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SYNTHESIS AND PROPERTIES OF EPOXYDIZED COOLIGOMERS OBTAINED FROM PETROLEUM RESINS SYNTHESIZED BY HETEROGENEOUS CATALYTIC OLIGOMERIZATION

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Abstract. Synthesis of cooligomers with epoxy groups was studied. They were synthesized by epoxidation of unsaturated petroleum resins obtained by heterogeneous catalytic oligomerization of liquid pyrolysis products C₉ fraction. The degree of unsaturated bonds conversion and the selectivity of epoxidation process were calculated. The properties of epoxydized cooligomers were determined and analyzed. The structurization of synthesized products in the composition of composites with epoxy resin ED-20 was studied.

Keywords: petroleum resins, C₉ fraction, heterogeneous catalytic oligomerization, epoxidation, epoxydized petroleum resins.

1. Introduction

The fractions of liquid pyrolysis products (LPP) utilization is an actual problem having both economical and ecological importance. Production of the cooligomeric products (petroleum resins) from these fractions, having a wide range of applications, is the most reasonable way of their utilization [1]. Our approach consists in synthesis of named products by heterogeneous catalytic method. Silica-alumina materials including natural and synthetic zeolites and various clays were used as acid type catalysts. The catalysts were activated by the acid before use. The activated natural clays such as bentonite clay and palygorskite were found to be the most effective ones among the studied catalysts [2].

The important feature of petroleum resins (PR) obtained from C₉ fraction of LPP by heterogeneous catalytic method is their high unsaturation. The products bromine number reaches 83.8 g Br₂/100g when using activated bentonite clay as a catalyst [2, 3] and 73.8 g Br₂/100g when using activated palygorskite [3].

Presence of unsaturated bonds in the mentioned cooligomers enables their further modification to produce functionalized products.

Oxygen-containing functional groups (carboxyl, hydroxyl, epoxy groups, *etc.*) among the functionalized oligomers products arouse considerable interest. Among them oligomers with epoxy groups deserve special attention. The reason of such attention to these materials is that the products made of them have universal mechanical, electrical and physical properties. Besides, these oligomers have good compatibility with various polymer materials, facilitating properties of the latter ones. The important property of oligomers having epoxy groups is their ability to come to crosslinked state. Moreover this transition proceeds at low temperatures (including ambient temperature) and in a relatively short time.

Considering above said, the task was set to produce cooligomers with epoxy groups from the petroleum resins previously synthesized by heterogeneous catalytic oligomerization method.

Crosslinking of polymers is one of the main ways of their structure modification that allows to facilitate a number of operational properties: strength, cracking resistance, resistance to solvents and high temperatures, *etc.* [4]. The chemical structurization including carrying out of chemical reaction between monomers or oligomers in a thin layer on a substrate is the most widespread. As a result, branched and crosslinked polymers are formed. It is known from literature [1] that PRs are used as additions to polymer mixtures in case of protective coatings (including anticorrosive coatings) based on them. The industrial epoxy resins are the main component of the mentioned composites. Products based on the epoxy resins are notable for good physico-mechanical properties as well as resistance to corrosive mediums [5]. Combination of epoxy resins with various oligomers allows to facilitate the properties of composites based on such mixtures

compared to epoxy resin itself [6]. The composites prepared from the industrial epoxy resins and unsaturated oligomers with functional groups deserve special attention. To produce composites with good physico-chemical and mechanical properties as well as high resistance to corrosion mediums, it is necessary that all components were interlinked into a single three dimensional polymer. The synthesized epoxydized petroleum resins (EPR) suit well for this purpose. The second part of the current paper is devoted to the issue of preparing composites based on EPR and the industrial epoxy-diane resin ED-20 as well as their structurization.

2. Experimental

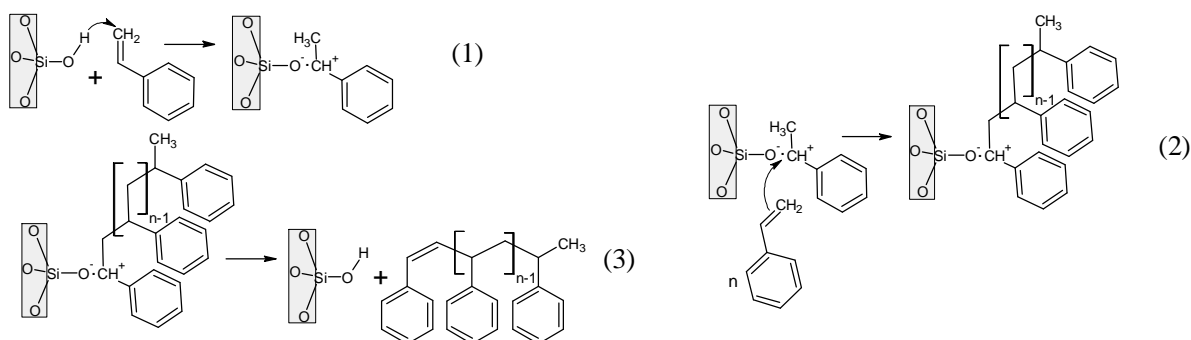
2.1. Object of Research

The objects of our research were cooligomers obtained from C₉ fraction of diesel fuel pyrolysis liquid products in the presence of heterogeneous catalysts of sili-

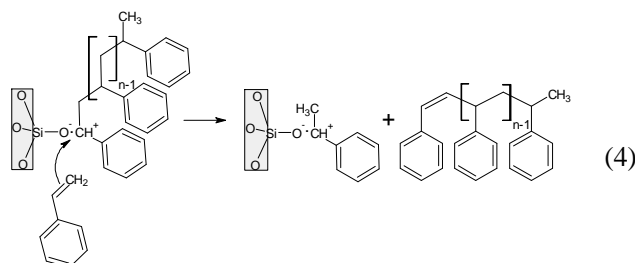
ca-alumina nature. These cooligomers mainly consist of styrene and dicyclopentadiene links as the main unsaturated components of the C₉ fraction are styrene (19 wt %), dicyclopentadiene (18 wt %) and vinyltoluenes (8 wt %). Besides these substances, α -methylstyrene (< 2 wt %), indene (< 2 wt %), allylbenzene (< 1 wt %) and other unsaturated compounds are constituents of C₉ fraction. The total content of unsaturated substances in the fraction is about 56 wt %. The main saturated component of C₉ fraction is xylene. The catalysts preparation and synthesis of cooligomers were carried out by technique described in our earlier papers [2, 3, 7].

Before further statement the nature of synthesized cooligomers unsaturation should be analyzed considering C₉ fraction composition and heterogeneous catalytic oligomerization mechanism.

Oligomerization on Bronsted active surface side may be represented by the following scheme (e.g. for styrene), where (1) – chain initiation, (2) – chain growth, (3) – stopping of the chain.



Stopping of the chain mainly occurs by the reaction (3), although transition of the chain to the monomer (4) is also possible:

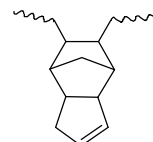


Knowing C₉ fraction composition, we can analyze the nature of all possible unsaturated bonds present in the final cooligomer product.

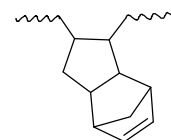
The main source of cooligomers unsaturation obtained from C₉ fraction is dicyclopentadiene (IUPAC name is [5.2.1.0^{2,6}]deca-3,8-diene). It contains the strained

ring, and two double bonds (norbornene and cyclopentene ones) in its molecule.

Thus, when oligomerization of dicyclopentadiene (DCPD) occurs by norbornene bond the cyclopentene bond remains:



When DCPD oligomerization occurs by the cyclopentene bond the norbornene bond remains:

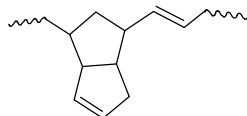


During DCPD oligomerization rearrangement of growing carbocation, resulting in somewhat different links formation, is also possible. But in case of heterogeneous

catalysis such rearrangement is much less probable compared to homogeneous catalysis.

In addition to above mentioned mechanisms of DCPD oligomerization, which consist in oligomerization by norbornene or cyclopentene double bond, another mechanism of its oligomerization is possible, namely the ring opening metathesis oligomerization with the opening of norbornene ring.

In this case the cyclopentene double bond remains and vinyl bond arises instead of norbornene one:



In case of such mechanism of DCPD oligomerization the number of double bonds is not reduced and consequently the most unsaturated product is obtained.

Oligomerization by both DCPD double bonds, that is theoretically possible in a homogeneous catalytic process, is impossible in this case. Since active sites are

fixed on the surface, DCPD molecule bound with a surface active site by one unsaturated bond cannot interact with other site by another double bond.

The other C₉ fraction components such as styrene, α -methylstyrene, vinyltoluenes, allylbenzene and indene cannot be the source of unsaturated bonds in the middle part of macromolecule. However, they (as well as dicyclopentadiene) may be the source of unsaturation at the end of cooligomer chain. Unsaturated bond arises at the end of oligomer chain when it is detached from the active site with proton detachment and active site regeneration (see oligomerization scheme given above). Regarding above said, it may be concluded that some part of total unsaturation is caused by this unsaturated bonds at the end of the cooligomer molecules. The contribution of these bonds in total unsaturation is greater the cooligomer molar weight is smaller.

Considering C₉ fraction composition we can state that cooligomer may involve the following double bonds, depending on which the compound is at the end of cooligomer chain.

Styrene	Allylbenzene	α -Methylstyrene	Indene	Dicyclopentadiene

When vinyltoluen link is at the end of PR molecule everything is similar as for styrene.

All possible unsaturated bonds, present in molecules of cooligomer obtained from C₉ fraction, are listed above. They determine PR unsaturation. Based on C₉ fraction composition it is evident that most of double bonds belong to dicyclopentadiene and styrene links.

High bromine number values of PR, obtained by heterogeneous catalytic oligomerization, indicate the possibility of effective DCPD oligomerization by this method. Besides, there is reason to assume that DCPD oligomerization largely occurs by metathesis mechanism with opening of the norbornene ring.

2.2. Technique of Cooligomers Epoxidation

4 g of cooligomer sample were dissolved in 17 ml of toluene. The prepared 20 % solution was loaded into the reactor and thermostated to the temperature of 303 K.

After establishing the process temperature required quality of oxidant was added to the reactor drop by drop. An oxidant mixture of the following composition: peracetic acid – 16.6 wt %, hydrogen peroxide – 21.0 wt %, acetic acid – the rest, was used. Besides, the mixture contains small amount of water. In this case the peracetic acid is the main reactant. The choice of this oxidant is motivated by the fact that it is produced commercially with the brand name “Dezosept Forte” and is rather cheap. Moreover this product is adapted for transportation and long storage. The oxidant was added in such amount to provide 15 % molar excess of peracetic acid relative to the number of unsaturated bonds of PR. After the oxidant addition the reaction mixture was kept under intensive stirring for 1.5 h. The reaction conditions (temperature of 303 K and duration of 90 min) are the optimal conditions of cooligomers epoxidation, obtained from LPP C₉ fraction [8].

After the end of the process the reaction mixture was transferred to a separating funnel where the water layer was separated. Then the organic layer was washed

four times with fivefold excess of water. For the first time washing was carried out with addition of small amount of soda (Na_2CO_3) to neutralize acids. After each washing the mixture was separated in the separating funnel into water (bottom) and organic (upper) layers and the water layer was poured out.

The epoxydized cooligomer was precipitated from the solution by the petroleum-ether. The precipitated product was dried in the exiccator at room temperature and residual pressure of 1.3–3.9 kPa to the constant weight.

2.3. Determination of the Epoxydized Cooligomer Properties

The cooligomers colour was determined comparing 10 % cooligomer solution in benzene with standard iodine scale. Unsaturation (bromine number (BN)) was determined by iodometric back titration according to the common technique [9]. The epoxy number (EN) of epoxydized cooligomers was determined by back potentiometric titration with the base by the technique given in [10].

2.4. Structurization of Epoxyoligomeric Compositions

The composites on the basis of epoxydized cooligomers were prepared by mixing of EPR 30 % benzene solution with industrial epoxy resin ED-20 until homogeneous state. Then the hardener was added and the mixture was stirred. After that the obtained mixture was applied onto glass plates. Polyethylenepolyamine (PEPA) was used as a hardener. Its amount in all composites was 16 g per 100 g of resin (or mixture of resins). The composites structurization was carried out at room temperature over a period of 1–5 days. After films structurization the gel-fraction content was determined.

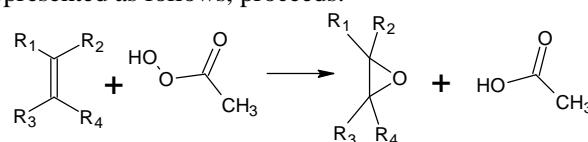
The gel-fraction content in composites based on EPR and ED-20 was determined by the extraction of ground samples with 1,4-dioxane in the Soxhlet extractor for 14 h, as described in [11].

3. Results and Discussion

3.1. Synthesis and Properties of Epoxydized PR

Eight samples of PR, having high unsaturation and obtained under conditions allowing to achieve high yields [3], were chosen for epoxidation.

The chosen PR samples epoxidation was carried out under conditions and by the technique given above. During the process the reaction, that may be schematically represented as follows, proceeds:



Colour, residual bromine number (BN_R) and epoxy number (EN) were determined for the obtained products.

By the value of cooligomers bromine number before epoxidation and residual bromine number (BN_R) of epoxydized cooligomers, the degree of unsaturated bonds conversion was calculated (X):

$$X = \frac{BN - BN_R}{BN} \cdot 100\% \quad (1)$$

By the values of BN and BN_R it is possible to calculate the value of epoxy number under condition of complete conversion of unsaturated bonds into epoxy groups (EN_{BN}):

$$EN_{BN} = \frac{M_{EG}}{M_{Br_2}} (BN - BN_R) = \frac{40}{160} (BN - BN_R) \quad (2)$$

Having determined the true value of EPR epoxy number, and taking into account the EN_{BN} value, the epoxidation process selectivity (S) may be calculated:

$$S = \frac{EN}{EN_{BN}} \cdot 100\% \quad (3)$$

The definitions and calculation results are given in Table 2.

Table 1

Properties and synthesis conditions of cooligomers chosen for epoxidation

PR synthesis conditions			PR yield, wt %	PR properties		
Catalyst	Temperature, K	Duration, h		BN , $\text{gBr}_2/100\text{g}$	Colour, $\text{mgI}_2/100\text{ml}$	M
ABC	373	3	29.9	68.5	150	550
ABC	393	3	35.1	78.0	280	571
ABC	413	3	45.3	75.1	500	742
ABC	373	1	23.5	79.6	130	707
ABC	393	1	27.9	83.8	160	678
AP	373	3	24.4	71.8	150	661
AP	393	3	29.6	69.8	220	701
AP	413	3	38.9	73.8	260	722

Notes: ABC – activated bentonite clay; AP – activated palygorskite.

Table 2

PR epoxidation process indicators and properties of obtained EPR

Colour, mgI ₂ /100ml	BN _R , gBr ₂ /100g	EN, %	EN _{BN} , %	X, %	S, %
20	26.3	10.4	10.6	61.56	96.10
25	28.8	7.9	12.3	63.08	64.56
60	32.6	7.8	10.6	56.56	73.78
15	28.4	8.7	12.8	64.35	68.07
20	39.0	6.7	11.2	53.44	60.23
15	27.3	9.5	11.1	61.96	85.77
20	24.3	10.5	11.4	65.21	91.97
25	33.9	6.8	10.0	54.06	68.37

The obtained epoxydized cooligomers are the solid crystalline light-yellow products. The colour of the EPR is about the order of magnitude lower than the colour of the PR which they are obtained from (see Table 1). Such lightening of EPR compared to initial PR indicates the reducing of the double bonds number in cooligomer, *i.e.* epoxidation process passing.

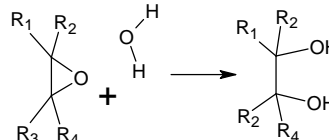
The epoxydized cooligomer bromine number remains sufficiently high. So, while in case of the epoxidation of cooligomer obtained by initiated oligomerization the residual bromine number does not exceed the value of 9.5 gBr₂/100g [8], in our case it ranges from 24.7 to 39.0 gBr₂/100g depending on the source PR properties determined by its production conditions.

The values of double bonds conversion degrees, calculated by BN and BN_R values range from 53.4 to 65.2 %. Thus, about half of unsaturated bonds remains unreacted. PR obtained at high temperatures has higher unsaturation. Besides, in case of these cooligomers epoxidation the unsaturated bonds conversion degree is less than in case of epoxidation of PR obtained at lower temperatures. So, those unsaturated bonds, which content is increased due to the PR synthesis temperature rise, are worst epoxydizable. Probably, unsaturated bonds of certain types are badly epoxydized (or cannot be epoxydized at all) because of their structure features. Most likely, unsaturated bonds of styrene, vinyltoluene, α -methylstyrene, allylbenzene and other similar links on the cooligomer macromolecules ends are epoxydized easily. Difficulties are possible in interaction with unsaturated bonds of dicyclopentadiene having strained rings in its structure. So such unsaturated bonds are badly epoxydized. Besides, the steric hindrances caused by neighboring groups may take place when epoxidizing the unsaturated bonds in the middle part of the chain, while in case of vinyl and allyl groups epoxidation there are no such hindrances at the ends of the chain. Such considerations explain why double bonds conversion degree is 81.4 % [8] in case of PR epoxidation obtained from C₉ fraction by initiated cooligomerization, since

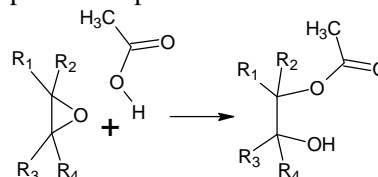
dicyclopentadiene links are absent in such cooligomers composition.

The epoxy number of EPR obtained by us ranges from 7 to 10 % that is approximately equal to EN values of products, production of which has been described in [8]. Comparing the EN values of our EPR with the EN of cooligomers synthesized by initiated cooligomerization in the presence of initiators with epoxy groups [12, 13] we can state that EN of our cooligomers is at the average 1.5–2.5 times higher.

The epoxidation selectivity is slightly lower than in case of epoxidation of PR obtained by homogeneous catalytic, initiated or thermal oligomerization [8]. And the PR synthesized by us at higher temperature are epoxydized with lower selectivity. The main side reactions, occurring along with the basic one, are the hydrolysis of epoxy groups:



and their acidolysis by acetic acid present in the oxidation system composition and produced from peracetic acid during the epoxidation process:



In particular, the stage of EPR solution washing with large excess of water contributes to the hydrolysis reaction. Consequently, besides the epoxy groups and unreacted double bonds, the little amount of hydroxyl and ester groups appears in EPR composition. Proceeding of these reactions decreases the epoxidation process selectivity. And the most strained epoxy groups hydrolyze most readily. The most strained epoxy groups appear as a result of cyclopentene and norbornene double bonds of cyclopentadiene links epoxidation. Thus, high

content of double bonds in PR contributes to the epoxidation selectivity decrease.

3.2. The Structurization of EPR and the Composites Based on EPR and Epoxy Resin ED-20

The further task of our research was the preparation of composites based on the industrial epoxy-diane resin ED-20 and EPR obtained by us, as well as their structurization studying.

For this purpose we chose the epoxydized cooligomer obtained by PR epoxidation from C₉ fraction in the presence of the activated bentonite clay as a catalyst at oligomerization temperature of 373 K and duration of 1 h. This epoxydized resin was chosen because it has an epoxy number approximately equal to the average *EN* value for all EPR obtained by us (see Table 2). Besides, this epoxydized cooligomer has a very light colour that enables their use in coatings of any colour, including decorative coatings of light colours.

Table 3

Composition of the epoxy-petroleum resin composites

Composites designation	I	II	III	IV	V	VI
Weight ratio ED-20/EPR	100/0	90/10	80/20	50/50	20/80	0/100

The structurization was studied at room temperature over a period of 1–5 days. The kinetics of composites hardening was monitored by the content of gel-fraction in it. The research technique is given above. The obtained results are given in Fig. 1.

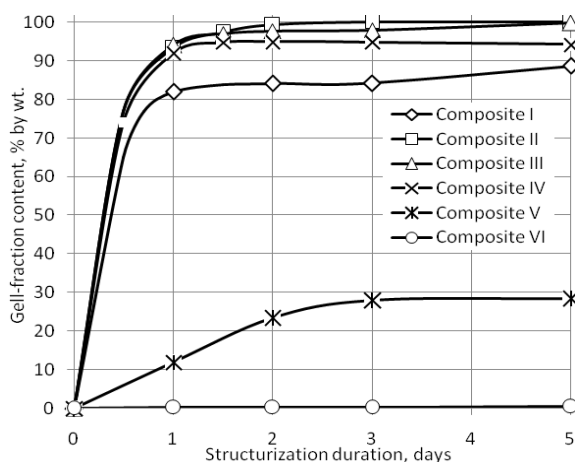


Fig. 1. The relationship between gel-fraction content and structurization process duration for composites with different ED-20/EPR weight ratio

As can be seen, the composites II-IV have higher gel-fraction content compared to the composite I containing no EPR. The gel-fraction content in the composite VI consisting only of EPR and the hardener is less than 1 wt % even after 5 days. In case of the ED-20/EPR ratio equal to 20/80 (composite V) structurization goes slower compared to composites I-IV, and the gel-fraction content does not exceed 29 wt %. The composites I-IV structurization goes rapidly and is almost completed during the first day. The further growth of gel-fraction content occurs very inessentially and practically stops after the second day of structurization. Thus, it may be stated that the structurization duration of 2 days is quite enough for structurization of the composites I-IV. Obtained results allow to draw the following conclusions. The EPR addition to ED-20 definitely increases the gel-fraction content in the composite but this happens only to a certain limit. The higher the EPR content, the lower is the gel-fraction content, and when EPR content in the composite is too high the gel-fraction content becomes lower than in case of composite I containing no EPR. Thus, the composite II has the highest gel-fraction content, the composite III has very close but a little lower gel-fraction content, and when the ED-20/EPR ratio is 50/50 (composite IV) the gel-fraction content is already significantly lower. So, the presence of EPR at the ED-20/EPR ratio up to 50/50 promotes the ED-20 resin structurization. But in case of the ED-20/EPR ratio of 20/80 the gel-fraction content dramatically decreases compared to the ED-20 structurization. Thus we may conclude that the EPR molecules interlink each other badly (they almost don't interlink). The results obtained for the composites VI and V indicate this fact. But the EPR molecules interlink with the ED-20 molecules very well. When the EPR content becomes higher than its content in the composite IV, the number of EPR molecules which cannot interlink with the ED-20 molecules increases, and these excessive EPR molecules dissolve during extraction, *i.e.* compose a sol-fraction.

Hardness of the composites I-V after 10 days of structurization at room temperature was determined with a pendulum device M-3.

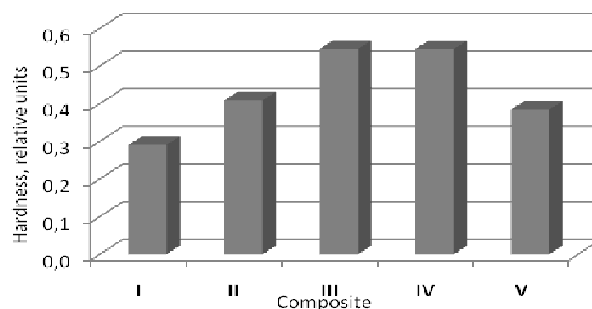


Fig. 2. Hardness of the epoxy-petroleum resin composites

The obtained results indicate that the composites prepared with EPR addition to the industrial epoxy-diane resin ED-20 (composites II-V) have an appreciably higher hardness compared to the composite containing no epoxydized PR. And the dependence between hardness and EPR content in the composites has an extremum. The increase of ED-20/EPR ratio from 100/0 to 90/10 results in hardness increase of 40.6 %, the increase of the ratio from 90/10 to 80/20 leads to hardness increase of 33.6 %, and its increase from 80/20 to 50/50 has no longer any effect. Further increase of the EPR content leads to the composite hardness decrease. Thus, in case of such structurization conditions and such amount of hardener (16 g per 100 g) a composite with the highest hardness may be obtained when the ED-20/EPR weight ratio ranges from 80/20 to 50/50. Generally, the EPR addition to ED-20 allowed to increase the composite hardness almost by 88 % compared to the structurized ED-20. Decrease in hardness of the composites having the ED-20/EPR ratio more than 50/50 is obviously explained by low cross-linking degree of such composites (see Fig. 1).

4. Conclusions

The cooligomers, synthesized from C₉ fraction by the heterogeneous catalytic method developed by us, have high unsaturation and thus may be epoxydized to obtained epoxydized products. In case of mixture containing peracetic acid use as an oxidant the unsaturated bonds conversion of 65 % with the selectivity of more than 90 % may be achieved. The epoxy number reaches 10 % in this case. The epoxidation conditions: temperature 303 K and duration 1.5 h are mild that suggests the low energy intensity of this process. It may also be concluded, that the epoxidation of cooligomers synthesized by heterogeneous catalytic oligomerization allows to obtain the products with both rather high epoxy number and high residual unsaturation.

The obtained EPR may be used for preparation of the composites with the industrial epoxy-diane resin ED-20. The EPR addition to the ED-20 resin appreciably increases the gel-fraction content in case of EPR/ED-20 weight ratio no more than 50/50.

In view of the fact that the EPR addition makes the end composite cheaper, the high content of EPR is desirable. However the composite must have good hardness and gel-fraction content. Considering the gel-fraction content and hardness determination results, the composite IV, having EPR/ED-20 weight ratio 50/50,

may be said to be the most preferred. The preferred structurization duration is 2 days.

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СИНТЕЗ ТА ВЛАСТИВОСТІ ЕПОКСИДВМІСНИХ КООЛІГОМЕРІВ, ОТРИМАНИХ НА ОСНОВІ НАФТОПОЛІМЕРНИХ СМОЛ, СИНТЕЗОВАНИХ МЕТОДОМ ГЕТЕРОГЕННОКАТАЛІТИЧНОЇ ОЛІГОМЕРИЗАЦІЇ

Анотація. Вивчено синтез коолігомерів із епоксидними групами внаслідок епоксидування ненасичених нафтополімерних смол, отриманих гетерогенно-каталітичною коолігомери-зацією фракції C₉ рідких продуктів піролізу. Розраховано ступінь конверсії ненасичених зв'язків та селективність процесу епоксидування. Визначено та проаналізовано властивості епоксидованих коолігомерів. Досліджено процес структурування синтезованих продуктів у складі композицій із епоксидною смолою ЕД-20.

Ключові слова: нафтополімерні смоли, фракція C₉, гетерогенно-каталітична олігомеризація, епоксидування, епоксидовані нафтополімерні смоли.