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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS OF NANOCARBON BASED ON LIQUID PRODUCTS OF PYROLYSIS AND STUDY IN SUNFLOWER OIL PURIFICATION PROCESSES

Speciality: 2314.01 - Petrochemistry

Field of science: Chemistry

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The work was performed at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan.

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GENERAL DESCRIPTION OF THE WORK

The actuality of the topic and elaboration rate. In modern times, the synthesis of high-quality nanoscale carbon materials has led to fundamental changes in all areas of industry. The development of the effective production technology of carbon nanoparticles opens wide prospects for their use in hydrogen energy, in various fields of medicine, in the creation of electrodes for fuel cells, in catalysis - as a catalyst carrier, in nanoelectronics - in the preparation of one-dimensional conductors (wires), nano-sized transistors, cold emitters of electrons and supercapacitors, in technology - to increase the mechanical strength, electrical conductivity, and heat resistance properties of products, as an additive to polymers, and inorganic composites[1].

The rapid development of the chemical industry, the daily increase in the demand for technology and innovations forces researchers to improve existing systems, create high-efficiency systems, use intermediate products as raw materials, to realize environmental protection, and to acquire advanced technologies in the direction of purchasing low-cost products. From this point of view, development of the nanocarbon processing process, increasing yield by optimizing the process, conducting research in the directions of improving the quality indicators of nanocarbon are among the urgent issues of the petrochemical industry.

It is known that one of the main indicators characterizing the consumption properties of vegetable oils is its transparency, which is determined by the presence of pigments, lipids and resin-like substances in the oil. Cleaning oils from pigments, phospholipids, as well as salts of fatty acids and metal ions formed in them during the processing stages is considered one of the important stages. For this

[1] Jeevanandam, J. Review on nanoparticles and nanostructured materials: history, sources, toxicity and regulations / J.Jeevanandam, A.Barhoum, Y.S.Chan, A.Dufresne, M.K.Danquah // Beilstein Journal of Nanotechnology, - 2018. 9, - p. 1050 - 1074.

purpose, the bleaching earth is widely used[2]. Bleaching earth is mainly produced in the United States of America, Malaysia, and China, the selling price of bleaching earth is high, and it is often impossible to obtain the desired quality indicators during use.

The object and subject of the research. The main research object of the dissertation work is obtaining nanocarbon from liquid products of pyrolysis, activated carbon (AC) from heavy pyrolysis resin (HPR), and the subject of the research study is the study of synthesized carbon materials as adsorbent in the process of bleaching sunflower oil.

The purpose and objectives of the research. The purpose of the research is to synthesize nanocarbon and activated carbon from the liquid products of pyrolysis and heavy pyrolysis resin by CVD method, study the regularities of the process, analyze the structure and properties of the obtained carbon particles, analyze the surface properties through physical methods, and develop effective methods of the refining process using new highly efficient adsorbents in the existing technological processes of oil processing.

The following research works have been conducted to achieve the goal:

- Conducting pyrolysis of C_5 fraction in CVD with the presence of a catalyst placed on the carrier for obtaining carbon nanoparticles;
- Determining the effect of gas mixture composition, process temperature, and time on the yield of carbon nanoparticles;
- Studying the physicochemical properties of the obtained particles, confirming their composition and structure with modern physical research methods;
- Synthesis of carbonized material with excellent surface properties from HPR in the presence of NaOH;
- Studying the properties of activated carbon obtained from the thermal processing of HPR by physical methods;

[2] Topkafa, M. Role of Different Bleaching Earths for Sunflower Oil in a Pilot Plant Bleaching System / M.Topkafa, H.F.Ayyildiz, F.N.Arslan, S.Kucukkolbasi, F.Durmaz, S.Sen, H.Kara // Polish Journal of Food and Nutrition Sciences, - 2013. 63 (3), - p. 147 - 154.

- Selection of effective, high-quality adsorbents for the process of bleaching of oils in order to improve the quality of the finished product;

- Studying the effect of technological regimes on the quality of oils, their antioxidant stability, and storage time;

- Comparative study of nanocarbon with bleaching earth widely used as an adsorbent in practice in the bleaching of vegetable oils, determination of the effect of their minimum concentration and other parameters on the process.

Research methods. Chromatographic, X-ray phase, X-ray fluorescence, Raman spectrometry, Inductively coupled plasma mass spectrometry, thermogravimetry, scanning electron, tunneling microscopes, and other physicochemical analysis methods were used to determine and identify the structural and physical properties of primary components, synthesized carbon nanomaterials and refined oils.

The main provisions for the defence.

- Nanocarbon was obtained from the C_5 fraction of pyrolysis liquid products, which are produced in our country and require efficient processing;

- Activated carbon with high surface properties was synthesized as a result of the thermal treatment of heavy pyrolysis resin with alkalis;

- The physico-chemical properties, composition and structures of the synthesized carbon samples were determined;

- The possibility of using the obtained nanocarbon and activated carbon materials in the bleaching of oils was investigated;

- The effect of the amount of adsorbent, temperature and time on the bleaching process was studied;

- In order to increase the efficiency of the process, the refining process was carried out in mass ratios of 95÷5, 96÷4, 97÷3, 98÷2 and 99÷1 of bleaching earth and nanocarbon.

Scientific novelty of the research.

- The nanocarbon synthesized from the C5 fraction was used as an adsorbent in the process of refining food oils;

- Nanocarbon particles were synthesized at high temperatures with the presence of iron-based catalysts, their dimensions and properties were studied, and it was determined that in the process of pyrolysis of the C5 fraction at temperatures of 750-920ºC in the CVD reactor, the addition of inert gas and hydrogen gas to the system at different rates regulate the size of the nanocarbon growing on the catalytic centers, and at the same time to the increase in the purity of the obtained nanocarbon;

- The adsorption isotherms for the bleaching stage of the oil refining process were determined, which made it possible to precisely control the necessary parameters of the process;

- Due to the high adsorption capacity of the nanocarbon obtained from the C_5 fraction, it was determined that it has a high efficiency in the decolorization process of oil compared to bleaching earth.

Theoretical and practical significance of the research. Compared to other adsorbents, an adsorbent with higher efficiency than other adsorbents - nanocarbon - was synthesized from the liquid products of pyrolysis and its application was carried out in the sunflower oil purification process. The application areas of the obtained nanocarbon were investigated. The use of nanocarbon, activated carbon and their mixture as a highly adsorbing reagent in the vegetable oil industry has been suggested.

Personal participation of the author. Collection and summarization of literature data, preparation of samples for research, preparation and conducting of experiments, analysis of the obtained results, writing of articles and theses, and preparation for printing were performed with the direct participation of the author.

Approbation and application of research. 21 scientific works including 8 articles and 13 theses, were published on the subject of the dissertation.

The main results of the work were reported and discussed at the following various scientific and international conferences:

Republican scientific-practical conference on petrochemical synthesis dedicated to the 100th anniversary of Academician S.C.Mehdiyev (Baku 2014), IX Republican scientific conference "Actual problems of chemistry" of doctoral students, masters and young researchers dedicated to the 92nd anniversary of the birth of national leader Heydar Aliyev (Baku 2015), Republican Scientific Conference dedicated to Academician T. Shakhtakhtinsky's 90th anniversary (Baku 2015), Scientific conference "Actual problems of modern chemistry and biology" dedicated to the 93rd anniversary of the birth of the national leader Heydar Aliyev (Ganja 2016), 6th Rostocker International Conference: "Thermophysical Properties for Technical Thermodynamics" (Germany 2017), International scientific conference on "Actual problems of modern natural sciences" (Ganja, 2017), International Scientific and Technical Conference "Petrochemical Synthesis and Catalysis in Complex Condensed Systems" dedicated to the 100th anniversary of Academician B.K.Zeynalov (Baku, 2017), 7th Rostocker International Conference: "Thermophysical Properties for Technical Thermodynamics (Germany 2018), International scientific conference "Actual problems of modern natural and economic sciences" (Ganja 2018), International scientific conference "Actual problems of modern natural and economic sciences" (Ganja 2019), The International Scientific Conference "Actual Problems of Modern Chemistry" dedicated to the 90th Anniversary of the Academician Y.H.Mammadaliyev Institute of Petrochemical Processes (Baku 2019).

8 scientific articles related to the dissertation were published in "Processes of Petrochemistry and Oil Refining" (3), "International Journal of Science and Research Methodology" (1), "International Journal of Industrial Chemistry" (1), "Pharmaceutical Sciences & Analytical Research Journal" (1), "Journal of Advances in Chemisty" (1) and "Oil Industry" (1) journals.

The name of the organization where the dissertation work was performed. The dissertation work was carried out in the "Catalytic cracking and pyrolysis" laboratory and in the Testing laboratory of Azersun Holding LLC according to the plan (State registration No. 0113 Az 2039) of scientific research works conducted at the Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan.

Volume and structure of the work:

The dissertation consists of an introduction, 4 chapters, conclusions, and a bibliography of 184 cited titles. The work consists of 169323 (introduction 11300, chapter I 50932, chapter II 27683, chapter III 47302, chapter IV 29873, results 2233), includes 24 tables, 35 graphs and 27 figures.

In the introductory part of the dissertation, the relevance of the work, the purpose of the research, issues ahead, the scientific novelty of the work, practical importance, approbation of the work and areas of application are described.

In the first chapter - in the literature review, the synthesis of carbons and nano-sized carbon materials based on pyrolysis liquid products (PLP) and their processing, properties and fields of application were explained and the mechanism of nanocarbon growth was investigated. Also, the composition and properties of vegetable oils, their refining process and methods were investigated.

In the second chapter, the characteristics of the used raw materials and the physico-chemical properties of the obtained activated carbon, nanocarbon and sunflower oil, the general rules of conducting experiments and the analysis methods of the obtained products were discussed.

In the third chapter, the structure and properties of activated carbon obtained from HPR and nanocarbon obtained from the liquid products of pyrolysis were studied, the diversity of the influence of various factors on their formation and development was investigated, and the scientific basis of these factors was presented.

In the fourth chapter, the application of processed activated carbon and nanocarbon in the process of bleaching sunflower oil in industry and the obtained results were presented.

Literature information related to the specific topic of each chapter is given both at the beginning of the topic and during the discussion of the results throughout the research.

The indicators presented in the dissertation work are the average results of several parallel experiments and are stably repeated.

THE MAIN CONTENT OF THE WORK

The amount of HPR obtained as a by-product in the EP-300 complex varies from 4,5 to 8,2% of the total raw material, depending on the nature of the raw material taken, including the temperature in the pyrolysis furnace. The hydrocarbon composition of heavy pyrolysis resin is very complex, but in all cases polycyclic aromatic compounds dominate the composition. Examples of condensed cyclic hydrocarbon systems include naphthalene, anthracene and phenanthrene, naphthacene, pentacene, and their alkyl- and alkenyl derivatives.

Liquid products of pyrolysis - pyrocondensate and HPR obtained at the EP-300 unit are sold abroad at a low price, but it is possible to obtain valuable products for the petrochemical complex and industries.

Cleaning and activation of carbon using alkali

In several literatures, it has been mentioned that the activation of carbon with NaOH has several advantages compared to KOH. For example, the price of NaOH is cheap, effective results can be achieved even at low doses, it is relatively less harmful to the environment, and it is less corrosive. Considering what has been said, in our research, carbon samples obtained from HPR were activated with NaOH solution, then the chemical and textural indicators of AC were studied, and the effectiveness of vegetable oil refining as an adsorbent was comparatively studied.

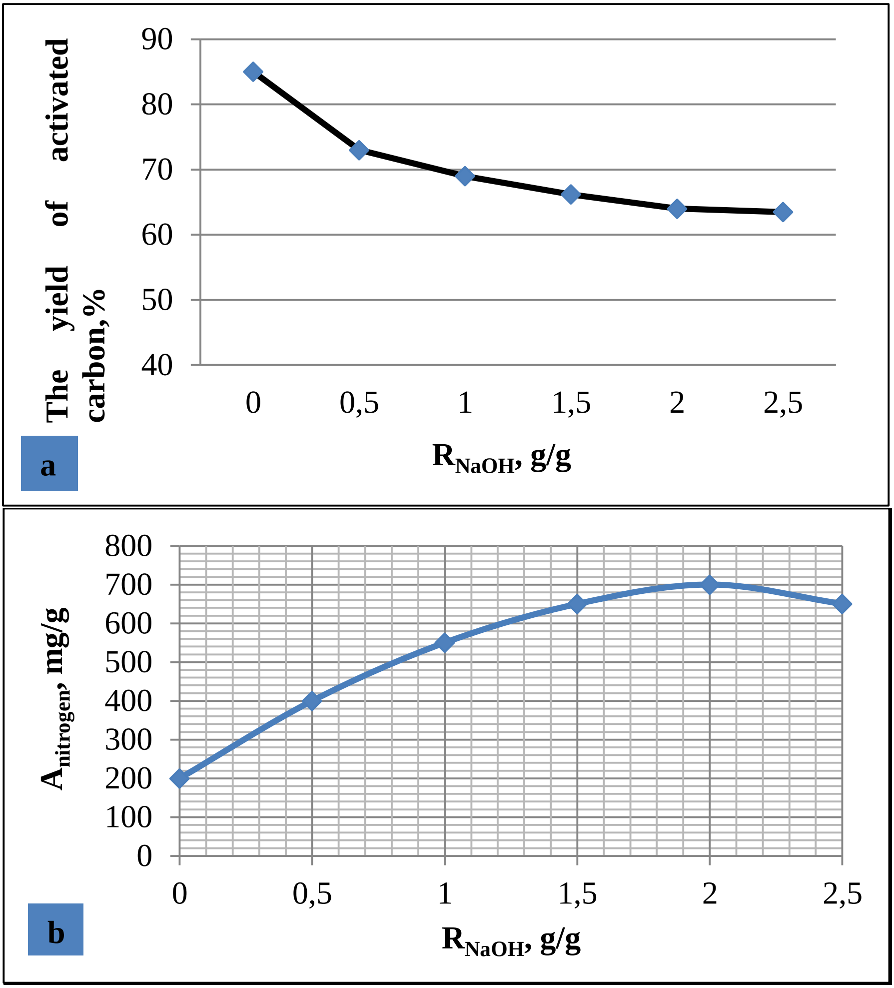
The carbon material synthesized from the HPR was mixed with pure NaOH and 10 ml of water in certain ratios of 0,5:1; 1:1; 1,5:1; 2:1 and 2,5:1 (NaOH:carbon) were mixed in a vertical steel reactor with a magnetic stirrer for 2-3 hours and dried at 130°C for 4 hours. The reactor consisting of a dry mixture was placed in a furnace with a nitrogen gas flow of 100 cm^3 per minute and heated to 700°C by increasing the temperature by 20°C per minute and treated at this temperature for 1,5 hours. After cooling, the mixture was washed with 0,1 M HCl solution and then with hot distilled water until the pH value was about 6,5, to remove residues of the activating agent and other inorganic compounds formed during the process. During the washing phase, AC was separated using a 0,45 μm membrane filter, and the obtained carbon samples were dried at 110°C for 24 hours.

The influence of the amount of alkali, as well as temperature and thermoprocessing time, on the yield of AC was studied experimentally. In order to determine the dependence of the thermal treatment of HPR on the amount of NaOH used, their mechanical mixtures in the ratio of 0,5-2,5 g/g were prepared and they were pyrolyzed in nitrogen atmosphere at different temperatures.

The effect of alkali amount on activated carbon yield and adsorption properties is shown in graph 1. It was determined that when increasing the amount of alkali, as a result of its destructive effect, the output of AC decreases 1,3 times (Graph 1 (a)). Decreasing the amount of alkali deteriorates the sorption properties of the material and, accordingly, the development structure of micropores, and also directs the process to the unstable regulation region. By increasing the amount of alkali to 2,0 g/g, it was possible to increase the nitrogen adsorption capacity of AC up to 700 mg/g (Graph 1 (b)). If the amount of the final product decreases as a result of the effect of alkali, the adsorption capacity increases, that is, radical changes occur in the surface properties of activated carbon.

It should be noted that in addition to increasing the amount of alkali, increasing the temperature also has a strong effect on the yield and adsorption capacity of the obtained carbonized material. Although increasing the pyrolysis temperature to 800°C improves the adsorption properties of the obtained AC, it has a negative effect on the yield and greatly complicates the selection of the reactor for the process due to chemical corrosion or abrasion.

Thus, if activated carbon is obtained with an 80% yield during processing of alkali in the amount of 2,0 g/g and at 400°C for 2,5 hours, the carbonization process is accelerated by increasing the processing temperature, and as a result of the separation of unstable compounds from the system, 700°C processing temperature, the carbon yield is 62%. At this time, the adsorption capacity of AC increased from 300 mg/g to 750 mg/g. No significant change in carbon output and adsorption capacity was recorded during the subsequent temperature increase.



Graph 1. The effect of the amount of alkali on the yield (a) and adsorption capacity (b) of activated carbon (processing conditions: T-700°C; t=2,5 hours)

The effect of the thermoprocessing period on the yield and adsorption capacity of the obtained AC was investigated. During the pyrolysis of HPR, the decomposition and structuring processes begin after 30 minutes, end within 2,5 hours, and during this time the output of activated carbon stabilizes, the adsorption capacity of the obtained activated carbon for nitrogen reaches a maximum of 700-750 mg. Further increase of the thermotreatment time has little effect on the yield and adsorption capacity of activated carbon.

According to the presented results, it can be said that thermotreatment with NaOH should be carried out under the following conditions:

1. The dosage of NaOH relative to carbon is 2,0 g/g;
2. Chemical activation temperature in the reaction mass - 700^{o}C;
3. Thermoprocessing time – 2,5 hours

Increasing the amount of NaOH also affects the specific surface area, pore diameter and volume of carbon. The BET specific surface area, total pore volume and average diameter of the samples are given in table 1.

Table 1.
Surface parameters of carbon samples

Samples	C_{BET}	S_{BET} (m^2/g^{-1})	V_P (cm^3/g^{-1})	d_p (nm)
Activated carbon	105,4	80	0,109	48,18
AK (R_{NaOH})=1,0 g/g	123,2	105	0,2328	39,09
AK (R_{NaOH})=2,0 g/g	131,8	210	0,3651	29,12

Here, C - energy constant (first layer); V_p - total volume of pores; d_p is the mean diameter of the pore.

Thus, when 2,0 g/g NaOH is used, S_{BET}=210 m^2/g^{-1}, while the pore diameter is d_p=29.12 nm, with 1,0 g/g NaOH, these indicators are respectively 105 m^2/g^{-1} and 39,09 nm. It is clear from the table that the adsorption capacity of the carbon sample obtained at low amounts of NaOH (1,0 g/g) and, accordingly, the reduction of the specific surface area, pore diameter and volume.

Effect of alkali/carbon ratio on the morphology of the obtained activated carbon

Pyrolysis of HPR with alkaline presence has a great effect on the surface morphology of sorbent. When we look at the microscopic images of AC obtained at different R_{NaOH} values in Figure 1, we see that a large amount of small pores are formed in the sorbent during the treatment process with the presence of alkali, and the sorbent has a perfect sponge structure.

a	b	c

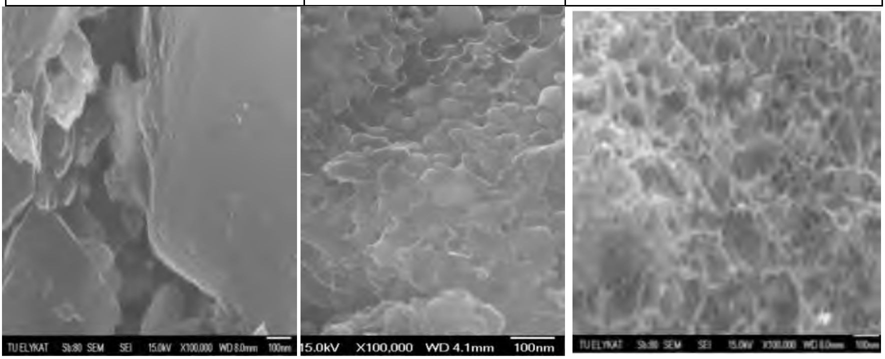


Figure 1. SEM images of carbon samples obtained from thermal processing with heavy pyrolysis resin a - alkali-free, b - R_{NaOH}=1,0 g/g and c - R_{NaOH}=2,0 g/g amounts

It should be noted that in Figure 1 (a), only large-sized pores in the form of cracks exist in the primary carbonized material. In the initial sample not treated with alkali, the presence of aggregates with primary amorphous particles, characterized by a characteristic fibrous-needle-like structure, is clearly visible. This confirms the lack of order in the structure of the crystal lattice.

It is clear from the images of the scanning electron microscope that the samples treated with alkali have a micro- and mesoporous structure, and the microporous structures are dominated here. As a result of increasing the amount of alkali to 1,0 g/g in the pyrolysis process in Fig. 1 (b), the reduction of the pores of the particles and the uniform distribution along the surface are clearly visible in the electron microscope images.

As can be seen from Figure 1 (c), the microstructure of carbon obtained from the thermal treatment of HPR with an alkali content of 2,0 g/g resembles a porous skeleton with partitions, and the granulometric composition is homogeneous. Here, thin thread-like fibers are arranged parallel to each other, the pores have a spherical shape, and nano-sized particles with sizes of 30-45 nm are predominant.

Study of chemical composition of carbon and activated carbon obtained from heavy pyrolysis resin

Table 2 shows the chemical composition of the carbon obtained on the basis of (HPR) and the sorbents obtained from its processing with alkali, determined by XGT-7000 device by X-ray fluorescence analysis method.

Table 2.
Metal content of carbon samples

Samples	The amount of metals, %							
	C	H	S	Ti	Cr	Mn	Fe	Ni
Carbon from HPR	89,3	10,34	0,35	0,004	0,01	0,011	0,07	0,02
Activated carbon, (R_{NaOH}=2,0 g/g)	95,6	4,21	0,11	0,002	0,01	0,07	0,04	0,01

From the results of the elemental analysis, it appears that the degree of carbonation is higher in the presence of alkali. While the amount of carbon in the carbonized material is 89,3% without alkali, it is 95,6% in the carbonized material obtained with the presence of alkali. At the same time, a small amount of Ti, Cr, Mn, Fe, Ni, Co elements are also found in the carbonized material. It should be noted that a decrease in the amount of sulfur and metals was recorded in the carbon sample taken in the presence of alkali.

Synthesis of nanocarbon, study of its structure and properties

A number of factors have a strong influence on the process of obtaining carbon nanoparticles by chemical vapor deposition (CVD) method, and by choosing them correctly, it is possible to control the parameters of the obtained nanocarbon. In order to determine the role of the catalyst in obtaining nanocarbon, the pyrolysis process was carried out in the temperature range of 750-920°C without a catalyst and with the presence of a catalyst.

50 ml of C_5 fraction without catalyst was pyrolyzed at 750°C and 920°C temperatures and under 1 mm Hg pressure for 30 minutes. Experiments have shown that regardless of the temperature, nanocarbon particles without a catalyst are practically not obtained. In this case, the hydrocarbons contained in the C_5 fraction break down and turn into soot, smoke, and other gas hydrocarbons with an amorphous structure. The processed solid particles settle on the walls of the quartz tube, where the amount of nanoparticles formed is negligible. These facts show that the catalyst has a special role in the formation of nanocarbon particles.

From the analysis results mentioned in Table 3, it is clear that it was practically impossible to obtain nanocarbon from the pyrolysis of the C_5 fraction without a catalyst. By increasing the pyrolysis temperature from 750ºC to 920ºC without a catalyst in the presence of MgO carrier, it was possible to increase the product yield to only 2,84% by mass.

The results of experiments conducted at 750, 850 and 920ºC using ferrocene, an easily sublimated compound of iron, as a catalyst show that the yield of the product at low temperatures with the presence of the catalyst is up to 3,93%, while at 920ºC this indicator is 9,37%. At the same time, the carbon content of the obtained product is higher – 99,1%. The amount of the catalyst is 2% based on the raw material. As the temperature rises, an increase in the amount of carbon from 97,1% to 99,1% and a decrease in the amount of hydrogen from 2,4% to 0,6% is observed. The obtained results show that temperature and catalyst play a special role in carbon synthesis.

Table 3.

Amount of C and H elements in carbon samples obtained from pyrolysis of C_5 fraction under different conditions

Samples	Output, %	Amount of elements, %	
		Carbon	Hydrogen
Product obtained with MgO carrier without catalyst at 750ºC	0,068	-	-
Product obtained with MgO carrier without catalyst at 920ºC	2,84	-	-
Product obtained in the presence of MgO carrier and ferrocene catalyst at 750ºC	3,93	97,1	2,8
Product obtained in the presence of MgO carrier and ferrocene catalyst at 850ºC	4,07	98,2	1,72
Product obtained in the presence of MgO carrier and ferrocene catalyst at 920ºC	9,37	99,2	0,7
Product obtained in the presence of halloysite carrier and ferrocene catalyst at 920ºC	7,37	98,3	1,4
Product obtained in the presence of halloysite carrier and manganocene catalyst at 920ºC	8,8	97,2	1,3
Product obtained in the presence of MgO carrier and manganocene catalyst at 920ºC	7,95	98,4	1,2

When using a manganese-containing compound as a catalyst and halloysite ($Al_2Si_2O_5(OH)_4$) as a carrier, a decrease in the yield of pyrolysis products and the amount of carbon, and an increase in the amount of hydrogen is observed. This fact shows that the recovery of nanocarbon from the pyrolysis process also depends on the nature of the catalyst and carrier. The mass fraction of carbon in the sample taken at 920ºC with the presence of halloysite carrier and manganese catalyst is relatively low - 97,2%.

The transfer of elements from the catalysts and carriers used in the synthesis process to the composition of carbon nanostructures was determined by doing analyses. As a result of iron and magnesium analysis, the carbon sample taken at 920ºC with MgO carrier and ferrocene catalyst contained a small amount of 0,03% Fe and 0,002% Mg, and the carbon sample taken at 920ºC with MgO carrier and manganese catalyst was found 0,05% Mn and 0,009% Mg. Al content was 0,004%, Si content was 0,007%, and Fe content was 0,07% in the carbon synthesized with the presence of halloysite carrier and ferrocene catalyst at 920ºC. As a result of the comparison of the obtained results, it is clear that a certain amount of metal passes from the carrier and catalyst into the carbon samples.

Table 4 shows the results obtained from the C_5 fraction pyrolysis process in the CVD unit at a temperature range of 750-920ºC. If we look at the table, we can see that at 750ºC, in the ratio of H_2:Ar=1:1, the yield of nanoparticles is very low and is only 7,8%. As a result of raising the temperature to 920°C, the increase in conversion in the pyrolysis process is noticeable, and already at 850°C, the yield of nanoparticles reaches 13,4% even in the ratio of H_2:Ar:C_5=1:1:1 within 10 minutes. The effect of time on the process showed that when the duration of the pyrolysis process is increased to 20 minutes, the amount of nanoparticles increases to 15,9%. It should be noted that increasing the concentration of hydrogen in the system increases the yield of nanoparticles, and as can be seen from the table, at a temperature of 920°C, this indicator is 18,7% in the ratio of 2:2:1, and 19,8% in the ratio of 4:2:1. Thus, it was determined that the gas mixture ratio affects the yield of the target product.

Table 4.

The output of carbon nanotubes at different temperatures and gases supply volumes

Temperature, °C	H_2:Ar:C_5, volume	Reaction time, min.	Output of carbon nanotubes, %
750	1:1:1	10	10,2
	1:1:1	20	12,8
	2:2:1	20	13,5
	4:2:1	20	15,1
850	1:1:1	10	13,4
	1:1:1	20	15,9
	2:2:1	20	17,4
	4:2:1	20	18,6
920	2:2:1	20	18,7
	4:2:1	20	19,8

From the experiments, it was determined that when the duration of the process is increased from 10 to 20 minutes, there is a linear increase in the diameter and height of the obtained carbon nanoparticles, although there is no change in the diameter in the interval of 20-30 minutes, there is a density in the heights. Thus, since new nanotubes are formed in the first moments, this leads to an increase in the total number of tubes and finally the diameter of carbon nanotubes (CNT) increases. As the CNTs approach their critical size, distortions in the orientation of the tubes appear, which leads to an increase in "defective" structures and, as a result, carbon nanoparticle growth slows. In the time period above 20 minutes, the carbon bonds are broken, as a result, the amount of carbon particles with an amorphous structure increases.

In the first moments of the process, although the amount of H_2 gas supplied to the reactor is kept constant throughout the process, the amount decreases during the first 20 minutes, but with time, the amount of gas begins to increase linearly. This is explained by the fact that at the beginning of the process, since the pyrolysis process in the hydrocarbon is not yet at the required level and therefore hydrogen is not obtained, the hydrogen gas introduced into the process is used to remove the amorphous carbon particles contained in the synthesized nanoparticles from the system. As the pyrolysis process intensifies, hydrogen gas begins to form in the system as a result of decomposition, and since H_2 gas is constantly supplied to the system from outside, this gas increases in the system. Thus, the supply of hydrogen gas to the system performs two main functions: it ensures the formation of synthesized CNTs, and it regulates the amount of free carbon received by preventing the rapid decomposition of the hydrocarbon feedstock supplied to the system.

Based on the obtained results, it was determined that the optimal ratio of precursor and hydrogen supply to the system for the process of obtaining CNTs is 4:2:1.

Study of the physicochemical parameters of the synthesized nano carbons

In order to remove the amorphous carbon particles and catalyst fragments formed in the synthesized process in the sample, it was treated with concentrated sulfuric acid at a temperature of 90°C for 2 hours, and a 15-17% decrease in the mass of the sample was recorded as a result of the treatment. Thus, it was determined that the dissolved part mainly belongs to amorphous carbon.

The phase composition of the obtained carbon samples was studied using the X-ray phase analysis method. Figure 2 shows the comparative diffractograms of nanocarbon samples synthesized and purified from mixtures by acid. It is clear from the picture that as a result of treatment with acid, structuring occurs in the carbon nanostructure as a result of impurities removal, a crystalline phase characteristic of nanocarbon is formed.

In the diffractogram of nanocarbon, peaks corresponding to the metal phase were detected at 36,89, 42,87 and 62,27 degrees. At the same time, a broadened peak is observed at θ= 26 and 44,59 degrees corresponding to the nanocarbon phase with dispersed graphite structure. As can be seen from Figure 2, after purification, only the peaks corresponding to the crystalline carbon phase are observed.

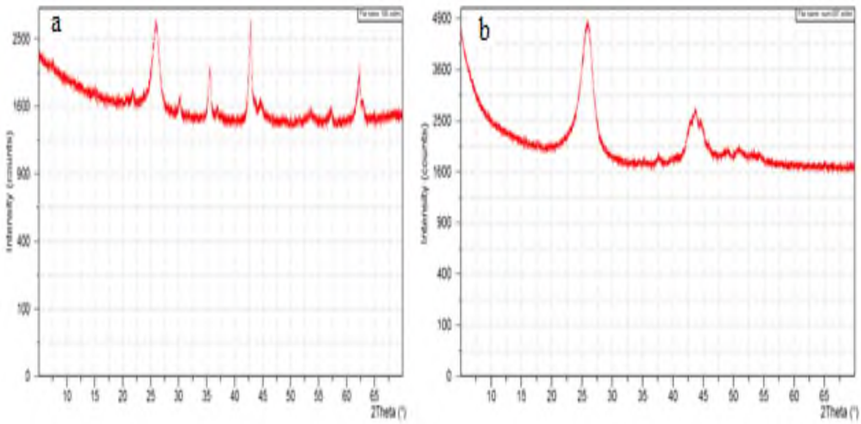


Figure 2. The diffractogram of the nanocarbon sample obtained from the pyrolysis of the gas mixture in the ratio H_2:Ar:C_5=4:2:1 at 920°C and purified from the mixtures: a) initial; b) after treatment with sulfuric acid

A scanning electron microscope was used to determine the shape, structure, and size of the particles in the obtained carbon samples. As a result of the analysis of SEM images in Figure 3 (a), it was determined that the height of the nanocarbon particles synthesized at 750°C was 70±5 nm, the diameter was 31±3 nm, and they were composed of CNTs oriented in different directions. By increasing the temperature to 850°C (Fig. 3 (b)), this disadvantage - irregular orientation is eliminated, and the geometric dimensions of CNTs are approximately in the same range (65±5 nm height and 27±4 nm diameter). This effect can be associated with the easy desorption of hydrogen formed during the decomposition of the C_5 fraction from the catalytic centers, because the carbon layer formed on these centers is less "defective" and does not allow the carbon nanotube particles to branch or lose orientation. Increasing the temperature to 920°C (Figure 3 (c)) leads to an increase in the flexibility of the active centers, as a result of which the small catalytic centers are combined with each other and become larger centers. The increase in the size of the active centers is also manifested in the obtained nanoparticles, so the diameter of CNTs increases to 52±5 nm, and the height to 86±8 nm. Also, there are carbon particles with larger sizes here. The growth recorded with increasing temperature confirms the growth of carbon nanotubes during the activation and heating stages. Indeed, the virtual absence of particles smaller than 25 nm in CNTs proves absorption by smaller active centers or sublimation of smaller particles at that temperature. At the same time, increasing the temperature accelerates the process of desorption of the hydrocarbon feedstock from the surface of the samples, which results in a relative reduction of the yield at higher temperatures.

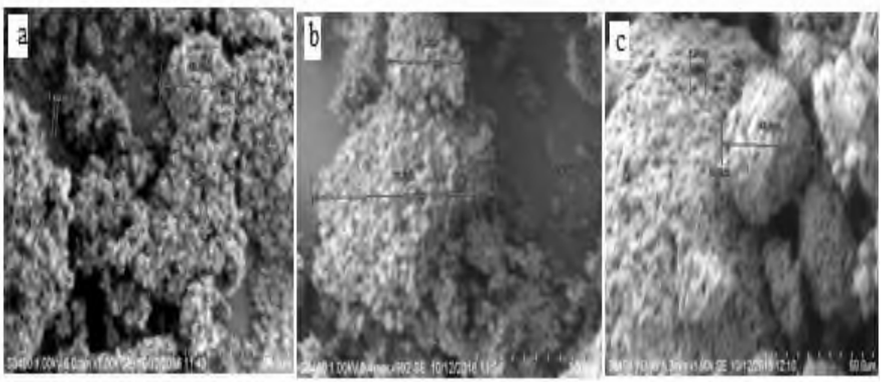
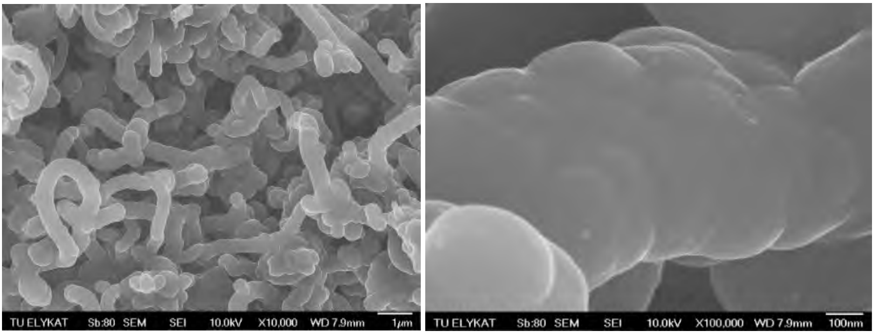


Figure 3. SEM images of carbon nanostructures obtained at different temperatures: a) 750°C; b) 850°C; c) 920°C (1000 times enlarged)

In Figure 4 (a), the tubes are clearly visible in the microscopic images of the particles at 10,000 times magnification. The length of the tubes is 60-100 nm, and the diameter is 3-10 nm. If you look closely at the 100,000 times magnified image in Figure 4 (b), it can be seen that the surface of the tubes consists of oval-shaped parts. Such a structure further increases the surface area and pores of the carbon particles.

Thus, as a result of the experiments, it is clear that when comparing nanocarbons synthesized at temperatures of 750, 850 and 920°C, increasing the temperature, hydrogen and argon gases has a positive effect on the yield of nanocarbon. After cleaning with acid, the diameter and height of CNTs obtained at 920°C are higher than those obtained at other temperatures.



a) b)

Figure 4. SEM image of carbon nanotubes obtained at a temperature of 920°C: a) 10000 times enlarged; b) 100,000 times enlarged

Based on experiments with HPR, the adsorption capacity of carbon thermotreated with 2,0 g/g of NaOH relative to carbon at a temperature of 700°C for 2.5 hours was the highest - 750 mg, and this activated carbon was marked with AK1, and involved in the bleaching process of sunflower oil.

Nanocarbon samples NK1, NK2 and NK3 obtained on the basis of nanocarbon synthesis experiments from liquid products of pyrolysis at temperatures of 920, 850 and 750°C, with the presence of MgO carrier and ferrocene catalyst in H_2:Ar:C_5= 4:2:1 volume ratios, during 20 minutes processing time marked with and involved in the bleaching process of sunflower oil to determine the adsorption properties.

A commercial pure multi-layered carbon nanotube (≥ 98% pure) with an average diameter of 6-13 nm, an average length of 100 nm, and a BET surface area of about 240 m^2/g was obtained and compared with carbon samples synthesized by us. This CNT used in the experiments is labeled KNB1.

Activated carbon with a commercial particle size of 0,4-2,4 mm and a BET surface area of about 220 m^2/g was obtained, and this carbon used in the experiments was designated AK2.

A general comparison of synthesized and purchased adsorbents used in the bleaching process is given in Table 5.

Table 5.

Comparative table of adsorbent samples

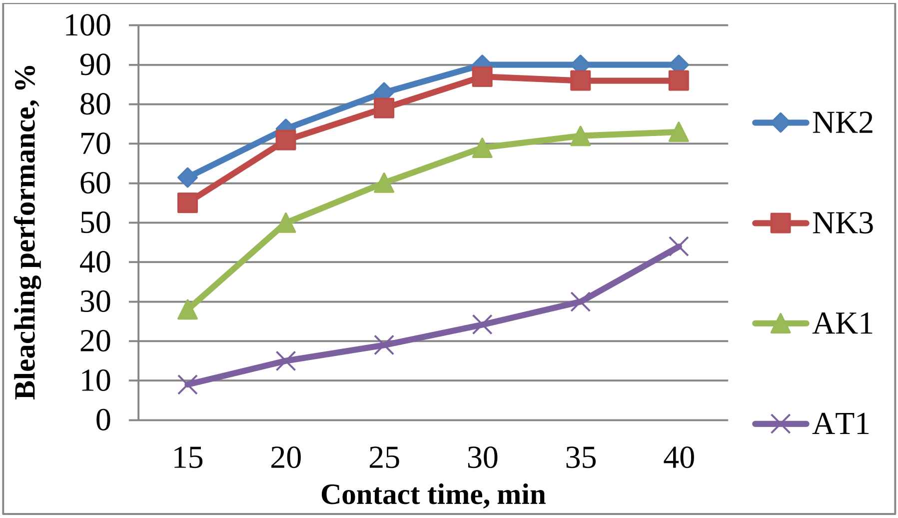
Adsorbents	Amount of carbon, %	Diameter, nm	Average length of tubes, nm	BET surface area, m^2/g
NK1	99,1	9±3	86±8	220
NK2	98,2	9±3	70±5	210
NK3	97,1	7±3	65±5	210
KNB1	≥ 98%	6-13	100	240
AK1	95,5	29,1	-	210
AK2	≥ 98,5	34	-	220

STUDY OF CARBON MATERIALS AS ADSORBENT IN THE BLEACHING PROCESS OF EDIBLE OILS

The carbon materials obtained from the pyrolysis of HPR and C_5 fraction of pyrocondensate were studied as adsorbents in the bleaching process of sunflower oil, the effect of temperature, contact time, amount of adsorbent on the process was studied, the obtained results were compared with commercial bleaching earth.

The effect of contact time on the bleaching process

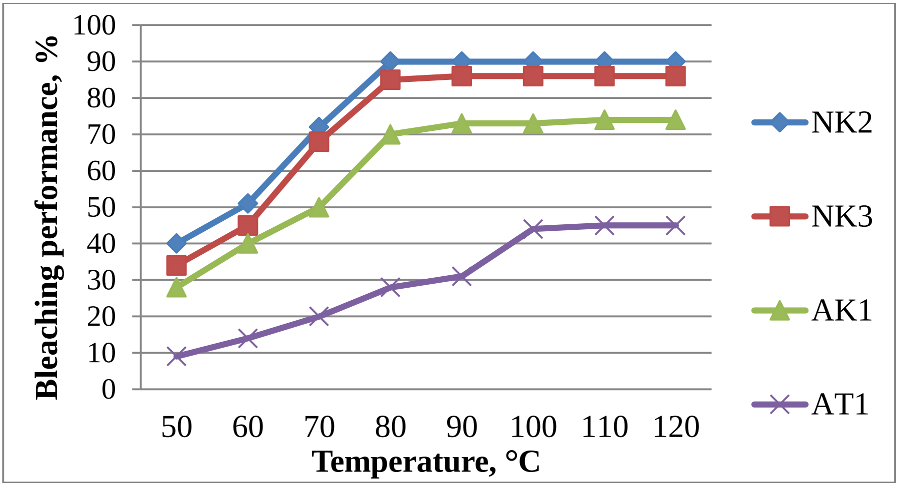
Synthesized nanocarbons - NK2, NK3, activated carbon (AK1) and commercial activated carbon (AK2) and bleaching earth (AT1) samples were compared. In this process, the temperature is 100°C based on literature data, and the adsorbent amount is 1,0 grams, and the bleaching performance is calculated based on the obtained color changes and a graph is made. It is clear from graph 2 that the bleaching performance of vegetable oil in the first 15 minutes is 61% with NK2, 55% with NK3, 28% with AK1, and 9% with bleaching earth AT1. When the duration is increased to 30 minutes, these indicators increase to 90%, 86%, 70% and 23%, respectively. It is clear from the graph that while further increase in time had little effect on the adsorption capacity of the carbon samples, an increase in bleaching performance up to 44% was recorded with bleaching earth within 40 minutes. Thus, as the contact time increased, it was observed that the absorption of pigments also increased.



Graph 2. Effect of contact time on the bleaching process: with 100°C temperature of and 1% mass content

The effect of temperature on the bleaching process

Graph 3 shows the effect of temperature on the bleaching process of sunflower oil with the presence of synthesized carbon samples and bleaching earth. The bleaching process was carried out for 30 minutes, in the amount of 1% of adsorbents. As can be seen from the graph, as the temperature rises, the degree of bleaching also increases, as a rule. 90% and 85% maximum bleaching was achieved with NK2 and NK3 adsorbents at 80°C. 70% and 28% bleaching rate was achieved with AK1 and AT1 samples at 80°C temperature. The maximum performance during bleaching with activated carbon was at 90°C, and when using bleaching earth at 100°C. As reported in the literature, color absorption from oil increases as the interaction between earth and oil increases with increasing temperature, and only 44% bleaching was achieved at 100°C. No color change was observed during further temperature increase.

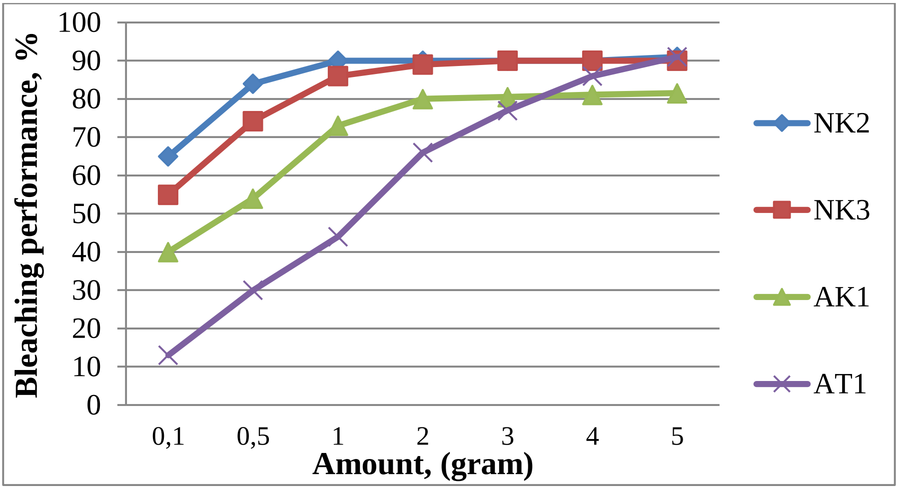


Graph 3. Effect of temperature on the bleaching process: with 30 minutes contact time and 1% mass content

The effect of the amount of adsorbent on the bleaching process

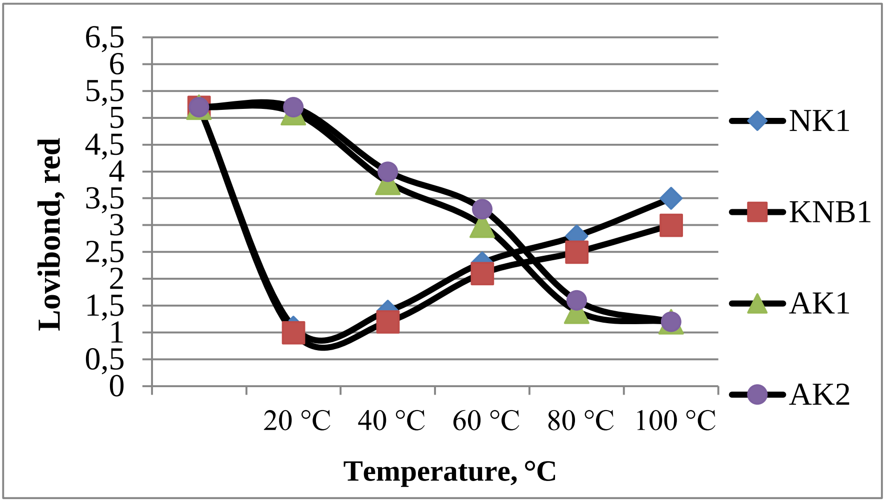
The bleaching of the oil is directly proportional to the increase in the amount of adsorbent. From graph 4, it is clear that a sharp difference between adsorbents is observed when using adsorbent at 0,1%. When this amount was increased to 0,5%, a sharp increase in NK2 and NK3 adsorbents was observed. While NK2 sample 1%, NK3 sample 2%, AK1 sample 2% provided the maximum bleaching of oil, the best result with bleaching earth was recorded at 5%. A further increase in the amount has very little effect on the bleaching capacity of the carbon samples. With bleaching earth, this indicator increased to 90% with an increase in amount up to 5%, and further increase in amount had little effect on bleaching.

The carbon nanotube sample (NK1) synthesized at 920°C was found to show different properties compared to NK2 and NK3, and a comparison with commercial carbon nanotube (KNB1) and activated carbon (AK2) was also conducted. The influence of temperature on the bleaching process is shown in graph 5. The bleaching process was carried out for 30 minutes using 1,0% adsorbent at temperatures between 20 and 100°C. From the graph, it can be seen that carbon nanotubes (NK1) adsorbed colored particles from oil at 20°C, that is, at room temperature, and the red color decreased from 5,2 to 1,1. That is, by absorbing the colored particles in the oil, about 79% bleaching performance was achieved.



Graph 4. The effect of the amount of adsorbent on the bleaching process: with 30 minutes contact time and 80°C temperature

However, as can be seen from the figure, increasing the temperature has a negative effect on the process. That is, increasing the temperature does not increase the adsorption capacity of carbon nanotubes, on the contrary, the desorption process in oil begins. The reason for this may be that increasing the processing temperature negatively affects the surface properties of carbon nanotubes and disrupts the structure of the tubes. However, NK2 and NK3 samples maintain their adsorption property as the temperature increases and reach maximum adsorption at 80°C. When comparing samples of AK1 and AK2, it is clear that the adsorption capacity of both adsorbents is directly proportional to the temperature and reaches the maximum adsorption (1,2 red color) at 100°C.



Graph 5. Color temperature dependence curves: with 30 minutes contact time and 1% mass content

Experiments were conducted at 20°C and 30 minutes of mixing time, and it was found that 1,3 grams of carbon nanotube NK1 and 1.2 grams of KNB1 completely decolorized the oil. From the obtained results, it is clear that carbon nanotubes have the ability to adsorb pigments from oil even at room temperature. After bleaching with NK1 adsorbent, physico-chemical analyzes were conducted and the composition was studied, the results are given in table 6.

As can be seen from the table, a decrease in free fatty acids

(FFA) and peroxide analyzes was observed after bleaching. It shows that the oxidized compounds and free fatty acids contained in the oil are also adsorbed by NK1. Also, complete decolorization of oils by removing the pigments contained in the oil and giving it color proves the effectiveness of the process. Also, no increase in the anisidine value was observed as a result of the bleaching process at room temperature. The fact that the analysis results of the fatty acid composition are the same before and after bleaching indicates that there is no change in the chemical composition of the oils.

Table 6.

Quality parameters of sunflower oil before and after bleaching with NK1

Samples	GOST 1129-2013	Before bleaching	After bleaching with NK1
FFA, %	≤ 0,3	0,24	0,06
Peroxide value, mmolO_2/kg	≤ 10	0,9	0,83
Anisidine value	≤ 3	4,27	4,19
Chlorophyll content, mg/kg		0,32	0,00
β-carotene content, mg/kg		0,35	0,00
The amount of color		5,2 red / 30 yellow	0,2 red / 0 yellow
The fatty acid content, %			
Palmitic acid	4,0-7,6	6,60	6,71
Stearic acid	2,1-6,5	4,48	4,55
Oleic acid	14,0-71,8	35,2	34,8
Linoleic acid	18,7-74,0	51,5	51,9

Thus, carbon nanotubes synthesized from C_5 fraction at 920°C have high efficiency in oil decolorization compared to bleaching earth and AC due to their high adsorption capacity. Also, it was shown the possibility of reducing the energy cost of the production process and preventing financial loss by ensuring that the nanocarbon samples obtained from the liquid products of pyrolysis in oil production are carried out in a small amount and at a low temperature.

Adsorption of polyaromatic hydrocarbons (PAH)

Taking into account the high adsorption properties of NK1 and AK1 adsorbents, their mixtures with bleaching earth in different proportions were prepared and applied in the adsorption of PAHs in the bleaching process of sunflower oil. Carbon nanotube (NK1) was mixed with bleaching earth (AT1) in the following percentages and labeled: B1 - 1% NK1 and 99% AT1 mixture; B2 - a mixture of 2% NK1 and 98% AT1; B3 - a mixture of 3% NK1 and 97% AT1; B4 - a mixture of 4% NK1 and 96% AT1; B5 - a mixture of 5% NK1 and 95% AT1. Activated carbon (AK1) was mixed with bleaching earth (AT1) in the following proportions and labeled: C1- a mixture of 1% AK1 and 99% AT1; C2 - a mixture of 2% AK1 and 98% AT1; C3- a mixture of 3% AK1 and 97% AT1; C4 - a mixture of 4% AK1 and 96% AT1; C5- a mixture of 5% AK1 and 95% AT1.

The process was carried out at a temperature of 100°C, 1% amount of adsorbents, and a contact time of 30 minutes, and the effect of nanocarbon on the adsorption property of bleaching earth was investigated. Initially, no change in PAH levels was seen when using bleaching earth (AT1) as a bleaching agent in the process. However, maximum adsorption was observed when nanocarbon (NK1) and activated carbon (AK1) were used. Comparing the mixtures B1 and B5, C1 and C5, it became clear that the adsorption of PAHs increases with the increase in the amount of carbon in the mixture. The lowest PAH absorption was obtained when using C1, and the highest absorption was obtained when using B5. The results of B4 and B5 are almost similar, but the adsorption of benzo (b) fluoranthene of B5 is higher than that of B4.

It is clear that nanocarbon adsorbs especially heavy PAHs. Adsorption of heavy PAHs is greater than light PAHs when using B5.

Additionally, the effect of temperature on the absorption of PAHs was investigated and it was determined that increasing the temperature had almost no effect on the adsorption of PAHs. That is, there is no difference in PAH adsorption between the bleaching process at 40°C and 100°C, but the color absorption at 100°C is greater than that at 40°C due to bleaching earth comparing B1 and B5.

CONCLUSIONS

1. It was determined that under optimal conditions with NaOH alloy of pre-thermally treated heavy pyrolysis resin: it is possible to get activated carbon with 62% yield from pyrolysis with T=700°C; alkali:raw material=2:1 and 2,5 hours [17, 21].
2. It has been shown that the alkali metal intercalates into the carbon cage and interacts with it to create perfect micropores, and as a result, the adsorption capacity of activated carbon for nitrogen increases up to 750 mg/g [17].
3. The structure and properties of the activated carbon obtained from heavy pyrolysis resin were studied by physical methods and the surface area of the obtained carbon particles was S_{BET}=210 m^2/g^{-1}, the average diameter of the pores d_p=29,12 nm, and the volume V_p=0,3651 cm^3/g^{-1} [5, 17, 21].
4. Multi-walled nanocarbon was synthesized as a result of pyrolysis with a ferrocene catalyst placed on MgO in a temperature range of 750-920ºC in an argon atmosphere in a pyrolysis unit based on the chemical vapor deposition method (CVD) of the C_5 fraction of pyrolysis liquid products to obtain nano-sized carbon particles, the optimal conditions for the process are T=920°C, 20 minutes reaction time and H_2:Ar: C_5=4:2:1 gas mixture ratio were selected [12, 14].
5. It was determined that the nanocarbon particles synthesized at temperatures of 850 and 920°C from the C_5 fraction have a high dispersity, so they provide both a high efficiency of the bleaching process and a high filtration rate of oil at lower temperatures than known adsorbents [7, 11, 20].
6. Experimental results determined the optimal technological

regimes for the bleaching stage of sunflower oil: the temperature for nanocarbons is 80°C, the duration is 30 minutes, the amount is 1 g, and the bleaching efficiency is 86-90% [6, 16].

7. According to the Frendlix isotherms established for the bleaching process, the highest adsorption capacity occurred when using nanocarbon obtained at a temperature of 920°C (1/n value=0,81, K_f=5,36, R^2=0,9942). The obtained results show that nanocarbon and activated carbon are more effective than bleaching earth [13, 18].
8. It was determined that the refined sunflower oil obtained from the bleaching stage, which is one of the refining stages using nanocarbon and activated carbon, has a low prooxidant potential and fully meets the requirements of GOST 1129-2013 in terms of quality and safety [3, 9, 15, 19].

The main results of the dissertation are published in the following scientific works:

1. Ibragimov, H.C., Ibragimova, Z.M., Gasimova, K.M., Gasimova, G.F., Aliyev, B.M., Kolchikova, I.V., Guliyev N.G. Investigation of the process of obtaining electrode coke from heavy pyrolysis resin by catalytic treatment // Materials of the conference dedicated to the 100th anniversary of Academician S.C. Mehdiyev, - Baku: - 2 - 3 December, - 2014, vol. II, - p. 9-13.

2. Ibragimov, H.C., Ibragimova, Z.M., Gasimova, K.M., Kolchikova, I.V., Guliyev N.G. Study of the process of obtaining nano-sized carbon particles from heavy pyrolysis resin // Materials of the conference dedicated to the 100th anniversary of Academician S.C. Mehdiyev, - Baku: - 2 - 3 December, - 2014, vol. II, - p. 14-17.

3. Ibragimov, H.C., Alakbarov, C.A., Gasimova, K.M., Guliyev N.G., Kolchikova, I.V. Study of carbon obtained from heavy pyrolysis resin as an adsorbent in the process of decolorization of sunflower oil // IX Republican scientific conference of doctoral students, masters and young researchers "Actual problems of chemistry" dedicated to the 92nd anniversary of the national leader

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