Extractive Dearomatization of Naphthalene Oil Fraction with Ionic Liquid and -N-Methyl-2-Pyrrolidone

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ABSTRACT

In the article are given an analysis of the results of researches carried out for the purpose of selective treatment of the Naphthalene oil fraction boiling at 260-340°C with ionic liquid (IL) -morfoliphormiate synthesized on the basis formic acid + morpholine and - N-methyl-2-pyrrolidone (N-MP) and as an extractant. The aim is to remove poisonous components - sulfur compounds and toxic carcinogenic polynuclear aromatic hydrocarbons in order to improve the therapeutic properties of Naphthalene oil cut. For this purpose we have used extraction method. On the basis of the conducted researches the role of -N-methyl-2-pyrrolidone and IL-morpholinephormiate in extraction conditions have been determined in selective treatment of Naphthalene oil, according to the results of spectral analysis residual amount of aromatic hydrocarbons decreased from 18.5% wt accordance to 2%, 1.5%. As well as the amount of sulphur decreased from 0.0354% to 0.010%, 0.011% and 0.019%.

In the next stage the treated raffinates with N-methyl-2-pyrrolidone has been purified on silica. In a result amount of aromatic hydrocarbons drastically decrease to be 0.07% and 0.02%. As a result we acquire transparency and at the same time the percentage amount of aromatic hydrocarbons drastically decreased from 2% to 0.07%, 1.5%, to 0.02%. Raffinates obtained in a 2-4 stage dearomatization with N-MP and IL. Raffinate are measured on NMR, UV and IR spectral analysis.

Keywords: Naphthalene oil; dearomatization; extraction; extractant; selective treatment; raffinate; ionic liquid; carcinogenic compound; silica.
1. INTRODUCTION

As it is known there are a number of oil fields both on the land and also in the Caspy sea basin of Azerbaijan. Some of them has a special peculiarities, including Naphthalene oil. Naphthalene oil is differed from other oils in its significancy, therapeutic effect and physico-chemical properties. It doesn't contain paraffin hydrocarbons and petroleum fraction, has a high beginning of boiling point (190-2000C). Naphthalene oil therapeutic effect is connected to the naphthen hydrocarbons contained in it.

Beside the biological active naphthene hydrocarbons also poisonous components in the content of Naphthalene oil were determined, that have negative impact on its treatment properties. These are asphaltenes, resins, naphthenic acids and polynuclear aromatic hydrocarbons, sulfur compounds [1,2]. As mentioned Naphthalene oil contains toxic components and toxic carcinogenic polynuclear aromatic hydrocarbons are considered the most dangerous ones.

So, the most dangerous carcinogens are dimethyltetraphenes, dibenzpirenes, antracenes.

As the result of scientific researches relative carcinogenic activity of eight polynuclear aromatic hydrocarbons have been determined; carcinogenic properties have been detected in 3,4-benzyrene > anthracene > 2,3-benfluorantene > ortophenylpyrene > 11,12- benzfluorantene, and 10,11- benzfluorantene, 1,2 benzyrene and 1,12- benzperylene [3].

But depending on boiling temperature the amount of polynuclear aromatic hydrocarbons in Naphthalene oil fractions varies (from 2000C - up to 4500C). Thus, by increasing of boiling temperature, the amount of aromatic hydrocarbons increases. Benzene, naphthalene, phenantrone, benzofluorene, chryzene, anthracene, 1,2-benzantracene, 3,4-benzphenantrone, pyrene aromatic hydrocarbons were detected in all fractions of naphthenale oil.

As seen, if Naphthalene oil is used naturally for the purpose of therapeutic, it is great importance conducting researches related to its purificaction. Thus, in study of structural-group composition of Naphthalene oil the extensive researches has been carried out, refining technologies from asphaltenes, resins, naphthenic acids, sulphur compounds, mainly polynuclear aromatic hydrocarbons were developed by using acid-contact, hydrogenation, hydrotreatment methods. However, the processes requiring the use of many solvents, adsobents, acid, high pressure, is not efficient ecological and economically and there is not use area of wastes produced from purification processes in industry. So, hydrogenation and hydrotreatment processes accompanied by the loss of high-energy could not be considered the most efficient treatment method of Naphthalene oil.

Should be specifically noted that, as a result of creating perspective treatment technology of therapeutic Naphthalene oil and production of naphthenic hydrocarbons it is possible to use this in the treatment of some diseases (eye diseases, gynecological diseases, burns, open wounds, and nose-throat diseases etc.) and cosmetic industry [4]. In the recent years for purification of oil fraction used environmentally ionic liquid [5-7]. The aim of this work is to study the capabilities of N-methyl-2-pyrrolidone and ionic liquid for the dearomatization and desulphurization of Naphthalene oil fraction by extraction method.

2. EXPERIMENTAL PROCEDURE

In this view we partially realized purification process by using Naphthalene oil fraction ( \( \alpha^2_0 = 0.8900 \text{ g/cm}^3 \), \( \eta^2_0 = 1.4870 \), M-274, amount of aromatic h/c, by sulfurization method, % -18.5) boiling at 260-340°C. We have detected benzene, naphthalene, phenantrone, benzofluorene, chryzene, anthracene, 1,2-benzantracene/3,4-benzphenantrone, pyrene aromatic hydrocarbons in Naphthalene oil fraction boiling at 260-340°C.

Morpholine and formic acids are used for the synthesis of the morpholinephormiate-containing ionic liquid. Physical-chemical properties of the morpholine and formiat acid used for the synthesis of ionic liquid (IL) are compatible to the references.

Morpholine and formic acid boiling points allows high yield synthesis of the corresponding IL. So, there is no synthesis evaporation and as a result of its no loss. Besides the high density 1000.5 and 1219.6 kg/m3 both of two chemicals also testifies to high density of the ionic liquid which will be synthesized from them. Phase separation after the extraction depends on the difference of the densities and higher density of solvent than density of oily fraction is one of the factors leading to enough less residue amount in the raffinate solution.

Laboratory synthesis of morpholinephormiate-containing ionic liquid is carried out as below.

IL synthesis on the basis of formic acid and morpholine is carried out at the same ratio (molar), 60-700C temperature during 1.5 h. At first calculated amount of morpholine (87.12 g) is poured to the reaction flask. Then formic acid (52.94 g) is added on the morpholine gradually by mixing. The formic acid of 85% is used in the reaction.

The fraction boiling above 1350C is used as an IL. Otherwise non-reactive morpholine can be remained at 1350C boiling fraction.
Here is the reaction general scheme:

\[
\text{NH}_2\text{O} + \text{H-O-C=O} \rightarrow \text{H}^+\text{NH}_2\text{O} \quad \text{O=C-}\text{H}
\]

Influences of different factors (temperature, molar ratio of components, reaction duration) on the IL synthesis have been studied and corresponding optimal conditions have been determined. The components same ratios (molar), 60-70°C temperature and 1.5 h duration are considered the optimal conditions for IL synthesis. The optimal yield has been got by different temperature syntheses of morpholine-containing IL synthesized on the basis of formic acid and morpholine. The IL yield obtained at the optimal conditions is 95.07% (mas.). The proses general material balance has been compiled according to the optimal conditions researched. Table 1 presents material balance of obtaining of morpholinephormiate-containing IL:

| Taken:                  | Mass |
|-------------------------|------|
| morpholine              | g    |
| formic acid (85 %)      |      |
| Total                   |      |
| Obtained:               |      |
| IL-morpholinephormiate  | g    |
| water                   |      |
| Total                   |      |

As Table 1 presents, morpholine-containing IL synthesized on the basis of formic acid and morpholine amounts to 95.07% (mas.), but water separated from the mixture – 4.93%. Thus, the water separated from the mixture is the same one in the containing of the formic acid (15%).

It's important to note that synthesized morpholinephormiate-containing IL has higroscopicity. The occurrence of 0.047% (mas.) water absorption has been observed while keeping morpholine-containing IL at 20-25°C in the open air for 90 hours.

IL obtained on the basis of formic acid and morpholine is transparent white viscous liquid.

The morpholinephormiate-containing IL obtained has all quality properties characterized for IL type salts, such as liquid state at very wide temperature limits (-5°C-250°C), low vapor pressure, high and selective solvent ability, thermal stability (more than 300°C).

Low viscousity of morpholinephormiate-containing IL provides ideal mixing with oil fractions during extraction. This peculiarity causes its easier connection with the aromatic hydrocarbons and sulfur compounds in the containing of oil fractions during extraction processes.

| Properties                  | Values of properties |
|-----------------------------|----------------------|
| Density at 20°C, kg/m³      | 1135                 |
| Coefficient $n_D^{20}$      | 1.4850               |
| Boiling point, ºC           | -                    |
| Melting point, ºC           | -5                   |
| Critical point, ºC          | 426                  |
| Critical pressure, Mpa      | 5.24                 |
One of the important requirements for the solvents is a safety problem that is met positively in morpholinephormiate-containing IL.

Morpholinephormiate-containing IL obtained on the basis of N-methyl-2-pyrrolidon and formic acid + morpholin is used as an extractant for the purification of Naphthalene oil. For this purpose extraction of selected extractants – N-methyl-2-pyrrolidon (N-MP) and morpholine-containing IL is carried out by Naphthalene oily fraction. For the definition of the process optimal conditions intensive mixing of the components is carried out stage-by-stage in three-necked flask at room temperature and 60°C by N-MP, but at 50-90°C by IL-morpholiniformiat, by N-MP: Naphthalene oil fraction (at room temperature, 20°C – from 1:0.5 1:2.5 mas.; 60°C – from 1:0.5 1:3 mas.), and at different ratios by IL-morpholinephormiate:Naphthalene oil fraction (from 1:1 1:2, mas.), by N-MP extractant for 30 minutes contacting duration, by IL-morpholinephormiate about an hour. Then the mixture obtained is kept in an alternative funnel until absolute separation of raffinate and extractant solutions phases. The bottom layer is the extract-, but the top layer is raffinate solutions.

The raffinate and extract quality properties and structural-group compositions are studied by the modern physical-chemical analysis methods after the separation of them from the solvent by corresponding methods.

It is carried out NMR-, UV- and IG-spectral analyses of the raffinate obtained by the fraction purification through preliminary and selecting extractant – N-methyl-2-pyrrolidon. Besides, the raffinate purified through the extractant - N-methyl-2-pyrrolidon has been passed through the silica produced by Sigma-AldRICH Company of Germany.

UV-analyses have been carried out by UV 6850 UV/Vis (Jenway company) spectrophotometer, at 190-350 nm spectral area, room temperature. The samples spectra have been taken in different concentrations (c₁=10.75g/ml, c₂=2.52 g/ml, c₃=0.40 g/ml) quartz bathtubs of 1.9 cm thickness, benzene and polycyclic aromatic hydrocarbons amounts have been defined by the help of iodoxane solvent.

IR-spectrum of the fraction boiling at 260-340°C has been taken at 600-4000 cm⁻¹ wavelength range by ALPHA IR-Furye spectrometer of BRUKER company of Germany and the following absorption bands have been observed:

![Figure 1. IR-spectrum of the fraction boiling at 260-340°C](image_url)
NMR spectra have been taken by Furye spectrometer (production of “Bruker” company) of 300.18 MHs frequency working in pulsed mode at room temperature. Deuterium acetone has been used as a solvent.

### 3. RESULTS AND DISCUSSION

Results of selective purification of 260-340°C fraction of Naphthalene oil by N-MP and IL are presented in Table 3-5.

#### Table 3. Stage-by-stage selective purification of Naphthalene oil by N-methyl-2-pyrolidon as an extractant (at 20°C temperature, every stage duration is 30 minutes)

| Oil | Mass ratio feedstock:extractant | Stage number | Average molecular weight | Extractant | Yield, % | Refrinate | Coefficient, η₂₀ | Density, g/cm³ | Kinematic viscosity, mm²/s | Sulfur | Aromatic h/c, % |
|-----|---------------------------------|--------------|--------------------------|-------------|---------|-----------|----------------|----------------|-----------------------------|--------|----------------|
| Oil | N-methyl-2-pyrolidon            | Stage number | Average molecular weight | Extractant | Yield, % | Refrinate | Coefficient, η₂₀ | Density, g/cm³ | Kinematic viscosity, mm²/s | Sulfur | Aromatic h/c, % |
| 1   | 0.5                             | 1            | 272                      | 54.34       | 93.83   | 1.4760    | 0.8828         | 5.14           | 0.022                       | 12     |                |
| 1   | 1                               | 2            | 272                      | 196         | 84.00   | 1.4760    | 0.8827         | 5.20           | 0.017                       | 10     |                |
| 1   | 2                               | 4            | 272                      | 223.99      | 76.01   | 1.4750    | 0.8825         | 5.28           | 0.014                       | 0      |                |
| 1   | 2                               | 2            | 270                      | 223.83      | 76.17   | 1.4740    | 0.8815         | 5.35           | 0.010                       | 0      |                |
| 1   | 2.5                             | 5            | 268                      | 190.1       | 59.90   | 1.4719    | 0.8807         | 5.40           | 0.012                       | 0      |                |

#### Table 4. Stage-by-stage selective purification of Naphthalene oil by N-methyl-2-pyrolidon (at 60°C temperature, every stage duration is 30 minutes)

| Oil | Mass ratio feedstock:extractant | Stage number | Average molecular weight | Extractant | Yield, % | Refrinate | Coefficient, η₂₀ | Density, g/cm³ | Kinematic viscosity, mm²/s | Sulfur | Aromatic h/c, % |
|-----|---------------------------------|--------------|--------------------------|-------------|---------|-----------|----------------|----------------|-----------------------------|--------|----------------|
| Oil | N-methyl-2-pyrolidon            | Stage number | Average molecular weight | Extractant | Yield, % | Refrinate | Coefficient, η₂₀ | Density, g/cm³ | Kinematic viscosity, mm²/s | Sulfur | Aromatic h/c, % |
| 1   | 0.5                             | 1            | 266                      | 0.2         | 99.8    | 1.4819    | 0.8800         | 5.14           | 0.036                       | 8      |                |
| 1   | 1                               | 2            | 263                      | 1.46        | 98.54   | 1.4788    | 0.8818         | 5.18           | 0.028                       | 6      |                |
| 1   | 2                               | 4            | 265                      | 27.98       | 72.02   | 1.4732    | 0.8796         | 5.21           | 0.020                       | 0      |                |
| 1   | 2                               | 2            | 254                      | 32.44       | 67.56   | 1.4731    | 0.8742         | 5.26           | 0.012                       | 0      |                |
| 1   | 3                               | 5            | 258                      | 190.1       | 64.57   | 1.4730    | 0.8759         | 5.35           | 0.011                       | 0      |                |
Table 5. Selective purification of Naphthalene oil by IL – morphinephormiate (3 staged, every stage duration is 60 min.)

| Mass ratio feedstock: extractant | Temperature °C | Average molecular weight | Yield, % | \( \eta^{0.20} \) | \( d_{40}^{20} \) | Kinematic viscosity, 40 °C mm²/s | Sulfur | Aromatic h.c. by sulfurization method |
|---------------------------------|----------------|--------------------------|----------|----------------|--------|---------------------------------|--------|-----------------------------------|
| Oil                             | Morphinephormiate | Extractant                | Raffinate|                |        |                                 |        |                                  |
| 1:1                             | 50             | 250                      | 10.49    | 89.51          | 1.4781 | 5.01                            | 0.8749 | 0.022                            | 6      |
| 1:1                             | 60             | 250                      | 14.06    | 85.94          | 1.4778 | 5.28                            | 0.8750 | 0.014                            | 8      |
| 1:2                             | 60             | 250                      | 16.08    | 83.92          | 1.4761 | 5.19                            | 0.8751 | 0.013                            | 6      |
| 1:1                             | 70             | 250                      | 15.76    | 84.24          | 1.4770 | 5.20                            | 0.8751 | 0.020                            | 8      |
| 1:2                             | 70             | 250                      | 17.32    | 82.68          | 1.4781 | 5.07                            | 0.8751 | 0.019                            | 2      |
| 1:1                             | 80             | 250                      | 14.83    | 85.17          | 1.4781 | 5.10                            | 0.8751 | 0.024                            | 4      |
| 1:2                             | 80             | 250                      | 15.45    | 84.55          | 1.4775 | 5.20                            | 0.8751 | 0.018                            | 8      |

There are not aromatic hydrocarbons in the last 3 samples of Table 3 and Table 4 (sulfurization method, 98.6% H₂SO₄).

We have taken IR-, UV- and NMR-spectra of 5 staged raffinates at 1:2.5 from Table 3 and 5-staged raffinates at 1:3 from Table 4. IR-spectrum of 1:2.5 sample (Table 3) is presented on Figure 2, and 1:3 sample – on Figure 3.

**Figure 2.** IR-spectrum of Naphthalene oil fraction purified at room temperature by N-MP

**Figure 3.** IR-spectrum of Naphthalene oil fraction purified by N-MP at 60°C
IR-spectra of Naphthalene oil fraction (boiling at 260-340°C) raffinate purified through selective extractant and adsorbent have been studied and determined that there isn’t absorbent bands characteristic for aromatic hydrocarbons (1604 cm⁻¹) on the raffinate spectrum. We have also carried out UV-spectral analyses of these samples. We have observed mono- and polycyclic aromatic hydrocarbons corresponding to benzenes 1.57% and 0.94%; naphthalenes – 0.39% and 0.33%; phenanthrenes – 0.07% and 0.11%; benzophularenes – 0.02% and 0.03%; chrizenes – 0.05% and 0.07%; anthrenes – 0.007% and 0.01; 12 benzantrasenes / 3.4 benzphenantherenes – 0.02% and 0.05% in the sample (Table 3 and 4) purified by the extractant – N-methyl-2-pyrrolid. Percentage amount of the above listed is 2.13% and 1.5%. Only mononuclear aromatic hydrocarbons (benzenes – 0.07% and 0.02%) are observed after the purification by an adsorbent.

As a result we have taken NMR-spectra of Naphthalene oil fraction preliminary and after purification samples (Table 3 and 4). The most important parameters values of hydrocarbon composition observed on NMR-spectra are presented in Table 6.

Physical-chemical and NMR-spectroscopy characteristics of the raffinate of Naphthalene oil fraction boiling at 260-340°C, obtained after the purification by selective solvent – N-methyl-2-pyrrolidon (N-MP) and silica are presented in Table 6.

**Table 6.** Physical-chemical and NMR-spectroscopic characteristics

| Fraction Number | Average molecular weight | Hydrogen distribution for the structural groups, % | The number of the rings | Carbon atoms number in molecular fragments, % | Aromaticity |
|-----------------|--------------------------|-----------------------------------------------|-------------------------|---------------------------------------------|-------------|
|                 | H⁺ | H₋ | Hγ | Kₐ | K_saturated | Cₐ | C_N | C_alk | f_α |
| I               | 292.0 | 2.8 | 3.2 | (15.5) | 35.9 | 42.6 | 0.40 | 3.73 | 17.0 | 58.6 | 24.4 | 0.17 |
| II              | 275.0 | 1.2 | 2.1 | (17.0) | 34.2 | 45.5 | 0.05 | 3.29 | 2.0 | 73.3 | 24.7 | ~0.02 |
| III             | 249.0 | 0.5 | 0.9 | (19.6) | 38.8 | 40.2 | - | 3.07 | 0.07 | 77.1 | 22.9 | 7.10⁺ |
| IV              | 246.5 | 0.2 | 0.7 | (20.3) | 38.1 | 40.7 | - | 3.09 | 0.02 | 79.4 | 20.3 | 2.10⁺ |

H⁺, H₋, Hγ - indices of naphthenic structures are given in the brackets

H innovate, H₋n, Hγ - relative share of hydrogen atoms in the different structural groups (aromatic, alkyl and etc.)

Kₐ and K_saturated - aromatic and saturated cycles number

Cₐ, C_beta, C_alk - carbon atoms share in aromatic, naphthene and alkyl fragments, correspondingly

f_α - aromatities of the fractions

As it is presented, the main part of hydrogen atoms are in the saturated structures of the average molecules, but the rest ~ 2-3% are in aromatic rings in the samples studied by us. And it is compatible to low aromaticity of molecules. The fractions structural parameters have been calculated by the method [8].

This method allows definition of aromatic (Kₐ) and saturated (K_saturated) rings numbers in carbon and hydrogen atoms aromatic (H⁺ and Cₐ), naphthene (C_N) and alkyl (C_alk) structures of the average molecules of the fractions and also the share of terminal methyl groups (Hγ) in alkyl rings of hydrogen atoms.

We have also estimated the share of alkyl fragments (-CH₃, -CH₂, -CH) situated on α and β states in respect of the aromatic nucelars.

The analysis of the results presents that the structure of the fraction average molecule (before the purification) consists of 3 and 4 cyclic naphthene rings (Kₐ=3.73) and it may be imagined as condensed by aromatic structures (Kₐ=0.40). The share of carbon atoms in naphthene (C_N) and alkyl (C_alk) structures consists of ~59% and ~24% correspondingly.

The basic parameters in 1→ II→ III→ IV are changed in naphthene hydrocarbons favor after the purification and they exceed other structural fragment (Cₐ increases from ~58.6% up to ~79.4%).

Aromatic hydrocarbons share is nearly 0 (C_alk=0.02%), but alkyl fragments are reduced (C_alk from 24% to 20%).

Tricyclic naphthenes mostly exceed in the fractions compositions. But tetracyclic naphthenes aren’t more than ~10%.
4. CONCLUSIONS

As a result of the investigations it has been determined that although long contacting duration and wide temperature range of Naphthalane oil with IL-morpholinephormiate, there isn’t any positive impact on the percentage amount of aromatic hydrocarbons in the raffinate. And also percentage amount of aromatic hydrocarbons of the raffinates obtained by the purification of Naphthalane oil fraction through N-methyl-2-pyrrolidon has seriously minimized in comparison with IL-morpholinephormiate. Thus, the raffinates purified by N-methyl-2-pyrrolidon have been studied by NMR-, UV- and IR-spectrophotometer methods. As the affirmative results residue amount of the aromatic hydrocarbons of the raffinates obtained is reduced from 268 down to 249, from 258 down to 246, and percentage amount of aromatic hydrocarbons – from 2% down to 0.07%, from 1.5% down to 0.02%.

As a result of the investigations percentage amount of naphthene hydrocarbons in therapeutic Naphthalane oil fractions samples has increased from 58.6% up to 79.4%, reflecting vitally important and therapeutic properties for human organism.

As the final result we may say that the selective purification and adsorption method studied by us can be considered the most successful and perspective purification method for therapeutic Naphthalane oil. The experimental results proves it visually, too.

1) Naphthalane oil fraction boiling at 260-340°C has been studied by spectroscopic methods (NMR-, UV-, IR) before and after the purification.

2) It has been determined that as carcinogenics it’s possible to minimize aromatic hydrocarbons amount in the fraction using these methods.

3) The results allow wide usage of purified Naphthalane oil fraction samples for medicine and cosmetics industry.

Thus, we produce environmentally-friendly, non-waste, having high therapeutical properties white Naphthalane oil without using any catalyst, pressure, adsorbent and acid by energy-saving methods.

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