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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

DEVELOPMENT OF TECHNOLOGY FOR THE PROCESS OF EXTRACTIVE DEAROMATIZATION OF DISTILLATES OF NAPHTHALAN OIL

Specialty: 2314.01 - Petrochemistry

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GENERAL DESCRIPTION OF THE WORK

Relevance of the topic: Naphthalan oil is one of the rare oil fields in Azerbaijan, which has long been widely used in many fields of medicine. At the end of the 19th century, therapeutic properties of Naphthalan oil found out. Already in the middle of the 20th century, Azerbaijani scientists established that the therapeutic properties of Naphthalan oil are closely related to its chemical composition. For the first time, acad. Yu.H.Mammadaliyev hypothesized that the therapeutic properties of Naphthalan oil are associated with the biologically active naphthenic hydrocarbons contained in it. By the studies conducted on Naphthalan oil, acad. Yu.H.Mammadaliyev discovered that it contains aromatic compounds, sulfur, resinous compounds, naphthenic acids and very small amount of paraffin hydrocarbons. He found out that aromatic compounds contained in Naphthalan oil has a negative impact on the properties of naphthenic hydrocarbons and its therapeutic properties[1].

But, the most important requirement of the contemporary period is to minimize the amount of aromatic compounds using the most effective methods without polluting environment and the simultaneously production of highly yield products. That is why, the purification technology of Naphthalan oil distillates has actuality both from a scientific-practical and ecological points of view and it is necessary to develop a new scientific research and create favorable technologies in order to eliminate aromatic and sulphour compounds from them. The presented dissertation has studied the modern methods for structural-group composition of the distillates obtained from Naphthalan oil mixtures extracted from various wells and purification process of corresponding distillates from aromatic and sulfur compounds has been studied experimentally using environmentally friendly selective extragents. On the bases of the results, the most effective solvent has been selected and presented for the development of Naphthalan oil distillates refining technology

[1] Мамедалиев. Ю.Г. К теории механизма действия нафталанской нефти // - Баку: Известия Академии Наук СССР, - 1946. № 5, - с. 560-562.

and the calculation of material balance of the process.

Object and subject of research. By extraction methods, purification of Naphthalan oil from aromatic hydrocarbons, sulfur compounds with environmentally friendly solvents and presentation of the conceptual technological scheme of oil production "White Naphthalan".

Purpose and objectives of work. The purpose of the dissertation work is obtaining distillates from therapeutic Naphthalan oil mixtures and studying percentage changes of naphthenic hydrocarbons, aromatic, sulfur, oxygen and nitrogen compounds in the content of them depending on the boiling points and simultaneously improving the properties of therapeutic “White Naphthalan” oil suitable for use in cosmetics, perfumes and medical fields by releasing aromatic and sulfur compounds, and providing an environmentally and economically efficient block scheme for purification technology.

The objectives of the research are: studying physical and chemical properties of the initial sample and its distillates by modern methods and carrying out spectral analyses; using some solvents for the determination of the most efficient extractant for the purification of NO distillates by extraction; using ionic liquids synthesized on the bases of various amines and formic acid and selective extragent N-methyl-2-pyrrolidone which applied widely in the oil refining industry as extractants[2]; application of extraction method to Naphthalan distillates using IM and NMP solvents; studying the raffinates and extract solutions by modern methods; determining residual aromatic and sulfur compounds amounts in the raffinate (the raffinate has been purified under optimal conditions by the most efficient solvent selected in the result of extraction process) by modern methods; purification of Naphthalan distillate under various temperature conditions and volume ratios; adsorption of the mononuclear aromatic hydrocarbons remaining in the raffinate

[2] Ангели, Е.А. Расширение сырьевой базы для производства минеральных масел / Е.А.Ангели, А.Р.Ханов, Л.А.Насырова [и др.] // Нефтегазовое дело, - Москва: - 2018. №4, - с. 61-83

refined under optimal conditions and spectral analysis; presentation of a block scheme of the technology for purification of Naphthalan distillate and the calculation of material balance of the process.

Research methods. Naphthalan distillates were studied by Direct Inlet Probe-High Resolution Mass Spectrometry (DIP-HR-MS), gas chromatography-mass spectroscopy (GC-MS), ultraviolet (UV), infrared (IR), nuclear magnetic resonanse (NMR) methods. Besides of, were investigated the physical-chemical properties of initial and purified raffinates with standard methods (GOST və ASTM D).

The main provisions submitted to the defence:

- obtaining and analysis of distillates from native Naphthalan oil taken from various wells;
- purification of Naphthalan distillates with ionic liquid extractant by extraction at different mass ratios and temperature conditions;
- purification of Naphthalan distillates using N-methyl-2-pyrrolidone extractant at different mass ratios, temperature conditions, also at room temperature by extraction method;
- influence of contact durations, temperature, amount of extractant on the output and quality of rafined distillate by extraction process.
- selection of an effective extractant in extraction processing and an investigations with spectral analysis of the "White Naphthalan" oil non-sulfonated aromatic hydrocarbons quality obtained under optimal conditions;
- eliminate by adsorption methods of non-sulfonated aromatic hydrocarbons in "White Naphthalan" oil obtained under optimal conditions;
- presentation of a block scheme of obtained technology of therapeutic"White Naphthalan oil" and calculation of material balance of the process.

The scientific novelty of the work. For the first time, ionic liquids and N-methyl-2-pyrrolidone have been used for purification of Naphthalan distillates from aromatic hydrocarbons and sulfur compounds and on the bases of the researches, it has been proved

that N-methyl-2-pyrrolidone is an effective extractant.

-In the content of the distillate of Naphthalan oil boiling at 200-450°C, the amount of aromatic hydrocarbons (by UV spectral analysis – 22.26% mas.) and sulfur compounds (by ASTM D D4294-16e1 methods are 423 ppm) only at room temperature by extraction using extractant N-methyl-2-pyrrolidone was minimized (non-sulfonated aromatic hydrocarbons are 1.56% mas., sulfur compounds are 100 ppm in the absence of a catalyst, pressure, temperature or physical exposure;

- As a result of adsorption of the obtained "White Naphthalan" oil purified with N-methyl-2-pyrrolidone extractant, on silicagel, the content of non-sulfonated aromatic compounds reached 0.02% mas. (according to NMR and UV spectroscopy data), and no sulfur compounds were detected;

- At result is obtained a high-yielding therapeutic "White Naphthalan" oil (72.52%) both from a scientific, practical and from an environmental points of view;

- The block-scheme of "White Naphthalan" oil obtaining technology is presented.

The theoretical and practical value of the research.

- The technology of purification of Naphthalan distillates with N-methyl-2-pyrrolidone extractant is effective in terms of ecology and is not dangerous for the environment.

- Obtained in the purification processes the extract N-methyl-2-pyrrolidone after distillation under vacuum condition has been useful same effective for reuse 3-4 times.

- A high-yielding "White Naphthalan" oil obtained the base of Naphthalan distillates by purification of aromatic carbohydrogens and sulfur compounds using extraction and adsorption processes can be easily applied in many fields of medicine, as well as for the treatment of nose-throat diseases, skin diseases, gynecology, etc. diseases.

- Technology block scheme presentation based on the scientific results obtained on the dissertation work can be useful in future for the oil chemistry and petroleum refining plants.

Approbation of the work:

25 scientific papers were published on the results of the dissertation including 1 Azerbaijan patent, 10 articles (2 single authored) and 14 abstracts were represented in republican and international conferences.

The main results of the dissertation work were presented at international and national conferences: Akad. Ə.M.Quliyevin 100 illik yubileyinə həsr olunmuş respublika elmi konfransı (Bakı, 2012); VIII Бакинская международная Мамедалиевская конференция по нефтехимии (Баку, 2012); Шестая Всероссийская научно-практическая конференция. "Добыча, подготовка, транспорт нефти и газа" 24-26 сентября (Томск, 2013); Ulu Öndər Heydər Əliyevin anadan olmasının 90 illik yubileyinə həsr olunmuş gənc tədqiqatçiların beynəlxalq elmi konfransi. Qafqaz universiteti. (Bakı, 2013); Международная Научно-Техническая конференция ”Ресурсо-и энергосберегающие, экологически безвредные композиционные материалы” (Ташкент, 2013); Akad. S.C.Mehdiyevin 100 illiyinə həsr olunmuş elmi-praktiki konfrans (Bakı, 2014); Ümummilli lider H.Əliyevin anadan olmasının 91 illiyinə həsr olunmuş III Respublika elmi konfransının materialları. Bakı Dövlət Universiteti (Bakı, 2014); Müasir biologiya və kimyanın aktual problemləri” mövzusunda elmi-praktiki konfrans. Gəncə Dövlət Universiteti (Gəncə, 2014); Azərbaycan Respublikasının Neftçilər gününə həsr olunan "The role of multidisciplinary approach in solution of actual problems of fundamental and applied sciences (Earth, Technical and Chemical)" mövzusunda gənc alim və mütəxəssislərin I Beynəlxalq Elmi Konfransı. (Bakı, 2014); Ümummilli lider H.Əliyevin anadan olmasının 92-ci ildönümünə həsr olunmuş “Müasir biologiya və kimyanın aktual problemləri” mövzusunda elmi-praktiki konfrans (Gəncə, 2015); International Turkic World Conference on Chemical Sciences and Technologies”, (Bosnia and Herzegovina, Sarajevo. 2015); IX Бакинская Международная Мамедалиевская конференция по нефтехимии (Bakı, 2016); Akademik Toğrul Şaxtaxtinskinin 90 illik yubileyinə həsr olunmuş Respublka Elmi konfransı. (Bakı, 2015); Neft-Kimya Prosesləri İnstitutunun 90 illik yubileyinə həsr olunmuş “Müasir kimyanın aktual problemləri” mövzusunda Beynəlxalq Elmi

Konfrans (Bakı, 2019).

The organization where the work was performed in. The studies presented in this dissertation were carried out according to the program of scientific research of the Institute of Petrochemical Processes of ANAS 9/2009, in 2011-2013 years (State registration number 0106Az00017).

Personal contribution of the applicant. Scientific research on the topic of the dissertation, the experimental research and analysis, design of the dissertation had been directly by the applicant.

Scope and structure of the work: The dissertation printed on 180 pages, consists of an introduction part and 5 chapters: - literature review, research methods, physical methods applied researches and their discussion, introduction of block scheme, the final results, a list of 194 references, a list of abbreviations, including 49 tables, 46 figures, 8 scheme, 2 diagrams and 2 graphs. The total volume of the dissertation is 191682 with the exception of tables, graphs, figures, scheme, diagrams and references.

In the introduction part of the dissertation the actuality of the problem is substantiated, the purpose and responsibilities of the research, research methods, main provisions of the defense, scientific novelty of the work, theoretical and practical significance of the research, approbation and application, name of the organization where the dissertation work is performed and is noted informations about on the volume of the dissertation.

The first chapter was touched to the connection with the well-known issues of purification of various petroleum products using traditional solvents, the physicochemical properties of the purification method, the physicochemical properties of polar and non-polar solvents, the purification of petroleum products with ionic liquids and the application of the obtained distillates.

The second chapter is discussed of the compositions and the most important physicochemical properties of initial materials, applied physicochemical analitic methods, used equipment during carry out, as well as curried methods of experiment.

The third chapter Naphthalan oil and its distillates the study structure and properties of phiysical methods were reviewed.

The fourth chapter is devoted to determining the optimal conditions for the purification of Naphthalan oil distillates by ionic liquid compositions, the effect of contact time, temperature, amount of solvent on the extraction process, qualitative indicators of raffinate and extracts and analysis of their hydrocarbon group by modern physicochemical research methods.

The fifth chapter is discussed the purifications of therapeutic Naphthalan oil distillates with selective extractant N-methyl-2-pyrrolidone in 40-90^{o}C temperature intervals, the stages extractions at room temperature of Naphthalan oil distillate boiling at 200-450^{o}C with extractant N-methyl-2-pyrrolidone, the analysing of “White Naphthalan” oil with modern specral methods, the adsorbsion of non-sulfonated aromatic hydrocarbons in the content of “White Naphthalan” oil obtained at optimal conditions, the presentation block-scheme of of “White Naphthalan” oil obtaining technology and calcullation material balance.

THE MAIN CONTENT OF THE WORK

1. Initial raw materials, research areas, the equipments and rules for carrying out the tests

A mixture of Naphthalan oil (NO) extracted from different wells has been used for the researches. The analysis of physical and chemical properties of the NO mixture by various methods has been followed by the distillation on the device A-2 (GOST 11011-85) and obtaining the distillates boiling at different temperature ranges. Percentage amount of the hydrocarbons and heteroatomic compounds in the content of the distillates due to the increase in their boiling temperatures (260-300°C, 300-340°C, 340-400°C, 400-450°C, 450-500°C) has been determined by co-use of Direct Inlet Probe-High Resolution-Mass Spectrometry (DIP-HR-MS) and GC-MS (gas chromatography-mass spectroscopy) for identification increasing intensity (5°C distillates boiling at 260-340^{o}C) of naphthenic hydrocarbons. The main parameters for the distillates (260-340, 210-390 və 200-450^{o}C) and purified samples have been confirmed by NMR, IR, UV spectral analyses and approved by modern standard methods. It was determined that aromatic hydrocarbons (AH) in distillates boiling at 260-340, 210-390 and 200-450°C were 18.86%, 20%, 22.26% mas., and sulfur compounds (SCs) were 354, 374 and 423 ppm respectively The reagents used for the purification process were the ionic liquids (IL) synthesized on the bases of various amines, formic acid and N-methyl-2-pyrrolidone (NMP) extractant. Critical solution temperature (CST) of NO distillate, boiling at 260-340 ^{o}C with IL of anilineformiate, morfolineformiate and NMP extractant has been calculated (the upper critical solution temperature for NMP extractant has been as 167^{o}C), the upper critical solution temperature for NMP extractant has been determined as 167^{o}C), extraction and adsorption processes have been realized. At first, extraction process has been carried out for releasing aromatic and sulphurous compounds from the content of the distillates.

Effect of contact time, temperature condition, mass ratio of Naphthalan distillate (ND) of the extractant on the extraction

process, the yield of the purified raffinate, its main parameters during the extraction process has been studied by contemporarary methods. The extraction has been carried out for 0.5-3 h of contact time, at 1:1-3 mas ratio of ND:extractant in 1 stage and each stages at 1:0.5 ratio of ND:extractant 90-60°C and room temperature. NMP has been selected as the most efficient extractant in obtaining "White Naphtalan" oil from ND boiling at 200-450°C as a result of the researches. Adsorption process has been conducted in order to elimunate of non-sulfonated aromatic hydrocarbons the "White Naphthalan" oil obataining on the basis of NMP extractant from ND and a block-scheme of the "White Naphthalan" oil obtainig technology has been presented.

2. The results of the studies carried out on the extraction processes of Naphthalan distillates by ionic liquids

For the first time, ionic liquid compositions (morpholineformiate, diethylamineformiate, triethylamineformiate, piperidineformiate, pyperidineformiate, anilineformiate, N-methyl-2-pyrrolidoneformiate) have been used for NO distillates extraction. For this purpose, the extraction process has been carried out at 1:3 mas ratio of ND:IL, 80°C, 1h of contact time in a single stage. The researches have revealed, that ionic liquids of diethylamineformiate, triethylamineformiate, piperidineformiate have low selectivities in extraction process and the obtained raffinate contains 22 mas.% of sulfonated aromatic hydrocarbons (AH) residue amount and 306, 311 and 296 ppm of sulphurous compounds (SC), correspondingly. This can be clearly seen from the yield extracts obtained during purification (16.3; 17.9; 19.5% mas. correspondingly). However, compared to the primary raw material, the amount of AH (22.26%) decreased by only 2-4 mas.%. This means that the solubility of these solvents is relatively high and selectivity low.

But the ionic liquids of piperidineformiate, anilineformiate, morpholineformiate have had higher selectivity and AH residue amount of 10%, 14%, 16 mas.%, correspondingly and SC of 216, 264, 259 ppm. The results showed that the minimum amount of AH

(10%) was raffinate purified with piperidineformate ILs. At the same time, the residual sulphur content in the obtained raffinate decreased in comparison with other raffinates and amounted to 216 ppm, the refraction index was 1.4925, and the color was 2 points. There was also a sharp decrease in the density of the obtained raffinate (888.2 kg/m^3). It should be noted that aromatic hydrocarbon compounds have the highest density among hydrocarbon compounds. This indicates that the amount of AH at the NO raffinates was significantly lower than at other raffinates (10%). Thus, the content of carcinogenic aromatic hydrocarbons required to be removed from the raw material was 22 mas.%, the amount of sulfur compounds was 423 ppm, density 914.6 kg / m3, refraction index was 1.5030, color was 8 points.

As is evident from the results, the raffinate purified by the IL of piperidineformate in 62.0% mas. of yield, has minimum amount of AH (10% mas.). Extraction of ND by piperidineformiate IL at 80ºC, 1:3 molar ratio of ND:IL causes passing of necessary components (NH-naphthenic hydrocarbons) into the extractant simultaneously with decreases in AH and SC amount of the raffinate.

Just this allowed us to carry out the next extraction process by IL of piperidineformiate in 1 h of contact time, at 1:1-1:3 mas ratio of ND:IL, 40-80ºC temperature range and 1:2 mas ratio of ND:IL and temperature of 80ºC have been determined as an optimal condition. However, under these conditions, the yield of the extract (33.1% mas) was about 3 times higher than the amount of residual AH (10% mas.) extracted from the raw material (22%).The obtained raffinate yield has amounted to 66.9% mas., residue AH 10% mas (density 889.1 kg/m^3, refraction index 1.4931, color was 2.5 points) has been observed under these condition.

As seen, solubility exceeds selectivity in extraction of ND by IL of piperidineformiate at 80ºC, 1:2 mas ratio of ND:IL. This is undesirable for the extraction process.

Extraction of the NO distillates, boiling at temperature range of 260-340°C and 200-450°C by IL of anilineformiate has been carried out under the condition of 1 h of contact time, at 1:1-1:3 mas ratio of ND:IL at 40-80°C and room temperature. During extraction

process the optimal condition for both of the distillates has been 80°C, mas ratio of ND:IL as 1:3 (260-340°C) and 1:2 (200-450°C). Residue amount of AH in the obtained raffinates has been determined 8 mas.% versus18 mas.%; SC 354 ppm versus 174 ppm; refraction index 1.4878 versus 1.4864; density 890.4 versus 889.0 kg/m^3; colour 3 versus 5 for the distillate boiling at 260-340°C temperature range; and AH 10% versus 22 mas%; SC 423 ppm versus 228 ppm; refraction index 1.5030 versus 1.4932, density 914.6 versus 888.9 kg/m^3; colour 8 versus 4.5 for the distillate boiling at 200-450°C temperature range. The yields of raffinate in these distillates are 85.8% and 87.6% mas, respectively. As can be seen, the anilineformate IL instead of ensuring the complete transfer of AC to the extract solution, but also allowed the transfer of important components (NH) to the solution. This means that the solvent is less selective against AH. The raffinate has been also analyses by IR-, UV-spectroscopy. According to UV spectroscopy, the amount of AH in the raffinate was 10.21% mas.

Extraction of NO distillates boiling at temperature ranges of 260-340°C, 200-450°C and 210-390°C by IL of morpholineformiate has been carried out for 1 h of contact time, at 40-90°C and 1:1-1:4 mas ratio. The studies have been resulted in obtaining 89.1%, 84.9%, 84.0% mas. of the raffinates yields in the distillates boiling at 260-340°C, 200-450°C and 210-390°C under the optimal condition of 80°C - 1:4; 90°C - 1:4; 80°C – at 1:3 mas ratios, correspondingly. In this case, AH residue amount has been determined 8% (9.93% according to UV spectroscopy) versus 18%; 10% versus 22% (10.66% according to UV spectroscopy); 8% versus 20%; and SC 232 ppm. versus 354 ppm; 277 ppm.versus 423 ppm; 207 ppm. versus 374 ppm.

As is evident, temperature range of 80-90°C is considered the optimal condition for a single-stage purification of ND by IL. A solution has amounted 3-4 times more than ND under these temperature conditions. On the other hand, as is seen from the results, the highest yield is achieved although AH in the obtained raffinate is minimum in extraction of ND by IL of

morpholineformiate. It's understood so, that IL of morpholineformiate has more selectivity to AH than the other ILs used for extraction process and naphthenic hydrocarbons (NH) doesn't pass into the extractant. That's why the following studies on NO distillates boiling at temperature ranges of 200-450°C and 260-340°C by morpholineformiate IL have been tested in 2-4 stages under the conditions of 1:0.5-1:1 mas ratios of ND:IL at 40-90°C, contact time for each of the experiments – 0.5-1 h.

By 0.5 h-contact time of every stage, extraction process optimal conditions for the distillates with boiling points of 200-450°C and 260-340°C have been determined as: in 4-stages, 70°C, 1:0.5 mas ratio (AH amount is 8% of 22%, SCs–189 ppm of 423 ppm) the yield is 80.3% mas.; in 3-stages, 60°C, 1:0.5 mas ratio (AH amount is 4% of 18%, SCs – 135 ppm. of 323 ppm.) the yield has been raffinates of 80.44% mas.

But 1 h-contact time for every stage in extraction of an NO distillate boiling at 200-450°C by IL of morpholineformiate causes selection of the raffinate obtained in 4 stages with the yield of 72.65% mas. under the condition of 60°C, at mas ratio of 1:1, 3.23% (according to UV-spectroscopic analysis) amount of AH in the raffinate of 22.26% and 167 ppm. of SC of 423 ppm. have been determined.

Thus, in the process of purification of NO boiling distillate at 200-450ºC, morpholineformate IL behaved more selectively against AH than other ionic liquids.

However, due to the loss of time, a large amount of solvent, and also incomplete purification of aromatic compounds from ND, the next extraction process was carried out with a selective solvent N-methyl-2-pyrrolidone, which is widely used in the world oil industry.

3. Purification of Naphthalan distillates by selective solvent of N-methyl-2-pyrrolidone

N-methyl-2-pyrrolidone has been used as an extractant for

extractive purification of NO distillates from AH and SCs. The NO distillates, boiling at temperature ranges of 260-340°C and 210-390°C have been used for extraction process. At first, the studies have been carried out for 3 h of contact time, in a single-stage, at temperature range of 40-90°C, 1:1-1:4 mas ratios of ND:NMP. The effect of contact time on extraction process has been observed and the obtained raffinates have been studied by modern methods. As a result of the studies, an optimal condition determined for the distillates boiling at 210-390°C and 260-340°C for 3 h of contact time is 90°C, 1:3 – ND:NMP; 80°C, 1:2 – ND:NMP, correspondingly. The distillates raffinates, obtained under these conditions contains: AH residue amount – 4% of 20%, SCs residue amount – 78 ppm of 374 ppm, refraction index 1.4820, colour 1 for the distillate of 210-390°C; AH residue amount is 2% of 18%, SCs residue amount – 74 ppm of 354 ppm, refraction index 1.4811, colour 1; for the distillate of 260-340°C and; and the raffinates yields are 78.0% and 79.2% mas., correspondingly. As a result of the studies, there have been determined, that although the raffinates, purified by NMP extractant of ND contain minimal amounts of AH and SCs, higher temperature and longer contact time has been caused passing a few of NH into the extractant. At first, re-purification of ND has been carried out by decreasing of contact time from 3 h to 1 and 0.5 h. Following studies were carried out by purifying NO distillates boiling at 260-340, 200-450 and 210-390ºC with contact times of 1 and 0.5 hours.1 h of contact time provides the optimal condition for the NO distillate boiling at 260-340°C temperature range as 80°C, 1:3 mas ratio, 2% of AH residue amount, 81 ppm SC, colour 1, refraction index 1.4850, 78.9% mas. of the raffinate yield and the optimal condition for the distillate boiling at 210-390°C as 60°C, 1:2 mas ratio, 6% of AH residue amount, 93 ppm of SC, colour 1.5, refraction index 1.4838 and 81.1% mas. of the raffinate yield. It was found that the reduction of the contact time from 3 to 1 h led to an increase in the yield of obtained raffinate, the amount of SCs , as well as AH. However, it seems that under these conditions, NMP extractant has not lost its selectivity against AH.

The studies have been continued by purification of an NO distillate, boiling at the temperature range of 200-450°C for 0.5 h, at 40-90°C, 1:3-1:4 mas ratios. In this case, effect of contact time, extraction temperature simultaneously with amount of solvent on extraction process has been studied. It has been determined, that although contact time and also AH and SC are decreased to the minimum amounts, higher extraction temperature and solvent amount (it should be remembered, that solvent amount has been taken 3-4 times more than raw material) have caused decrease in the yields of the obtained raffinates. On the other hand, NMP extractant has higher selectivity by lower extraction temperature. So, that 3-4 times more amount of solvent and 40-60°C of extraction temperature causes 6-8% mas. of AH residue amount in the raffinate. In this case, the yield of the raffinates amounts 80.1-78.7 % mas. As is evident, by a single-stage purification of ND by NMP extractant even at low extraction temperatures, AH amount is minimal in the purified raffinates. Staged extraction of NO distillate boiling at the temperature range of 200-450°C has been carried out for the purpose of providing decrease in AH and SC residue amounts in the raffinates to the minimum values simultaneously with loss decrease in the yields of the purified raffinates by the experiments carried out by NMP extractant. Based on the results of numerous studies in the field of purification of therapeutic naphthalene oil distillates, it was determined that the selectivity of NMP extractant exceeds the solubility at low temperatures (60-40ºC). For this purpose, cleaning of ND with NMP extractant at room temperature was carried out in the ratio ND: NMP-1:3-1:4 by weight with contact time of 0.5 hours.

It was found that one-step extraction of NMP solvent 3-4 times more than ND at room temperature reduced the amount of AH in the purified raffinate by 2.78-3.71 times (8-6% by weight by sulfonation), and SC caused a decrease of approximately 3-4 times (~ 120-94 ppm). It should be noted that the amount of SC in the primary raw material was 423 ppm.

At the same time, the yield of refined products was relatively high. (83.3 in the ratio of 1:3 by weight and 81.1% by weight in the ratio of 1:4). Taking into account the reduction of the content of AK

and KB in the raffinate obtained during the single-stage purification of naphthalene distillate with NMP extractant in the ratio ND:NMP-1:0.5, the studies were carried out in stages at 60ºC. For this purpose, extraction processes were continued in 2-4 stages in the ratio of ND: NMP-1:0.5 and 1:1 mass. In this case, the minimum amount of aromatic compounds was found during 4-stage purification in the mass ratio ND:NMP-1:0.5. Thus, in the obtained raffinate AH was not observed by sulfonation, KB - 97 ppm, and the yield of raffinate was 70.16%. As a result of UV-spectral analysis of the obtained raffinate was determined to be non-sulfonated aromatic hydrocarbons -2.13% (NSAH). As can be seen, the residual content of NSAH in the raffinate was quite small during the phased purification. In order to remove NSAH (2.13%) from the raffinate obtained under optimal conditions, it was silicagel in a glass column. UV and NMR spectral analysis revealed that the residual NSAH in the silica gel raffinate was 0.07% against 2.13%, and no traces of SC were observed. As can be seen, the low-temperature stages purification. As is evident, NMP extractant is more selective with against to AH and SC during by a staged purification and low temperature (60ºC). To increase the efficiency of the results obtained, the purification process was carried out in stages at room temperature.

4. Purification of Naphthalan oil distillate boiling at 260-340°C at room temperature by N-methy-2-pyrrolidone extractant

Based on the results of numerous studies in the field of purification of therapeutic Naphthalan petroleum distillates it was found that the selectivity of the extractant NMP (mainly morpholine formate) is higher than the solubility of ionic liquids used in the staged purification process at lower temperatures. For this purpose, purification of ND by NMP extractant has been carried out in 4-stages, for 0.5 h of contact time at 1:0.5 mass ratio of ND:NMP at room temperature (Table 1).

As is seen from Table 1, during extraction process of the Naphthalan distillates by extractant NMP at room temperature by 4 stages purification has maximum effect on the content of the harmful

components (sulfur, aromatic hydrocarbons, acids, etc.) at mass ratio of 1:0.5. So, that SCs (ASTM D4294-16e1) 100 ppm in this obtained raffinate (it should be remembered, that ND has amounted to 423 ppm of SC before purification). Besides, colour index of 1 against 8 (GOST 20284-74), reduction of acid number from 0.72 to 0.03 mgKOH/gr (GOST 5985-79), decrease in refractive index from 1.5030 to 1.4824 (ASTM D5006-96) are the main factors demonstrating a significant decrease in AH amount in the content of the purified raffinate. It is considered the increase in percentage amount of NH in the content of the purified raffinate. The amount of aromatic compounds (recall that in the primary raw material by the method of sulfonation AH-22% mas.) according to the method of sulfonation in the obtained raffinate was not observed. The obtained raffinate have been analyzed by IR, UV, NMR spectroscopies.

Table 1.
A staged purification of Naphthalan distillate by N-methyl-2-pyrrolidone at room temperature (contact time for every stage was 30 min.)

Ratio of components ND:NMP	Extract yield, % (mas.)	Indices of raffinate						
		yield %, (mas.)	Density, at 20°C kg/m^3	Refraction index, n_D^{20}	Colour	Degree desulfirization, ppm	Acid number, mgKOH/gr	Non-sulfonated aromatic hydrocarbons, %
Initial	-	-	914.6	1.5030	8	423	0.92	22
4-stage								
I stage	12.01	87.56	888.2	1.4890	3	226	0.17	12
II stage	4.49	82.57	888.2	14869	2.5	158	0.11	6
III stage	3.42	78.78	887.1	1.4842	1.0	114	0.09	2
IV stage	3.96	73.41	887.1	1.4824	1.0	100	0.03	0

According to UV spectral analysis observed 1.56% NSAH. At this time, the yield of raffinate was 73.41% mas.

Thus, studies on ND have shown that NMP extractant is a more effective extractant than morpholineformate ionic liquid. Table 2 presents the results of a comparative study of the staged purification process by NMP and morpholineformate IL of Naphthalan oil distillates boiling at temperature range 200-450ºC under optimal conditions.

As can be seen, the degree of aromatization of raffinate (AH-3.13%, according to the results of UV-spectral analysis) obtained under optimal conditions during purification of Naphthalan distillate with morpholineformate IL in 4 stages (ND: IM-1:1 mass ratio) at 60ºC, desulfurization rate was 55.32%, acid number was 0.12 mgKOH/gr, and yield was 72.65% mas. The yield of the extract was 24.25% mas.

Table 2

Comparative study of the results of purification of Naphthalan oil boiling at 200-450ºC with ionic liquid morpholinformate and extractant N-methyl-2-pyrrolidone

NO, (gram)	Consumed extractant (grams)	Temperature, ºC	Contact time, h	Indices of raffinate				
				Refraction index, n_D^{20}	Colour	Degree SCs, ppm	Acid number, mgKOH/gr	According to UV-spectral analysis, the amount of AH,%
	-	-	-	1.5030	8	423	0.92	22.26
NO:IL-1:1								
(obtained raffinate from IV stage) 72.65	Total: 335.07	60	4	1.4855	2	167	0.12	3.23
NO:NMP-1:0.5								
(obtained raffinate from IV stage) 73.41	Total: 174.45	Room temperature	2	1.4824	1	100	0.03	1.56

But during the purification of Naphthalan distillate with N-methyl-2-pyrrolidone at room temperature, the degree of

aromatization of raffinate (according to the results of UV-spectral analysis AH-1.56%) obtained under optimal conditions (ND: NMP-1:0.5 mas ratio) was 93%, and the degree of desulfurization was 77%, acid number 0.03 mgKOH/gr, yield 73.41%, extract yield was 23.91% mas.

As can be seen, the total yield of the extract (23.91%) corresponds approximately to the amount of residual aromatic hydrocarbons in the raw material (22.26% by UV spectroscopy).

This means that the yield of therapeutic "White Naphthalan" oil obtained during purification is high (73.41%, Table 1). In addition, the extraction process with NMP extractant was spent 2 hours and the extraction process with morpholineformate ion liquid was spent twice as long - 4 hours. Despite the fact that the amount of NMP (174.45 grams in total) extractant is 2 times less than in morfolineformiate IL the content of aromatic hydrocarbons in the obtained raffinate is 1.56%. As can be seen from the table, was used 335.07 g during 4-stage purification with morpholinformate ionic liquid, 174.45 g during extraction with NMP extractant. This means ~ 50% savings in solvent during Naphthalan distillate extraction with NMP-extractant.

Thus, taking into account the results obtained, it was determined that the extractant NMP is a more promising extractant in the production of the therapeutic "White Naphthalan" oil.

In order to completely remove NSAH from the composition of "White Naphthalan" oil obtained under optimal conditions, it was passed once at room temperature with W.R.GRACE & CO.-CONN silica gel produced by the German company Sigma-AldRICH (model 5183338). At that time, the yield of obtained "White Naftalan" oil was 72.52%, The density obtained refined – 866.7 kg/m^3, refraction index – 1.4731, molecular weight – 240, acid number – 0.001 mgKOH/gr proved by modern standard methods.

The purified raffinat was analyzed by IR, UV, NMR spectroscopy. 1.56% of NSAH in the content of ND raffinate (at mas ratio 1:0.5 of ND:NMP, in 4-staged), purified by NMP has been determined by UV spectral analysis the content of residual

mononuclear aromatic hydrocarbons in silicagel-purified raffinate was 0.02% (Table 3).

As is seen from Tab. 3, ND contains minimum amount of NSAH after the purification and only a few amount of mononuclear NSAH ~0.02% mas. remains in the raffinate.

Table 3.

The results of UV spectral analyses of Naphthalan distillate and its raffinate purified by N-methyl-2-pyrrolidone (raffinat) and silica gel (White Naphthalan oil)

ND	Non-sulfonated aromatic hydrocarbons, %						
	Benzene derivatives	Naphthalines	Phenanthrenes	Benzofluorens	Chrysenes	Anthracenes	Total content of arom. hydro-carbons, %
200-450°C	9.25	6.18	4.61	0.040	0.82	1.36	22.26
Raffinat	1.27	0.16	0.06	0.035	0.026	0.009	1.56
White Naphthalan oil	0.02	-	-	-	-	-	0.02

As a confirmation of UV spectral analysis results, NMR spectra of ND and the "White Naphthalan" oil have been drawn (Fig. 1, 2) and spectroscopic characteristics of the hydrocarbons in them have been set into Tab. 4.

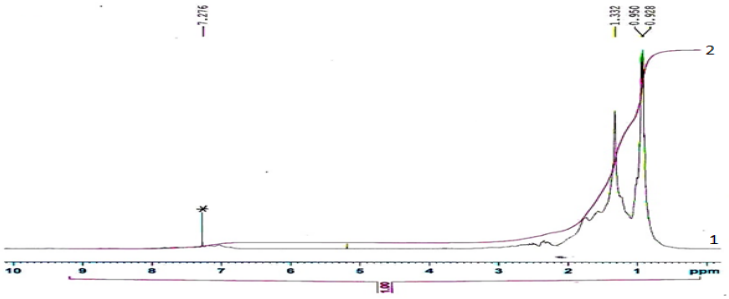


Figure 2. NMR spectrum of Naphthalan oil distillates boiling at 200-450°C

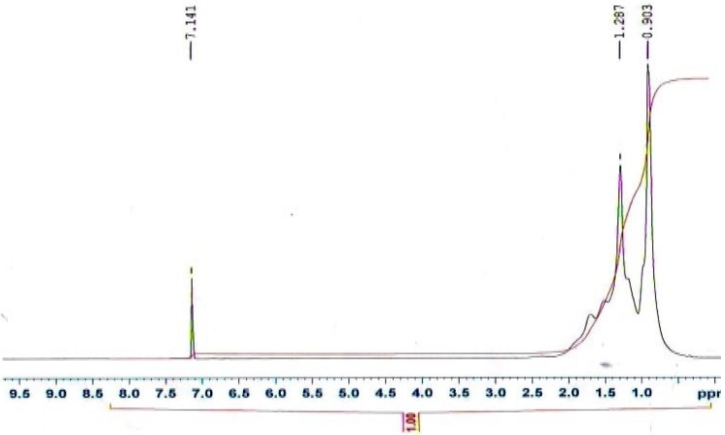


Figure 1. NMR spectrum of White Naphthalan oil

Table 4

NMR structural characteritics of Naphthalan distillate (I), its raffinate purified by N-methyl-2-pyrrolidone (II) and White Naphthalan oil (III)

Samples	Distribution of hydrogen on structure groups, %					Number of rings		The proportion of carbon atoms in molecule fragments, %		Degree of aromati-zation
	H_a	H_{doy}	H_α	H_β	H_γ	K_a	K_n	Naphthe-nic-alkyl	Aroma-tics	f_a
I	4.9	95.1	8.5	(18.5) 40.3	27.8	0.53	3.74	78.0	22.0	0.22
II	1.8	98.2	1.7	(18.8) 32.5	45.2	0.31	3.51	98.5	1.5	1.5×10^{-2}
III	0.2	99.8	0.7	(20.3) 38.1	40.7	-	3.09	99.98	0.02	2×10^{-4}

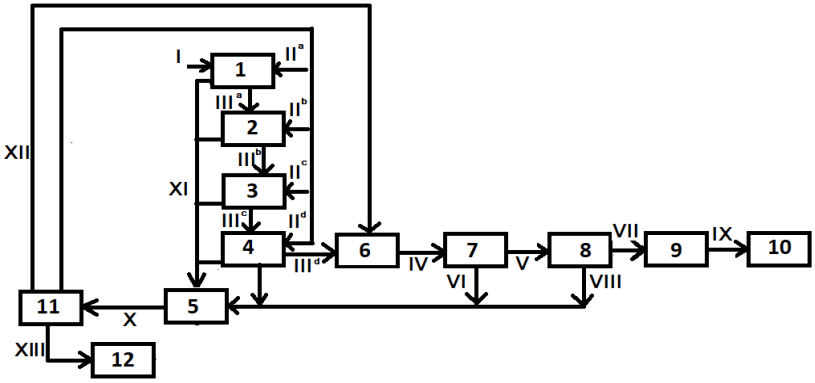
Note: Distillation of I-NO boiling at 200-450°C; II-raffinate; III-White Naphthalan oil

As is evident from Tab.4, saturated structures of average molecules ($H_{sat.}$=98.2-99.8%) are a major portion of hydrogen atoms in comparison with initial raw material, but the rest portion is aromatic hydrocarbon rings (H_a=1.8-0.2%) in the purified samples. It proves low aromaticity degree of purified samples ($f_\alpha=1.5\times10^{-2}$ - 2×10^{-4}).

Simultaneously, it has been determined by NMR spectroscopy, that a share of naphthenic-alkyl hydrocarbons (it should be remembered, that a share of naphthenic-alkil hydrocarbons was 78.0% in the initial raw material) has increased after purification by 20.8 və 21.98% and become 98.5-99.98%. On the bases of the calculations, carried out by NMR spectral analysis, it has been determined, that the major part of naphthenic hydrocarbons consists of tricyclic naphthenes. But the share of tetracyclic naphthenes is not more than ~10%.

This proves higher structural parameters of naphthenic hydrocarbons than the other fragments. The share of aromatic hydrocarbons has been almost zero (0.02%) in comparison to the initial raw material (22.0%).

Thus, taking into account the advantages of the extraction process with NMP-extractant, a scheme for the production of "White Naphthalan" oil has been developed (Scheme 1).



Scheme 1. Block scheme of obtained technology of treatment “White Naftalan oil” from Naphthalan oil distilllates boiling at 200-450°C of temperature ranges

1, 2, 3, 4 - extractors; 5 - device for separating extractant + water mixture from extract 6 - mixing tank provided with distilled water; 7 - device for separating raffinate from the mixture of extractant + water; 8 - device for perecipitating residual water in content of raffinate; 9 - adsorbent; 10 - capacity of “White Naphthalan” oil; 11 – device regenerating NMP-extractant; 12 - NMP reserve; I - line passing the raw material to the system; II^a, II^b, II^c, II^d - lines transmitting the extractant consumed in the 1st, 2nd, 3rd and 4th stages; III^a, III^b, III^c, III^d - line passing raffinate from stages 1, 2, 3 and 4; IV - line passing raffinate + extractant + water mixture; V - line of non-dehydrated raffinate; VI - line passing the extractant + water mixture to the regeneration unit; VII - line for transfer of fully dehydrated raffinate to the adsorbent; VIII - extractant + water separated from raffinate; IX - line for transfer of adsorbed "White Naphthalan" oil to the tank; X - line passing the extractant + water mixture; XI - line passing the extract solution; XII - line passing distilled water to the system; XIII - line providing recovery of extractant consumption.

As a result, we can say that the method of selective purification and adsorption, studied to obtain the medicinal oil "White Naftalan", can be considered the most successful and promising purification technology among the purification methods used today. This is confirmed by experimental results. Thus, no catalyst, without the application of high pressure, and at the same time environmentally friendly technology is realized by the method of extraction. This is also the economic value of the NMP extractant

Thus, the technological block-sheme created on the basis of the research is environmentally effective and ensures the production of the therapeutic “White Naftalan” oil with a yield of 72.52%.

CONCLUSIONS

1. Physicochemical, spectral properties and structures of distillates obtained from mixtures of Naphthalan oils were determined using modern methods of analysis [14, 20, 22].
2. For the first time, to remove aromatic and sulfur compounds from naphthalan oil distillates, a one-stage extraction process was carried out using a number of ionic liquids (morpholineformate, diethylamineformate, triethylamineformate, pyridineformate, piperidineformate, anilineformate) at room temperature and temperature range. 40-90^oC, contact time 1 h, at a mass ratio of

ND: IM 1:1-1:4, and was found the ionic liquid morpholineformate (compared to other ionic liquids) to be the most effective extractant at a mass ratio of ND: IM- 1:4 at 80ºC. [4, 5, 8, 16, 18, 24].

3. Extraction process of Naphthalan oil distillates by N-methyl-2-pyrrolidone has been carried out under the condition of 1-3 h of contact time, at 1:1-1:3 mass ratio of ND:NMP at 40-90ºC. In this case, the optimal conditions were determined by the contact time of 1 h, at 80°C and the mass ratio of ND: NMP-1:3. The content of aromatic hydrocarbons have been determined in the obtained raffinate 2% mas., sulfur compounds 74 ppm and the yield of raffinate is 79.2% mas. [1, 2, 3, 6, 7, 12, 20, 23, 25].
4. Morpholineformate ionic liquid of Naphthalan oil boiling 200-450ºC with mass ratio ND:IM-1:1, temperature 60ºC, contact time 1 hour, as a result of 4-stage purification, the residual content of aromatic compounds 22.26% of 3.23% (dearomatization 86%), the amount of sulfur compounds was 167 ppm of 423 ppm (desulfurization 60.52%) the acid number was 0.12 mg KOH/gr, the raffinate yield was 72.65 % mas., and the extract yield was 24.25% mas [11, 17,].
5. Naphthalan oil distillates boiling at temperature ranges 200-450ºC single-stage extraction process was carried out with a NMP extractant at room temperature, contact time 0.5 h and mas ratio 1:4 of ND:NMP. The degree of aromatization of raffinate obtained during single-stage purification was determined to be 72.73%, sulfur compounds 90 ppm, acid number 0.18 mgKOH/gr, yield 81.1% mas [11, 12, 13, 18, 19, 23, 25].
6. Naphthalan oil distillates boiling at temperature ranges 200-450ºC, 4-stage extraction process was carried out with a NMP extractant at room temperature, contact time 0.5 h and mass ratio 1:0.5 of ND:NMP. At that time, the degree of aromatization of "White Naftalan" oil was 93%, the degree of desulfurization was 76.4%, the acid number was 0.03 mgKOH/gr, the yield was 73.41% mas, and the yield of the extract was 23.91% mas [9, 10, 11, 13, 15, 19].
7. N-methyl-2-pyrrolidone extractant used to purify the distillate of

Naphthalen oil boiling at 200-450ºC from aromatic hydrocarbons and sulfur compounds used in the production of therapeutic "White Naphthalan" oil as a result of spectral analysis (-UV, -IR, -NMR) has been shown to be the most effective extractant compared to ionic liquids morpholineformate. The use of N-methyl-2-pyrrolidone extractant resulted in approximately 50% solvent savings [17, 21, 13].

8. In order to remove non-sulfonated aromatic hydrocarbons (1.56%) from "White Naphthalan" oil purified on the basis of N-methyl-2-pyrrolidone from 200-450ºC boiling distillate of Naphthalan oil, it was passed through silicagel at room temperature. The yield of "White Naphthalan" oil obtained at this time was 72.52%, monoaromatic hydrocarbons 0.02%, and sulfur compounds were not observed [17, 21, 13].
9. For the first time, a block-scheme of the technology for the production of therapeutic "White Naphthalan" oil with a yield of 72.52% for complete dearomatization and desulfurization of the distillate of Naphthalan oil boiling at 200-450ºC was presented and the material balance for the process was calculated [17, 21].

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