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ILMIY AXBOROTNOMA

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SEPARATION OF ORGANIC DYES FROM SOLUTIONS USING MODIFIED ACTIVATED CARBON

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Abstract. Activated carbon samples were obtained from walnut shells by pyrolysis. Activated carbon samples were modified with polyvinyl alcohol to increase the sorption capacity and improve the textural characteristics. After modification, the sorbent samples had a specific surface area (S_{BET}) of 980 m^2/g, an average pore diameter (d,nm) of 1.8÷2.2 nm, a pore volume (Vs, cm^3/g) of 0.85÷0.96 cm^3/g, and an increase in the adsorption capacity for benzene vapors to 3.2 mmol/g. Pseudo-second-order kinetic adsorption of methylene blue dye onto modified carbon was studied. From the results obtained, it was determined that the equilibrium adsorption amount (qe) of the adsorbed dye was 0.32 mg/g, the rate constant was 0.021 g/mg min, and R^2 = 0.882.

Keywords. pyrolysis, activated carbon, adsorption capacity, BET isotherm model, SEM analysis, X-ray microanalysis.

Modifikatsiyalangan faollashgan ko‘mir yordamida eritma tarkibidagi organik bo‘yoqlarni ajratish

Annotatsiya. Piroliz yo‘li bilan yong‘oq mevasi qobig‘idan faollangan ko‘mir namunalari olindi. Faollangan ko‘mir namunalarini sorbsiya sig‘imini oshirish va tekstur xarakteristikalarini yaxshilash uchun polivinilspirti bilan modifikatsiyalandi. Modifikatsiyalangandan so’ng sorbent namunalarida solishtirma sirt yuza (S_{BET}) 980 m^2/g, g‘ovaklar o‘rtacha diametri (d,nm) 1,8÷2,2 nm, g‘ovaklar hajmi (Vs, cm^3/g) 0,85÷0,96 cm^3/g, benzol bug’lari bo‘yicha adsorbsiya sig‘imi esa 3,2 mmol/g gacha ortishi aniqlandi. Modifikatsiyalangan uglerodga metilen ko‘ki bo‘yog’i yutilishining psivdo ikkinchi tartibli kinetik adsorbsiyasi o’rganildi Olingan natijalardan yutulgan bo‘yoqning muvozanatdagi adsorbsiya miqdori (q_e) 0,32 mg/g, tezlik doimiysi 0,021 g/mg·min va R^2 =0,882 ekanligi aniqlandi.

Kalit so’z: piroliz, faollangan ko‘mir, adsorbsiya sig‘imi, BET izoterma modeli, SEM tahlili, rentgenmikrotahlil.

Разделение органических красителей из растворов с использованием модифицированного активированного угля

Аннотация. Образцы активированного угля были получены из скорлупы грецкого ореха методом пиролиза. Образцы активированного угля были модифицированы поливиниловым спиртом для увеличения сорбционной емкости и улучшения текстурных характеристик. После модификации образцы сорбента имели удельную площадь поверхности (S_{BET}) 980 $м^2/г$, средний диаметр пор (d, нм) 1,8÷2,2 нм, объем пор (Vs, $см^3/г$) 0,85÷0,96 $см^3/г$ и увеличение адсорбционной емкости по парам бензола до 3,2 ммоль/г. Изучена псевдовторая кинетическая адсорбция красителя метиленового синего на модифицированном угле. Из полученных результатов

определено, что равновесное количество адсорбции (qe) адсорбированного красителя составило 0,32 мг/г, константа скорости 0,021 г/мг мин, а R^2 = 0,882

Ключевые слова: пиролиз, активированный уголь, адсорбционная способность, модель изотермы БЭТ, анализ СЭМ, рентгеновский микроанализ.

Introduction. In recent years, the accelerated growth of the chemical industry and manufacturing sectors has resulted in the unsystematic exploitation of fossil natural resources. This, in turn, has led to the emergence of economic and many environmental problems. Therefore, the processing of local and secondary raw materials, increasing the range of products obtained from them is one of the urgent problems of our time. In this context, the synthesis of activated mesoporous carbon with enhanced structural properties through pyrolysis of various fruit husks considered as secondary raw materials offers a cost-effective and sustainable approach.

Mesoporous carbon, which has high sorption properties, is used for various purposes depending on its mechanical and thermal strength, hydrophobicity, and pore size. Notably, this type of activated carbon finds broad application across various fields: in medicine for the removal of toxic liquids, in genetic engineering for the isolation of specific genes and enzymes, in electronics for the development of high-capacity energy storage devices such as batteries and supercapacitors, in liquid chromatography as an efficient adsorbent, in wastewater treatment for the separation of hydrophobic compounds, and in environmental protection for the purification of toxic volatile organic compounds from ambient air.

The reason for the widespread use of mesoporous carbon is its high specific surface area and pores in the nanometer range. At the same time, modifying the surface of mesoporous carbon with various polar and non-polar substances improves their physical and chemical properties and further increases their potential applications. Therefore, in recent years, scientific research conducted in various research centers around the world has been aimed at expanding the possibilities of using activated carbon in catalysis, adsorption, energy storage and environmental restoration by modifying its surface. In this work, mesoporous carbon was obtained by pyrolysis from walnut shell, which is a secondary raw material, and by modifying its surface with various polar group substances, an adsorbent with improved textural characteristics was obtained.

Active carbon obtainment and surface modification. Charcoal was obtained from walnut shell by pyrolysis at 550°C. The obtained charcoal samples were modified with polyvinyl alcohol by post-synthesis method. The mesoporous activated carbon was dried at 105°C for 12 hours to remove moisture and was tableted. A 5% by weight polyvinyl alcohol (PVS) solution was prepared by dissolving the PVS in deionized water at 80°C by stirring. The solution was stirred for 1 hour until completely homogeneous and transparent. The mixture was left in solution for 3 hours to ensure sufficient adsorption of PVS onto the surface of the carbon tablets. To enhance the bonding, the mixture was then heated at 100°C for 2 hours to initiate partial cross-linking. The solid product was filtered and washed several times with distilled water to remove excess PVS. The sample was then dried in an oven at 60 °C for 24 hours. The PVS-modified mesoporous carbon shows improved hydrophilicity and functional group presence.

Results and discussion. The surface morphology and pore structure of the synthesized sorbent were analyzed using a SEM EVO MA 10 (Carl Zeiss) scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (EDS Aztec Energy Advanced X-Act, Oxford Instruments). The accelerating voltage was increased to 1 kV, allowing magnification up to ×200,000. Both secondary electron (SE2) imaging and backscattered electron (ESB) analysis were performed to investigate the structural features. (Figure 1). The composition of the sorbents was also studied by EDS X-ray microanalysis. According to the results, the carbon content in the adsorbents obtained from almond shells is 74.9% before carbonization, and 92.3% after carbonization and modification (Figure 2).

The SEM image shows that spherical and aggregated carbon particles with relatively smooth surfaces are formed after PVA modification. The visible globular morphology indicates that PVA has formed a uniform coating layer on the carbon structure. The surface porosity is slightly reduced, indicating that the pores are partially covered by polyvinyl alcohol.

No large cracks or delaminations are observed, suggesting a stable bond between PVS and the carbon surface. The presence of fine surface textures and interconnected structures confirms the preserved porosity. This morphology indicates improved hydrophilicity and functionality due to the -OH groups from PVA. The uniform particle size distribution indicates effective dispersion during modification. The process probably occurs in an aqueous medium at ~80 °C, providing strong hydrogen bonding interactions.

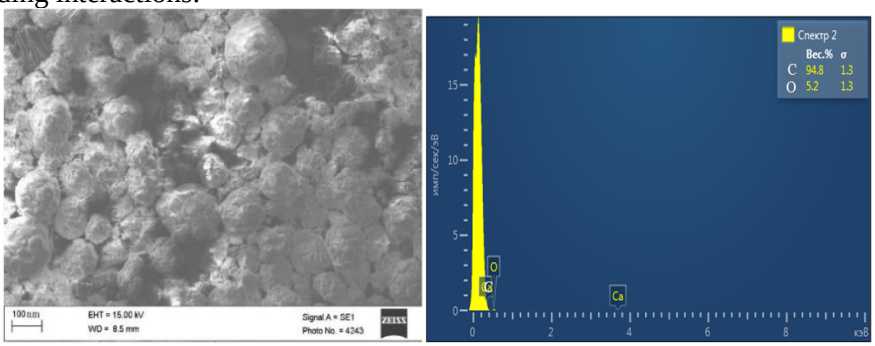

Figure 1. SEM and EDS image of PVS-modified activated carbon

The sorption and textural properties of the activated carbon sample were studied using a sensitive quartz spiral structure of Mak-ben-Bakra using benzene vapor adsorption. Prior to use as an adsorbate, benzene was purified under vacuum conditions and frozen until its vapor pressure equaled that of pure benzene, ensuring the removal of dissolved gases. Before initiating the adsorption measurements, each adsorbent system was degassed to a residual pressure of 1.33×10^{-3} Pa and thermally treated at 323 K for 6 hours. Following this preparation, benzene vapor adsorption isotherms were recorded (see Figure 2).

The adsorption/desorption isotherms of the modified carbon clearly show a sharp increase in benzene vapor uptake up to a relative pressure of $p/p_0 = 0.6$, followed by a tendency toward saturation as p/p_0 approaches 0.9. The overlap of the adsorption and desorption branches in the pressure range $p/p_0 = 0.5–0.8$, caused by capillary condensation of benzene vapor, results in the formation of a characteristic hysteresis loop.This indicates a high proportion of mesoporous pores in them.

Based on the sorption isotherms and the BET equation, the specific surface area (SBET), pore volume (Vs) and average diameter (D), monolayer capacity (α∞) and saturation adsorption (as) of the sorbents were calculated (Table 1).

Table 1

Textural characteristics of mesoporous carbon and modified carbon

Sorbent	a_∞, mol/kg	S_{BET} m^2/g	a_s, mol/kg
Mesoporous carbon	0,50 ± 0,1	700 ± 30	1,08 ± 0,5
modified carbon	0,84 ± 0,01	980 ± 20	3,2 ± 0,1

It can be seen from the table that the specific surface area of mesoporous carbon modified with polyvinyl alcohol increases.

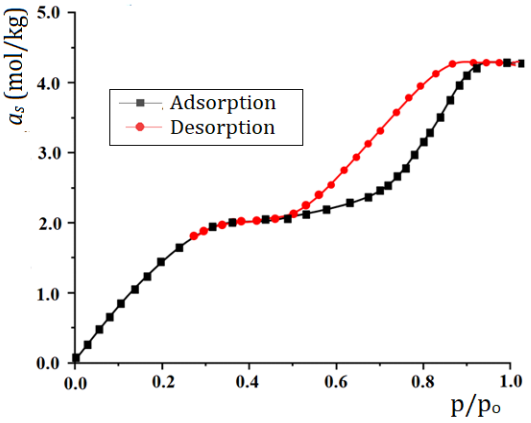

Figure 2. Benzene vapor adsorption isotherms

In addition, the micropore volume (W_0), mesopore volume ($W_{me}z$), and saturation adsorption volume (V) were calculated from the sorption isotherm data obtained through benzene vapor adsorption on the activated carbon sample at varying relative pressures (see Table 2).

Table 2

The pore size distribution of mesoporous and surface-modified carbon materials was determined through benzene vapor adsorption analysis

Sorbent	W0 ·10^3, m^3/kg	W_{me}·10^3, m^3/kg	V_s·10^3, m^3/kg
meoporous carbon	0,14 ±0,02	0,6 ± 0,01	0,205 ± 0,103
modified carbon	0,32 ± 0,01	0,86 ± 0,05	0,28 ± 0,01

The separation of dyes from wastewater using a sorbent was studied.

The adsorption capacity of dyes on activated carbon was calculated as follows.

$$a_s = \frac{C_0 - C_e}{m} \cdot V (1)$$

In the equation, C_0 is the initial concentration of dyes (mg/l), C_e is the equilibrium concentration of the dye (mg/l), m is the mass of activated carbon (gr), and V is the volume of the dye solution (l).

The efficiency of dye removal from aqueous solutions was evaluated using the corresponding calculation formula.

(2):

$$R = \frac{C_0 - C_e}{C_0} \cdot 100\% (2)$$

The R values were determined based on the adsorption of dye solutions with both the lowest and highest initial concentrations onto the activated carbon.

The adsorption isotherm shows the mechanism of interaction between adsorbate molecules and the adsorbent. The adsorption equilibrium of different dyes on activated carbon was assessed using the linear forms of the Langmuir and Freundlich isotherm models.

$$\frac{C_e}{a_m} = \frac{1}{K \cdot a_{max}} + \frac{C_e}{a_{max}} (3)$$

In Equation (3), a_m represents the monolayer adsorption capacity of the adsorbent, K denotes the Langmuir equilibrium constant (L/mg), and a_{max} refers to the maximum adsorption capacity of the adsorbent expressed in mg/g. The logarithmic expression of the Freundlich isotherm model is given as follows:

$$loga_s = logK_f + \frac{1}{n} \cdot logC_e (4)$$

In equation (4), K_f is the Freundlich equilibrium constant, 1/n is the adsorption intensity.

Activated carbon was used to separate the organic dye methylene blue from wastewater. The dye adsorption experiments were conducted by adding 25 mg of the synthesized activated carbon into 100 ml Erlenmeyer flasks, each containing 100 ml of dye solution with initial concentrations ranging from 5 to 100 mg/L. The mixtures were stirred continuously for 24 hours at a controlled temperature of 22 ± 2°C to ensure adsorption equilibrium. Upon reaching equilibrium, the solid adsorbent was separated from the liquid phase via centrifugation at 6000 rpm for 3 minutes. The remaining dye concentration in the supernatant was then determined using a UV-Vis spectrophotometer.Two parallel series of measurements were performed and the results presented are the arithmetic mean of those obtained in each series.

The values of physical quantities obtained from the adsorption of dyes are presented in Table3.

Table 3

Physical quantities obtained from the adsorption of methylene blue on activated carbon

Paints	C_0 (mg/l)	Lengmyur				Freyndlix			
		a_m	a_{max}	K	R^2	a_m	a_{max}	K	R^2
Methylene blue	10÷100	12.1	50,2	0,073	96,4	12,4	45,9	0,078	98,4

According to the data presented in the table, activated carbon demonstrated the highest adsorption capacity for methylene blue compared to the other tested dyes. The sorption isotherms describing the dye adsorption behavior on the activated carbon were consistent with both the

Langmuir and Freundlich models, with the best fit and most pronounced parameters observed specifically for methylene blue.The isotherms obtained from the adsorption of dyes are presented in Figure 2.

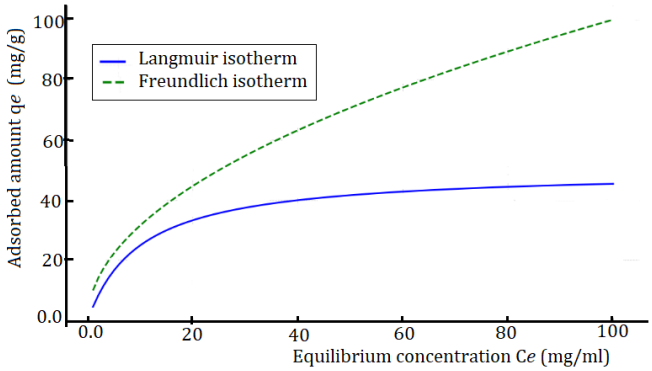

Figure 2. Isotherms obtained from the adsorption of dyes on activated carbon

Also, when the phase composition of the sorbent was studied on a Pananalytical XRD X-ray diffractometer, it was found that its composition consists of amorphous phases (Figure 2). From the figure, mesoporous carbon usually shows amorphous properties in XRD. The broad peaks in the 2θ 23° and 43° regions of the diffractogram are considered to be phases belonging to mesoporous carbon. The high-intensity peaks at 30° of 2θ are considered to be phases belonging to polyvinyl alcohol. The low-intensity peaks throughout the entire diffraction pattern indicate that the sorbent formed from mesoporous carbon and polyvinyl alcohol is amorphous in nature.

The mechanism of adsorption and the factors determining the adsorption rate were evaluated using experimental data interpreted through the pseudo-second-order kinetic model. This model suggests that the adsorption process is likely governed by chemisorption, which involves valence interactions—specifically, the sharing or transfer of electrons between the adsorbent and the adsorbate.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} (5)$$

k_2- rate constant (1/min), q_t- adsorption capacity at time t (mg/g).

The values obtained based on the experimental results and calculations are presented in Table 4:

Table 4

Kinetic quantities of methylene blue adsorption on the sorbent over time

Vaqt (min)	q_t (mg/g)	$\frac{t}{q_t}$ (min·)
5	12,4	0,403
10	18,9	0,529
20	25,6	0,781
30	28,5	1,053
45	30,1	1,495

From the table, it can be seen that the adsorption of methylene blue dye on modified carbon for a pseudo-second-order reaction is chemisorption on polar groups, and its adsorption increases over time.

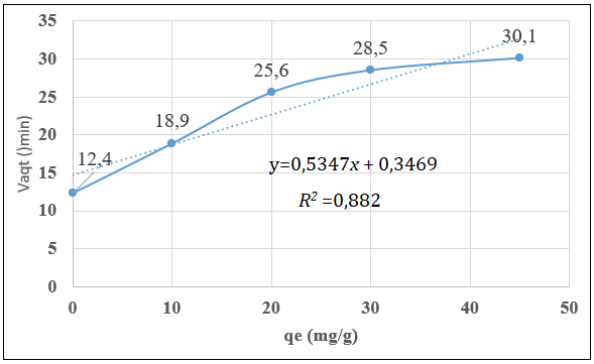

Figure 3. Kinetic curve of methylene blue adsorption on modified coal

From the obtained results, it was found that the equilibrium adsorption amount (qe) was 0.32 mg/g, the rate constant was 0.021 g/mg·min, and R2 =0.882.

Conclusion. After modification with polyvinyl alcohol, the specific surface area (SBET) of the mesoporous sorbent increased to 980 m2/g, the average pore diameter (d,nm) to 1.8÷2.2 nm, the pore volume (Vs, cm3/g) to 0.85÷0.96 cm3/g, and the adsorption capacity for benzene vapor increased to 3.2 mmol/g. The pseudo-second-order kinetic adsorption of methylene blue dye on modified carbon was studied. From the results obtained, it was found that the equilibrium adsorption amount (qe) of the absorbed dye was 0.32 mg/g, the rate constant was 0.021 g/mg min, and R2 =0.882.

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