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SupercapBattery performances of rGO/Fe_2O_3/PEDOT hybrid nanocomposites

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Abstract **–** In this study, 3 different materials were combined to form hybrid nanocomposite for SupercapBattery applications. Reduced graphene oxide (rGO), iron (III) oxide (Fe_2O_3), and poly(3,4-ethylenedioxythiophene) (PEDOT) were used as a component of nanocomposites. Electrochemical performances were performed by cyclic voltammetry (CV), galvanostatic charge / discharge (GCD) and electrochemical impedance spectroscopy (EIS) measurements. EIS measurements were analyzed by Nyquist, Bode-magnitude, Bode-phase, and Admittance plots. Long-term stability tests were obtained by CV method using 1000 charge/discharge performances at a scan rate of 100 $mV \times s^{-1}$. The highest specific capacitance was calculated as C_{sp}= 1129.38 $F \times g^{-1}$ at 2 $mV \times s^{-1}$ (electrode weight was obtained as 15,7 mg).

Keywords – Supercapbattery, 2032 Coin Cell, PEDOT, Hybrid Nanocomposite, Energy Storage

I. INTRODUCTION

Energy storage systems are critical importance for supercapacitors and batteries [1, 2]. Carbon materials, such as reduced graphene oxide (rGO) is an electrode material for electric double layer capacitance (EDLCs) because of its high electrical conductivity, high specific area, chemical stability, and mechanical strength, eco-friendly, etc [3-6]. Graphene has an important material in various areas since it was found in 2004 because of its unique physical and chemical properties [7].

II. MATERIALS AND METHOD

Electrochemical measurements were performed with 2032 coin-type cells. The slurry was obtained by mixing the as-synthesized materials, acetylene black and *N*-methyl-2-pyrrolidone (NMP) as a solvent for materials. Polyvinyl pyrrolidone (PVP) was performed by binder. Then the slurry was pasted onto Al and Cu foils and dried at 60 ^oC in a vacuum oven for 12 h. The electrolyte was ionic liquid (IL). And the mass loading of electrode of 13 mg. Galvanostatic charge/discharge, rate performance and cyclic performances were tested between 0.0 and 0.8 V by using ivium-vertex potentiostat-galvanostat instrument.

A. GO and rGO synthesis

For this purpose, 3 g of graphite was mixed with 70 ml of 3 M H_2SO_4 (11.7 ml of H_2SO_4, completed to 70 ml with 58.3 ml of DI water in an ice-bath at 15 ^oC for 4 h. $KMnO_4$ (9 g) was gradually added to the mixture within 1 h while stirring was continued. The mixture was stirred for an additional 3h in an ice-bath to avoid explosions. The temperature was raised to 35 ^oC and then 150 ml of DI water was added to the mixture. After adding DI water, the temperature of mixture was increased to 97 ^oC. Afterwards, stirring was continued and the solution was allowed to cool to room temperature.

DI water (500 ml) was added to the solution and the mixture was placed in an ice-bath. The mixture temperature was reduced to 15 ^oC. Then, while the solution was in the ice-bath, H_2O_2 (10 ml of 30%) was added dropwise and stirring was

continued for 4 h. After mixing process was completed, it was waited for some time (1 h) and it was observed that the solution separated into 2 phases. The upper aqueous phase was first poured and then the Pasteur was removed with a pipette and separated from the lower solid phase. The remaining phase at the bottom was placed in 4 centrifuge tubes. It was centrifuged at 4000 rpm for 15 min. The remaining part was centrifuged (up to 12 ml). Washing process with DI water was repeated 10 times (until pH reached 5 and above). The metal ions in the solid precipitated by the washing process were removed from the environment. The solution was washed with 10 ml, 1 M HCl, 5% NH_3, 5% ethanol and finally DI water. Finally, it was taken into a petri-dish and dried in an oven at 70 ^oC. Afterwards, GO materials were obtained by reducing GO with microwave oven (Bosch or Samsung) with the microwave method [8].

B. *Fe_2O_3 nano-material synthesis*

$FeCl_3 \times 6H_2O$ (1.5 g) was dissolved in 2 ml of HCl solution (30 ml). This mixture was obtained as pH= 11 adding dropwise to 25% NH_4OH solution (75 ml) with stirring of solution. Afterwards, 2 ml of HCl was added dropwise until pH=2 and the mixture was stirred for 2h. Then by centrifugation and the collected solid was washed with DI water to remove access NH_4OH. The obtained product was dried at 80 ^oC for 8 h. Thus, Fe_2O_3 nanomaterials was synthesized [9, 10].

C. *PEDOT synthesis*

Polyvinylpyrrolidone (PVP, 30 mg) was placed in 20 ml of DI water in 250 ml flask and dispersed by ultrasonication device for 30 min. EDOT monomer (80 mg) and naphthalene-1, 5-disulfonic acid tetra hydrate (60 mg) were added into the flask and mixed for 30 min. Ammonium persulfate (APS, 500 mg) was added to the flask and mixed. The polymerization was carried out at room temperature for 15 h. As a result, PEDOT in solid form was obtained and dried in vacuum oven for 15 h at 60 ^oC by filtration through cellulose membrane.

D. *Electrochemical performances of SupercapBattery device*

rGO/Fe_2O_3/PEDOT nanocomposite were measured by CV, GCD and EIS measurements.

E. *CV measurements*

CV plots of rGO/Fe_2O_3/PEDOT nanocomposite at different scan rates from 1000 $mV \times s^{-1}$ to 2 $mV \times s^{-1}$ were given in Figure 1.

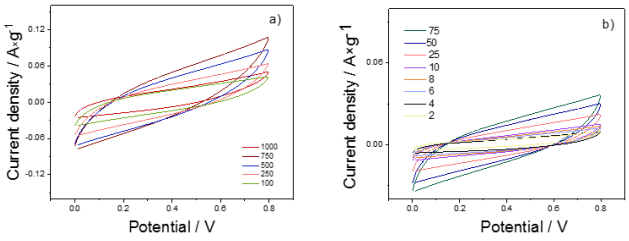

Fig. 1 CV plots of rGO/Fe_2O_3/PEDOT nanocomposite at different scan rates, a) 1000-100 $mV \times s^{-1}$, b) 75-2 $mV \times s^{-1}$.

The lowest specific capacitance was obtained as C_{sp}= 11.67 $F \times g^{-1}$ at 1000 $mV \times s^{-1}$. However, the highest specific capacitance was found as C_{sp}= 1129.38 $F \times g^{-1}$ at 2 $mV \times s^{-1}$. There is a logarithmic decrease by increasing of scan rate due to fast ion movement from one compartment to another compartment (Figure 2).

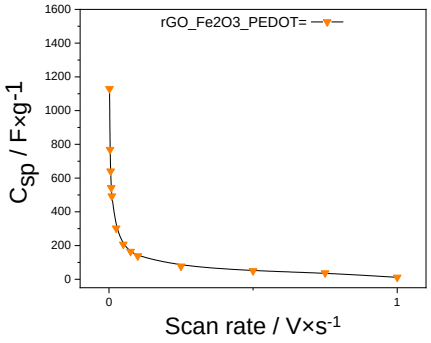

Fig. 2 C_{sp} vs. Scan rate plot of rGO/Fe_2O_3/PEDOT nanocomposite at different scan rates from 1000 to 2 $mV \times s^{-1}$.

F. *GCD measurements*

GCD plots of rGO/Fe_2O_3/PEDOT nanocomposite was given at constant current density from 0.1 $A \times g^{-1}$ to 10 $A \times g^{-1}$ as shown in Figure 3. The highest specific capacitance was obtained as C_{sp}= 1.74 $F \times g^{-1}$ at 0.1 mA by GCD measurements.

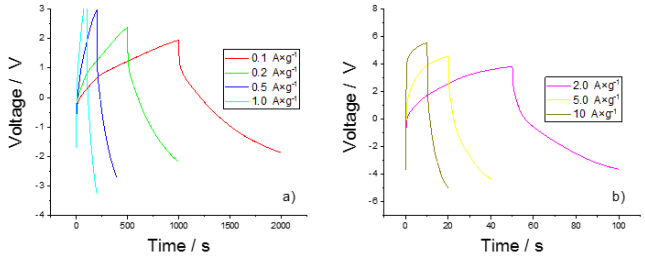

Fig. 3 GCD plots of rGO/Fe_2O_3/PEDOT nanocomposite at constant current density from 0.1 A×g^{-1} to 10 A×g^{-1}.

G. *EIS measurements*

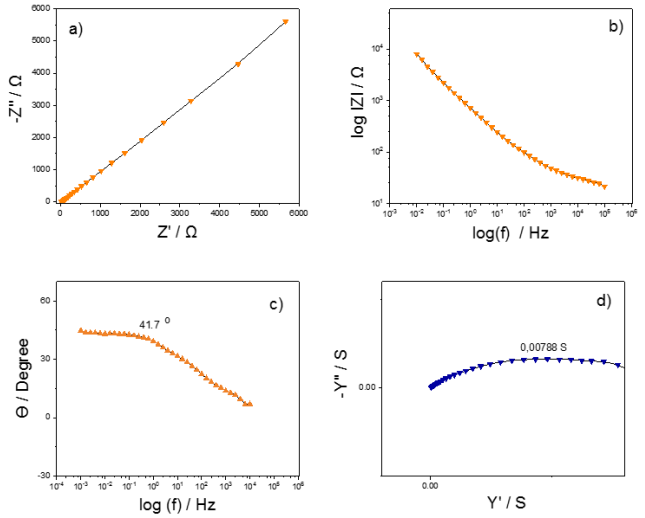

Fig. 4 EIS plots of rGO/Fe_2O_3/PEDOT nanocomposite a) Nyquist plot, b) Bode-magnitude plot, c) Bode-phase plot, d) Admittance plot.

EIS plots of rGO/Fe_2O_3/PEDOT nanocomposite were given in Figure 4. Specific capacitance was obtained as C_{sp}= 0.18 F×g^{-1} from Nyquist plot. Double layer capacitance and phase angle were obtained as C_{dl}= 0.089 F×g^{-1} and θ= 41.7º at 0.258 Hz from Bode-magnitude and Bode-phase plots, respectively. Admittance plots defined conductivity of nanocomposite material (Y″= 0.00788 S).

H. Stability tests

The stability graphs of the rGO/Fe_2O_3/PEDOT nanocomposite for 2032 coin cell and SS electrodes were given charge/discharge device performances for 1000 cycles (Fig.5). The first capacitance value after 1000 charge/discharge performances were obtained as 88.49% for the SS electrode in ionic liquid in 2032 coin cell.

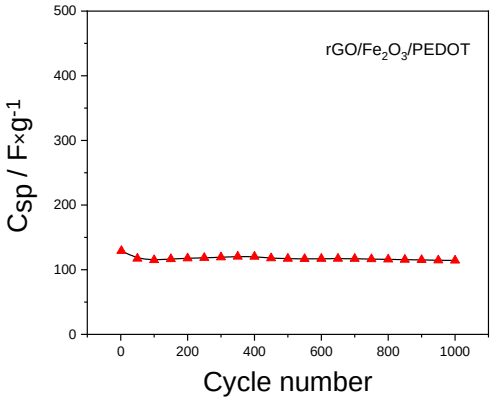

Fig. 5 Stability tests of rGO/Fe_2O_3/PEDOT nanocomposite at a scan rate of 100 mV×s^{-1}, 1000 charge-discharge measurements.

III. RESULTS & DISCUSSION

SupercapBattery device performances were obtained by 2032 coin cell. The highest energy and power densities were obtained as E= 6.01 Wh×kg^{-1} at 10 mA and P= 2210.19 W×kg^{-1} at 10 mA. EIS data were also presented as θ= 41.7º at 0.258 Hz and 88.49% for initial capacitance preservation for 1000 charge-discharge measurements.

IV. CONCLUSION

Our results have demonstrated that rGO/Fe_2O_3/PEDOT nanocomposites will be considered as a promising symmetrical electrode materials for the next generation of supercapacitor applications.

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