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Structure, morphology and conductive properties of thermochemically activated carbon material from walnut shells

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The method of thermochemical activation with orthophosphoric acid was used to obtain porous carbon material from walnut shells. The effect of the activation temperature on the structural, morphological and conductive properties of the carbon material was studied. The structural and morphological characteristics of the carbon samples were characterized using low-temperature adsorption porometry, SEM and Raman spectroscopy. It was shown that thermochemical activation with orthophosphoric acid allows obtaining porous carbon materials with a specific surface area of 734 - 1385 m^2/g, depending on the activation temperature. The conductive properties of the obtained porous carbon materials were studied using the impedance spectroscopy method.

Keywords: porous carbon material, thermochemical activation, specific surface area, fractal dimension, impedance spectroscopy, specific electrical conductivity.

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Introduction

In the modern context, the development of environmentally friendly materials from renewable raw materials for electrodes of electrochemical power sources, in particular, supercapacitors, is becoming increasingly important. Materials based on porous carbon material (PCM) obtained from biomass of plant origin are a promising solution due to their low cost, availability of raw materials and high porosity and specific surface area. The key advantages of such carbon materials are: 1) the possibility of using agricultural and food waste as raw materials, which contributes to reducing the environmental load and reducing the cost; 2) the high specific surface area of PCM and developed porosity, which provide a large electrochemically active volume for adsorption/desorption of electrolyte ions or for the formation of an electric double layer; 3) the potential for surface modifications (functional groups, doping, etc.) to improve electrical conductivity, electrode interaction and cyclic stability [1, 2].

One of the most effective approaches to obtaining PCM from biomass is chemical activation. Chemical activation has a number of advantages compared to physical activation, which include a single-step and short-term activation process, low activation temperatures, higher mass yield of material, and the ability to control the porous structure of the obtained PCM. H_3PO_4, $ZnCl_2$, NaOH, or KOH are most often used as a precursor for chemical activation. These substances act as dehydrating agents that prevent the formation of resin and reduce the formation of other volatile products. In particular, activation of biomass with orthophosphoric acid promotes the formation of interchain phosphate bonds between the polymer structures of cellulose, which leads to the "loosening" of the structure, expansion of the intermicrofibrillar space, and the creation of meso- and micropores. The disadvantage of the chemical activation process is the need for a long stage of washing the obtained material to completely remove the activation agent from the obtained PCM [3-5].

In the context of electrode materials, the activation method is of great importance: well-developed porosity and a large specific surface area contribute to effective electrolyte contact with activated carbon, reduce diffusion limitations, improve electronic conductivity and increase the number of available electrochemically active sites [6, 7]. Combined with the availability of food or agro-industrial waste as a raw material, this approach has the potential to become a commercially viable option for the fabrication of high-performance electrodes. However, despite all the advantages, challenges remain in matching the properties of the biomass and the final electrochemical characteristics of the material, stability of cyclic operation, compatibility with different electrolytes and integration into commercial systems [1, 8, 9].

Thus, the aim of this work is to study the obtained activated carbon material from plant raw materials (walnut shells) by the method of thermochemical activation using orthophosphoric acid, and to evaluate it as an electrode material for electrochemical power sources with an emphasis on the structural and morphological characteristics of the PCM and its conductive properties.

I. Materials and methods of research

To obtain chemically activated PCM, walnut shells were mixed with orthophosphoric acid. The ratio of the mass of the activator to the mass of the raw material m(H_3PO_4)/m(biomass) was 1.5:1. The resulting mixture was thoroughly mixed for 1-2 hours and left for 48 hours. Then the mixture was dried at 100°C for 24 hours until a constant mass was reached. The mixture was divided into parts of the same mass with an accuracy of 10^{-3} g for further carbonization. Carbonization was carried out in a muffle furnace at temperatures of $400 \div 900$ °C with a step of 100°C. The heating rate of the furnace was 10 °C/min. After cooling the obtained carbon material to room temperature, it was washed with distilled water to a neutral pH and dried at 80°C until a constant mass was reached. The obtained PCM samples are labeled by activation temperature (Table 1). The carbon materials were ground in a ceramic mortar and their bulk density d_0 was measured.

Table 1.

Marking and bulk density of chemically activated PCM

PCM	NP1	NP2	NP3	NP4	NP5	NP6
Activation temperature, °C	400	500	600	700	800	900
d_0, g/cm^3	0.33	0.34	0.34	0.47	0.55	0.60

The structural adsorption characteristics of the PCM were determined by the method of low-temperature N_2 adsorption on the Autosorb Nova 2200e analyzer (Quantachrome Instruments, USA), and the parameters of the porous structure were calculated using the NOVA 2200e software. The specific surface area (S_{BET}) was determined by the Brunauer-Emmett-Teller multipoint method [10]. The total pore volume (V_Σ) was calculated from the volume of adsorbed N_2 at $P/P_0 \approx 1$. The Halsey t-method was used to determine the micropore volume (V_{mic}) [11]. The size distribution of mesopores was studied using the Barrett-Joyner-Halend method, and the density functional theory (NLDFT method) was used to determine the micropore distribution [12]. The fractal dimension (D) was calculated using the Frenkel-Halsey-Hill method [13, 14].

To study the surface morphology of the obtained carbon samples, a scanning electron microscope from ZEISS (Germany) was used.

Raman spectra were obtained using a Horiba Jobin-Yvon T-64000 triple spectrometer equipped with an Olympus BX41 confocal microscope.

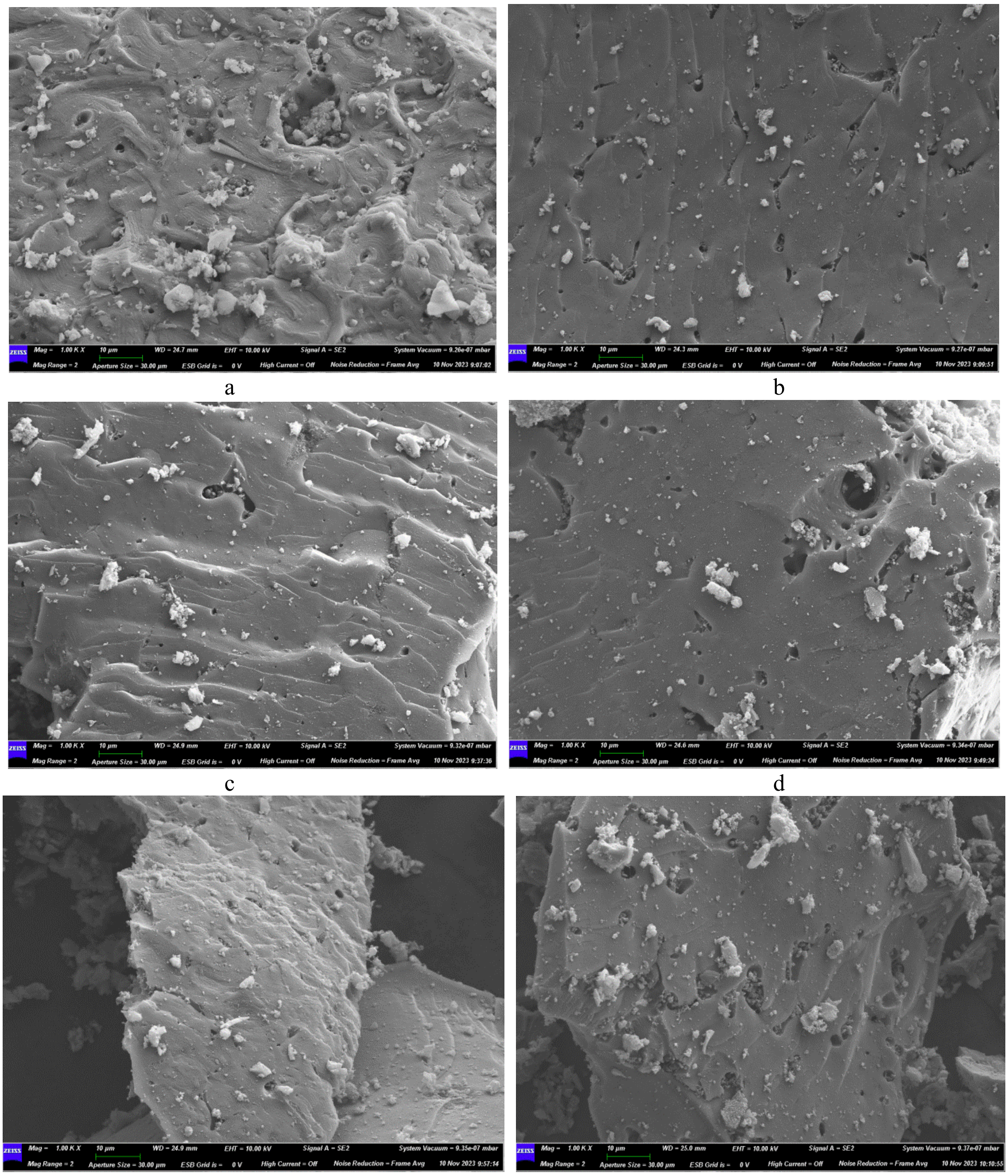
Impedance spectra in the frequency range $f = 10^{-2}–10^5$ Hz were obtained using the AUTOLAB PGSTAT complex (ECO CHEMIE, the Netherlands). The method described in [15] was used to calculate the specific electrical conductivity values.

II. Results and discussion

Fig. 1 shows SEM images of NP samples. The figure shows that the surface of chemically activated PCM is quite rough. White inclusions are visible on the carbon surface, which, in the vast majority, are residues of burnt ash. Carbon particles have surface microcracks and round (oval) pores, the average diameter of which is less than 10 μm.

It can be assumed from the obtained data that activated PCM are characterized by a developed porous structure not only on the surface, but also in the volume of the carbon material. The characteristics of the porous structure of PCM activated with orthophosphoric acid were determined by measuring nitrogen sorption. The N_2 adsorption/desorption isotherms for NP samples (Fig. 2) have a form corresponding to type II according to the IUPAC classification system and are typical for porous materials [16]. A sharp increase in the amount of nitrogen adsorbed at low relative pressure indicates the presence of micropores in the material. The progressive increase in pore volume with increasing relative pressure indicates the presence of mesopores and the multilayer adsorption process. The formation of a hysteresis loop during desorption indicates a type IV isotherm. This type of isotherm is a general characteristic of porous carbon materials with large pores.

The structural and adsorption characteristics of the activated PCMs are given in Table 2. Nitrogen adsorption/desorption isotherms were recorded in the range of relative pressures $P/P_0 = 0.0 \div 1.0$ at a temperature of -196 °C. The specific surface area S_{BET} was calculated in the range of relative pressures P/P_0 from 0.05 to 0.35. The total pore volume V_Σ and the average pore diameter d_{pore} were calculated by the BJH method. According to the data in Table 2, the specific surface area and total pore volume reached the highest value for the sample activated at 400°C, sample NP1: $S_{BET} = 1385$ m^2/g, $V_\Sigma = 1.113$ cm^3/g; and the lowest value for the sample NP5 activated at 900°C: $S_{BET} = 892$ m^2/g, $V_\Sigma = 0.62$ cm^3/g. It should be noted that the specific surface area and total pore volume change randomly with an increase in the activation temperature from 400 to 900°C, i.e. up to 600°C the corresponding values decrease, at 700°C they increase and then decrease again with an increase in the activation temperature. This can be



a

b

c

d

e

f

Fig. 1. SEM-images of PCM samples: a – NP1, b – NP2, c – NP3, d – NP4, e – NP5, f – NP6.

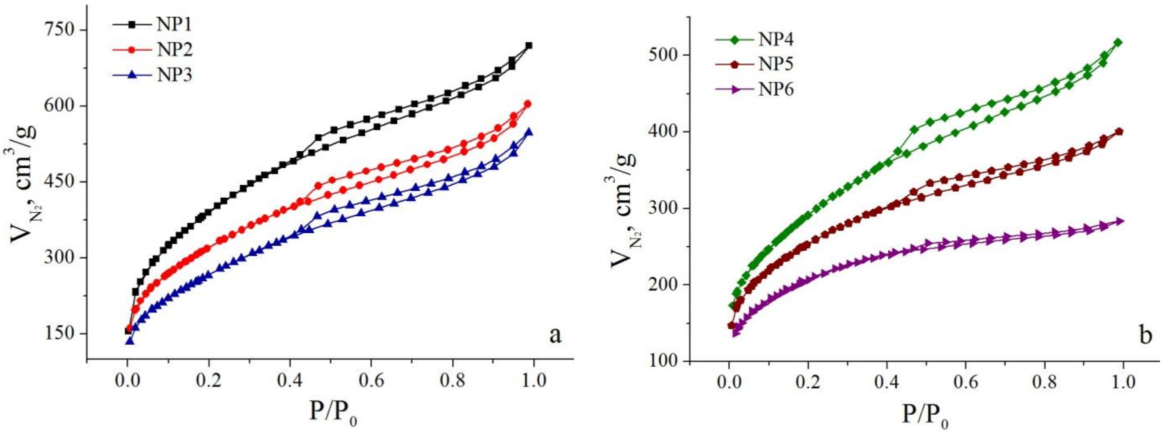


Fig. 2. N_2 adsorption/desorption isotherms for PCM samples.

explained by the fact that the thermal decomposition of cellulose in the walnut shell at an activation temperature of 400 ÷ 600 °C creates small and partially closed primary pores inside the structure of the walnut fiber. But at a carbonization temperature of 700 ÷ 900°C the thermal decomposition occurs more intensively and creates a more open primary porous structure of the PCM. Since the BJH method is applicable only to determine the size of pores distributed in the regions of meso- and macropores, the calculated average pore size (Table 2) does not change much for all samples and fluctuates within the range of 2.4 - 3.6 nm.

The BJH pore size distribution (Fig. 3(a)) confirms the presence of meso- and macropores in the range from 2 to ~100 nm for all NP samples. However, the largest pore volume is in the 3.5–3.6 nm pores, as evidenced by the peak in Fig. 3(a). Since classical macroscopic theories, such as the BJH method, do not provide a realistic description of the filling of micropores and narrow mesopores, this leads to an underestimation of the pore sizes. For a more detailed description of the micro-mesoporous structure of NP samples in the 0.35-40 nm pore size range, the NLDFT method (slit-like pore model) was used. The pore size distribution in the form of histograms (Fig. 3 (b-d)) for samples NP1 – NP5 demonstrates the presence of one large and two smaller "peaks" for pores of 1.2, ~2.6 and ~3.6 nm, respectively. An increase in the activation temperature from 400 to 600°C is accompanied by a decrease in the volume of the corresponding pores (Fig. 3 (a, c – NP3)). For sample NP4, obtained at an activation temperature of 700°C, a sharp increase in the volume of pores of 1.2 nm is observed, but the number of pores of ~2.6 and ~3.6 nm sizes practically does not change (Fig. 3(c)). However, the porous structure of samples NP1 – NP4 is similar. Carbon materials NP5 and NP6 differ from the previous PCMs. In particular, in NP6 samples activated at 900°C, a significant volume is occupied by pores with an average diameter of ~0.5–1 nm (Fig. 3(d)), which are absent in other samples of this series.

To quantitatively describe the surface roughness of the PCM, the fractal dimension D was determined using

Table 2.

Parameters of the porous structure of chemically activated PCM

PCM	S_{BET}, m^2/g	V_Σ, cm^3/g	V_{mic}, cm^3/г	V_{mic}/V_Σ, %	d_{pore}, nm
NP1	1385	1.113	0.292	26	3.2
NP2	1135	0.934	0.248	27	3.3
NP3	952	0.847	0.104	12	3.6
NP4	1015	0.800	0.179	22	3.2
NP5	892	0.620	0.212	34	2.8
NP6	734	0.439	0.177	40	2.4

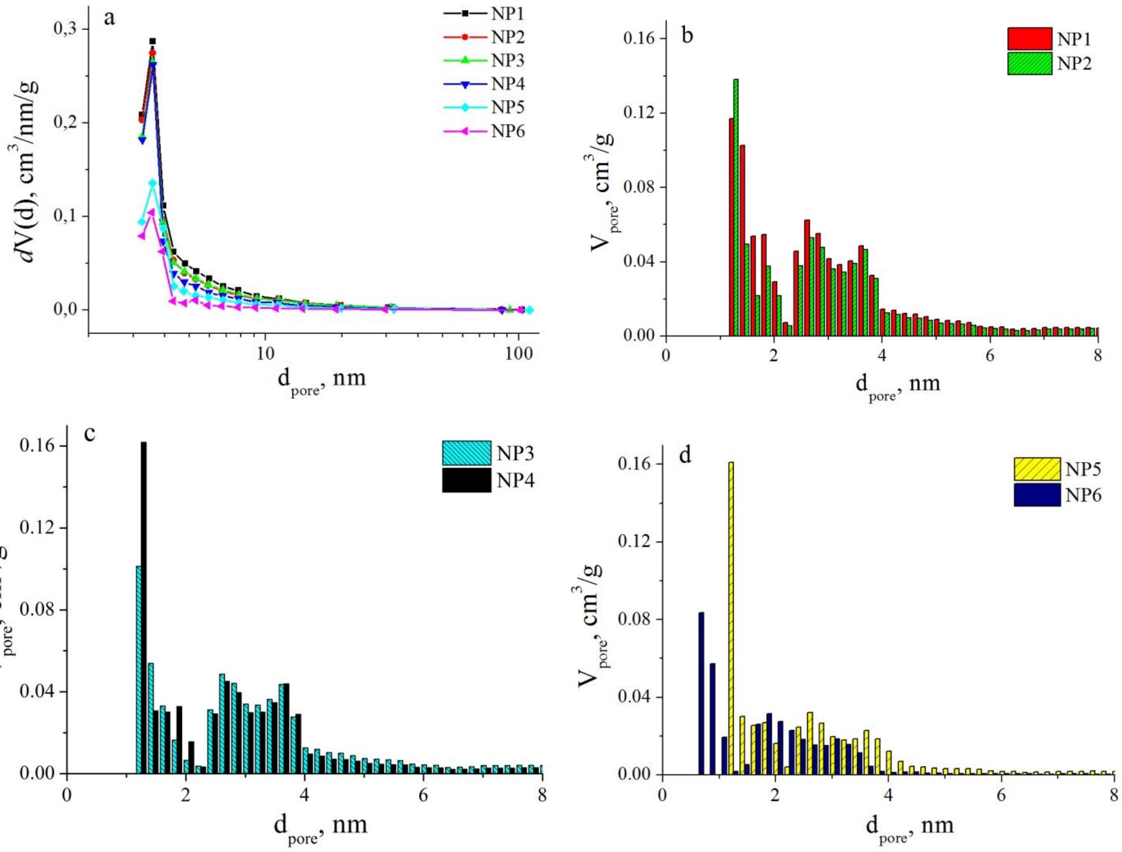


Fig. 3. Pore size distribution of chemically activated PCM calculated by the BJH method (a) and the NLDFT method (b-d).

the Frenkel-Halsey-Hill method, according to which the value of D can be calculated using data from low-temperature N_2 adsorption, according to the equation:

$$\ln V = \ln K + \frac{A}{3-D}\left[\ln\left(\ln\left(\frac{P_0}{P}\right)\right)\right],$$

where V is the volume of nitrogen adsorbed by the PCM at equilibrium pressure; P_0 is the pressure of saturated nitrogen vapor; A is the power exponent, which depends on the fractal dimensions and the adsorption mechanism; K is an empirical constant that depends on the nature of the adsorbent and the measurement conditions ($K = e^b$, where b is the intercept); D is the fractal dimension [17, 18].

One can distinguish linear sections in the region of low and high relative pressures P/P_0 at the graphs of the dependence of the logarithm of the adsorbed amount of nitrogen V_{ads} on the logarithm $[\lg(P_0/P)]$ (Fig. 4). These sections correspond to the fractal dimension of the microporous (D_{F1}) and mesoporous (D_{F2}) structures of chemically activated carbon material (Table 3).

As can be seen from the Table 3 the activation temperature has little effect on the change in the values of fractal dimension for NP samples. The value of D_{F1} increases slightly with increasing activation temperature from 2.42 for NP1 to 2.57 for NP6 samples, i.e. the surface of the micropores of the PCM becomes rougher, and their internal structure becomes more complex. The value of D_{F2} for the PCM samples ≥ 2.9, which indicates a complex structure of mesopores. Therefore, chemical activation of walnut shells with orthophosphoric acid allows obtaining PCM with a complex and developed micro- and mesoporous structure, regardless of the activation temperature.

The degree of graphitization of activated PCMs is estimated according to the results of Raman scattering spectroscopy. For all NP samples, two characteristic diffraction peaks are present in the Raman spectra (Fig. 5), located at 1590 cm^{-1} (G-band) and 1340 cm^{-1} (D-band). The maximum intensity of these peaks changes with increasing temperature of chemical activation of biomass.

Typically, the intensity ratio (I_D/I_G) between the D- and G-bands is used to estimate the degree of graphitization of PCM. The integral intensities of I_D and I_G were determined based on two Lorentzians. The correlation coefficient at the approximation ≥ 0.98. A lower value of I_D/I_G indicates a higher degree of graphitization, since the G-band is associated with the vibration of sp^2 hybrid carbon atoms in the graphite layer, and the D-band is associated with the existence of a disordered carbon structure [9, 19]. When the activation temperature increases from 500 to 900°C, the I_D/I_G values for PCM samples change from 2.3 to 1.8, indicating a constant increase in the degree of graphitization with increasing temperature in this range (Fig. 6).

The size of graphite fragments L (nm) along the basal plane (002) of graphite was determined using the equation

$$L(нм) = (2{,}4 \cdot 10^{-10})\lambda^4 \frac{I_G}{I_D},$$

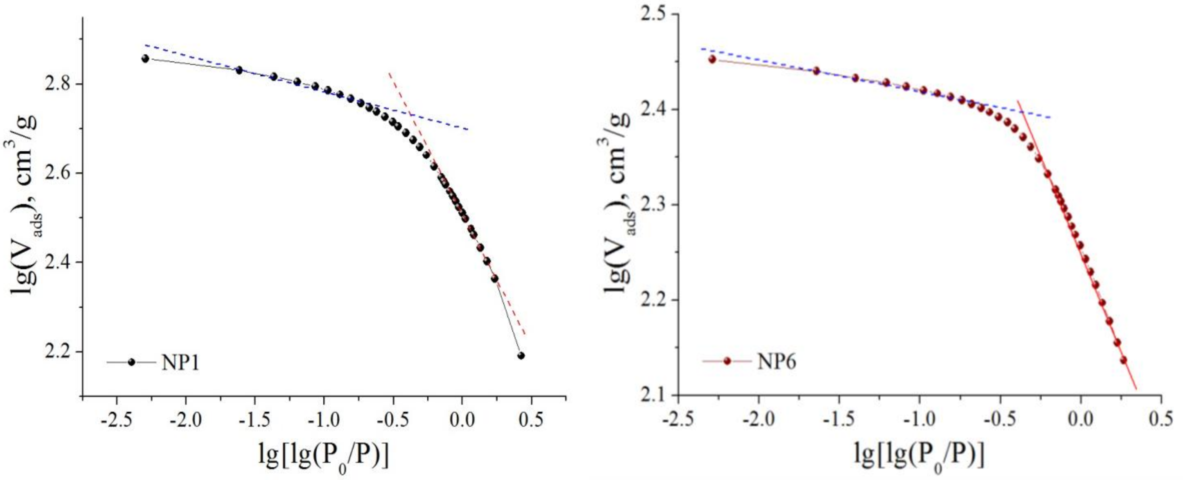


Fig. 4. Typical graphical dependences of $\lg(V_{ads})$ on $\lg[\lg(P_0/P)]$ for determining fractal dimension using the Frenkel-Halsey-Hill method.

Table 3.

Fractal dimension for PCM

PCM	Low-pressure region ($P/P_0 \leq 0.4$)		High-pressure region ($P/P_0 > 0.4$)	
	D_{F1}	Correlation coefficient	D_{F2}	Correlation coefficient
NP1	2.42	0.99	2.91	0.95
NP2	2.48	0.99	2.90	0.97
NP3	2.46	0.99	2.90	0.96
NP4	2.54	0.99	2.91	0.95
NP5	2.58	0.99	2.93	0.95
NP6	2.57	0.99	2.96	0.95

where λ = 488 nm is the wavelength of the excited laser radiation [20]. The dependence of L on the activation temperature (Fig. 6) demonstrates a decrease in the average transverse size of graphite particles from 6.9 to 6 nm with an increase in the activation temperature from 400 to 500ºC. In this case, the decrease in the value of L may be associated with the processes of gas release, splitting or removal of some components of the material. A further increase in the activation temperature (600 ÷ 900ºC) leads to an increase in the average size of graphite fragments, which can be explained by the process of sintering of the carbon material [9].

To study the conductive properties of the activated PCM, the impedance spectroscopy method was used (Fig. 7). As can be seen from the figure, the carbonization temperature significantly affects the conductive properties of the obtained PCM. This is evidenced by the quantitative changes in the real Z' and imaginary Z'' components of the complex resistance of the system. At an activation temperature of 400-600°C, the dielectric properties of the PCM are predominant. A higher carbonization temperature leads to lower values of the real resistance Z'. As noted above, the carbonization of the starting material at high temperatures (700 ÷ 1000 °C) is accompanied by the nucleation of graphite inclusions and the formation of ordered graphite layers. However, in this case, the exception is sample NP6, for which the value of the real resistance increases slightly compared to samples NP4 and NP5. It can be omitted that this is due to the peculiarities of the porous structure of this sample of carbon material, namely the presence of a significant number of micropores approximately 40%.

The dependences of the specific electrical conductivity of chemically activated PCM in semi-logarithmic coordinates are presented in Fig. 8 (a). From this graph it is clear that the highest value of electrical conductivity (25 Ohm^{-1}·m^{-1}) is possessed by the PCM sample obtained at 800°C. The dependence of the total electrical conductivity σ* on the temperature of chemical activation is shown in Fig. 8 (b). The errors in the

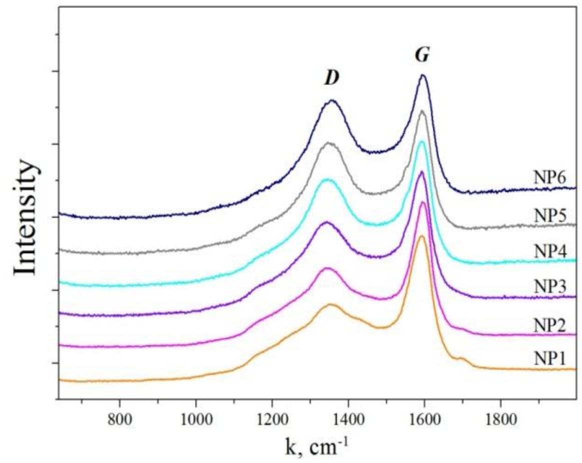


Fig. 5. Raman spectra for NP samples.

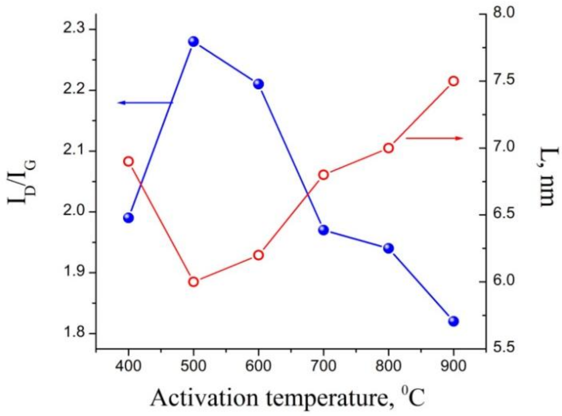


Fig. 6. Dependence of the ratio of integrated intensities (I_D/I_G) and the average size of graphite fragments (L) on the chemical activation temperature.

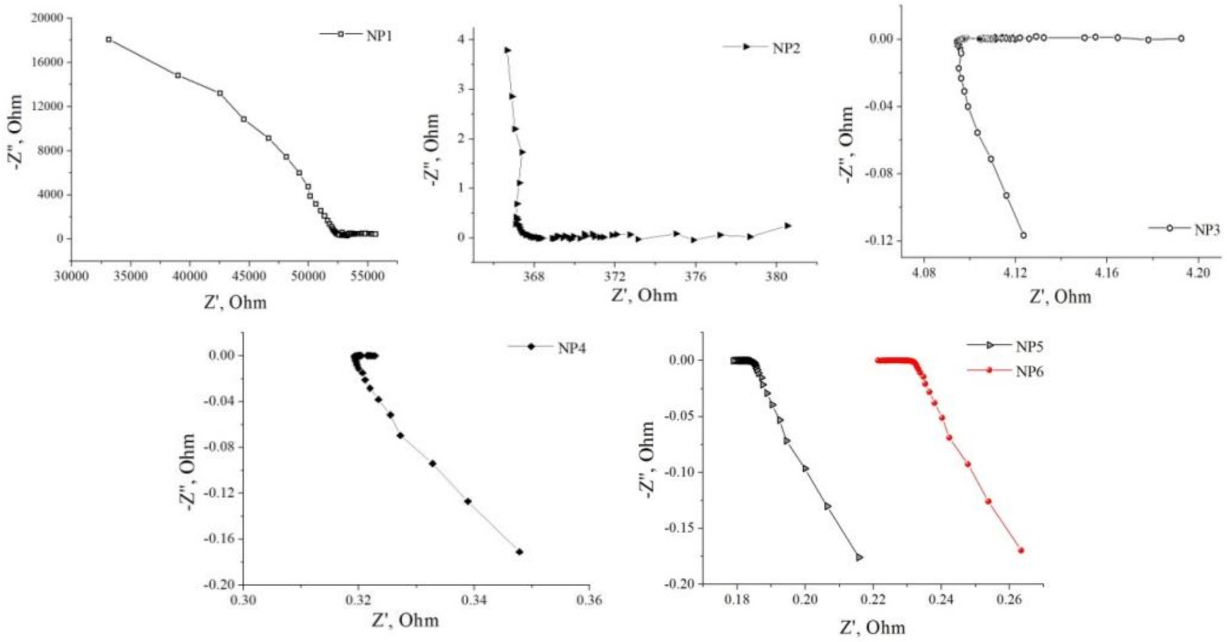


Fig. 7. Nyquist diagrams for thermochemically activated PCM.

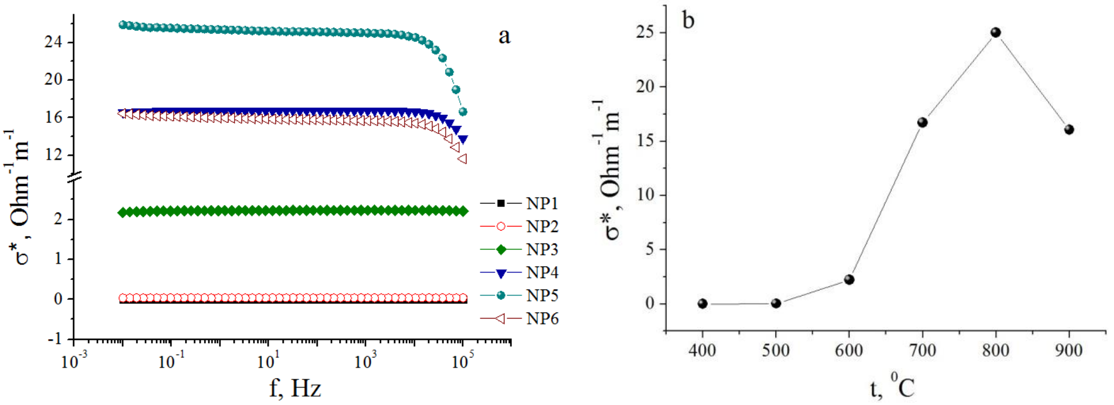


Fig. 8. Frequency dependences of specific electrical conductivity for PCM (a) and dependence of specificelectrical conductivity on activation temperature (b).

approximation for obtaining the specific conductivity values do not exceed 1%.

Conclusions

Mesoporous carbon materials with a specific surface area of 734 – 1385 m^2/g were obtained by thermochemical activation of walnut shells with orthophosphoric acid.

It was shown that the surface of the obtained PCM is quite rough, and the particles of the carbon material have surface microcracks and round (oval) pores with an average diameter of less than 10 µm.

Analysis of the pore size distribution for PCM samples demonstrates the presence of a significant number of pores with a diameter of 1.2 nm, ~2.6 nm and ~3.6 nm, and an increase in the activation temperature from 400 to 600°C is accompanied by a decrease in the volume of the corresponding pores.

The activation temperature has little effect on the change in the values of the fractal dimension for NP samples. The values of the fractal dimension of the microporous structure of the carbon samples range from 2.42 to 2.57. The fractal dimension of the mesoporous structure is within 2.90 - 2.96, which indicates a complex structure of mesopores.

Increasing the activation temperature from 400 to 900°C causes first a decrease, and then an increase in the average size of graphite fragments, which is associated, respectively, with the processes of degassing and subsequent sintering of the carbon material.

The analysis of impedance spectra made it possible to determine the specific electrical conductivity of the PCM samples. It was set that the highest value of electrical conductivity of 25 Ohm^{-1}·m^{-1} is possessed by the carbon material activated at 800°C.

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Структура, морфологія і електропровідні властивості термохімічно активованого вуглецевого матеріалу із шкарлупи волоських горіхів

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Для отримання пористого вуглецевого матеріалу зі шкаралупи волоських горіхів було використано метод термохімічної активації ортофосфорною кислотою. Досліджено вплив температури активації на структурні, морфологічні та електропровідні властивості вуглецевого матеріалу. Структурно-морфологічні характеристики вуглецевих зразків було охарактеризовано за допомогою низькотемпературної адсорбційної порометрії, СЕМ та раманівської спектроскопії. Показано, що термохімічна активація ортофосфорною кислотою дозволяє отримати пористі вуглецеві матеріали з питомою площею поверхні 734 - 1385 м^2/г в залежності від температури активації. Методом імпедансної спектроскопії досліджено електропровідні властивості отриманих пористих вуглецевих матеріалів.

Ключові слова: пористий вуглецевий матеріал, термохімічна активація, питома площа поверхні, фрактальна розмірність, імпедансна спектроскопія, питома електропровідність.