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Electrical Properties of Composites Based on Nanoporous Carbon Material

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In this work, the morphological and electrical properties of the composite nanoporous carbon material/thermally expanded graphite or acetylene black have been investigated. Nanoporous carbon material was obtained from plant materials by its thermochemical activation based on potassium hydroxide. The dependence of the specific capacity of the nanoporous carbon/electrolyte electrochemical system on the applied potential was determined by the impedance spectroscopy method. Furthermore, the concentration of charge transfer and the density of states, as well as the flat-band potential of the system under research, were determined based on the Mott-Schottky model.

Keywords: Mott-Schottky model, nanoporous carbon material, flat-band potential.

Introduction

The energy characteristics of electric energy storage devices are largely dependent on the electrode material and the electrolyte used as well as the electrochemical processes occurring at the electrode/electrolyte interface. The key tasks of electrochemical electrode/electrolyte systems research are to investigate the mechanisms of capacitive accumulation of electric charge, due to the formation of a double electric layer (DEL) and a pseudocapacitance, under redox reactions procedure. Of primary importance in the development of the maximum operating voltage value of a single cell and the amount of stored energy and power of the device is based on the selection of electrolyte solution. Since these parameters depend on the decomposition voltage of the electrolyte and its electrical conductivity [1, 2]. Thus, the duration of the cyclic stability of the electrochemical capacitor (EC) depends on the degradation of the electrode material, which is largely dependent on the electrolyte

used. Therefore, one of the most promising directions of research in electrochemical charge storage systems is the investigation of the physical and chemical processes as well as electrical properties of ECs electrode materials. It is advisable to use the method of impedance spectroscopy (IS) to investigate the electrochemical electrode/electrolyte interaction, determining the voltage at which charge transfer occurs in this system, as well as the value of the accumulated charge and the electrical resistance of the system [3]. Based on nanoporous carbon material, which is chemically resistant to most electrolytes, the IS method can determine the dependence of the specific capacity of the system on the applied potential and the potential region in which charge accumulation occurs due to of DEL formation. The magnitude of the accumulated charge and the order of the reaction as well as the probability of its occurrence depend on the applied potential in electrochemical systems in which the accumulation of charge occurs due to faraday reactions. In this case, it is one of the key

concepts associated with the chemical state of the system and the reactions that occur at a given potential. It is possible to determine the potentials of the passage of faraday reverse redox reactions and calculate the charge accumulated due to pseudocapacitance and also to research the range of working potentials of the electrode material based on using the impedance spectroscopy and the Mott-Schottky model [4]. The Mott-Schottky model is commonly used to measure of the flat-band potential of semiconductor material and to determine the density of charge into semiconductors/electrolytes interface system [5, 6]. In this work, the composite material formed based on of nanoporous carbon material (NCM) and thermally expanded graphite (TEG), or acetylene carbon black (ACB) has been selected as the object of the research. The Mott-Schottky model was used to research the mechanisms of charge accumulation in the electrochemical systems investigation, evaluate the electrical conductivity of the system, and determine the potentials at which the transfer of electrons and ions through the composite/electrolyte interface occurs.

I. Materials and methods

As the active material, was used NCM obtained from raw materials of plants by carbonization and activation with potassium hydroxide. Dried apricot seeds as a feedstock were crushed to fractions of 0.25 to 1 mm and carbonized in a close furnace at 330°C to 350°C with a heating rate of 10°C/min. The resulting carbon was mechanically crushed to fractions of 200 to 250 μm and mixed with potassium hydroxide and water in a weight ratio: $m(C):m(KOH):m(H_2O) = 1:1:2$. The resulting mixture was thoroughly stirred for 1 to 2 h, after which it was dried in the thermostat to constant weight. The dry material was placed in a furnace and heated in argon atmosphere at 900°C to 920°C with the heating rate of 10°C/min and kept at this temperature for 20 min. After cooling, the resulting material was washed with 5 % aqueous HCl and distilled water to neutral pH and again dried at 90°C to constant weight.

The characteristics of porous structure (surface area and total pore volume) of NCM were determined by analyzing the adsorption/desorption isotherms of nitrogen at its boiling point, obtained using Quantachrome Autosorb Nova 2200e (Quantachrome Instruments, Boynton Beach, FL, USA).

The electrochemical research of composite/electrolyte system were performed used the three-electrode cells by Autolab PGSTAT/FRA-2 spectrometer in galvanostatic and potentiodynamic modes in the frequency range from 10 mHz to 100 kHz. A 25 mg working electrode was made based on a composite NCM and TEG in a proportion 75:25

respectively, which was selected due to of the optimum ratio between the internal resistance and the specific capacity of the storage devices. Moreover, a similar ratio was used for the system based on NCM / acetylene carbon black composite. The specific surface area of the NCM was about 1240 m^2/g. Thus, the composite material was pressed into a nickel wire mesh with the size of 5*5 mm^2. After that, the formed electrodes were dried and placed in the test cells. Moreover, (Ag/AgCl) was used as a reference electrode which was filled by 3.5 M aqueous KCl. The (platinum electrode) was used as counter electrode. In this case, it was determined that the potential of the composite carbon material relative to the reference electrode is from -0.33 to -0.28 V under electrochemical research. The impedance spectra of the composite material under research were obtained in the potential region -1 V to 0.5 V in 0.1 V increments. The concentration of charge transfer and the flat-band potential were determined using the Mott-Schottky model at a frequency of 100 mHz.

II. Discussion of results

The morphology of the obtained NCM was investigated by the method of low-temperature adsorption/desorption of nitrogen, its quantitative characteristics of the porous structure are presented in Table 1. In particular, the value of specific surface area was determined using the BET method with a linear graph of the dependence of $1/[W(p_0/p)-1]$ on p_0/p in the range of adsorption isotherm for the ratio p_0/p in the range from 0.05 to 0.35 [7]. To research the structure of the microporous carbon materials, it is also advisable to use the Langmuir method, DFT, t-method, since the BET method does not completely take into account the contribution of micropores [7].

In the obtained NCM, the specific mesopore area is up to 15 % of the total surface area, which is equal to 1200 - 1300 m^2/g. It is defined by three independent methods and correlates within a margin of error of 10 %. On the one hand, the micropore volume determined by the t-method is 0.41 cm^3/g, which is about 60% of the total pore volume determined at maximum pressure. On the other hand, mesopores play the role of transport channels for the ingress of adsorbate or electrolyte into micropores. The investigation of the efficiency of the formed composite as the electrode material of the EC was carried out by impedance spectroscopy. Thus, the amount of charge accumulated in the DEL, which is formed at the composite/electrolyte interface, depends on the applied electrode potential [10]. Therefore, the determination of the dependence of the specific capacity of the composite/electrolyte system on the applied potential made it possible to evaluate the relationship

Table 1

The NCM sorption characteristics.

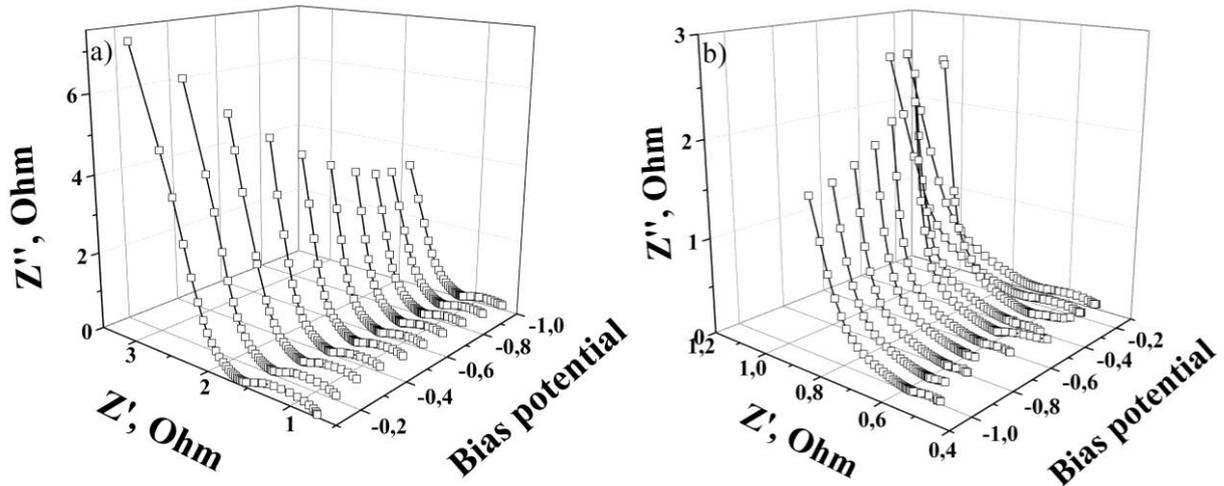| Sample	S_{BET}, m^2/g	S_L, m^2/g	S_{DFT}, m^2/g	$S_{t\text{-micro}}$, m^2/g	$S_{BJH\text{-meso}}$, m^2/g	S_{micro}, %	V_{total}, cm^3/g	V_{micro}, cm^3/g
NCM	1236	1405	1085	1040	160	84	0.69	0.41

Fig. 1. The Nyquist curves of the CT (a) and CA (b) systems in the negative potential area.
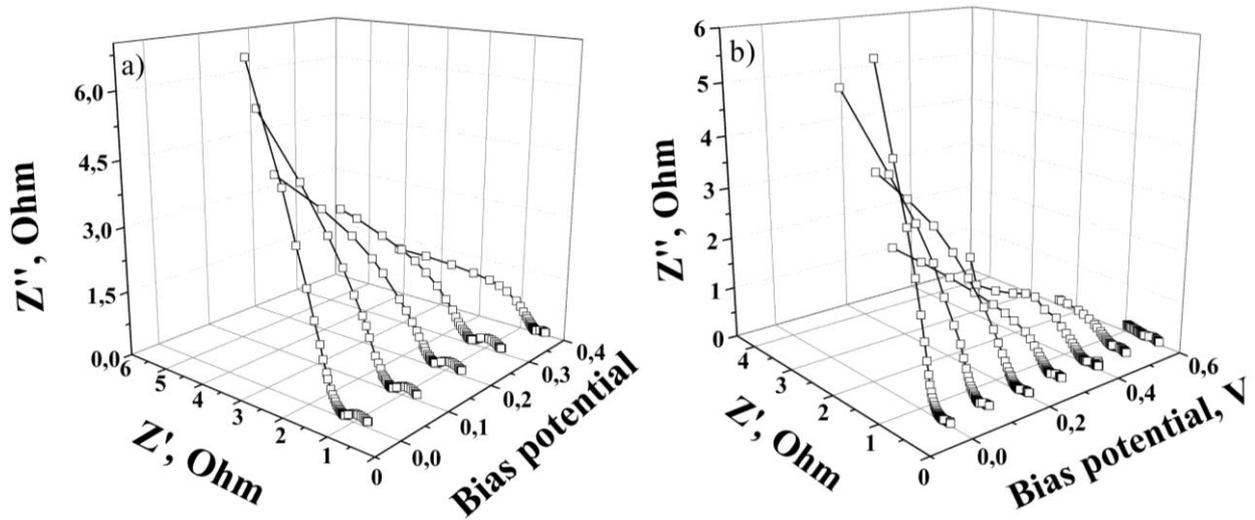

Fig. 2. The Nyquist curves of the CT (a) and CA (b) systems in the positive potential area.

between the electrical conductivity and the porous structure of the composite and the properties of the electrolyte used and to obtain information on the efficiency of the joint functioning of the components of the system under research. The Nyquist diagrams of the investigated electrochemical systems of NCM/thermally expanded graphite (CT) and NCM/acetylene carbon black (CA) composites in an aqueous electrolyte of 6 M KOH are presented in Fig. 1.

The impedance spectra for both systems (Fig. 1) have a typical form of capacitive accumulation of electric charge at the composite/electrolyte interface, as evidenced by the fact that at low frequencies the angle of inclination of the diagrams tends to 90º. Moreover, from the IS, the internal electrical resistance (0.25 - 1.25 Ohms) is determined, which depends on the contact of the compounds and the electrolyte used. In the high-frequency region, IS in the form of a semicircle is present in all diagrams due to the limitations of ion transport (H^+, K^+) through the carbon matrix, as a result in additional resistance characterizing the redox reaction at the composite/electrolyte interface [11]. In the middle frequency range, the straight line at an angle close to 45°

is responsible for diffuse processes that occur in the pores of the carbon composite. In the low-frequency region, the apparent impedance component increases, indicating the dominance of the capacitive behavior of the composite/electrolyte system. The region of positive potentials is represented by the Nyquist diagrams in Fig. 2. The potential growth from 0 to 0.5 V is accompanied by the formation of two semicircles in the Nyquist plots. The semicircle in the high-frequency area is associated with the course of faraday processes based on OH⁻ groups. At the medium and low frequencies of IS, the linear dependence of the Nyquist diagrams at 0 - 0.2 V, passes into a semicircle at potentials (0.2 - 0.5 V), probably due to diffusion processes OH⁻ groups.

The total capacity of the electrochemical system (C) was calculated based on the values of the apparent resistance at a frequency of 100 mHz using the formula,

$$C = \frac{1}{2\pi \cdot f \cdot Z^{//}}$$ where f is the frequency and $Z^{//}$ is the

imaginary resistance (Fig. 3).

Figure 3 show that the specific capacity of the research systems depends on the magnitude of the

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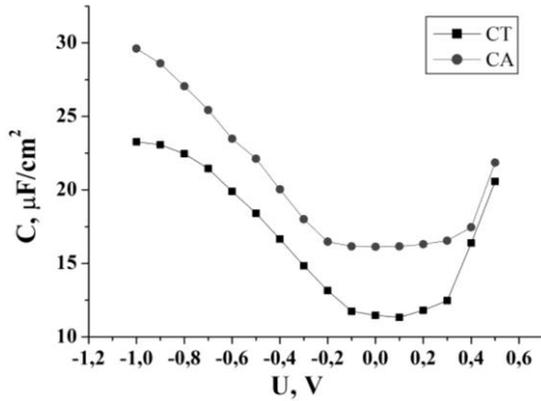
Fig. 3. The specific capacity of the investigated composites dependence on the applied potential.

applied potential. In this case, due to a change in the cell potential, the surface of the carbon material is polarized in the negative or positive direction, which leads to a change in the surface free charge and the formation of DEL with different signs of electrolyte ions. Therefore, in the range of potentials research (from -1 to 0.6 V) on a faraday voltage curve the minimum value of specific capacitance is observed, which corresponds to the zero charge potential in the absence of specific effects on the surface of the carbon electrode [13, 14]. In the obtained curves (Fig. 3), the minimum of the specific capacitance is shifted in the positive direction. Moreover, that shift is probably associated with the adsorption of molecular oxygen in the KOH electrolyte on the surface of the carbon material [14]. Considering that, depending on the conductive additive used, the specific capacity of the carbon material has different values, so at negative potentials the capacity of the CA system is $18 \div 29$ μF/cm^2, which is 25 - 30 % more suitable capacity for the CT system. Thus, for both systems, the specific capacitance decreases with increasing electrode potential and the minimum is 11.3 - 11.8 μF/cm^2 and 16.1 - 16.5 μF/cm^2 for CT and CA systems, respectively, in the range from -0.1 to 0.3 V. In this case, accumulation of charge in the region of zero potential occurs arbitrarily by the electrostatic mechanism. The further increase in potential in the positive region leads to an increase in specific capacitance due to the passage of an oxidative reaction at the electrode/electrolyte interface with the evolution of oxygen. In the general case, the dependence of the specific capacity of the carbon material/electrolyte system on the applied potential can be considered close to parabolic. Similar are the parabolic dependences of the specific capacitance on the applied potential for pure highly oriented graphite and pyrolytic graphite [15, 16], as well as for semiconductors in aqueous electrolytes [17]. It should be noted that a decrease in specific capacitance at positive potentials is characteristic of n-type semiconductors [16]. Assuming that the capacitive behavior of the research carbon systems is close to the behavior of semiconductors, the carbon electrode/ electrolyte interface can be described by the Mott-Schottky model. To determine the concentration of charge carriers and the flat-band potential, the dependence of the specific capacitance of

the spatial charge on the applied potential is used:

$$C_{sc} = \left(\frac{2\varepsilon\varepsilon_0 e^2 N}{kT} \right)^{1/2} \cosh\left[\frac{\left(U - U_{fb}\right)e}{2kT} \right], \qquad (1)$$

where N, U, and U_{fb} are the charge carrier concentration, the electrode potential, and the flat-band potential, respectively. The value of T is the absolute temperature and k is the Boltzmann constant. In the region of negative potentials for n-type semiconductors, an accumulation of electrons occurs on its surface, as a result of which the capacitance increases exponentially, which is confirmed by equation (1). It should be noted that this area is called the accumulation region for semiconductors system. The process of charge accumulation due to K$^+$ ions at the interface of carbon material/electrolyte in the region of negative potentials is similar. Therefore, increasing the potential in the positive region leads to a decrease in electrons in the near-surface region, which leads to a decrease in the specific capacitance. In the range of potentials of the depletion region, the specific capacity of the system does not change, and the parabolic dependence of the capacity passes into a straight line parallel to the axis of potentials. However, the next is the inversion region in which an increase in the positive potential leads to the dominance of holes in the semiconductor and the capacitance grows exponentially according to equation (1). Additionally, the capacity of the system carbon material/electrolyte increases due to the reaction of oxygen evolution (Fig. 3). Figure 4 shows the dependence of the inverse square of the specific capacity of the composite/electrolyte system on the applied potential. According to the dependencies data, it is possible to establish the type of conductivity of the system under research.

The semiconductor properties of the n-type carbon material/electrolyte systems are confirmed by the linearity of the Mott-Schottky graphs in the potential range (from -0.4 to 0 V) (Fig. 4a) and their positive slope. The behavior characteristic of n-type semiconductors is associated with the presence of valence electrons in thermally expanded graphite and acetylene black, which are part of the carbon composite, as materials for which sp^2 bonds are characteristic. The value of C^{-2} in the potential range from 0.2 to -1 V (the region of the DEL capacitance) is maximum for the CT composite and lower for CA (Fig. 4, b), which indicates a higher capacity of the CA composite. According to the Mott-Schottky theory, for n-type semiconductors, capacitance and potential are related by the equation:

$$\frac{1}{C^2} = \left(\frac{2}{\varepsilon\varepsilon_0 A^2 e N_D} \right)\left[U - \left(U_{fb} + \frac{kT}{e} \right) \right], \qquad (2)$$

where A is the surface area of the investigated composite, N_D is the concentration of charge carriers.

The charge carrier concentration (N_D) and the flat-band potential (U_{fb}) for the systems under research were calculated from the slope of the Mott-Schottky graph and the intersection of the approach curve with the potential axis (Fig. 4b, Table 2).

The data obtained based on carbon composites formed with the addition of acetylene black have a carrier concentration of 2.5 times higher than the
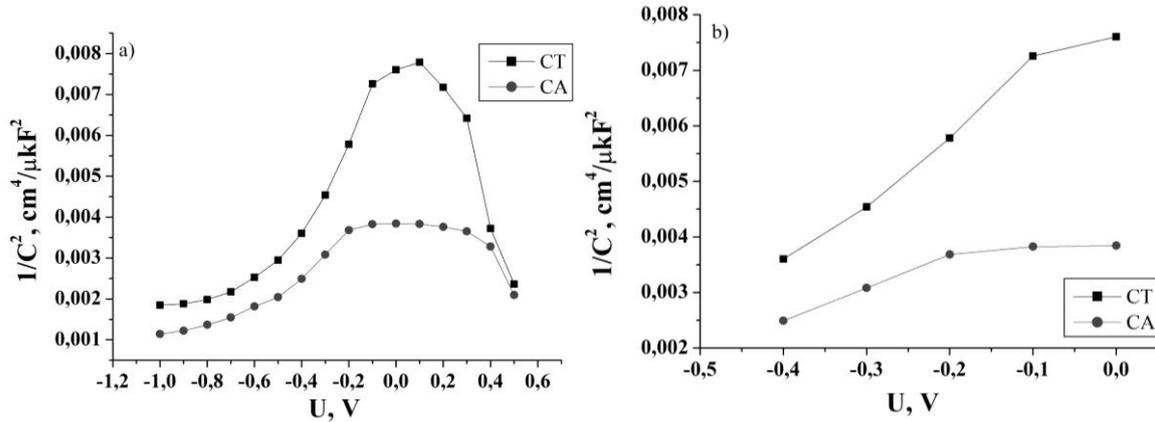
Fig. 4. The Mott-Schottky plots of the NCM/electrolyte system, potential range from -0.4 to 0 V a) and from -1 to -0.6 V b).

Table 2

The characteristics of carbon materials

Composite	N_D, cm^{-3}	U_{fb}, V	C_{SC}, μkF/cm^3	$D(E_f)$ eV^{-1}cm^{-3}
CT	$3.51 \cdot 10^{21}$	-0.635	13.15	$0.32 \cdot 10^{23}$
CA	$8.96 \cdot 10^{21}$	-0.825	16.48	$1.87 \cdot 10^{23}$

corresponding composites formed from thermally expanded graphite. The flat-band potential for the CT composite is (-0.635 V) and indicates a lower over potential compared to the CA composite (-0.825) for the charge transfer reaction. To determine the density of states and the capacity of the space charge region, the specific capacity of the NCM/electrolyte electrochemical system is presented as the sum of series-connected capacities: C_{SC} is the capacity of the space charge region in a solid; C_H is the capacity of the dense part of the DEL (Helmholtz layer) C_G is the capacity of the diffuse part of the DEL (Gui-Chapman layer). The total capacity of the system is determined from the following expression:

$$\frac{1}{C} = \frac{1}{C_{SC}} + \frac{1}{C_H} + \frac{1}{C_G}.$$

The diffuse part of the DEL can extend to 1–10 nm depend on the electrolyte concentration and practically disappears at a concentration exceed 10^{20} cm^{-3} [18]. Moreover, in concentrated electrolyte solutions used in practice $C_G \gg C_H$, and therefore the capacity of the Gouy-Chapman layer can be ignore. Therefore, the capacity of the space charge can be calculated using the

equation: $\frac{1}{C} = \frac{1}{C_{SC}} + \frac{1}{C_H}$. To determine the capacity

of the space charge, it was assumed that the capacity of the DEL for KOH is 20 μF/cm^2 when using an aqueous solution of KOH as the electrolyte and size of hydrated K$^+$ ions is equal to 0.53 nm [19]. The density of states is calculated based on the equation: $C_{SC} = \sqrt{\varepsilon\varepsilon_0 D(E_F)} \cdot e$

where ε, ε_0 and e are the dielectric constant, electric constant and electron charge respectively (Table 2). It was determine that for CA composites, the specific capacitance and density of states is higher than in CT

composites, probably due to the fact that acetylene black has a higher concentration of free charge carriers (Table 2).

Conclusions

It was determine that the maximum specific capacity of electrode materials formed based on composites NCM/TEG and NCM/ACB are 23 and 30 μF/cm^2 respectively. Moreover, the electrical properties of the composite material in a 6 M aqueous KOH electrolyte were investigated. Furthermore, the potentials of flat-band potential of -0.635 V and -0.825 V for materials CT and CA, respectively, were determined using the Mott-Schottky model. On the one hand, the concentration of charge carriers is $3.5 \cdot 10^{21}$ cm^{-3} for the composite based on TRG. On the other hand, the concentration of charge carriers increases 2.5 times based on the acetylene carbon black applications.

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Електричні властивості композитів на основі нанопористого вуглецевого матеріалу

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У роботі досліджено морфологічні та електричні властивості композиту нанопористий вуглецевий матеріал/терморозширений графіт або ацетиленова сажа. Нанопористий вуглецевий матеріал отриманий із рослинної сировини шляхом її термохімічної активації з використанням гідроксиду Калію. Методом імпедансної спектроскопії визначено залежність питомої ємності електрохімічної системи нанопористий вуглець/електроліт від прикладеного потенціалу. Використовуючи модель Mott-Schottky визначено концентрацію носіїв заряду, густину станів та потенціал плоских зон досліджуваної системи.

Ключові слова: модель Мотта-Шотткі, нонопористий вуглецевий матеріал, потенціал плоских зон.