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MORPHOLOGY, CONDUCTIVITY AND ELECTROCHEMICAL PROPERTIES OF HYDROTHERMAL CARBONIZED POROUS CARBON MATERIALS

N.I. NAGIRNA, V.I. MANDZYUK

Abstract. The paper studies the morphology, conductivity and electrochemical properties of carbon materials, obtained from raw plant materials at different condition of hydrothermal carbonization, using low-temperature porometry, impedance spectroscopy and galvanostatic charge/discharge. It is set, that in porous structure of carbon materials micropores are dominant; when carbonization temperature increased the specific surface and pore volume decrease more than 10 times. The temperature growth results in increasing the electrical conductivity of the carbon material more than 6 orders. It is found, that the maximal value of specific capacity (1138 mA·h/g) has an electrochemical system based on porous carbon carbonized at 1023 K.

Keywords: porous carbon material, morphology, conductivity, lithium power source.

1. INTRODUCTION

As know [1], current creative process in lithium power sources (LPS) is a result of lithium ions insertion (intercalation) into cathode material. Energy potential of current sources is determined by possibility of cathode absorb reversibly a certain number of "guest" ions, which is characterized by insertion degree x . To increase significantly the amount of absorption centers and increase the specific energy parameters of the power sources the maximum possible dispersion of cathode material in order to increase the specific surface of the material is used. When used as a cathode porous carbon material (PCM) the value of its specific surface area can be effectively controlled by selecting of carbonization temperature [2, 3]. The question is, how carbonization temperature effect on the electrochemical processes in the LPS based on PCMs and its specific energy characteristics, and is the subject of this paper.

2. EXPERIMENTAL

Mechanically crushed apricot pits, placed in an autoclave filled with distilled water, were the initial material for carbonization. Tightly sealed autoclave is placed in the furnace, where PCM was obtained at a certain temperature. Hydrothermal carbonization of the material was carried out in the temperature range $T = 873 - 1373$ K (temperature change every 50 K) at a pressure of water vapor in the

range $(6 - 8) \cdot 10^5$ Pa. The regimes of hydrothermal carbonization of feedstock are described in detail in [4]. After the heat treatment material obtained has undergone mechanical milling twice.

The textural characteristics of samples were analyzed on the basis of low-temperature (77.4 K) nitrogen adsorption-desorption isotherms recorded using a Quantachrome Autosorb Nova 2200c adsorption analyzer. Before measurements, the samples were heated at 180°C for 24 h. Quantitative analysis of adsorption/desorption isotherms was held according to the standard BET- [5] and t- [6] methods. This made it possible to calculate the parameters of porous structure of carbon materials: surface area (S_{sp}), micropore surface (S_{micro}), the external pore surface (S_{ext}), micropore volume (V_{micro}). The total pore volume V was evaluated from the nitrogen adsorption at $p/p_0 \approx 0.99$, where p and p_0 denote the equilibrium and saturation pressure of nitrogen at 77.4 K, respectively. The pore size distribution (PSD) was calculated using nonlocal density functional theory method [7] using equilibrium model with slit pores.

To study the electrical parameters of PCMs used the capacitor system consisted of two copper electrodes and a sample in between. Nyquist diagrams $Z' = f(Z'')$, where Z' and Z'' – real and imaginary parts of complex resistance of system, was carried out using a Autolab FRA-2 (Frequency Response Analyzer) at $10^{-2} - 10^5$ Hz and voltage amplitude of 10 mV. The specific resistance, conductivity and frequency dependence of the electrical parameters were calculated taking into account the geometric parameters of samples according to equation

$$\rho^* = \rho' - j\rho'',$$

where $\rho' = Z'A/d$ and $\rho'' = Z''A/d$, and A , d – electrode surface area and sample thickness respectively. Complex conductivity was determined from the ratio

$$\sigma^* = 1/\rho^* = \sigma' + j\sigma'',$$

where $\sigma' = \rho'/M$, $\sigma'' = \rho''/M$, $M = |Z^*|^2 (A/d)^2$, and total conductivity $\sigma^* = \sqrt{(\sigma')^2 + (\sigma'')^2}$ [8].

The cathode was made from a mixture of PCM and a binder component (teflon) at a ratio of 96%: 4% respectively. These components mechanically mixed to obtain a homogeneous mixture, then acetone was added to achieve a paste-like consistency and the resulting slurry was applied to the nickel mesh size of 5×5 mm² and pressed. The anode from a lithium foil was pressed at nickel net. After drying electrodes impregnate into the electrolyte solution and element was sealed. 1M solution of lithium tetrafluoroborate (LiBF₄) in γ -butyrolactone was used as an electrolyte. All operations for LPS manufacturing was carried out in a glove box with dry atmosphere. The equilibrium electrode potential of explored materials regarding lithium reference electrode was 3.2-3.4 V. Electrochemical insertion of lithium ions into PCM carried out in galvanostatic mode at a current density of 40 μ A/cm² (2 mA/g).

Specific discharge capacity C_{sp} of LPS was calculate as

$$C_{sp} = It/m,$$

where I – discharge current, t – discharge time, m – mass of PCM.

Specific energy E_{sp} of LPS was determined as area under discharge curve $U = f(C_{sp})$.

3. RESULTS AND DISCUSSION

The primary step in the study of porous structure of any material is to obtain adsorption/desorption isotherms on which you can make further calculations. Typically, sorption isotherm is dependence of quantity of gas adsorbed by material on relative pressure p/p_0 at constant temperature.

Isotherms of nitrogen adsorption/desorption for PCMs (Fig. 1), obtained at different temperatures carbonization, is similar quality. However, a more detailed analysis, based on a quantitative comparison of the magnitude of adsorbed nitrogen and form of adsorption curve, gives reason to assert that for PCMs received at carbonization temperatures of 873-1173 K adsorption isotherm can be classified as I type according to the IUPAC classification [9], which is typical for microporous solids

having a relatively small external surface (activated carbons, zeolites, organic metal grating). The adsorption isotherm for PCMs, obtained at higher temperatures, can already be attributed to the II type that is characteristic of non-porous adsorbents. This difference is due to processes that occur during the carbonization of feedstock. In particular, temperature range, which corresponds to a change of adsorption isotherm type, is in the region where the transition from polycondensation of aromatic molecules to the stage of first carbonization take place, during which begins the formation of carbon layers and the formation of primary graphite nuclei. The removing of volatile components at the stages of cyclization, aromatization and polycondensation leads to the formation of developed microporous structure with small external surface in the raw material. The reduction of micropores number, their merging with transition in mesopores and the formation of microparticles of non-porous PCM are possible at higher temperatures. It should be noted that all isotherms are characterized by a mismatch of adsorption and desorption branches, especially in the area of low pressure, called low pressure hysteresis. This can be due to the presence of long and narrow pores with narrow necks close in size to the nitrogen molecules [9].

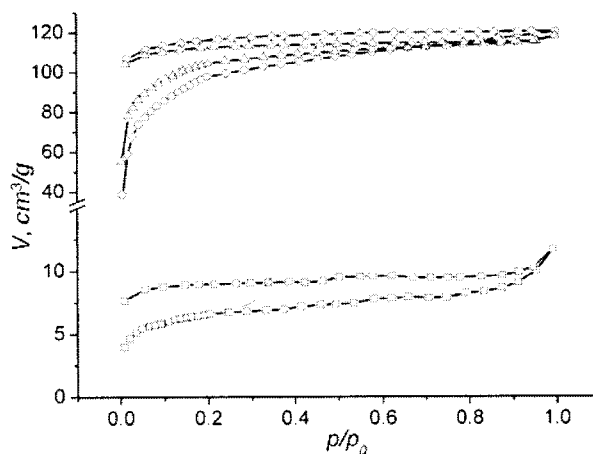


Fig. 1. Nitrogen sorption isotherms for PCMs: 873 K (-○-), 1073 K (-△-), 1323 K (-□-).

Quantitative analysis of sorption isotherms shows that PCMs obtained at carbonization temperatures of 873-1173 K, characterized by high specific surface area, the value of which gradually decrease with increasing temperature (Tab. 1). The specific surface area decreases more than an order at the temperatures of carbon layers formation (first carbonization). The data of Tab. 1 confirm the assumption of the mesopore existence in PCM, in addition to micropores; the number one, so other decreases with increasing temperature. Somewhat different is the situation regarding the ratio of micro- and mesopores surface. For samples obtained in the temperature range 923-1123 K, the proportion of the surface occupied by micropores is greater than 85% of the surface, whereas this value is 60% when the temperature increases. It is clear, that when carbonization temperature is increased the parameters that characterize the pore volume of PCM will be altered so. In particular, there is a tendency to decrease of the total volume of pores V (Tab. 1). The micropore volume V_{micro} is changed similarly. Having the last two parameters, you can determine the amount of meso- and macropores. However, the absolute values are less informative; more interesting is the change in the micropore fraction in the total pores at temperature increasing. As follows from Tab. 1, in the temperature range of 873-1123 K fraction of micropores is from 64 to 88 %, while at higher temperatures their content sharply reduced and does not exceed 50 %. This behavior is similar to that described above change of the proportion of surface micropores S_{micro} relation to the total surface S_{sp} .

Carbonization temperature T , K	S_{sp} , M^2/g	S_{micro} , m^2/g	S_{ext} , m^2/g	V , cm^3/g	V_{micro} , cm^3/g
873	357	282	75	0,186	0,119
923	407	354	53	0,193	0,149
973	374	338	36	0,174	0,143
1023	343	292	51	0,166	0,123
1073	382	338	44	0,182	0,143
1123	331	314	17	0,148	0,131
1173	265	185	80	0,151	0,078
1223	168	96	72	0,116	0,040
1273	45	34	11	0,031	0,014
1323	24	18	6	0,018	0,008
1373	12	9	3	0,011	0,005

Tab. 1. Structure-sorption parameters of PCMs.

PSD (Fig. 2) indicates that the in explored samples micropores dominate, which is in the range of $d = 1.25-1.65$ nm; the amount of adsorbed nitrogen remains unchanged to a temperature of 1173 K. Above this temperature gas volume decreases sharply and it content decreases in 10 times at $T = 1323$ K.

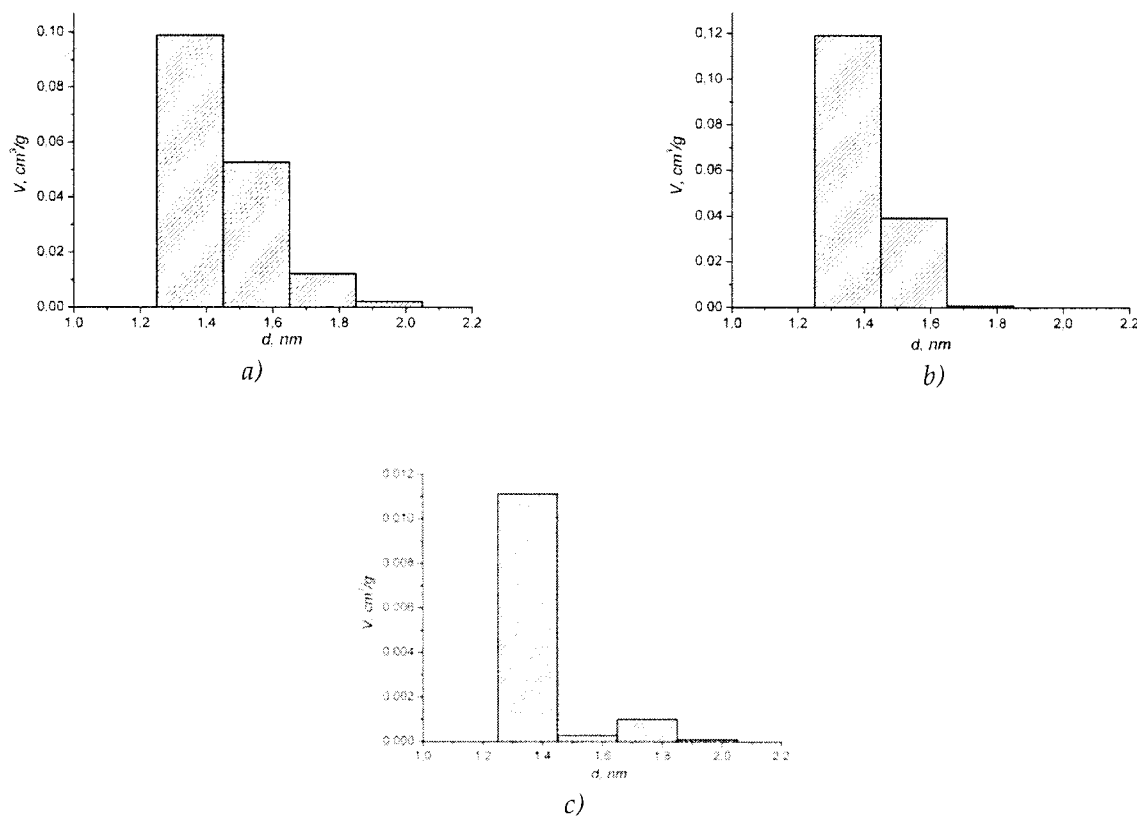


Fig. 2. Pore size distribution for PCMs carbonized at 873 (a), 1073 (b), and 1323 K (c).

As the main factors that affect the electrical properties of the PCMs is thermal treatment of feedstock, which detects the presence on the surface of different functional groups, chemical composition and morphology of the material, and the pressure applied to the packing of particles, so it is necessary to establish relation between conductivity of PCMs and temperature carbonization of feedstock.

As follows from the results of the research, increasing of PCM carbonization temperature greatly changes the character of the complex impedance system (Fig. 3), thus reflecting the possible changes in

the electrical conductivity of carbon materials. These are the real and imaginary reduce the system resistance more than 4-6 order, and the general view of hodograph curve.

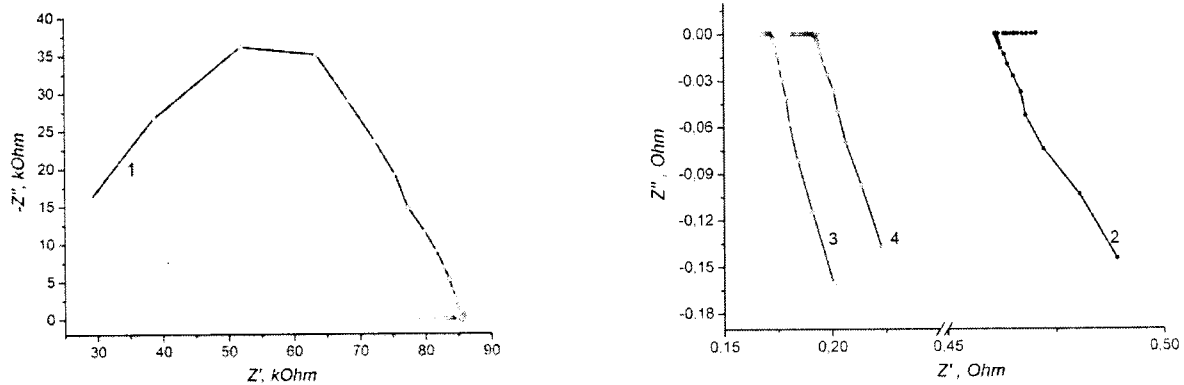


Fig. 3. Nyquist diagrams for PCMs, obtained at 923 (1), 1073 (2), 1223 (3) and 1373 K (4).

A characteristic feature of the frequency behavior of resistances with temperature increasing is the transition of imaginary resistance values Z'' from negative half plane in a positive one, indicating the dominance of inductive behavior over capacitive one at frequencies $f = 10^5$ -400 Hz. The values of real resistance Z' for PCMs, obtained at temperatures above 923 K, are independent on frequency. Based on the ratios described above the values of real, imaginary and complete conductivity of PCMs and their dependence on the frequency were calculated (Fig. 4). Because the value of the real part of the electrical conductivity (Fig. 4, a) is approximately in two orders greater than the imaginary conductivity (Fig. 4, b), the contribution of the latter in full conductance (Fig. 4, c) of PCM is insignificant. As follows from the data, values σ' , σ'' and σ^* are independent on frequency in the frequency range $f = 10^{-2}$ - 10^3 Hz for all materials. In the range of low frequencies ($f < 10^{-2}$ Hz) for PCMs ($T > 1123$ K) is a slight increase in value σ' and σ^* accordingly.

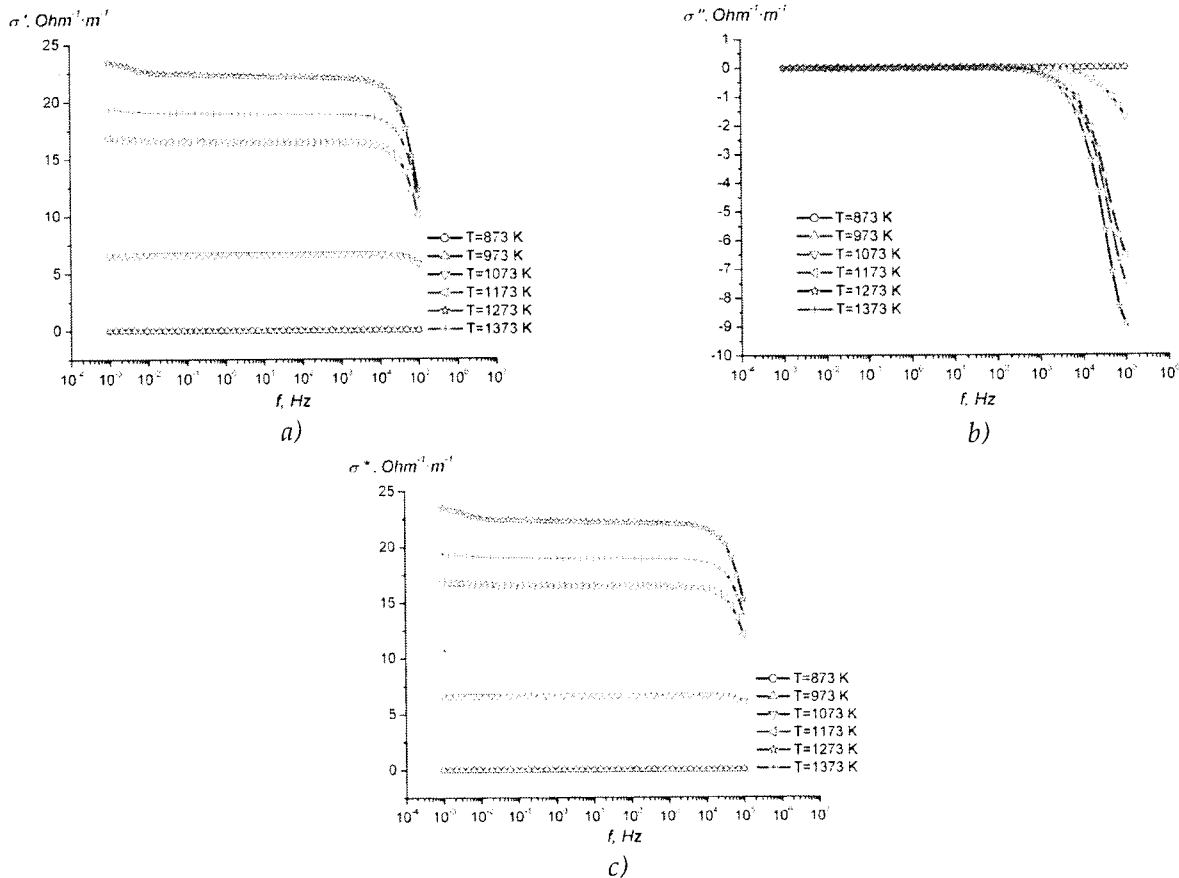


Fig. 4. The frequency dependences of real (a), imaginary (b) and complete (c) conductivities of PCMs obtained at different carbonization temperatures.

Interesting is the behavior of the conductivity in the frequency range 10^3 - 10^5 Hz. For PCMs, carbonized at a temperature below 1023 K, a sharp drop of conductivity with decreasing frequency is observed in this frequency range, whereas conductivity increases when frequency decreasing at $T > 1023$ K. This is because during carbonization is thermal decomposition of raw materials, at which a removing of volatile materials take places. Further increase in temperature leads to nucleation of graphite inclusions, which increases when heated and form ordered graphite layers.

The kind of raw materials and treatment conditions determine the size and number of graphene layers and relative orientation of crystallites. The size and orientation of crystallites are important because they determine the texture of the material and mechanisms of electrical conductivity [10]. It is believed that at 1023 K a change of PCM conductivity is observed caused by the above-mentioned reasons.

Submission of frequency dependence of conductivity in semi-logarithmic coordinates makes it possible to determine the value of the electrical conductivity of the material by extrapolating of frequency independent area of curve to its intersection with the σ^* -axis (when $f \rightarrow 0$ we have access to the direct current) (Fig. 5).

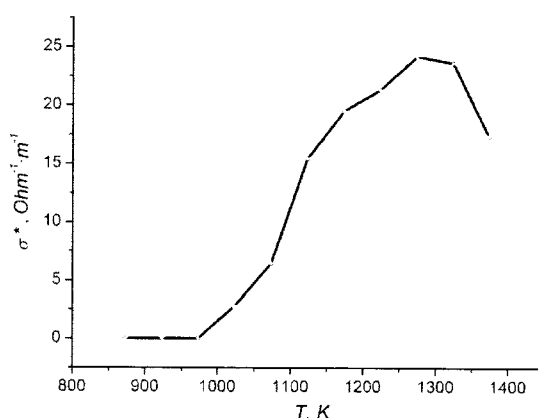


Fig. 5. The complete conductivity of PCMs as function of carbonization temperature.

As can be seen from this dependence, the increasing of carbonization temperature leads to a significant increase in conductivity compared to the temperatures $T \leq 973$ K. Most probably, this is because at lower carbonization temperatures is a partial burning organic base of feedstock and incomplete formation of the complex frame from condensed aromatic layers formed by carbon atoms. As a result, the complete resistance of aggregate particles of PCM, which depends on a resistance of each particle and the contact resistance between them is quite high (the conductivity in this temperature range is between $0.31 \div 1250 \mu\text{Ohm}^{-1}\cdot\text{m}^{-1}$). There is an increase in conductivity of PCM to the value of $24,23 \text{ Ohm}^{-1}\cdot\text{m}^{-1}$ ($T = 1273$ K) with increasing temperature, then there is a slight decline – its value is $17,38 \text{ Ohm}^{-1}\cdot\text{m}^{-1}$ at $T = 1373$ K.

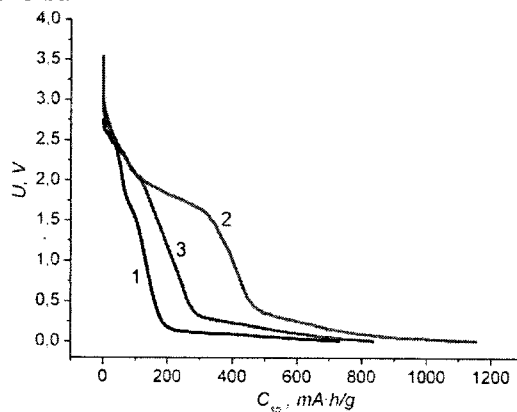


Fig. 6. Discharge curves of LPS based on PCM carbonized at 873 (1), 1073 (2), and 1373 K (3).

Electrochemical power sources were made on the bases of PCMs. As follows from the results of the experiment, the electrochemical process of lithium ions insertion into PCM has a stage character. There are 3-4 areas at discharge curves with different slope ratio (Fig. 6), at which speed the flow of electrochemical processes are different. For all material the main part of discharge capacity is provided at voltages less than 0.5 V. The wide horizontal plateau on the discharge curve answers this process. This behavior is typical for almost all carbon materials used in LPS [11-13]. The forming of insertion compounds as Li_xC takes place in this voltage range. Unlike natural (synthetic) graphite or highly ordered carbon materials for which this area is divided further into several horizontal plateau [14], for explored PCMs there is monotonically downward profile of this area of discharge curve. The latter is because the structure of the PCM is highly disordered and no clear boundaries of transition from one intercalation compound to another. Something different is character of discharge curves at voltages above 0.5 V. For PCMs, carbonized at temperatures of 1023-1173 K, can distinguish two areas in the voltage range of 2.8-1.6 V and 1.6-0.5 V (Fig. 6, curves 1 and 3); the first range can also be further divided into two ones (from 2.8 to 2 V and 2 to 1.6 V). For PCMs, carbonized at temperatures of 873-973 K and 1223-1373 K, such separation is difficult, because the first three intervals are merged into one, which corresponds to the voltage range 2.8-0.5 V. It is clear that such behavior is due morphological features of PCMs. In particular, PCM has a disordered structure and highly developed microporosity at low temperature carbonization (873-973 K), while at high temperatures (1223-1373 K) the surface area of the material decreases sharply, the process of material compacting and primary nucleation of graphite nanoclusters and layers take place. At carbonization temperatures of 1023-1173 K carbons are derived, which have properties of PCMs carbonized both at low and high temperatures. This feature provides the highest value of discharge capacity of these materials (Fig. 6, curve 2). As follows from the data (Tab. 2), the energy parameters of LPS depend on temperature carbonization non-monotonically. The greatest value of specific capacity has electrochemical system based on PCM, carbonized at 1023 K. However, due to higher discharge voltage the system based on PCM, carbonized at 1123 K, has a maximum specific energy. This behavior is caused mainly by the morphology of the material – specific surface, pore volume and PSD.

Carbonization temperature T , K	C_{sp} , mA·h/g	E_{sp} , W·h/kg
873	730	325
923	800	445
973	866	575
1023	1138	690
1073	1025	779
1123	984	977
1173	593	643
1223	571	500
1273	545	553
1323	877	754
1373	835	547

Tab. 2. Discharge parameters of LPS based on PCMs.

Comparing the results of low-temperature porometry (Tab. 1) with data of Tab. 2, it can be state that there is no clear correlation between the value of the discharge capacity and structural morphological parameters of PCMs. The cumulative impact of all these parameters creates preconditions for larger values of discharge parameters.

Determine what fraction of the total capacitance is a value of specific capacity of the initial stage of lithium ions insertion $\varepsilon_{in} = \frac{C_{in}}{C_{sp}} \cdot 100\%$ and on the plateau $\varepsilon_{pl} = \frac{C_{pl}}{C_{sp}} \cdot 100\%$, where C_{in} – capacity spent in voltage range of 2.8-0.5 V, C_{pl} – capacity spent at voltages less than 0.5 V (Tab. 3).

Carbonization temperature T , K	ε_{in} , %	ε_{pl} , %
873	0,39	0,61
923	0,37	0,63
973	0,30	0,70
1023	0,28	0,72
1073	0,45	0,55
1123	0,53	0,47
1173	0,54	0,46
1223	0,52	0,48
1273	0,55	0,45
1323	0,48	0,52
1373	0,34	0,66

Tab. 3. Capacity distribution in LPS based on PCMs.

The analysis made gives the possibility to establish that the C_{in} value satisfactorily correlated with the value of specific surface of PCM. This fact indicates that in this voltage range the basic electrochemical processes associated with the occurrence of reactions at the electrolyte/PCM boundary. A proof of this is the behavior of the discharge curve, i.e. a slope ratio that characterizes the rate of the electrochemical reaction. It is clear that due to the high mobility of ions in the electrolyte and absence of diffusion limitations fastest processes will occur exactly at the interface of two phases. Lithium ions inserted into PCM to be affected by the carbon matrix and electrostatic interactions with other lithium ions. Therefore, these processes will occur in time slowly, that meet nearly horizontal area at the discharge curve. Regarding the relation between ε_{in} value and other structural and morphological parameters, the precise relationship between them does not exist. There is no clear relation between these parameters and ε_{pl} value. It is logical to assume that the value of the specific capacity on the plateau should not depend on variables that describe the properties of the carbon material surface, since, as noted above, the passing of electrochemical processes in a voltage range below 0.5 V, is associated with the involvement of microcrystalline structure of PCM particles, but not their surface.

4. CONCLUSIONS

PCMs, which have developed porous structure, obtained in the temperature range 873-1373 K by the method of hydrothermal carbonization of raw plant materials. This structure is formed by both micro- and mesopores. The increasing of carbonization temperature leads to a reduction in both types of pores with a simultaneous increase in the part of mesopores (from 12 % at $T = 873$ K to 55 % at $T = 1323$ K) relative to the total pore volume.

The growth of carbonization temperature of feedstock in the range of 873-1373 K results in increasing of PCM conductivity in more than 6 orders. It value achieves maximum at 1273 K and is $24 \text{ Ohm}^{-1}\cdot\text{m}^{-1}$.

Specific energy parameters of LPS depend largely on the structure and morphology of PCMs. The maximum value of specific capacity (1138 mA·h/g) has electrochemical system based on a PCM obtained at 1023 K. For this carbon material the main part of the transferred charge (72 %) spent on the formation of Li_xC insertion compounds. It is set, that the process of lithium ions electrochemical insertion into PCMs has stage nature. The electrochemical processes related to the reactions on the material surface in the voltage range of 3.3-0.5; at voltages less than 0.5 V, is the formation of a continuous series of non-stoichiometric insertion compounds as Li_xC according to the reaction $x\text{Li}^+ + xe^- + \text{C} \rightarrow \text{Li}_x\text{C}$.

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Address: N.I. Nagirna, The Electronic Device College of IFNTUOG', 223 Vovchynetska Str., Ivano-Frankivsk, 76000, Ukraine;
V.I. Mandzyuk, Vasyl Stefanyk Precarpathian National University, 57, Shevchenko Str., Ivano-Frankivsk, 76018, Ukraine.

E-mail: n.nagirna@mail.ru; mandzyuk_vova@ukr.net.

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Нагірна Н.І., Мандзюк В.І. Морфологія, провідність та електрохімічні властивості гідротермально карбонізованих пористих вуглецевих матеріалів. *Журнал Прикарпатського університету імені Василя Стефаника*, **3** (1) 2016, 46–55.

У роботі досліджено морфологію, провідність та електрохімічні властивості вуглецевих матеріалів, отриманих із сировини рослинного походження за різних умов гідротермальної

карбонізації, з використанням методів низькотемпературної порометрії, імпедансної спектроскопії та гальваностатичного заряду/розряду. Встановлено, що у пористій структурі вуглецевих матеріалів домінують мікропори; при збільшенні температури карбонізації питома поверхня та об'єм пор зменшуються більш ніж у 10 разів. Ріст температури також призводить до збільшення питомої електропровідності вуглецевого матеріалу більш ніж на 6 порядків. З'ясовано, що максимальним значенням питомої ємності (1138 мА·год/г) володіє електрохімічна система на основі пористого вуглецю, карбонізованого за температури 1023 К.

Ключові слова: пористий вуглецевий матеріал, морфологія, питома електропровідність, літієве джерело струму.