Production of Nanocarbon from Local Raw Materials

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Abstract

This research aims at extraction of nanocarbon from local raw materials through a detailed study of its characteristics. The research involves study of thermal properties of fruit kernels (apricots) and walnut kernels, determining the optimal modes of the carbonization process for each type of raw material, developing a method for the synthesis of spherical granules by liquid granulation and determining the optimum concentration of the sulfuric acid solution used in granulation. Analysis of the results on the dynamic sorption of petroleum products showed that maximum sorption is achieved when using a sorbent obtained at $400°C$ for 30 minutes. Temperature regimes have been optimized for obtaining sorbent from walnut shells with different carbon content. The hydrophobicity and oleophilic of their surface are common to all these materials. The dynamic conditions of the adsorption capacity processes under the sorbents were studied. Sorption efficiency and

the specific capacity of sorbents were determined. It has been shown that the technological cycle of the studied sorbents can be repeated by repeated purification. When activated charcoal is used to purify water from petroleum products, the process of leaching with colloidal and finely dispersed mixtures – it was found that deaeration is necessary. The individual composition of the ether fraction OV-1 has a fixed liquid phase, Connected to Finuigan 4023 Automated GC/MS System mass spectrometer, It was determined by chromato-mass spectrometry in a capillary column 40 m long and 0.25 mm in internal diameter. The results of the Fure-IR spectroscopy study of samples based on the apricot peel, walnut peel, and walnut peel showed that winning symmetrical bands (2920 and 2851 cm^{-1}) were observed in them, these bands belong to the C-H-valence oscillation belonging to the methyl and methylene groups. In the spectrum taken from a sample of apricot peel, absorption bands belonging to cellulose (1377 cm^{-1} and 1069 cm^{-1}) were observed.

Thermogravimetric studies were performed on an STA 449C Jupiter synchronous thermal analysis instrument in the inert environment at a heating rate of 10°C/min in the temperature range of 25–1000°C. Analysis of the porous structure parameters of carbon sorbents were carried out using the high-speed analyzer of gas sorption NOVA-1200e by the method of imaging nitrogen adsorption isotherms at a temperature of 77K.

1 Introduction

Nowadays with growing techniques and technologies, in various sectors of the economy including pharmaceuticals, cosmetology, oil and gas refining industry, the demand for effective environmentally safe adsorbents is increasing [1–5]. Preparation of adsorbents that meet such requirements, the study of colloid-chemical properties of adsorbents, the study of the mechanism of adsorption processes in them pose new challenges to scientists and researchers in the field. A colloid is a heterogeneous combination with particles that are half the size of a solution and half the size of a suspension. The scattering of visible light by colloidal particles is known as the Tyndall effect. A colloidal dispersion of a liquid in either a liquid or a solid is known as an emulsion. Currently, activated charcoal is used in the food industry, medicine, and other fields for the treatment of gases and sewage, and other

waters [6–8]. Carbonation is the chemical process that produces carbonates, bicarbonates, and carbonic acid from carbon dioxide.

The use of charcoal, for example, in purifying water, allows drinking water to meet basic requirements [9–12], the use of charcoal as electrodes of supercapacitors allow the creation of inexpensive rechargeable electrochemical devices with high power and energy properties [13–15]. Therefore, the study of the raw material base for obtaining activated charcoal and improving its physical and technical characteristics are of particular interest [16]. Its black ash, obtained by burning walnut shells in the absence of oxygen, has sorption activity. Walnut Kernels are the wrinkled, brown-colored nuts with a brown seed coat that are edible. They come from walnut trees of the genus Juglan, which belong to the Juglandaceae family. Two rough lobes like abstract butterflies adorn the kernels.

Experimental Part

Crushed shells of walnuts, dried to a constant weight at 105°C to a size of 0.5-2.0 mm was used as a raw material. An initial shell of 100–120 g was placed in a heated hermetic aluminum reactor, argon, or helium inert gas was sent to it at a rate of 40 ml/min. The reactor was shut down and heating was carried out for 5–10 min to the set temperature after complete loss of air. Pyrolysis of walnut shells was carried out at temperatures of 200, 250, 300, 350, 400, 450, and 500°C until the separation of liquid and gaseous products ceased. The duration of pyrolysis ranged from 2 to 5 hours. The liquid products of pyrolysis were collected in a tube dried to constant weight. The gases were quantitatively collected in an oxygen bag with a capacity of 50 dm3. The gas volume was determined using a GSB-400 gas meter with a drum-type liquid valve. Identification and quantitative composition of gases have a detector for thermal conductivity, zeolite CaA (CO), and 1.5 m Ø 5 mm columns filled with Separon BD (CH4, CO2) was performed using Crystallex 4000 m chromatography. The calculation of the amount of gaseous individual substances were determined by the method of absolute calibration of the chromatograph detector. The introduction of the gaseous sample was carried out through a dispenser with a volume of 1.0 ml. The employment of chemical reactions of test specimens directly in a chromato-mass-reaction spectrometer's system. When pyrolysis was completed, the flask was weighed with liquid products, and the output of liquid products were calculated according to the formula.

$$C = (m\_1 - m)/g \cdot 100\%,$$

Where m1 is the mass of the tube with the liquid product, g; m is the mass of the empty tube, g; g is the mass of the dry shell gravity.

The mixture in the flask was then divided into two fractions: acid fraction and phenol fraction plus neutral substances. The proportion of dissociation of an acid is used in weak-acid calculations to compute the fraction of weak-acid that dissociates in a solution. The mass balance and charge balance equations may also be used to assist us solve for the unknowns. Phenolic components, which function as natural antioxidants, make the oil more resistant to storage and heating. To do this, dry sodium bicarbonate was sprayed into the flask until the carbon dioxide separation was stopped under constant stirring and extracted three times with diethyl ether (organic acids in the aqueous part in the form of sodium salts, in the extract – phenols and neutral substances remain).

The acidity of the aqueous portion was increased to pH=2 with sulfuric acid and extracted three times with diethyl ether. The individual composition of the ether fraction OV-1 has a fixed liquid phase, Connected to Finuigan 4023 Automated GC/MS System mass spectrometer, 40 m long, and was determined by chromato-mass spectrometry in a capillary a column with an inner diameter of 0.25 mm. The level of lipophilic compounds can be used to control the quality of wild carrot seeds using chromato-mass spectrometry.

Analysis conditions: linear programming model with speed from 20 to 500°C to 5°C/min; 30 minutes isothermal holding at 250; test volume at 3–5 μl 1/50 discard.

The catalyst was prepared as follows: dry mixture 200 g $Cu(NO_3)_2 \cdot 6H_2O$, 7.40g $Co(NO_3)_2 \cdot 6H_2O$, 13.68g $(NH_4)_2Mo_2O_7$ per 48 ml of water, and a mixture containing 33.20 g of glycine and 45.36 g of citric acid were added. Stirring the mixture vigorously, a viscous black-raspberry-colored mass was formed, we put it in a porcelain bowl and processed it at 250°C–260°C for 5 hours. The result is a white-gray powder. To remove organic residue, we heated it at 500°C for an additional 5 hours. The resulting catalyst was placed in a tubular quartz reactor with a diameter of 150 mm, we performed nano glycerol synthesis. To remove the catalyst compounds, we boiled the nanocarbon in concentrated HCl for 3 h, filtered and washed in distilled water until the pH was neutral. We used a Teflon filter funnel for filtering and washing. For size-selective samplers, PTFE filters offer a flexible option. The particular features of the material make it appropriate for gravimetric, chemical, and/or microscopic investigation of sample particles. Aerosol sampling, air venting, and gas filtering all require PTFE filters. As shown in Figure 1.
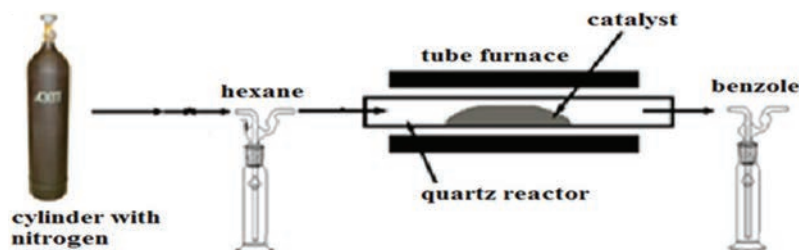
Figure 1   Nanocarbon extraction device.

Extraction of sorbents. A batch of walnut and apricot shells was selected to obtain sorbents.

Plant waste: walnut shell, apricot kernels, as well as CCOM brand coal – air-free initial heat treatment corresponding to the maximum yield of volatile substances for 4–6 hours. After grinding and sieving, the powdered carbon material was mixed with the binder in a 1:5 mass ratio and dispersed in the sulfuric acid solution for solidification. When sampling based on walnut kernels the ratio of coal powder and binder mass in the composition was changed in the range of 1:10 ÷ 1:12. Walnut Kernels are the wrinkled, brown-colored nuts with a brown seed coat that are edible. They come from walnut trees of the genus Juglan, which belong to the Juglandaceae family. Two rough lobes like abstract butterflies adorn the kernels.

The effect of sulfuric acid solution concentration on the strength of the obtained granules were studied. The acid concentration was changed in the range of 15–40%. Granules obtained at concentrations less than 30% have low strength after carbonization and scattered at a very small load. Increasing the concentration to 35% and above does not affect the quality of the granule obtained and leads to unreasonable excess consumption of acid and an increase in the amount of wastewater in the washing of the granules. The optimum concentration of the sulfuric acid solution is 30–35%, therefore, further studies were performed using this concentrated solution. Spherical granules of 0.5–3.0 mm in size were stored in an acid solution for 24–30 hours, then separated from the liquid and washed with distilled water to pH=5–6. Washed granules were first dried in the air, then in an oven for 3 hours at a temperature gradually increasing from 100°C to 300°C.

Thermal modification of spherical granules to create a porous structure is in two stages: Carbonation in a carbon dioxide atmosphere at a temperature of 1 h (500 ± 50 hour) and activation in a carbon dioxide atmosphere at the temperature of 800 ± 50°C for 1 h. Carbonate release was recorded during

the carbonation phase and the actual and imaginary (apparent) densities of the semi-finished products were measured. Carbonation is the chemical process that produces carbonates, bicarbonates, and carbonic acid from carbon dioxide. The creation of metal CO2 complexes activates carbon dioxide. Direct coordination of a transition metal–CO2 complex is one of the most potent and ubiquitous methods for inducing chemical reactions in the inert CO2 molecule. Thermogravimetric studies were performed on an STA 449C Jupiter synchronous thermal analysis instrument in an inert environment at the heating rate of 10°C/min in the temperature range of 25–1000°C.

The analysis of the porous structure parameters of carbon sorbents were carried out using a high-speed analyzer of gas sorption NOVA-1200e by the method of imaging nitrogen adsorption isotherms at a temperature of 77K.

Study of the amount of carbon in sorbents: To determine the amount of carbon and ash in the resulting sorbent, their thermal processing was carried out, for 2 hours processing time at 800°C. At this temperature, the carbon burns completely and the carbonized shell ash remains almost white. Based on the method of separation of petroleum products from water by extraction with hexane, gravimetric method, the initial concentration of oil in water was determined by the method of chromatographic separation of petroleum products from compounds of other classes in a column filled with aluminum oxide. graphy is a method for separating compounds in a mixture by having them move through the substrate at various rates, leading them to separate from one another.

The study of the effectiveness of sorbents was evaluated at a concentration of 23.5 mg /l of oil for contaminated water. Initially, studies were conducted to assess the degree of water purification, depending on the rate of passage through the immobile sorbent layer. The volumetric velocity of the water through the immobile layer of the sorbent was (6, 15, and 32 ml/min).

A column 50 cm high and 3 cm in diameter (Figure 2) was filled with sorbent 20 cm high, the starting solution is sent from above and it passes by its flow through the sorbent layer.

1,3 – glass fiber, 2 – sorbent, 4 – contaminated water.

Purified water was collected in 250 ml portions in a collector. At the exit of the column, the water is transparent – under GOST 3551-46, there is no smell in the purified water.

The small amount of residual oil in the water leads to the use of instrumental methods of analysis. Now it is not possible to determine this concentration by the gravimetric method. The mass of an ion in a pure compound may be calculated by gravimetric analysis, which can then be used
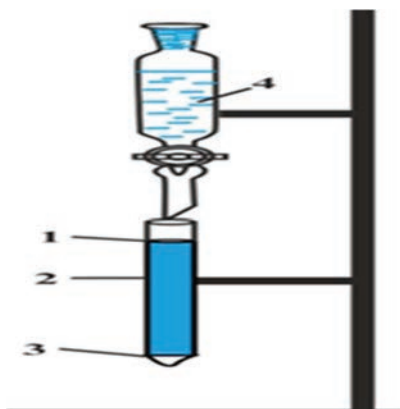
Figure 2 Device for filtering contaminated water through a stationary sorbent layer.

Table 1 Sorbent extraction mode

No.	Carbonation Temperature, °C	Carbonation Time, Min	Amount of Carbon, %	Sol, %
1	450	40	94	2,4
2	450	80	90	4
3	550	30	80	10
4	550	60	60	10

to calculate the mass percent of the same ion in a known quantity of an impure molecule. The ion under investigation has entirely precipitated. It must be a pure chemical that forms the precipitate. It is usually used in the analysis of heavily contaminated samples and cannot be used in the analysis of samples containing petroleum products because the lower limit of the measurement range is 0.3 mg/dm3 when the volume of the analyzed a sample is 3–5 dm3. the maximum allowable concentration (REK) of oil in water intended for fisheries is 0.05 mg/l.

The main methods of quantitative chemical analysis currently used in the determination of petroleum products in water is gravimetric, IR spectroscopy, gas chromatography, and fluorimetric methods. For this purpose, we conducted a water analysis for the residual amount of oil in a liquid analyzer "Fluorat-02".

Experimental Results and Their Discussion.

Data on sorbent extraction modes are summarized in Table 1.

As can be seen from Table 1, coal below No. 1 is the most efficient. Sorbent with the highest carbon content for further research on oil and
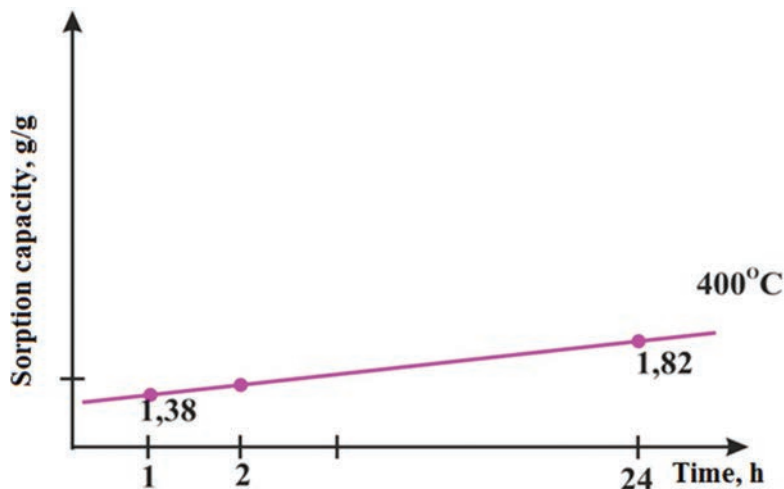
Figure 3   The time dependence of the sorption capacity of the sorbent.

water purification, a sorbent obtained at 400°C for 30 min was selected. The main indicator that characterizes the efficiency of sorbents is their sorption capacity, that is, the ability of the sorbent to absorb the maximum possible amount of sorbate per unit mass [5].

The kinetic dependences of the sorption capacity of the synthesized sorbent were studied. The quantity of adsorbate taken up by the adsorbent per unit mass (or volume) of the adsorbent is known as adsorption capacity (or loading). The mass of water absorbed per mass of desiccant is the adsorption capability of a solid desiccant for water. Such dependencies are shown in Figure 3.

For this purpose, studies on the overall absorption of oil and petroleum products (gasoline) by sorbents were conducted.

The surface capacity of the obtained sorbents for oil and petroleum products was checked. The evaluation of the efficacy of the sorbents studied in this paper was determined according to TU 214-10942238-03-95.

The initial shell of the nut had the following composition as shown in Table 2. The actual density of extractable charcoal was carried out according to generally accepted methods following GOST 7657-84.

From the data in Tables 2 and 3, it can be seen that the raw material is low-ash, high content of volatile substances, which allows the formation of various pores. It can therefore be assumed that activated charcoal from the walnut shell, in the food industry and medicine for the treatment of various

Table 2   Material balance of walnut shell

Components	Walnut shell	
	G	%
Solid residue (woodcoal)	134.5	31.28
Resin	73.9	−17.2
Water soluble substances	139.4	32.4
Pyrolysis gas, losses	81.8	19.02
Gray substances	0.12	0.027
Total	429.72	99.93

Table 3   Physicochemical properties and yield of charcoal from the walnut shell at different temperatures of pyrolysis

Pyrolysis Temperature, °C	Output of Coal, Weight %	Coal Sol, Weight %	Actual Density, g/cm^3
100	100	0.53	1.43
250	79.5	0.79	1.33
300	43.7	1.41	1.04
350	41.6	1.59	1.13
400	35.3	1.73	1.47
450	34.7	1.89	1.33
500	32.3	1.93	1.41
550	31.4	1.92	1.42

gases and effluents, such as many charcoals, can also, be used in coal-pulp and coal-smelting technologies to extract gold, because such activated carbon has a large volume of pores. Smelting is a type of extractive metallurgy that involves extracting a metal from its ore. Smelting decomposes ore using heat and a chemical reducing agent, releasing other elements as gases or slag and leaving just the metal left.

It is known that the development of a porous structure in the carbonization of carbon-containing materials depends to a certain extent on the final temperature of heat treatment. And it also depends on the heating rate to the final temperature, but the primary raw material is of predominant importance. It is the volume of micro-pores in cm^3/g that determines the distribution of the volumes of the pores of different views according to their dimensions. Loss of the main part of volatile substances in the initial heat treatment of plant raw materials lead to the formation of primary micro-porosity of the carbonaceous material, as shown in the example of the fruit seed. In addition, the loss of the bulk mass of the volatiles ensures a smooth transition of their carbonization process without compromising the integrity of the granules.
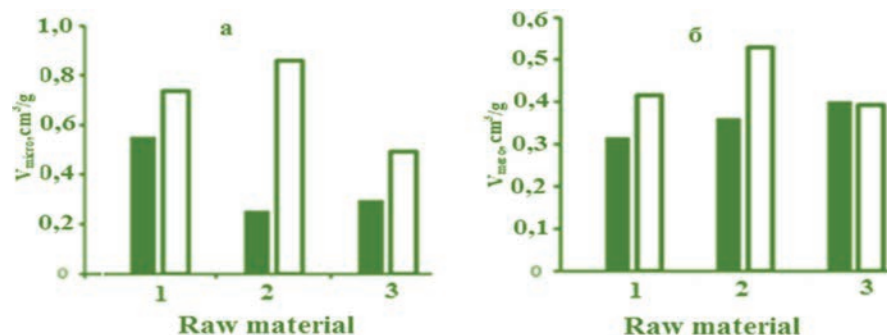
Figure 4 Changes in the volume of AK micro-pores (a) and mesopores (b) during the activation of carbonate: 1 walnut shell; 2 fruit seeds; 3 coal CCOM; Carbonate is marked in black, activated carbon is marked in white.

Carbonization is a process that involves heating biomass fuel in the absence of air at temperatures about 400°C in a kiln or retort.

Samples with the highest density of fibrous structure, with the largest release of volatile substances in the initial heat treatment, observed for walnut shells and apricot kernels and is 73–75%.

The value of the marginal volume (Wo) of the activated carbon adsorption cavity obtained during the activation process increases insignificantly, in addition to activated charcoal from apricot kernels, for which the value of Wo increases by about 2 times due to the increase in the volume of the micropores (Figure 4a).

After proper washing with 10% sulfuric acid, by subsequent rinsing with distilled water, the washed batch of the shell was ignited at a temperature of 400–500°C for 30–60 minutes to remove volatile substances in the air atmosphere, because at this temperature the formation of adsorption centers according to the literature source data occurs.

During the carbonization phase, a future activated carbon carcass – primary porosity and strength – is formed.

Nanocarbon functionalization is a common method of oxidation:

Processes in this form break down the top layer and form functional groups that retain oxygen. The functionalization of nanotubes is a different way to tune their interfacial characteristics. Aromatic chemicals, surfactants, and polymers are used to functionalize in a non-covalent way. However, the choice of oxidizer and the method of oxidation directly affects the amount and type of group. Figure 5, illustrates the groups formed on the surface of a nanocarbon as a result of its oxidation, as shown in Figure 6.
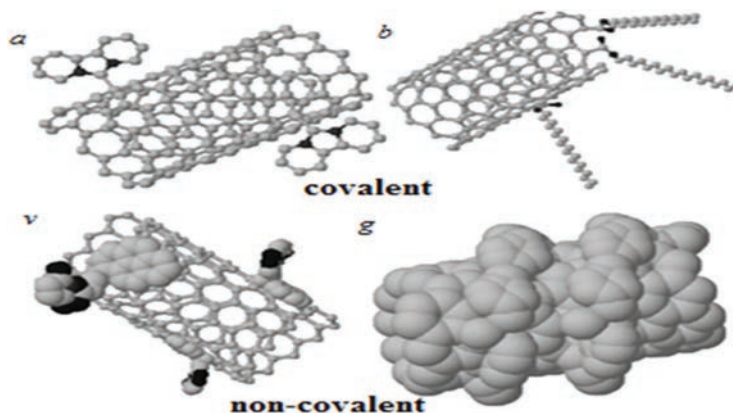
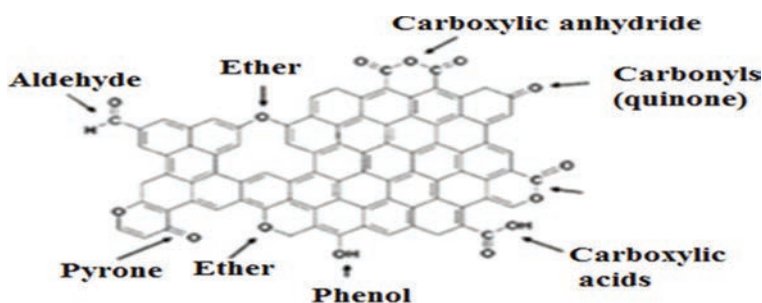**Figure 5** Different options for nanocarbon functionalization.

Figure 6 Functional groups formed on the nanocarbon surface after treatment with oxidants.

Nitric acid was used as an oxidizer along with a mixture of nitric and sulfuric acids, potassium permanganate, potassium dichromate, hydrogen peroxide.

According to the thermal analysis, the bulk of the organic matter that makes up the walnut shell is removed at a temperature of 450–550°C. Thermal analysis procedures are analytical experimental techniques that analyses a sample's characteristics in relation to its temperature.

A necessary part of carbonized organic matter is the determination of the amount of carbon because the amount of carbon depends on the degree of purification of water from oil and oil products. For this purpose, we conducted studies on the amount of carbon in sorbents synthesized by us.

Activated charcoal from apricot kernels shows the most developed micro- and mesoporous structure. Coal from walnut shells have an advanced macroscopic system. As the proportion of bark in activated carbon increases,

Table 4   Walnut shell pyrolysis gas emissions (weight% heavy)

Gaz Release	Temperature, °C					
	300	350	400	450	500	550
CO	0.24	2.02	5.63	7.2	8.70	11.25
CO_2	2.26	6.02	11.12	12.35	12.96	12.79
CH_4	–	–	0.10	0.93	2.62	3.4
	2.50	8.04	16.85	20.48	24.28	27.31

the volume of micro-and macro-pores increases, the size of the mesentery varies after passing through the maximum. The results of the study showed the possibility of obtaining spherically granulated activated carbon with an advanced system of micro-and macro-pores, their formation begins at the carbonization stage. In spherically activated coals, the average radius of the pores varies in the range of 0.527–0.635 nm, regardless of the raw material. When the amount of buckwheat husk in the composition is increased from 1:10 to 1: 2, the radius of the micro-pores in the sample increases from 0.490 to 0.724 nm.

The process of the formation of charcoal is followed by the destruction of the lingo carbohydrate complex of wood biomass.

Lignocellulose decarboxylation is the main process in the thermal transformation of raw materials at low temperatures and decarbonization, resulting in carbon monoxide and the decomposition of carbon dioxide and methane testifies to deeper changes in the ligno-carbohydrate complex at higher temperatures. It is the world's most plentiful raw material for the creation of biofuels, namely bio-ethanol. Carbohydrate polymers and an aromatic polymer make up this substance. The qualitative and quantitative composition of walnut shell pyrolysis gaseous products in the temperature range of 450–550°C is given in Table 4.

As can be seen from Table 4, carbon monoxide and methane are the carbon-shielding gases, in which methane formation is observed at 350°C and above.

Liquid, as well as gaseous products of pyrolysis and solid products are also formed. In this case, the liquid products are organic matter and pyrolysis water, and the solid product is the solid residue of pyrolysis (Table 5). The maximum yield of liquid products of thermal destruction of nuts is observed at a temperature of 300°C and 49.2% of the absolute dry gravity (m.q.t.) mass, but as the pyrolysis temperature increases, the yield of liquid products decreases to 36.0% at 500°C. The decrease in the yield of liquid products with increasing temperature may be since with the increase of the pyrolysis

Table 5 Walnut shell pyrolysis products

Products of Pyrolysis	Pyrolysis Temperature, °C							
	150	250	300	350	400	450	500	550
Solid residue	100	93.4	80.9	42.6	40.7	36.2	35.6	34.6
Liquid products	–	5.5	16.7	49.2	41.7	40.2	37.9	36.0
Pyrolysis gas	–	1.1	2.4	8.2	17.6	23.6	26.5	29.4

Table 6 Composition of fractions of neutral and phenolic compounds of liquid products of pyrolysis in the temperature range 350–550°C

	Pyrolysis Temperature, °C					
	350		450		550	
Compound	From Liquid Products %	From a.d.w %	From Liquid Products %	From a.d.w %	From Liquid Products %	From a.d.w %
2-ethyl-5-methylfuran	0.34	0.17	0.28	0.11	0.21	0.08
phenol	0.06	0.03	0.11	0.05	0.13	0.05
2,5-dimethylfuran	0.42	0.21	0.33	0.13	0.23	0.08
methylphenol	0.10	0.05	0.13	0.05	0.31	0.1
guaiacol	4.35	2.14	3.27	1.32	1.55	0.56
cresol	1.73	0.85	1.72	0.69	0.97	0.35
pyrocatechol	–	–	–	–	0.06	0.02
2,3-dimethoxytoluene	0.35	0.17	0.16	0.06	0.13	0.05
2,5-dimethoxytoluene	4.14	2.04	2.28	0.92	2.00	0.71
2,4-dimethoxyphenol	0.32	0.16	0.22	0.09	0.13	0.05
eugenol	0.46	0.23	0.27	0.11	0.26	0.09

m.q.t. – absolute dry weight.

temperature, the subsequent destruction of the liquid products occur with the coaling of a part of the liquid products or the formation of gaseous products.

It was established by chromato-mass spectrometry that, the main liquid products present in the fraction of phenols + neutral substances are aromatic phenolic derivatives hydrocarbons and furan series compounds. The identified compounds of this fraction by the mass spectrometry method is given in Table 5. The maximum yield of a liquid product at 300°C is 15.23% of the sum of all liquid products and decreases legally to 7.77% at 500°C with increasing pyrolysis temperature.

The total amount of phenolic compounds in liquid products was 60–65% of the mass of all liquid products of pyrolysis. High yield of phenols is obtained in thermal destruction of walnut shell lignin-phenol and is associated with a high amount of the main component that forms its derivatives.

Table 7   Oil concentration in water

The Name of the Sorbent	The Concentration of Oil in the Water at Different Filtration Speeds		
	6 ml/min	15 ml/min	35 ml/min
Sorbent obtained at 400°C for 30 min	0.045	0.07	0.25

As can be seen from Table 5, compounds of this fraction are represented by two groups of substances: The formation of furan derivatives occurs due to the thermal conversion of cellulose present in 32.4% of walnut shells, the phenol-derived hydrocarbons occur due to the release of phenylpropane structural units of lignin.

To determine the possibility of purification of wastewater from oil and oil products using sorbents, their sorption properties were studied under dynamic conditions by filtering the purified solution through an immobile layer of adsorbent. Water velocity under dynamic conditions in determining the properties of sorption and the oil concentration in the treated water were considered.

The data obtained on the concentration of oil in the treated water is shown in Table 7. The data obtained show that the oil concentration is lower than the REM level when the water is filtered at a rate of 6 and 15 ml/min (volume filtering level is selected). At a high filtration rate of 35 ml/min, the oil concentration is 0.25 mg/l, the REM of these fishery waters is lower than the REM of 0.5 mg/l.

Filtration rate plays an important role in water purification because the duration of the interaction of water with the sorbent is seconds and this is very important, it is necessary to have time to completely clean the water from oil in a short time. The filtration rate is the amount of water that goes through a specified sized filter in a given amount of time. Gallons per minute per square foot of filter area (gpm/ft2) is a common unit of measurement for filtration rates. It is the flow of water across a filter's surface area. The dependence of the filtration rate on the treatment of the contaminated water is shown in Figure 7.

Chemical regeneration sorbents are usually a liquid at a temperature not exceeding 100°C or gaseous organic or inorganic reagents. Chemical regeneration of the sorbents listed above is not economically feasible, because they require large amounts of reagents, and recycling the waste that is generated again becomes a problem.

To clean the contaminated coal, the most efficient and inexpensive methods were selected. Regeneration of the used sorbent was carried out by
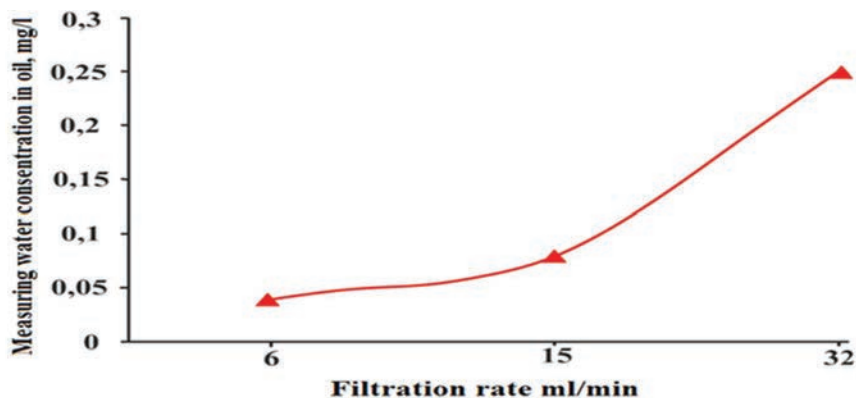
Figure 7 Dependence of the filtration rate on the treatment of contaminated water with an initial concentration of 23.5 mg/l through the sorbent layer.

mixing the used sorbent with gasoline, after its purification, the sorbent was heat-treated at 500°C to completely remove the oil products. Thus, it was found that it could be regenerated again and again due to its high strength.

According to the results of sorption properties, the sorbent should be noted separately based on carbonized walnut shells. As a product of recycling of plant raw materials, sorbent provides high purification of water from petroleum products.

Another obvious advantage of this sorbent is the active hydrocarbons, which are widely used to purify water from organic pollutants, are associated with the aeration of waters.

The surface drains are saturated with air. The presence of dissolved air in the fluctuations (vibrations) of the carbon crystal lattice leads to the spontaneous separation of micro-fabrication, their accumulation passivates the active sorption centers of activated carbon. For this reason, the necessary stage of water purification is the deaeration (degassing, deaeration) of the activated carbon filter. In addition, the process of collating the activated carbon with colloidal and finely dispersed compounds take place.

Research in the field of physical adsorption of petroleum products gives us the basic requirements for the parameters of carbon sorbents, allows us to determine their porous structure.

These micro-pores (mainly super-pores with r>0.6–0.7 nm) and meso-pores (r = 2.0–2.5 nm) feature such a maximum volume development. At the same time, the improvement of the carbon sorbents through the selection of raw materials and technological processing modes are almost
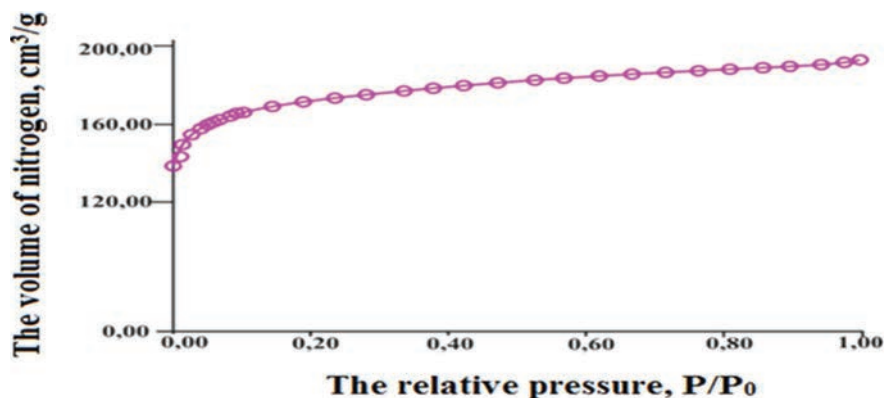
Figure 8   Nitrogen adsorption isotherms in samples of activated carbon micro powder.

over. Additional increase in the absorption properties of sorbents concerning the organic matter in water, is directly related to the directional change of their surface properties. The property that the intersection of a set, A, with any set is equal to A, or that the intersection of A with the union of A and any set is likewise equal to A.

Information on the structure of small powders and meso-powders of samples were obtained by low-temperature nitrogen adsorption method using the Nova 1200 device. The samples wer e clarified several times to obtain the temperature of 300°C and a residual pressure of not less than 0.001 mm. sim. Figure 8 shows nitrogen adsorption isotherms in samples of activated carbon when the micro powder is activated by slowly heating the apricot kernel, pre-carbonizing it and mixing it with carbon dioxide, then soaked in water vapor. In both cases, it was vacuum-sucked, and processing was carried out at the temperature of 850°C. In this case, of course, when heated slowly, the microparticle system will have a minimal amount of meso and micro-powder.

Examination of the structure of powdered materials by low-temperature nitrogen adsorption showed that samples obtained based on apricot peel has an improved micro powder structure. Activated carbon from walnut shells are in the form of meso powder, and it is characterized by hysteresis in the adsorption isotherm as shown in Figure 9.

The results of the Fure-IR spectroscopy study of samples based on the apricot peel, walnut peel, and walnut peel showed that winning symmetrical bands (2920 and 2851 cm^{-1}) were observed in them. These bands belong to the C-H-valence oscillation of the methyl and methylene groups. In the spectrum taken from a sample of apricot peel, shown in Figure 10, absorption
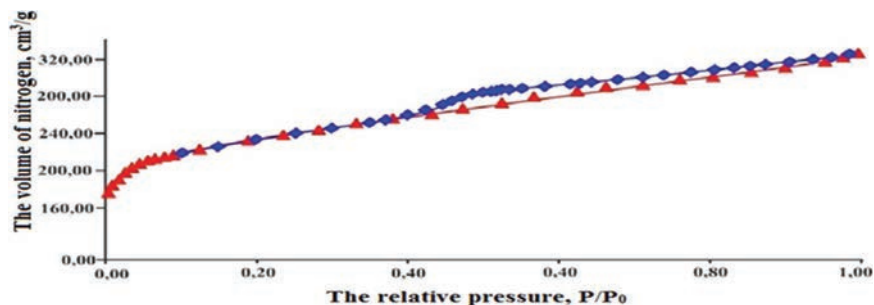
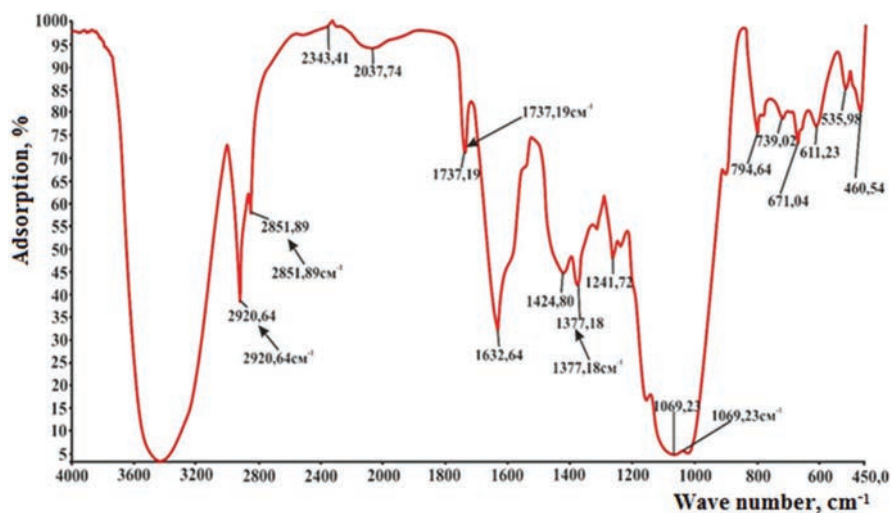**Figure 9** Nitrogen adsorption isotherms in samples of coal micro powder obtained from walnut shell.

Figure 10 IR-Fure spectrogram of apricot peel.

bands belonging to cellulose (1377 cm^{-1} and 1069 cm^{-1}) were observed (Figure 10).

IR spectra determine the absorption band of the three hydroxyl groups in each glucopyranose network. In the case of walnut shells, this absorption band is 1376 cm^{-1} and 1064 cm^{-1} was observed in the area. Xylan-specific 1737 cm^{-1} absorption band, which is the main component of hemicellulose was observed, and its intensity characterizes the C=O bond oscillation. A possible explanation for this fact is the process of carbonation reaction of condensed gas in appearance and solid pyrolysis of apricot peel leads to the formation of carbon-retaining microcrystals.
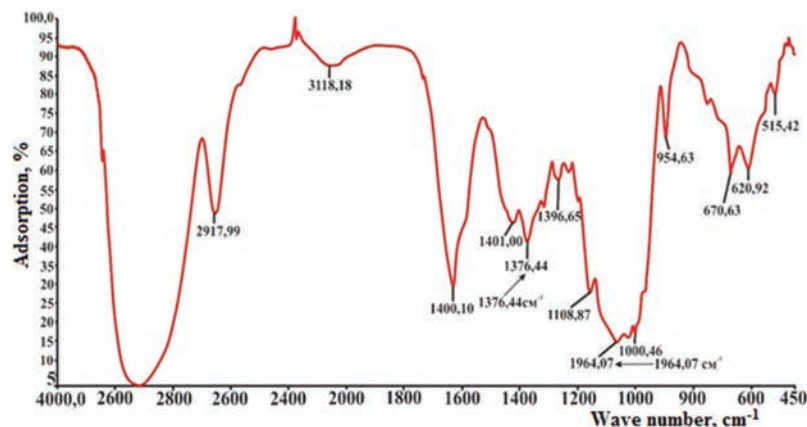
Figure 11   IR-Fure spectrogram of nuts.

As shown in above Figure 11, A thermally processed modification of the plant cliché shows a decrease in the proportion of xylan in it. This can be explained by a decrease in the intensity of the 1734 cm^{-1} absorption band. The half-width of IR spectra in the experiment and the activity of carbon nanotubes was evaluated according to the change in intensity. Carbon nanotubes have the same half-widths of IR spectra, Infrared spectroscopy is a useful method in chemistry because it makes detecting the presence of specific functional groups in a molecule simple. And the band intensities in the 1300–1600 cm^{-1} area vary. Since the change in intensity represents the vibration of the structure, a change in the IR spectrum in the 1300–1600 cm^{-1} field indicates a structural change. The properties of carbon nanotubes of different sizes were studied.

Figures 12 and 13 shows the electronic data of the fibrous structure of multilayer carbon pipes. In this type of UNT crystallization, the fiber thickness is 0.03–0.1 microns, the length is several times greater than the thickness and is ≈5 mkm. UNTs form aggregates of different sizes from 5 mkm to 2.5 mkm in the form of bundles. Aggregates are formed based on Van der Waals forces. Van der Waals forces are weak electric forces that attract neutral molecules in gases, liquefied and solidified gases, and practically all organic liquids and solids to one another. A net attractive force is created by the tendency of such persistent dipoles to align with one another. As can be seen from Figures 12 and 13, the multilayer nanotubes are homogeneous, other modifications were observed to consist of an amorphous carbon fullerene particle.
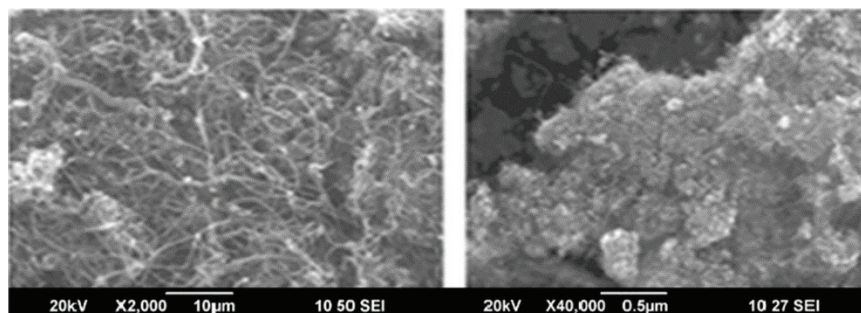
Figure 12   Microphotographs of the samples: 1-5000 times magnified: 2-1000 times magnified.
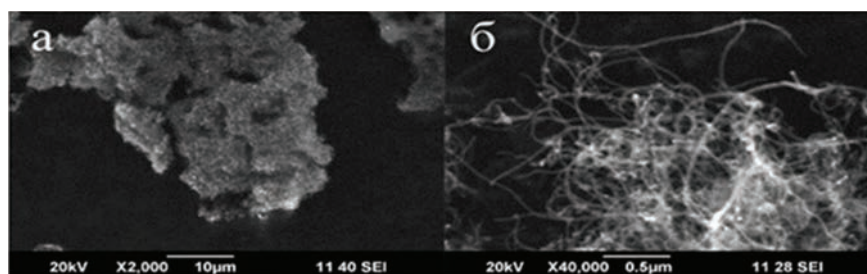

Figure 13   The CEM micro photograph of nanocarbon.

Synthesis of carriers in nanocarbon-based catalysts and after functionalization, the nanocarbon was condensed, their outer diameter remained unchanged and was 10–30 nm (Figure 13).

The compaction is explained by the formation of hydrogen bonds on the surface of the nanocarbon between the polar functional groups. The specific surface area of nanocarbon was measured by the BET method from physical adsorption isotherms. The Brunauer-Emmett-Teller (BET) theory tries to explain the physical adsorption of gas molecules on a solid surface and serves as the foundation for a critical analytical approach for calculating the specific surface area of materials. Statistical morphological characteristics of the porosity of the material were calculated: The specific surface area of BET is 157.2 m^2/g, the specific porosity is 0.36 cm^3/g, the average hole diameter is 2.23 nm. Nanocarbon samples synthesized to determine thermal properties were examined using thermogravimetric analysis (TGA/DTG) scanned in a nitrogen atmosphere. TGA is an analytical method that monitors the weight change that happens while a sample is heated at a constant pace to assess a
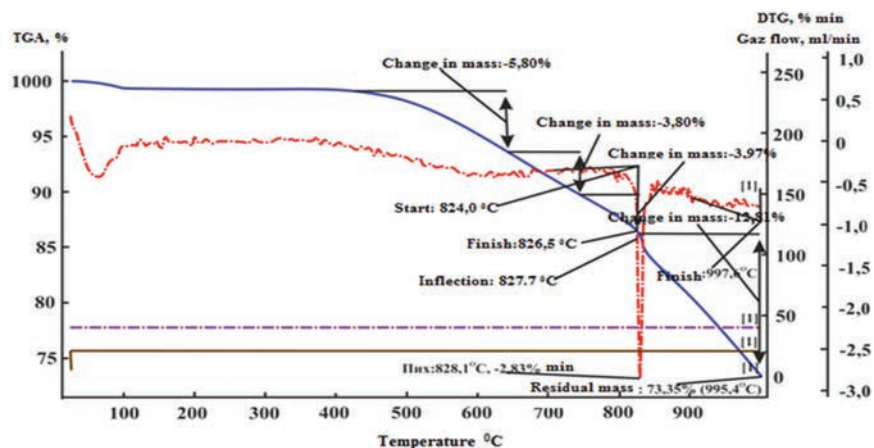
Figure 14 TG/DTG-analysis of nanocarbon in nitrogen medium. Structural properties of carbon nanostructures.

material's thermal stability and proportion of volatile components. Figure 14 shows a graph of the loss of nanocarbon mass obtained by the TGA method. As can be seen from the graph, at temperatures below 100°C and T = 250°C, mass loss is observed, which is responsible for the evaporation of moisture and the presence of water. At a temperature of 425°C, the destruction of the product begins and reaches a maximum mass reduction rate at a temperature of 825.5°C. In this case, the weight loss is 9.6%. In the range of 400–840°C, the rate of mass change (DTG curve) changes its value several times. This is evidence of ongoing structural changes in the carbon part, for example, more defective carbon formation, amorphous carbon, as well as highly structured formations, e.g., nanocarbon.

Since carbon nanotubes have several forms of carbon components, the decomposition process proceeds in 2 stages: in the first stage (up to 825°C), the more active phase (amorphous carbon) decomposes, in the second (above 840°C), decomposition of carbon nanotubes occurs. The residual mass at 998.4°C is 73.35%.

As shown in Above Figures 15 and 16, Adsorption processes for the purification and separation of gas mixtures include various masses forming the mixture, based on molecules in size and structure. The molecular masses of the macro components of air (nitrogen and oxygen) are close, 28 and 32 units, respectively, and the difference in the properties of these substances are not used during their adsorptive separation. However, it should be noted that
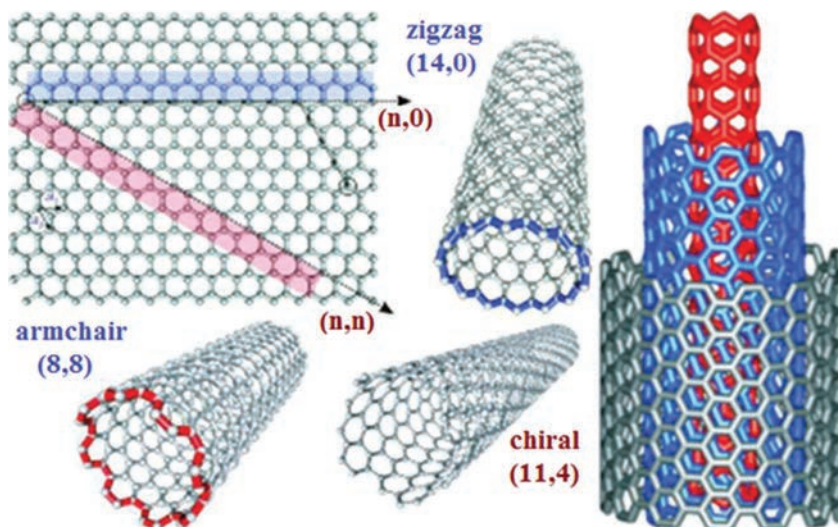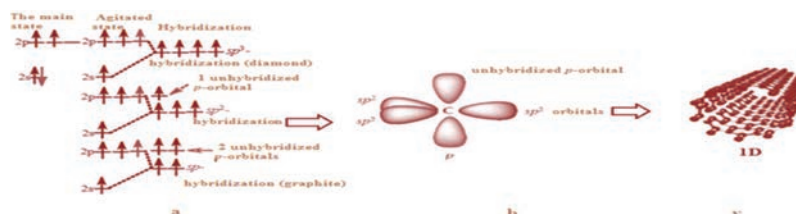
Figure 15 Structure of nanotubes.

Figure 16 Electronic structure of nanotubes.

it is this factor that ultimately determines the efficiency of low-temperature air separation.

Conclusion

The results of the Fure-IR spectroscopy study of samples based on the apricot peel, and walnut peel showed that winning symmetrical bands (2920 and 2851 cm^{-1}) were observed in them, these bands belong to the C-H-valence oscillation belonging to the methyl and methylene groups. In the spectrum taken from a sample of apricot peel, absorption bands belonging to cellulose (1377 cm^{-1} and 1069 cm^{-1}) were observed.

The thermal properties of fruit kernels (apricots) and walnut kernels were studied. Optimal modes of the carbonization process have been determined for each type of raw material.

A method for the synthesis of spherical granules by liquid granulation has been developed. The optimum concentration of the sulfuric acid solution used in granulation has been determined.

The analysis of the results on the dynamic sorption of petroleum products showed that maximum sorption is achieved when using a sorbent obtained at 400°C for 30 min.

Temperature regimes have been optimized for obtaining sorbent from walnut shells with different carbon content. The hydrophobicity and oleophilic of their surface are common to all these materials.

The dynamic conditions of the adsorption capacity processes under the sorbents were studied. Sorption efficiency and the specific capacity of sorbents were determined. It has been shown that the technological cycle of the studied sorbents can be repeated with repeated purification.

Colloid with activated charcoal is used to purify water from petroleum products through the process of collimation with finely dispersed mixtures in which deaeration is necessary.

Declarations

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Conflict of interest

There is no conflict of interest among the authors.

Data Availability

All data generated or analysed during this study are included in the manuscript.

Code Availability

Not applicable.

Author's Contributions

Kholmirzaeva H.N, Methodology, Project Administration, Manuscript editing; Fayzullaev N.I, Software, Validation; Normurodov O.O, Visualization, Manuscript Review and editing; Haydarov G.Sh, Design Framework, Resources, Validation.

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