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Properties of Modified Petroleum Resins Based on Various Fractions of Liquid Pyrolysis Products

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Abstract. This article addresses the use of aliphatic, cycloaliphatic and aromatic petroleum resins obtained by radical (thermal and initiated) and ionic polymerization of unsaturated compounds of various fractions of liquid pyrolysis products as starting materials for modification by nitration with nitric acid and subsequent reduction with iron in the presence of hydrochloric acid.

It has been established that nitration of petroleum resins leads to the appearance of nitro-, nitrate-, carbonyl and carboxyl groups in the polymer chain. Further modification of petroleum resins allows the introduction of amino groups into the composition of resins and significantly improves the performance characteristics of coatings based on the obtained resins: hardness, adhesion, and bending and impact strength.

Keywords: petroleum resin, fractions of liquid pyrolysis products, modification, nitration, reduction, coatings.

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Свойства модифицированных нефтеполимерных смол на основе различных фракций жидких продуктов пиролиза

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Аннотация. Рассмотрено использование алифатических, циклоалифатических и ароматических нефтеполимерных смол, полученных радикальной (термической и инициированной) и ионной полимеризацией непредельных соединений различных фракций жидких продуктов пиролиза, в качестве исходных для проведения модификации нитрованием азотной кислотой и последующим восстановлением железом в присутствии соляной кислоты. Установлено, что нитрование нефтеполимерных смол приводит к появлению нитро-, нитрат-, карбонильных и карбоксильных групп в составе полимерной цепи. Дальнейшая модификация НПС позволяет ввести аминогруппы в состав НПС и значительно улучшить эксплуатационные характеристики покрытий на основе полученных смол: твёрдость, адгезию, прочность при изгибе и при ударе.

Ключевые слова: нефтеполимерная смола, фракции жидких продуктов пиролиза, модификация, нитрование, восстановление, покрытия.

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Introduction

Highly aggressive operating environments are one of the main reasons for premature failure of industrial equipment [1].

To protect metals from corrosion in various aggressive aqueous environments, paint coatings and inorganic and organic corrosion inhibitors are used. The use of corrosion inhibitors is one of the most effective ways to combat metal corrosion. Corrosion inhibitors are designed to reduce the aggressiveness of gas and electrolytic medium, as well as to prevent active contact of the metal surface with the environment.

Currently, there are many different substances that can be used as inhibitors to block the corrosion process on the surface of metal objects [2].

Inorganic corrosion inhibitors include phosphates, bichromates, chromates, polyphosphates, nitrites, silicates, molybdates, etc. These are generally oxidizing inorganic substances that provide a protective film on the metal surface. This type of protection is considered very effective [3, 4].

Organic corrosion inhibitors slow down cathodic and anodic reactions. Most often used are aliphatic and aromatic compounds that have nitrogen, sulfur and oxygen atoms in their composition. Amines are used as inhibitors of iron corrosion in acids and aqueous media. Thiols (mercaptans), organic sulfides and disulfides show stronger inhibitory effect than amines. Organic acids and their salts are used as inhibitors of iron corrosion in acids, oils and electrolytes, as well as inhibitors of the hydrogenation process. The presence of amino- and hydroxyl- groups in organic acids improves their protective properties.

Electrophilic substituents that increase the positive charge on the adsorption center should facilitate the adsorption of molecules on a negatively charged surface and reduce corrosion, but they often increase it, which is explained by the peculiarities of hydrogen evolution on the metal.

Electron-donating molecules have a lower ionization potential, a larger negative charge on the nitrogen atom of the active center, and a more positive charge on the hydrogen atom in the protonated form and should be better inhibitors due to the formation of strong coordination or hydrogen bonds with the metal surface [5].

In terms of production volume, corrosion inhibitors are much inferior to paint coatings or protective coatings based on various hydrocarbons (oils, greases). As a rule, the coating does not affect the corrosion mechanism, but only acts as a barrier layer. If the integrity of the paint coating is broken, its protective properties are significantly reduced. However, the use of inhibiting additives in the composition of the paint coating provides sufficient protection of metal surfaces even if the coating itself is destroyed.

It has been established that the mechanism of action of the inhibitory additive depends on the structure of its molecules, the nature of the corrosive environment and the metal [6].

The products of nitration and oxidation process of natural and synthetic oils and resins are widely used as surfactants, detergents and corrosion inhibitors. In the paint and varnish industry, these products are also widely used as anticorrosion additives [7].

Nitrated ceresin and petrolatum have found application in the composition of protective coatings. The authors of [8] showed the possibility of using nitrated mineral oils with calcium stearate and calcium sulfonate as protective compositions.

Mixed corrosion inhibitors are widely spread. For example, work [9] shows the use of nitrated oligomers of ethylene and amides of petroleum acids.

Compositions based on nitro derivatives of C_{12}-C_{18} α -olefins and aminoamides obtained from technical petroleum acids and polyethylene polyamine [10] or nitration products of higher α-olefins and amides or aminoesters obtained from petroleum acids and di- and triethanolamines [11] can be used as an inhibiting mixture. Thus, most of the works propose the joint application of components containing nitro- and amino (amido)-functional groups.

Products based on modified petroleum polymer resins can also be used as anticorrosion additives. Petroleum polymer resins are resins obtained by polymerization of unsaturated compounds of liquid pyrolysis products. The monomer composition of various fractions of liquid pyrolysis products provides the production of aliphatic, aromatic, and cycloaliphatic hydrocarbon resins [12].

However, according to the elemental composition, IR, NMR and UV spectroscopy data, and chemical analysis of functional groups, PR is a statistical copolymer of styrene, as the main monomer in the fractions of liquid pyrolysis products, with other unsaturated compounds of the fractions (α-methylstyrene, vinyltoluene, indene, dicyclopentadiene and their derivatives). A solution of this non-polar polymer in white spirit is the most non-polar film-forming substance of all natural and synthetic boiled oils used.

Great possibilities for using PR can be realized after targeted chemical modification of resins in order to improve their characteristics and expand their areas of application. In connection with this arises the problem of increasing the polarity of PR. Analysis of literature data shows that chemical modification by oxidation, maleinization, addition of unsaturated acids, halogens, and introduction of sulfo groups allows to change the properties of PR and expand the areas of their application by creating weather-resistant materials with improved adhesive and cohesive properties.

Unfortunately, information on the functionalization of resins by nitration and the subsequent reduction of nitro groups to amino groups is limited in the literature.

Therefore, the purpose of this work is the synthesis and characterization of petroleum resins modified by nitration and subsequent reduction of nitrated petroleum resins.

Experimental

Objects of research

Petroleum polymer resins were obtained by polymerization of unsaturated compounds from various fractions of liquid hydrocarbon pyrolysis products: aromatic C_9, aliphatic C_5, cycloaliphatic with increased content of dicyclopentadiene DF and broad fraction C_{5-9} containing aliphatic, aromatic and cyclic monomers.

The polymerization of unsaturated compounds of the C_9 aromatic fraction was carried out under the influence of radical type initiators (PR_{C9_IN}), temperature (PR_{C9_T}), and $TiCl_4$-$Al(C_2H_5)_3$ catalysts (PR_{C9_ION}). Unsaturated compounds of fractions C_5, DF and C_{5-9} were polymerized by the ionic mechanism using the $TiCl_4$-$Al(C_2H_5)_3$ catalytic system, thereby obtaining the following petroleum polymer resins, respectively: PR_{C5}, PR_{DF}, PR_{C5-9}.

The nitration of all PR was carried out in a three-necked flask equipped with a mechanical stirrer, a dropping funnel and a reflux condenser according to the following procedure: a 64 % solution of nitric acid (30 % of the weight of resin) was slowly added to a 30–50 % solution of PR in petrol with vigorous stirring at a temperature of 25 ^{o}C. When the acid addition was complete, the temperature in the flask was raised to 60 °C and was maintained for 180 minutes. The reaction mass was then washed with water to a neutral medium, the solvent was distilled off and the resin dried to constant weight.

The reduction of the nitrated resin was carried out in a three-necked flask equipped with a mechanical stirrer, a dropping funnel and a reflux condenser, according to the following procedure: iron filings (25 % of the weight of the resin) were added to a 20–30 % solution of nitrated PR in a mixed solvent (solvent and butanol in a ratio of 90:10). Then a 37 % solution of hydrochloric acid (65–70 % of the weight of the iron filings) was added slowly, drop by drop, with stirring and at a temperature of 25 ^{o}C. When the acid addition was complete, the temperature was raised to 60 °C and was maintained for 180 minutes. The reaction mass was then washed with water or soda lye until neutral medium, the solvent was distilled off and the resin was dried to a constant weight.

Methods of research

The physicochemical properties of the original and modified resins were determined using standard methods [13].

IR spectra were recorded on a Nicolet 5700 IR-Fourier spectrometer, samples were resin films, spectral range 400–4000 cm^{-1}.

^{1}H NMR spectra of RP were recorded on an AVANSE AV 300 spectrometer from Bruker with an operating frequency of 300 MHz; HMDS was used as an internal standard.

The softening temperature of PR was determined according to GOST 32054–2013 "Petroleum bitumen. Determination of softening temperature by ring and ball."

Adhesion was determined by the pull-off test – GOST 32299–2013 (ISO 4624: 2002): Paint materials. Pull-off test for adhesion.

The flexural strength of coatings was determined according to GOST R 52740–2007 (ISO 1519–2002) "Paints and varnishes – Bend test (cylindrical mandrel)".

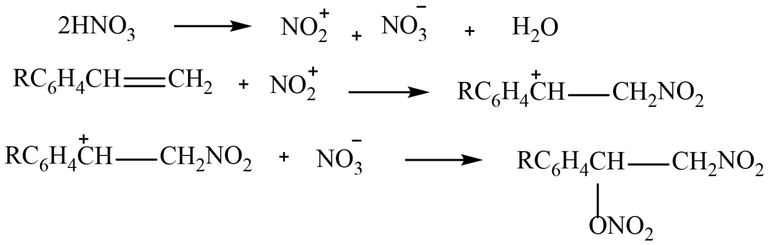
The hardness of the coating was determined by the Knoop method, ISO 6441 "Paints and varnishes – Indentation test (spherical and pyramidal)".

The impact resistance of the coating was determined according to GOST 4765–73 "Pain and lacquer materials. Method of determination impact resistance".

Results and Discussion

Various compounds are used as nitrating agents: nitric acid, a mixture of nitric and sulfuric acids, nitrogen oxides, nitrate salts, etc. In industrial processes, the cheapest and most accessible substances, such as nitric acid, are most often used. The use of nitric acid has the following advantages over other nitrating agents: high efficiency, ease of dosing and availability. However, nitration with nitric acid can be accompanied by oxidation processes and the formation of oxygen-containing insoluble products. Increasing the nitration temperature from 60 to 90 °C leads both to an increase in the content of nitrated products and to a sharp increase in the oxidation rate [14].

The mechanism of the nitration reaction of unsaturated aromatic compounds with the formation of nitro and nitrate groups is given in [15].



$$2HNO_3 \longrightarrow NO_2^+ + NO_3^- + H_2O$$

$$RC_6H_4CH{=}CH_2 + NO_2^+ \longrightarrow RC_6H_4\overset{+}{C}H{-}CH_2NO_2$$

$$RC_6H_4\overset{+}{C}H{-}CH_2NO_2 + NO_3^- \longrightarrow RC_6H_4\underset{|\,ONO_2}{CH}{-}CH_2NO_2$$

The resins obtained after nitration of PR (NPR) and subsequent reduction (APR) were studied in comparison with the initial resins (PR) by spectral and chemical methods.

The reduction of nitro groups in NPR was carried out by iron in the presence of hydrochloric acid by the following reaction:

$$R\text{-}NO_2 + 3Fe + 6HCl \longrightarrow R\text{-}NH_2 + 3FeCl_2 + 2H_2O$$

The results of the elemental composition analysis prove the carried out the nitration process of resins. The elemental composition of initial and modified aromatic resins is given in Table 1. It has been established that simultaneously with the appearance of nitrogen in the resin composition (3.8–4.1 %), the oxygen content also increases (by 10.9–14.8 %).

Figure 1 shows the IR spectra of the initial and modified aromatic resins (PR_{C9_ION}, NPR_{C9_ION}) and resins based on a wide fraction of hydrocarbons ($PR_{C5–9}$, $NPR_{C5–9}$).

Table 1. Elemental composition of petroleum resins

Element	Elemental composition of aromatic petroleum resins					
	PR_{C9_IN}	NPR_{C9_IN}	PR_{C9_T}	NPR_{C9_T}	PR_{C9_ION}	NPR_{C9_ION}
C	83,1	68,3	83,5	68,1	81,9	68,8
H	6,9	5,1	7,7	4,5	7,0	5,2
N	0,0	4,1	0,0	3,8	0,0	4,0
O	10,0	22,5	8,8	23,6	11,1	22,0

The presence of intense absorption bands in the region of 1551 cm^{-1}, characteristic of asymmetric valence vibrations of the $C–NO_2$ bonds, and medium intensity absorption bands in the region of 1350 cm^{-1}, corresponding to symmetric valence vibrations of the $C–NO_2$ nitro group, confirms the obtaining of nitrated resins. The spectra also contain absorption bands in the region of 1632–1633 cm^{-1} and 1278–1279 cm^{-1}, characteristic of asymmetric and symmetric valence vibrations of the alkyl nitrate group $C–O–NO_2$, respectively. As the nitration process deepens, an increase in the intensity of the absorption band of the C–N group valence vibrations in the region of 851–852 cm^{-1} is observed in the IR spectra.

In addition, in the IR spectra of nitrated PR, an increase in the absorption bands was noticed in the regions of 3428–3433 cm^{-1} and 1702–1707 cm^{-}1, characteristic of the valence vibrations of the hydroxyl group OH and the carbonyl group C=O, respectively. The weak intensity absorption band in the region at 1074 cm^{-1} is attributed to the valence vibrations of the C–O bond. The presence of carbonyl and hydroxyl groups in the composition of nitrated resins is explained by the possibility of oxidation of the resins with nitric acid (Fig. 1).

Similar characteristic absorption bands are observed in the IR spectra of other nitrated resins obtained on the basis of the aromatic fractions C_9 (NPR_{C9_T}, NPR_{C9_IN}), the aliphatic fraction C_5 (NPR_{C5}), and the dicyclopentadiene fraction DF (NPR_{DF}).

Analysis of the normalized integral intensity values of 6 types (A, B, C, D, E, F) protons in the ^1H NMR spectra of the initial and modified resins (Table 2) showed that during nitration the number of olefinic (type B) and aromatic (type A) protons in the hydrocarbon chains of the resin (type D, E protons) increased. This fact indicates the possibility of introducing a nitro group into the aromatic rings of monomer units (for example, styrene, vinyltoluene) or the addition of a nitro group to the double bonds of diene monomers (for example, cyclopentadiene). A decrease in the integral intensity of the olefin and methylene protons at the α-position to the double bond (type B, C) also does not exclude the possibility of oxidation at the site of the double bonds.

Spectral methods for studying the reduction products of nitrated petroleum resins, unfortunately, do not give unambiguous results. This is due to the fact that the characteristic absorption bands of the NH_2 group bending vibrations (medium or weak intensity) are in the region of the NO_2 group asymmetric valence vibrations (strong intensity), and the absorption band of the NH_2 group valence vibrations lies in the region of the absorption band of the OH group valence vibrations.

The chemical analysis of the modified resins is in agreement with the results of the spectral studies (Table 3).

The aromatic petroleum polymer resins obtained by the ionic mechanism have the maximum unsaturation (bromine number) and a higher acid number after nitration (Table 3). The ionic mechanism

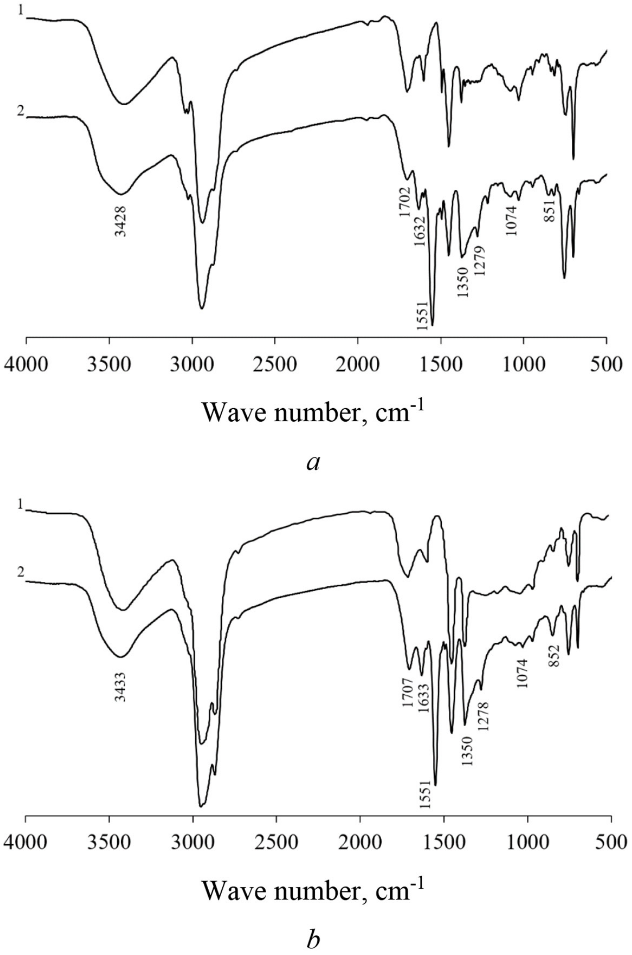


Fig. 1. IR spectra of PR_{C9_ION} (1a), NPR_{C9_ION} (2a), $PR_{C5–9}$ (1b), $NPR_{C5–9}$ (2b)

Table 2. Values of normalized integral proton intensity (%) for initial and nitrated petroleum resins

Proton type (δ, ppm)	PR_{C9_ION}	NPR_{C9_ION}	PR_{DF}	NPR_{DF}
A (6,5–7,8) – aromatic ring protons	25,9	23	1,3	1,1
B (4,5–6,5) – olefinic protons	4,5	3,3	10,8	7,5
C (2,5–4,5) – methyl and methylene protons at the α-position to the aromatic ring or at the α-position to the double bond	23,4	22,4	9,8	9,5
D (2,0–2,5) – methine protons of paraffins and naphthenes	12,7	12,4	19,4	19
E (1,1–2,0) – methylene protons of paraffins and naphthenes	22,2	23,4	33,2	35
F (0,1–1,1) – methyl protons	11,3	15,5	25,5	27,9

allows the most complete incorporation into the polymer chain of all the monomers present in the initial fraction of the liquid pyrolysis products; for example, dicyclopentadiene, which is incapable of copolymerisation by the radical mechanism, is copolymerised by the ionic mechanism. Of the remaining resins, the resin obtained from the dicyclopentadiene fraction is noteworthy: the resin is highly unsaturated,

Table 3. Physicochemical properties of initial and nitrated petroleum resins

Property	Properties of aromatic petroleum resins									Properties of aliphatic, cycloaliphatic resins and resins based on a wide fraction of hydrocarbons								
	PR_{C9_IN}	NPR_{C9_IN}	APR_{C9_IN}	PR_{C9_T}	NPR_{C9_T}	APR_{C9_T}	PR_{C9_ION}	NPR_{C9_ION}	APR_{C9_ION}	RR_{C5}	NRR_{C5}	ARR_{C5}	RR_{C5-9}	NRR_{C5-9}	ARR_{C5-9}	PR_{DF}	NPR_{DF}	APR_{DF}
Bromine number, g Br_2/100 g	42	12	13	44	13	14	49	15	16	58	11	12	60	15	15	84	16	17
Acid number, mg KOH/1g	5	12	14	6	14	15	8	17	17	4	8	7	7	9	9	3	6	5
Softening point, °C	104	135	68	110	134	76	76	120	65	75	125	72	70	127	69	79	131	73
Reference		16			16						16			16				

but both the initial and the nitrated resins are slightly subject to oxidation. All resins after nitration have a high softening point (above 100 °C), which further confirms the introduction of polar nitro groups.

The products of the reduction reaction of nitro groups have close bromine and acid numbers, which indicates the absence of reactions at double bonds. Only the softening temperature changes significantly after the reduction of nitrated resins. The decrease in softening temperature can probably be explained by the possibility of destruction of physical bonds between macromolecules or supramolecular structures of resins under the influence of hydrogen released during reduction.

In order to assess the possibility of using modified resins in film-forming compositions, coatings based on modified PR were obtained and their physical and mechanical characteristics were compared with the properties of coatings based on the initial resins (Table 4). The thickness of the coatings was 30 microns.

It follows from the data of Table 4 that coatings based on nitrated PR have improved hardness, adhesive tear-off strength and impact resistance. The hardness of nitrated aromatic petroleum resins (0.6–0.8 kg) is slightly higher than that of aliphatic PR (0.4 kg). Simultaneously with the increase of

Table 4. Physical and mechanical characteristics of coatings based on initial and modified resins

Property	Properties of coatings based on aromatic petroleum resins									Properties of coatings based on aliphatic, cycloaliphatic and broad fraction hydrocarbon petroleum resins								
	PR_{C9_IN}	NPR_{C9_IN}	APR_{C9_IN}	PR_{C9_T}	NPR_{C9_T}	APR_{C9_T}	PR_{C9_ION}	NPR_{C9_ION}	APR_{C9_ION}	RR_{C5}	NRR_{C5}	ARR_{C5}	RR_{C5-9}	NRR_{C5-9}	ARR_{C5-9}	PR_{DF}	NPR_{DF}	APR_{DF}
Bending strength, mm	20	20	12	20	20	16	6	12	10	10	12	12	12	16	16	10	12	10
Impact resistance, cm	2	3	8	2	3	7	3	4	7	6	7	9	5	6	9	6	7	10
Hardness, kg	0,4	0,6	0,8	0,4	0,6	1,0	0,7	0,8	1,0	0,7	0,4	0,6	0,2	0,4	0,8	0,2	0,4	0,6
Adhesion, MPa	0,7	0,8	2,3	0,7	0,8	2,3	0,9	1,1	2,2	1,4	1,2	1,6	1,0	1,2	1,8	1,3	1,4	1,6

strength characteristics (impact resistance, adhesion strength) and hardness of PR nitration products, a deterioration of elastic properties (bending strength) is observed: nitrated resins are more brittle compared to the initial ones.

Further modification: the reduction of nitrated PR to aminated resins leads to an improvement in the key performance characteristics of the coating: hardness, elasticity, adhesion and impact resistance. It has been found that the adhesion of coatings based on aminated PR increases from 0.8–1.1 MPa to 2.2–2.3 MPa for aromatic resins (APR_{C9_IN}, APR_{C9_T}, APR_{C_ION}) and from 1.2–1.4 MPa to 1.6–1.8 MPa for aliphatic, cycloaliphatic and wide fraction resins (APR_{C5}, APR_{C5-9}, APR_{DF}). The impact resistance of coatings based on aminated APR increases from 3–4 cm to 7–8 cm for aromatic resins and from 6–7 cm to 8–9 cm for other resins. The elasticity (bending strength) of coatings based on aminated resins increases significantly from 20 mm to 10–16 mm.

Conclusion

Nitration of petroleum resins leads to a significant change in their functional composition: the appearance of nitro-, nitrate-, carbonyl and carboxyl groups, which makes it possible to use them for further modification and expansion of the range of resins.

Nitrated petroleum resins has improved hardness, adhesive peel strength and impact resistance compared to the initial ones.

The introduction of amino groups into petroleum resins leads to an improvement in the key performance characteristics of the coating: hardness, adhesion, bending strength and impact resistance.

Modified petroleum resins can be recommended for use as components of paints and varnishes for iron and reinforced concrete structures.

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