

- bisphenol A (purity 97 %, crystalline with melting point 426 K) was purchased from Sigma Aldrich (Taufkirchen, Germany).
- formalin stabilized: mass fraction of formaldehyde 37 %, acid content per ant 0.1 %;
- concentrated hydrochloric acid: HCl content – 37 % and d_4^{20} 1.19 (used as a catalyst).

2.2. Experimental Procedure

Bisphenol-formaldehyde resin (BPhFR) was obtained by polycondensation of bisphenol A with formaldehyde according to the method.[37,38] For synthesis we used a laboratory facility for the synthesis (see Fig. 1). Bisphenol A, formalin, and hydrochloric acid are loaded into the reactor, a mechanical stirrer is turned on, and the reactor is heated in a boiling water bath. Mixing of the reacting components was carried out for 30 minutes. After that, the resin was washed with warm distilled water. The resulting resin was poured into a porcelain bowl and dried under vacuum at a temperature of 333 K to a constant mass. The conditions of resin synthesis are given in Table. 2.

2.3. Test methods

FTIR spectra were recorded on a Spectrum Two spectrometer (PerkinElmer, USA) using a diamond U-ATR single reflection accessory. PerkinElmer Spectrum software was used to draw the spectra. The spectra (16 scans per spectrum) of the samples were collected in the mid-infrared wavenumber range from 1800 to 400 cm^{-1}, with a spectral resolution of 4 cm^{-1}.

The physical and mechanical properties of bituminous material and methods of their implementation are given in Table 1.

3. Results and Discussion

On the basis of experimental results the mass balance for obtaining BPhFR was calculated (see Table 3). The yield of the resulted resin was 71.05 wt.%.

Table 1. Physico-chemical characteristics of OB

Index	Unit of measurement	Value	Method
Penetration at 298 K	dmm	82.5	EN 1426:2015[29]
Softening point (SP)	K	322.5	EN 1427:2015[30]
Ductility at 298 K	cm	>150	According to[9]
Dynamic viscosity at 333 K	Pa·s	96	EN 12596:2018[31]
Kinematic viscosity at 408 K	mm^2/s	283	EN 12595:2018[32]
Solubility	%	99.95	EN 12592:2018[33]
Adhesion to gravel	mark	3.0	DSTU 8787[34]
Fraas breaking point (FBP)	K	261	EN 12593:2018[35]
Plasticity interval (PI)	K	59.2	PI = SP – FBP
Penetration index	–	-0.05	EN 12591[36]

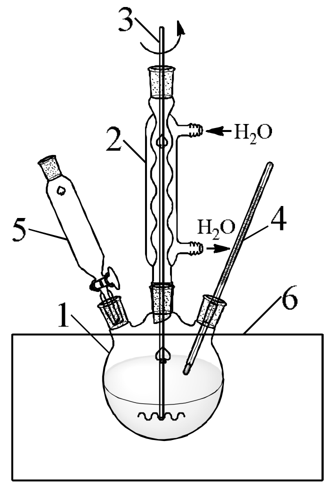

Fig. 1. Laboratory facility for the synthesis of BPhFR:
1 – three-necks flask; 2 – Allihn condenser; 3 – mixer;
4 – thermometer; 5 – dropping funnel; 6 – thermostat

Table 2. Conditions for BPhFR synthesis

Parameter	Unit of measurement	Value
Weight ratio of "raw" phenols / formalin (formaldehyde content in formalin – 37 wt.%)	–	1.92
Catalyst weight content (concentrated HCl) by bisphenol A	wt.%	3.0
Temperature	K	353.0
Process duration	min	30.0

Table 3. Mass balance for obtaining BPhFR

Components	g	wt.% relative to the reactor loading
Loaded		
Bisphenol A	24.00	64.34
Formalin	12.50	33.51
Concentrated HCl	0.80	2.14
Total:	37.30	100.00
Obtained		
BPhFR	26.50	71.05
Water and unreacted components	10.80	28.95
Total:	37.30	100.00

The condensation reaction of bisphenol A with formaldehyde using an acid catalyst is as follows:

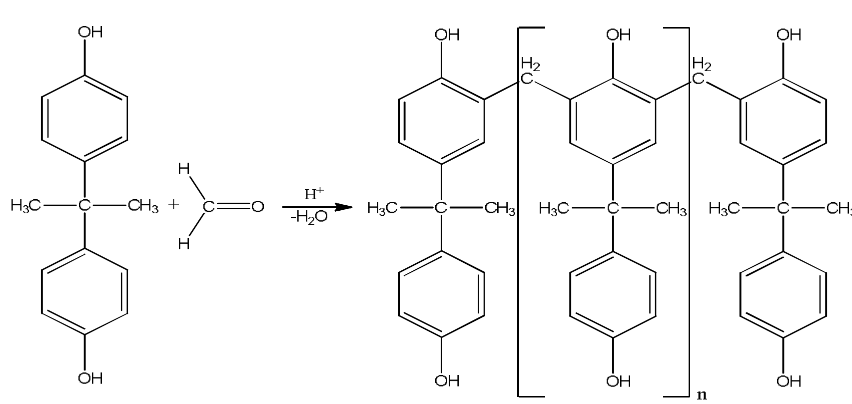

Table 4. Comparison of the main characteristics of pure and BPhFR modified bitumen

Index	Unit of measurement	BND 60/90 (OB 4)	BND 60/90 + BPhFR (0.5 wt.%)	BND 60/90 + BPhFR (1.0 wt.%)
Penetration at 298 K	dmm	82.5	54.5	30.0
Penetration decline	%	–	33.9	63.6
Softening point	K	322.5	327.3	337.2
Softening point increase	%	–	9.7	29.7
Ductility at 298 K	cm	>150	72	46
Adhesion to gravel	mark	3.0	3.0	3.5
Penetration index	–	-0.05	0.02	0.64

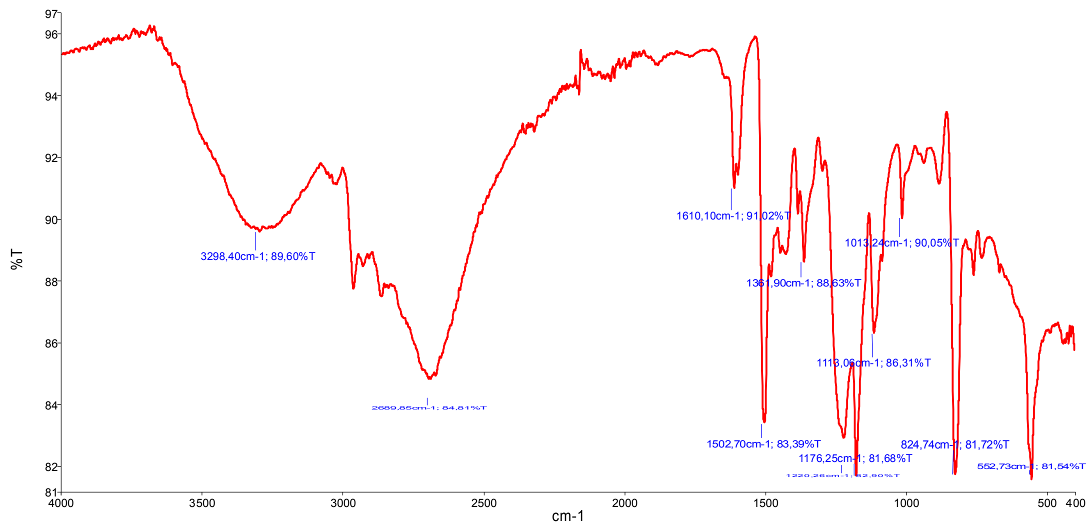

Fig. 2. FTIR spectrum of BPhFR

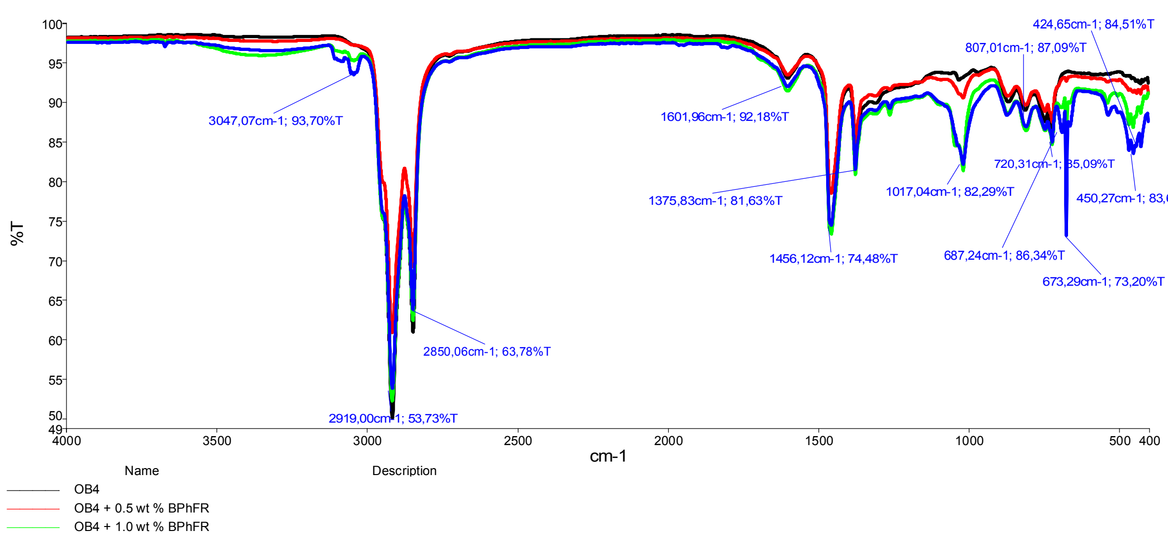

Fig. 3. FTIR spectrum of bitumen modified with BPhFR in range 4000-400 cm^{-1}

To establish the possibility of using BPhFR as a modifier of petroleum bitumens, modification of oxidized bitumen with the obtained resin was carried out. Bitumen modification was carried out at 463 K and a modifier content of 0.5 and 1.0 wt.%. Table 4 shows the main characteristics of the obtained bitumen-polymer compositions.

Based on the data given in Table 4, it can be argued that addition of the synthesized BPhFR resin into bitumen significantly increases the heat resistance of modified bitumen. The softening temperature increases by 4.8 K at 0.5 wt.% of BPhFR and by 14.7 K at 1.0 wt.% of BPhFR. On the other hand, addition of the synthesized resin into the bitumen composition leads to a decrease in the penetration of modified bitumens. It is worth noting that the introduction of only 1.0 wt.% of the obtained resin into the bitumen composition leads to a slight increase in adhesion with the surface of the stone material.

In order to establish the nature of both the BPhFR resin itself and the process of bitumen modification, the IR spectroscopic studies have been carried out according to the methodology described above. The IR spectra of resin and bitumen modified with this resin are shown in Figs. 2-4. Identification of IR absorption bands was carried out according to works.[39]

As shown in Figs. 2-4 results of the IR spectroscopic study, the spectra of BPhFR and bitumen modified by it are not identical. In the spectrum of BPhFR there is a broad absorption band at 3298 cm^{-1}, which is characteristic of valence vibrations of the hydroxyl group in phenols. Such a band is absent in modified bitumens. At the same time, in the spectra of BPhFR and bitumens modified by it, there is an absorption band at 3047 cm^{-1}, which corresponds to valence vibrations of –CH_2– bonds in aromatic structures, while the intensity of the band is insignificant in the unmodified sample, as well as at the content of 0.5 wt.% BPhFR in bitumen , and increases significantly with increasing modifier content, reaching a maximum at 1.0 wt.%.

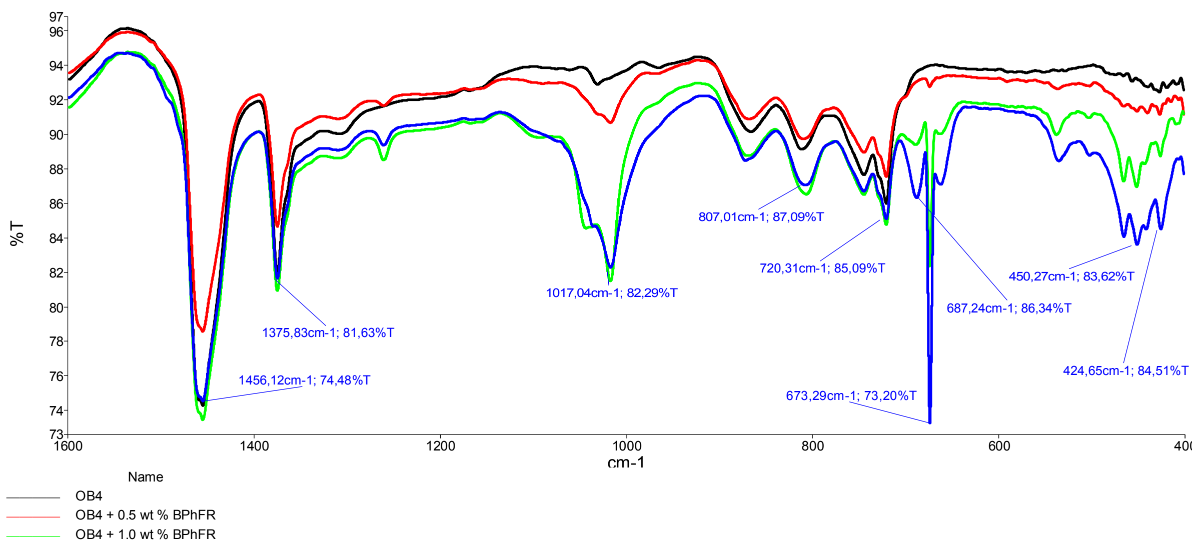

Fig. 4. FTIR spectrum of bitumen modified with BPhFR in range 1600-400 cm^{-1}

In the BPhFR sample, there are absorption bands that are absent in the modified bitumens:

1502 cm^{-1} – valence vibrations of C=C bonds in aromatic rings;

1361 cm^{-1} – doublet of heme-dimethyl deformation vibrations characteristic of the $(CH_3)_3C$– group;

1220 cm^{-1} – valence vibrations of C−O phenolic bonds in the aromatic ring Ar–O–;

1176 cm^{-1} – deformational vibrations of the C−H bonds of the –CH=CH– group in the plane of the aromatic ring Ar;

1113 cm^{-1} – deformational vibrations of C−H bonds in the plane of the aromatic ring;

824 cm^{-1} – deformation vibrations of C−H bonds out of the plane of the aromatic ring (most characteristic for the presence of alkyl substituents).

The absorption band of 1610-1602 cm^{-1} indicates the presence in all samples of aromatic compounds containing methyl substituents (1456 cm^{-1}) and which are connected to each other by –CH_2– bonds (1375 cm^{-1}). Moreover, the intensity of these bands increases with an increase in the amount of the BPhFR modifier in the bitumen. The systematic increase in intensity for the bands with maxima at 1602, 1456, 1375, 1017, 720, 450, and 424 cm^{-1} with an increase in the amount of BPhFR in the bitumen is obviously explained by a significant increase in the background absorption There is a clear increase in the number of groups characteristic of resins, which is possible only when the modifier is dissolved in bitumen or mixed with it.

The absorption band at 1017-1013 cm^{-1} detected in the spectra indicates the presence of monosubstituted benzene derivatives, and, as the spectra show, with an increase in the amount of BPhFR used, their intensity increases, which confirms the chemical interaction of BPhFR molecules with bitumen molecules.

Absorption bands at 824-807 cm^{-1} (trisubstituted benzene derivatives), 750-720 cm^{-1} (*mono*- or trisubstituted benzene derivatives), 552, 520 cm^{-1} (*para*-disubstituted derivatives) also indicate the presence of substituted aromatic structures in the spectra benzene), 450, 424 cm^{-1} (*ortho* and *meta*-disubstituted benzene derivatives).

4. Conclusions

Bisphenol-formaldehyde resin was synthesized using the method of polycondensation of bisphenol A with formaldehyde, the yield of which is 71.05 wt.%.

Oxidized road bitumen was modified with bisphenol-formaldehyde resin. It was established that bisphenol-formaldehyde resin slightly improves the adhesion of bitumen modified by it with the crushed stone surface. On the other hand, it can be seen that the introduction of synthetic resin into the composition of bitumens significantly increases their heat resistance. At the same time, the introduction of the obtained resin into the composition of bitumen leads to a significant decrease in the plasticity of the obtained bitumen-polymer composition. Thus, with a bisphenol-formaldehyde resin content of 0.5 wt.% in bitumen, the softening temperature increases from 322.5 to 327.3 K, and with a content of 1.0 wt.% to 337.2 K.

The IR spectroscopic analysis showed the presence of an absorption band at 1017-1013 cm^{-1} in the spectra, which indicates the presence of monosubstituted benzene derivatives. Moreover, as the spectra show, with an increase in the amount of BPhFR used, their intensity increases, which confirms the chemical interaction of BPhFR molecules with bitumen molecules.

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ВПЛИВ БІСФЕНОЛО-ФОРМАЛЬДЕГІДНОЇ СМОЛИ НА ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ ДОРОЖНІХ БІТУМІВ

Анотація. *Методом поліконденсації бісфенолу А з формальдегідом синтезовано бісфеноло-формальдегідну смолу. Проведено модифікування дорожніх бітумів цією смолою. За різного вмісту синтезованої смоли встановлено можливість її використання як модифікатора дорожнього нафтового бітуму. Встановлено, що введення в склад бітуму синтезованої бісфеноло-формальдегідної смоли значно підвищує його теплостійкість. Проведено ІЧ- спектроскопічний аналіз синтезованої смоли та модифікованих нею бітумів. Описано зміну складу та властивостей бітуму, модифікованого бісфеноло-формальдегідною смолою.*

Ключові слова: *бісфенол А, модифікування бітуму, ІЧ-спектроскопія.*