INVESTIGATION OF THE PROPERTIES OF PRODUCTS AND REGULARITIES OF COOLIGOMERIZATION OF ALKENES-1 WITH DICYCLOPENTADIENE

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The binary cooligomerization reactions have been carried out with the participation of alkenes-1 with dicyclopentadiene and the Lewis acid-type catalyst BF₃·O(C₂H₅)₂ and the regularities of this reaction, the copolymerization constant values and the composition of the prepared products have been determined. The binary copolymer samples with average molecular weight 1600–6150 have been obtained and investigated as a synthetic component in the M-6 oil. It has been shown that by addition of alkenes-1-dicyclopentadiene binary oligomer as a synthetic component to M-6 oil, it is also possible to obtain both the base oil meeting the requirements for viscosity-temperature properties and to give the corrosion resistance to the obtained oil.

**Keywords:** octene-1, dicyclopentadiene, cooligomerization, viscosity additives.

**Introduction**

The oligomers or cooligomers of alkenes-1 are used to increase lubricating oils, so that the use of getting additives attracts attention for macromolecular compounds, synthetic components and polyfunctional additives [1–3]. Creation of polyfunctional additives, which improve the quality of the lubricants by using such compounds is economically efficient [4–6].

The polymerization of multicomponent polymer additives is accompanied by polymerization, together with vinyl aromatic, carbocyclic and alkyl acrylate monomers of the alkenes-1 [7, 8]. In connection with this, binary cooligomerization reaction with alkenes-1 dicyclopentadiene, which has more raw material resources have been carried out, regularities and products of this reaction have been investigated. In turn, the use of dicyclopentadiene is associated with its incorporation into the cooligomeric macromolecule, which gives anticorrosion property to the macromolecular compound in the composition of lubricants and it can be used as an additive with bifunctional effect. The dicyclopentadiene has been extracted from the distillation of coal for a long time, and recently the petrol pyrolysis is obtained from liquid pro-duits. The dicyclopentadiene has two various reactive π-bonds and can be converted into π-bonds for providing the unsaturation to high molecular compounds (for example to vulcanize the ethylene-propylene cooligomer). The cyclic compounds are more resistant to oxidation than open-chain hydrocarbons and inclusion of dicyclopentadiene links into alkenes-1 macromolecular chains should increase the durability of the end product to thermal and thermochemical effects.

**Experimental**

The dicyclopentadiene is a condensation product of the two cyclopentadiene molecules (melting point 305–306 K, boiling point 443 K).

For oligomerization it were used octene-1 (hexene-1) and cyclopentadiene and BF₃·O(C₂H₅)₂ – as catalyst. The process was carried out as methodologically as follows. After addition of calculated quantity of monomer mixture of octene-1 and cyclopentadiene and hexane or heptane as a solvent to a flask equipped with mixer, thermometer and drop funnel the mixer is activated and the reaction temperature in the flask is created by cooling bath. BF₃·OEt₂ catalyst is introduced into the system as a solution through the drop funnel. The catalyst feed rate is adjusted so that the temperature in the tube does not rise above 415 K. After the temperature rise, the
reaction mass is added for 1 hour to complete the reaction, so that the reaction is complete. Then the catalyst complex is decomposed by 2–3% in alkaline solution, washed with water-isopropyl alcohol (no emulsion) to neutral medium, filtered, solvent and easy part is distilled, the yield of prepared oligomer compound, kinematic viscosity and value of molecular weight is determined. The cooligomerization process has been carried out by change of the temperature, the ratio of monomers and the catalyst quantity within certain ranges.

The composition and structure of synthesized compounds have been investigated by methods of IR spectroscopy and NMR, elemental analysis (determination of composition on carbon) and fractional composition.

The IR spectra have been taken on Varian 640 IR FTIR spectrometer, Specord M 80 (Carl Zeiss Jena) and Spectroscopy FTIR Nicolet is 10 devices.

The spectra NMR have been taken on the 300 MHz "Bruker" device using CCl₄. 90 MHz Perking Elmer has been used to explore some points.

The thermal analysis of alkenes-1 and cyclopentadiene cooligomers has been carried out in nitrogen atmosphere by heating at temperature of 293–773 K in derivatography of OD-102T type (Paulic-Paulic-Erdey system) at dynamic regime of 5 K/min. The aluminum oxide was used as ethanol and a quantity of the sample was 0.100g, the temperature rise rate was 5 K/min. For each sample, DTA, TGA, and DTG curves were obtained, the thermal or thermal-oxidative destruction of the sample was estimated based on the temperature corresponding to the mass loss of 5, 10 and 50% of the TGA and DTG curves.

**Results and discussion**

The effects of several factors on the reaction have been investigated. The process has been carried out with the participation of Lewis acid type catalysts BF₃O(C₂H₅)₂ and the obtained results are presented in Table 1. The molar ratio of dicyclopentadiene and the temperature of the process in the mixture of the initial monomers are more prominent. Increasing the temperature at 253–293 K practically does not influence on yield of the targeted product, but the resulting copolymer samples reduce the average molecular weight from 6150 (5800) to about 3140 (3000). Naturally, an increase in temperature intensifies both chain elongation and breaking reactions, but the chain breaking reactions rate increases more rapidly than the elongation reaction, which results in a decrease of the molecular weight of the cooligomer. An increase of the quantity of dicyclopentadiene in the mixture of the initial monomers from 0.10 to 0.30 mol %, leads to the decrease of yield and molecular weight of the prepared cooligomer (from 86.5% to 75.4% and from 3140 (3000) to 1670 (1600) and it is explained by retarding effect of dichloropentadiene on cooligomerization.

The polymerization constants have been determined together with alkene-1 and dicyclopentadiene by using the Fayneman-Ross method to describe the obtained results. These copolymer constant values, which are determined by the participation of various catalysts, do not differ from each other (Table 2). The obtained values show that the copolymer constant of hexene-1 with the use of both catalysts is greater than the unit, but the copolymer constant of dicyclopentadiene is smaller than unit, i.e. the connection rate of the alkene-1 monomer in the active centers is higher than the dicyclopentadiene monomer. Thus, the formed copolymer macromolecules should be rich with links, which made from alkene-1.

Because the co-polymerization constant \( r_2 \) for the dichloropentadiene monomer is zero, it can not generate homopolymer, but this monomer is only included in the composition of the cooligomer. It should also be noted that when the homopolymerization of dicyclopenta diene is carried out with the participation of catalysts, there are obtained the solid substances of dark amber color, insoluble in the petroleum oils with molecular weight in the interval 300–600. The copolymerization products of hexene-1 with dichloropentadiene are good compounds in the oils.
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Table 1. Conditions for preparation of cooligomers of octene-1 (1–8) and hexene-1 (9–16) (M₁) with dicyclopentadiene (M₂), yield of copolymers and some indicators; catalyst BF₃·O(C₂H₅)₂

| №  | Temperature, K | M₁: M₂ mol. p. | Quantity of catalyst, % | Yield of cooligomer, % | Average molecular weight Mₐ | Bromine number of cooligomer, mg Br₂/100 g |
|----|----------------|----------------|------------------------|-----------------------|-----------------------------|---------------------------------------------|
| 1  | 293            | 0.9:0.1        | 0.5                    | 81.5                  | 3140                        | 11.6                                        |
| 2  | 273            | 0.9:0.1        | 0.5                    | 79.9                  | 3730                        | 11.1                                        |
| 3  | 263            | 0.9:0.1        | 0.5                    | 82.7                  | 4580                        | 10.6                                        |
| 4  | 253            | 0.9:0.1        | 0.5                    | 85.1                  | 6150                        | 10.2                                        |
| 5  | 293            | 0.8:0.2        | 0.5                    | 78.6                  | 2920                        | 16.9                                        |
| 6  | 293            | 0.7:0.3        | 0.5                    | 71.3                  | 1670                        | 21.8                                        |
| 7  | 293            | 0.9:0.1        | 0.3                    | 58.5                  | 2680                        | 17.2                                        |
| 8  | 293            | 0.9:0.1        | 0.7                    | 91.4                  | 2150                        | 19.1                                        |
| 9  | 293            | 0.9:0.1        | 0.5                    | 85.8                  | 3000                        | 12.4                                        |
| 10 | 273            | 0.9:0.1        | 0.5                    | 84.8                  | 3600                        | 11.8                                        |
| 11 | 263            | 0.9:0.1        | 0.5                    | 86.1                  | 4500                        | 10.9                                        |
| 12 | 253            | 0.9:0.1        | 0.5                    | 88.3                  | 5800                        | 10.4                                        |
| 13 | 293            | 0.8:0.2        | 0.5                    | 82.6                  | 2800                        | 18.9                                        |
| 14 | 293            | 0.7:0.3        | 0.5                    | 75.4                  | 1600                        | 25.0                                        |
| 15 | 293            | 0.9:0.1        | 0.3                    | 60.8                  | 2500                        | 11.6                                        |
| 16 | 293            | 0.9:0.1        | 0.7                    | 95.9                  | 2000                        | 11.7                                        |

Table 2. Composition of the oligomer depending on the initial ratio of monomers in cooligomerization of hexene-1 with cyclopentadiene (catalyst BF₃·O(C₂H₅)₂)

| Ratio of monomers in the initial mixture, mol. p. | Composition of oligomer, mol. p. | r₁ | r₂ |
|-----------------------------------------------|---------------------------------|----|----|
| M₁               | M₂               | m₁  | m₂  |
| 0.85             | 0.15             | 0.84 | 0.16 | 1.28 | 0  |
| 0.75             | 0.25             | 0.73 | 0.27 |     |    |
| 0.35             | 0.65             | 0.36 | 0.64 |     |    |
| 0.25             | 0.75             | 0.24 | 0.76 |     |    |
| 0.15             | 0.85             | 0.19 | 0.81 |     |    |
| 0.05             | 0.45             | 0.05 | 0.95 |     |    |

The observed results can be explained by the fact that when a quantity of dicyclopentadiene in the comonomer mixture is high, it is possible to obtain dicyclopentadiene dimer and trimer at the initial time of the reaction, but they are converted into compounds soluble in the oils in interaction with hexene-1 molecules.

As a result, during copolymerization with hexene-1 (in the presence of Lewis acid-type catalysts) a preparation of homopolymer (or homooligomer) of dicyclopentadiene is not observed.

The cooligomer macromolecules synthesized under various conditions, especially, in the small quantity of dicyclopentadiene monomer is enriched by links forming from alkene-1 monomer.

It should be noted that in carrying out of the copolymerization of dicyclopentadiene with hexene-1 in the presence of BF₃·O(C₂H₅)₂ the copolymerization constant values are r₁ = 1.28 and r₂ = 0. At the same time, the copolymerization constants determined by taking the catalyst BF₃·O(C₂H₅)₂ in octene-1 of the dicyclopentadiene have r₁ = 1.23, r₂ = 0. Comparison of these values shows that r₁ and r₂ constant values do not practically depend on the nature of the catalyst (both catalysts are of cation type), but r₁ constant value in passing from hexene-1 to octene-1 as comonomer is slightly decreased, so when a length of the alkyl radical containing π-bond connected to carbon is increased, an activity of 1-alkene in the cation copolymerization reaction with dicyclopentadiene is decreased.

The reason for such change of the relative activity of alkenes-1 with dicyclopentadiene in the polymerization reaction is likely to be the electron-donor effect of the alkyl substituent to the alkene-terminated carbocation, more precisely, to the carbon atoms carrying the positive charge. With increasing a number of carbon atoms in this substituent its electron-donority increases. At the same time, the increase in alkaline substituent increases the spatial difficulty created by it. These factors determine the copolymerization constant values, i.e., their relative activity in the polymerization reactions with dicyclopentadiene of 1-alkenes used as a comonomer.
The dicyclopentadiene has two π-bonds differing from its activity and π-bond in the six-membered ring is more reactive than π-bond in the five-membered ring. During copolymerization, the opening of both π-bonds is not observed in a molecule. Thus, the prepared cooligomers have an unsaturation (the bromine value is between 10.2–25.0 mg in Br/100g – Table 1) and forming the branched or cross-linked structure is not noted and the cooligomers are well dissolved in the oil. However, the result of spectral investigations showed that each of the double bonds of dicyclopentadiene can participate in the copolymerization, but two π-bonds in a molecule are not opened at the same time; when one of the π-bonds is opened, the other remains untouched and finally the unsaturated compounds are formed.

These suggested ideas have been confirmed by the investigations of the IR and NMR spectra of the cooligomers. In the IR spectrum the absorption bands at 1610 and 1640 cm\(^{-1}\) are observed, which correspond to the dicyclopentadiene link. In the spectrum NMR the signals of chemical displacement 5.4 and 5.86 mln\(^{1}\) are observed and these signals can be referred to protons being in double bond in dicyclopentadiene rings. The NMR spectra of the synthesized cooligomers have been taken at different frequencies in order to confirm that these displacements belong to protons being in a separate π-bond. The distance between these signals in the spectra of the separate frequencies is changed, i.e. these signals belong to separate protons: the signal observed at 5.86 mln\(^{1}\) corresponds to the protons of π-bond in the six-membered ring, and the signal observed at 5.4 mln\(^{1}\) corresponds to the protons of π-bond in the five-membered ring. It has been revealed on the basis of the results of the corresponding calculations that during the cooligomerization 75% of π-bonds in the six-membered ring, 25% of π-bonds in the five-membered ring in the dicyclopentadiene molecule are opened. Thus, it has been once again confirmed that the reactivity of π-bonds in the dicyclopentadiene molecule is determined by various factors, including reaction conditions. The study of influence of the quantity of the taken catalyst on cooligomerization process shows that no more 3% of a quantity of AlCl\(_3\) [8], a quantity of BF\(_3\)O(C\(_2\)H\(_5\))\(_2\) is less 1.0% in the system and does not influence on molecular weight of the cooligomer, but decreases a yield of the cooligomer (from 83.5% to 50.3%). Increasing of quantity of the catalyst increases a yield to 93.7%.

The physico-mechanical properties of the polymer compounds are determined by the presence of their macromolecular composition along with average molecular weight of them and whether it is homogeneous or not. In this regard, the composition of the studied cooligomer has been investigated. For this purpose, the sample to be investigated has been dissolved in hexane and passed through column of ACK mark filled with silicagel.

It has been determined by investigation of IR spectrum of the obtained fractions that there is no fraction containing dicyclopentadiene links, at the same time the fraction corresponding to the homooligomer consisting of the appropriate links of dicyclopentadiene monomer only. Thus, as a result of polymerization of octene-1(hexene-1) with dicyclopentadiene in the presence of Lewis acids-type catalysts there are formed the real binary cooligomers, enriched with formed links of octene (hexene-1) monomers, in this or other degree depending on polymerization conditions.

The alkenes-1-dicyclopentadiene binary cooligomers have a low average molecular weight (1600–6150) and such compounds are not important as viscous additives for oils. The polymers of relatively low molecular weight are usually used as primary raw materials to prepare synthetic components and polyfunctional polymer additives for oil [9–14].

The octene-1-dicyclopentadiene binary cooligomers have been investigated as a synthetic component in the M-6 oil and the obtained results are given in Table 4. The cooligomer with molecular weight 2150 containing 0.265 mol.p. of dicyclopentadiene links has been used as a synthetic component.

The results show that in increasing the concentration of hexene-1-dicyclopentadiene cooligomer used as a synthetic component in the composition of M-6 oil an improvement of viscosity-temperature properties of the composition takes place.
Table 3. Yield, molecular weight and element composition of cooligo(octene-1-dicyclopentadiene). Reaction time is 5 hours, catalysts BF3·O(C2H5)2.

| № | Ratio of monomers of cooligomerization, mol. p. | Yield of cooligomer, % | Average molecular weight of cooligomer | Link composition of cooligomer, % |
|---|---|---|---|---|
| 1 | 0.9:0.1 | 81.5 | 3200 | \( m_1 \) (octene-1) 76.3
| 2 | 0.85:0.15 | 80.1 | 2050 | \( m_2 \) (dicyclopentadiene) 23.7
| 3 | 0.8:0.2 | 78.6 | 2920 | 73.5
| 4 | 0.75:0.25 | 75.2 | 2130 | 26.5
| 5 | 0.7:0.3 | 71.3 | 1670 | 70.8

Table 4. Influence of cooligo(octene-1-dicyclopentadiene) on the viscosity-temperature properties of M-6 oil

| Concentration of cooligomer in the oil, % | Kinematic viscosity of oil, mm²/s | Viscosity index | Corrosion in lead boards, GOST 20502-75, g/m² |
|---|---|---|---|
| | 373 K | 313 K | | |
| 0 | 5.8 | 39.2 | 80 | 177 |
| 3.5 | 6.2 | 42.7 | 84 | 145 |
| 7.0 | 6.7 | 48.7 | 87 | 74 |
| 10.5 | 7.2 | 56.2 | 95 | 47 |
| 14 | 8.0 | 62.0 | 105 | 3.5 |
| 17.5 | 8.6 | 63.1 | 106 | 1.0 |
| 21.0 | 9.0 | 64.3 | 107 | — |
| 24.5 | 9.3 | 67.6 | 108 | — |

For example, increasing the concentration of the cooligomer in the M-6 oil from 3.5 to 10.5–14% increases the value of the viscosity index from 84 to 95–105. These results show that it is possible to obtain the base oil with the kinematic viscosity \( \pm 0.5 \; \text{mm}^2/\text{s} \) at 373 K, meeting the modern requirements, as according to the norm a viscosity index value of such oils should be higher 93.

The influence of the cooligomer on viscosity index value of M-6 oil is more effective at concentration of 10–15%. A further increase of concentration of the cooligomeres does not cause a sharp change in the viscosity index value. Thus, after a certain value of concentration of macromolecular additive (higher than limit value of concentration) an influence between macromolecules is intensified and they lose their independence somewhat.

At the same time, it should be noted that the investigated cooligomer demonstrates an anticorrosion property in the composition of M-6 oil. Thus, the cooligomer with dicyclopentadiene structure fragmentary has an unsaturation, i.e. in opening of one of \( \pi \)-bonds of dicyclopentadiene, the other remains untouched. Due to an unopened \( \pi \)-bond, the cooligomer is chemically sorbed on the metal surface, forming a thin protective coating layer, which protects the metal from external influences and prevents a corrosion. For complete prevention of corrosion it is enough to add 15% of alkene-1-dicyclopentadiene cooligomer as a synthetic component to the M-6 oil. Thus, according to the available standard, 5 g/m² corrosion is accepted as "non-existent".

Thus, by adding a cooligo(alkene-1-dicyclopentadiene) as a synthetic component to M-6 oil, it is also possible to obtain both the base oil meeting the requirements for viscosity-temperature properties and also to give the corrosion-resistant properties to the obtained oil.

For determination of influence of value of average molecular weight of the synthesized cooligo(octene-1-dicyclopentadiene) on viscosity-temperature parameters of the transformer oil, Table 5 is presented for the results of the investigations of these oils.

Table 5. Influence of average molecular weight of cooligo(octene-1-dicyclopentadiene) on the viscosity-temperature parameters of the transformer oil

| Average molecular weight of cooligomer | Concentration of cooligomer in the oil, % | Viscosity of concentrated oil, mm²/s | Ratio of viscosities at 313 and 373 K | Viscosity index |
|---|---|---|---|---|
| 2150 | 20 | 62.0 | 8.41 | 7.34 |
| 2920 | 15 | 56.2 | 8.52 | 6.42 |
| 4580 | 10 | 54.9 | 8.61 | 6.46 |
| 6150 | 7 | 56.0 | 8.78 | 6.35 |

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It is seen from Table that by increasing the average molecular weight of the cooligomer from 2150 to 6150, the cooligomer concentration used for mixing with oil decreases from 20 to 7%. Using 7% of the cooligomer with molecular weight 6150, it is possible to obtain the oil with kinematic viscosity 8.78 at 373 K and viscosity index 125.

Conclusions

1. The binary cooligomerization reactions have been carried out with the participation of alkenes-1 with dicyclopentadiene and the Lewis acid-type catalyst BF₃·O(C₂H₅)₂ and the regularities of this reaction, the constant values of copolymerization and compositions of the prepared products have been determined.

2. The binary cooligomer samples with average molecular weight 1600–6150 have been obtained and investigated as a synthetic component in the oil. It has been shown by addition of cooligomers as a synthetic component to M-6 oil that at the same time, it is also possible to obtain both the base oil meeting the requirements for viscosity-temperature properties and to give the corrosion resistance to the obtained oil.

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1-ALKENLƏRİN DİTSİKLOPENTADIENLİ BİRGƏ POLİMERLƏŞMƏSİNİN QANUNAĞYÜNLUQLARININ VƏ MƏHSULLARININ XASSƏLƏRİNİN TƏDQİQİ

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Heksen-1-in ditsiklopentadienli Lyus turşuları tipli katalizatorun [BF₃·O(C₂H₅)₂] istirikı ilə birinigəpolimerləşmə reaksiyaları həyata keçirilmiş, bu reaksiyanın qanunəyünluqları, birgo polimerləşmə səbətlərinin qiymətləri, alnan məhsulların tərkibləri və qurulları müəyyən edilmişdir. Orta molekül kütülərinin qiymətləri 1600–6150 təşkil edən birinigə oligomer nümünələri alınıb və vəzifələrin tərkibində sintetik komponent kimə tədqiq edilmişdir. Göstərilmişdir ki, M-6 yağın sintetik komponent kimə səoliq(alken-1-ditsiklopentadien) əlavə edilmiş, eyni zamanda, ham özülük-temperatür xassələrinin göra tələbləri cavab verən baza yağlı thin bağlanıb, ham da alınıb vəya korroziyaya qarşı davamlıq xassası da aşılamaq mümkündir.

Açar sözlar; okten-1, ditsiklopentadien, birgəpolimerləşmə, özülük aşqarları.

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Осуществлены реакции бинарной сополимеризации алкенов-1 с дициклопентадиеном в присутствии катализаторов типа кислот Льюиса – BF₃·O(C₂H₅)₂, и установлены закономерности, значения констант сополимеризации, составы и структуры продуктов этих реакций. Получены образцы бинарных соолигомеров со значением средних молекулярных масс 1600–6150 и исследованы в качестве синтетического компонента в составе масел. Показано, что путем добавления бинарного соолиго(октен-1-дициклопентадиен)-а в состав масла М-6 в качестве синтетического компонента одновременно можно получать базовые масла, отвечающие требованиям по вязкостно-температурным свойствам, и придать им антикоррозионные свойства.

Ключевые слова: октен-1, дициклопентадиен, сополимеризация, вязкостные присадки.