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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

PRODUCTION OF MEDICINAL AND COSMETIC OILS FROM BALAKHANI, OIL STONES AND NAPHTHALAN OILS

Specialty: 3303.01. – "Chemical technology and engineering"

Field of science: Technics

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Baku - 2022

The work was performed at "Biologically active natural substances" laboratory of the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Azerbaijan National Academy of Sciences.

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GENERAL CHARACTERISTICS OF THE WORK

Relevance of the topic and the degree of elaboration. Cosmetic and medical oils have been widely used in medicine and cosmetology for many years. The main function of medical oils is to be a carrier in medicinal preparations. But in many cases they perform a therapeutic function at the same time. It should be noted that low-sulfur and low-paraffin oils are more suitable for the obtain of medical and cosmetic oils. In this regard, low-sulfur Azerbaijani oils rich in naphthenic hydrocarbons are more favorable. Balakhani, Neft Dashlari, Naftalan and Zaghli oils of Azerbaijan are more suitable for the obtain of medical and cosmetic oils. These oils are rich in naphthenic hydrocarbons, which contain optically active components. For this reason, the obtain of medical and cosmetic oils rich in naphthenic hydrocarbons is an actual problem[1]. Unfortunately, the production of medical and cosmetic oils does not exist in the Republic of Azerbaijan and in fact the country is completely dependent on foreign countries in this matter[2]. If we take into account that medical oils are used for various purposes in the cosmetics industry, medicine and pharmacy, food industry, agriculture, then the seriousness of the problem becomes clear.

On the other hand, the acquisition of essential oils from medicinal plants, the preparation and research of natural cosmetic and medicinal compositions based on them and white oils are also of great scientific and practical importance.

Object and subject of research. Oil distillates of Balakhani, Neft Daslari, Zaghli and Naftalan oils, which are naphthenic oils of Azerbaijan, as well as rosemary, eucalyptus, pine, thuja, orange, lavender and rose essential oils of vegetable origin were considered

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[1] Самедова, Ф.И. Нефти Азербайджана / Ф.И.Самедова. –Баку: Элм, – 2011. – 412 с.

[2] Щепалов, А.А. Современное состояние производства белых масел: зарубежные и отечественные технологии / А.А.Щепалов, Е.С.Котлова, А.С.Новоселов [и др.] // Вестник ПНИПУ. Химическая технология и биотехнология. Технология переработки нефти и газа, – Москва: – 2018. №4, – с. 138-150.

suitable for the purpose. These mentioned plants are widespread in Azerbaijan.

Goals and objectives of the study. Development of methods of purification of aromatic hydrocarbons from oil distillates separated from Balakhani, Neft Dashlari, Zaghli and Naftalan oils, rich in naphthenic hydrocarbons of Azerbaijan and separation of essential oils from various plants (rosemary, eucalyptus, pine, thuja, orange, rose and lavender), obtained medical and cosmetic oils consists in creating compositions based on essential oils and researching their antimicrobial effects against pathogenic microbes and proposing a field of application. For this purpose, the solution of the following tasks was set:

– Obtaining the oil distillate of Balakhani oily petroleum and studying the structure-group composition;

– Researching the possibilities of cleaning with adsorbents and acid for the dearomatization of T-46 oil distillate and determining the optimal parameters;

– Obtaining oil distillate based on therapeutic Naphthalan oil, researching acid-contact processes for its dearomatization and choosing optimal parameters;

– Obtaining essential oils from rosemary, eucalyptus, pine, thuja, orange, rose and lavender plants, studying their composition;

– Preparation of compositions based on medical and cosmetic oils, research of their antimicrobial properties.

Research methods: The integrity of the results obtained in the dissertation work was proven by the most modern spectroscopic methods, such as gas chromatography-mass (GC-MS), infrared (IR), nuclear magnetic resonance (NMR). The physico-chemical properties of primary and refined oils were determined by the following test methods: ASTM D445, ASTM D5002, GOST 4333-87, GOST 20287-91, ASTM D4294-06.

The main provisions of the defense:

– Development of technologies for obtaining medical and cosmetic oils based on Balakhani, Neft Dashlari, Zaghli and Naftalan oils;

– Acquisition and study of essential oils of rosemary, eucalyptus, pine, thuja, orange, rose, lavender plants;

– Preparation of compositions based on medical, cosmetic oils and essential oils and research of their antimicrobial properties.

Scientific novelty of the research:

– Development of technologies for obtaining medical and cosmetic oils based on Balakhani, Neft Dashlari, Zaghli and Naftalan oils;

– The technology of separation of essential oils of rosemary, eucalyptus, pine, thuja, orange, rose and lavender plants was developed and their composition was studied;

– Compositions based on medical, cosmetic oils and essential oils were prepared, their antimicrobial properties were studied and effective compositions were determined;

– On the basis of essential oils obtained from eucalyptus, rosemary, orange, lavender, cumin, peppermint, lemon and geranium plants, natural deodorants without carcinogenic components have been developed.

Theoretical and practical significance of the research: The results of the conducted theoretical studies are that the possibility of obtaining high-quality medical and cosmetic oils from naphthenic Baku oils has been confirmed. The comparative analysis of essential oils obtained from medicinal plants increases the possibilities of creating more effective compositions.

As a result of the dissertation work, medicines and cosmetics based on Naftalan oil and essential oils (Naftakosmet-P, Naftakosmet-L, Naftaderm-La, Naftaterm-Ch, Naftakandid-C, Gold-Naftalan), as well as natural deodorants without carcinogenic components (Atilla, Shahrizad, Mirvari, Karvan, Javadkhan, Maiden's Castle, Simurgh, Cleopatra) were created. The technical conditions created for their production have been approved by the Institute of Standardization and Metrology. White Naftalan oil was registered as a cosmetic in Saudi Arabia in 2017.

Approbation and application of research. 18 scientific works, including 10 articles, 6 theses, 1 patent, as well as 1 book on oil research were published on the dissertation work.

The articles containing the main results of the dissertation work were published in the scientific journals mentioned below: "Oil and gas technologies and analytics: scientific, technical and analytical

journal", "The world of oil products", "Scientific Works of the Central Botanical Garden of the Azerbaijan National Academy of Sciences", "News collection of Ganja branch of ANAS", “Processes of petrochemistry and oil-refining”.

The main results of the work were presented in the materials of the following scientific conferences: "Actual problems of modern natural sciences" international scientific conference dedicated to the 94th anniversary of the birth of national leader Heydar Aliyev (Ganja, May 04-05, 2017), "Floriculture: history, theory, practice" IX international scientific conference (St. Petersburg, September 7-13, 2019), The international scientific conference on "Actual problems of modern chemistry" dedicated to the 90th anniversary of the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of ANAS (Baku, October 2-4, 2019), "Science-intensive research as the basis for the innovative development of society" international scientific and practical conference (Sterlitamak R.F., April 4, 2022), "Molecular, membrane and cellular foundations of the functioning of biosystems", to the 100th anniversary of the Belarusian academic science, abstracts of the international scientific conference (Minsk, June 15–17, 2022) .

The name of the organization where the dissertation work was carried out. The dissertation work was carried out at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Azerbaijan National Academy of Sciences.

Personal participation of the author. The main scientific ideas implemented in the dissertation, the setting of issues and their implementation, the directions of conducted research, conducting experimental tests and their implementation, summarizing the obtained results were carried out by the author personally.

Volume and structure of the work. The dissertation consists of an introduction, 5 chapters, conclusions, 110 references, as well as 23 pictures, 4 technological schemes, 14 tables, a total of 147 printed sheets and a 55-page appendix to the dissertation. Dissertation consists of 194278 signs (Introduction- 8867, Chapter I - 32941, Chapter II - 21717, Chapter III - 90699, Chapter IV - 18790, Chapter V - 17856 and Conclusions - 3408), excluding figures, schemes,

tables and bibliography.

In the introduction, the relevance of the topic and degree of development, the object and subject of the research, the goals and objectives of the research, research methods, the main propositions defended, the scientific innovation of the work, the theoretical and practical importance of the research, the publication and approbation of the work, the name of the organization where the dissertation work was carried out, the scope and structure of the work, essence of the chapters are briefly described.

In the first chapter, the results of modern researches in the field of naphthenic oils of Azerbaijan, oil distillates obtained from these oils, purification of oil distillates, vegetable essential oils, medical and cosmetic oils, and the compositions based on them were analyzed.

In the second chapter, the properties of the raw materials and intermediate products used for the research were studied and information about the analysis methods was given.

The third chapter presents the results of the research conducted in the field of purification of oil distillates and extraction of essential oils of plant origin. In this chapter, general research of various oils, oil fractions purification methods were investigated, acid-contact method of purification technology was developed, directions of beneficial use of the formed by-product were proposed.

In the fourth chapter, the results of the study of the antimicrobial effect of the compositions based on medical and cosmetic oils obtained from Baku oils and essential oils obtained from plants against Pseudomonas aeruginosa, Staphylococcus aureus, Escherichia coli bacteria and Candida albicans fungus and the research of preparations that have a repulsive effect on mosquitoes were explained.

In the fifth chapter, the technological scheme of the Naftalan oil processing plant, the plant created for the purchase of vegetable essential oils and the purchase of cosmetic ointment is presented, and information about their working principles is explained in detail. Also, the material balance and technical-economic evaluation of the performed works were mentioned.

BRIEF CONTENT OF THE WORK

Methods of research and purification of Balakhani, Naftalan, Neft Dashlari and Zaghli oils

In order to obtain medical and cosmetic oil that meets modern requirements with selective solvents, adsorbents, acid-contact method from oil distillates separated from naphthenic oils of Azerbaijan, researches were carried out in the following sequences.

Oils were studied in order to determine the appropriate oil sample for conducting research. Thus, the research indicators of the oil of wells No. 2659 and 2662 of the Neft Dashlari field (Tab. 1) and No. 755, 1223, 1228, 1305, 1307, 1324, 1483 of the Zaghli field are given in table 2.

Table 1

Research of oil of some wells of the Neft Dashlari field

No. of the well	Oil extraction depth, meters	Oil density, kg/m^3	Kinematic viscosity, at 20ºC, mm^2/sec	Amount, mass %				Ignition temperature in a closed bush, ºC	Acid number, mg KOH/g mass	Fraction yield, in % (GOST 2177-99)	
				Resin	Asphaltene	Paraffin	Sulphur			Up to 200ºC	Up to 300ºC
2659	886-875	825,4	27,6	7,84	0,11	1,88	0,2144	Below -5	1,89	17,5	42,5
2662	1241-1235	845,6	37,9	8,48	0,074	1,73	0,2642	Below -5	0,23	10,0	36,0

Table 2

Oil research of some wells of the Zagli field

No. of the well	Oil extraction depth, meters	Oil density, kg/m^3	Kinematic viscosity, at 20ºC, mm^2/sec	Amount, mass %					Ignition temperature in a closed bush ºC	Acid number, mg KOH/g mass	Fraction yield, in % (GOST 2177-99)	
				Resin	Asphaltene	Paraffin	Sulphur	Coke			Up to 200ºC	Up to 300ºC
755	2338-1035	849,3	3,9	4,75	0,49	2,23	0,1858	2,18	-5	0,88	34,0	54,0
1223	2063-1520	851,4	3,4	4,38	0,52	0,75	0,138	2.19	-5	0,55	38,0	60,0
1228	2208-1790	845,4	4,1	3,98	1,49	6,35	0,1321	1,86	-5	0,07	37,5	41,8
1305	2070-1216	842,8	4,9	4,89	0,38	7,54	0,2875	1,6	-5	0,07	33,5	56,0
1307	2086-1350	843,0	4,4	5,32	0,88	6,31	0,1779	1,86	-5	0,1	35,0	59,0
1324	2030-1309	843,8	6,1	5,26	0,68	6,43	0,237	2,02	-5	0,12	12,5	42,0
1483	1168-660	897,3	18,1	8,27	0,95	1,89	0,2304	3,37	-5	0,56	12,5	42,0

A sample of medicinal Naftalan oil taken from the total capacity was analyzed.[3] Oil sample density was determined by ASTM D5002, total sulfur content ASTM D4294, total nitrogen

[3] Abbasov, V.M. Research of structure-group composition of narrow fractions of therapeutic Naphtalan oil / V.M.Abbasov, G.A.Isayeva, A.E.Alizadeh [et al.] // Processes of petrochemistry and oil-refining, – Baku: –2018. №1, p.3-16.

ASTM D5762 and water content ASTM D4928 methods. It was determined that the density of this oil sample at a temperature of 15ºC was 935.8 kg/m^3, total sulfur was 0.15%, total nitrogen content was 2126 mg/kg and water content was 0.48%.

The oil sample was fractionated by ASTM D2892 & 5236 method. As a result of the expulsion, 9 fractions and one cubic residue were obtained. The density of the obtained fractions, the amount of total sulfur, nitrogen, mass and volume yield were determined (Tab. 3)

Table 3

Indicators of fractions of naphthalene oil

Fractions of Naphthalan oil, ºC	Density at 15ºC temperature, kg/m^3	Total sulfur content, %	Amount of total nitrogen, mg/kg	Yield, in %	
				Mass	Volume
116-150	807,4	0,0026	<0,3	1,63	1,89
150-200	841,4	0,0052	< 0,3	3,03	3,37
200-250	867,2	0,0119	2,0	9,02	9,74
250-300	896,5	0,0364	72	12,02	12,55
300-350	922,5	0,106	208	14,16	14,36
350-400	936,8	0,126	612	12,81	12,80
400-450	949,3	0,160	1297	12,89	12,71
450-500	960,0	0,194	2146	10,1	9,85
500-550	971,0	0,231	2863	8,53	8,22

Examining the results of the fractions shows that as the boiling temperature of the fractions increases, their density and the amount of sulfur and nitrogen in their content increase.

In the sample taken from Balakhani oil, the density of crude oil was 886.5 kg/m^3, determined by ASTM D-1298 test method. The amount of water in the sample was 0.1% and was determined by the ASTM D-4006 method.

As a result of extraction of Balakhani oily oil sample in PILODIST PD 100CC/200CC apparatus, it was separated into 2 fractions and 1 residue:

Fraction I is expelled at 68-350ºC (AEP);

Fraction II is expelled at 350-580ºC (AEP);

The residue boils at > 580ºC (AEP).

(AEP) – means equivalent to atmospheric pressure.

The total amount of alkanes in fraction I is 24.747% (mass).

18.307% of them are normal alkanes, 6.44% are iso-alkanes.

The total amount of cycloalkanes in this fraction is 60.623% by mass, including monocycloalkanes – 32.78%, bicyclo alkanes – 17.72%, tricyclo alkanes – 0.135%, tetracycloalkanes – 6.528%, pentacycloalkanes – 3.96%.

The total amount of aromatic hydrocarbons in this fraction is 7.623%, including benzene series aromatic hydrocarbons – 0.922%, naphthalene series aromatic hydrocarbons – 3.665%, tricyclic aromatic hydrocarbons – 3.036%.

As can be seen, paraffin hydrocarbons and aromatic hydrocarbons are few and naphthenic hydrocarbons are abundant in the fraction obtained from the Balakhani oil sample and boiling at 68-350^{o}C. It contains 2,843 times more paraffins of normal structure than saturated hydrocarbons of iso-structure.

It is interesting that among the various naphthenic hydrocarbons in this fraction, tricyclic naphthenic hydrocarbons are the least amount (0.135%).

In this fraction, the amounts of naphthalene series and tricyclic aromatic hydrocarbons are approximately equal, 3.665% and 3.036%, respectively, while benzene series hydrocarbons are in the minority and amount to only 0.922%.

The total amount of alkanes in the fraction boiling at 350-580 ^{o}C is 36,666%, of which 31,183% are normal saturated hydrocarbons and 5,483% are iso-saturated hydrocarbons. The total amount of cycloalkanes in this fraction is 49.739%. The composition of these hydrocarbons is as follows:

Monocycloalkanes – 8.847%, bicycloalkanes – 6.773%, tricyclo alkanes – 1.265%, tetracycloalkanes – 19.985%, pentacycloalkanes – 12.869%.

The total amount of aromatic hydrocarbons in this fraction is only 4.357%, including monocyclic aromatic hydrocarbons – 0.106%, bicyclic aromatic hydrocarbons – 0.043%, tricyclic aromatic hydrocarbons – 4.208%.

As can be seen, in the fraction boiling at 350-580^{o}C, paraffin hydrocarbons are 36.666%, naphthenic hydrocarbons are 49.739%. As in the first fraction, in the second fraction, normal alkanes are

more than iso-alkanes, tetracyclic and pentacyclic naphthenic hydrocarbons are more than bicyclo and tricyclo alkanes.

It should also be noted that the fractions were tested on an Agilent 7890 B mass spectrometer: the temperature was raised to 20ºC at a rate of 5ºC per minute. The volume of the test sample is one milliliter.

The test was performed in TLC (total lon current) mode. Test results are given in relative (%) unit.

The spectrum obtained for fraction 1 in the mass spectrometer is given in figure 1 and the spectrum obtained for fraction 2 is given in figure 2.

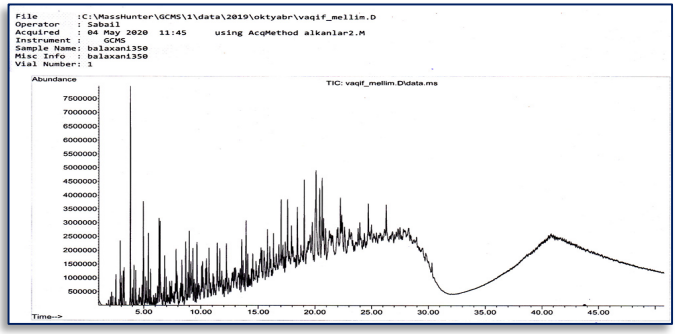


Figure 1. Mass spectrum of the fraction boiling at 68-350ºC of Balakhani fatty petroleum

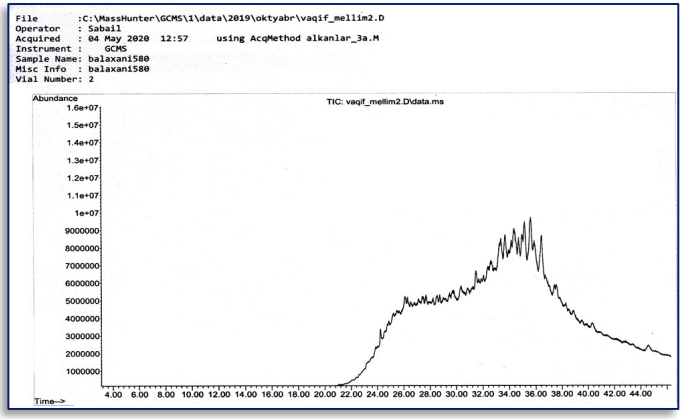


Figure 2. Mass spectrum of the fraction boiling at 350-580ºC of Balakhani oil

Desirable hydrocarbons in oil distillates are naphthenic hydrocarbons, liquid paraffin hydrocarbons. Undesirable hydrocarbons are polycyclic aromatic hydrocarbons, short side-chain naphthene-aromatic hydrocarbons, solid paraffins, serosins.

Oil distillates also contain oxygen-sulfur-nitrogen compounds. Medicinal and cosmetic oils generally should not contain aromatic hydrocarbons, oxygenated and sulfur compounds.

Initial studies involved purifying T-46 oil distillate containing 15.0% aromatic hydrocarbons. The distillate was purified in a glass calon containing silica gel. 1440 grams of silica gel was filled in a glass calon with a height of 150 cm and a diameter of 4.6 cm. The height of the part filled with silica gel in the calon was 113.5 cm. 2 liters of T-46 oil distillate was passed through this calon at room temperature. T-30 oil distillate and oil distillate obtained from Zagli oil were also released from the same type of calons.

To start the research, the physico-chemical properties of the identified oil samples were first studied (Tab. 4).

Table 4

Results of dearomatization of oil distillates of T-46, T-30 and Zagli oil with silica gel in a calon

The name of the indicators	The name of the sample					
	Primary T-46 oil distillate	Refined T-46 oil distillate	Primary T-30 oil distillate	Refined T-30 oil distillate	Primary Zaghli oil distillate	Refined Zaghli oil distillate
Kinematic viscosity, mm^2/sec, 40ºC	77.750	57.712	49.642	36.784	6.6126	6.0806
Kinematic viscosity, mm^2/sec, 100ºC	8.0356	7.1480	6.1185	5.5075	2.0065	1.9296
Viscosity index	56.5	75.7	50.3	78.7	88.4	98.1
Density, kg/m^3 20ºC	906,6	885,9	906,4	880,2	882,2	837,5
Freezing temp.ºC	-27	-30	-35	-35	-20	-50
Ignition temp.ºC	236	232	218	217	126	132
Amount of aromatic hydrocarbons, %	15,0	0,0	14,0	0,0	25,0	0,0

As can be seen from the table, after removing all three distillates from the silica gel-filled calon, the amount of sulphable aromatic hydrocarbons in the purified product was 0.0%, that is, no carcinogenic aromatic hydrocarbons remained in the purified product (Tab. 4).

Other studies have been the purification of oil distillates with adsorbents. The cleaning was carried out in a special mixer for 2, 3 and 4 hours at temperatures of 20 and 80^0C (Tab. 5).

Table 5

Refinement stages and properties of T-46 oil distillate under different conditions

Raw material		Indicators of oils									
		% of adsorbent with respect to the	Temperature, ºC	Duration of the experiment, hours	Yield, %-lə	Kinematic viscosity, mm^2/sec,		Density at 20ºC, kg/m^3	Total sulfur,%	Color, score	Amount of aromatic k/h+in, %
						40ºC	100ºC				
T-46 oil distillate initially		-	-	-	-	75,856	7,9121	906,6	0,1789	2,5	15,0
T-46 oil distillate + bentonite		25,0	80	3	86,5	74,358	7,6394	906,3	0,1275	2,5	8,0
T-46 oil distillate + bentonite		11,0	80	4	77,0	75,978	7,6796	906,4	0,1359	2,5	10,0
T-46 oil distillate + bentonite	I step	25,0	80	2	90,86	74.331	7.7921	906,1	0.1148	2,5	6,5
	II step	25,0	80	2	73,57	74.728	7.8275	904,9	0,1034	2,5	5,6
T-46 oil distillate + bentonite		20,0	20	2	88,0	75,856	7,3461	905,5	0,1220	2,5	6,5
T-46 oil distillate +silica gel		25,0	20	2	93,0	76,033	8,0191	902,1	0,0840	2,5	11,0

As a result of the research, it was determined that the amount of aromatic hydrocarbons decreased from 15.0% to 6.5% after the first stage during the treatment of T-46 oil distillate with 25.0% bentonite at 80^0C. After stage II, the amount of aromatic hydrocarbons was 5.6%.

Bentonite cleaning was also carried out in one step at room

temperature. Bentonite was taken in the amount of 20% of T-46 oil distillate and the cleaning time was 2 hours. The amount of aromatic hydrocarbons in the purified product was 6.5%. As can be seen, the result is the same as the result of one-step cleaning at 80^0C. Thus, it is not advisable to generate additional temperature during cleaning with bentonite.

It should be noted that bentonite is a natural adsorbent, produced in Azerbaijan, and its price is many times cheaper than silica gel. On the other hand, the bentonite used in the purification of oil distillate can be used in the preparation of asphalt-concrete pavement in road construction without being regenerated.

As can be seen in Table 5, the amount of sulfonated aromatic hydrocarbons decreases from 15.0% to 11.0% during purification with silica gel and from 15.0% to 6.5% during purification with bentonite. However, less total sulfur remains in the purified product after silica gel purification. Thus, while the amount of total sulfur in the product obtained by purification with silica gel is 0.0840%, in the product obtained by purification with bentonite, 0.1220% of total sulfur remains (that is, 20.6% more).

The structural parameters of the product obtained from the purification of T-46 oil distillate with adsorbents were confirmed by NMR spectroscopy.

In the first direction, adsorption of T-46 oil distillate from aromatic hydrocarbons was carried out from a column filled with silica gel. The structural indicators of refined oil are H_{Ar} – 0.0%, Hα – 0.0%, Hβ:$H_{naphthene}$ – 14.3%, $H_{paraffin}$ – 51.9%, Hγ – 33.8%, isoparaffin index – 0.43, fα – 0.0%. As it can be seen, the oil obtained by this purification method is completely purified from aromatic hydrocarbons (Figure 3).

In the second direction, the T-46 oil distillate was purified by stirring with silica gel taken at 25.0% of the oil distillate at 20ºC for 2 hours. The structural parameters of the purified distillate are H_{Ar}- 2.0%, Hα- 3.0%, Hβ:$H_{naphthene}$- 12.7%, $H_{paraffin}$- 49.4%, Hγ- 32.9%, isoparaffin index- 0.44, fα- 0.11%. This means that in this case the purification is practically not possible (Figure 4).

In the third direction, T-46 oil distillate was purified by mixing

with bentonite taken at 25.0% by oil distillate at 20ºC for 2 hours. In the purified product, H_{Ar}- 1.8%, Hα- 4.5%, Hβ:$H_{naphthene}$- 12.8%, $H_{paraffin}$- 31.0%, isoparaffin index- 0.41, fα -0.09%. This means that the cleaning did not go well (Figure 5).

In the fourth direction, gasoline was first passed through the silica gel filled in the calon and then it was purified by releasing T-46 oil distillate. In the purified product, H_{Ar} – 0.6%, Hα – 1.5%, Hβ:$H_{naphthene}$ – 14.3%, $H_{paraffin}$ – 51.2%, Hγ – 32.4%, isoparaffin index – 0.42, fα – 0, It was 01%. That is, the total amount of aromatic hydrocarbons was 1%. The advantage of this method is that gasoline lowers the viscosity of the oil distillate, which is why the removal of aromatic hydrocarbons is faster. The downside is that this method uses a gasoline fraction that can cause a fire (Figure 6).

Note: H_{Ar} is the amount of hydrogen in the aromatic structure

Hα – amount of CH_3, CH_2 and CH groups in the α-state in the aromatic nucleus

Hβ – the amount of CH3, CH2 and CH groups in the β-state in the aromatic nucleus

$H_{naphthene}$ - the amount of hydrogen in naphthene-containing hydrocarbons

Hγ – CH_2 is the amount of hydrogen in the last groups

fα – degree of aromaticity

İ – isoparaffin index

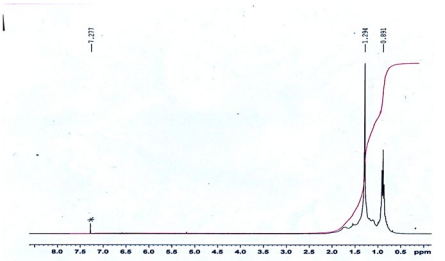


Figure 3. NMR spectrum of T-46 oil passed through silica gel in calon

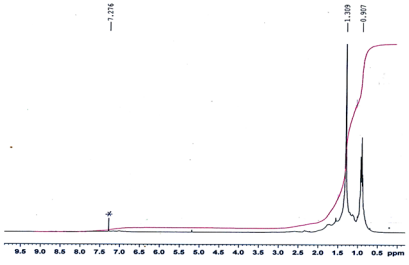


Figure 4. NMR spectrum of T-46 oil purified with 25.0% silica gel

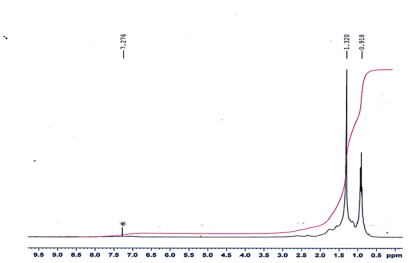


Figure 5. NMR spectrum of T-46 oil treated with 25.0% bentonite

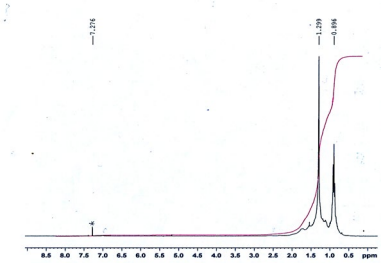


Figure 6. NMR spectrum of T-46 oil purified first by gasoline and then by silica gel

Thus, to obtain dearomatized oil based on T-46 oil distillate, the method of passing through calon filled silica gel is more appropriate.

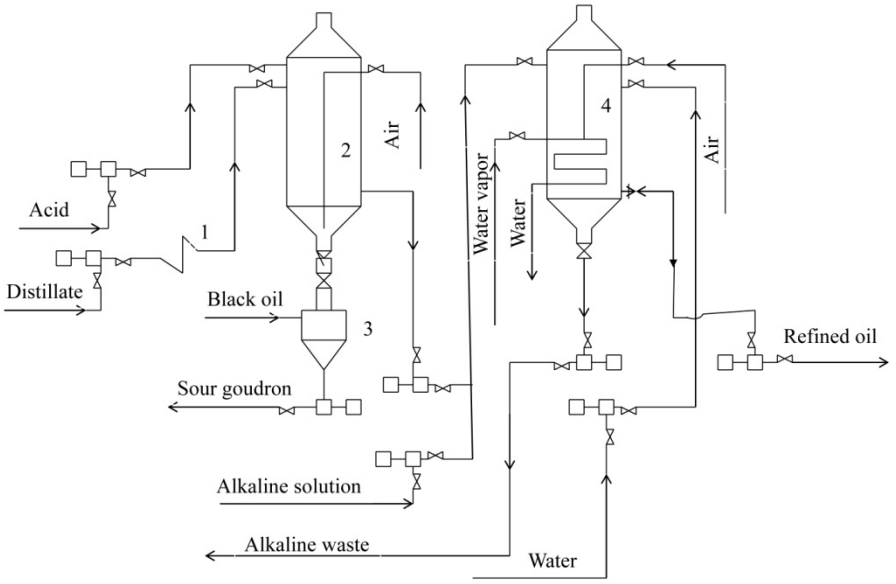
Another cleaning method is the extraction cleaning method, a mixture of 90.0% N-methylpyrrolidone and 10.0% ethylene glycol was taken as a solvent. For purification, oil distillate of Balakhani oil containing 15.0% aromatic hydrocarbons and extractant-solvent were taken in a ratio of 1:3 by mass. The temperature was 30ºC and 60ºC with stirring for 2 hours.

In Stage I, 100 grams of oil distillate, 270 grams of N-methylpyrrolidone and 30 grams of ethylene glycol were taken. The amount of aromatic hydrocarbons in the purified oil distillate was 8.0%, the purification temperature was 30ºC and the degree of purification was 46.67%.

Stage II was carried out at 60ºC taking 100 grams of oil distillate, 270 grams of N-methylpyrrolidone and 30 grams of ethylene glycol. At this time, the amount of aromatic hydrocarbons in the purified oil distillate was 7.0% and the degree of purification was 53.34%.

The purification product of Stage II was purified a second time at 60ºC. The amount of aromatic hydrocarbons in the purified oil distillate was 4.0% and the degree of purification was 73.34%.

Other researches have been in the direction of purification of T-46 oil distillate with sulfuric acid.

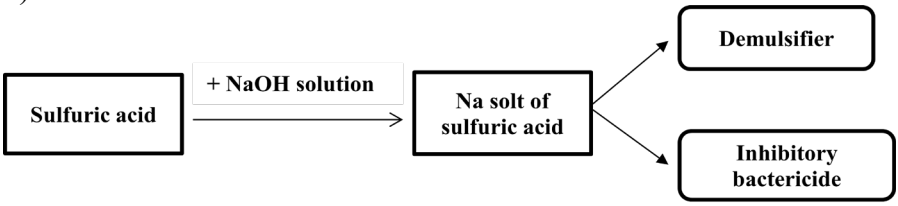


Scheme 1. Technological scheme of purification of oil distillates from aromatic hydrocarbons by acid-alkaline method

The process was carried out in five stages:

1. To dearomatize the oil distillate and get sulfoacid; (1,2)
2. Separate sulfuric acid and get 20-25% sodium sulfonate salts from them; (3)
3. Neutralize the dearomatized oil from acid residue with 5% NaOH; (4)
4. Wash the neutral oil with water; (4)
5. Hot air drying of oil desalted with hot water. (4)

The main shortcoming of the method of dearomatization of oil distillates by the acid-contact method was the fact that the obtained by-product - acid tar - was still not offered for efficient use (scheme 2).



Scheme 2. Block diagram of the purposeful use of sulfuric acid

During acid-contact cleaning, neutralization should be carried out in such a way that a 20-25% sodium sulfonate solution is obtained. T-46 oil distillate was sulfonated at room temperature by adding 98.0% sulfuric acid (10% by oil distillate) for 3 hours and then neutralized with 5.0% sodium hydroxide solution. It was determined that in this case aromatic hydrocarbons were completely removed from the oil (scheme 1).

The density of the salt solution obtained during cleaning at room temperature was 1.035 g/cm^3 at 20ºC, the freezing temperature was minus 8ºC and the pH was 9.524

2.0% solutions were prepared from 20.0% salt solutions and their demulsifying properties were checked.

The bactericidal property of 20.0% sodium sulfonate solutions against sulfate-reducing bacteria was studied.

During the experiments, in the environment without sulfate-reducing bacteria (SRB), the amount of hydrogen sulfide was 24 mg/l, in the second control environment with SRB, the amount of H_2S was 375 mg/l and in the third control environment, the amount of SRB was 108 cells/ml.

The sodium sulfonate solution obtained as a by-product during the purification of T-46 oil at room temperature was added to the nutrient medium in the amount of 100, 200, 300, 400, 500 and 600 mg/l.

Depending on the concentration, the amount of SRB decreases sharply when sulfosalt solution is added to the nutrient medium. Thus, when the solution is given at 100 mg/l, the amount of SRB decreases from 10^8 cells/ml to 10^4, the amount of hydrogen sulfide decreases from 375 mg/l to 136 mg/l, the bactericidal effect is 63.7%.

When the concentration of the reagent is 200 mg/l, SRB decreases to 10^3 cells/ml, H_2S to 110.5 mg/l, the bactericidal effect is 71.0%. When 300 mg/l reagent is added to the environment, the number of SRB decreases to 10^3 cells/ml, H_2S is 93.5 mg/l and the bactericidal effect is 75.0%.

When 400 mg/l is added to the reagent medium, the amount of SRB decreases from 10^8 to 10^1 cells/ml, the amount of H_2S decreases

from 375 mg/l to 8.5 mg/l, the bactericidal effect is 97.7%.

The further continuation of the experiments showed that when the reagent was given to the medium in the amount of 500 and 600 mg/l, there were no cells of SRB in the nutrient medium and H_2S was not formed in the medium. Note that the absence of SRB in the environment eliminates the danger of biocorrosion, the absence of H_2S, hydrogen sulfide corrosion, and electrochemical corrosion as a whole.

Studies have shown that a two-step purification with bentonite at 80ºC and purification with 98.0% solid sulfuric acid (taken at 10% by oil distillate) at room temperature can obtain medical and cosmetic oil. The used bentonite can be used in road construction, and the obtained by-product-sulfo acids mixture can be used as a demulsifier and corrosion inhibitor-bactericide.

Research of the antimicrobial effect of compositions based on purified White Naphthalan, T-46, Balakhani oils and essential oils

It is known that the effectiveness of drugs used for the treatment of pathologies caused by microorganisms in any country of the world, as well as in Azerbaijan, decreases over time. The reason for this is the constant formation of resistant forms of microorganisms. It is these reasons that make it necessary to constantly keep these issues in the center of attention, to constantly search for new methods and approaches to eliminate the pathologies caused by microorganisms.

As a result of research, in order to confirm the effect of combined compositions of oil distillates of refined oil and essential oils obtained from plants at a high level, their antimicrobial effects against pathogenic microbes were studied.

In the course of research, White Naphthalan oil (WNO), dearomatized T-46 and Balakhani oils (BO) and rosemary, eucalyptus, pine, thuja, orange, lavender, etc. each of oils were mixed 1:0,04; 1:0.08 and 1:0.1 mass ratios were prepared, their effects against Pseudomonas aeruginosa, Staphylococcus aureus, Escherichia coli bacteria and Candida albicans fungus at different

exposure times (15 min., 30 min., 1 hour, 2 hours, 3 hours, 4 hours, 5 hours, 6 hours) were studied. For this purpose, the test-object method was used.

As a result of the research, during the effect of compositions of WNO and rosemary oil in different proportions on the biological activity of Staphylococcus aureus bacteria, it was observed that the proportions of 1:0.08 and 1:0.1 begin to have a bactericidal effect already within 1 hour. However, compositions with the same proportions of WNO and pine oil began to have a destructive effect on bacteria starting from the exposure time of 5 hours.

During the effect of different ratio compositions of WNO and eucalyptus oil on Staphylococcus aureus, it was determined that the composition with a mass ratio of 1:0.08 has an antimicrobial effect starting from the exposure time of 3 hours.

During the study of the antimicrobial effect of 0.04%, 0.08% and 0.1% solutions based on dearomatized T-46 oil and eucalyptus oil on Pseudomonas aeruginosa bacteria, it was determined that the 0.04% solution was only 3 hours and 0.1% solution for the initial 15 min. showed a higher antibacterial effect starting from the exposure period. During the study of the antimicrobial activity of these solutions on Staphylococcus aureus bacteria, we observed that they had a 100% bactericidal effect on the inoculated bacterial cultures starting from the exposure period of 5 hours in the 0.1% composition.

In the course of the research, during the antibacterial effect of compositions of WNO with tuya oil, it was found that only the composition prepared in a ratio of 1:0.1 enhanced the antibacterial activity of Pseudomonas aeruginosa bacteria starting from the exposure period of 6 hours.

The effect of compositions with dearomatized T-46 oil and tuya oil on the biological activity of Pseudomonas aeruginosa was also studied. It was determined that the composition prepared in a ratio of 1:0.1 destroyed Pseudomonas aeruginosa bacteria at all exposure times and had a 100% bactericidal effect.

The first 1:0.04 ratio composition of WNO and tuya oil caused an increase in the antimicrobial activity of Staphylococcus aureus

bacteria starting from 5 hours and after increasing concentrations, the 1:0.08 and 1:0.1 ratio compositions increased from 1 hour.

During the study of the antibacterial activity of compositions of WNO with orange oil against Pseudomonas aeruginosa from Gram (-) bacteria, it was determined that the composition taken at a mass ratio of 1:0.1 has a high antimicrobial effect against the mentioned bacteria after 30 minutes.

Certain changes were observed during the effect of 0.04%, 0.08% and 0.1% solutions prepared on the basis of WNO and orange oil on the biological activity of Staphylococcus aureus. Thus, the 0.04% solution had an antibacterial effect starting from 5 hours, the 0.08% solution from 3 hours, and the 0.1% solution from 15 minutes of exposure.

The effect of different ratio compositions of WNO with orange oil on Candida albicans was also studied. It was determined that only the 1:0.1 composition had a 100% antimicrobial effect after 3 hours of exposure.

0.04%, 0.08% and 0.1% solutions of lavender oil with dearomatized BO were prepared and the effect on microbes was also studied. It was found that only 0.1% solution has an antimicrobial effect on Pseudomonas aeruginosa bacteria after 6 hours of exposure.

It was determined from the researches that compositions made in different proportions have different effects on microbes.

In the course of research, natural and harmless preparations have been created that repel mosquitoes. A mixture of white Naphthalene oil with eucalyptus oil, rosemary oil, and common pine oil had a repellent effect against mosquitoes of up to 5-6 hours, while the effect of a mixture made with tuya oil and peppermint oil was 2-3 hours.

The technology of obtaining cosmetics and medicines

It should be noted that although various mineral oil distillates are used as raw materials for the preparation of cosmetics and pharmaceuticals, as a result, the general fraction of therapeutic Naftalan oil was chosen as a more favorable raw material. This is due to several reasons:

- The oil obtained from therapeutic Naftalan oil has not only carrier but also therapeutic value.
- The initial boiling temperature of the total fraction obtained from this oil is 116°C and the final boiling point is 550°C. When this fraction is sulfonated, phasing is faster at the end of the process. This is due to the fact that, in contrast to mineral oil distillates, the density of the total fraction of therapeutic Naphthalan oil is lower. It is for this reason that it is easier to get medical oil from distillate by adsorbent method. Thus, this fraction is purified at a greater speed in the column containing silica gel as an adsorbent.
- After therapeutic Naftalan oil has been used for a certain period of time in a bath, a therapeutic oil fraction can be obtained from it.

The experience of preparing certain cosmetic products based on the oils of various medicinal plants has been around for a long time, and more and more attention is being paid to this field. Extensive research in this field is carried out at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of ANAS. Essential oils are obtained from mint, eucalyptus, rosemary, pine, juniper, thuja, cumin, anise, geranium, lemon, orange, tea tree, rose, lavender, thyme, wormwood plants. The composition of the obtained essential oils is studied by mass-spectroscopy, as well as by preparing compositions based on de-aromatized cosmetic oil and essential oils in different ratios, their antimicrobial effects against pathogenic microbes are studied.

Figure 7 shows the technological scheme of the apparatus created for the extraction of essential oils from plants. The main part of this apparatus, i.e., the water vapor extraction column, is made of copper, and the vapor cooling and condensation system is made of stainless steel. The core reactor, made of copper, consists of two parts inside, with a very small porous partition between the parts. The plant whose oil is to be extracted is placed in the reactor through a special cover on the upper part. Water vapor is supplied to the lower part. Essential oils expelled by water vapor pass through a water-cooled refrigerator and are collected in a special container.

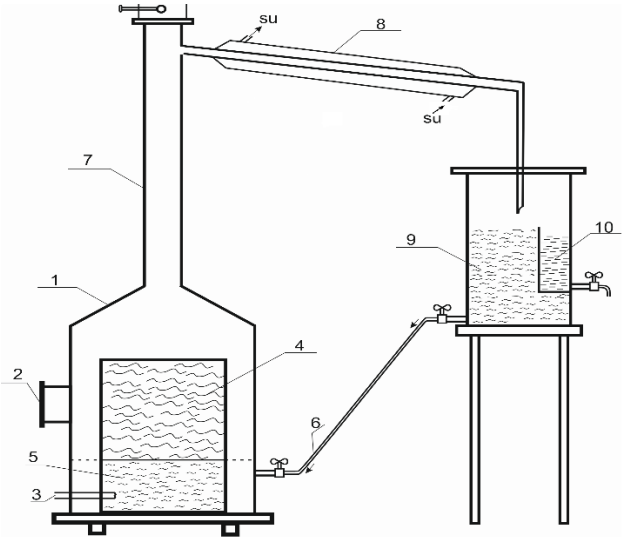


Figure 7. New technological scheme for obtaining essential oils from essential oil plants: 1- reactor, 2- hatch for filling the reactor, 3- heater, 4- plant to be extracted, 5- water for evaporation, 6- condensed water returned to the reactor, 7- column, 8- calon, 9- condensate, 10- condensate separator ether.

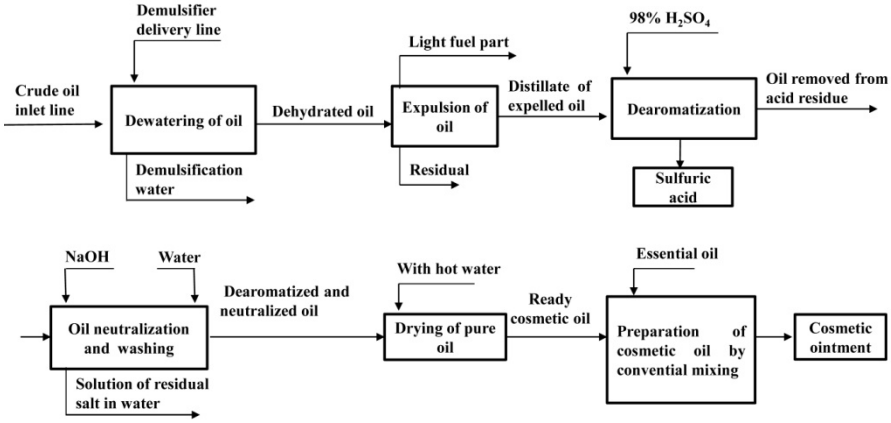
On the other hand, the previously used classic method for removing the water used in the steam cooling system - the chemical water treatment method - has been abandoned. So, in this method, energy and reagent costs are high, and the cost increases. In the course of research, it was proposed to use river and lake water, water from artesian wells only after cleaning the suspended substances, in order to prevent the possibility of mold forming on the inner walls of the pipes of the cooling system of the created device and, therefore, to prevent deterioration of the cooling system. In order to solve this problem, we suggested using the "Baku" scale solvent twice a year, created at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of ANAS. It should also be noted that the composition obtained during the cleaning of the cooling system (amide nitrogen and simple superphosphate) can be used as a fertilizer for plants in agricultural fields. Thus, a low-energy, low-cost, low-cost method of obtaining essential oils of plants has been

developed. It should be noted that the cleaning process of the cooling system of the unit is carried out without stopping the main process, which increases the overall useful efficiency of the unit. On the other hand, the used "Baku" scale solvent protects the inside of the cooling system from the corrosion process at a high level, so the service life of the unit increases.

In addition, it is recommended to add "Azeri", a multifunctional corrosion inhibitor created at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of ANAS (in the amount of 300 g per m^3 of cooling water), so that the water used in the cooling system does not form sludge and does not cause corrosion.

"Azeri" inhibitor has a dual function. This inhibitor prevents both scale formation and corrosion process. This leads to better cooling and no loss of light components in essential oils. To obtain different essential oils in the same reactor, the main reactor of the unit is washed with a special detergent before changing the raw materials, so that the smell of the essential oil of the previous plant does not remain. For this, cotton or sunflower oil is hydrolyzed with alkali and the reactor is washed with the obtained solution, then cleaned with steam.

In the course of research conducted in accordance with the purpose of the thesis, the purchase of cosmetic ointments went according to the following scheme (Scheme 3).



Scheme 3. Block diagram of the obtain of cosmetic ointment

As can be seen from the scheme, the crude oil dehydration process is underway at the initial stage. For this purpose, crude oil is collected in the reactor and a demulsifier is added to it. The demulsified water is separated from the oil. This process is continued until the amount of water in the oil is 1%. In the second reactor, the process of expelling dehydrated oil is underway. Dehydrated oil is separated from the light fuel part and fractionated. The expelled oil fraction is sent to the third apparatus and undergoes a chemical cleaning process with 98% sulfuric acid. Oil purified by precipitation from the residue of the ink is separated and sent to another reactor. To remove the acid residue, the refined oil is first washed with 5% NaOH and then with plain water until a neutral medium is formed. The finished product, completely cleaned from the reagent with water and dried with hot air, is sent to the next apparatus and undergoes the stabilization process. Cosmetic ointments (Naftakosmet-P, Naftakosmet-L, Naftaderm-La, Naftaterm-Ch, Naftakandid-C, Gold-Naftalan) were developed based on the prescribed recipes based on the obtained dearomatized cosmetic oil and essential oils of medicinal plants. Technical conditions have been developed for the created cosmetics.

CONCLUSIONS

1. Technologies for obtaining medical and cosmetic oils based on oil distillates of dearomatized T-46, T-30, Balakhani, Zaghli and Naftalan oils were developed and the structure-group compositions of purified oils were studied by modern research methods [3, 4, 13, 14, 17, 18].
   When oil distillates were released from a glass calon filled with silica gel with a height of 150 cm and a diameter of 4.6 cm, the residual content of sulfonated aromatic hydrocarbons in the obtained oil was 0.0%.
2. A waste-free cleaning method with solid sulfuric acid was developed to obtain medical and cosmetic oil. It was determined that when T-46 oil is processed by adding 98.0% sulfuric acid in the amount of 10.0% to the oil for 3 hours at room temperature,

the oil becomes completely dearomatic, and the sodium salt solution of sulfuric acids obtained as a by-product has both demulsifying and bactericidal properties. It was determined that sulfonate salt is added to nutrient medium at 100 mg/l; 200 mg/l; 300 mg/l; When given in the amount of 400 mg/l, the amount of SRB in the medium is 104, 103, 101 cells, respectively. The amount of H_2S in the environment is 136 mg/l in the first four cases; 110.5 mg/l; 93.5 mg/l and 8.5 mg/l. When the reagent is given at 500 mg/l and 600 mg/l, H_2S is not formed in the environment [16].

3. An acid-contact purification method was developed for the dearomatization of the oil fraction of naphthalene oil [2, 17].
4. We created a device for extracting essential oils from medicinal plants and for the first time, the use of "Azeri" inhibitor was proposed for the cooling system of this device.
5. Rosemary, thuja, pine, juniper, eucalyptus, mint, lemon, orange, wormwood, thyme, lavender, rose, geranium, cumin, anise, tea tree oils were obtained [1,5,6,7,8,9,10,15] and cosmetics were obtained by preparing compositions with biologically active White Naphthalan oil.
6. Biologically active White Naphthalan oil and cosmetic oils containing composition were obtained [9] and the technical condition for them is TC AZ 2000146301. 264-2019 "Cosmetic oils based on White Naphthalan oil" (on 27.11.2019, with registration number 1874); Perfume-cosmetic deodorants that do not have carcinogenic components have been prepared based on vegetable oils, the technical condition for their production is TC AZ 2000146301.265-2020. "Perfumer-cosmetic deodorants in aerosol packaging" (on 04.09.2020, with registration number 1946); "Lemon, orange, tuya oils" TC AZ 2000146301.276-2022 (on June 8, 2022, with registration number 2086); The technical condition TC AZ 2000146301.277-2022 "Gold Naftalan cosmetic oils" for cosmetics made on the basis of biologically active White Naftalan oil and essential oils obtained from various plants was prepared (with registration number 2087 on 8.06.2022) and approved and applied at the Azerbaijan

Standardization Institute.

7. As a result of research, it was determined that from the analyzes conducted on 4 microbes (Pseudomonas aeruginosa, Staphylococcus aureus, Escherichia coli bacteria and Candida albicans fungus) cosmetic oil obtained from petroleum oil distillates and plants (rosemary, eucalyptus, pine, thuja, juniper, orange, lemon), mint, geranium, jasmine, thyme, lavender) compositions of essential oils obtained in different proportions (1:0.04; 1:0.08; 1:0.1) have a more effective bactericidal and fungicidal effect. So that:
   Against Staphylococcus aureus bacteria – compositions prepared with essential oils of rosemary, pine, eucalyptus, thuja, orange, lemon, mint, geranium, lilac, thyme - plants;
   Against Pseudomonas aeruginosa bacteria – compositions prepared with essential oils of thuja, orange, mint, lavender, thyme, lilac, juniper plants;
   Compositions made with essential oils of lemon, mint, geranium, thyme, jasmine, juniper plants – against Escherichia coli bacteria;
   Compositions made with essential oils of orange, lemon, mint, geranium, thyme, juniper plants have an antimicrobial effect – against Candida albicans fungus..
8. In the course of research, preparations with a repulsive effect on mosquitoes were created from the compositions of WNO with rosemary oil, eucalyptus oil and common pine oil and they were patented [11,12].

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