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PETROLEUM RESINS FOR BITUMENS MODIFICATION

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Abstract. The process of tar and industrial petroleum resin “Piroplast-2” joint oxidation has been investigated. The experimental results of the main regularities researches of the obtaining process of petroleum resins with carboxyl groups based on the pyrocondensate C₉ fraction of hydrocarbon feedstock pyrolysis have been shown. It has been established that synthesized petroleum resins may be used for the modification of petroleum bitumens.

Keywords: tar, blown bitumen, petroleum resin, carboxyl group, modification, bituminous -polymeric composition.

1. Introduction

Petroleum resins obtained via oxidation, deep vacuum distillation or other methods sometimes do not meet demands for the marketable products [1, 2]. Bitumen produced today in Ukraine has low adhesive activity due to the peculiarities of its obtaining, as well as to the properties of initial raw material. This fact results in the bitumen film intensive lift-off from the surface of solid materials under the action of temperature difference, water, mechanical load etc. Moreover, bitumen can not be produced using any kind of feedstock. Therefore, different additives able to improve bitumen properties are used for the production of blown and residual bitumens [3-6]. Energy saving (temperature decrease, reduction of oxidation time) is also the main direction of process improvement.

Petroleum resins (PRs) are among additives which may be used for the modification of petroleum bitumens. Such oligomeric products are obtained via oligomerization of unsaturated compounds presented in the liquid products of oil stock pyrolysis. The C₅ and C₉ fractions of pyrocondensate or pyrolysis heavy resin are used for the PRs production.

The Department of Chemistry and Technology of Petroleum at Lviv Polytechnic National University has carried out a raw of investigations concerning the modification of blown bitumens produced by Ukrainian refineries by different petroleum resins.

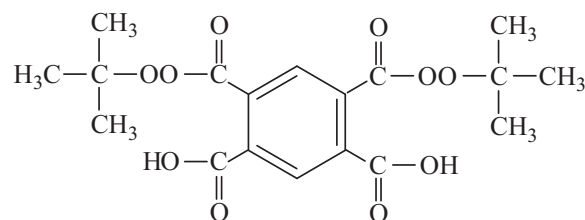
The bitumens were modified using two methods:

- PRs introduction into feedstock for the oxidation process;
- PRs introduction into finished bitumen.

2. Experimental

The joint oxidation of oil tar and petroleum resins was carried out at laboratory-scale plant consisting of reactor block, air supply and cooling systems, as well as trapping unit for oxidation volatile products. A tar, extracted from Russian export oil mixture was taken at JSC “Ukratnafta”, Kremenchuk with the following characteristics: viscosity 23.2 s, flash point 525 K, softening temperature 303 K, $d_4^{20} = 0.991$. “Piroplast-2” industrial petroleum resin with softening temperature 358 K was obtained via thermal oligomerization of the pyrocondensate C₉ fraction of gasoline pyrolysis. Ductility, penetration and softening temperature were determined for blown petroleum bitumens by standard procedures [7].

The C₉ fraction obtained at the pyrolysis of hydrocarbon feedstock at JSC “Lukor, Kalush, was used as the raw material for the production of petroleum resins with carboxyl groups (PRCs). The characteristic of initial material is presented in [8]. Peroxide with carboxyl end groups was the initiator of cooligomerization process. Its formula is given below.



Di-*tert*-butyldiperpyromellitate (PPM)

PRCs were synthesized in glass ampoules by volume of $5 \cdot 10^{-5} \text{ m}^3$. The ampoules were blown with inert gas and the sample of the C₉ fraction was loaded. Then the initiator was added, the ampoules were soldered and loaded into a thermostat. After the experiment the ampoules were cooled and the contents were precipitated with petroleum ether. The solvent was separated, the obtained residue was dried at 323 K under vacuum and the product was analyzed.

We investigated 3 commercial oil bitumens produced by Ukrainian refineries, namely:

Table 1

Characteristics of commercial blown bitumens

Index	BN-70/30 bitumen	BNK-45/190 bitumen	BN-60/90 bitumen
Depth of needle penetration at 298 K, 0.1 mm	21-40	160	90
Softening temperature determined by ring and ball method, K	343	323	333
Tension (ductility) at 298 K, no less than	3	–	5
Solubility in toluene or chloroform, %, no less than	99	99,6	99
Mass change after heating, %, at most	1.0	0.23	0.85
Flash point, K, no less than	503	525	518
Water mass part, at most	–	absence	–
Depth of needle penetration at 298 K in the residue after heating, % calculated from initial value, no less than	–	80	–
Paraffin mass part, %, at most	–	4.7	–
Penetration index	–	2.5	–

BN-70/30 bitumen, produced at JSC “Galychna”, Drogobych;

BNK-45/190, produced at Shebelinka Gas-Works;

BN-60/90, produced at JSC “Ukratnafta”, Kremenchuk.

The main characteristics of the mentioned bitumens are presented in Table 1. Five different types of industrial and synthesized petroleum resins were used as modifiers:

Pyroplast-2 PR – solid, light-yellow, industrial product obtained via thermal oligomerization of the C₉ fraction. The softening temperature is 358 K.

Dark PR was obtained via thermal oligomerization of pyrolysis heavy resin. It is a dark-brown substance with the softening temperature 378 K.

Light PR was obtained via thermal oligomerization of the C₅ and C₉ fraction of pyrolysis pyrocondensate. It is a light-yellow substance with the softening temperature 355 K.

PR with epoxy groups was obtained at the Department of Chemistry and Technology of Petroleum at Lviv Polytechnic National University. It is light-brown substance with softening temperature 355 K. Its characteristics: molecular mass (M_n) is 850, epoxy number (e.n.) is 8.2 %, bromine number (b.n.) is 9.3 g Br/100 g.

PR with carboxyl groups was obtained at the same Department. It is a light-brown substance with the softening temperature 358 K. Its characteristics: M_n is 1320, e.n. is 8.2 %, b.n. is 9.3 g Br/100 g.

Bitumen-polymeric compositions were prepared by mixing the components on the laboratory-scale plant consisting of electrical heater, metal reservoir equipped with stirrer and thermometer. Samples were weighted, loaded into the reservoir and heated to 383 K with constant stirring. Then the calculated amount of PR was added. The components were stirred until the mixture became homogeneous at 393 K (the temperature has to be higher than PR softening temperature).

3. Results and Discussion

3.1. Joint oxidation of heavy residues and PR

In order to study joint oxidation the main regularities of heavy residues and PR it was necessary to determine the PR optimum content in the feedstock for obtaining blown oil bitumens and to find out how the main processing factors influence the oxidation rate and thus characteristics of obtained bitumens.

The effect of Pyroplast-2 content in the feedstock (tar) on the properties of blown bitumens was studied with the aim of determination of PR optimum content. So, PR was introduced into the feedstock in amount of 1–10 mass %. The oxidation was carried out at 523 K for 3 h. The volumetric flow rate was 2.5 min⁻¹. The obtained results are presented in Table 2.

One can see from Table 2 that introduction of Pyroplast-2 resin into the feedstock essentially affects the properties of obtained oil bitumens. The increase of PR amount increases bitumen softening temperature and ductility but decreases penetration which determines bitumen hardness. At the same time the increase of PR content in the feedstock increases acid number of modified bitumens. This fact has a positive effect on the adhesive properties which are characterized by the index “adhesion to glass”. Thus, the introduction of PR into the feedstock essentially affects the characteristics of obtained bitumens and increases their adhesion to the solid surfaces. The mixture with the tar:PR ratio equal to 95:5 was used for the further investigations.

In order to establish the process optimum temperature we examined the effect of oxidation temperature on the properties of obtained oil bitumens. The investigations were carried out in two parallel directions: joint oxidation of tar and PR with the ratio 95:5 and tar oxidation without PR. The oxidation process was carried out for 3 hrs within the temperature range of 483–543 K. The volumetric flow rate was 2.5 min⁻¹. The obtained results are presented in Table 3.

Table 2

Effect of PR amount in the feedstock on the properties of blown oil bitumens

Index	PR amount in the feedstock, mass %				
	0	1	3	5	10
Softening temperature determined by ring and ball method, K	308	308	310	311	317
Ductility at 298 K, cm	40	41	42	64	93
Penetration at 298 K, 0.1 mm	260	214	210	168	68
Adhesion to the glass, %	14.5	19.6	31.6	36.9	75.3
Acid number, mg KOH/g	0.28	0.31	0.39	0.48	1.06

Table 3

The effect of oxidation temperature on the properties of oil bitumens

Index	Temperature, K				
	483	503	523	533	543
Tar + 5 mass % PR as feedstock					
Softening temperature determined by ring and ball method, K	309	310	311	312	313
Ductility at 298 K, cm	44	55	64	45	41
Penetration at 298 K, 0.1 mm	260	210	168	147	123
Tar as feedstock					
Softening temperature determined by ring and ball method, K	308	308	308	309	310
Ductility at 298 K, cm	35	38	40	46	44
Penetration at 298 K, 0.1 mm	280	280	260	255	250

Table 4

The effect of oxidation time on the properties of blown oil bitumens

Index	Oxidation time, h		
	3	6	9
Tar + 5 mass % PR as feedstock			
Softening temperature determined by ring and ball method, K	311	317	344
Ductility at 298 K, cm	64	88	9
Penetration at 298 K, 0.1 mm	168	93	14
Tar as feedstock			
Softening temperature determined by ring and ball method, K	306	314	326
Ductility at 298 K, cm	40	73	69
Penetration at 298 K, 0.1 mm	257	134	35

At joint oxidation of tar and 5 % of petroleum resin main the characteristics of obtained bitumen are changed with the temperature increase. We can see from the experiments that the softening temperature increases smoothly. At the same time ductility has an extreme character. This value increases till 523 K and then decreases. Bitumen penetration decreases with the temperature increase. At tar oxidation without modifier (see Table 3) the bitumen characteristics are changed in a lesser degree. Evidently, that tar oxidation with PR and without modifier proceeds in a different way. Tar oxidation without PR proceeds with lower rate. So PR affects the

oxidation mechanism and reduces the process duration. Further investigations were carried out at 523 K as an optimum temperature.

The effect of the process time was examined during 3–9 hrs at 523 K and air flow rate 2.5 min⁻¹. The obtained results are presented in Table 4.

As we can see from Table 4, oxidation time essentially affects the properties of obtained bitumens. With the increase of oxidation time the bitumen softening temperature increases and the penetration decreases. The dependence of ductility upon oxidation time has an extreme character, the same as in the previous case.

The comparison of bitumens obtained by different ways shows that PR introduction into the feedstock allows to achieve definite operational characteristics in a shorter period of time, that, in its turn, intensifies the process and saves energy supply. So, PRs may be used as modifiers of oil bitumens.

3.2. Obtaining of petroleum resins with carboxyl groups

Since there is a direct dependence between acid number of modified bitumens and their ability to hold on the surface (index “adhesion to glass”) it should be necessary to synthesize petroleum resins with carboxyl groups (PRC) and to investigate them as modifiers of bitumens. To our mind, the introduction of oligomers with carboxyl groups will exactly improve the adhesion properties of oil bitumens.

PRC obtaining was studied via initiated cooligomerization of unsaturated hydrocarbons presented in the C_9 fraction in the presence of PPM functional peroxide. The experiments were carried out in the temperature range of 393–413 K and PPM amount 0.25–5.0 mass %. The process time was 5–50 h.

Functional oligomers with different characteristics (Tables 5 and 6) were obtained under different conditions (temperature, initiator amount, process time). The main characteristics are: molecular mass, bromine number

which corresponds to the resin unsaturation and acid number which characterizes the carboxyl group content in the cooligomer molecule.

We can see from Table 5 that the increase of the initiator amount increases the PRC yield and acid number but decreases its molecular mass. Obviously, it is the result of molecules formation with less size but greater amount of carboxyl groups. The increase of functionality (f) reveals the same fact. The increase of the initiator amount decreases PRC bromine number.

The increase of oligomerization temperature (see Table 5) increases PRC yield, decreases molecular mass and increases acid number, it results in the increase of PRC functionality. The unsaturation of PRC is reduced because we observe the decrease of bromine number.

The results from Table 6 show that the increase of synthesis time increases PRC yield, molecular mass and bromine number but decreases acid number and PRC functionality.

Obtained results allow to offer the optimum conditions for PRC obtaining: temperature 393 K, time – 50 h, PPM content – 2.5 mass %, calculated on the feedstock. Synthesized PRC is a solid, light-brown substance, which is soluble in aromatic solvents, dioxane, carbon tetrachloride etc. Its characteristics are: yield is 20.2 mass %, $M_n = 1320$, br.n. = 23.8 gBr₂/100g, a.n. = 51.9 mg KOH/100g, functionality is 1.22.

Table 5

The effect of initiator amount on the PRC yield and main characteristics

Initiator content, mass %	T, K	Yield, mass %	M_n	a.n., mg KOH/g	Br.n. g Br ₂ /100g	f
0.5	393	13.3	1390	34.8	31.5	0.86
1.0		13.9	1370	39.5	28.1	0.97
2.5		14.7	1320	51.9	23.8	1.22
5.0		16.6	1260	68.2	17.3	1.53
7.5		17.4	1180	84.5	13.2	1.78
0.5	403	14.7	1270	44.5	27.1	1.01
1.0		15.2	1250	47.9	24.0	1.07
2.5		16.5	1180	60.2	18.8	1.27
5.0		18.1	1080	78.4	14.2	1.51
7.5		18.8	1030	97.1	11.3	1.79
0.5	413	16.1	1130	56.0	21.7	1.13
1.0		16.7	1100	62.3	19.0	1.22
2.5		17.7	1060	73.4	16.4	1.39
5.0		19.2	990	94.8	11.9	1.67
7.5		20.2	920	111.9	9.2	1.84

Note. Process time is 50 h.

Table 6

The effect of cooligomerization time on the PRC yield and main characteristics

Time, h	T, K	Yield, mass %	M _n	a.n., mg KOH/g	Br.n. g Br ₂ /100g	ƒ
5	393	9.4	1030	127.6	5.7	2.35
10		11.5	1100	118.6	6.6	2.33
20		14.2	1150	104.9	8.1	2.15
30		15.7	1210	88.2	12.1	1.19
40		16.3	1230	75.5	14.5	1.66
50		16.6	1260	68.2	17.3	1.53
5	403	10.9	870	139.2	3.8	2.17
10		13.1	930	130.9	4.9	2.17
20		15.3	990	118.0	7.0	2.09
30		16.5	1060	104.0	8.6	1.97
40		17.4	1070	90.2	12.2	1.72
50		18.1	1080	78.4	11.9	1.51
5	413	12.3	770	153.8	2.1	2.12
10		14.6	810	145.7	3.9	2.11
20		17.0	890	132.9	5.8	2.10
30		18.3	920	114.0	7.1	1.92
40		18.9	970	105.3	8.3	1.82
50		19.2	990	94.8	6.1	1.67

Note. C_{in} = 5.0 mass % calculated for the feedstock.

3.3. Modification of oil bitumens by petroleum resins

Synthesized petroleum resins with carboxyl groups were used for the modification of oil bitumens. The modification of blown oil bitumens was carried out via PRC introduction into BN-70/30 industrial bitumen produced at JSC “NPK-Galychyna”.

The PRC amount ranged from 1 to 10 mass %. Four other types of PR were introduced in the same bitumen in the same amount for the comparison. The results are presented in Table 7. As we can see from the experiments the introduction of petroleum resins increases bitumen softening temperature. Moreover such increase is correlated with PR softening temperature; it means bitumen-polymeric composition containing dark petroleum resin has the maximum softening temperature.

The investigated bitumen has the minimum ductility – 3 cm. The introduction of PR does not change the ductility. The increase of PR amount increases the bitumen hardness (index “penetration”). Bitumen with 10 mass % of dark PR has the minimum penetration (1 mm).

The ability to hold on the surfaces (adhesion ability) is one of the main characteristics of building bitumens. This ability is characterized by the index “adhesion to glass”. One can see from Table 7 that in all cases introduction of PR increases the adhesion. Moreover, the greater PR amounts, the greater index “adhesion to glass”. The presence of functional groups in the investigated PRs has a stronger influence on the bitumen adhesion. The best results are obtained using PR with carboxyl groups.

For instance, the introduction of 10 mass % of such oligomer increases the adhesion from 81.9 to 99.6 %, that is also correlated with acid number of bitumen-polymeric composition. This fact confirms the hypothesis about the improvement of adhesion properties by carboxyl groups.

Other bitumen-polymeric compositions on the bases of BNK-45/190 and BN-60/90 commercial bitumens (produced at Shebelinka Gas-Works and JSC “Ukratnafta, correspondingly) were obtained and analyzed for the comparison. The results are presented in Tables 8 and 9.

BNK-45/190 bitumen is very similar by its characteristics to BN-70/30 bitumen produced at JSC “NPK-Galychyna”. That is why obtained values characterizing the effect of different types of PRs on the bitumen properties are also similar to the mentioned values. One can see from Table 8 that the increase of PR amount in the bitumen-polymeric composition increases its softening temperature and decreases penetration. The composition ductility actually is not changed. Like in the previous case, the increase of PR amount improves the adhesion characteristics of the bitumen-polymeric compositions, which result in the increase of the “adhesion to glass” index. The best results have been obtained using PR with carboxyl groups.

BN-60/90 bitumen produced at JSC “Ukratnafta” (see Table 9) has the same regularities except ductility. In contrast to the previous cases the introduction of PR increases the ductility but at the PR amounts more than 5-7 mass % the ductility is reduced.

Table 7

The effect of PR amount on the characteristics of BN-70/30 produced at JSC “NPK-Galychyna”

Index	PR amount in the composition, mass %					
	0	1	3	5	7	10
Pyroplast-2 PR						
Softening temperature determined by ring and ball method, K	345	347	347	348	348	348
Ductility at 298 K, cm	3	4	4	4	2	1
Penetration at 298 K, 0.1 mm	15	13	9	7	6	4
Adhesion to glass, %	81.9	83.2	85.5	87.1	89.7	89.9
Dark PR						
Softening temperature determined by ring and ball method, K	345	347	347	348	348	349
Ductility at 298 K, cm	3	3	4	4	3	2
Penetration at 298 K, 0.1 mm	15	13	10	7	5	1
Adhesion to glass, %	81.9	82.8	84.6	86.5	88.1	88.7
Light PR						
Softening temperature determined by ring and ball method, K	345	347	347	348	348	348
Ductility at 298 K, cm	3	4	4	4	3	2
Penetration at 298 K, 0.1 mm	15	12	8	6	3	2
Adhesion to glass, %	81.9	83.1	85.3	86.8	89.4	89.7
PR with epoxy groups						
Softening temperature determined by ring and ball method, K	345	347	347	348	348	348
Ductility at 298 K, cm	3	3	3	3	2	2
Penetration at 298 K, 0.1 mm	15	14	12	9	6	4
Adhesion to glass, %	81.9	84.3	87.6	89.7	91.1	92.5
PR with carboxyl groups						
Softening temperature determined by ring and ball method, K	345	346	346	347	348	348
Ductility at 298 K, cm	3	4	4	3	3	1
Penetration at 298 K, 0.1 mm	15	13	11	8	5	3
Adhesion to glass, %	81.9	89.20	97.7	99.3	99.5	99.6
Acid number, mg KOH/g	0.8	3.8	5.4	6.4	7.1	9.7

Table 8

The effect of PR amount on the characteristics of BNK-45/190 bitumen produced at Shebelinka Gas-Works”

Index	PR amount in the composition, mass %					
	0	1	3	5	7	10
Pyroplast-2 PR						
Softening temperature determined by ring and ball method, K	350	356	359	362	365	–
Ductility at 298 K, cm	3	2	4	2	2	–
Penetration at 298 K, 0.1 mm	40	35	31	25	15	–
Adhesion to glass, %	95.8	96.5	96.7	97.5	98.1	–
Dark PR						
Softening temperature determined by ring and ball method, K	350	357	361	363	368	–
Ductility at 298 K, cm	3	2	2	1	2	–
Penetration at 298 K, 0.1 mm	40	34	29	22	14	–
Adhesion to glass, %	95.8	96.1	96.7	97.1	97.4	–
Light PR						
Softening temperature determined by ring and ball method, K	350	352	357	360	363	–
Ductility at 298 K, cm	3	3	4	3	3	–
Penetration at 298 K, 0.1 mm	40	35	30	24	18	–
Adhesion to glass, %	95.8	96.3	96.9	97.3	97.7	–
PR with epoxy groups						
Softening temperature determined by ring and ball method, K	350	359	360	362	363	–
Ductility at 298 K, cm	3	3	4	3	2	–
Penetration at 298 K, 0.1 mm	40	36	32	30	27	–
Adhesion to glass, %	95.8	96.0	96.8	97.2	98.0	–
PR with carboxyl groups						
Softening temperature determined by ring and ball method, K	350	357	359	360	362	363
Ductility at 298 K, cm	3	2	2	2	2	2
Penetration at 298 K, 0.1 mm	40	37	34	30	27	24
Adhesion to glass, %	95.8	96.0	96.9	97.5	98.4	99.8
Acid number, mg KOH/g	5.2	6.9	8.2	10.0	12.1	14.5

Table 9

The effect of PR amount on the characteristics of BN-60/90 bitumen produced at JSC “Ukratnafta”

Index	PR amount in the composition, mass %					
	0	1	3	5	7	10
Pyroplast-2 PR						
Softening temperature determined by ring and ball method, K	325	326	327	328	329	331
Ductility at 298 K, cm	77	78	80	83	84	83
Penetration at 298 K, 0.1 mm	81	80	77	71	66	61
Adhesion to glass, %	71.5	73.6	78.9	81.6	85.4	91.3
Dark PR						
Softening temperature determined by ring and ball method, K	325	326	328	329	331	333
Ductility at 298 K, cm	77	77	76	75	73	71
Penetration at 298 K, 0.1 mm	81	78	73	68	62	54
Adhesion to glass, %	71.5	72.4	76.5	79.8	82.6	86.1
Light PR						
Softening temperature determined by ring and ball method, K	325	325	326	328	329	330
Ductility at 298 K, cm	77	78	79	80	81	80
Penetration at 298 K, 0.1 mm	81	79	75	72	66	60
Adhesion to glass, %	71.5	73.5	78.7	81.4	85.3	91.1
PR with epoxy groups						
Softening temperature determined by ring and ball method, K	325	325	326	327	328	329
Ductility at 298 K, cm	77	77	78	80	81	81
Penetration at 298 K, 0.1 mm	81	80	77	74	71	65
Adhesion to glass, %	71.5	74.0	80.1	82.8	88.3	93.4
PR with carboxyl groups						
Softening temperature determined by ring and ball method, K	325	325	326	327	328	329
Ductility at 298 K, cm	77	78	80	82	82	81
Penetration at 298 K, 0.1 mm	81	79	76	73	70	64
Adhesion to glass, %	71.5	75.3	80.8	84.1	89.7	95.5
Acid number, mg KOH/g	2.6	3.9	5.1	6.5	8.2	10.4

4. Conclusions

On the basis of above-mentioned results we may assert that petroleum resins may be used as active additives of bitumen-polymeric compositions. Their introduction to the bitumen structure has a positive effect on the operational characteristics, especially on the adhesion properties. The optimum amount is 5 mass % but this value may be changed depending upon the expected result.

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ВИКОРИСТАННЯ НАФТОПОЛІМЕРНИХ СМОЛ ДЛЯ МОДИФІКАЦІЇ БІТУМІВ

Анотація. Вивчено процес сумісного окиснення гудрону та промислової нафтополімерної смоли «Піропласт-2». Наведено результати експериментальних досліджень основних закономірностей процесу одержання нафтополімерних смол з карбоксильними групами на основі фракції C_9 піроконденсату піролізу вуглеводневої сировини. Встановлено, що створені нафтополімерні смоли можуть бути використані для модифікації нафтових бітумів.

Ключові слова: гудрон, окиснений бітум, нафтополімерна смола, карбоксильна група, модифікація, бітумно-полімерна композиція.