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S- rGO/Fe_2O_3/PANI nanocomposite Synthesis and Energy Storage Applications

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Abstract – In this study, 3 different materials were combined to form hybrid nanocomposite for SupercapBattery applications. Sulfur doped reduced graphene oxide (S-rGO), iron (III) oxide (Fe_2O_3), and poly(aniline) (PANI) 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}= 157.42 $F\times g^{-1}$ at 2 $mV\times s^{-1}$ (electrode weight was obtained as 13.4 mg).

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

I. INTRODUCTION

Both supercapacitors and batteries are used as an energy storage systems [1, 2]. Reduced graphene oxide (rGO) is an electrode material for electric double layer capacitance (EDLCs) due to its high electrical conductivity, high specific area, chemical stability, and mechanical strength, eco-friendly, etc [3]. Graphene has an important material in various areas since it was found in 2004 because of its unique physical and chemical properties [4].

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 °C in a vacuum oven for 12 h. The electrolyte was ionic liquid (IL). And the mass loading of electrode of 13.4 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*

Graphene oxide (GO) was synthesized from graphite powder according to modified Hummers method [5].

B. *S-rGO synthesis*

In literature, the conductivity of GO increases by doped of S and N elements [6, 7]. As a result, S doped GO have a superior performance compared to other additives (N, B or P) in terms of capacitor performance [8]. 5 ml Na2S was mixed in various sources (0.5 M). It was synthesized in a microwave oven at 180 Watt and 20 min. So, S-GO was obtained by centrifuged 3 times [9].

C. *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 °C for 8 h. Thus, Fe_2O_3 nanomaterials was synthesized [10, 11].

D. *PANI synthesis*

Aniline (0.2 ml) and sodium dodecyl benzene sulphate (SDBS, 3.3 g) as a surfactant will be mixed with 0.06 ml of H_3PO_4 in 10 ml of DI water in an ice-bath. The resulting ANI/H_3PO_4 salt will be added to the 0.46 g / 5 ml DI water soluble ammonium persulfate (APS) mixture. Polymerization will continue in the ice-bath for 12 h. A green colour solid PANI will be formed. The resulting solid PANI will be dried by DI water and ethyl alcohol [12].

E. *Electrochemical performances of SupercapBattery device*

S-rGO/Fe_2O_3/PANI nanocomposite were measured by CV, GCD and EIS measurements.

F. *CV measurements*

CV plots of S-rGO/Fe_2O_3/PANI nanocomposite at different scan rates from 1000 mV×s^{-1} to 2 mV×s^{-1} were given in Figure 1.

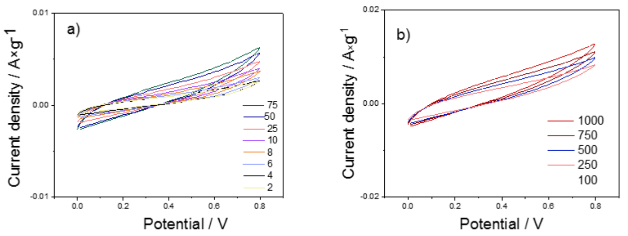

Fig. 1 CV plots of S-rGO/Fe_2O_3/PANI nanocomposite at different scan rates, a) 75-2 mV×s^{-1}, b) 1000-100 mV×s^{-1}.

The lowest specific capacitance was obtained as C_{sp}= 1.69 F×g^{-1} at 1000 mV×s^{-1}. However, the highest specific capacitance was found as C_{sp}= 157.42 F×g^{-1} at 2 mV×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).

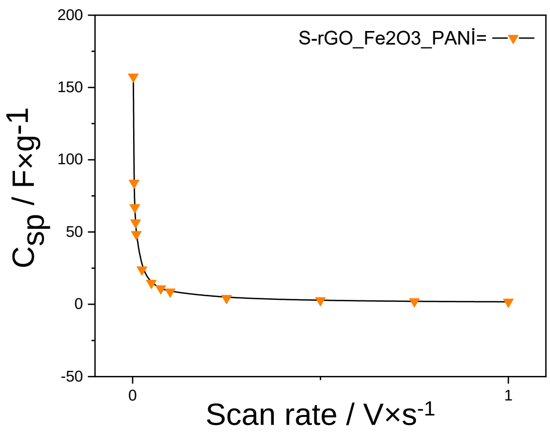

Fig. 2 C_{sp} vs. Scan rate plot of S-rGO/Fe_2O_3/PANI nanocomposite at different scan rates from 1000 to 2 mV×s^{-1}.

G. *GCD measurements*

GCD plots of S-rGO/Fe_2O_3/PANI nanocomposite was given at constant current density from 0.1 A×g^{-1} to 10 A×g^{-1} as shown in Figure 3. The highest specific capacitance was obtained as C_{sp}= 2.26 F×g^{-1} at 0.5 mA by GCD measurements.

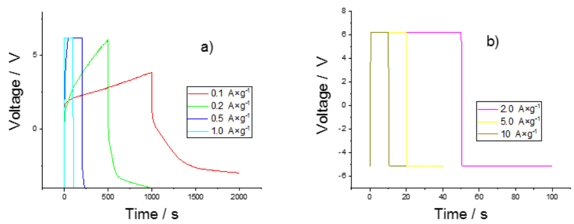

Fig. 3 GCD plots of S-rGO/Fe_2O_3/PANI nanocomposite at constant current density from a) 0.1 A×g^{-1} to 1.0 A×g^{-1}, b) 2.0 A×g^{-1} to 10 A×g^{-1}.

H. *EIS measurements*

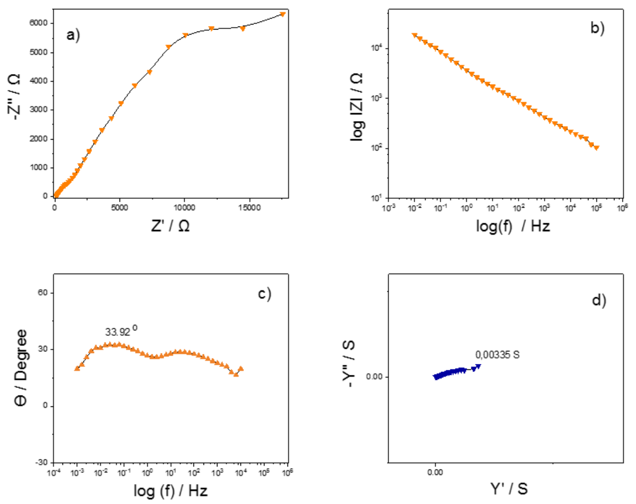

Fig. 4 EIS plots of S-rGO/Fe_2O_3/PANI nanocomposite a) Nyquist plot, b) Bode-magnitude plot, c) Bode-phase plot, d) Admittance plot.

EIS plots of S-rGO/Fe_2O_3/PANI 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}= 0.020F×g^{-1} and θ= 33.92^o at 0.0392 Hz from Bode-magnitude and Bode-phase plots, respectively. Admittance plots defined conductivity of nanocomposite material (Y″= 0.00335 S).

İ. Stability tests

The stability graphs of the S-rGO/Fe_2O_3/PANI 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 144.7% for the SS electrode in ionic liquid in 2032 coin cell.

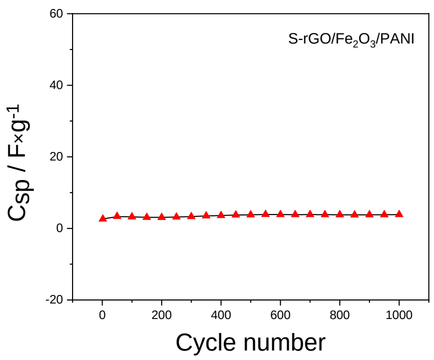

Fig. 5 Stability tests of S-rGO/Fe_2O_3/PANI nanocomposite at a scan rate of 100 mV×s^{-1}, 1000 charge-discharge measurements.

The initial capacitance value was exceeded to 100% due to the wettability and electrode activation process depending on the cycle increase [13, 14].

III. Results & Discussion

SupercapBattery device performances were obtained by 2032 coin cell. The highest energy and power densities were obtained as E= 4.94 Wh×kg^{-1} at 0.1 mA and P= 61.57 W×kg^{-1} at 0.5 mA. EIS data were also presented as θ= 33.92^o at 0.0392 Hz and 144.7% for initial capacitance preservation for 1000 charge-discharge measurements.

IV. Conclusion

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

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