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Investigation of the effect of carbonization temperature of plant biomass on the electrochemical properties of carbon material

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Porous carbon materials (PCM) with different pore distributions in size and size of the specific surface area up to 250 m²/g were obtained by changing the carbonization temperature of plant biomass, namely walnut shells. The electrodes of electrochemical supercapacitors are formed based on the obtained carbon materials. The electrochemical behavior of PCM in 33% aqueous KOH solution has been studied by cyclic voltammetry and galvanostatic discharge-discharge methods and the value of their specific capacitance. The physicochemical processes occurring at the carbon electrode/electrolyte interface have been investigated by the method of impedance spectroscopy.

Keywords: porous carbon material, supercapacitor, cyclic voltammetry, specific capacitance

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Introduction

An electrochemical capacitor (EC), also known as supercapacitor is a new energy storage that fills the gap between a conventional capacitor and a battery [1, 2]. Obtaining new or modifying known materials for supercapacitors is a necessary method to achieve high power-intensive characteristics of both the material and the electrochemical system [3, 4]. To improve the electrochemical characteristics of EC, numerous strategies are used that are associated with a change in the structure and an increase in the surface area of materials for electrodes [5–7].

It is known that charge accumulation at the electrode/electrolyte interface mainly depends on the surface area, pore size distribution, and electrical conductivity of the electrode material. Based on many scientific papers, it has been proven that an improvement in capacitance can be achieved by increasing the surface

area with a minimal decrease in electrical conductivity [8]. However, the rate of charge accumulation processes due to the formation of a electric double layer (EDL) is also one of the key factors, mainly depending on the compatibility of the size of electrolyte ions with the distribution of pores over the size of the material [9]. A complex of electrochemical studies, such as cyclic voltammetry, galvanostatic cycling and impedance spectroscopy have been carried out in order to understand the fundamental relationship between structural characteristics (surface area and pore size distribution) and electrochemical properties (high specific capacitance). In this study, a PCM where obtained by the method of thermal carbonization in a wide range of temperatures (400 - 1000 °C), which is much cheaper than most methods - thermochemical, chemical-physical or catalytic activation. The influence of the carbonation temperature on the structural and adsorption characteristics of PCM was studied. The relationship between the structure of PCM and the energy-intensive characteristics of this

material in 33% KOH aqueous solution was established.

I. Materials and methods of research

Carbon materials were obtained by thermal carbonization of plant biomass (walnut shells) in the temperature range from 400°C to 1000°C with a step of 100°C. During the production process, the feedstock with a fraction of 5 - 15 mm was poured into an autoclave, placed in a furnace, and heated to a predetermined temperature at a rate of 5 °C/min. The applied methodological approach is close to that described in [8].

The morphology of the investigated porous carbon materials was studied using a JSM-6700F field emission scanning electron microscope. Secondary electron images were obtained on carboncoated samples under operating conditions: an accelerating voltage of 10 kV and a beam current of 0.75 nA.

The characteristics of the porous structure of the obtained PCM samples were determined based on the adsorption-desorption isotherm of nitrogen at its boiling point using an Autosorb Nova 2200e automatic gas analyzer (Quantachrome, USA). The sample was preliminarily degassed at a temperature of 180 °C for 18 hours. With the help of experimental nitrogen adsorption-desorption isotherms, the standard structural-adsorption parameters characterizing porous carbon materials were determined: specific surface area BET (Brunauer, Emmett, Teller) – S_{BET} [10], total pore volume – V_{total} – was determined on the basis of single-point adsorption isotherms for the relative pressure $p/p_0 \approx 1$, the t-method proposed by de Boer and Lippens – to determine the volume of micropores – V_{micro} [11]. The pore size distribution functions were determined by the desorption curve of the adsorption-desorption isotherm using the

DFT (Density Functional Theory) method [12].

Electrochemical properties of PCM were studied by galvanostatic charge-discharge cycling, potentiodynamic method, and electrochemical impedance method using two- and three-electrode cells using an Autolab PGSTAT/FRA-2 spectrometer (Netherlands). For research, the working electrode was made in the form of lamellae from a mixture of PCM (75% by mass) and a conductive additive (25% by mass). The electrolyte is a 33% aqueous solution of potassium hydroxide.

II. Results and discussion

In the process of obtaining carbon materials, a cellular structure is formed, which includes pores of various types, sizes and shapes. Determining the structure of materials is an important factor in the further study of the electrochemical behavior of the obtained PCM in an electrolyte. For this purpose, the methods of scanning electron microscopy and low-temperature porometry were used, and the structural and adsorption characteristics of PCM were determined.

High spatial resolution and depth of field on the surface of the PCM samples have been obtained by SEM investigation. Moreover, based on the principle of interaction of an electron beam with the substance, SEM is a relatively simple experiment and requires minimal preparation of a solid material sample and provides reliable information about the surface structure in the form of an "unprojected" three-dimensional image [13]. Typical SEM images of the surface of PCM carbonized at 800°C at different magnifications are shown in Fig. 1. Thus, the figure shows that this material is characterized by a meso- and microstructure, on the surface there are microcracks, round and oval-shaped pores, and the size is about

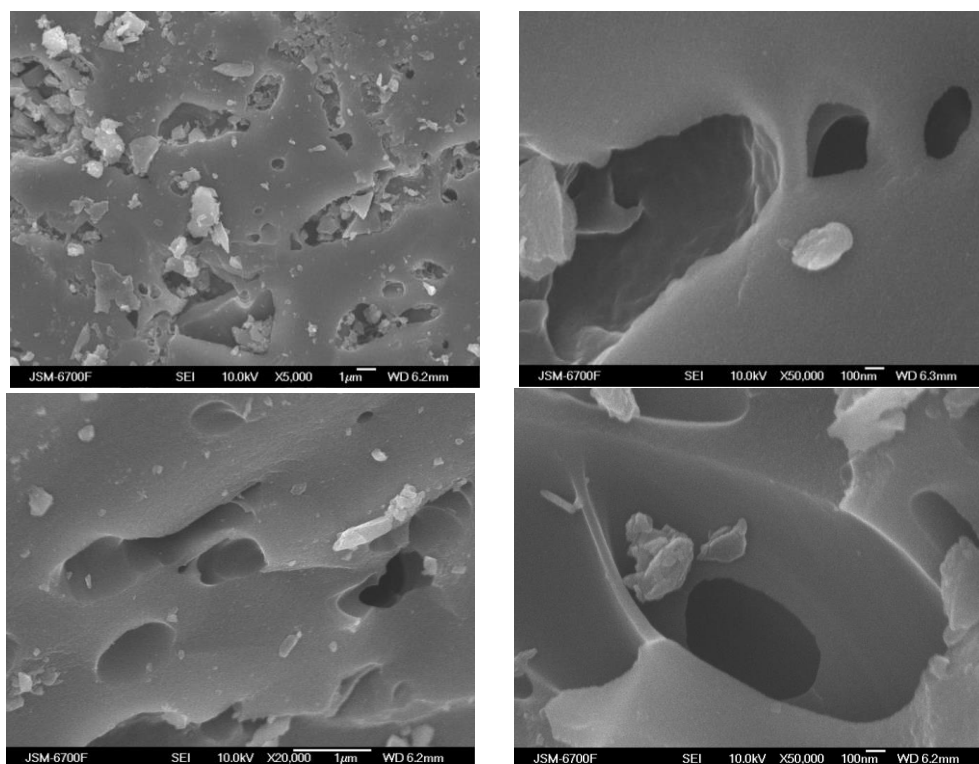


Fig. 1. SEM image of PCM obtained at 800°C.

0.2 microns to 45 microns. Overall, the isolated white inclusions observed on the surface are probably associated with ash residues formed during the thermal carbonization process.

Figure 2 shows a typical nitrogen sorption isotherm for PCM. Moreover, it belongs to the type II isotherm according to the IUPAC classification [14]. For this type of materials, the isotherm shows a divergence of the adsorption and desorption branches in the region of low relative pressures p/p_0 – low pressure hysteresis, which is associated with capillary condensation in mesopores. It is a more detailed description of the evolution of the porous structure of PCM was presented in our previous work [15].

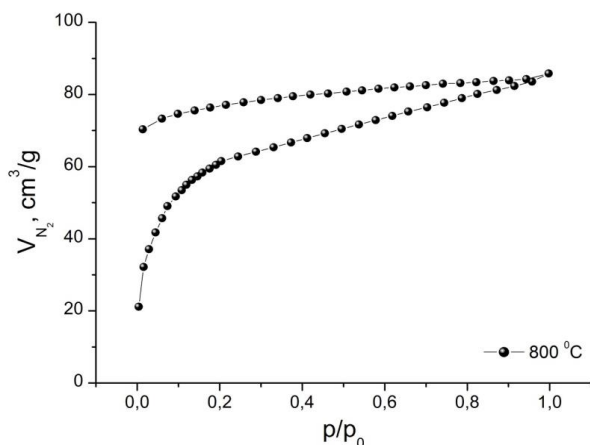


Fig. 2. Nitrogen sorption isotherms of PCM obtained at 800°C.

The BET equation $\frac{p/p_0}{A(1-p/p_0)} = \frac{1}{A_m C} + \frac{C-1}{A_m C} \cdot \frac{p}{p_0}$, which was used to calculate the specific surface area of PCM, is linear for narrow range of relative pressures (0.05–0.3) p/p_0 . Below this region, the BET equation predicts too little adsorption, and at high relative pressures – too high [10]. Despite these deviations, to determine the specific surface of carbon materials, the BET equation is usually used for the linear section of the adsorption isotherm with a linear dependence $\frac{p/p_0}{A(1-p/p_0)}$ on p/p_0 . Studies have shown that for the obtained PCM samples in this range, the BET graph is linear with the correlation coefficient $R^2 = 0.999$. This indicates the good applicability of this model and the reliability of the obtained specific surface area values. The main characteristics of the nanoporous structure of PCM are given in Table 1.

Table 1.

Structural and adsorption characteristics of PCM

Carbonization temperature, °C	S_{BET} , m^2/g	V_{total} , cm^3/g	d , nm
400	39	0.063	6.5
500	80	0.138	6.9
600	207	0.370	7.2
700	223	0.251	4.5
800	238	0.133	2.2
900	80	0.156	7.8
1000	47	0.068	5.9

The size distribution of pores in carbon materials was estimated using the NLDFT model (slit-like pores) (Fig. 3). Figure 3 show that the carbonization temperature of the feedstock affects the structure of the resulting PCM. Thus, increasing the carbonization temperature from 400°C to 800°C promotes the formation of micropores with a diameter of 1-1.5 nm, and the largest volume (0.064 cm^3/g) of micropores of the corresponding size, constituting 48% of the total pore volume, has a carbon material obtained at 800 °C. It should be noted that mesopores 2.5–5 nm in size also makes an important contribution to the specific surface area of the PCM under study.

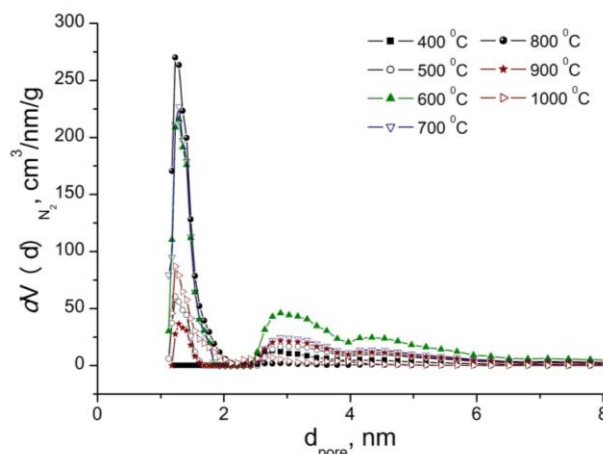


Fig. 3. Pore size distribution according to the DFT method.

PCM is a promising electrode material for electrochemical energy storage systems; therefore, further studies are devoted to studying the electrochemical behavior of PCM/electrolyte (33% KOH aqueous solution) systems.

Figure 4 shows discharge curves for PCM/electrolyte electrochemical systems. The studies were carried out using a two-electrode cell. Moreover, figure 4 shows that for electrochemical systems formed based on of the obtained PCM in 33% KOH has a characteristic capacitive behavior. The carbonization temperature affects the value of the specific capacity and the internal resistance of the electrochemical system. The voltage drop does not exceed 5% of the maximum at a discharge current of 10 mA. The specific capacity (C_{sp}) of the PCM was calculated based on the data obtained by the galvanostatic method at a discharge current of 10 mA, according to the following relationship: $C_{sp} = 2It/(U_{max} - \Delta U)m$, where I is the discharge current, t is the discharge time, $(U_{max} - \Delta U)$ is potential difference at the extreme points of the discharge curve – operating voltage window, ΔU is voltage drop, m is PCM mass. It has been determined that PCM obtained at 800 °C has the maximum specific capacity (118 F/g) for 33% KOH. This sample of carbon material was chosen for further detailed studies (hereinafter C-800).

The cyclic voltammetry method in the range of the scan rate from 1 to 50 mV/s was used to study the dependence of the specific capacitance charge/discharge process of the EC. The shape of the potentiodynamic curves indicates capacitive charge accumulation in the

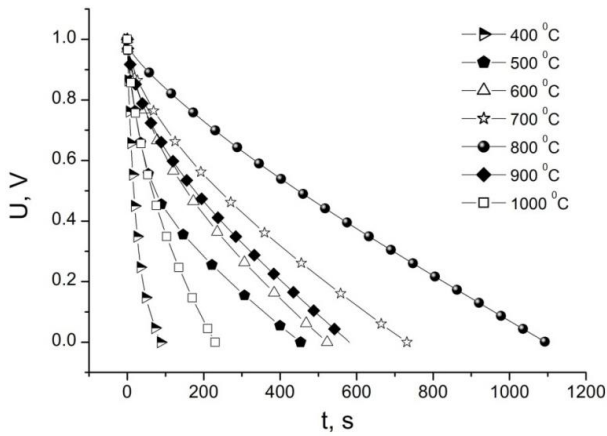


Fig. 4. Discharge curves at a current of 10 mA for PCM/electrolyte systems.

C-800/KOH/C-800 system (Fig. 5). The obtained voltammograms have a relatively symmetrical shape, there are no redox peaks. This indicates the quasi-reversibility of the charge/discharge process of the EDL and is typical for carbon electrodes operating on the principle of charge/discharge of the EDL. At a scan rate $s = 1\div 10$ mV/s, the curves are close to rectangular, which indicates that the total electrical capacity is provided by the capacitance of the EDL. Therefore, at a scan rate of $s \geq 15$ mV/s, the curves deviate from a rectangular shape, but the ability to reverse cycle is retained, which indicates the possibility of using this material as EC electrodes at high operating currents [16]. In fig. 6 shows the decrease in the specific capacity of a sample of carbon material with an increase in the scanning speed s . This is explained by the fact that with an increase in the scan rate s , the number of pores inaccessible to the electrolyte (mainly ultra- and micropores) increases, and, consequently, a smaller surface area of the PCM will participate in the formation of the EDL.

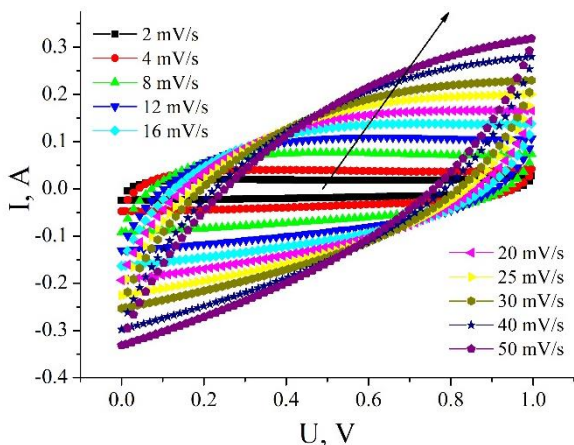


Fig. 5. Potentiodynamic curves for the C-800/electrolyte system

The maximum specific capacitance for the C-800 sample in aqueous KOH electrolyte was calculated based on the data obtained by cyclic voltammetry (Fig. 6) using the technique described in [17]. In the kinetic model [17], it is assumed that the scan rate s affects the total specific capacitance C_{sp} of the electrochemical system. Furthermore, a decrease of parameter s leads to an increase

in C_{sp} , and the dependence of C_{sp} on s can be extrapolated to $s \rightarrow 0$ using the functional dependence on s : $1/C_{sp} = 1/C_{s \rightarrow 0} + b\sqrt{s}$, where $C_{s \rightarrow 0}$ is the maximum specific capacity that can be get, b is a constant value. Therefore, as a result of extrapolation of C^{-1} from $s^{1/2}$ to the Y axis (Fig. 7), the maximum specific capacitance of the C-800/electrolyte electrochemical system was determined to be 125 F/g.

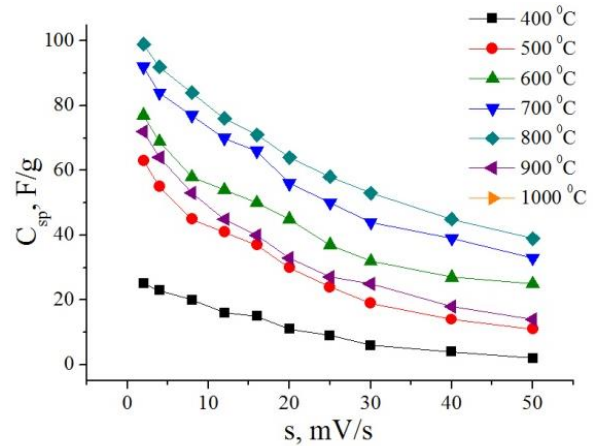


Fig. 6. Specific capacity of PCM C-800 depending on s .

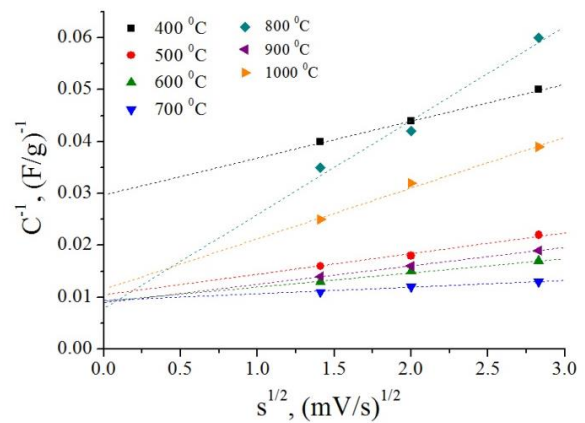


Fig. 7. Dependence of C^{-1} on $s^{1/2}$.

The dependence of the specific capacitance of the C-800 material on the value of the charge/discharge current was studied by the method of galvanostatic cycling. Moreover, the discharge specific capacity and internal resistance of the carbon material C-800 were determined based on the obtained chronopotentiograms (Fig. 8). The dependence of the values of specific capacitance and resistance on the magnitude of the discharge current is shown in Fig. 9. A sharp drop in voltage ΔU_R (Fig. 8, insert) at a charge/discharge current indicates the presence of ohmic resistance in the PCM/electrolyte electrochemical system. Since the voltage drop occurs in the electrochemical system when the galvanostatic charge/discharge current increases, the discharge current value did not exceed 100 mA for all ECs, keeping the voltage drop value below 20% [18]. However, it should be noted that the value of the charge/discharge current has practically no effect on the internal resistance of the electrochemical system, the value of which is 1 - 1.5 Ohm with an error of 0.2 Ohm (Fig. 9).

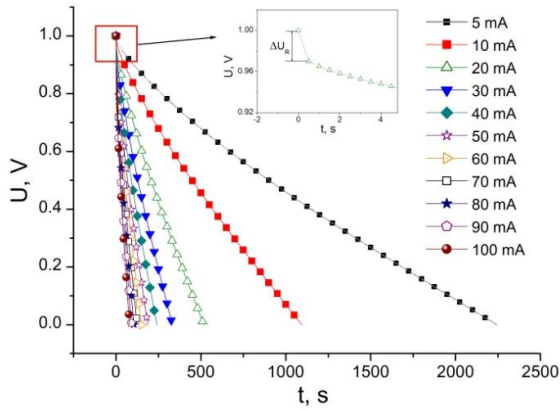


Fig. 8. Chronopotentiograms for sample C-800 obtained at different discharge currents.

The physicochemical processes occurring at the carbon electrode/electrolyte interface were studied using electrochemical impedance spectroscopy. A three-electrode cell was used for these studies. The cell was constructed using a C-800 sample as a working electrode, a platinum electrode as an auxiliary electrode, and silver chloride (Ag/AgCl) as a reference electrode. The material of the working electrode was made from the PCM C-800 under study, a conductive additive, and a binder material (in a ratio of 75:20:5) and pressed into a nickel mesh $5 \times 5 \text{ mm}^2$ in size. Electrolyte – 33% KOH. The open circuit potential for the working carbon electrode was -0.23 V with respect to the Ag/AgCl reference electrode. All studies were carried out at room temperature.

Presented in fig. 10 Nyquist graphs for the C-800/electrolyte electrochemical system in the frequency range from 0.01 Hz to 100 kHz show that in the high frequency range a small area resembling a semicircle is observed (inset in Fig. 10). This indicates insignificant Faraday processes at the PCM/electrolyte interface [19]. Then, behind the semicircle, you can observe an inclined linear section, the presence of which is associated with the process of electrolyte penetration into pores of different diameters and shapes and, as a result, frequency dispersion. This is the main reason for the impedance deviation from the theoretical vertical line (ideal capacitance behavior) at low frequencies. This behavior of impedance spectra is inherent to porous carbon electrodes. However, the maximum specific capacitance can be achieved at a very low frequency, so EC should be used under DC conditions or at very low frequencies [20].

Increasing the voltage applied to the working carbon electrode leads to an increase in the number of potassium ions that participate in the formation of EDL at the electrode/electrolyte interface. Thus, the capacity accumulated on the surface of the carbon electrode increases. When the bias voltage increases, changes in the shape of the Nyquist diagram occur, which are most noticeable in the low-frequency section (Fig. 10). A small deformed semicircle in the high-frequency range of the Nyquist diagram indicates that the EDL capacitance is dominant and the accumulated pseudocapacitive charge is negligible [3].

The optimal results of fitting the experimental curves

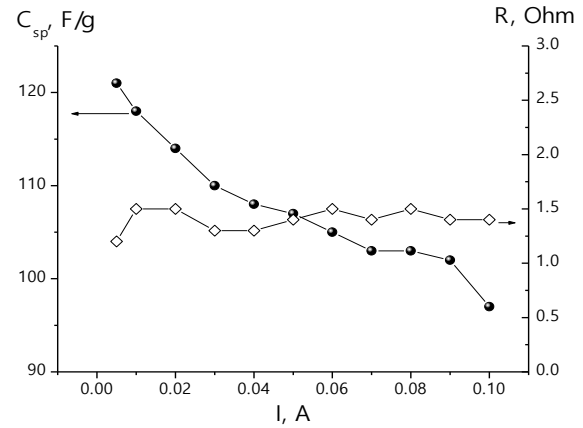


Fig. 9. Dependence of the specific capacitance of the C-800 sample on the current.

Z' ($-Z''$) (Fig. 10) were obtained using the electrical equivalent circuit (EEC) shown in Fig. 11. The EEC includes a series equivalent resistance R_1 (resistance of the electrolyte, contacts and underwater wires), $CPE_1 \parallel R_2$ links, which is responsible for diffusion processes in the macropores of the carbon material and charge accumulation at the carbon material/electrolyte interface, and $C_1 \parallel R_3 - CPE_2$ is responsible for processes in meso- and micropores. The elements of the constant phase of the CPE are used to provide flexibility in the simulation. The CPE₁ are a capacitive-type constant phase element reflecting the processes of charge accumulation in macropores of a carbon material, CPE₂ is a diffusion-type constant phase element associated with processes of limited diffusion of K^+ ions in micropores, R_2 and R_3 are charge transfer supports in a carbon material.

The proposed EEC (Fig. 11) is well appropriate for approximating the impedance spectrum (Fig. 10) in a wide frequency range, and the squared deviation of the approximation curve from the experimental spectrum is 3×10^{-4} . Furthermore, obtaining the values of the parameters included in the circuit is due to the use of EES (Table 2).

Since at low frequencies the active resistance is practically independent of frequency, it is possible to estimate the approximate capacitance values of the studied PCM, which will depend on the applied voltage. According to the ratio $C = 1/2\pi\nu Z''$ and taking into account the mass of the electrode, the specific capacitance of the PCM was calculated. Moreover, at an open circuit voltage, the specific capacitance of the studied PCM is 3.3 F/g . It should be noted that this capacitance is provided only as a result of physical absorption at the electrode/electrolyte interface of carbon. By applying a voltage to the PCM-electrode, we cause the appearance of electrostatic forces, which encourage a greater number of ions to participate more intensively in the formation of the EDL at the PCM/electrolyte interface. This fact leads to an increasing specific capacitance of the material, which at a bias voltage of -1 V is 84 F/g .

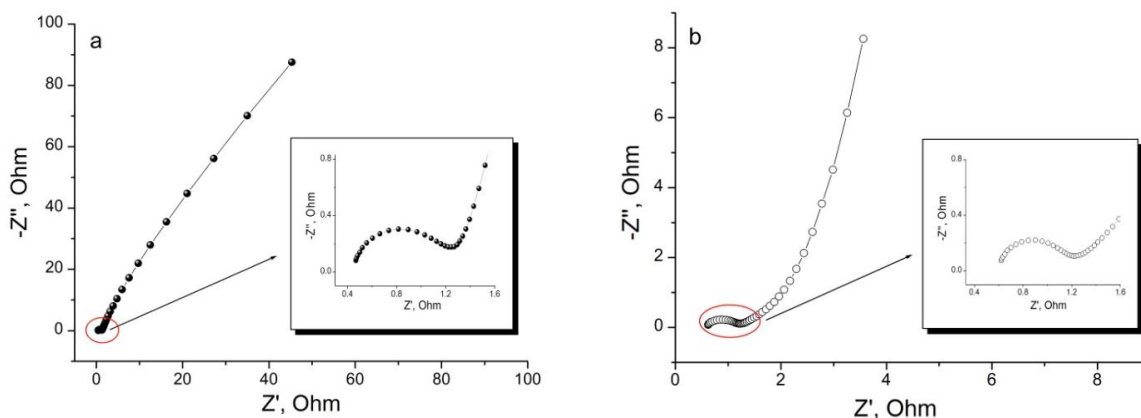


Fig. 10. Nyquist diagram for the C-800/electrolyte system at an open circle voltage of -0.23 V (a); at bias voltage -1 V(b).

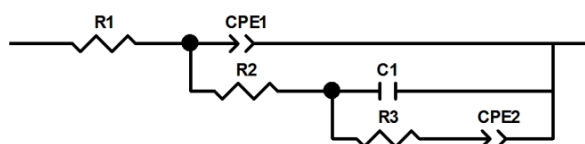


Fig. 11. EEC for approximate the impedance spectra.

Table 2.

EES parameters for the C-800/KOH electrochemical system

Potential, V	R ₁ , Ohm	CPE _{1T} , mF	CPE _{1P}	R ₂ , Ohm	C ₁ , F	R ₃ , Ohm	CPE _{2T} , F	CPE _{2P}
-0.23	0.41	1.9	0.72	0.95	0.03	0.55	0.05	0.67
-1	0.55	4.8	0.63	0.77	0.19	0.60	0.98	0.79

Conclusions

The galvanostatic and potentiodynamic methods were used to research the energy-intensive characteristics of PCM samples in 33% KOH. It was determined that the most effective electrode materials for EC based on KOH electrolyte is carbon material obtained by carbonization at 800 °C, which has the largest specific surface area (238 m²/g). Moreover, it is determined that the primary carbonization of plant biomass at a temperature of 800 °C makes it possible to obtain PCM with a specific capacity of up to 97 – 121 F/g in the range of operating currents of 5 – 100 mA at a maximum charge/discharge voltage of 1 V.

The method of impedance spectroscopy was applied, which makes it possible to obtain information on the kinetics of the formation of EDL at the PCM/electrolyte interface depending on the pore size. The proposed EEC for describing the electrochemical processes occurring in the PCM/electrolyte system is in agreement with the results of impedance spectroscopy. The physical interpretation of the circuit elements is presented.

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Дослідження впливу температури карбонізації рослинної біомаси на електрохімічні властивості вуглецевого матеріалу

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Змінюючи температуру карбонізації рослинної біомаси, а саме шкарлупи волоських горіхів, отримано пористі вуглецеві матеріали (ПВМ) з різним розподілом пор за розміром та величиною питомої площі поверхні до 250 м²/г. На основі отриманих вуглецевих матеріалів сформовано електроди електрохімічних суперконденсаторів. Методами циклічної вольтамперометрії та гальваностатичного заряду-розряду вивчено електрохімічну поведінку даних ПВМ у 33 % водному розчині КОН та визначено значення їх питомої електроємності. Методом імпедансної спектроскопії досліджено фізикохімічні процеси, що відбуваються на межі розділу вуглецевий електрод/електроліт.

Ключові слова: пористий вуглецевий матеріал, суперконденсатор, циклічна вольтамперометрія, питома ємність.