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B. P. Bakhmatyuk, Cand. Sc. (Chem.), I. Ya. Dupliak, Ya. M. Voytovich Lviv Polytechnic National University, Lviv, Ukraine

IMPROVED PERFORMANCE ELECTRODE BASED ON ACTIVATED CARBON FOR ELECTROCHEMICAL ENERGY SOURCES

Б. П. Бахматюк, канд. хім. наук, І. Я. Дупляк, Я. М. Войтович Національний університет "Львівська політехніка", м. Львів, Україна

ЕЛЕКТРОД З ПОКРАЩЕНОЮ ЕФЕКТИВНІСТЮ НА ОСНОВІ АКТИВОВАНОГО ВУГЛЕЦЮ ДЛЯ ЕЛЕКТРОХІМІЧНИХ ДЖЕРЕЛ ЖИВЛЕННЯ

Purpose. To investigate the effectiveness of the commercial activated carbon material "Norit DLC Super 30" as an electrode for electrochemical energy sources in 25 % aqueous solution of ZnBr₂.

Methodology. For the research standard electrochemical methods of galvanostatic charge-discharge and cyclic voltammetry was used.

Findings. Theoretical adsorption isotherms of bromine and depending of specific pseudocapacity on the fractional surface coverage of bromine on the nanoporous activated carbon material "Norit DLC Super 30" was built. Using data of galvanostatic charge-discharge at a current density from 5 to 50 mA × cm⁻² the experimental adsorption isotherms and depending of specific pseudocapacity on the fractional surface coverage was received. The comparison of theoretical and experimental dependences shows that the investigated process is the process of electrosorption according to Frumkin model with parameter of interatomic interactions in adsorption layer (g = 0.5). The Ragone depending of the electrode in the system of electrochemical energy source was studied. The increase of specific power (P) from 1.1 to $9.6W \times g^{-1}$ leads to a slight decrease of specific energy (W) from 2251 to $2056J \times g^{-1}$. The obtained experimental value of W is equal to 84 % of the theoretical value of W_{Theor} .

Originality. The effectiveness of the electrode based on commercial activated carbon material "Norit DLC Super 30" in the system of electrochemical energy source and the mechanism of bromine electrosorption process on the surface of microporous activated carbon material was studied for the first time.

Practical value. The obtained high values of specific power, energy, capacity and electrode performance based on commercial activated carbon material "Norit DLC Super 30" can be considered as a promising positive electrode for electrochemical energy source.

Keywords: Frumkin model, adsorption isotherm, Ragone plot

Introduction. Carbon materials, due to environmental friendliness, low cost, high corrosion resistance, good electrical conductivity, and a highly porous structure, remain the most attractive and accessible materials for electric double layer capacitors [1]. Also, they are used for gasification, as it is shown in [2], the experiment parameters of mathematical design of underground gasgenerator and process of coal gasification were corrected for the borehole underground coal gasification station which is described in [3]. Alongside with oxides and nitrides of metals, conductive polymers. ACMs are used for electrosorption of hydrogen and iodine in asymmetric systems of electrochemical supercapacitors [4]. In [4] high values of C and W for the process of electrosorption of iodine in microporous ACM were obtained. The influence of ACM nanocomposites with bromine on the increase

in energy density of a supercapacitor in non-aqueous electrolyte is investigated in the work [5]. Zinc-bromine batteries (ZBB) are well known. The Br_2/Br^- redox couple is employed in the positive electrode of ZBB based on the following reaction [6]

$$2Br^- \leftrightarrow Br_2 + 2e^- (E^0 = 1.07 \text{ V vs. SHE}).$$

And the Zn^{2+}/Zn redox couple is employed in the negative electrode of ZBB based on the following reaction

$$Zn^{2+} + 2e^- \leftrightarrow Zn \ (E^0 = -0.76 \text{ V vs. SHE}).$$

In [7], it is shown that the addition of microporous ACM to positive electrode of ZBB increases *i* to 20 mAcm⁻² (up to 40 mAcm⁻² in [6]), due to the adsorption of bromine in ACM micropores. In [6], the determined steps of the Br⁻ oxidation reaction and Br₂ reduction reaction are proposed as follows:

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Br-oxidation reaction

$$Br^- \rightarrow Br_{ads} + e^-;$$
 (1)

Br₂ reduction reaction

$$Br_2 + e^- \rightarrow Br_{ads} + Br^-$$

Analysis of the literature data shows prospects of research process of bromine electrosorption in micropores of ACM. The influence of electrosorption processes on specific capacitance and specific energy of supercapacitors is studied in [1]. In the work [4], the process of electrosorption of iodine at a surface of micropores of ACM is investigated according to Langmuir model. According to the model, which does not take into account interatomic interaction in the adsorptional layer (g = 0), the Langmuir isotherm was presented by the well-known relation from [4]

$$\theta_{\rm I}/1 - \theta_{\rm I} = (Kc_{\rm I}) \times \exp\left\{(EF)/(RT)\right\}, \qquad (2)$$

here $0 < \theta < 1$; *K* is the constant of adsorptional equilibrium; $c_{1^{-}}$ is the concentration of iodine ions in the solution; *E* is the electrode potential; *F* is the Faraday constant (hereinafter); *R* is the gas constant (hereinafter); *T* is the temperature is *K* (hereinafter). *E* can be presented in the following form

and

$$E = E^{0} + (RT)/F \times \ln (\theta/1 - \theta),$$

$$E - E^0 = (RT)/F \times \ln(\theta/1 - \theta),$$

here E^0 is the equilibrium electrode potential. In the work [4], the well-known formula was used

$$C_P = (q_{\rm I}F)/(RT) \times \{\theta_{\rm I}(1-\theta_{\rm I}),\tag{3}$$

which indicate the maximum of the pseudocapacitance at $\theta = 0.5$, and the electrode potential of the maximum is considered as the E^0 ; q_1 is maximal value of NS commercial ACM specific surface charge ($\theta =$ = 0.99) by atoms of iodine.

The aim of this study is to investigate the process of electrosorption of bromine in microporous NS by means of the known physical adsorption model and to study the possibility of obtaining the maximum practical values of C, W, P per unit mass of ACM in EES system.

Materials and methods. The "Norit DLC Super 30" commercial ACM, ZnBr_2 (99 %, Aldrich), and Zn foil (99.999 %, Aldrich) were used for our experiments. NS commercial ACM was microporous activated carbon with highly developed total surface area (TSA BET = $S_{\text{micro}} + S_{\text{mezo}} = 1540 \text{ m}^2\text{g}^{-1} + 60 \text{ m}^2\text{g}^{-1} = 1600 \text{ m}^2\text{g}^{-1}$) [8]. The total pore volume of this sample was of 0.6 cm³ g⁻¹ and the average pores size was 1.24 nm [9]. All the chemicals were of analytical grade, and they were used without further purification.

For investigations, there were used film-like electrodes of the active mass (m_a) of 14 mg (NS1) and the thickness (d) of 0.2 mm (S was 1.0 cm²) and of $m_a =$ = 7 mg (S = 1.0 cm², d = 0.1 mm: NS2) with the added binder of 5 wt. % of Teflon and 10 wt % of acetylene carbon black. The electrodes had been assembled by

pressing them to graphite foil. Electrochemical investigations were carried out in a two-electrode and in a three-electrode glass cells with a Zn foil counter electrode (1 cm²). As a reference electrode, Ag|AgCl was used. As the electrolyte, aqueous solution of 25 % ZnBr₂ was used. All measurements were taken at room temperature. The electrochemical properties of the EES were investigated by cyclic voltammetry (CV), by galvanostatic charge-discharge (GCD), the investigations have been carried out with a help of AUTOLAB measuring complex made in Netherlands by "ECO CHEMIE" in combination with GPES computer program. Theoretical and experimental of adsorption isotherms are presented in the form of the relation (2) for the electrode polarization $\Delta E = E - E^0$. Experimental values of C_P were determined with the use of discharge voltage steps of dU = 0.01 V and corresponding to them differentials $d\theta_{\rm Br}$ according to the following known formula

$$C_P = q_{\rm Br}(d\theta_{\rm Br}/dU),$$

here q_{Br} is the maximal value of NS commercial ACM specific surface charge with bromine atoms ($\theta_{Br} = 0.99$).

C, *W*, *P*, θ_{Br} , η of GCD were determined according to the following known formulas

$$C_{c} = I_{c} \times t_{c}(m_{a})^{-1}(a); C_{d} = I_{d} \times t_{d}(m_{a})^{-1}(b);$$

$$\theta_{Br} = C_{d} \times (C_{Br, \max})^{-1}(c); \qquad (4)$$

$$W = i \int_{t_{1}}^{t_{2}} U(t) dt (a); P = W t_{d}^{-1}(b);$$

$$\eta = (C_{d}) \times (C_{c})^{-1} \times 100 \% (c). \qquad (5)$$

Here C_c , C_d are the specific capacities of charge and discharge, respectively; I_d , I_c are the currents of charge and discharge, respectively; t_c , t_d are the times of charge and discharge, respectively; $t_{d,s}$, $t_{d,f}$ are the times of start and finish of discharge, respectively; $C_{\text{Br,max}}$ is the maximal value of specific gravimetric charge of NS commercial ACM with bromine atoms ($\theta_{\text{Br}} = 0.99$); $U_{d,\text{max}}$ is the maximal value of the discharge voltage; $U_{d,\text{min}}$ is the minimal value of the discharge voltage; $dU = U_{d,\text{max}} - U_{c,\text{min}}$ denotes the discharge voltage range; m_a is the mass of NS commercial ACM.

Results and discussion. NS was tested in twoelectrode system using aqueous $ZnBr_2$ electrolyte, which is the prototype of a HC in charged state

$$Zn|25\% ZnBr_2|C^*Br.$$
(6)

This corresponds to the current generating process

$$C^*Br + 0.5Zn = C^* + Br^- + 0.5Zn^{2+}.$$
 (7)

The process which proceeds during anode polarization of NS in 25 % $ZnBr_2$ is considered in this work as a process of electrosorption of Br⁻ (transition of Br⁻ into adatomic state) according to the schemes from [1, 4, 6]

$$C^*Br + e^- = C^* + Br^-,$$
 (8)

where C^* is the surface of the carbon material, θ is fractional coverage by Br at C^* (0 < θ < 1). Some

amount of Br_3^{-1} is being formed during the charging of the electrode. Taking into account the ions size (Br^{-1} is smaller than Br_3^{-1}), this makes the penetration of Br_3^{-1} into the carbon network more poor. It is clear that the micropores and small mesopores of carbon are the most adapted for electrosorption according to equation (8) because, besides electrostatic attraction of ions, the faradaic reactions related with bromide/bromine electron transfer are involved in the charging process (8). On the basis of the aforesaid, the influence of Br_3^- is not examined. They, probability, are located in macropores of NS commercial ACM.

As is shown in Fig. 1, *a*, galvanostatic charge (GC) to 1.93 V (1.08 V vs Ag/AgCl) of NS1 electrode at the i_c of 14 mA cm⁻² in EES system ensures galvanostatic discharge (GD) at the i_d of 7 mA cm⁻² with the high value of C_d (equal to 083 Cg⁻¹) in the voltage window from 1.71 V (0.9V) to 1.05V (0.27 V) with wide polarization ($\Delta E = 0.66V$) of electrode. The wide discharge ΔE , which amounts to 0.66 V at sufficiently great value of C_d (equal to 1083 Cg⁻¹), considerably reduces W. The galvanostatic cycle (GC) under the given condi-

tions ($i_c = 14 \text{ MAcm}^{-2}$, $i_d = 7 \text{ MAcm}^{-2}$) of Zn electrode (Fig. 1, b) shows a ΔE close to 0. Identical general appearance of CVs of NS2 electrode, which are plotted according to two-electrode and three-electrode measuring circuits, can be easily seen in Fig. 1, c and in Fig. 1, d. With this, the CVs have equal anode maxima ($i_a = 47.9 \text{ mAcm}^{-2}$) and close cathode minima ($i_c =$ = 40 mAcm $^{-2}$, Fig. 1, c, and $i_c = 33.6 \text{ mAcm}^{-2}$, (Fig. 1, d). The obtained galvanostatic data of NS1 and Zn electrodes and data of CVs of NS2 electrode (Fig. 1, a-d) indicate that the polarization (ΔU) of the whole HC system is equal to the ΔE of NS electrode.

The use of half-thickness NS2 electrodes essentially changes the GD (Fig. 2, *a*). GCD cycles (Fig. 2, *a*) at i_d from the range of 5–50 mA cm⁻² were recorded in the system of EES prototype. The galvanostatic curves have their plateau of the discharge with moderate-valued ΔE . With this, the increase in i is accompanied by the increase in θ of the electrode by bromine atoms from 0.8 to 0.9, and the ΔE increases from 0.15 to 0.31 V.



Fig. 1. ACM "Norit DLC Super 30" and Zn electrodes in the system of EES: galvanostatic cycles ACM "Norit DLC Super 30" (a), Zn (b) at 14 mAcm⁻² and GDs at 7 mAcm⁻² (with the use of 3-electrode measurement); CVs of NS "Norit DLC Super 30" recorded for scan rate 10⁻³ V s⁻¹ with the use of 2-electrode measurement (c) and of 3-electrode measurement (d)

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Fig. 2. ACM "Norit DLC Super 30" electrode in the system of EES: (a) GD at $i_d = 5 \text{ mAcm}^{-2}(1)$, $i_d = 20 \text{ mAcm}^{-2}(2)$, $i_d = 30 \text{ mAcm}^{-2}(3)$, $i_d = 45 \text{ mAcm}^{-2}(4)$, $i_d = 50 \text{ mAcm}^{-2}(5)$; galvanostatic cycles at $i_c = i_d = 40 \text{ mAcm}^{-2}$: 24 GCs (b) and 24th cycle (c); (d) Ragone plot (1) and dependence of C_d on to the P (2)

(9)

Fig. 2, b shows 24 identical GSs at i equal to 40 mA cm⁻². Fig. 2, c shows the typical 24^{th} -GCD profiles of the NS2 electrode in a voltage window of 1.02-2.07 V and at i equal to 40 mA cm⁻². The measured value of electromotive force (EMF = 1.77 V) in the EES system (6) is less than 1.83 V of the galvanic couple $Zn|Br_2$. The plateau on the GSs correspond to high-capacity charge and discharge according to the equation (4, a-c). Discharge (Fig. 2, c) on the plateau from 1.58 to 1.42 V is characterized by the values of C_d = 1488 Cg⁻¹, W = 2245 J g⁻¹, P = 8.6 Wg⁻¹, and by the η of 91 %. To calculate the maximal value of NS commercial ACM specific surface charge ($\theta = 0.99$) according to the formulas $q_{\rm Br} = (s_{\rm Br})^{-1}e^{-} = (2 \times 0.196 \text{ nm})^{-2} \times 1.6 \times 10^{-19} \text{ C} = 1.04 \text{ Cm}^{-2} \text{ and } C_{\rm Br,max} = q_{\rm Br}S_{\rm total} = 1.04 \text{ Cm}^{-2} \times 1600 \text{ m}^2\text{g}^{-1} = 1664 \text{ Cg}^{-1}$, the well know value of bromine ion radius (r_{Br}- equals 0.196 nm) was used. Formula (1) for electrosorption of bromine can be presented in the following form

and

$$E - E^0 = RT/F \times \ln(\theta_{\rm Br}/1 - \theta_{\rm Br}).$$

 $E = E^{0} + RT/F \times \ln(\theta_{\rm Br}/1 - \theta_{\rm Br}),$

By means the use of the formula (9) and by substituting $q_{\rm Br}$ into the formula (3), the theoretical adsorption isotherm (TAI) and theoretical dependence of C_P on θ were plotted.

The theoretical specific pseudocapacitance has its maximum value of 10.1 Fm⁻² at $\theta = 0.5$ (Fig. 3, curve 4). Comparison of the experimental value of C_d = = 1488 Cg⁻¹ with maximum theoretical value of $C_{\text{Br,max}} = 1664 \text{ Cg}^{-1}$ ($\theta = 0.99$) gives a high practical value of fractional surface coverage of $\theta = 0.89$. This is well illustrated by the experimental desorption isotherm (EDI), Fig. 3, a, curve 3. And the plotted according to GR ($i = 40 \text{ mA cm}^{-2}$) dependence of the $C_{P,d}$ on the θ shows the maximum of $C_{P,d} = 8.4 \text{ Fm}^{-2}$ at $\theta = 0.63$ (Fig. 3, curve 6), which is less than the maximum $C_{P,\text{theor}} = 10.1 \text{ Fm}^{-2}$ at $\theta = 0.5$ (Fig. 3, curve 4) of the theoretical dependence drawn according to the formula (3). According to the Frumkin model of physical adsorption $(g \neq 0)$, the equations (2, 3, 9) take the form

$$\theta_{\rm Br}/(1-\theta_{\rm Br}) = (\mathbf{K} \times \mathbf{c}_{\rm Br}) \times \exp(-g\theta_{\rm Br}) \times \exp(EF/RT);$$

$$E - E^0 = (RT/F) \ln(\theta_{\rm Br}/1-\theta_{\rm Br}) + (RT/F)g\theta_{\rm Br}; (10)$$

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Fig. 3. ACM "Norit DLC Super 30" electrode in the system of EES:

(a) TAIs at g = 0 (1), g = 0.5 (2), and EAI g = 0.5 (3); theoretical dependences of the C_P on θ : g = 0 (4), g = 0.5 (5) and experimental dependence of the $C_{P,d}$ on θ at g = 0.5 (6); (b) theoretical discharge (g = 0.5) (curve 1) and and experimental GD (2) at 40 mAcm⁻²

$$C_P = q_{\rm Br} F \times (RT)^{-1} \times \theta_{\rm Br} (1 - \theta_{\rm Br}) \times \\ \times \{ (1 + g \theta_{\rm Br} (1 - \theta_{\rm Br}) \}^{-1}.$$
(11)

Substituting the experimental values into the formula (11), the practical value of g = 0.5 was calculated. The parameters of g which were calculated at other i_d have values from -1 to 0.6. Positive and negatives values of the parameter g are the cause of the changes ΔE of the electrode according (10). Small deviations between TAIs at g = 0 and g = 0.5 can be seen in Fig. 3. And more considerable deviations take place between EAI and theoretical ones. Fig. 3, b shows theoretical and experimental discharge curves (g = 0.5). Theoretical discharge curve was plotted with the use of the measured value of electromotive force of the galvanic couple (6) (EMF = 1.77 V for θ = 0.9) and ΔE_{Theor} = = 0.27 V (Fig. 3, *a*, curve 2). Theoretical specific energy (W_{Theor}) and the experimental one W were calculated with the use of data of Fig. 3, b and the use of the formula (5, *a*). They are of the values of 2682 Jg^{-1} and 2253 Jg^{-1} , respectively. The obtained experimental value of W is equal to 84 % of the theoretical value of W_{Theor} . The W_{Theor} which were obtained at other i_d have values from 2680 to 2715 Jg^{-1} . The Ragone plot (Fig. 2, d, curve 1), which is plotted according to the data of GD (i from 5 to 50 mAcm⁻²) shows a good high - W characteristics of NS commercial ACM in an EES system. The increase of P from 1.1 to 9.6 Wg^{-1} leads to a slight decrease of W from 2251 to 2056 Jg⁻¹. And the increase of P from 1.1 to 9.6 Wg⁻¹ leads to a increase of C_d from 1336 Cg⁻¹ (371 mAhg⁻¹) to 1512 Cg^{-1} (420 mAhg⁻¹ (Fig. 2, *d*, curve 2).

Conclusion. The process of electrosorption of bromine by NS commercial ACM surface ensures a high capacity discharge of 1336–1512 Cg⁻¹ range at i_d from 5 to 50 mA cm⁻². Comparison of experimental desorption isotherm and dependence of the specific capacitance on the fractional surface coating of NS

commercial ACM with the corresponding theoretical dependencies (g = 0, g = 0.5) indicates the mechanism of bromine physical adsorption in accordance with Frumkin's model. The mechanism of bromine adsorption ensures the high-valued fractional coverage from 0.8 to 0.9 of NS commercial ACM surface and 77–84 % discharge of the values W_{Theor} . All of this gives us the reason to consider the investigated activated material in bromide electrolytes as a prospective positive electrode for EES constructions.

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Мета. Дослідити ефективність роботи комерційного активованого вуглецевого матеріалу "Norit DLC Super 30" як електрода електрохімічного джерела енергії у 25 % водному розчині ZnBr₂.

Методика. Для досліджень використані стандартні електрохімічні методики гальваностатичного заряду-розряду та циклічної вольтамперометрії.

Результати. Побудовані теоретичні ізотерми адсорбції брому та залежності питомої псевдоємності від фракційного покриття брому нанопористої поверхні активованого вуглецевого матеріалу "Norit DLC Super 30". Використовуючи дані гальваностатичного заряду-розряду при густинах струму від 5 до 50 м $A \times cm^{-2}$, отримані експериментальні ізотерми адсорбції та залежності питомої псевдоємності від фракційного покриття поверхні. Зроблене порівняння теоретичних і експериментальних залежностей показує, що досліджений процес відповідає моделі електросорбції за Фрумкіним з параметром міжатомної взаємодії в адсорбційному шарі (g = 0.5). Проведено аналіз залежності Рагоні дослідженого елетрода в системі електрохімічного джерела енергії. Збільшення питомої потужності з 1,1 до 9,6 Вт \times г⁻¹ призводить до незначного зниження питомої енергії з 2251 до 2056 Дж \times г⁻¹. Отримане експериментальне значення питомої енергії складає 84 % від її теоретичного значення.

Наукова новизна. Уперше вивчена ефективність роботи електрода на основі комерційного активованого вуглецевого матеріалу "Norit DLC Super 30" в системі електрохімічного джерела енергії та механізму процесу електросорбції брому на мікропористій поверхні активованого вуглецевого матеріалу.

Практична значимість. Отримані високі значення питомих потужнісних, енергетичних, ємнісних показників електрода на основі комерційного активованого вуглецевого матеріалу "Norit DLC Super 30", що дозволяють розглядати його перспективним додатнім електродом для електрохімічного джерела енергії.

Ключові слова: модель Фрумкіна, ізотерма адсорбції, залежність Рагоні

Цель. Исследовать эффективность работы коммерческого активированного углеродного материала "Norit DLC Super 30" в качестве электрода электрохимического источника энергии в 25 % водном растворе ZnBr₂.

Методика. Для исследований использованы стандартные электрохимические методики гальваностатического заряда-разряда и циклической вольтамперометрии.

Результаты. Полученные теоретические изотермы адсорбции брома и зависимости удельной псевдоемкости от фракционного покрытия брома нанопористой поверхности активированного углеродного материала "Norit DLC Super 30". Используя данные гальваностатического зарядаразряда при плотностях тока от 5 до 50 м $A \times cm^{-2}$, получены экспериментальные изотермы адсорбции и зависимости удельной псевдоемкости от фракционного покрытия поверхности. Сделанное сравнение теоретических и экспериментальных зависимостей показывает, что исследованный процесс соответствует модели электросорбции за Фрумкиным с параметром межатомного взаимодействия в адсорбционном слое (g = 0.5). Проведен анализ зависимости Рагони исследованного электрода в системе электрохимического источника энергии. Увеличение удельной мощности с 1,1 до 9,6 Вт × г⁻¹ приводит к незначительному снижению удельной энергии с 2251 до 2056 Дж \times г⁻¹. Полученное экспериментальное значение удельной энергии составляет 84 % от её теоретического значения.

Научная новизна. Впервые изучена эффективность работы электрода на основе коммерческого активированного углеродного материала "Norit DLC Super 30" в системе электрохимического источника энергии и механизма процесса електросорбции брома на микропористой поверхности активированного углеродного материала.

Практическая значимость. Получены высокие значения удельных мощностных, энергетических, емкостных показателей электрода на основе коммерческого активированного углеродного материала "Norit DLC Super 30", что позволяют рассматривать его перспективным положительным электродом для электрохимического источника энергии.

Ключевые слова: модель Фрумкина, изотерма адсорбции, зависимость Рагони

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