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# Analysis of the parameters of supercapacitors containing polypyrrole and its derivatives

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## ABSTRACT

**Purpose:** Investigation of some parameters of energy storage devices (so-called supercapacitors) in which the structures of selected conductive polymers were implemented. Researchers were interested in the relationship between the parameters of the supercapacitor and the molecular structure of the conductive polymer used as the electrode material.

**Design/methodology/approach:** The polypyrrole and its derivatives were produced by an electropolymerization process performed with cyclic voltammetry. During the research, polymer supercapacitors were created containing collectors made of ITO plates. Measurement of device parameters using cyclic voltammetry and chronopotentiometry. In addition, the devices use polymer electrolytes based on poly (methyl methacrylate) (PMMA).

**Findings:** Devices containing polypyrrole have the best electrochemical parameters, while supercapacitors containing poly (phenylpyrrole) have the lowest parameters. This parameter is due to the high hindrance in the poly(phenylpyrrole) molecule in the form of an aromatic ring.

**Research limitations/implications:** The most significant limitation of the devices is their durability due to the low strength of the conductive layer on ITO plates. This layer was easily degraded with too many test cycles.

**Practical implications:** It was confirmed that polypyrrole and its derivatives could be used as electrode materials in energy storage devices.

**Originality/value:** One of the few studies that allow the evaluation of the molecular structure of polypyrrole and its derivatives as electrode material in symmetrical supercapacitors.

**Keywords:** Polymersupercapacitors, Conductive polymers, Cyclic voltammetry, Chronopotentiometry

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## MATERIALS



## 1. Introduction

In the future, renewable energy sources, such as wind, sun, and water, will increasingly play an indispensable role in the functioning of the energy sector. An essential problem of renewable energy sources is their variable activity because solar power plants generate the most electricity on a sunny day. In contrast, wind power plants only during stronger gusts of wind. To distribute energy from renewable sources efficiently, energy storage is needed, which must be able to accumulate electricity during low energy demand, and at the same time be able to generate electricity in peak energy demand [1].

Supercapacitors, also known as ultracapacitors, play an increasingly important role in the electrochemical energy storage devices market. A higher power density than traditional batteries characterizes devices. They have a power density of up to 100 kW/kg, while conventional lead-acid batteries are only 450 W/kg. Ultracapacitors are also characterized by an increased cycle speed (charge-discharge), a massive number of work cycles, and a low voltage of current between 2 and 3V [2].

High-power density systems (supercapacitors) are used in recuperative braking systems in electric vehicles. They were initially used in traction units, but with the development of electromobility, they have found applications in electric cars and buses. The ideal material for the supercapacitor electrode should have low electrical resistance, high resistance to mechanical impact, and a well-developed surface [3]. Conducting polymers have them in the context of the desired energetic properties. These substances form an excellent material that functions as an electrode. Some conjugated polymers have such good electrochemical and strength properties that they are used as single-component electrodes. To obtain better device parameters, composites of conductive polymers and carbon nanostructures are used as electrode materials. Such carbon structures can be produced in plasma reactors [4]. In addition, it is possible to improve the properties of composite materials containing carbon materials by ultrasonic radiation. The research by scientists from Lviv Polytechnic National University has shown the possibility of the interaction of radiation on the phase boundary of the carbon material and the electrolyte [5].

The first study discovered electric conduction through polymers in the USA in 1977. First, the research shows the process of polyacetylene oxidation with iodine vapor. Then, during the oxidation process (so-called doping), the modified polymer gained electrically conductive properties. At the same time, the electric conductivity of polyacetylene increased to 38 S/cm, which was an outstanding

achievement because pure polyacetylene has a conductivity value of  $10^{-9}$  S/cm. The first research on conductive polymers such as polyacetylene, polypyrrole, polythiophene, and polyaniline won the Nobel Prize in Chemistry in 2000. The laureates of this award were Alan J. Heeger, Alan G. MacDiarmid, and Hideki Shirakawa [6].

The analysis of the available literature showed that conductive polymers are also used in photovoltaic systems. Such systems use polymers such as poly(3,4-ethylene-1,4-dioxythiophenolthiophene (PEDOT) and polypyrrole [7-10].

Electroactive polymers are also used as active materials in temperature sensors in tribological systems. For example, professor Sławomir Zimowski from the AGH University of Science and Technology, in his article published in the journal "Tribologia," presented the possibility of diagnosing the operation of the tribological system based on temperature and mechanical pressure analysis [11]. At that time, a polyimide composite layer was used as the temperature sensor in the tests, in which the control of the composite resistance caused by temperature change allowed us to estimate the correctness of the system operation [12].

In the scientific literature, polypyrrole (PP) was first mentioned in 1919. At that time, the Italian chemists A. Angeli and A. Pieroni carried out a synthesis of the so-called pyrrole black. Its structure includes a heterocyclic pyrrole system. This monomer consists of a nitrogen atom and a heterocyclic system containing delocalized electrons. The poly (3-n-octyl pyrrole) has eight carbon-saturated hydrocarbon chain substituents. The third material is poly (phenylpyrrole). The phenyl group acts as the substituent in this polymer. The tested materials are used as electrode material in supercapacitors. Their electrically conductive properties allow the achievement of suitable parameters for devices in which these materials are used.

The pyrrole molecule and its derivatives undergo an electropolymerization reaction according to the standard mechanism [13]. First, the monomer is oxidized to a radical cation, and then in the addition reaction step, the two oxidized pyrrole structures form a dimer with two positive charges. The reaction of the addition of subsequent cation radicals continues until the macromolecule becomes insoluble in the system and the polymer is precipitated [14].

The heterocyclic polymer containing a nitrogen atom has also found its application in other supercapacitor systems. For example, a team of researchers from the School of Mechanical Engineering at the University of Shanghai for Science and Technology synthesized polypyrrole nanotubes by chemical polymerization using  $\text{FeCl}_3$  as an oxidant. Supercapacitor electrodes made of this material have excellent electrochemical performance and a specific capacitance of 281.2 F/g [15]. In addition, the team led by

Rizwan Ullah from the University of Peshawar in Pakistan developed unique composites of polypyrrole and gum arabic prepared by emulsion polymerization. The produced material achieved excellent electrochemical properties; supercapacitors containing polypyrrole had a specific capacity of 168 F/g, while systems with a polypyrrole/gum arabic composite reached a particular span of approximately 368 F/g [16].

Polyaniline is one of the best electrode materials in supercapacitor systems. However, the structure of its polymer chain disintegrates after the cell's charging and discharging cycle. One of the solutions to the above problems is a nanocomposite consisting of PANI inorganic metal oxides with excellent capacity and high stability. Such a nanocomposite was created by an international team of scientists from Algeria, Spain, and Germany led by Hafid Belhadj from the University of Mustapha Istanbul. The material was based on a combination of titanium dioxide (TiO<sub>2</sub>) and titanium carbide (TiC<sub>2</sub>) with polyaniline; such a composite achieved stability of 88% after 1500 cycles of charging and discharging the cell. The created nanocomposite is a potential electrode material in supercapacitors [17].

In the latest research, polymer electrolytes are used in energy storage devices. Such electrolytes have a solid state, which prevents environmental contamination during device failure. Most often, electrolytes consist of poly (methyl methacrylate) (PMMA) and poly (oxyethylene) (POE) [18].

Two research methods were used in the research - cyclic voltammetry and chronopotentiometry. Cyclic voltammetry (CV) is one of the electrochemical techniques carried out in a three-electrode system. A CV consists of registering a working electrode's current depending on the given electrode potential [19]. This technique was refined in 1958 by Polish chemists Zenon Kublik and Wiktor Kemula. It has found its application in the study of electrochemical processes, determination of the specific capacity of supercapacitors, carrying out electrochemical polymerization, determination of the electron stoichiometry of the reaction, and determination of the diffusion coefficient of the analyte.

On the other hand, the chronopotentiometry measurement determines the electric potential caused by the current flow between the electrodes (working electrode and reference electrode) at a given constant intensity. The results are obtained by examining the change of this potential as a function of time. This measuring technique is implemented with a three-electrode system [20]. In addition, a given potential caused by the flow of current through the system is monitored concerning the reference electrode.

In contrast, the flow of an electric charge of a constant intensity determines the increase in the value of the potential, which is caused by the decrease of the electrolyte

resistance and the charging of the working electrode. The result of testing the circuit with this technique is presented with the help of the so-called chronopotentiometry curve. Chronopotentiometry is a measurement method used to determine the electrode capacitance, ion-selective membrane resistance, and electrochemical parameters of energy storage devices [21].

## 2. Research materials

In this study, a group of supercapacitors was prepared. Three supercapacitors were developed containing selected polymers as electrode material. The devices use collectors made of ITO plates (glass plates covered with a conductive layer of a tin oxide containing an admixture of indium) by MALDI.

To carry out the research, electropolymerization of selected conjugated polymers was performed.

The electropolymerization process was performed by cyclic voltammetry using CHI 660 potentiostat and a three-electrode system. The polymers were applied to the working electrodes - ITO plates. In each of the prepared systems, the function of the auxiliary electrode (AUX) was performed by the platinum electrode. In contrast, the reference electrode (REF) was the silver electrode (the electrode made of metallic silver). The first stage of the electropolymerization process was to prepare an electrolyte solution containing the monomer to be tested. The electrolytes intended for electropolymerization contained a mixture of the monomers to be tested [pyrrole (Acros Organics, extra pure, 99%), 3-n-octyl pyrrole (Janssen Chimica, 99%) or phenylpyrrole (TCI, > 95%)] at a concentration of 0.005 M dissolved in acetonitrile (Acros, 0.99%) and a solution of tetrabutylammonium hexafluorophosphate (Bu<sub>4</sub>NPF<sub>6</sub>) (TCI, 0.98 %) at a concentration of 0.1 M in acetonitrile (Acros, 0.99%) in a ratio of 1: 1. The same scan rate of 0.1 V/s was used during each polymerization. The potential window range during the electropolymerization process was -0.2V to 1.4 V. The electropolymerization used in the research allowed the deposition of conjugated polymers on the tested plates. During this process, thin polymer films were formed on the electrode surface. This phenomenon was a key stage in producing the studied polymer supercapacitors.

The mass of polymer deposited on the electrode due to electropolymerization was calculated from the first law of Faraday's electrolysis. According to equation No. (1), the mass of the electrically conductive substance deposited on the working electrode (m) is directly proportional to the product of the electrochemical equivalent (k) and the electric charge that has flowed through the electrolyte in this system (Q).

$$m = k * Q \quad (1)$$

$m$  – a mass of the substance, g;

$k$  – the electrochemical equivalent, g/C;

$Q$  – electric charge, C.

Equation No. (2) presents the possibility of calculating the electrochemical equivalent ( $k$ ), which is the quotient of the molar mass of the electroactive component ( $M$ ) and the product of the number of electrons ( $n$ ) transferred in the electrode reaction and the Faraday constant ( $F$ ).

$$k = \frac{M}{n * F} \quad (2)$$

$k$  – the electrochemical equivalent, g/C;

$M$  – molar mass, g/mol;

$n$  – number of electrons, mol;

$F$  – Faraday's constant, 96500 C/mol.

Considering that during the electrode reaction of one mole of pyrrole, one electron is transferred, this value should be used when calculating the electrochemical equivalent for the response of the tested monomer and its derivatives.

The charge ( $Q$ ) that flows through the electrolyte in the system is represented by the integral equation of the current ( $I$ ) limited by the time range ( $t_1$ ) and ( $t_2$ ). This relationship is presented in formula No. (3). For the tests, the value of the charge that flowed through the electrolyte was read using the EC-Lab software.

$$Q = \int_{t_1}^{t_2} I dt \quad (3)$$

where:

$Q$  – electric charge, C;

$I$  – current, A;

$t_1$  – initial measurement time, s;

$t_2$  – end time of measurement, s.

The electropolymerization process of individual polymers is shown in Figures 1, 2, and 3.

In the tested systems, an electrolyte contains poly (methyl methacrylate) (AlfaAestar, MW = 550,000), acetonitrile (Acros, 0.99%), propylene carbonate (Acros ORGANICS, 99.5%) and lithium perchlorate ( $\text{LiClO}_4$ ) (Acros, clear). In the first stage of electrolyte preparation, the weighed amount of lithium perchlorate needed for the research was dissolved in an appropriate amount of propylene carbonate. This amount was due to the molar ratio of the lithium salt and the solvent. Then, acetonitrile and powdered poly (methyl methacrylate) was added to the mixture. The molar ratio of the substances contained in the electrolytes used in the tests is presented in Table 1.

The electrolyte was placed in an ultrasonic washer and then introduced into a dryer to evaporate the solvent. The drying process lasted about 12 hours and was supposed to increase density.

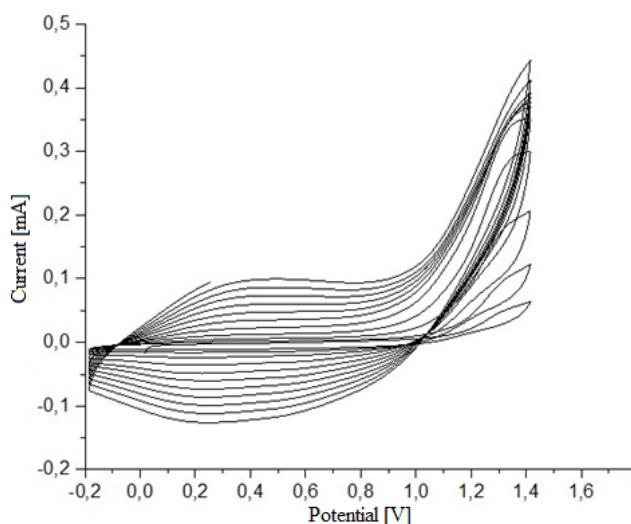


Fig. 1. Voltamogram of pyrrole electropolymerization

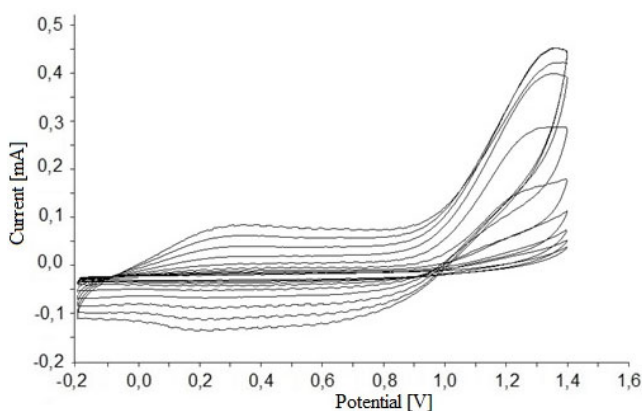


Fig. 2. Voltamogram of 3-n-octylpyrrole electropolymerization

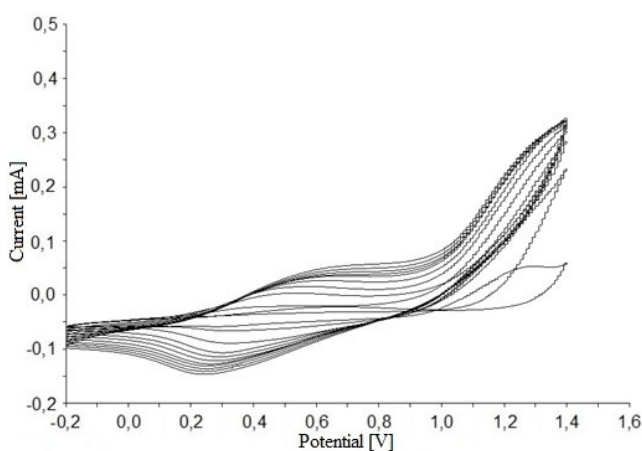


Fig. 3. Voltamogram of phenylpyrrole electropolymerization

Table 1.  
The molar ratio of substances contained in the electrolyte

Compound	Molar ratio
ACN	0.5130
PC	0.0617
PMMA	0.0228
LiClO <sub>4</sub>	0.0034

The electrolyte used in the experiment was applied with syringes. On each surface of the prepared polymer, 0.1 milliliters [ml] of the tested electrolyte was applied. After 10 minutes from the end of the application and the simultaneous evaporation of a part of the electrolyte, an additional application of one drop of electrolyte was performed on each polymer layer. The plates prepared in this way were brought into contact with each other on the contact surface, corresponding to approximately 1/3 of the length of the tested element. The distance between the two plates of the created supercapacitor constituting its electrodes in this system was about 70 micrometers [ $\mu\text{m}$ ]. On the ITO plates, the polymer was deposited on a surface area of about 6 mm<sup>2</sup>. Figure 4 shows a diagram of a complex supercapacitor.

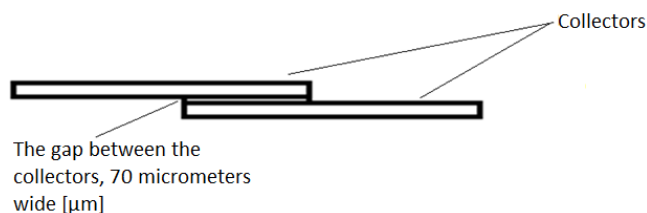


Fig. 4. Diagram of a complex supercapacitor

### 3. Methodology

Each of the created systems was tested by cyclic voltammetry and chronopotentiometry. Potentiostat CHI 660, CHI 620, and potentiostat SP-150 by Biologic were used for the tests.

The first method by which supercapacitor systems were investigated was cyclic voltammetry (CV). This method was used in laboratory work as it is one of the primary methods of testing conductive polymers.

As a result of measuring cyclic voltammetry for each system, a volt amperometric loop was obtained. During the tests of polymer supercapacitors, a potential window ranging from -0.2 V to 0.8 V was used because such a possible range ensured the correct ranges of the recorded current intensity and, at the same time, was safe for the tested materials. There

is a risk that the polymers could be damaged at a higher given potential.

Cyclic voltammetry makes it possible to determine each tested system's specific capacity easily. Therefore, the individual capacity of the tested supercapacitors was calculated using equation no. 4; in the literature, it is the primary method of determining the particular capacitance of supercapacitors [22].

$$C_p = \frac{P}{2 * m * \mu * (V_2 - V_1)} \quad (4)$$

where:

$C_p$  – supercapacitor capacity, F/g;

$P$  – voltamogram loop area, A\*V;

$m$  – a mass of the electrode substance, g;

$\mu$  – system scanning speed, V/s;

$(V_2 - V_1)$  – the value of the potential window, V.

The above dependence is the result of mathematical transformations having their genesis in the basic equation describing the capacitance of a supercapacitor and presented in No. (5).

$$C_p = \frac{I}{m * \mu} \quad (5)$$

We were rearranging equation no. (5) we get equation no. (6).

$$I = C_p * m * \mu \quad (6)$$

The current during a given test cycle shall be expressed as the value of the integral equation bounded by the importance of the boundary potentials. The relationship in question appears in equation no. (7).

$$\int_{V_1}^{V_2} I(v) dv = \int_{V_1}^{V_2} (C_p * m * \mu) dv \quad (7)$$

The surface area of the voltammetric loop ( $P$ ) obtained during the measurement of the tested systems is equal to the total current in a given cycle. This relationship is expressed by equation no. (8)

$$P = \int_{V_1}^{V_2} (C_p * m * \mu) dv \quad (8)$$

After solving the integral equation, we get equation no. 4.

The second method of testing the electrochemical parameters of the tested devices was the method of chronopotentiometry. The tested devices' specific capacity and energy density were calculated using this method. The result of measurements of energy storage devices by chronopotentiometry are galvanostatic charge and discharge curves. During the measurements, the current value was  $5 * 10^{-5}$  A, while the range of the set potential was from -0.2 V to 0.8 V. As a result of the analysis of research data carried out with the method of chronopotentiometry, it was possible

to calculate the specific capacitance of supercapacitor systems. They were calculated using equation No. (9) [22].

$$C_p = \frac{I \cdot \Delta t}{m \cdot (V_2 - V_1)} \quad (9)$$

where:

I – current in the system, A;

$\Delta t$  – time range, s;

m – a mass of active substance, g.

The energy density was calculated based on the research results on the specific capacitance obtained from the experiment; it is necessary to characterize the supercapacitor systems using the chronopotentiometry method. The significant parameters of the supercapacitor include the energy density, which was calculated using equation No. (10) [22].

$$E_g = \frac{1}{2} * C_p * (V_2 - V_1)^2 \quad (10)$$

where:

$C_p$  – specific capacity, F/g;

$(V_2 - V_1)$  – potential range, V.

#### 4. Results

To approximate the morphological structure of the polymers, pictures were taken using a scanning electron microscope (SEM) by Phenom ProX (Figs. 5-7).

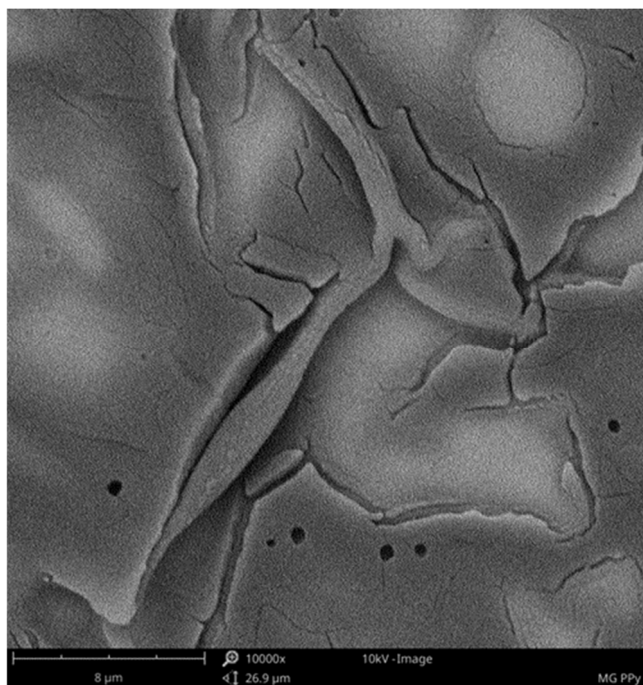


Fig. 5. SEM photo of polypyrrole

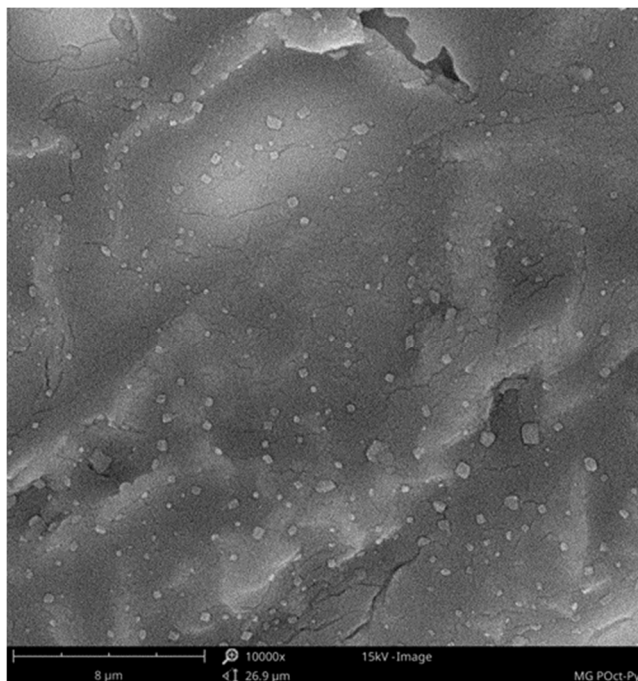


Fig. 6. SEM photo of poly(3-n-octyl pyrrole)

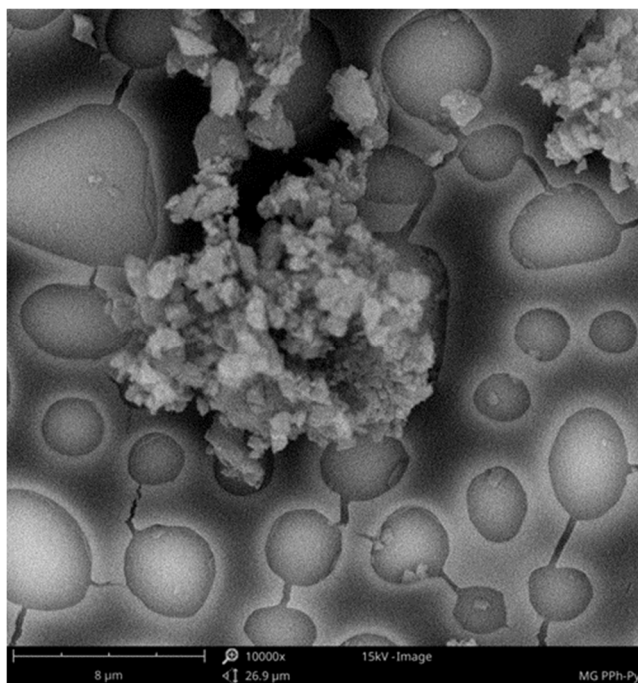


Fig. 7. SEM photo of poly(phenylpyrrole)

The devices were tested using cyclic voltammetry and chronopotentiometry (Figs. 8 and 9). The thickness of the coating was calculated using the weight of the polymer and

its density. The weight of the polymer in each device and the thickness of the coating layer are shown in Table 2. The

Parameters of supercapacitors containing collectors made of ITO plates are presented in Table 3.

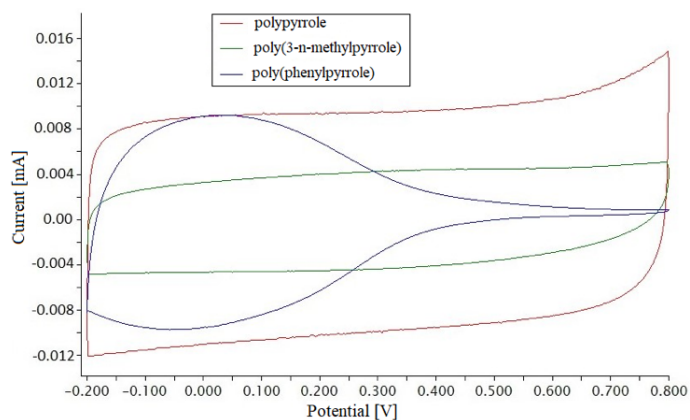


Fig. 8. Voltammetric loops of individual supercapacitors containing collectors made

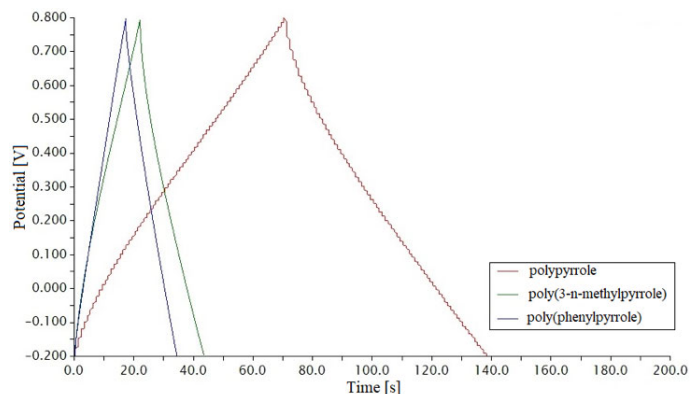


Fig. 9. Chronopotentiometric curves of the investigated supercapacitors

Table 2.

Parameters of the tested material

Collector	Parameters of the tested material			
	Polymer	Polypyrrole	Poly(3-n-octylpyrrole)	Poly(phenylpyrrole)
Mass of polymer, g		1.96E-05	1.49E-05	2.10E-05
The average thickness of the polymer layer, $\mu\text{m}$		0.11	0.10	0.12

Table 3.

Parameters of supercapacitors containing collectors made of ITO plates

Collector	Parameters of the tested supercapacitors			
	Polymer	Polypyrrole	Poly(3-n-octylpyrrole)	Poly(phenylpyrrole)
Specific capacity with CV, F/g		49.04	10.23	6.78
Specific capacity with CP, F/g		52.32	9.11	5.61
Energy density, Wh/kg		26.16	4.56	2.81

## 5. Description of the results and discussion

The best electrochemical parameters were obtained for a supercapacitor containing polypyrrole as the electrode material. Therefore, a high specific capacity and energy density characterize this device. On the other hand, the lowest electrochemical parameters were obtained in a device containing poly (phenylpyrrole) while A supercapacitor with poly (3-n-octyl pyrrole) as the electrode material generated intermediate values of electrochemical parameters.

The voltammetric loop obtained by measuring a supercapacitor containing polypyrrole has the most similar shape to the so-called loop - the perfect supercapacitor. An ideal supercapacitor has a square-shaped voltammeter loop because the charge of a perfect capacitor is directly proportional to the voltage generated by such a device. The device containing poly(3-n-octyl pyrrole) has a characteristic volt amperometric loop. The loop for this device reduces its surface area at higher potential values. A device containing poly(phenylpyrrole) generates a voltammetric loop with the smallest surface area of the tested devices.

The chronopotentiometry curves for the tested devices differ in the discharge time from 0.8 V to -0.2 V. The polypyrrole device has the longest discharge time, while the poly (phenylpyrrole) has the shortest discharge time.

The dependence on the different characteristics of the electrochemical properties of polymers is due to the other molecular structures of the tested compounds. Polypyrrole has the best electroactive properties of the tested polymers. Lower values of the tested parameters for pyrrole derivatives occur due to the steric hindrance in the structure of poly(3-n-octyl pyrrole) and poly (phenylpyrrole). The lower specific capacity in the case of poly(3-n-octyl pyrrole) and poly (phenylpyrrole) is probably due to the charge transfer resistance during electrochemical reactions generated by their substituents. In poly(3-n-octyl pyrrole), there is a chain of eight carbons; in poly(phenylpyrrole), there is an aromatic ring.

The characteristic voltammetric loop generated for a supercapacitor containing poly(3-n-octyl pyrrole) is due to the high resistance caused by this material at higher potential values. For the other two devices, the voltammeter loops are correctly shaped.

In Figure 5, we can see clear three-dimensional structures that indicate good material deposition on the conductive layer. At the same time, we will not notice any crystalline structures on the surface that can disrupt electrical conductivity. For this reason, polypyrrole has the best electrochemical properties. In the SEM photo of poly(3-

n-octyl pyrrole), we can see the even surface of the polymer. However, in some places, there are defects in the form of polymer crystals. These defects may negatively affect the electroconductive properties of the material. Polyphenylpyrrole showed the lowest electrochemical parameters. In the SEM image of this polymer, large crystalline formations can be observed, which could negatively impact the material's electrochemical reactions. The electrolyte used in the tested devices helped to obtain the appropriate electrochemical parameters. A well-chosen molar ratio of suitable solvents, polymer, and active salt (lithium perchlorate) results in the proper operation of individual electrochemical devices.

Analysing the results, we can conclude that the individual components of the electrolyte have been selected correctly. In this research, it was possible to make supercapacitor systems with comparable electrochemical parameter values available in the literature.

## 6. Summary

After analysing the results obtained from the conducted research, it should be concluded that the assumptions of the analysis in the form of positive verification of the possibility of using various types of polymer materials in supercapacitor systems containing polypyrrole, poly (3-n-octyl pyrrole) and poly (phenylpyrrole) have been fully confirmed. Moreover, the research managed to obtain information on the parameters of supercapacitors containing different types of conductive polymers and develop conclusions demonstrating the benefits of researching issues related to conductive polymers.

Comparing the results of the experimental work with the literature data, the parameters of the devices made have the values of specific capacities, suggesting the possibility of further targeted research on the use of conductive polymers in supercapacitors.

The capacitance results of the selected supercapacitors have more than twice the specific capacity of the systems configured by the researchers from other research teams. These teams include scientists, among others, from the Department of Chemistry and Biochemistry of the University of California in Los Angeles, who constructed a device containing a polypyrrole and copper (II) oxide nanocomposite with a specific capacity of 21 F/g [23]. In contrast, devices based on poly (phenylpyrrole) made by a team of scientists led by Hakan Gorcay from the Faculty of Chemistry of the Anadolu University in Eskişehir, Turkey, had a capacity of 0.8 F/g [24]. Supercapacitors constructed for this research containing polypyrrole had a capacity of 34-



49 F/g, while the supercapacitor based on poly(phenylpyrrole) reached a capacity with a value of 2.8 F/g.

The supercapacitor containing polypyrrole achieved excellent energy density results. The highest energy density measurement by chronopotentiometry was 26.16 Wh/kg. Flexible and high energy density asymmetrical supercapacitors based on manganese dioxide created by a team of scientists from the University of Jinan showed an energy density of 25.8 Wh/kg. The discussed values are similar, so we can conclude that the electrochemical system is comparable to the devices described in the literature [25].

However, the tested systems achieved much lower specific capacitance values than supercapacitors containing porous materials (such as carbon nanotubes). For example, supercapacitors containing polypyrrole and CNT structures (carbon nanotubes) made by a team of scientists from the Laboratory of Electronic Materials Research in China are characterized by a specific capacity of as much as 304 F/g.

Summarizing the research, the structure of polymer molecules determines the electrical properties of devices in which the tested materials were used. At the same time, the obtained research results clearly show that conductive polymers are materials with great potential for electrical energy storage applications.

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