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CONTENT

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SYNTHESIS OF UNSATURATED ETHERS OF INDIVIDUAL ALICYCLIC CARBOXYLIC ACIDS IN THE PRESENCE OF IONIC LIQUID CATALYST

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Etherification reactions of individual alicyclic carbonic acids -cyclohexane, methylcyclohexane, biticloheptene with unsaturated allyl alcohol in the presence of ionic liquid catalysts- N-methylpyrrolidone hydrosulfate were carried out and esters were synthesized with high yield (85-90%). The structures of the obtained ethers were confirmed by modern analytical methods and physical and chemical parameters were determined. The ionic liquid catalyst is environmentally friendly, and since the reaction goes faster, no additional resin products are formed, and the yield is high, non-toxic, can be separated after the reaction and used repeatedly. Since the compounds containing unsaturated bonds have a high reactivity in petrochemistry.

Keywords: *cyclichexane-. bicycloheptene - methyl cyclohexane carbonic acids, ionic liquid, allyl, propargyl alcohols, N-methylpyrrolidone hydrosulfate.*

INTRODUCTION

Carbonic acid esters are valuable and widely used substances in the petrochemical industry as solvents, plasticizers for polymeric materials, antioxidants and depressants for diesel fuel, as additives to mineral and lubricating oils, and they are among the valuable products used in various sectors of the economy and are in high demand. In recent years, the growth of industrial production, the increase in chemical waste, the synthesis of carbon dioxide derivatives, especially esters with low-cost waste, multi-stage methods of corrosion of equipment with the participation of acid catalysts create "nature and society" problems in the petrochemical industry. In this regard, the development of an environmentally and economically efficient, simplified method of obtaining complex ethers with high yield in the presence of effective catalysts is one of the urgent problems of today. Given that esters of carbonic acids have a wide range of applications in the national economy, it is expedient to synthesize them using high-yield, environmentally friendly and economically viable methods using efficient catalysts.

In the article [1], the scientists carried out the process of ethering of long-chain carbonic acids in equivalent amounts with alcohols in the presence of the catalyst $ZrOCl_2$ $8H_2O$ by the method of direct condensation and studied the activity of the catalyst. At this time, it was determined that the highest yield was observed in the reactions between monobasic carbonic acids and alcohols, but the lowest yield was observed between the branched acids and binary alcohols.

Scientists have carried out reactions of esterification of aliphatic and aromatic carbonic acids with alcohols with the catalytic presence of the $[(NH_4)H_2PW_{12}O_{40}]$-complex. Also, comparative studies with classical catalysts have been conducted and it has been observed that ether reactions in the presence of these [2] catalysts are long-lasting.

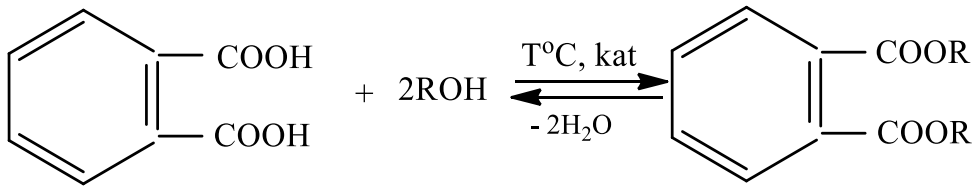
In the literature, esterification reaction of sebacic acid with aliphatic alcohols have

been carried out. The properties of the synthesized esters were studied, the activity of highly dispersed catalysts used for etherification processes, their effect on the process and the yield of the target product and their applications were studied [3].

Esterification of aromatic carbonic acids with aliphatic alcohols (2-propanol, 1-butanol, iso-butanol, 3-pentanol, 1-hexanol, heptanol, octanol and decanol) was carried out in the presence of H_3PO_4/TiO_2-ZrO_2 catalyst. In this case, high-yield esters were synthesized using 2.5% of the catalyst, and it was determined to recycle this catalyst to make it suitable for reuse [4].

The esterification reactions of carbonic acids are mainly carried out in the presence of acidic and basic catalysts, but it should be noted that some reactions can be carried out without a catalyst, such reactions are carried out mainly at very high temperatures and pressures. In the literature [5] an etherification reaction of various carbonic acids, propanol or hexanol was carried out in an autoclave at a temperature of 150 ° C and ether was obtained with high yield.

The main purpose of the study [6] is to study the catalytic activity of nano-sized TiO_2 (PC-500, PC-105, L-181) in the esterification reactions of phthalic acid with monohydric alcohols and the selection of a more effective catalyst. The etherification reaction is carried out according to the following scheme at a temperature of 110-120° C, in a 1: 2.5 mol ratio of acid and alcohol, the amount of nano-TiO_2 (PC-500, PC-50, L-181) taken as 1-2% (with respect to the acid), with high yield for 3-4 hours:



here R: C_6H_{13} - $C_{11}H_{23}$

It should be noted that despite the presence of a number of effective catalysts in organic chemistry, the acquisition of more effective, environmentally friendly catalysts has always been in the spotlight.

Today, the use of ionic liquid catalysts stimulates the emergence of new processes in "green chemistry". Ionic fluids are widely used in organic chemistry, biochemistry, petrochemistry and a number of syntheses [7]. Note that proton ion fluids show higher acidity.

V.M.Abbasov and his colleagues synthesized esters of C1-C3 series aliphatic alcohols on the basis of kapron acid using ionic liquids (N-methyl pyrrolidone hydrosulfate and 1,4-dimethylpyperazine hydrosulfate) as catalysts, the effect of the reaction time was studied and it was determined that the yield was 85%. The values of the synthesized ethers were also determined by analytical and spectral analysis methods (IR-, 1H and 13C NMR) [8-11].

By saying ionic liquids, what is considered is their salts, which are ionic in nature and have a low boiling point (<100°C) and belong to a new class of solvents [12-14].

Today, ionic liquids have been used successfully in a number of processes known in the foreign literature, replacing organic solvents. Ionic fluids form a two-phase system with a number of organic products, allowing for multiphase reactions. In terms of physical and chemical properties, ionic liquids do not have vapor pressure, which simplifies the expulsion of products, which is due to their chemical activity, unlike

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traditional solvents, which stimulates the creation of new class of solvents in catalytic systems.

Three types of highly acidic ionic liquids were synthesized on the basis of 4-sulfobenzyl imidazole hydrosulfate [15] and the catalytic activity of oleic acid as a catalyst in the esterification reaction with methanol was determined. Ionic fluids have been confirmed by modern UV, IR, 1H, 13C, NMR spectral analysis methods. Reaction parameters were studied, for example: temperature, molar ratio, catalyst load, time and stability. Experimental results have shown that acidic ionic liquids with a long alkyl chain are the best catalysts. Finally, under optimization of the reaction, the methyl ester of oleic acid (6 hours) yielded up to 95.0%. In addition, the esterification reactions of various alcohols containing fatty acids have been studied, and it has been shown that the catalytic activity is affected by the saturation rate of the fatty acid, which is a long alkyl chain, and that the catalyst is the best.

Taking into account that ionic liquids are more relevant catalysts in the reactions carried out, we carried out our syntheses in that direction.

EXPERIMENTAL PART

In the present study, unsaturated esters of carbonic acids were synthesized in the presence of N-methylpyrrolidone hydrosulfate, an ionic liquid catalyst.

IR-spectrum of samples have been registered on Fourier transform infrared spectroscopy LUMOS (firm BRUKER Germany) 600-4000 sm^{-1}within wave frequency.

Some physical-chemical indices of complex compounds, received on base of Baku natural oil acids have been determined. Crystallization temperature have been determined by standard – 5066-91, index of refraction n_D^{20} on refractometer IRF-22 N700060, relative density (d_4^{20}) by standard 3900-2000.

For the synthesis of allyl esters of alicyclic carbonic acids (cyclohexane, methylcyclohexane, biticloheptene) 75 g (1.2 mol) of allyl alcohol, 156 g (1 mol) of cyclohexane carbonic acid, 4.68 g (3% acid) of ionic liquid (IL) -N-methylpyrrolidone hydrosulfate and 100-150 ml of benzene were added into a three-necked flask. The mixture was heated to 80-90 ° C, the reaction was continued for 2- 2.5 hours and the acid number was 0.6 mg KOH / g. The end of the reaction is determined by the acid number. At the end of the reaction, the reaction product was filtered, purified from the catalyst, separated from the solvent at atmospheric pressure, neutralized with 2% KOH solution of alkali, expelled in a vacuum and separated into fractions. The properties of the obtained ether were studied, physical and chemical parameters were determined. Physicochemical parameters of alicyclic carbonic acids are given in the table, their yield of allyl esters is 85-90%. Synthesis of ethers was carried out according to the following scheme.

Some physical and chemical parameters of the synthesized ethers are given in the table 1.

The following absorption bands were observed in the IR spectral analysis of allyl esters of individual alicyclic carbonic acids: 2924, 1455 sm^{-1} for C-H bond in CH_2 group, 2862, 1375 sm^{-1} for C-H bond in CH_3 group, 988, 926 for C = C bond in α-position. sm^{-1}, 1239, 1162 sm^{-1} for C = C communication in CH_2 group, 1736 sm^{-1} for C = O ether group, characteristic absorption bands for C = C communication were observed in the cycle.

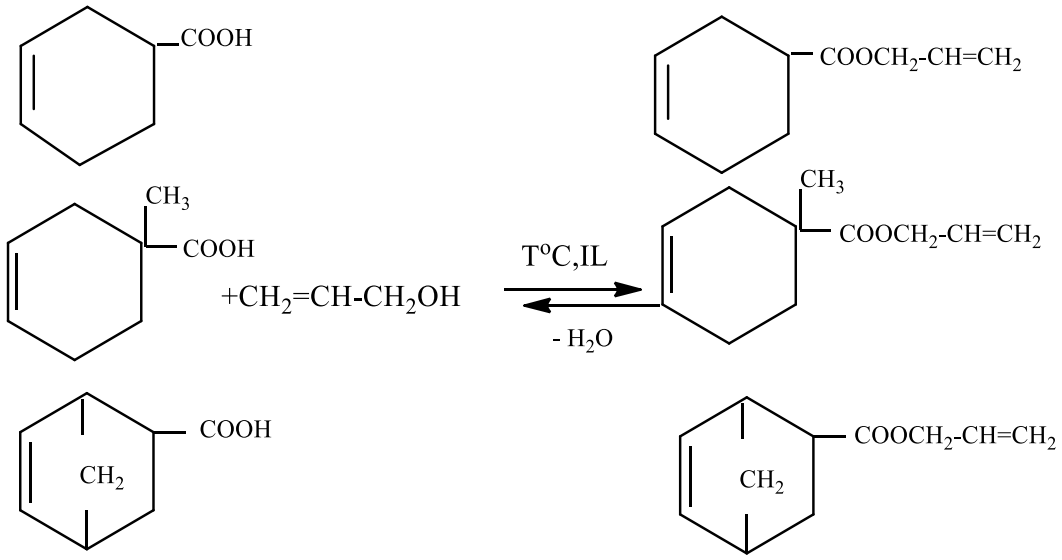

Table 1

Physicochemical properties of some alicyclic carbonic acids and esters

Name of the substance	Yield, wt%	$T_{boiling}$, ^0C mmHg	n_d^{20}	d_4^{20}
Allyl ester of cyclohexane carbonic acid	86	87/7	1,4753	0,9993
Allyl ester of methylcyclohexane carbonic acid	85	76/6	1,4648	0,9668
Bicycloheptene carbonic acid	73	86/2	1,4602	1,0131

In the NMR ^1H analysis of allyl esters of individual alicyclic carbonic acids signals have been observed for protons of the methylene group of the cycle and the side chain of the cycle, respectively at δ 1,5-3.50 (m) ppm. and δ4.00-4.35 (m) ppm, for the protons of vinyl C = CH_2 group at δ 4.70-4.90 (c) ppm, for protons of double bond in the cycle (2H, CH = CH-) at δ5.95-6.10 (m).

RESULTS AND DISCUSSION

Although the applications of alicyclic carboxylic acids, including unsaturated esters of cyclichexene and bicyclichehtene, are relatively little studied. It should be noted that unsaturated esters of alicyclic carbonic acids are used in polymerization and copolymerization reactions as monomers, corrosion inhibitors, modifiers and plasticizers in polymeric materials.

Cyclichexene, methyl cyclohexene, bicycloheptene carbonic acids and allyl alcohol were used as primary raw materials. Physicochemical parameters of primary raw materials are given in the table 2.

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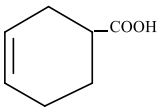
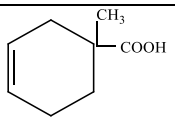
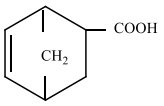
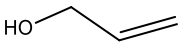
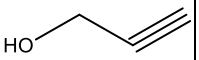
Table 2

Physicochemical parameters of primary raw materials

Parameters					
$T_{boiling}$.^{0}C	132,8/20	72,8/12	77/20	129/6,65·10^{-4} MPa	149-150/6,65·10^{-4} MPa
n_D^{20}	1,4814	1,4818	1,4843	1.4132	1.4315
d_4^{20}	1,0814	1,0119	1,19171	0.7580	0.9700

N-methylpyrrolidone hydrosulfate, an ionic liquid catalyst for the synthesis of esters, was synthesized according to a known methodology[15]. 20 g (220 mol) of N-methylpyrrolidone hydrosulfate is poured into a 250 ml flask fitted counter-refrigerator and a thermometer, sulfuric acid is added drop-by-drop in stoichiometric amount at a temperature of 0 ° C for 1 hour and stirred continuously for 24 hours. The resulting crystal catalyst is filtered, washed to remove residues that do not react with ethyl acetate, and dried at 70 ° C for 3 hours under a vacuum of 2 mm Hg. Electrical conductivity at a temperature of 30^{0}C is 16.62 S/m. The acid number of the catalyst is 548 mg KOH / g.

IR spectrum of N-methylpyrrolidone hydrosulfate (KBr, sm^{-1}): 3600–3300, 1660 (C = O), 1509, 1302, 1114, 962, 610.

In the NMR ^{1}H-spectral analysis of N-methylpyrrolidone hydrosulfate (300 MHz, D_2O), 6 methylene groups were found to be multiplet 1.83-1.91 m.h. (m., 2H, CH2) in the interval, in the form of a triplet 2.15 m.d. (t, 2H, N-CH_2) and 3.25 m.d. (t., 2H, CO-CH_2) in the interval, the methylene group together with the pyrrolidone ring singlet 2.65 m.d. (c, 3H, CH_3-N) oscillations are observed. HSO_3 and NH acid groups are observed in singlet form 9.78 m.h.

CONCLUSION

In conclusion, we can explain that the N-methylpyrrolidone hydrosulfate catalyst, which is an ionic liquid catalyst, is practical and environmentally friendly, non-toxic, and can be separated after the reaction and used repeatedly. Also, due to the short reaction time, the resin does not react during the reaction and the ethers are obtained at a high yield. Since the compounds containing unsaturated bonds have a high reactivity, their synthesis has aroused our interest and research has continued.

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СИНТЕЗ НЕНАСЫЩЕННЫХ ЭФИРОВ АЛИЦИКЛИЧЕСКИХ КАРБОНОВЫХ КИСЛОТ В ПРИСУТСТВИИ ИОННО-ЖИДКОСТНОГО КАТАЛИЗАТОРА

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Проведены реакции этерификации индивидуальных алициклических карбоновых кислот – (циклогексеновой, метилциклогексеновой и бициклогептеновой кислот) с аллиловым спиртом в присутствии ионной жидкости N-метилпирролидона гидросульфата в качестве катализатора, в результате чего получены сложные эфиры с высокими выходами (85-90%). Структура полученных эфиров подтверждена современными аналитическими методами и определены их физико-химические параметры. Одним из преимуществ ионной-жидкости катализатора является тот факт, что он экологически безвреден. Из-за короткой продолжительности реакции с этим катализатором, во время реакции не образуются дополнительные осмоляющиеся продукты, и катализатор можно использовать многократно. Синтезированные сложные эфиры представляют собой высокореакционные соединения из-за их ненасыщенных связей, которые представляют большой интерес в синтезе органических и нефтехимических продуктов.
Ключевые слова: *циклогексен-, бициклогептен, метилциклогексен карбоновые кислоты, ионная жидкость, алллиловый спирт, N-метилпирролидон гидросульфата*

İON MAYESİ KATALİZATORU İŞTİRAKINDA FƏRDİ ALİTSİKLİK KARBON TURŞULARININ DOYMAMIŞ EFİRLƏRİNİN SİNTEZİ

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Fərdi alitsiklik -metiltsikliheksen, tsikloheksen və bitsiklohepten karbon turşuları ion mayesi katalizatoru olan N-metilpirrolidonhidrosulfat iştirakında allil spirti ilə efirləşmə reaksiyası aparılmış və yüksək çıxımla (85-90%) efirlər sintez edilmişdir. Alınan efirlərin quruluşları müasir analiz üsulları ilə təsdiq edilmiş və fiziki-kimyəvi göstəriciləri müəyyən edilmişdir. İon mayesi katalizatoru ekoloji cəhətdən zərərsiz olduğu üçün, eləcə də reaksiya daha tez getdiyi üçün əlavə qətranlaşma məhsulları əmələ gəlmir. Sintez edilmiş efirlər tərkibində doymamış rabitə saxladıqları üçün yüksək reaksiyaya qabiliyyətli birləşmələr olub, neftkimya sintezində böyük maraq doğurur.
Açar sözlər: *tsikliheksen-. bitsiklohepten- metil tsikloheksen karbon turşuları, ion mayesi, allil, spirti, , N-metilpirrolidon hidrosulfat.*

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PHYSICO-CHEMICAL AND STRUCTURAL PROPERTIES OF ZEOLITE-CONTAINING ISOMERIZATION CATALYSTS

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In this research work, the physicochemical and phase-structural properties of zeolite-containing catalysts used as catalysts in the process of isomerization of normal paraffinic hydrocarbons were studied. For the purpose of comparative analysis, in all studies, the properties of both the initial support and the catalysts we synthesized were studied. Thus, the physicochemical and structural properties of various zeolite-containing nickel catalysts utilized within the isomerization of the pentane-hexane fraction were investigated using IR spectroscopic and X-ray methods, and the particular surface zone of the catalysts was studied using thermal desorption of nitrogen. Based on the studies carried out, the stage and basic composition of the catalysts was determined. It was found that, in comparison with the initial supports taken for synthesis, the synthesized zeolite-containing nickel catalysts exhibit high activity and selectivity in the process of isomerization of the pentane-hexane fraction.
Keywords: *pentane-hexane fraction, nickel based catalyst, isomerization, IR-spectroscopy, X-ray analysis, specific surface.*

INTRODUCTION

In the global production of motor gasolines, there is a steady drift towards toughening not only their performance, but also environmental characteristics. At the same time, international and residential standards for motor gasoline significantly limit the substance of benzene, sulfur, unsaturated hydrocarbons, general aromatics, oxygen-containing compounds. The control of such parameters as saturated vapor pressure and fractional composition is expanding. These factors have a significant impact on the production strategies and composition of motor fuels.

Modern requirements increase the demand for a highly efficient technology for the isomerization of light gasoline fractions boiling up to 70 °C and consisting of C_5-C_6 paraffins of predominantly linear structure (n-paraffins), due to their ability to reduce the concentration of benzene in gasoline while maintaining or increasing the octane characteristics of commercial gasoline. The purpose of catalytic isomerization processes in modern oil refining is to obtain high-octane isocomponents of motor gasolines or petrochemical raw materials. In the catalytic isomerization process, the molecular structure of n-paraffins is modified into their isomers (iso-paraffins) with a higher octane number, which are great components of motor gasolines [1-2].

The process is carried out using bifunctional catalysts with dehydrogenating and acidic exercises, which are a composition of a metal component, mainly platinum, and a supported promoter. Al_2O_3, aluminosilicates, zirconium oxide stabilized with aluminum oxide are used as a carrier [3-4].

The point of this work is to determine the relationship between the catalytic and physicochemical properties of zeolite-containing nickel catalysts in the process of isomerization of the pentane-hexane fraction.

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EXPERIMENTAL PART

To carry out the isomerization of the pentane-hexane fraction, a zeolite-containing nickel catalyst was synthesized by impregnation methods [5]. The activity of the synthesized catalysts was examined in a laboratory flow setup. To study the activity, 5 ml of catalyst, grain measure 1-2 mm, was loaded into the reactor. Analysis of liquid and gas products of the process was carried out by chromatographic method [6-7].

In order to reveal the relationship between the catalytic and physicochemical properties of the catalysts, we used analysis methods such as X-ray and IR spectroscopic [8-9]. IR spectra were recorded on a "Nicolet-IS10" IR-Fourier spectrophotometer ("Thermo scientific" firm) in the range 400-4000 sm^{-1}. X-ray studies of catalysts of various compositions were carried out on an automatic powder diffractometer "D2 Phaser" from "Bruker" (Germany) with CuK_{α}-radiation and a nickel filter. The specific surface area of the catalysts was determined by the method of thermal desorption of nitrogen in a helium stream in a special installation [10]. The test samples are placed in a stream of a mixture of nitrogen and helium and after that cooled to the temperature of liquid nitrogen. When the samples are heated to room temperature, nitrogen desorption occurs from the catalyst surface, which is recorded in the form of a desorption peak on the tape of a self-recording device. The peak area (S_{des}) depends on the amount of desorbed nitrogen, the latter, in turn, is directly proportional to the surface of the sample under study.

RESULTS AND DISCUSSION

In recent years, IR spectroscopy has taken an important place in the study of heterogeneous catalytic reactions. One of the main areas of application of IR spectroscopy is the study of surface properties, as well as the study of the mechanism of catalytic reactions. For the purpose of a comparative study, in this work, we took the IR spectra of the OMNICAT catalyst utilized as a support, its H-form, and the active H-OMNICAT/ $Ni(NO_3)_2$ catalyst synthesized by the impregnation method (fig.1).

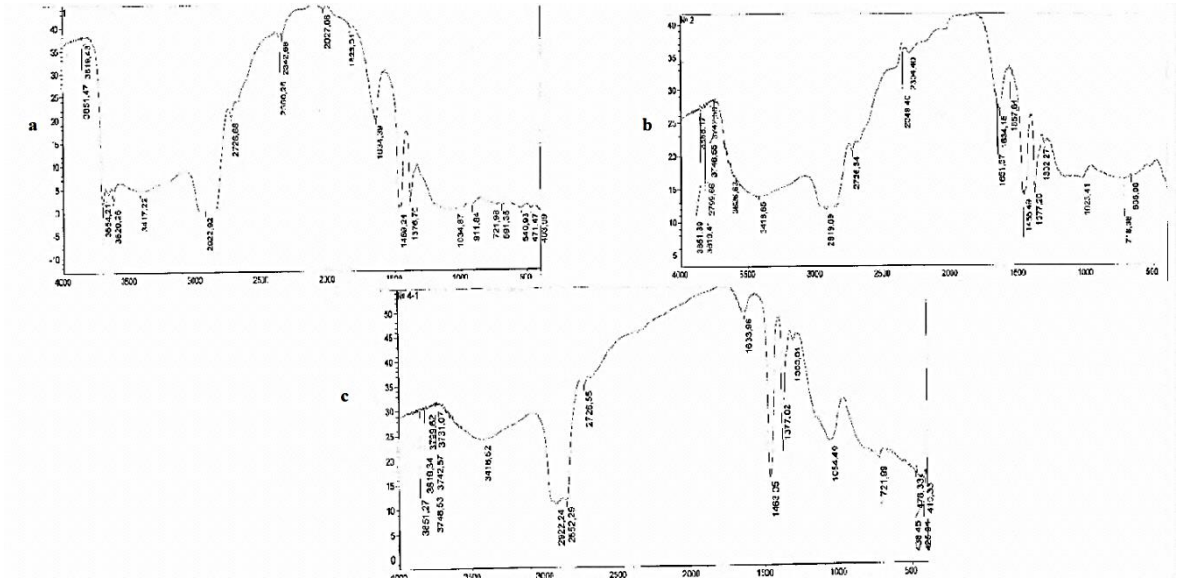

Fig. 1.IR spectrum of various zeolite-containing catalysts:
a – OMNICAT 210P catalyst; b – H-OMNICAT; c – H-OMNICAT/$Ni(NO_3)_2$

As can be seen from the figure (fig. 1, a), the absorption bands in the range 3700-3500 sm^{-1} refer to the stretching vibration of the OH group. Absorption bands at 3417, 1634 and 691 sm^{-1} indicate the presence of water in the sample. Since the samples are dissolved in vaseline oil, absorption bands at 2922, 1459 and 721 sm^{-1} are reasonable for vaseline oil. As is known, the OMNICAT catalyst contains aluminosilicate. Therefore, very intense absorption bands in the range of 1100-900 sm^{-1} were found for them. In addition, absorption bands of the Si - O valence bond are visible in the region of 500-300 sm^{-1}.

As mentioned above, for the preparation of H-OMNICAT, the commercial OMNICAT catalyst was treated with an NH_4NO_3 solution. Consequently, the characteristic absorption bands found in the range of 1380-1350 sm^{-1} indicate the presence of nitrates (fig.1,b). Since the process of isomerization of the pentane-hexane fraction occurs in a hydrogen atmosphere, some of these nitro compounds are converted to amines upon interaction with hydrogen. Thus, absorption bands in the region of 1650-1590 sm^{-1} indicate the presence of single amines, and bands in the region of 1650-1550 sm^{-1} indicate the presence of double amines. Absorption bands (1100-900 sm^{-1}) characteristic of silicates were also found in the catalyst sample.

The H-OMNICAT/$Ni(NO_3)_2$ sample synthesized by the absorption method is considered to be a more active catalyst, therefore the IR spectrum of this sample was taken. It can be seen from the figure (fig.1,c) that the absorption bands in the 1380-1350 sm^{-1} and 1300-1255 sm^{-1} regions refer to nitro compounds (R – NO_2). In the high-frequency region of the spectra, a wide band at 3200-3500 sm^{-1} is also obvious, which, according to the literature, refers to the stretching vibrations of hydroxyl (OH group). As can be seen from the figure, the initial samples contain absorption bands in the range of 480-410 sm^{-1}, which indicates the presence of nickel compounds. So, at ~ 475 sm^{-1} NiO, 471 sm^{-1} NiO, and at ~ 420 sm^{-1} $NiCl_2$ were found. After catalysis, these bands were not found (not shown in the figure). This is due to the fact that the isomerization process is carried out in a hydrogen atmosphere. Therefore, nickel oxides react with hydrogen and are reduced to metallic nickel.

For a comparative analysis of the phase composition, an X-ray analysis of the initial OMNICAT 210P catalyst, its hydrogen form H-OMNICAT, and the active H-OMNICAT/$Ni(NO_3)_2$ catalyst synthesized by the impregnation method was also carried out (fig. 2).

It can be seen from the figure (fig.2,a) that the initial OMNICAT 210P catalyst consists of the sodium-aluminosilicate form Y, a small amount of lithium aluminosilicate, and kaolinite clay. Then the original form was transferred to the hydrogen form. For this, the introductory OMNICAT 210P was treated with NH_4NO_3 solution, then the isolated sodium was washed out with distilled water, dried at 120-150 °C and calcined at 500 °C for 4 hours [5].

The X-ray diffraction pattern of H-OMNICAT (fig.2,b) contains phases of Na-aluminosilicate of form Y and a small amount of lithium-aluminosilicate. The absence of a phase of kaolinite clay is clarified by the fact that as a result of repeated processing and washing, this phase is completely removed from the catalyst composition. It is also seen from the figure (fig.2,c) that the synthesized catalyst consists of an aluminosilicate phase and nickel oxide. The presence of nickel oxide on the surface was also confirmed by the IR spectroscopic method. In the course of X-ray diffraction analysis, we calculated the degree of crystallinity of all formed phases using the DIFFRAC.EVA program table 1).

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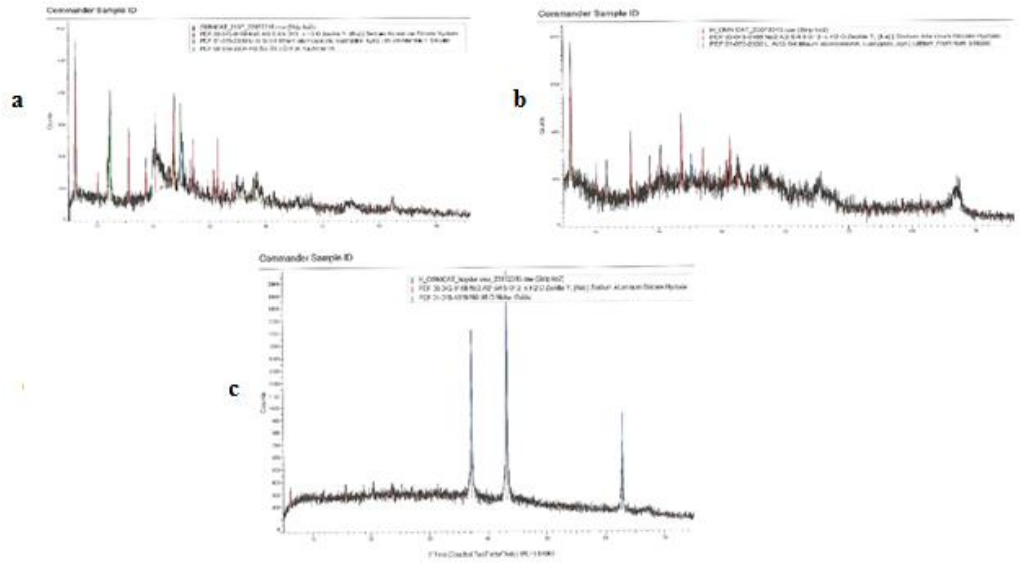

Fig. 2. X-ray analysis of zeolite-containing catalysts:
a – OMNICAT 210P catalyst; b – H-OMNICAT; c – H-OMNICAT/$Ni(NO_3)_2$

Table 1

Crystallinity of zeolite-containing nickel catalysts

Catalyst	***Crystallinity, %***
OMNICAT 210P	59,9
H-OMNICAT	45,3
H-OMNICAT/$Ni(NO_3)_2$	33,1

The table shows that, in comparison with other catalysts, the initial OMNIKAT 210P has the highest degree of crystallinity. A gradual decrease in the degree of crystallinity is due to the fact that, as a result of processing processes, the phase compositions of the samples are weakened.

The specific surface area of the catalysts is calculated by the following formula (table 2):

$$S_{sp} = \frac{S_{des} \cdot 4{,}35}{S_d^0 \cdot n}$$

where: S_{sp} – specific surface area of the catalyst, m^2/g;
S_{des} – desorption peak area, mm^2;
S_d^0 – desorption peak area corresponding to 1 m^2 of surface, mm^2;
n – amount of catalyst, g.

Table 2

Specific surface area of zeolite-containing catalysts

Catalyst	***Specific surface, m^2/g***
OMNICAT 210P	69,2
H-OMNICAT	49,4
H-OMNICAT/$Ni(NO_3)_2$	81,6

As can be seen from the table, the specific surface area of the OMNICAT catalyst taken as a carrier is 69.2 m^2/g. When the carrier catalyst is converted to the H-form, its specific surface area decreases to a certain extent (to 49.4 m^2/g). The specific surface area of the zeolite-containing nickel catalyst synthesized for isomerization of the pentane-hexane fraction is the highest (81.6 m^2/g).

Thus, studies show that the specific surface area of catalysts, both taken as a carrier and synthesized, affects their isomerization activity. That is, with an increase in the specific surface area of the catalyst, its isomerization activity increases.

CONCLUSION

Thus, based on the physical and chemical studies carried out, the following conclusions can be drawn:

1. On the basis of IR spectroscopic studies, it was determined that the synthesized catalysts contain absorption bands related to nickel compounds, especially nickel oxide. After catalysis, the absence of these bands is explained by the fact that by interacting with hydrogen it is reduced to a metallic form;
2. Using X-ray studies, the phase composition and the degree of crystallinity of the catalysts were determined. It is shown that the catalysts synthesized by the impregnation method have the highest degree of crystallinity in comparison with the initial samples;
3. On the basis of research, it was determined that the specific surface area of a zeolite-containing nickel catalyst synthesized by the impregnation method is the highest.

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ФИЗИКО-ХИМИЧЕСКИЕ И СТРУКТУРНЫЕ СВОЙСТВА ЦЕОЛИТСОДЕРЖАЩИХ КАТАЛИЗАТОРОВ ИЗОМЕРИЗАЦИИ

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В данной исследовательской работе изучались физико-химические и фазово-структурные свойства цеолитсодержащих катализаторов, используемых в качестве катализаторов в процессе изомеризации парафиновых углеводородов нормального строения. С целью сравнительного анализа во всех исследованиях изучались свойства как исходного носителя, так и синтезированных нами катализаторов. Так, исследованы физико-химические и структурные свойства различных цеолитсодержащих никелевых катализаторов, используемых в процессе изомеризации пентан-гексановой фракции, с помощью ИК-спектроскопического и рентгено-графического методов, а удельная поверхность катализаторов изучена с помощью тепловой десорбции азота. На основании проведенных исследований определен фазовый и структурный состав катализаторов. Установлено, что по сравнению с исходными носителями, взятыми для синтеза, синтезированные цеолитсодержащие никелевые катализаторы проявляют высокую активность и селективность в процессе изомеризации пентан-гексановой фракции.

Ключевые слова: *пентан-гексановая фракция, никельсодержащий катализатор, изомеризация, ИК-спектроскопия, рентгеноструктурный анализ, удельный поверхность.*

SEOLİTTƏRKİBLİ İZOMERLƏŞMƏ KATALİZATORLARININ FİZİKİ-KİMYƏVİ VƏ STRUKTUR XASSƏLƏRİ

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Mövcud tədqiqat işində normal quruluşlu parafin karbohidrogenlərinin izomerləşmə prosesində katalizator kimi istifadə olunan seolittərkibli katalizatorların fiziki-kimyəvi və faza-struktur xassələri tədqiq edilmişdir. Müqayisəli analiz aparılması məqsədilə, bütün tədqiqatlarda həm ilkin daşıyıcı, həm də tərəfimizdən sintez edilmiş katalizatorların xassələri tədqiq edilmişdir. Belə ki, İQ-spektroksopiya və rentgen üsullarının köməyi ilə pentan-heksan fraksiyasının izomerləşmə prosesində istifadə olunan müxtəlif seolittərkibli nikel katalizatorlarının fiziki-kimyəvi və struktur xassələri tədqiq edilmiş, katalizatorların xüsusi səthi isə azotun istilik deorbsiyası üsulunun köməyilə öyrənilmişdir. Aparılmış tədqiqatlar əsasında katalizatorların

faza və struktur tərkibi müəyyən edilmişdir. Aşkar edilmişdir ki, sintez üçün götürülmüş ilkin daşıyıcılar ilə müqayisədə, sintez edilmiş seolittərkibli nikel katalizatorlar pentan-heksan fraksiyasının izomerləşməsi prosesində yüksək aktivlik və selektivlik nümayiş etdirir.
Açar sözlər: *pentan-heksan fraksiyası, nikeltərkibli katalizator, izomerləşmə, İQ-spektroskopiya, rentgen struktur analizi, xüsusi səth.*

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INFLUENCE OF TEMPERATURE AND CONTACT TIME ON THE PROCESS OF CONVERTING ETHANOL TO ACETONE

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In recent years, scientists have been paying more and more attention to the process of obtaining various organic compounds from ethanol, which is produced in large quantities by processed plant raw materials. One of the valuable monomers, which can be obtained from alcohol, is acetone. The process of obtaining acetone from ethanol was carried out using catalysts of various compositions Catalysts were prepared by the method of coprecipitation. In the course of studying the activity of catalysts, it was found that catalysts based on zinc are most active in the reaction of converting ethanol to acetone. Among them, the highest activity in the process is shown by the catalyst composition of the ZnO:CaO=9:1. So the research was conducted on this catalyst. Many different parameters affect the reaction of converting ethanol to acetone. Some of them are parameters such as temperature and contact time. Research has been carried out in the range of different temperatures and in the range of different contact times. Investigations were carried out to identify the optimal process conditions, during which optimal conditions were established for the maximum acetone yield on an active catalyst in the reaction of ethanol to acetone conversion.

Keywords: *temperature, contact time, catalyst, ethanol, acetone.*

INTRODUCTION

Studies of the influence of technological parameters in catalytic processes are of great practical importance. So, studying the influence of technological parameters, it is possible to identify the optimal conditions for the process, as well as some kinetic laws of the process [4].All studies on the effect of temperature and contact time were carried out on a catalyst – ZnO: CaO = 9: 1.

Ethanol conversions on zinc-calcium catalyst depend on various factors. The conversion of ethanol to acetone is influenced by temperature, contact time, partial pressure of oxygen, water vapor, and ethanol [2]. Since compaction products are formed on the catalyst surface during the conversion of ethanol, their influence on the conversion of ethanol to acetone cannot be ruled out. This work discusses the effect of temperature and contact time.

EXPERIMENTAL PART

The studies were carried on the combined laboratory installation and a flow-through installation with integral reactor. The analysis was carried on the chromatograph LHM-8M [5].

The temperature dependence of the ethanol conversion on the ZnO:CaO catalyst was studied in the range of 300-550°C. Figure 1 shows the influence of temperature on the conversion of ethanol. As you can see, the conversion of ethanol to acetone is observed at a temperature of 300°C and is 20%. At this temperature, in addition to acetone, acetaldehyde and also a small amount of CO_2 and ethylene are formed. An increase in the temperature of the process leads to the yield of acetone, ethylene and

carbon oxide, while the yield of acetaldehyde decreases. The maximum acetone yield, equal to 86.5%, is observed at a temperature of 425°C. Carbon dioxide, ethylene, propylene and other hydrocarbons are formed as by-products [1].

As the temperature rises above 425°C, the acetone yield decreases and at a temperature of 525°C it is 28%. It should be noted that at this temperature, the ethylene yield increases sharply and amounts to 41%. The decrease in acetone is probably due to the conversion of ethanol to by-products. The acetaldehyde yield, as can be seen, at a temperature of 425°C first decreases to 2%, and then begins to increase again, and at a temperature of 500°C it reaches 13.8%. The yield of carbon dioxide in the entire studied temperature range increases.

The influence of the contact time during the catalytic conversion of ethanol was investigated in the range of 0 ÷ 5 sec. Special experiments have established that there are no diffusion complications at a catalyst grain size of 1–3.0 mm. The studies were carried out at temperatures of 400, 425 and 450°C, and partial pressures of ethanol and water vapor of 10 kPa and 40 kPa, respectively [3].

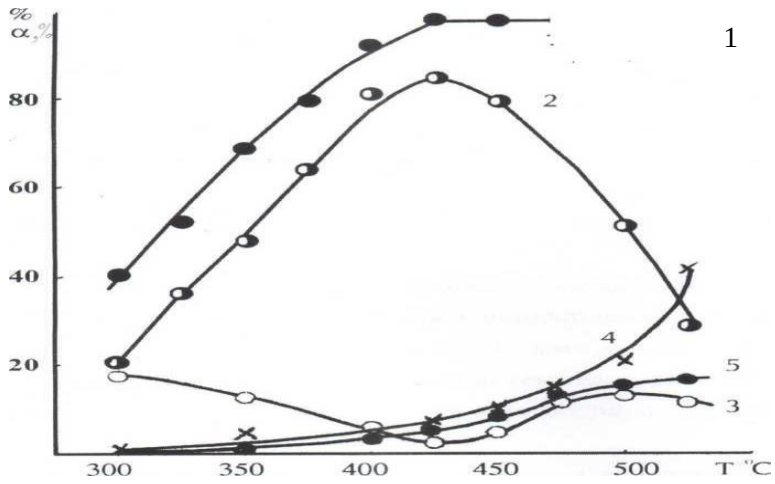

Fig.1. Influence of temperature on the reaction of ethanol conversion on ZnO:CaO = 9: 1 catalyst.

1- ethanol conversion; 2 - acetone; 3 - acetaldehyde;
4 - ethylene; 5 - CO_2

The contact time was changed by adjusting the amount of catalyst. Figure 2 shows the effect of the contact time on the conversion rate of ethanol to acetone, acetaldehyde, CO_2 and the conversion of ethanol. As can be seen from fig.2 at 400°C and τ = 0.5 sec. is 19% with an ethanol conversion of 30%. In this case, acetaldehyde, CO_2 and gaseous hydrocarbons are formed as by-products. With an increase in contact time from 0.5 sec. up to 1.5 sec. the ethanol conversion is doubled and amounts to 60%. The acetone yield is 50%. Acetaldehyde and carbon dioxide are formed as by-products. The total yield of by-products is no more than 10%. With contact time τ = 1.5 sec. and a temperature of 400 ° C, the selectivity for acetone is 83.3%. At 400°C, with an increase in the contact time to 3 sec., the ethanol conversion increases to 97% and the acetone yield is 85.4%. With a further increase in the contact time to 4 sec., the conversion of ethanol increases, while the yield of acetone decreases. The selectivity for acetone is 73.6%. With an increase in contact time up to 5 sec. the yield

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of by-products increases and at this contact time the yield of acetone is 60% with an ethanol conversion of 96%, and the total yield of by-products reaches 36%.

RESULTS AND DISCUSSION

As can be seen from fig. 2, at a temperature of 425 °C, the maximum acetone yield is observed at a contact time of 3 sec. and is 83%. At 425 °C (τ = 0.5 sec.), the acetone yield is 22% with an ethanol conversion of 38%. In this case, the acetone selectivity is 58%. The minimum yield of by-products is observed at a contact time of 3 sec. and a temperature of 425 °C.

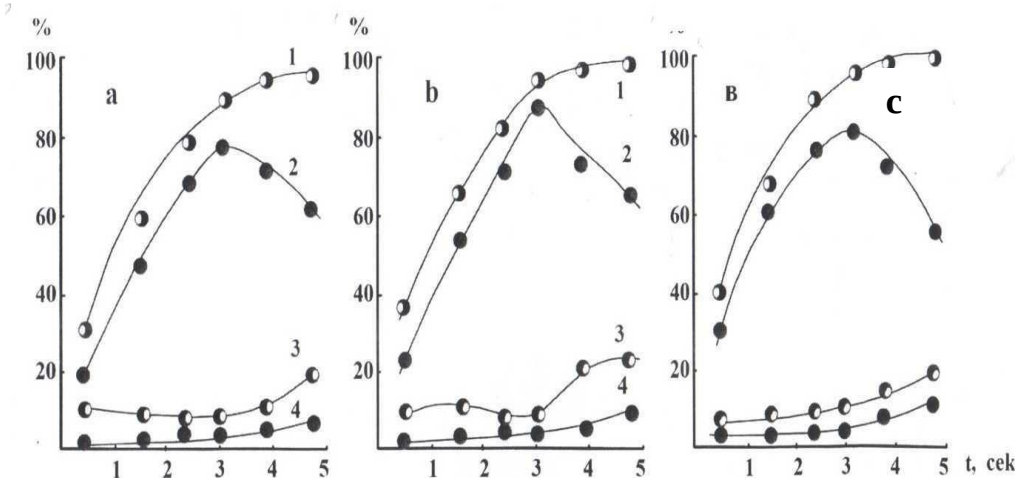

Fig. 2. Influence of contact time on the transformation process ethanol at 400 °C (a), 425 °C (b) and 450 °C (c).
1- conversion of ethanol; 2- acetone; 3- acetaldehyde; 4 - CO_2 (C_2H_4).

At 450 °C and contact time 0.5 sec. the acetone yield is 27% at 40% ethanol conversion. In contrast to the previously considered temperatures, at 450 °C and a contact time of 1.5 sec. the yield of acetone reaches 60%, while at 400 and 425 °C it is 45 and 53%, respectively. The maximum acetone yield is achieved at a contact time of 3 sec. (as at 400 and 425 °C). A further increase in the contact time leads to a sharp decrease in the yield of acetone, and the conversion of ethanol and the yield of by-products increase.

Studies have shown that in addition to the formation of by-products, compaction products are also formed. The formation of compaction products does not exceed 0.6%.

Thus, examining the contact time at various temperatures, it was found that the optimal conditions for the yield of acetone are: T = 425 ° C, τ = 3 sec.

Under these conditions, the acetone yield is 86.5% at 98.9% ethanol conversion. Acetaldehyde, ethylene and carbon dioxide are formed as by-products.

CONCLUSION

The influence of technological parameters, contact time and temperature, which influence the process of ethanol conversion into acetone, is investigated.

The optimal conditions for the maximum yield of acetone have been established.

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ВЛИЯНИЕ ТЕМПЕРАТУРЫ И ВРЕМЕНИ КОНТАКТА НА ПРОЦЕСС ПРЕВРАЩЕНИЯ ЭТАНОЛА В АЦЕТОН

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В последние годы всё большее внимание учёных обращается к процессам получения различных органических соединений из этилового спирта, который вырабатывается в больших количествах переработкой растительного сырья. Одним из ценных мономеров, который можно получать из этилового спирта является ацетон. Процесс получения ацетона из этанола проводили при помощи катализаторов различного состава. Катализаторы готовились методом соосаждения. В ходе исследования активности катализаторов установлено что наибольшую активность в реакции превращения этанола в ацетон проявляют катализаторы на основе цинка. Среди них наиболее высокую активность в процессе проявляет катализатор состава ZnO:CaO=9:1.Поэтому исследования велись на этом катализаторе. На реакцию превращения этанола в ацетон влияет много различных параметров. Одними из них являются такие параметры как температура и время контакта . Проводились исследования в области различных температур и в области разного времени контакта. Проведены исследования по выявлению оптимальных условий процесса, в ходе которых были установлены оптимальные условия для максима-льного выхода ацетона на активном катализаторе в реакции превращения этанола в ацетон.
***Ключевые слова**: температура, время контакта, этанол, ацетон, катализатор.*

ETANOLUN ASETON ÇEVRİLMƏSİ PROSESİNƏ TEMPERATUR VƏ KONTAKT VAXTININ TƏSİRİ

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Son illərdə alimlərin getdikcə daha çox diqqəti etil spirtindən müxtəlif üzvi birləşmələrinin alınması prosseslərinə yönəlir. Son zamanlar etil spirti bitki xammaldan emal edərək, çox miqdarda istehsal olunur. Etanoldan alına bilən qiymətli monomerlərdən biri də asetondur. Etanoldan asetonun alınması prossesi müxtəlif tərkibli katalizatorların üzərində aparılmışdı. Katalizatorlar çöküntü şərtləndirmə üsulu ilə hazırlanmışdır. Katalizatorların fəaliyyətinin öyrənilməsi zamanı, etanolun asetona çevrilməsi reaksiyasında ən çox fəaliyyətini sink tərkibli katalizatorlar göstərmişdi. Ən yüksək fəaliyyəti ZnO:CaO=9:1 tərkibli katalizator göstərmişdir. Ona görə bütün tədqiqatlar bu katalizatorun üzərində aparılmışdı. Etanolun asetona çevrilməsi reaksiyasına bir çox müxtəlif parametrlər də təsir edir. Bu parametrlərdən biri temperatur və kontakt vaxtıdır. Tədqiqatlar müxtəlif temperatur aralığında və müxtəlif kontakt vaxtı aralığında aparılmışdı. Etanolun asetona çevrilmə reaksiyasında, ən aktiv katalizatorun üzərində asetonun maksimal çıxışı üçün optimal şəraiti müəyyənləşdirmək üçün tədqiqatlar aparılmışdır.
***Açar sözlər**: temperatur, kontakt vaxtı, etanol, aseton, katalizator.*

UDC:665.753.502

OBTAINING OF ECOLOGICALLY PURE DIESEL FUEL BY USING N-METHYLPYRROLIDONE UNDER THE INFLUENCE OF MAGNETIC FIELD

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The purification process of polycyclic aromatic hydrocarbons was carried out of diesel fraction from the primary oil refining to get the ecologically clean diesel fuel which meets newest standarts. The extraction process is performed under influence of magnetic field by using N-methylpyrrolidone as an extractant. The process is carried out in different durations, temperatures, ratios without use of magnetic field effect to identify the optimal parameters. Then the obtained best result exposed to different inductions magnetic field effect to enhance further removal of aromatic hydrocarbons and sulfuric compounds. The content of aromatic hydrocarbons in the diesel fraction decreased by 45% under normal conditions and by 67% under the influence of magnetic field after extraction. The obtained diesel fuel content the 6% of polycyclic aromatic hydrocarbons and it conforms the Euro-5 diesel fuel standard for polycyclic aromatic hydrocarbon content (less than 8%).
Keywords: *diesel, extraction method, magnetic field, N-methylpyrrolidone.*

INTRODUCTION

The intensification of chemical-technological processes is one of the important tasks of science and technology. The basis for increasing the productivity of equipment and reducing energy consumption for carrying out chemical-technological processes can be the creation and implementation of efficient technological devices with a low specific energy consumption and material consumption, a high degree of impact on the processed substances. Such developments are based on irinpipially new engineering solutions, theoretical and experimental studies of physicochemical processes in processed media under intense impulse influences [1].

Traditional technologies for the preparation and processing of hydrocarbon raw materials do not meet the achievement of the expected indicators, and special processes with the use of new technological and structural solutions require large capital investments and a huge amount of time, therefore, at present, more and more attention is paid to unconventional methods of hydrocarbon raw materials activation: acoustic, mechanical , electrical, thermal, radiation, magnetic [2].

In spite of with fairly wide practical application of the methods of magnetic treatment of liquids, there is currently no generally accepted theory that explains all aspects of the effect of a magnetic field on hydrocarbon dispersed systems. In this regard, it is relevant to study the magnetic processing of liquids as a method of preparing hydrocarbon raw materials for further processing, analysis of the development and improvement of technical means for magnetic processing and assessment of the feasibility and prospects of its further development.

The author [2] found that the use of the method of dehydration of oil-water emulsions with preliminary treatment in a magnetic field of a dissolved demulsifier increases the degree of dehydration of the oil-water emulsion by 5-30%, and with the

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same degree of dehydration it reduces its consumption by 1.3-2 times compared to traditional technologies.

Preliminary magnetic treatment of the residues of atmospheric distillation of gas condensate and oil leads to an increase in the selection of distillate fractions up to 6% vol., And with equal selection, the process temperature decreases by 5-25 ° C, which significantly reduces energy consumption for the vacuum distillation process [3].

In the process of visbreaking of hydrocarbon residues under the influence of a magnetic field, the yield of light products increases by 4-8% of the mass, and at the same time, coke formation decreases by 1.2-2.3 times, which leads to an increase in the overhaul life of visbreaking installations.

A strong constant magnetic field significantly improves the properties of hydrocarbons. As numerous studies show, they really and significantly improve the quality of preparation of any fuel and increase the calorific value and completeness of its combustion. Many different magnetic designs are known, but the general point is that any design contains powerful magnets [4].

Apparatus for magnetic processing of liquids can be classified according to several criteria: by the method of obtaining a magnetic field, by the location of magnets or electromagnets, by performance, by the direction of the magnetic field vector in relation to the moving fluid flow, by the number of intersections of the magnetic field, etc.[5, 6].

Devices for magnetic processing in the literature are called differently: magnetiser, magnetic activators, magnetrons, magnetizers, devices for magnetic processing, etc.

To create a magnetic field, permanent magnets or electromagnets are used from separate sections in the form of windings and magnetic circuits. Permanent magnets are undoubtedly easier to use, because do not require power supply, are easily mounted in devices for magnetic processing, do not pose a threat of explosion in the presence of flammable gases, and have a low cost. Their disadvantages include the inability to quickly adjust the magnetic field strength, a decrease in induction during prolonged operation at high temperatures, vibrations, and shocks. Unlike permanent magnets, electromagnets make it possible to regulate the intensity of the magnetic field when changing the parameters of the supply current, depending on the characteristics of the flow and the fluid being processed [7].

As the results of experimental studies show, the use of magnetic processing makes it possible to significantly increase the efficiency of the processing of petroleum raw materials. It has been established that when a magnetic field is applied to hydrocarbon systems, the decisive role is played by the magnitude of the magnetic induction, the rate of intersection of the magnetic field in the active zone (zone with maximum induction) by the flow of the processed liquid, and the number of intersections of the magnetic field by the processed liquid.

The effectiveness of the effect of a magnetic field on various hydrocarbon systems are not the same. So, as the viscosity increases, it takes longer to achieve the greatest effect of exposure. When studying the effect of magnetic processing parameters on various indicators of hydrocarbon processing processes, it was found that the rate of crossing the active gap is tenths and hundredths of m / s.

According to the literature [8], the value of the magnetic induction usually varies in the range of 0.1-0.3 T. At lower values, the effect of the influence of the magnetic field is reduced, large values are economically unjustified both in the case of using

electromagnets (high power consumption), and in the case of permanent magnets (high cost of a magnet).

In the oil and gas processing industry, the main task of the selective purification process with N-methylpyrrolidone is to increase the qualitative and quantitative indicators of the target product of the process (raffinate) and reduce energy consumption. This is achieved due to engineering solutions that complicate the technological scheme of the process, or due to the introduction of additives into the raw materials, which, due to the insufficient degree of influence on the selectivity of the solvent in relation to aromatic hydrocarbons, resins, asphalt-resinous substances, which is expressed in a slight increase in the yield of raffinate without improving its quality [9].

It is of interest to use wave effects, which allow carrying out technological processes with greater efficiency, as well as creating compact technologies. However, the main process of the influence of the magnetic field on the gradation of the extraction cleaning process, in particular, on the selective cleaning process, remains unclear.

In this work, the purification process of polycyclic aromatic hydrocarbons was carried out by extraction of diesel fraction obtained from the primary oil refining under the influence of magnetic field.

EXPERIMENTAL PART

The extraction process was obtained by effectively mixing the diesel distillate with the N-methylpyrrolidone under normal conditions and under the influence of magnetic field using glass mixer in 3-necked flask with a circular base.

After extraction, 15 minutes were allowed to release the extract and raffinate. The extract and raffinate are released by separating funnel. A special device made of electric coils was used to create the magnetic field. The tube was placed between the magnetized plates during the experiments.

RESULTS AND DISCUSSION

The main purpose of this study is to extract polycyclic aromatic hydrocarbons from the diesel fraction obtained from the primary refining of oil by extraction using N-methylpyrrolidone under the influence of magnetic field. The N-methylpyrrolidone used in the process meets the standards MS-TSH-KOMP-2-207-10.

The extraction process is achieved by effective mixing of diesel distillate with N-methylpyrrolidone under normal conditions and under the influence of magnetic field using glass mixer in 3-necked circular base flask.

The quality indicators of the primary processing diesel fraction used in the experiments are given in table 1.

The experiments were performed in different durations, temperatures and ratios. The best result achieved at room temperature (20^{0C}) under normal conditions in the ratio of 1:1 (N-methylpyrrolidone: diesel distillate). Then was started to perform extraction process under the influence of magnetic field with different induction rates (0.05-0.5 mT). Maqnetic field was measured by using “Gaussmeter Teslameter WT10A”device.

The best result achieved under the 0.2 mT magnetic field effect.

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Table 1

Quality indicators of primary processing diesel fraction

S/S	Indicators	Values
1	Density, at 20°C	0.8450
2	Total amount of sulfur, % (mass)	0.0895
3	Kinematic viscosity, mm^2/s	6.2
4	Freezing temperature, °C	-36
5	Turbidity temperature, °C	-25
6	Tlash point, °C	72
7	Iodine number	1.83
8	Acidity	57.7
9	Aromatic hydrocarbons	18.08
10	Actual resin	3.4
11	Fractional composition, % (mass)	
	Begining of boiling, °C	222
	50% boils , °C	296
	96% boils , °C	357
	End of boiling, °C	367
12	Residue	3.8

CONCLUSION

As a result of the analysis of refined diesel fuel by sulfation and iodine number methods, the content of aromatic hydrocarbons in refined diesel distillate decreased from 18.08% to 10% after extraction.

However, as a result of extraction of primary processing diesel distillate under the influence of magnetic field at 0.2 T induction a further decrease in the content of aromatic hydrocarbons is observed by 6% under the same conditions. It meets the Euro-5 diesel fuel standard for aromatic hydrocarbon content. The results are given in table 2.

Table 2

The amount of aromatic hydrocarbons after extraction in primary processing diesel distillate

Used extractant	Amount of aromatic hydrocarbons, %		Aromatization rate
	Normal condition	Magnetic field	
N-methylpyrrolidone	10	6	45/67*

* The result was obtained under the influence of magnetic field

According to table 1, the amount of aromatic hydrocarbons in the raw material is 18.08%. So, the extraction process with N-methylpyrrolidone under normal conditions showed 45% decrease in the amount of aromatic hydrocarbons and 67% decrease as a result of the extraction process using magnetic field.

Experiments have shown that the magnetic field influence the further increases the efficiency of the extraction process when using N-methylpyrrolidone as an extractant, and the extraction process is carried out at normal room temperature and atmospheric pressure.

The purification process of aromatic hydrocarbons of the diesel fraction by extraction using magnetic field will be possible at atmospheric pressure and ambient temperature, which will further simplify the process.

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ПОЛУЧЕНИЕ ЭКОЛОГИЧЕСКИ ЧИСТОГО ДИЗЕЛЬНОГО ТОПЛИВА ПРИ ИСПОЛЬЗОВАНИИ N-МЕТИЛПИРРОЛИДОНА ПОД ВОЗДЕЙСТВИЕМ МАГНИТНОГО ПОЛЯ

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Осуществлен процесс очистки от полициклических ароматических углеводородов дизельной фракции первичной переработки нефти с целью получения экологически чистого дизельного топлива, соответствующего новейшим стандартам. Процесс экстракции осуществляется под действием магнитного поля с использованием N-метилпирролидона в качестве экстрагента. Процесс осуществляется при различной

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продолжительности, температуре, соотношении без использования эффекта магнитного поля для определения оптимальных параметров. Затем полученный лучший результат подвергается воздействию магнитного поля различной индукции для усиления дальнейшего удаления ароматических углеводородов и серных соединений. Содержание ароматических углеводородов в дизельной фракции снизилось на 45% при нормальных условиях и на 67% под действием магнитного поля после экстракции. Полученное дизельное топливо содержит 6% полициклических ароматических углеводородов и соответствует стандарту дизельного топлива Евро-5 по содержанию полициклических ароматических углеводородов (менее 8%).
Ключевые слова: *дизельное топливо, метод экстракции, магнитное поле, N-метилпирролидон.*

MAQNİT SAHƏSİNİN TƏSİRİ ALTINDA N-METİLPİRROLİDONDAN İSTİFADƏ ETMƏKLƏ EKOLOJİ TƏMİZ DİZEL YANACAĞININ ALINMASI

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Yeni standartlara cavab verən ekoloji təmiz dizel yanacağının alınması üçün neftin ilkin emalından alınan dizel fraksiyasının tərkibindən politsiklik aromatik karbohidrogenlərin təmizlənməsi prosesi həyata keçirilmişdir. Ekstraksiya prosesi ekstragent kimi N-metilpirrolidondandan istifadə etməklə maqnit sahəsinin təsiri altında yerinə yetirilmişdir. Optimal parametrlərin müəyyən olunması üçün proses maqnit sahəsinin təsirindən istifadə olunmadan müxtəlif müddətlərdə, temperaturda və nisbətdə aparılmışdır. Alınan ən yaxşı nəticədə aromatik karbohidrogenlərin və kükürdlü birləşmələrin təmizlənməsini daha da artırmaq üçün müxtəlif induksiyalı maqnit sahəsinin təsirinə məruz qoyulur. Ekstraksiyadan sonra dizel fraksiyasında aromatik karbohidrogenlərin miqdarı normal şəraitdə 45%, maqnit sahəsinin təsiri altında 67% azalmışdır. Əldə olunmuş dizel yanacağının tərkibində 6% politsiklik aromatik karbohidrogenlər var və bu politsiklik aromatik karbohidrogenlərin miqdarına görə EURO-5 dizel yanacağı standartına cavab verir (8%-dən az).
Açar sözlər: *dizel yanacağı, ekstraksiya metodu, maqnit sahəsi, N-metilpirrolidon.*

UDC:621.318.2;541.128.13

CATALYTIC ACTIVITY OF COPPER FERRITE SYNTHESIZED WITH THE USING OF MICROWAVE TREATMENT IN THE OXIDATION REACTION OF CARBON MONOXIDE

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The catalytic activity of copper ferrite samples obtained by the "sol-gel with combustion" and "ceramic" methods from oxides in the oxidation of carbon monoxide to carbon dioxide has been investigated. Microwave radiation was used to intensifythe formation of a catalytically active component. It was shown that microwave treatment of the gel during the preparation of the catalyst noticeably affects the catalytic activity. It was found that copper ferrite samples prepared by the sol-gel method with combustion both in the traditional way and in a microwave oven are the most active in CO oxidation. On these samples, the complete conversion of CO into CO_2 occurs at a temperature 200-250°C, while the samples with additional afterburning in a microwave oven and obtained from oxides by solid-phase synthesis in a microwave field completely convert CO at temperatures 250-325°C. It is shown that the use of microwave treatment in the synthesis of ferrites by the sol-gel method with combustion makes it possible to ensure the uniformity of the chemical and phase composition, reduce energy consumption and shorten the duration of the preparation process.

Keywords: *copper ferrite, sol-gel method, microwave radiation, oxidation, solid-phase synthesis, catalyst, specific surface area*

INTRODUCTION

The problem of utilization of carbon monoxide, which makes a significant contribution to environmental pollution, is relevant to this day. For the oxidation of carbon monoxide into dioxide, mainly manganese, copper-chromium and platinum group metals containing catalysts are used [1]. Publi- cations in recent years point to the promising use of ferrite catalysts in the oxidation of carbon monoxide to its dioxide [2, 3]. Ferrites are obtained by the method of co-precipitation of metal salts or hydroxides with subsequent thermal decomposition and calcination of the resulting oxides, using the "ceramic technology" from the oxides of their constituents, etc. In all methods of obtaining ferrites, the formation of its structure occurs at a high temperature during sintering, which is a solid-phase process and requires prolonged heat treatment. Therefore, the search for new methods for the synthesis of ferrites with a developed surface and a texture acceptable for catalytic purposes continues to be relevant. From this point of view, the synthesis of ferrite nanoparticles by the sol-gel technology with combustion (solution or gel) is one of the most promising methods for obtaining nanomaterials and allows the synthesis of complex oxide systems, including ferrite powders of various chemical compositions [4,5]. In addition, recently, various types of irradiation, in particular microwave radiation, have been used to obtain catalysts and stimulate chemical reactions [6,7]. Microwave treatment has a number of advantages over conventional traditional heating methods, such as a uniform temperature distribution in the volume of the heated object, which is extremely important for heat treatment of catalysts, a high heating rate and low inertia.

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This work presents the results of the synthesis of copper ferrite from copper and iron oxides using ceramic and sol-gel technologies with combustion, studies of the phase composition, magnetic and optical spectra, texture and catalytic properties.

EXPERIMENTAL PART

The salts $Fe(NO_3)_3 \cdot 9H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$ and citric acid were used as precursors for the synthesis of ferrite by the sol-gel method with combustion. Combustion was carried out both by the traditional method - heating in an oven and in a microwave oven. Aqueous solutions of the calculated amounts of salts and organic reagent were stirred on a magnetic stirrer with heating for 1 hour. Then it was heated in a drying oven. A fire occurred at a temperature of 130-150 °C. Part of the resulting powder was additionally subjected to further heat treatment in an EM-G5593V microwave oven (Panasonic) with a resonator volume of 25 liters while varying the magnetron power 300-800 W with an operating frequency of 2450 MHz. Another series of experiments with drying a paste-like gel and igniting it was carried out in a microwave oven. To synthesize ferrite using ceramic technology, copper oxide CuO and magnetite Fe_3O_4 taken in stoichiometric ratios for an hour were homogenized by grinding in a porcelain mortar with ethyl alcohol until completely dry. Then the resulting mixture was placed in a quartz glass and subjected to microwave treatment.

X-ray phase analysis of the products was carried out on a Phaser D2 automatic diffractometer (Bruker). The measurement of the specific surface area of the samples was determined by low-temperature nitrogen adsorption by the multipoint BET method, the total pore volume by the BJH method on a SORBI-MS device (ZAO META, Russia). The texture indicators of copper ferrite samples with a ratio of Cu: Fe = 1: 2 and 2: 1, obtained by different methods, are shown in table 1. The obtained ferrite powders in an amount of 1 gram were mixed with a binder - alumogel, molded into granules, dried in air, further heat treatment was carried out in a drying cabinet and a muffle furnace. CO oxidation was carried out by the flow method at a CO:air ratio = 1: (3-5), a space velocity of 6000-12000 h^{-1}. The analysis was carried out on an LXM chromatograph, in two columns with CaA and poropak Q sorbents.

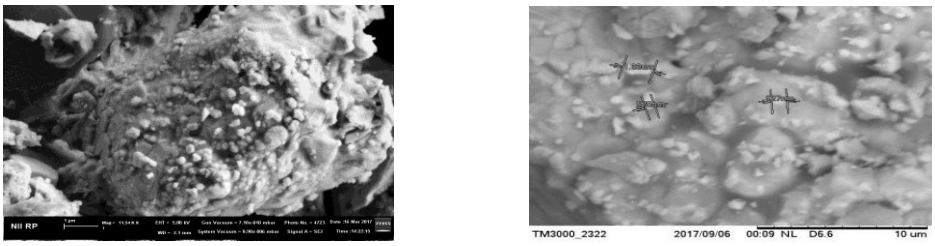
Table 1

Influence of the method of preparation of copper ferrite on its texture parameters

Synthesis method	Specific surface area, m^2 / g		Pore volume, cm^3 / g	
	Cu:Fe= 1:2	Cu:Fe= 2:1	Cu:Fe= 1:2	Cu:Fe= 2:1
1. "Ceramic method" of copper and iron oxides in a microwave field	0.4	0.6	-	-
2.Sol-gel method with combustion	18	15.4	0.09	0.083
3. Sol-gel method with combustion and additional microwave treatment	1.0	1.3	- 0.02	- 0.01
4. Sol-gel with burning in a microwave oven	8.0	5.5		

RESULTS AND DISCUSSION

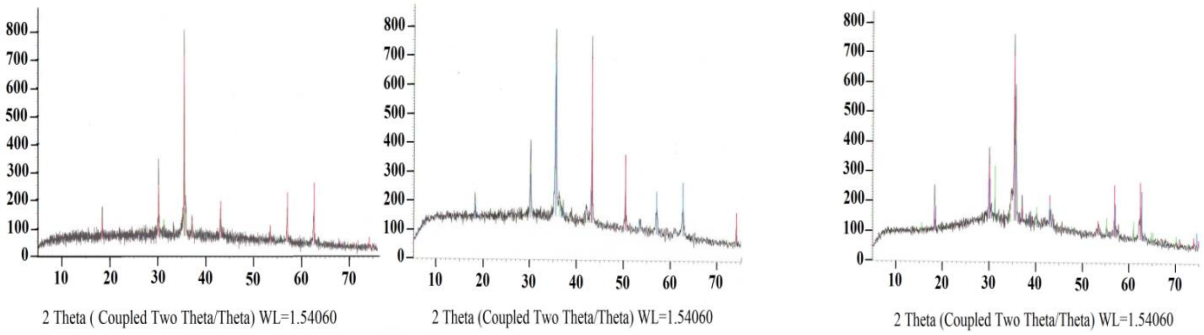
In fig. 1 a, b shows electron micrographs of copper ferrite samples synthesized by two different methods. The micrographs show that in both cases, ferrite particles are enlarged aggregates with a particle size from hundreds of nanometers to tens of micrometers, however, the sample obtained by the sol-gel method with afterburning in a microwave particles.



a) b)

Fig. 1. Micrographs of copper ferrite samples obtained by microwave synthesis from oxides (a) and by the sol-gel method with combustion and afterburning in the microwave field (b)

X-ray phase analysis showed that copper ferrite $CuFe_2O_4$ is formed both as a result of the solid-phase reaction of copper (II) oxide with magnetite and during the synthesis using the sol-gel technology fiq. 2). When copper ferrite is obtained by the sol – gel method, copper ferrite is formed already at the combustion stage. Along with it, hematite also forms, as indicated by the XRD data. The use of microwave heat treatment significantly accelerates the formation of copper ferrite. In solid-phase synthesis, ferrite formation with intermediate grinding of the mixture of samples takes only 8-10 minutes at a magnetron power of 800 W. Note that the use of microwave action on heterogeneous catalysts during their preparation makes it possible to obtain catalyst samples with a more uniform distribution of particles, accelerate the preparation of catalyst samples, and obtain a catalyst with a given dispersion. In the case of catalysts consisting of several phases, replacing the traditional heating with microwave heating can promote the preferred formation of any phase.



a) b) c)

Fig. 2. Diffraction patterns of copper ferrite obtained by microwave solid-phase synthesis from copper oxide and magnetite (a), sol-gel with combustion (b) and subsequent microwave treatment (c)

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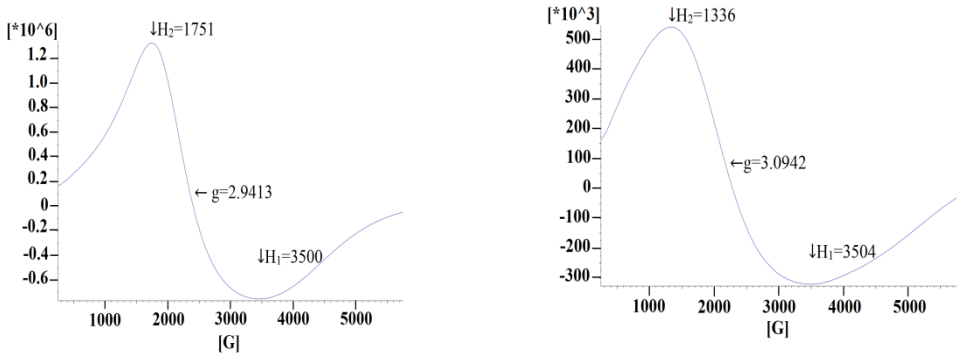
Figure 3 shows the EPR spectra of copper ferrite obtained by the sol-gel technology with combustion without and microwave treatment. The initial oxides - copper (II) oxide CuO, magnetite Fe3O4, as well as copper ferrite formed as a result of synthesis, are magnetic materials with a wide range of magnetic properties (from ferro (ferri) magnetism to superparamagnetism (depending on the particle size) and antiferromagnetism). As can be seen from Fig. 2, the intensity, width (ΔH = 2170 and 1740), and g-factors (g = 3.094 and 2.941) of the observed signals significantly depend on the method of sample



a) b)

Fig. 3. EPR spectra of copper ferrite, obtained by sol-gel technology with combustion without (a) and with microwave treatment (b)

As can be seen from the spectra presented, very intense, broad, and strongly asymmetric absorption lines are recorded. Similar electron magnetic resonance signals were recorded for a fairly large number of iron-containing samples with diamagnetic matrices. Since the samples under study contain at least two magnetic phases, the recorded spectra were considered to consist of two components. However, the curves plotted theoretically with allowance for two magnetic phases were unsatisfactory.

The IR spectrum in the range from 400 to 1100 cm^{-1} contains absorption bands (436.07; 473.61; 546.86; 692.87; 951.16 and 457.20; 548.78; 838.90; 890, 21; 1117.61cm^{-1}) due to lattice vibrations of M – O and M – OH bonds [8].

The catalytic activity of copper ferrite prepared by the above methods with the ratio Cu-Fe = 1: 2 and Cu-Fe = 2: 1 was investigated in the oxidation of carbon monoxide to dioxide. The temperature dependences of CO conversion for these copper ferrite samples are shown in fig. 4.

The results of CO oxidation on copper ferrite show that in the sol-gel method for producing ferrites, further thermal treatment of the gel (combustion, microwave treatment of the resulting powder after burning the gel and microwave treatment of the gel) affects the catalytic activity.

Figure 4 shows that all copper ferrite samples synthesized by different methods completely convert CO into CO_2. The difference in temperature at which complete CO conversion occurs. In CO oxidation, the most active copper ferrite samples prepared by the sol-gel method with combustion both in the traditional way and in a microwave oven. On these samples, the complete conversion of CO into CO_2 occurs at a temperature of 200-250°C, while the samples with additional afterburning in a microwave oven and synthesized from oxides by solid-phase synthesis in a microwave field completely convert CO at a temperature of 250-325°C. Comparison of the specific

surface area of the samples (table 1) shows that the samples obtained from oxides by solid-phase microwave synthesis have a very small specific surface area; additional microwave treatment of the copper ferrite powder obtained by the sol-gel method also leads to a decrease in the specific surface area in comparison with the samples obtained by combustion.gel, i.e. the influence of the sample preparation method on their textural parameters and catalytic activity is clearly manifested. A similar picture is observed with additional heat treatment of the ferrite powder obtained by the sol-gel method in a microwave oven.

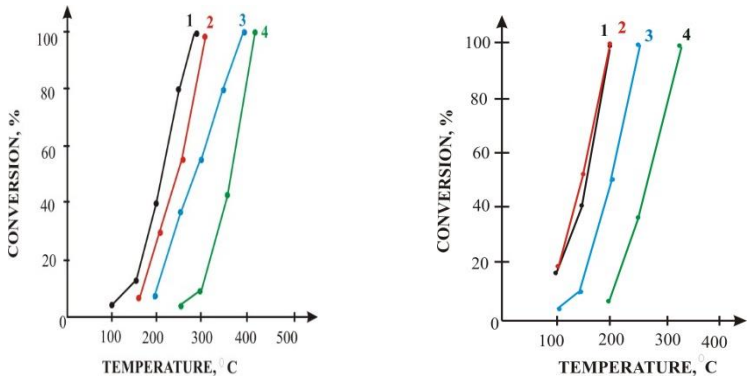


Fig. 4. Dependence of CO conversion on temperature for copper ferrite samples obtained: 1 - sol-gel method with combustion; 2 - sol-gel with combustion in a microwave oven; 3 -sol-gel method with combustion and afterburning in a microwave oven; 4-solid-phase synthesis from copper oxide and magnetite in a microwave oven: a) Cu: Fe = 1: 2, b) Cu: Fe = 2: 1. CO:air= 1: 3 (mol.), v.s. 10000 h^{-1}

Full conversion of CO on samples obtained by burning the gel in an oven and microwave oven is achieved already at a temperature of 200^0C, which is 100^0C lower than on samples with afterburning and solid-phase synthesis in a microwave oven. However, it takes much less time to ignite the gel in a microwave field due to the bulk absorption of microwave radiation and transform it into heat than with conventional heating, which makes this method more preferable.

The influence of the ratio of copper and iron in ferrite on the catalytic activity of the synthesized samples was also studied. At hyperstoichiometric copper content (Cu: Fe = 2: 1), the sample is more active in the CO conversion, which can probably be associated with a high content of the oxide phase in this sample. According to the X-ray diffraction pattern, along with ferrite during sol-gel synthesis with combustion, copper(II) oxide is also formed and its partial reduction to Cu_2O also occurs. Therefore, experiments were carried out with individual copper oxide, also obtained by the sol-gel technology. However, no increase in catalytic activity was found for the synthesized samples with a high content of the oxide phase of copper. So, if on copper oxide at 250 °C the conversion is 81%, on a copper ferrite sample already at a temperature 200°C the conversion reaches 100%.

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CONCLUSION

Summarizing the above, we can conclude that microwave radiation can be used to obtain active ferrite catalysts for the oxidation of CO to CO_2. It has been shown that microwave radiation provides the formation of catalytically active components in shorter periods of time. This method of obtaining catalytically active samples of copper ferrite can be considered more acceptable from the point of view of saving energy and time. Particular emphasis is placed on the creation of catalysts for the low-temperature conversion of carbon monoxide to dioxide. The above methods for the synthesis of copper ferrite using microwave energy can be successfully used for the preparation of catalysts for the utilization of vehicle exhaust gases and industrial emissions.

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КАТАЛИТИЧЕСКАЯ АКТИВНОСТЬ ФЕРРИТА МЕДИ, СИНТЕЗИРОВАННОГО С ПРИМЕНЕНИЕМ МИКРОВОЛНОВОЙ ОБРАБОТКИ, В РЕАКЦИИ ОКИСЛЕНИЯ МОНООКСИДА УГЛЕРОДА

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Исследована каталитическая активность образцов феррита меди, полученных «золь-гель с горением» и «керамическим» методом из оксидов, в реакции окислении монооксида углерода в диоксид углерода. Микроволновое излучение использовалось для

интенсификации образования каталитически активного компонента. Было показано, что микроволновая обработка геля при приготовлении катализатора заметно влияет на каталитическую активность. Установлено, что образцы феррита меди, приготовленные золь-гель методом с сжиганием как традиционным способом, так и в микроволновой печи, наиболее активны в окислении CO. На этих образцах полное превращение CO в CO_2 происходит при температуре 200-250°C, тогда как образцы с дополнительным дожиганием в микроволновой печи и полученные из оксидов твердофазным синтезом в микроволновом поле, полностью превращают CO при температурах 250-325 °C. Показано, что использование микроволновой обработки при синтезе ферритов золь-гель методом с сжиганием позволяет обеспечить однородность химического и фазового состава, снизить энергозатраты и сократить продолжительность процесса приготовления.
Ключевые слова: *феррит меди, золь-гель метод, микроволновое излучение, окисление, твердофазный синтез, катализатор, удельная поверхность.*

KARBON MONOOKSİDİN OKSİDLƏŞMƏ REAKSİYASINDA MİKRODALĞALI İŞLƏNMƏ İLƏ SİNTEZ OLUNMUŞ MİS FERRİT KATALİZATORUNUN AKTİVLİYİ

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Dəm qazının karbon dioksidə oksidləşməsində zol-gel yanma və "keramika" üsulu ilə oksidlərdən alınan mis ferrit nümunələrinin katalitik aktivliyi tədqiq edilmişdir. Katalizatorun hazırlanması zamanı gelin mikrodalğalı işlənməsi nümunənin katalitik aktivliyə əhəmiyyətli dərəcədə təsir etdiyi göstərilmişdir. Həm ənənəvi üsulla, həm də mikrodalğalı sobada zol-gel yanma üsulu ilə hazırlanmış mis ferrit nümunələrinin CO-nun oksidləşməsində aktiv olduğu müəyyən edilmişdir. Bu nümunələrdə CO-nun CO_2-yə tam çevrilməsi 200-250 ° C temperaturda baş verir, mikrodalğalı sobada əlavə yanma və oksidlərdən mikrodalğalı sahədə bərkfazalı sintezlə alınan nümunələr isə CO-nu 250-325°C temperaturda CO_2-yə çevirir. Ferritlərin zol-gel yanma ilə sintezində mikrodalğalı işlənmənin istifadəsi kimyəvi və faza tərkibinin bircinsliyini təmin etməyə və hazırlıq prosesinin müddətini qısaltmağa imkan verdiyi göstərilmişdir.
Açar sözlər: *mis ferrit, zol-gel metodu, mikrodalğalı emal, oksidləşmə, bərkfazalı sintez, katalizator, xüsusi səth.*

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OIL IN THE LIGHT OF PHILOSOPHIC CONSIDERATIONS AND ITS REFLECTION IN THE AZERBAIJANI ART

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The article considers the phenomenology of oil through the segments of the philosophy of culture, which by their origin and essence are closely related to the episteme of the world perception. Azerbaijani oil is a special page for our country, its historical past, modernity and future. In the XXI century, the idea of harmonious co-evolution of nature and society is gaining more and more popularity. The harmonious combination of natural resources and cultural values of the country is the philosophy of modern Azerbaijan, which is building the mechanism for distributing the natural resources for the purpose of social security of the population. Such strategy makes possible to shape a secure future for the nation. After all, oil is not only fuel, but also energy, life, the generator of the wheel of history, a kind of engine of progress in industrial society. Oil not only provides the daily life, but is also a starting point for understanding what our society will be like in the future.
The article considers the phenomenology of oil in the aspect of Azerbaijani art. The works of art of Azerbaijani authors testify to the heroes-workers of the oil industry, whose generalized images inspired many outstanding composers, artists, sculptors to create the works of deep philosophical sound. The artistic factor, to some extent present in them, contributes to a deeper phenomenological approach when reflecting the oil theme. Thanks to spiritual carriers, such as music, fine arts, cinematography, the images and names of the first well drillers and creators of oil fields have forever remained in the history of Azerbaijan.
Keywords: *the phenomenology of oil, Azerbaijani oil, oil in musical reflection, oil in the cinematography, oil in the visual arts.*

INTRODUCTION

Topicality of the research topic is that, oil itself has a huge impact on the life of the society. Oil becomes the object of consciousness, more and more penetrating into human thinking. That is its phenomenon. Oil is becoming the independent substance for study. For Azerbaijan, as the oil-producing country, oil is one of its symbols, and therefore its phenomenon is projected onto other strata of life, including culture and art.

The novelty of the problem «Phenomenology of oil and its features in the art of Azerbaijan», is as follows:

- the phenomenology of oil is presented as the primary direction not only in the national framework, but also in the spatial survey of the post – soviet states;
- for the first time, the phenomenon of oil was considered in the aspect of Azerbaijani art;
- for the first time, there was made the attempt to generalize the artistic character on the basis of works of various types of Azerbaijani art.

EXPERİMENTAL PART

Historical, logical, the method of systems analysis, as well as the methods of generalization, analysis and synthesis.

RESULTS AND DİSCUSSİON

The phenomenology, being one of the directions of modern Western philosophy, is presented through the scientific developments of such scientists as E. Husserl, M. Heidegger, E. Levinas, M. Merleau-Ponty and others. The distinctive feature of phenomenology is that, it is of a methodological nature, in other words, in fact, phenomenology is not just one of the philosophical trends, but it is the very methodology itself that was proposed by E. Husserl and M. Heidegger. Thanks to the works of these philosophers, phenomenology, as a whole, poured into a worldwide movement, and such a substance as oil turned into an object of comprehensive, dialectical study, which laid the foundation of science. Husserl was the first, who presented a methodically reflected view of the naturalistic theory of knowledge, which served as the impetus for the transcendental turn, which included the Descartes and Kantian curve at once, having in mind that, the meaning of transcendental reduction consists of two components: negative and positive. If the first hinders the transition of the main epistemological problem from the philosophical (phenomenological) dimension to the scientific one, then the second keeps the philosophical dimension of the problem in its purity open [1]. Moreover, Husserl analyzed the stages of the complex, polyphonic, creative process of transcending in art [2]. His student Heidegger went further, he refused the language of intellect, consciousness, experience and, etc. However, at the same time, he considered the phenomenology with new openness, new receptivity, and the sense of unity with the world [3]. Levinas expanded the concepts of living embodiment and more refined technique for suspending the conceptualization to reveal the experiences as they arise [4]. Sartre introduced an ethical dimension to what has traditionally been an epistemological project [5]**.** Merleau-Ponty made the important contributions to the philosophy of art, history, management of natural resources and politics, which played an important role in the spreading of phenomenology [6]**.**

Thanks to the works of the mentioned scientists in the philosophical science included such direction as the phenomenology of oil. On the one hand, oil is the welfare, but on the other hand, it harms the environment, which stipulates the search for its alternative. The nationwide leader of the Azerbaijani people Heydar Aliyev, starting the project «Contract of the Century» (2004 year), understood that it was the necessary stage in the economic breakthrough of Azerbaijan.

"Pumped out of nature and became, as a part, endowed with great importance for the world economy, geopolitics, social relations and political institutions, oil is acquiring its autonomous agency (if not to say “subjectivity”). Turning into a fetishized object of human desire, extracted with the help of technological operations, oil has a counter effect on a person. It would seem, that being the product of an active invasion of technology into the passive matter of nature, oil is capable of overturning subject-object relations, depriving a person of critical rationality and turning him into a hostage of his own passion / desire. The concept of "resource curse" despite of its metamorphism reflects well an unconscious supposition about the true relationship between oil and human beings"[7]. Here is another characteristic of oil: "Oil awakens the extraordinary emotions and passions, as oil is above all a colossal temptation. This is a promise of easy and crazy money, wealth and strength, happiness and power"[7].

In connection with the phenomenology of oil in a philosophical sense, the formulation of this problem itself, it should be better to highlight some sources that gave impetus to the understanding of oil. First of all, this is the book by Daniel Yergin “Oil

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output. The world history of the struggle for oil, money and power" (1990) for which he received the Pulitzer Prize [7]. The translation of the English title of the book «Prize» as output into Russian reveals the vision of oil as a phenomenon in the best possible way. The word "output", as we know, has two meanings in Russian. One of them is extraction, pumping out from the depths, and the other meaning is welfare, property, prize, reward, in other words, its ultimate outcome is the acquisition of power. The double-natured name of the Russian translation «output» very well reflects the essence of oil. In particular, D. Yergin made oil the subject of independent study and gave it the «status» of a phenomenon.

The phenomenology of oil in a philosophical sense has, based on stated in his book, three main aspects:

1) the progressive development of the capitalist formation and entrepreneurial activity; the dominance of the company «Standard Oil» in oil industry of the USA; the expansion from handcrafted oil production to technological progress in the local and the international economy of the XX century.

Throughout the history, contracts have been concluded between individual oil industrialists, large corporations and the countries.

However, by that time, oil had already won public recognition and became the most important source of energy. In the XX century it radically changed the life.

2) oil is associated with politics and power. This was confirmed by the First and Second World Wars. Certainly, Hitler was interested in the oil of Caucasus. Nevertheless, due to the oil advantage the USA, Germany and Japan ended in a fiasco.

During the Cold War, the USSR and the USA fought for oil domination. The Suez crisis in the 50s was also driven by oil. In the 70s, the struggle for oil became a global problem. Oil has also become «a bone of contention» and the cause of the war in the Persian Gulf.

However, oil continues to maintain its high commodity position; it is the decisive point in the national strategies and the international policies.

3) transformation of the world into a «hydrocarbonic society». Today people are so dependent on oil that they do not fully realize its enormous impact. Nevertheless, someday, perhaps in the near future they will have to take a decisive step towards switching to alternative energies, because, the «black gold» is losing its value [8,].

The phenomenology of oil receives the independent study and disclosure of the problem at the conference in St. Petersburg, in 2018, which was namely held under the name «Phenomenology of oil». At this forum, researchers from different countries (Russia, Italy, Germany, Serbia), the representatives of the world of art - artists, sculptors, as well as philosophers, writers, philologists and political scientists took part in an interdisciplinary discussion of oil. The phenomenon of oil was presented in the aspect of philosophical analysis through the prism of the humanities, and the reflection of oil in art was also considered. At the conference was emphasized that, oil has become an object of worship for various social groups, and religious confessions [9]. It should be mentioned that, the topic of this article to a certain extent was formed under the influence of familiarization with the materials of this conference, because for Azerbaijan as an oil-producing country, one of the symbols is oil.

It should be noted that, in the XX century, the demand for oil determined the supply, respectively, the growing dependence on it was noted positively and symbolized the progress. In the context of the struggle for state independence, the Republic of Azerbaijan made a qualitative breakthrough to such extent that it was called the

economic miracle. Here, of course, oil played the main role. The oil reserves of the Absheron Peninsula in the world dimension are one of the richest and most ancient. The ancient Greek historian Plutarch testified that the soldiers of Alexander the Great used oil brought from Absheron for lighting at night [10]. Oil was transported in wineskins or pottery vessels. Since then, the process of its extraction from the bowels of the earth and the Caspian Sea, as well as the transportation of raw materials, has changed significantly: the manual labor was replaced by modern mechanized installations; instead of pottery vessels and other containers, the pipelines were built to transport oil to the European countries.

In the aspect of the topic of the phenomenology of oil, it is impossible not to mention 2020 year. The pandemic, which touched precisely the human factor – the most vulnerable - struck down on all existing stereotypes, all hierarchies, it has significantly shaken the hegemony of oil, at least for the moment. There has been a big reassessment of values, but it is naturally impossible to realize this and see further prospects for how this will all develop; only time can show it. The Covid-19 pandemic, which has catastrophically collapsed global energy demand, extremely aggravated the already inexorably growing global overproduction of oil [11]. Most countries were not ready for such a sudden collapse and, not knowing what anti-crisis measures to take, suffered huge financial losses, while countries with developed economies, quickly navigating in the environment of pandemic continued along the path of decarbonization, in other words they refused of the fossil fuels. «The Stone Age ended not because of a shortage of stones, but simply it had to give the way to the new technologies» [11]. This associative parallel, even in an ironic form, depicts a very uncertain future of oil ...

The perception of Azerbaijan as the oil producer has become so deeply rooted in the world and in the mentality of the Azerbaijani people itself that it has integrated into the culture and become a part of life. Oil seemed to flow into the cultural space, turning into an independent substance, condensing in itself the historical energy, various ethical values and the ideological meanings. The artistic attributes of oil are present even in the design of a number of architectural structures in Baku. In particular, one of the striking examples is the building of the Central Bank of the Republic of Azerbaijan, which is located in the center of Baku, on one of the main streets – Rashid Behbudov street. Here, we can talk about the color layout, since it was built of golden glass and black alcapon, i.e. there are two colors here - black and gold, symbolizing the image of oil as «black gold» and, which is very important, this is the building of the Central Bank of Azerbaijan. It is also symbolic that behind the building is located the Azerbaijan State University of Oil and Industry – the talent foundry of the world's best oil specialists. Facing the building there is a sculpture with a raised hand of Heydar Aliyev, pointing the way forward to great achievements, to the harmony of nature and art. Another very interesting example, a more modern interpretation of the use of oil themes, can serve the installations with the participation of oil rigs, which are located in the park in front of the building of the new Palace of Water Sports. It really smells of fuel oil here and the pumps run into the well! It is impossible to imagine the Absheron landscape without oil derricks, which are spread like clusters along the entire coast… The whole history of oil began precisely from these rigs, from the time when there was so much oil that, the rigs were placed right on the ground and they pumped oil. And, therefore, in the 50s -60s, this Absheron landscape with an abundance of oil-derricks was very familiar and symbolic.

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Thanks to the spiritual media - music, painting, sculpture, etc., the images of the first well drillers, creators of oil fields have forever entered the history of Azerbaijan. The art community has dedicated and dedicate their works to them, the heroes of oil industry. During the silent film years, the famous photographer A. M. Michonne shot the newsreels «The Fire of the Oil Fountain at Bibi-Heybat» (1898) and «The Oil Fountain at Balakhany» (1898). The plot of these newsreels is about the hard everyday-life of oil workers, about sabotage at oil wells, about the heroic work of drillers and oil producers. The first demonstration of the «The Oil Fountain at Balakhany» took place on August 2, 1998. It is remarkable that, that based on this date, by the order of the President of the Republic of Azerbaijan Heydar Aliyev (December 18, 2000), August 2 was declared the Day of Azerbaijani Cinema. In 1916, the joint-stock company «Pirone» released the film based on the novel of the same name by the writer Ibragim bey Musabekov «The Kingdom of Oil and Millions». In the film is shown Baku at the beginning of the XX century, the life of Baku millionaires and the life of workers who earn their daily bread by hard labor in the oil fields. By the way, the shooting of this film was sponsored by oil industrialists [12]. In 1923, the documentary film “The Fire at the Surakhani Oil Fields” was shot. I would especially like to mention the documentaries "The Novel about the Caspian Oil Workers" (1953) and "Conquerors of the Sea" (1953). This film dilogy was directed by the famous filmmaker, one of the most famous documentary filmmakers of the XX century, Roman Carmen, which is about the life and extraordinary friendship of oil workers, about their heroic deed, every day "subduing" the sea weaves, producing oil in harsh conditions. R. Carmen is the author of such films as "The Great Patriotic War *," The Nuremberg Trials "and many others. As it is known, art as a form of reflection of reality carries the factor of artistry. The documentaries also carry the artistic message to one or another degree. In these films of Roman Carmen, the romantic feeling on the one hand (the romanticism of glorifying the labor of oil workers), and the maritime theme on the other hand, are very characteristic and they create the artistic mood of the films.

In 2015, on the occasion of the 70th Victory over Nazi Germany, with the support of the Heydar Aliyev Foundation, was shot the documentary film “Target is Baku. How Hitler lost the war”. The film was shot by the Baku Media Center and the famous French documentary film company “Clarke Costelle & Co” (CC & C). the project was directed by the producer, film director Arzu Aliyeva. The film states the fact that, during the war years, more than 70 percent of oil and 80 percent of gasoline consumed in the Soviet Union fell to the share of Azerbaijan [13]. At the same time, the company «Clarke Costelle & Co» (CC&C) released another film «Apocalypse: The Second World War», in which the battle for oil takes place as the main storyline.

The next type of art that gets involved in the orbit of “oil” influence is music. At first glance, it might seem that what could be the intersection points of seemingly incompatible things - oil and music? No, we are not talking about creating a piece of music like the famous "Bolero" by Ravel, which the composer, as it is known, wrote under the influence of a visit to a steel mill. Here, very interesting deep aspects of another plan arise. There are connections of a different nature between music and oil. The first aspect is historical. When the oil boom began, the large oil owners invested not only in education and enlightenment, but also in art - they built the Opera House, began to invite the musicians, artists, singers, etc. the European musical culture penetrated intensively in Azerbaijan.

One of the many reasons for this process was oil, which changed Baku into an important industrial city, multinational in appearance, which in its turn led to the emergence of typical attributes of European musical art here. However, at that time in Azerbaijan it existed in isolation from the national musical culture. The broad Azerbaijani public circles did not perceive the classical samples of European composer's creativity. The fusion of two musical cultures different in systemic qualities was carried out by the corypheus of Azerbaijani musical creativity - Uzeyir Hajibeyov. The appearance of this outstanding Azerbaijani composer was prepared by many historical factors. One of them, of course, not directly, but indirectly, was the active development of the oil industry in Azerbaijan, which contributed to the creation of conditions for the assimilation of professional European music in Azerbaijan [14].

The second aspect is associated with the musical creativity, which, to one or another degree, has in its content a reflection of the theme related to oil. Consequently, at the end of the 1920s and early in 1930s, U. Hajibeyov and M. Magomayev wrote the first Azerbaijani mass songs, including songs about oil. Later, the songs about oil workers were created by T. Kuliev and A. Rzayev. Music for two documentaries dedicated to oil workers was written by K. Karayev - "The Novel about the Caspian Oil workers" and "Conquerors of the Sea Weaves". Oil workers are the heroes of the film "My Favorite Song" (or Bakhtiyar), the music for which was written by Tofig Kuliev. The ballet of Tofig Bakikhanov "Caspian Ballad" is dedicated to Azerbaijani oil workers [14]. It is especially important to emphasize that the oil theme so inspired K. Karaev, so penetrated into his consciousness that years later, when he comprehended this problem in a purely musical aspect related to the development of folk music in composer's work, he was struck by a deep analogy of the connection between folklore and oil deposits.

If we already make comparisons, then, most likely, folk music can be likened to oil deposits, which are located in the earth in the form of layers located from the surface at different depths» [15]. And further K. Karaev continues to deepen this analogy: “Some 70-80 years ago in Absheron,” he argued, “it was enough to dig a well several meters long, and it quickly filled with oil ... People, in pursuit of profit, predatorily squander the upper, the most accessible to them layers. Oil began to go deeper, the upper layers were depleted, and now it can be produced only with the help of sophisticated technical devices. The situation is similar with folk music. Let's think about it, are not we getting it too easily, aren't we too mercilessly using the upper, easily accessible layers, isn't it time for us to think about the fact that folk music is really inexhaustible, but its main wealth is not only on the surface, but lies much deeper than we suppose and that it's time for us to get to them, armed with the necessary technical devices" [15]. By technical adaptations K. Karayev meant modern means of musical expression, which are not always quickly assimilated, not only by a wide audience, but even by professional musicians. Certainly, in order to do this, one must be a virtuoso, a foremost worker. In this respect, the statement of the outstanding Azerbaijani scientist A.Kh. Mirzadzhanzadeh is characteristic: “To reject new forms of writing, like everything new, only on the basis of what is perceived by an “average” person is not enough, of course, it is not correct. What is perceived today by the "eminent" representatives or the "elite", tomorrow will be perceived by the "average" person [16]. Azad Mirzajanzadeh is famous not only for being an outstanding corypheus of Azerbaijani oil and gas science, academician, oil worker, teacher, he is a man of encyclopedic knowledge, an unusually all-round mind, deeply versed in art and, in

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particular, a fine connoisseur of music. For example, the following phrase belongs to him: «Try to express music in any other form, - writes A. Kh. Mirzadzhanzadeh - Music does not convey the meaning of speech, but it is, to a much more powerful degree than speech, capable of conveying the tone of speech intonations» [16]. It is important to note that, Academician A. Mirzadzhanzadeh is the author of the manual "Introduction to the specialty", envisaged for students of technical universities. It is significant that he writes this manual through the prism of the humanities, literature and art, i.e. thus demonstrates that for the students of technical universities, future engineers and young technical specialists, the humanitarization of their thinking is very relevant and important. In his manual "Introduction to the specialty", he also quotes Kara Karayev about musical folklore, talks about the greatest cultural wealth of the Azerbaijani nation, which is subject to deep respect.

The third aspect is oil and charity. Very often it is the oil companies that sponsor music festivals, concerts, in addition, oil companies often sponsor talented children, their studies, etc. I would especially like to emphasize that the oil company “LUKOIL” financed the creation of films about the outstanding composer Kara Karaev and the famous conductor Niyazi.

The topic of oil also finds an interesting disclosure in the fine arts of Azerbaijan. The National Art Museum of Azerbaijan has collected the expositions of different times for future generations [17]. Not only music, but also works of art display the heroes -oil workers, whose individual and generalized images inspired many outstanding artists to create masterpieces of fine art. As the head of the Department of International Relations and Museum Innovations of the National Museum of Arts A. Melikova notes, the first "sketches" that have been preserved on postcards of the XIX - early XX centuries were made in the pre-revolutionary years by foreigners who came to Baku on various occasions. Thus, the British artist W. Simpson and the French photographer A, Michonne, being in Baku, photographed the oil fields in Surakhani and the hard work of the workers. A prominent Russian painter and graphic artist A. Ostroumova-Lebedeva, while being in Baku, during the First World War, painted a number of oil-related pictures. Among them we can note: "Bibi-Heybat" and "Oil platforms of Baku" and many others, reminding the sketches from nature. Currently, both paintings are not in Azerbaijan: one of them is in the Tretyakov Gallery, and the other is in the Art Museum of Estonia, in the Kadriorg Palace [18].

It should be especially emphasized that, the above-mentioned plots of the paintings cover the period when Azerbaijan ranked first place in the world in oil production. At that time, in some fields (Balakhani, Ateshgah-Shubany, Lokbatan), the daily oil production rate reached 16-20 tons. The famous well №1 in the Surakhany field produced 35 tons. of white oil. In the Ganja region of Azerbaijan, Naftalan medicinal oil, which has no analogue in the world, was produced. In Bibiheybat, 18-30 meters from the coast, for the first time in the world, oil was extracted from a hand-drilled well in the sea. Later, oil and gas field Neft Dashlary was explored in the Caspian Sea.

Already in the Soviet period, when a whole pleiad of Azerbaijani artists, having received their education in higher educational institutions in Moscow, Leningrad and Tbilisi, returned to Azerbaijan, and they immediately joined an active creative search. Furthermore, in the Azerbaijani fine arts, the next national stage of development began with the priority of the oil line. As a result, appeared the beautiful paintings by artists - S. Salamzadeh "A team of young oil drillers", "Baku” by T. Taghiyev. “Industrial

Landscape "," Hills "by L. Feyzullaeva," Oil Rocks "by K. Khanlarov," Trestle "by B. Mirzazadeh," Oil Rocks "by N. Ismayilov. In continuation of the series "Oil Rocks" M. Rahmanzadeh presented the graphic work "Our Guests", which depicts foreign representatives in the process of showing them a picture of a unique offshore oil field, symbolizing the national landmark of Azerbaijan. Then oil was a strategic resource for the huge Soviet power. The maestro of landscape painting, Sattar Bahlulzadeh, glorifying the living, realistic beauty of nature, brought concentration to the oil theme on the one hand and reverent, respectful attitude on the other. The artist devoted many paintings to the oil fields of the Absheron Peninsula. The canvases of our famous contemporaries - T. Javadov, N. Gasimov, M. Abdullayev, who also repeatedly refer to the oil theme, breathe realism.

It should especially be noted that the picture of the outstanding artist Tahir Salakhov "The Shift is Over". According to T. Salakhov himself, while painting the picture, the portraits of heroes-sailors flashed in his head - Mikhail Kaverochkin, drowned in a whirlpool of waves, fearless sailors – explorers from the boat "Sergey Chvanov", who gave their lives for the benefit of their native Azerbaijan [18]. In 2007, a new testimony to the large-scale personality of T. Salakhov appeared, his last work - the famous six-meter triptych "Land of Fires", in which the artist skillfully realized his idea of showing the past and present of the Azerbaijani people. One of the parts of the painting depicts a modern, powerful drilling rig equipped with the latest technology.

After breakup of the USSR, the modernist, postmodern and other trends and styles sounded in the visual arts, opening the new, bright pages of our modernity. The oil strategy of the century further emphasized the priority of "black gold" and inspired the honored artists to new creativity. Consequently, there appeared the installations by I. Eldarova, mosaic by K. Alieva, neon painting by F. Alekperov, as well as canvases painted in oil. The author of the unique technique of painting with oil is Sabir Chopuroghlu. He uses in his works instead of oil paints, watercolors, etc. directly the oil itself. His paintings are of interest all over the world. There is the painting "Caspian Symphony" among them which was awarded with the 1st degree Diploma [19]. This picture is filled with a philosophical meaning about the relationship between man and nature. After all, in fact, life is the same canvas that we paint ourselves.

CONCLUSION

The phenomenology of oil, presented in the aspect of Azerbaijani art, reveals associative parallels of the process of extracting "black gold" with the process of creating folk art - music, fine arts, etc. Extracting minerals from nature's pantries, we empty the upper layers, the pyramid of values subsides, goes deep, one has to use sophisticated techniques to extract the treasures of creativity or nature.

In the national framework and in the spatial survey of the post-Soviet states, the phenomenology of oil is a primary direction and is of great interest in the aspect of dialectical philosophy. Thus, the study of various works of art in Azerbaijan, representing its various types, is evidence of the generalization of the artistic order of this philosophical theme. The additional generalization carried out in the works of art of Azerbaijan is predetermined by the priority symbolic factor of oil and the State Strategic Policy of Harmonious Development in order to form a new cultural world and careful use of natural resources.

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According to the President of Azerbaijan Ilham Aliyev: "... We managed to direct the revenues from oil to the development of the non-oil sector. We have managed to invest large investments in human capital" [20].

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НЕФТЬ КАК ФИЛОСОФСКАЯ КАТЕГОРИЯ И ЕЕ ОТРАЖЕНИЕ В АЗЕРБАЙДЖАНСКОМ ИСКУССТВЕ

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В статье рассмотрена феноменология нефти через сегменты философии культуры, которые по своему происхождению и существу тесно связаны с эпистемой мировосприятия. Азербайджанская нефть – это особая страница для нашей страны, ее исторического прошлого, современности и будущего. В XXI веке всё более набирает популярность идея гармоничной коэволюции природы и общества. Гармоничное сочетание природных богатств и культурных ценностей страны – такова философия современного Азербайджана, выстраивающего механизм распределения природных ресурсов в целях социального обеспечения населения. Такая стратегия позволяет формировать надежное будущее нации. Ведь нефть – это не только топливо, но и энергия, жизнь, генератор движения колеса истории, своего рода двигатель прогресса индустриального общества. Нефть не только обеспечивает повседневную жизнь, но и является отправной точкой для понимания того, каким будет наше общество в будущем. В статье рассмотрена феноменология нефти а аспекте азербайджанского искусства. Произведения искусства азербайджанских авторов свидетельствуют о героях-нефтяниках, чьи собирательные образы вдохновили многих выдающихся композиторов, художников, скульпторов на создание глубоких по философскому звучанию творений. Художественный фактор в той или иной степени присутствующий в них, способствует более глубокому феноменологическому подходу при отражении нефтяной темы. Благодаря духовным носителям, таким как, музыка, изобразительное искусство, кинематография в истории Азербайджана навсегда остались образы и имена первых бурильщиков скважин, создателей
Ключевые слова: *феноменология нефти, азербайджанская нефть, нефть в музыкальном отражении, нефть в кинематографе, нефть в изобразительном искусстве.*

NEFT FƏLSƏFI KATEQORIYA KIMI VƏ ONUN AZƏRBAYCAN SƏNƏTINDƏ ƏKS OLUNMASI

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Məqalədə, mənşəyi və mahiyyəti baxımından dünya qavrayış epistemi ilə sıx əlaqəli olan mədəniyyət fəlsəfəsinin seqmentləri ilə neft fenomenologiyası nəzərdən keçirilir. Azərbaycan nefti ölkəmiz, tarixi keçmişi, müasirliyi və gələcəyi üçün xüsusi bir səhifədir. XXI əsrdə təbiətin və cəmiyyətin harmonik birgə təkamülü ideyası getdikcə daha çox populyarlıq qazanır. Ölkənin təbii sərvətləri ilə mədəni dəyərlərinin ahəngdar birləşməsi, təbii sərvətlərin əhalinin sosial təminatı məqsədilə bölüşdürülməsi mexanizmini quran müasir Azərbaycanın fəlsəfəsidir. Bu cür

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strategiya millətin təhlükəsiz gələcəyini formalaşdırmağa imkan verir. Axı neft təkcə yanacaq deyil, həm də enerji, həyat, tarix təkərinin yaradıcısı, sənaye cəmiyyətində bir növ irəliləyiş mühərriki. Neft gündəlik həyatı təmin etməklə yanaşı, gələcəkdə cəmiyyətimizin necə olacağını anlamaq üçün də bir başlanğıc nöqtəsidir.

Məqalədə neft fenomenologiyası Azərbaycan sənətinin aspektində nəzərdən keçirilir. Azərbaycan müəlliflərinin sənət əsərləri ümumiləşdirilmiş obrazları bir çox görkəmli bəstəkarları, rəssamları, heykəltəraşları dərin fəlsəfi səs əsərləri yaratmağa ruhlandıran neft sənayesi işçiləri qəhrəmanlarına şəhadət edir. Bunlarda müəyyən dərəcədə mövcud olan bədii amil, neft mövzusunu əks etdirərkən daha dərin bir fenomenoloji yanaşmaya kömək edir. Musiqi, təsviri sənət, kinematoqrafiya kimi mənəvi daşıyıcılar sayəsində ilk quyu qazmaçılarının və neft yataqlarının yaradıcılarının obrazları və adları Azərbaycan tarixində əbədi olaraq qalmışdır.

Açar sözlər: *neft fenomenologiyası, Azərbaycan yağı, musiqi əksində yağ, kinematoqrafiyada yağ, vizual sənətdə yağ.*

UDC:669.2;66.08

DERIVATOGRAPHIC STUDY OF THE MIXTURE OF PYRRHOTINIZED FILIZCHAY ORE WITH FERRIC SALT

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The process of oxidation in the solid phase of pyrrhotinazedpolimetalsulphide ore(the Filizchay deposit) with ferric salts ($FeCl_3$ and $Fe_2(SO_4)_3$) by the derivatographic method was studied.The transformations in the main sample were determined according to endo- and exo- effects observed in DTA curves and mass loss in TG curves. Reactions occurring in the solid phase are proposed and confirmed by X-ray phase analysis.
Keywords: polymetalsulfideore, pyrrhotine, thermal analysis, X-ray phase analysis, ferricsulphate, ferric chloride.

INTODUCTION

One of the important factors in processing of polymetal sulfide ores is maximum extraction of all valuable metals together with main components (iron, sulphur) from them. Presently one of the methods during processing sulfide raw materials is oxidation of them using ferric salts. Fe^{3+} ions oxidize metal sulfides and allow transforming them into more soluble compounds, and sulphur to a free state. This is due to high oxidative ability of iron (III) ions. On the other hand, ferric salts can be easily used for processing of sulfide minerals due to their cheapness and easy recovery.The reactions during the oxidation of sulfides in ore with ferric sulfate and ferric chloride can be expressed by the following general equations:

$$MeS+Fe_2(SO_4)_3 \rightarrow MeSO_4+2FeSO_4+S^0$$

$$MeS+2FeCl_3 \rightarrow MeCl_2+2FeCl_2+S^0$$

Me=Zn, Cu, Pb

There are various research works [1-6] in literature on processing of sulfide ores and concentrates with ferric salts.The kinetics of oxidation of various sulfide minerals with ferric ions was studied in these research works. Ferric sulfate [1-3] and ferric chloride [4-6] were used as a solvent, their effect on the dissolution of sulfides was studied.But research objects used in these workssharply differ from each other both for element and phase composition. Since Filizchaypolymetal ore has a complex composition, special researches must be performed and a new method must be chosen to determine optimum parameters during its processing. In our paper we have presented the results of derivatographic analysis of interaction of pyrrotinized product of Filizchay polymetal ore with ferric salts in solid phase. In this work the aim is to study the oxidation of pyrrotinized product of Filizchay ore in solid phase using physical-chemical analysis methods.

Mineralogical researches showed that the main mineral of Filizchay ore is pyrite and it is influenced by other minerals (ZnS, PbS, $CuFeS_2$). It is known that pyrite has a

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crystal cell and is poorly soluble in water and acids. These show that Filizchay ore belongs to complex ores. It is known that the processing of raw materials containing non-ferrous and precious metals includes the burning of ore, solution of a burn, extraction of metals from solution by various methods. Gases emitted to atmosphere during burning of sulfide ores mainly consists of sulfur tetroxide which deepens ecological crisis.Therefore, during researches we used pyrrhotinized product produced from thermal decomposition of Filizchay ore at inert atmosphere.

EXPERIMENTAL PART

In the work derivatographicanalysis of the mixture of ferric salts ($FeCl_3$, $Fe_2(SO_4)_3$) with pyrrhotinized product under non-isothermal condition was studied. Pyrrhotinized product contains iron (50.71%), zinc (7.44%), lead (3.9%), copper (0.73%), sulphur (33.2%) and SiO_2 (2.1%).

The mixture of pyrrhotinized product of Filizchay ore with "chemically pure" $FeCl_3 \cdot 6H_2O$, $Fe_2(SO_4)_3 \cdot 10H_2O$ crystal hydrates in 1:1 ratio was used as research object. Thermal analysis curves were obtained at 20-1000^{0}C in the air heating for 10deg./min on Paulic-Paulic-Erdeyderivatograph Q-1500. The sample and standard (thermo inert substance – α-Al_2O_3)are placed to heating furnace in a corundum crucible. Temperature is controlled by chromel/alumel thermocouple. Phase transformations in the material studied by thermograms are analyzed. In DTA curves endo- and exothermic effects are recorded. The samples taken at temperatures corresponding to endoeffects in DTA curves are studied by X-ray phase analyses. X-ray phase analyses are performed on copper cathode (CuK_a=1.54 A^0) DRON-2 diffractometer.

RESULTS AND DISCUSSION

Some endothermic and exothermic effects (fig.1 a and b) were observed in these samples. The first endoeffects observed at 90^{0}C correspond to physically linked water loss in the sample that is accompanied by weight loss in TG curves. In both samples mass reduction is found to be 1.025 and 11.17%, correspondingly. Crystallization in three endoeffect samples the peaks of which correspond to 160, 195 and 250^{0}C in figure 1a, and in four endoeffect samples the peaks of which correspond to 160, 180, 210 and 280^{0}C in figure 1 b is related to water evaporation. As seen from TG curves in both samples decomposition takes place beginning from 160^{0}C at a wide temperature range. Using these curves mass loss was calculated and it was approximately found to be 16.8% (fig.1a) and 25.2% (fig.1b). This corresponds to theoretically calculated values of crystallization water in the samplesIn DTA curve exothermic effects with maximum of 420^0 (fig.1a) at 350-500^{0}C and maximum of 520^{0}C (fig.2a) at 500-560^{0}C correspond to the conversion of free sulphur, which is formed due to interaction of sulfides with ferro-ions, to sulfite anhydride being oxidizing with oxygen. Results of X-ray phase analyses (fig.2a) of the sample produced from heating pyrrhotinized product and ferrichloride mixture at 420^{0}C shows that it contains ferrous chloride ($FeCl_2$) (d=5.90; 5.302; 3.96; 2.52; 1.83).

There are also the lines (d_n(A^0) =5.513; 3.725; 2.434) corresponding to basic iron sulfate in diffractogram. As seen from thermograms at a highertemperature mass decreases and weight loss is found to be 13.5% (fig.1a) and 7.18% (fig.1b),

correspondingly. This can be related to a hydrolytic destruction of ferric salts and crystal hydrates, as well as different reactions betweengas and solid phase.

Exothermic effect recorded at 670^0C in DTA curve (fig.1a) corresponds to the formation of hematite. The results of X-ray phase analyses (fig.2a) of the samples obtained from heating pyrrhotinized product and $FeCl_3$ mixture at650^0C shows the lines ($d_n(A^0)$ = 3.696; 2.70; 2.515; 1.84) corresponding to Fe_2O_3.

In DTA curve exothermic effect recorded at 650-720^0C with maximum at 685^0C corresponds to a mass loss in TG curve. The results of X-ray phase analyses (fig.2d) of the samples obtained from heating the mixture of pyrrhotinized product with $Fe_2(SO_4)_3$ at 650^0C show that this phaseconsists of only synthetic magnenite ($Fe_{2.9}O_4$) ($d_n(A^0)$ =5.453; 3.126; 2.202; 1.911; 1.695).

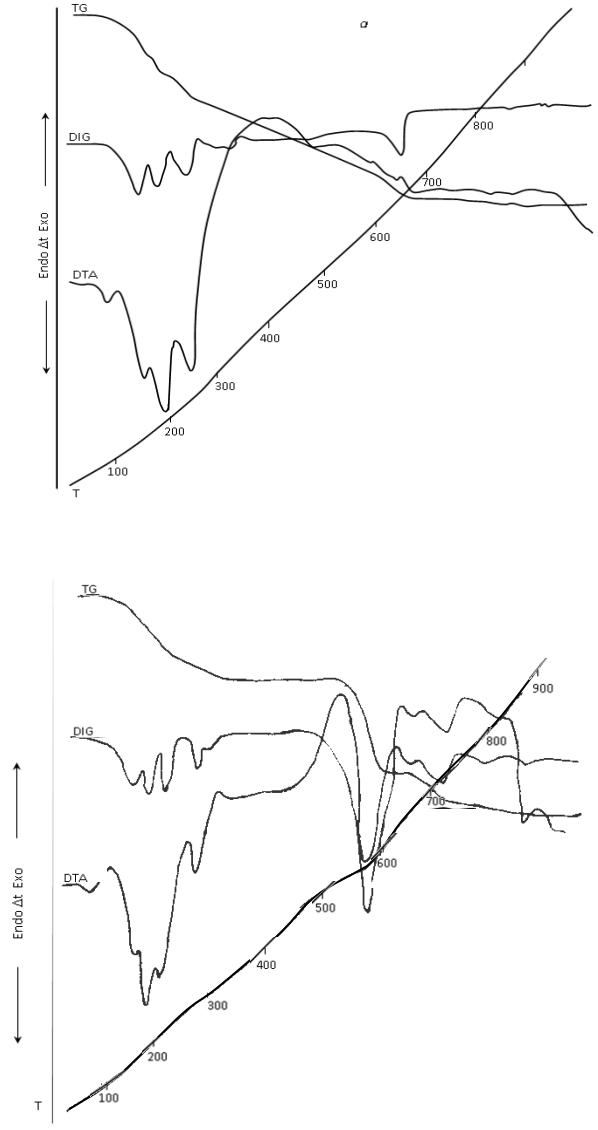

Fig.1. Derivatograph of the mixture pyrrhotinized product with $FeCl_3 \cdot 6H_2O$ (a) and $Fe_2(SO_4)_3 \cdot 10H_2O$ (b)

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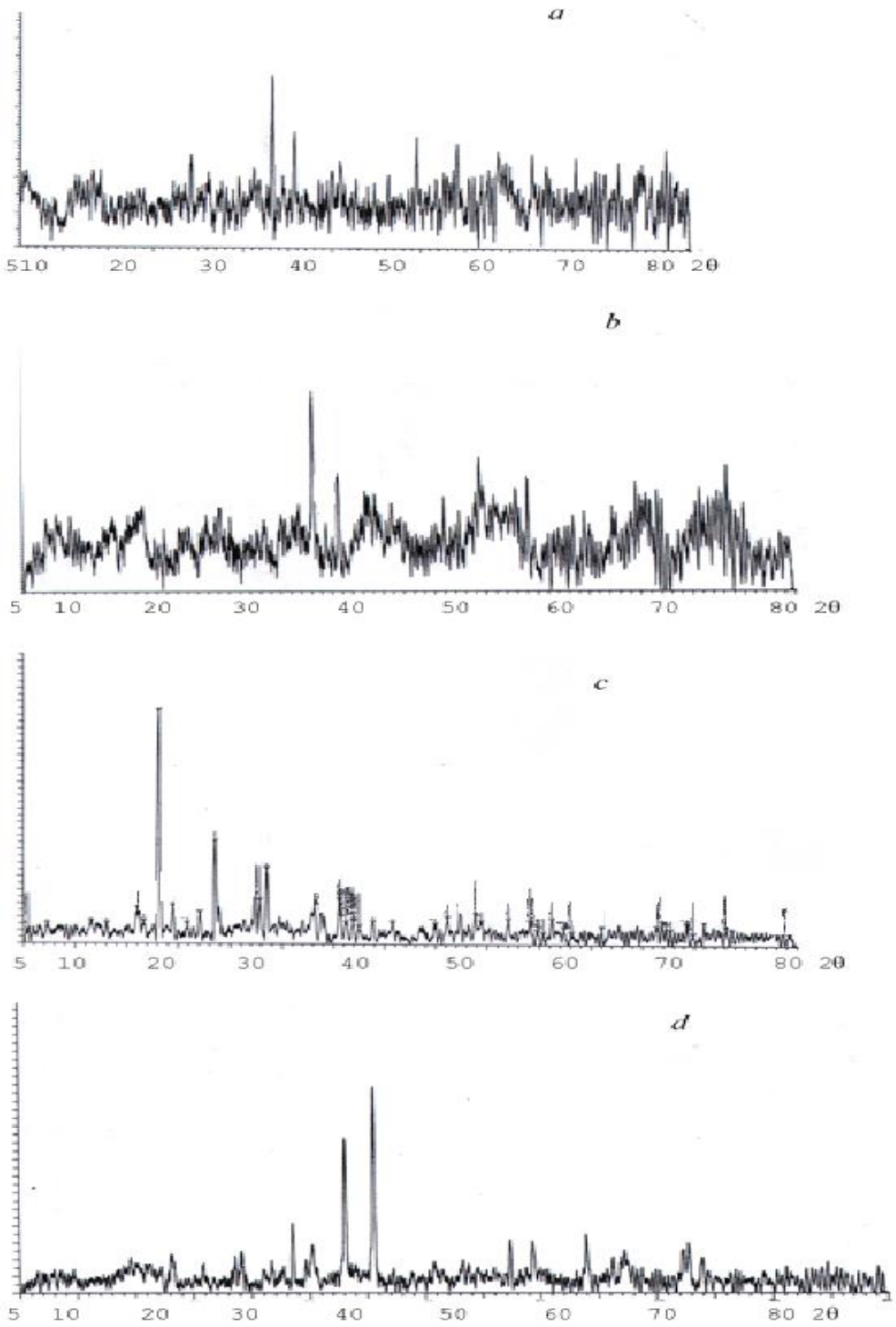

Fig. 2. X-ray pattern of the mixture of pyrrhotinized product with $FeCl_3 \cdot 6H_2O$ (a-420^{0}C, b-650^{0}C) and $Fe_2(SO_4)_3 \cdot 10H_2O$ (b c-520^{0}C, d-650^{0}C)

In diffractogram we observed the lines ($d_n(A^0)$ = 5.567; 2.344; 2.836) showing the presence of basic ferrous chloride along with the lines corresponding to ferric salts.

The results of X-ray phase analyses shows the presence of the phase α-Fe_2O_3 ($d_n(A^0)$ = 2.70; 2.515; 1.842; 1.489; 1.454) and synthetic Fe_2O_3 (maghemite) ($d_n(A^0)$

=3.765; 2.945; 2.086). It is supposed that magnetite is converted to maghemite and hematite at 650-720^{0}C and maghemite is also transformed to hematite.

CONCLUSION

1. According to the studies it was determined that the oxidation of pyrrotinized product of Filizchay ore with ferric sulfate and ferric chloride occurs with the same regularities.
2. Thus, in both cases the formation of basic ferric salts, normal ferric salts (relevantly chloride and sulfates) is observed and it is determined that the final phase of oxidation of metal sulfides consists of a stable phase – hematite.

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ДЕРИВАТОГРАФИЧЕСКИЕ ИССЛЕДОВАНИЯ ПИРРОТИНИЗИРОВАННОЙ ФИЛИЗЧАЙСКОЙ РУДЫ В СОЛЯХ ТРЕХВАЛЕНТНОГО ЖЕЛЕЗА

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Дериватографическим путем исследован процесс окисления пирротинизированной полиметаллической сульфидной руды (Филизчайского месторождения) солями трехвалентного железа ($FeCl_3$ и $Fe_2(SO_4)_3$) в твердой фазе. На основании –эндо и экзоэффектов наблюдаемых на кривых ДТА были установлены превращения происходяшие в твердой фазе, что также подтверждается убылю массы на кривых ТГ и подтверждается результатами РФА.
Ключевые слова: *полиметаллического сульфидного руда, пирротин, термический анализ, рентгенофазовый анализ, хлорид железа, сульфат железа.*

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PİRROTİNLƏŞMİŞFİLİZÇAYFİLİZİNİN ÜÇVALENTLİDƏMİRDUZLARI İLƏ QARIŞIĞININ DERIVATOQRAFİKTƏDQİQİ

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Pirrotinləşmiş polimetal sulfid filizinin (Filizçay yatağı) bərk fazada üçvalentli dəmir duzları ($FeCl_3$,$Fe_2(SO_4)_3$) ilə oksidləşməsi prossesi derivatoqrafik üsulla tədqiq edilmişdir. DTA əyrilərində müşahidə olunan endo- və ekzotermiki effektlərə və TG əyrilərindəki kütlə itkisinə əsasən nümunədə baş verən çevrilmələr müəyyənləşdirilmişdir və nəticələr RFA vasitəsilə təsdiqlənmişdir.

Açar sözlər: *polimetal sulfid filizi, pirrotin, termiki analiz, rentgenfaza analizi, dəmir 3-sulfat, dəmir 3-xlorid.*

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COLLOIDAL STABILITY OF COMPOSITIONS BASED ON INDUSTRİAL OIL, ZINC DIALKYL- DITHIOPHOSPHATE AND FUNCTIONALLY SUBSTITUTED SULFIDES ON DYNAMIC LIGHT SCATTERING DATA

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Colloidal stability of compositions based on industrial oil I-40A, dialkyl zinc diphosphate (DF11), and functionally substituted sulfides R_1 - S - R_2, where R_1 is $C_4H_9OCOCH_2$-, was studied by the method of dynamic light scattering (DLS). R_2 is CH2CH2X, where X is Cl (1), Br (2), OH (3), -CH_2-C_6H_4-OH (4). The structure of the composition before and after processing was characterized by IR and EPR spectroscopy. The size of the "hydrodynamic" particle diameters and their size distribution in the compositions were determined using a Horiba LB 550 particle size analyzer in the temperature range of 278-343K. DLS data show that the starting base oil and its compositions with the above sulfide compounds are molecular systems, and the particle size in these systems and compositions with zinc dialkyldithiophosphate is no more than 10 nm. Processing these compositions changes the spectral picture. During the operation of oils, paramagnetic asphaltene structures are formed. The interaction of these structures with each other and with diamagnetic molecules of the environment leads to the formation of colloidal particles of the order of 100 nm or more, the aggregation of which leads to their precipitation and deterioration of the operating properties of oils. The mechanism of interaction of the components of the compositions, leading to the stabilization of colloids, as well as the effect of treatment on their composition and structure are considered.
Keywords: *colloidal stability, industrial oil, zınc dıalkyldıthıophos- phate, functionally substituted sulfides, dynamic light scattering.*

INTRODUCTION

Modern oils are lubricants of complex composition containing an additive package [1]. The selection of additives is carried out mainly on the basis of their functional action. Very often, compositions obtained by simple mixing of commercial additives, due to insufficient colloidal stability, can delaminate under the influence of various factors. In the conditions of storage and use of commercial oils, precipitation is formed in them, their operational properties deteriorate. To create an effective and stable package of additives during storage and operation, it is necessary, first of all, to study the mutual influence of additives in the package, a system for selecting additives that are compatible with each other and, along with compatibility and the correct quantitative ratio of components in the composition, it is extremely necessary to take into account colloidal-chemical characteristics of the selected systems. Research in this direction requires the use of highly selective informative methods for studying the colloidal stability of commercial oils [2, 3].

In this work the results of studies by dynamic light scattering (DLS) methods of colloidal stability of a composition based on industrial oil I-40A, zinc (II) dialkyldithiophosphate, and functionally substituted sulfides are presented.

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EXPERIMENTAL PART

Table 1 shows the physicochemical parameters of the base industrial oil I-40A, which is a mixture of highly purified distillate and residual oils of selective purification.

Table 1

Basic physical and chemical indicators of industrial oil I-40A [4]

№	Indicators	Values
1	Density at 20 °C, кkg/m^3, no more than	900
2	Kinematic viscosity, at 40 ° C, mm^2 / s	61-75(51-75)
3	Acid number, mg KOH / g, no more than	0,05
4	Temperature, °C: flashes in an open crucible, not lower than	220 (200)
5	Pour point, ° C: not higher than	-15
6	Color, units CNT, no more than	3,0 (4,5)
7	Oxidation stability: acid number increment, mg KOH / g, no more than	0,4
8	Resin content,%, no more than	3,0

Zinc dialkyldiuthiophosphate DF11, “Kvalitet” company Groupe, Russia, is used as detergent additive. In the table 2 the main physical-chemical parameters of DF11 are given.

Table 2

The basic physical and chemical parameters of Zn(II) dialkylditiophosphat (DF11, Russia)

№	Indicators	Values
1	Zink, mas. %	9,6
2	Phosphor, mas. %	8,7
3	Sulphur, mas. %	15,0
4	Viscosity at 100°C, mm^2/s	7
5	Flash point in an open crucible, ºC	<170

Functionally substituted sulfides of the composition R^1 –S –H + R^2 -X → R^1 –S – R^2, where R^1 is C_4H_9-O-CO-CH_2-; R^2 is $CH_2CH_2CH_2X$ and X is Cl (1); Br (2); OH (3); -CH_2-C_6H_4OH (4), used as additives, were obtained by reacting butyl ester of mercaptoacetic acid with unsaturated compounds in the presence of triethylamine. The initial butyl ester of mercaptoacetic acid was obtained according to a known method [5] by the interaction of mercaptoacetic acid with butanol in the presence of HCl as a catalyst: its yield was 77% and T_b. 192-194 °C; n_D^{20} 1.4568; d_4^{20} 1027.8kg / m^3.

Compounds 1-4 were synthesized as follows: to a mixture consisting of 0.5 mol of butyl ester of mercaptoacetic acid and 0.5 mol of allyl halide with stirring, a few drops of triethylamine were added, stirring was continued for 1 h at room temperature and 3-4 h at 60-70 °C. Upon completion of the reaction, the target compound was isolated from the reaction mixture by vacuum distillation. Table 3 lists the characteristics of the used sulfides.

Table 3

Physicochemical indicators of functionally substituted sulfides*

A	B	C	D	Element composition,%				
				C	H	S	Cl	Br
1	88-92	1.4810	1088	48,07	7,52	14,02	15,54	-
2	62-65	1,4651	1209	40,03	6,29	11,68	-	29,54
3	148-150	1,4836	1073	52,31	8,59	14,95	-	-
4	197-200	1,5253	1145	63,73	7,57	11,17	-	-

* A - compounds, B - boiling point, respectively, at 12, 3, 3 and 4 mm Hg, in ºC, C - refractive index at 20 ºC, D - density at 20 ºC, in kg /m^3.

The structures of the synthesized sulfides were confirmed by IR and NMR spectroscopy. The IR spectra of the compounds contain absorption bands in the range of 1700-1710 cm^{-1}, characteristic of the C = O group, and there are no absorption bands from the SH-group. The IR spectrum of compound 3 clearly shows the bands absorptions characteristic of the ester group (1172 and 1239 cm^{-1}). The presence of a hydroxyl group is confirmed by a wide band in the range of 3500-3600cm^{-1}.

The values of the magnetic resonance characteristics of the synthesized compounds 1-4 are given below.

Compound 1: ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 0.85 t (3H, CH_3, 3JH-H = 6.45 Hs); 1.42 m (2H, CH_2); 2.05 m (2H, CH_2); 2.75 t (2H, CH_2 -S, 3JH-H = 6.1 Hs); 3.15 s (2H, CO-CH_2-S); 3.55 t (2H, CH_2 -Cl, 3JH-H = 6.25 Hs); 4.1 t (2H, CH_2O, 3JH-H = 6.38 Hs). ^{13}C NMR Spectrum: 12.7; 18.3; 28.7; 31.3; 32.2; 33.1; 42.9; 65.1; 169.8.

Compound 2: ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 0.6 t (3H, CH_3, 3JH-H = 6.45 Hs); 1.0 m (2H, CH_2); 1.25 m (2H, CH_2); 2.4 t (SCH_2); 3.05 s (CH_2S); 3.15 t (2H, CH_2Br); 4.2 t (2H, CH_2O). ^{13}C NMR Spectrum: 10 (CH_3); 19 (CH_2); 26 (CH_2); 30 (CH_2); 31 SCH_2); 34 (CH_2S); 41 (CH_2Br); 71 (CH_2)), 170 (COO).

Compound 3: ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 0.8 t (3H, CH_3); 1.1 m (2H, CH_2); 1.45 m (2H, CH_2); 1.75 m (2H, CH_2); 2.5 t (SCH_2); 3.2 s (2H, CH_2S); 4 t (2H, CH_2OH), 4.3 t (2H, CH_2O), 5.2 s (H, OH). ^{13}C NMR spectrum ($CDCl_3$, δ, ppm): 10 (CH_3); 11 (CH_3); 20 (CH_2); 28 (CH_2); 31 (CH_2); 32 (SCH2); 36 (CH_2S); 65 (CH_2OH); 70 (CH_2O)), 175 (COO).

Compound 4: ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 0.9 t (3H, CH_3); 1.3 m (2H, CH_2); 1.6 m (2H, CH_2); 2.0 m (2H, CH2); 2.7 t (4H, SCH_2 + CH_2Ar); 3.6 s (2H, CH_2S); 4.2 t (2H, CH_2O), 5.5-5.8 s (1H, OH); 6.7-7.4 m (4H, Ar). ^{13}C NMR spectrum ($CDCl_3$, δ, ppm): 14 (CH_3); 19 (CH_2); 29 (CH_2); 30 (CH_2); 32 (CH_2); 34 (SCH_2); 35 (CH_2, Ar); 55 (CH_2S); 116 (CH, Ar), 120 (CH, Ar); 127 (CH, Ar), 130.5 (CH, Ar), 136 (C, Ar), 154 (C, Ar), 171 (COO).

The elemental composition of the synthesized compounds (carbon, hydrogen, sulfur) was determined using a TruSpec Micro CHNS analyzer from LECO. The content of chlorine and bromine in the samples was determined using the combustion method in a flask with oxygen. This method was also used to determine the sulfur content in the samples. The results of determining the sulfur content in the synthesized samples by the above two methods did not exceed 10-15% [6].

NMR spectra of the synthesized compounds were recorded on a Bruker-300 spectrometer with an operating frequency of 300 MHz (Germany). The internal standard is tetramethylsilane. We used 1-5% solutions of the analyzed compounds in $CDCl_3$. IR

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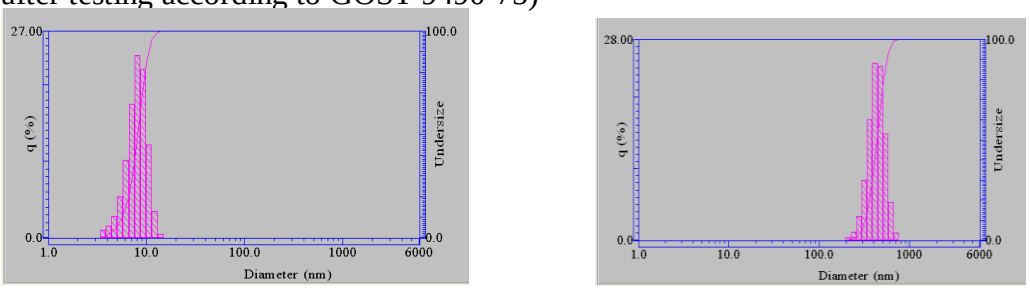
spectra were recorded on an FTIR LUMOS spectrometer, Bruker, in the frequency range 400-4000 cm^{-1}. EPR spectra were registered using EMXmicro, Bruker spectrometer. The "hydrodynamic" particle diameters of the base oil and waste oil samples and their size distribution were determined using a Horiba LB 550 particle size analyzer. The method is based on measuring the average diffusion rate of dispersed particles by registering fluctuations in the scattered light intensity. This method directly measures the diffusion coefficient of particles in a liquid. Since the diffusion coefficient of particles is unambiguously related to their size, the size of these particles is essentially determined by the method of dynamic light scattering (DLS) [7, 8].

The analyzer used makes it possible to study the processes of formation, decay of particle aggregates, complexes in the temperature range 278-343K. The range of determined particle sizes of this device is 0.001-6 microns. The power of the radiation source is 5 mW, the wavelength is 650 nm. The antiwear properties of the joints were determined using a four-ball friction machine ChShM-3/1 (GOST 9490-75). Test conditions: spindle rotation speed 1420 rpm, load 20 kg, room temperature. time 1 hour. The antiwear properties of the synthesized compounds were judged by the size of the wear spot diameter (mm), which were 0.42, 0.44, 0.54, and 0.60 mm for samples I-40A containing 1 wt% of compounds 1-4: 0.42, 0.44, 0.54, and 0.60 mm, respectively. Note that the size of the wear scar for the base oil samples is 0.81 mm. For comparison of antiwear properties, samples of I-40A oil containing 1.5 wt.% Of the known additives DF-11 were tested (the size of the wear scar was 0.81 mm).

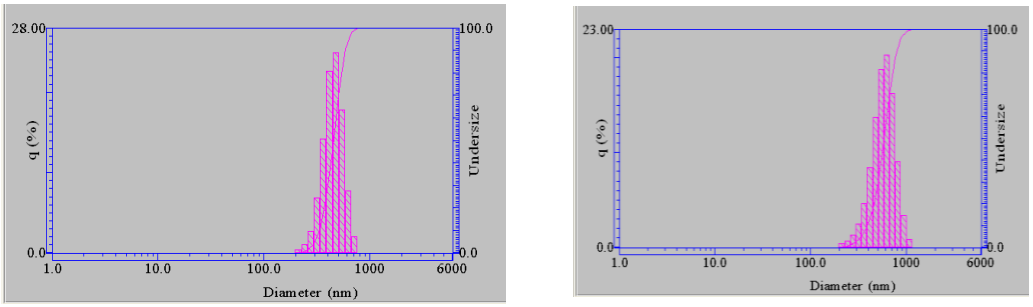
RESULTS AND DISCUSSION

In fig. 1 and 2 show, respectively, the dynamic light scattering spectra (DLS) of industrial oil I-40A and oil I-40A containing compound 1 and DF-11 in n-octane (before and after testing according to GOST 9490-75)



a) b)

Fig. 1. DLS spectra of sample I-40A in n-octane: a) before and b) after testing



a) b)

Fig. 2. DLS spectra of samples I-40A containing: a) 1 wt% compound DF11 and compound 4b) DF11 compound 4 in n-octane after testing

Table 4 shows the values of the DLS parameters for I-40A samples containing compounds 4 and DF11 in n-octane (before and after testing).

Table 4

Values of DRS parameters for I-40A base oil samples and its compositions with compounds 4 and DF11 (before and after testing) in n-octane*

Com posi-tion*	Diameter of the particles in liquid phase, nm			Median, nm	Average size, nm	Moda, nm	Span, rel.units	D**, E^{-11},m^2/s
	10	50	90					
1a	5,9	8,1	10,3	8,1	8,1	8,1	0,61	13
1b	295,6	582,8	870,0	582,8	590,4	619,6	0,71	0,18
2a	4,7	6,5	8,5	6,5	6,6	6,9	0,58	4,1
2b	234,3	450,9	667,5	450,9	455,6	470,2	9,55	0,23
3a	3,1	4,5	5,9	4,5	4,5	4,6	0,76	5,9
3b	196,6	433,9	671,2	433,9	439,4	424,5	0,58	0,29

* Samples of I-40A oil: 1 - initial, no additives, 2,3 - containing in the amount of 1 wt%, DF11 and DF11 + compound 4, respectively; a and b - samples of joints before and after testing. ** D is the value of the diffusion coefficient of particles in m^2/sec., Determined by the Stokes – Einstein formula $D = k_BT / 3\pi\eta d$, where k_B is the Boltzmann constant, T is the temperature in Kelvin degrees, η is the viscosity of the medium, d is the "hydrodynamic diameter" particles.

As can be seen from the above fig. 1, a and table. 4, the values of the average particle sizes before testing for oil samples vary in the range from 3 to 9 nm. These particles most likely belong to supramolecular formations. Note that in industrial oils, as in almost all similar liquid petroleum products, supramolecular structures are formed, most likely from resins with sizes up to 10 nm [9, 10]. These structures are paramagnetic and have characteristic EPR spectra [11]. For samples of fresh base oil I-40A, no signals in the EPR spectra recorded at room temperature are observed. However, the EPR spectra of the samples of treated oils are a superposition of at least three signals centered at g = 2.02 with a line width ΔH = 110 mT, g = 4.3 with a width ΔH = 630 mT and a narrow signal c g = 2.003 with a line width ΔH = 0.9 mT. The first two signals most likely belong to iron-containing particles [9] and a narrow signal to radical formations formed in the process of base oil development. Note that these signals are observed for all samples containing compounds 1-4, DF11, and base oil and only slightly differ from each other in the intensity of the observed signals. This picture allows us to conclude that the narrow signal is most likely due to asphaltene structures formed during the degradation of the I-40A oil samples, and the presence of broad signals belonging to the iron-containing structures introduced into the base oil samples during the test. As can be seen from the above fig. 1 and table 3, for the used oil samples, noticeable changes in the particle sizes are observed. The formation of particles of this size in used oils is the result of degradation of the base oil. During the operation of the IO, under the influence of external factors (air oxygen, temperature, etc.), its chemical composition changes due to the formation of paramagnetic asphaltene structures. The presence of absorption bands at 1710 and 1608 cm^{-1} in the IR spectra of waste oils indicate the formation of oxidized compounds (acids, aldehydes, ketones,

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etc.) and an increase in the content of polycyclic aromatic structures. For the samples of used oil, weak bands were also found at 3420 cm^{-1} and 1625 cm^{-1} of water adsorbed from the air, and also allowed bands at 1100-1010 cm^{-1} in the region of bending vibrations of OH groups linked by hydrogen bonds.

Note that the IR spectra of all the studied oil samples, both base and used, are typical for oils. They practically coincide, but differ in the intensities of the absorption bands. In addition, absorption bands at 2360 and 2340 cm-1 are also recorded in all spectra, due to the absorption of CO_2 in air, since the spectra were recorded under normal conditions with free access to air. In addition to the bands already described, absorption bands of stretching vibrations of C = C bonds (1605 cm^{-1}) of aromatic rings are observed in the IR spectra at an increased layer thickness. The presence of condensed aromatic compounds is also confirmed by the presence of bands at 873 and 815 cm^{-1}. In the IR spectra of waste oil samples, along with the above absorption bands, absorption bands of CH_2 and CH_3 groups of alkane structures (2952, 2935, 2872, 2848, 774 and 720 cm^{-1}) are clearly manifested. The absorption bands in the region of 930 and 890 cm^{-1} rather reliably indicate the presence of terminal vinyl and (or) methylene groups [12, 13]. A comparison of the IR spectra of samples of base and used oils shows that the intensity of most absorption bands belonging to aromatic structures, mainly at 1605 cm^{-1}, noticeably increases, which indicates an increase in aromatic compounds during processing. This process can lead to the formation of supramolecular structures due to intermolecular interactions of π -electrons of aromatic rings and their further agglomeration with precipitation.

CONCLUSION

Thus, the presented experimental results allow us to conclude the following.

1. During the operation of oils, paramagnetic asphaltene structures are formed. The interaction of these structures with each other and with diamagnetic molecules of the environment leads to the formation of colloidal structures with sizes of about 100 nm or more, the aggregation of which leads to precipitation and deterioration of the operating properties of oils.
2. Oils containing sulphides as an additive and subjected to treatment are characterized by a smaller size of colloidal structures in comparison with oils containing DF11.

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КОЛЛОИДНАЯ УСТОЙЧИВОСТЬ КОМПОЗИЦИЙ НА ОСНОВЕ ПРОМЫШЛЕННОГО МАСЛА, ДИАЛКИЛ-ДИТИОФОСФАТА ЦИНКА И ФУНКЦИОНАЛЬНО -ЗАМЕЩЕННЫХ СУЛЬФИДОВ ПО ДАННЫМ ДИНАМИЧЕСКОГО РАССЕЯНИЯ СВЕТА

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Методом динамического светорассеяния (ДРС) исследована коллоидная стабильность композиций на основе индустриального масла И-40А, диалкилдифосфата цинка (DF11) и функционально-замещенных сульфидов R^1 - S - R^2, где R^1 - $C_4H_9OCOCH_2$-, R^2 - CH_2CH_2X, где X - Cl (1), Br (2), OH (3), -CH_2-C_6H_4-OH (4). Структура композиции до и после обработки охарактеризована методами ИК- и ЭПР-спектроскопии. Размер «гидродинамических» диаметров частиц и их распределение по размерам в композициях определяли с использованием анализатора размера частиц Horiba LB 550 в диапазоне температур 278-343К. Данные DLS показывают, что исходное базовое масло и его композиции с указанными выше сульфидными соединениями представляют собой молекулярные системы, и размер частиц в этих системах и композициях с диал-килдитиофосфатом цинка составляет не более 10 нм. Обработка этих композиций меняет спектральную картину. В процессе эксплуатации масел образуются парамагнитные структуры асфальтенов. Взаимодействие этих структур друг с другом и с диамагнитными молекулами окружающей среды приводит к образованию коллоидных частиц порядка 100 нм и более, агрегирование которых приводит к их осаждению и ухудшению рабочих свойств масла. Рассмотрен механизм взаимодействия компонентов композиций, приводящий к стабилизации коллоидов, а также влияние обработки на их состав и структуру.

Ключевые слова: *коллоидная стабильность, индустриальное масло, цинк диалкилдитиофосфат, функционально-замещенные сульфиды, динамическое рассеяние света.*

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SƏNAYE YAĞI, SİNK DİALKİL DİTİOFOSFAT VƏ FUNKSİYONAL ƏVƏZLİ SÜFFİDLƏR ƏSASLI KOMPOZİSİYALARIN İŞIĞIN DİNAMİK SAPILMƏSİNƏ GÖRƏ KOLLOİD STABİLLİYİ

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İşığın dinamik səpilməsi (İDS) metodu ilə sənaye yağı I-40A, sink sink dialkilditiofosfat (DF11) və funksional əvəzli sulfidlərdən R^1 - S - R^2 (R^1 - $C_4H_9OCOCH_2$- və R^2 - CH_2CH_2X, haradaki X - Cl (1), Br (2), OH (3), -CH_2-C_6H_4-OH (4)) ibarət kompozisiyaların kolloid stabilliyi tədqiq edilmişdir. İşlənmədən əvvəl və sonra kompozisiyanın quruluşu IQ və EPR spektroskopiyası metodları, hissəciklərin "hidrodinamik" diametri və kompozisiyalarda ölçülərinə görə paylanması 278-343K temperatur intervalında Horiba LB 550 analizatorunun istifadəsi ilə müəyyən edilmişdir. İDS tədqiqatlarının nəticələri göstərir ki, qeyd olunan baza yağı və onun əsasında hazırlanmış və funksional əvəzli sulfidlərdən ibarət kompozisiyalar molekulyar sistemlərdir və bu sistemlərdə, onların sink dialkiditiofosfatlı kompozisiyalarında hissəciklərin ölçüsü 10 nm-dən çox deyil. Göstərilir ki, bu kompozisiyaların istismarı onların spektral mənzərəsini dəyişdirir. Yağların istismarı zamanı paramaqnit asfalten təbiətli quruluşlar yaranır. Bu strukturların bir-biri və ətraf mühitin diamaqnit molekulları ilə qarşılıqlı təsiri ölçüləri 100 nm və ondan daha çox ölçüsü olan aqreqatların əmələ gəlməsinə, çökməsinə və nəticədə yağların eksplutasion xüsusiyyətlərinin pisləşməsinə gətirib çıxarır. Kolloidlərin stabilliyinin kompozisiyaların komponentlərinin qarşılıqlı təsir mexanizmindən asılılığı və həmçin işlənmənin onların tərkib və quruluşuna təsiri araşdırılır.

Açar sözlər: *kolloid stabillik, sənaye yağı, sink dialkilditiofosfat, funksional əvəzli sulfidlər, işığın dinamik səpilməsi.*