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SELECTIVE PURIFICATION OF THE OIL FRACTIONS WITH USE OF IONIC LIQUID AS EXTRACTANT ON THE BASIS OF N-METHYL PYRROLIDONE

[1]Minaver J.Ibrahimova, [1]Vagif M.Abbasov, [1]Sayara G.Aliyeva, [1]Asadulla B.Khalilov, [2]Gusein Dzh.Guseinov, [1]Vagif A.Nagiyev, [3]Arzu E.Alizada, [1]Sabina A.Seidova

[1]Y.H.Mammadaliyev Institute of Petrochemical Processes of Azerbaijan National Academy of Sciences, AZ 1025, Baku, 30 Khojaly ave.
[2]Institute of Physics of Azerbaijan National Academy of Sciences, Baku
[3]Interdisciplinary Analytical Center attached to the Presidium of Azerbaijan National Academy of Sciences, Baku, AZ 1143115 Huseyn Javid ave.

e-mail: minaver-ibrahimova@rambler.ru

Abstract

In the paper the results of investigations on selective purification of the various oil fractions with preparation of base of diesel fuel and base oils of various purpose have been presented. The distillates prepared by compounding of ten-degree oil fractions of “Balakhany” and “Neft Dashlary (Oil Rocks)” deposits, oily distillates with viscosity 7.6 mm^2/s and 18 mm^2/s at 100^0C, distillate of diesel fuel produced by Baku ORP n.a. H.Aliyev there have been used as raw materials. On the basis of the obtained results the perspectivity of use of the ionic liquid on the basis of acetic acid and N-methyl pyrrolidone as a selective solvent in the processes of selective purification of these oil fractions has been shown

Keywords: *selective purification, extraction, distillate, ionic liquid, raffinate, extract, oil fractions*

Introduction

Environmental protection is a serious problem of humanity in the XXI century and one of the priority approaches to solution of this problem is the development of new ecologically and economically profitable technologies known as "green chemistry”.

In this aspect, a special attention should be paid to the use of so-called ionic liquids (IL) – melts of some organic salts possessing unique set of physical-chemical properties as alternative for classical organic solvents. Unlike the latter, ILs, as a rule, are characterized by thermal stability, non-volatility, have negligible vapor pressure, are non-toxic, non-combustible and are differed by simplicity of synthesis. Due to these properties they are ecological, i.e., "friendly to the environment", in connection with which during 20 years, the researchers' interest to ILs has grown significantly and consequently a number of works on synthesis, investigation of properties and areas of their application has increased [1-5].

With the rapid development of technology, scale growth of automobile parks the volume of consumption is increased, and also the requirements for quality, exploitation properties of fuels and base oils of various purposes become tougher. In the production process of high-

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qualitative fuels and oils with the aim of decrease of content of the aromatic hydrocarbons, sulphur-containing and resinous compounds in them, one of the main technological stages is the selective purification of the corresponding oil fractions. Usually, the following reagents are used as a selective solvent: furfurol, phenol, sulfolan, N-methyl pyrrolidone, morpholine, etc as a selective solvent, which are toxic, non-ecological, fire- and explosive.

A special attention should be paid to the investigations devoted to the development of ecologically safe methods of purification of the oil fractions with use of ionic liquids as a selective solvent.

The effectiveness of use of ILs as an extractant is based by their good solvent ability and high selectivity in relation to different classes of compounds and easiness of regeneration from composition of extract solution, which is characteristic for selective solvents used in the liquid extraction process.

It is known the use of ILs as extractant in the division processes of alkanes and aromatic hydrocarbons [6-10], ethers of organic acids [11-13], and also in the purification processes of the oil fractions, in particular, diesel fuels, various petrol fractions as well as oily distillates [3,7,14-19]. In particular, in work [18] the results of combined desulfurization of diesel fuel by extraction purification with use of IL extractant and oxidizer of hydrogen peroxide have been presented. At ratio diesel fraction: IL-tetrafluoroborate of N-butylpyridinium: hydrogen peroxide 1:1:0.4, temperature 55°C, test duration for 0.5 h sulphur content in the raffinate is decreased per 78.5-83.3% mass. The authors have showed the possibility of regeneration of ILs and recycling in the extraction process in 4 times.

In another paper, it is concluded that the extraction purification of diesel fractions is more economical than hydrogenation processes [19].

The works carried out at IPCP of Azerbaijan National Academy of Sciences in the field of selective purification of the various oil fractions with use of ILs synthesized by interaction of organic acids and amines of various composition as a selective solvent are of scientific and practical interest.

By carried out systematic investigations there have been developed the conditions of purification of distillate of diesel fuel (DF), petrol of catalytic cracking (PCC), reforming (PR), transformer oil, oily distillates with viscosity 6.89 mm^2/s and 8.5 mm^2/s at 100°C and it has been shown that at selective purification of the investigated oil fractions ILs on the basis of formic acid and aniline or morpholine [20-27] show relatively high extracting ability, which has been apparently connected with higher affinity of their cation-composed parts to aromatic hydrocarbons in simultaneous absence of alkyl or cycloalkyl radicals both in cation and in anion parts.

Experimental Part

In this paper the results of the investigations on preparation of base motor oils of various purpose, base of hydraulic oil AMG-10 and diesel fuel by selective purification of the corresponding oil fractions with use of ILs on the basis of N-methyl pyrrolidone as a selective solvent have been stated.

The selective purification of the investigated oil fractions by ion-liquid extractant has been carried out in glass extractor equipped with mixer, reflux condenser, thermometer and

jacket for heating. The water heated to the certain temperature circulating through ultrathermostat served as heat-barrier. The raw material and necessary quantity of the solvent were loaded into the extractor, after which the stirrer and heating were switched on. The necessary temperature in the system was held by continuous circulation of water heated to the required temperature. After mixing for 0.5-3 hours, the mixture was stood for 30-40 minutes and the raffinate phase was separated from the extract one.

As it was known as raw material for preparation of base of hydraulic liquid (HL) on oil basis widely used in modern aviation technology it is advisable to use paraffin oils.

At IPCP of Azerbaijan National Academy of Sciences the preparation process of hydroliquid AMG-10 by dewaxing of distillates isolated from Azerbaijan paraffin oils with subsequent hydropurification and hydrogenation on industrial catalysts and also sulphuric acid purification of distillate of Balakhany oil with subsequent product neutralization has been developed [26, 27].

The hydroliquid AMG-10 prepared from Balakhany oil on basic physical-mechanical indices is close to hydroliquid Aeroshellfluid-4, now used in hydrosystems of aircraft and can be mixed in any volume ratios, and with HL Aeroshellfluid-1 AC – only volume ratios 1:1.

The developed method of sulfuric acid purification by oleum is multistage, uneconomical due to the loss of hydrocarbon raw materials up to 20-25%, counting per initial distillate, and also is non-ecological as the wastes (acid tar, alkaline water, waste clay, etc.) prepared in a large quantity are not utilizable, lead to rapid corrosion of equipment and formation of a large number of acidic waste waters polluting the environment. The environment is also polluted by sulphur dioxide emissions.

Results and Discussion

Taking into account the above-mentioned one, there were carried out the investigations on selective purification of distillate isolated from Balakhany oil (temperature b.b. 234°C, b.e. 306°C, density at 20°C – 850 kg/m^3, flash temperature – 110°C, kinematic viscosity – 2.31 mm^2/s, at 50°C, congelation temperature – minus 75°C, content of the aromatic hydrocarbons – 17% mass) with use of ILs on the basis of N-methyl pyrrolidone and formic or acetic acid as selective solvent [28, 29].

Varying a ratio of raw material to ILs, extraction temperature, contact time of the components and their mixing rate, the conditions of selective purification of oily distillate AMG-10 to degree of dearomatization 88.2% mass with preparation of base of hydraulic liquid meeting the requirements corresponding to TU 0253-021-46693103-2006 have been determined (Tab.1).

Based on obtained results on selective purification of oily distillate AMG-10 isolated from oil of Balakhany deposit, the purification of distillate of the oil of “Neft Dashlary (Oil Rocks)” deposit has been also carried out with use of ionic liquid-N-methyl pyrrolidone acetate as extractant [29].

The distillate prepared by compounding of ten-degree fractions isolated from this oil was characterized by the following physical-chemical properties: boiling beginning temperature – 222°C, boiling end – 313°C, density at 20°C – 849.3 kg/m^3, kinematic viscosity at +50°C- 2.13 mm^2/s, at minus 50°C- 133.6 mm^2/s, flash temperature – 104°C, congelation temperature – minus 75°C, content of aromatic hydrocarbons –12% mass.

By study of influence of the various factors on process of selective purification with use of this IL as extractant the conditions of one-stage and step-by-step purification, dearomatization of raw material have been determined. It has been shown that in one-phase purification of distillate on the basis of oil from “Neft Dashlary” deposit with use of two-fold excess of ion-liquid extractant in relation to raw materials equal to 2,0:1 mass, at extraction temperature – 60°C and components contact time – 1.5-2.0 h the yield of raffinate with residual content of the aromatic hydrocarbons in the raffinate – 2% mass is 76%. At carrying out of the process at ratio IL to raw material equal to 2,5:1,0 or 3,0:1,0 in these conditions of extraction the complete dearomatization of the raffinate has been reached. The yield of raffinate in this case was 72.3 and 70.5%, respectively.

At step-by-step purification of oily distillate of this IL at equal mass ratio of components and components contact time for 1 h on each stage the yield of the raffinate after two-phase purification was 80.66%. The residual concentration of the aromatic hydrocarbons indicated to their absence in the purposeful product.

Table 1. Base quality of hydraulic oil AMG-10

Indices	Norm on TU 0253-021-46693103-2006	Oil bas AMG-10 After purification of IL on the basis of		On GOST (State Standard)
		Acetic acid	Formic acid	
Density, at 20°C, kg/m^3	no more 850	833.3	835.7	GOST 3800-85
Viscosity, mm ^{2}/s				
at 50°C	no less 2.2	2.25	2.40	GOST 33-2016
at minus 50°C	no more 200	200	190.85	
Temperature, °C:				
boiling beginning	no below 210	223	214	GOST 2177-99
Flash (in opened crucible)	no below 93	126	112.4	GOST 4333-2014
Congelation	no higher minus 72	minus 75	minus 75	GOST 20287-91
Total content of sulfurized, % mass	no more 2	2.0	1.9	GOST 6994-74
Acidic number, mg KOH/g	no more 0.3	0	0	GOST 5985-79
Content, % mass				
mechanical impurities	absent	absent	absent	
Moisture	absent	absent	absent	
Aniline point, °C	no below 76.2	77.9	79	GOST 12328-77
Corrosion test on copper plate	not normalized	sustains	sustains	GOST 2917-76

The IR-spectral analysis of raw material, raffinate and extract on IR-Fourier spectrometer LUMOS (firm BRUKER Germany) within the range of wave frequencies 600-4000 cm^{-1} also indicates to absence of the absorption bands referring to aromatic hydrocarbons (669, 741, 811, 873, 1602 cm^{-1}) observed in the IR-spectrum of raw material in the raffinate. The IR-spectrum of the prepared extract is characterized by availability of the absorption bands referring to deformation vibrations of C-H bond of substituted benzene ring (Fig. 1).

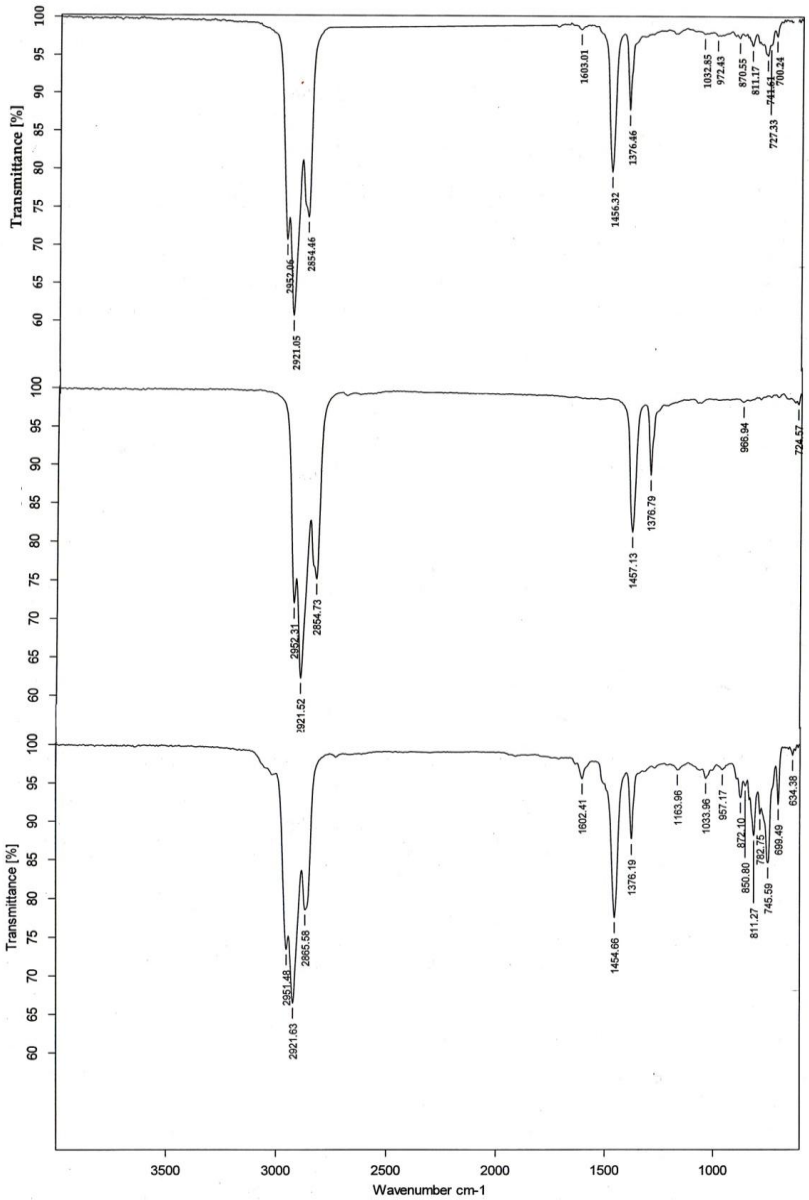

Fig 1. IR-spectra of initial distillate (a), raffinate (b), extract (c) prepared after ion-liquid purification

The physical-mechanical properties of raffinate prepared on the basis of distillate from “Neft Dashlary” oil field are presented in Tab. 2.

Table 2. Physical-chemical indices of base of hydraulic liquid AMG-10 prepared by ion-liquid purification

Indices	Norm TU 0253-0214693103-2006	Oil base AMG-10	Methods of tests
Density at 20°C, kg/m^3	850	839	GOST 3900-85
Kinematic viscosity, mm^2/s, at 50°C, not less at minus 50°C, no more	2.2 220	2.25 187	GOST 33-2016
Fractional composition, °C: boiling beginning , no below boiling end, no higher	 210 315	 225 302	GOST 2177-99
Flash temperature (in opened crucible), °C, no below	93.0	114	GOST 4333-2014
Congelation temperature, °C, no higher	minus 72	does not freeze at minus 75	GOST 20287-91
Aniline point, °C, no lower	78.0	79	GOST 12329-77
Corrosion test on copper plate	not normalized	sustains	GOST 2917-76
Total content of sulfurized, %, mass, no more	1.5	0.0	GOST 6994-74
Acidic number, mg KOH g of product, no more	0.03	0.006	GOST 5485-76
Refracion coefficient, n_D^{20}	–	1.4537	–
Total quantity of sulphur,%	–	0.0257	ASTMD 4294

By analysis on chromatomass spectrometer of mark “Clarus SQ 8T” of production of firm “Perkin Elmer” and “Clarus – 680” the group hydrfocarbon composition of raw material of the base hydraulic oil and raffinate prepared by selective purification by ion-liquid composition has been established [30].

The obtained results are presented in Tab. 3. As is seen, in the composition of raw material-distillate prepared on the basis of oil from “Neft Dashlary” deposit a content of the aromatic hydrocarbons is 27.75% mass, the main part of which (25.52%) falls on the share of disubstituted. A content of cyclic hydrocarbons is 64.93% mass; share of monocyclic hydrocarbons is 64-31% mass. A content of alkanes – 7.14% mass.

It has been shown the possibility of regeneration of ILs of extractant by treatment of extract solution with water and further rectification of the prepared aqueous solution [31]. The IR- and NMR-spectra of IL extractant isolated by this way have been compared with spectra of

synthesized IL and has been confirmed a complete accordance, which allowed the repeated use of IL as extractant.

Table 3. Group hydrocarbon composition of distillate by hydraulic liquid and prepared base hydraulic oil on its basis

Components	Group hydrocarbon composition, % mass	
	Raw material	Raffinate
Aromatic hydrocarbons, including:	27.75	0.0
monosubstituted	0.62	-
disubstituted	25.52	-
trisubstituted	1.61	-
Cyclic hydrocarbons, including:	64.93	87.96
monocyclic	6.93	-
bicycylic	0.61	-
tetracyclic	0.01	-
Alkanes	7.14	12.04

Due to the fact that one of the components, N-methyl pyrrolidone, used in the synthesis process of IL found wide application in the processes of selective purification of the oil fractions [32] the process of dearomatization-distillate isolated from oil of “Neft Dashlary” deposit by N-methyl pyrrolidone have been investigated. The extraction process has been carried out in the analogous conditions: ratio extractant:raw material – 2.5:1.0% mass, extraction temperature – 60°C, component contact time – 2 h and it has been established relatively low yield of raffinate (63% mass against 71% in a case of ion-liquid extractant), a high yield of extract (34%) mass and also availability in the raffinate, residual quantity of aromatic hydrocarbons – 3% mass. The obtained results show relatively low selectivity of N-methyl pyrrolidone in comparison with IL synthesized on its basis, when a high selectivity is one the basic requirements made to extractants.

Perspectivity of use of the ionic liquid on the basis of N-methylpyrrolidone as extractant has been also established by investigation of process of selective purification of oily distillate with viscosity 7.5 mm^2/s at 100°C, and also mixture of oily distillates prepared by compounding of oily distillates with viscosity 7.5 mm^2/s and 18.0 mm^2/s at 100°C at mass ratio 1-4:1 [33].

It has been shown that in a case of use of IL on the basis of formic acid and N-methyl pyrrolidone as extractant, depending on ratio of ionic liquid to raw material 1-2:1% mass, respectively, at extraction temperature 65°C the raffinate yield on the basis of oily distillate with viscosity 7,5 mm^2/s at 100°C is vibrated within the ranges of 92.0-93.0%, viscosity index – 76-76.4, content of total sulphur – 0.0623% mass.

With the aim of expansion of raw materials base of preparation of the base motor oil the possibility of ion-liquid extraction purification of above-mentioned mixture of oily distillates with viscosity 7.5 mm^2/s and 18.0 mm^2/s at 100°C has been investigated.

The characteristics of the base oils prepared by extraction purification of mixture of oily distillates at various molar ratios of distillates are presented in Tab. 4.

Table 4. Characteristics of raffinates prepared by selective purification by ionic liquids of mixtures of oily distillates taken in various mass fractions

Indices	Apparatus	ASTM	Ionic liquid					
			IL-1		IL -2		IL -3	
Freezing temperature, ^oC	Stanhope Seta	D97	-22*	-25	-22*	-24	-22*	-22
Acidic number, mg KOH/g	GOST 5985–79		0.0*	0.17	0.44*	0.32	0.0*	0,24
Refraction index at 20^oC	Abbemat	–	1.4956	1.4955	1.4954	1.4954	1.4938*	1.4937
Flash tempertaure, ^oC	Stanhope Seta	D92	230*	232	236*	222	232*	226
Kinematic viscosity, mm^2/s: at 40^oC at 100^oC	Stabinger SVM	D445	 97.98* 9.74*	 105.07 9.958	 102.44* 9.98*	 82.54 8.54	 114.15* 10.55*	 96.45 9.53
Viscosity index	Stabinger SVM	D2270	70*	64.7	69.3*	65.2	65.3*	67.8
Density at 20^oC, g/cm^3	DMA 4500 M	D5002	0.9028*	0.9013	0.9014*	0.9030	0.9031*	0.9011
Ash content, % mass	–	D482	abs.	abs.	0.01*	abs.	abs.	abs.
Coke content, % mass	Stanhope Seta	D189	1.25*	0.08	0.13*	0.07	0.0*	0.06
Total sulfur content, % mass	SLFA-20	D4294	0.1077*	0.1453	0.1412*	0.1401	0.1138*	0.1032
Color on method of NPA	GOST 20284–74		3.0*	3.5	3*	3	3.2*	+3

Note: IL-1 – on the basis of formic acid and morpholine; IL-2 on the basis of formic acid and N-methyl pyrrolidone; IL-3 on the basis of acetic acid and N-methyl pyrrolidone.
*The mixture of oily distillate has been prepared by compounding of oily distillates with viscosity 7.5 mm^2/s and 18 mm^2/s at 100°C and their mass ratio 1:1, and other composition at ratio 2:1.

As is seen, the raffinates prepared by selective purification of distillates mixture of IL composition independently of composition of oily distillate are characterized by sufficiently high flash temperature (222-236°C), viscosity index (64.7-70) and freezing temperature (minus 22 - minus 25°C).

By systematic investigations carried out at IPCP of Azerbaijan National Academy of Sciences the possibility of preparation of diesel fuel meeting the requirements of European standard by ion-liquid extraction purification has been also shown [34,35]. The best results have been reached by carrying out of selective purification process of IL step-by-step. In this case, a content of the aromatic hydrocarbons in diesel fuel is decreased to ~10.5% mass, sulphur-containing to ~ 0.034% mass.

The possibility of complete removal of the aromatic hydrocarbons from composition of diesel fuel has been shown with use of ion-liquid composition on the basis of acetic acid and N-methyl pyrrolidone as extractant. In particular, by study of influence of extraction temperature, ratio of extractant to raw material, components contact time, the possibility of regulation of

residual content of aromatics and sulphur-containing compounds in the purified diesel fuel has been revealed. It has been established that at extraction temperature 60°C in mass relation of IL to raw material equal 2:1 the residual content of the aromatic hydrocarbons in the raffinate prepared with yield 81.3% mass is 4%, sulphur-containing compounds – 0.0153% against 16% and 0.0181% mass in the initial raw material, respectively.

At extraction temperature 20-25°C and this ratio of components the purified diesel fuel is characterized by complete absence of the aromatic hydrocarbons, the residual content of the sulphur-containing hydrocarbons is 0.013% mass, cetane number – 52.

The realization of selective purification process of diesel fuel step-by-step with use of 1.5 weight parts in relation to raw material of new portion of IL extractant in each stage and contact time 1 h favors more deep fuel purification. A degree of purification from sulphur-containing compounds is 70.55% at residual content 0.0127%, and from aromatic hydrocarbons – 75% mass, residual aromatics content – 4% mass.

It is necessary to note that at ion-liquid extraction a removal of resinous compounds is also observed, the raffinate acquires a transparent color and is characterized by relatively low refraction index and specific weight.

Conclusion

By carried out cycle of the investigations it has been shown the possibility and perspectivity of use of the ionic liquids on the basis of N-methyl pyrrolidone in the selective purification processes of the various oil fractions with the aim of creation of more favorable technologies, which is determining one in the way of environmental protection:

- At extraction purification of distillate prepared by compounding of ten-degree fraction isolated from oil of “Neft Dashlary” deposit with use of two fold excess of ion-liquid extract on the basis of acetic acid and N-methyl pyrrolidone the selective purification of raw materials is achieved with preparation of the raffinate with residual content of the aromatic hydrocarbons 2% mass at carrying out of the process in one stage, at step-by-step purification, a complete dearomatization is achieved.
- The results of extraction purification of oily distillate with viscosity 7,5 mm^2/s, and also mixture of oily distillates with viscosity 7,5 mm^2/s and 18 mm^2/s at various mass ratios indicated to perspectivity of use of the ionic liquids on the basis of N-methyl pyrrolidone and formic or acetic acid as extractant.
- The optimal conditions of practically complete dearomatization of diesel distillate by a method of extraction purification use of the ionic liquids on the basis of N-methyl pyrrolidone and acetic acid as selective solvent with preparation of diesel fuel characterizing by cetane number 52 and residual content of sulphur-containing hydrocarbons 0.013% mass.

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