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Simple synthesis of Fe_3O_4@Fe_3S_4 nanocomposites coated with polyindole-polythiophene for high-performance supercapacitor

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ABSTRACT

To prevent a major energy crisis in the near future, it is necessary to assemble efficient electrochemical energy storage devices such as supercapacitors. Herein, a Fe_3O_4@Fe_3S_4 nanocomposites coated with polyindole-polythiophene on nickel foam (NF) has been prepared by following a low facile, multistep method. In terms of structural and electrochemical performance, the nanocomposites have been investigated. The electrochemical properties of as fabricated Fe_3O_4@Fe_3S_4@PIn-PTh are studied in 2M KOH. A specific capacitance of 100.83 F g^{-1} at a current density of 2 A g^{-1}. The results of the synthesized sample in comparison with Fe_3O_4 show that using sulfide metal and polymer component improve the electrochemical performance. These characteristics of Fe_3O_4@Fe_3S_4@PIn-PTh as the active material illustrate a sutiable performance as a cathode material for electrochemical energy storage.

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1. Introduction

Indiscriminate consumption of fossil energy due to the increase in consumption and development of industries has caused many problems, including the reduction of fossil fuel reserves as well as problems such as environmental pollution. Therefore, there is a need to develop clean and renewable energy generators such as solar cells, wind turbines, etc., and considering the dependence of these energies on atmospheric conditions, the issue of storing these energies as a priority is given. According to their characteristics, electrochemical energy storage devices are classified into various categories, such as supercapacitors, batteries, capacitors, and fuel cells [1–6].

Supercapacitors, which have a high power density, a long functional life, and no need for special maintenance equipment, have been the focus of industrial and academic research in recent years. The mechanism of operation of supercapacitors is based on the exchange of electrons between the electrolyte and the electrode of the supercapacitor, and this process depends on the active materials used in the electrode. Common electrode materials in the preparation of transition metal oxides and sulfides are conductive polymers or carbon materials with a large surface area [7–12] as they present multifold advantages of high power density, fast charging–discharging, and long cyclic stability. However, the intrinsically low energy density inherent to traditional supercapacitors severely limits their widespread applications, triggering researchers to explore new types of supercapacitors with improved performance. Asymmetric supercapacitors (ASCs).

Due to their ability to provide high capacity (compared to the oxide state, due to the lower electronegativity of sulfur compared to oxygen), metal sulfides are among the materials that are of interest in research in supercapacitor studies as active electrode materials. In these materials, based on fast and reversible reactions on the surface of the active material, it produces this capacity. In order to achieve high energy density and power, materials must have reversible reactions with electrolytes at high speeds. However, research is aimed at improving the properties of active materials used in supercapacitor electrodes. One of the things that is of interest in research today in order to improve the properties of these materials is the use of nanomaterials. Due to their porous and regular structure, these materials cause the structure to become porous and subsequently increase the electron exchange between the electrode and the electrolyte, which leads to an improvement in the electrochemical performance of the supercapacitor [13–15].

Another desirable thing in the synthesis of active materials is to functionalize the surface and increase the hydrophilic property due to the improvement of electron exchange in the electrochemical process, which is possible by coating the synthesized structure with a polymer layer. For the synthesis of iron oxide magnetic particles in controlled atmospheres, various methods such as hydrothermal, co-precipitation, sol-gel, and solvothermal are used. Among the mentioned methods, the co-precipitation method is a suitable option for synthesis on a large and industrial scale due to its ease of implementation and lower cost [16–18] a novel core-shell structure is developed based on CoS deposited on NiS nanosheets, which involves hydrothermal and electrodeposition method. The micromorphology of the composite electrode can be optimized by adjusting the cycles of electrodeposition. Taking advantages of the high-

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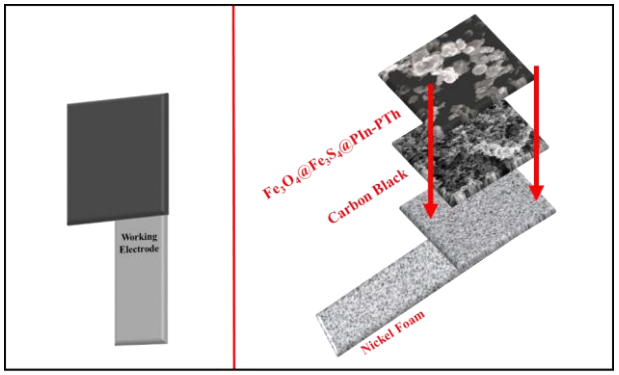

Fig. 1. Fabricated working electrode with its components.

ly conductive, open framework of the core-shell nanolayer, the 5-NiS@CoS electrode shows a specific capacitance of 1210 F g^{-1} at a current density of 1 A g^{-1} (retaining 82% from 1 to 10 A g^{-1} , while NiS substrate is only 39%.

Using two 2 and 3 valent iron salts and the co-precipitation method, particles for use as the active material in the hybrid supercapacitor electrode are prepared in this study. There is a need for identification and functional tests for electrochemical performance and electron exchange between electrode and electrolyte.

In this work, Fe_3O_4@Fe_3S_4 nanocomposites coated with polyindole-polythiophene (Fe_3O_4@Fe_3S_4@PIn-PTh) are synthesized as active materials and evaluated for supercapacitor application. This study consists of material Characterization and electrochemical investigation.

2. Experimental

2.1. Material and Electrode

Magnetic Fe_3S_4 nanoparticles (magnetite) were synthesized by the coprecipitation method. First, 2.7 g of $FeCl_3$ salt and 7.84 g of $FeCl_2.6H_2O$ with a Fe (III) to Fe (II) molar ratio of 2:1.Ions were dissolved in 400 ml of deionized water, and the solution was deoxygenated for 10 minutes before being heated to 80 degrees Celsius during simultaneous deoxygenation. After that, 20 ml of 25% ammonium hydroxide solution was added to the stirring solution (1000 rpm). The presence of oxygen causes the formation of Fe_2O_3 nanoparticles, which are not very desirable because they have a lower specific surface area and lower magnetic properties than Fe_3O_4. After the end of the reaction, the precipitate of magnetite nanoparticles was separated from the reaction environment with a magnet and washed several times with deionized water, and finally the obtained nanoparticles were dried at ambient temperature.

To prepare magnetic nanoparticles covered with a polymer layer and to increase the hydrophilic property and functionalize its surface, a mixture of thiophene and indole was added to the magnetic nanoparticles prepared in the previous step by the chemical oxidation polymerization method in an aqueous medium using Fe (III) chloride synthesized as an oxidant.Thus, 0.75 grams of magnetic nanoparticles were added to 100 ml of deionized water and subjected to ultrasonic waves for 10 minutes. Next, a solution consisting of 0.3 ml of thiophene and 0.19 g of indole was added to the reaction mixture, and the resulting mixture was stirred for 10 minutes. Next, 1.3516 grams of $FeCl_3$, which was dissolved in 25 ml of deionized water, was added dropwise to the aforementioned mixture over a period of 30 minutes, and the polymerization process was carried out for 6 hours at a temperature of 30 degrees Celsius. Grad continued. Then, in order to stop the polymerization reaction, acetone was added to the reaction mixture. Finally, the functionalized adsorbent was collected with a magnet and washed several times with deionized water and methanol to remove impurities.

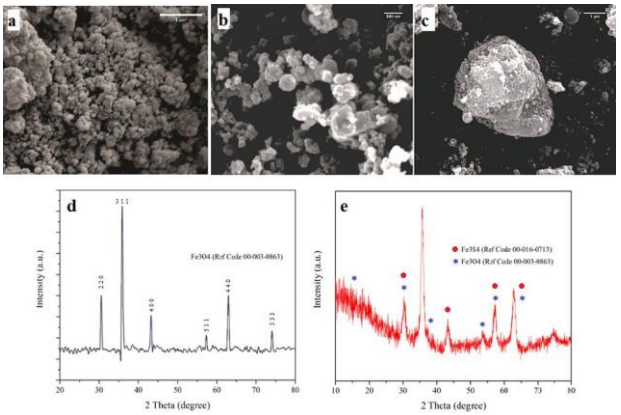

Fig. 2. SEM images of (a) Fe_3O_4 and (b-c) Fe_3O_4@Fe_3S_4@PIn-PTh, XRD patterns of (d) Fe_3O_4 and (e) Fe_3O_4@Fe_3S_4@PIn-PTh.

In order to produce the sulfide component, 0.25 g of Fe_3O_4@PIn-PTh in 300 ml of ethylene glycol was subjected to ultrasonic waves for 15 minutes to obtain a homogeneous mixture. Next, 2.25 g of thiourea was dissolved in 40 ml of ethylene glycol and added to the reaction mixture. The intended reaction continued for 4 hours at 150°C, and finally the synthesized material was washed with enough water and ethanol to remove impurities and unreacted materials.

2.2. Material Characterization and electrochemical investigation

The morphological investigation of the samples was done by scanning electron microscopy (SEM) (FEI SEM QUANTA 200). For electrochemical performance, Palmsens (Palmsens 4) was used for all evaluations, such as cyclic voltammetry (CV), charge and discharge (CD), and electrochemical impedance spectroscopy (EIS) in 2 M KOH as the electrolyte. The electrodes (active materials coated on nickel foam) are applied as a working electrode linking a reference electrode of Ag/AgCl. Platinum (Pt) wire was employed as a counter electrode in three-electrode cell mode. The active materials coated on nickel foam as working electrode was made as follows: active material, acetylene black, and polyvinylidene fluoride with mass ratio of 80:15:5 in N-Methyl-2-pyrrolidone on washed nickel foam electrode with thickness of 0.2 cm, for fabrication of samples as working electrode. Then, the slurries were coated on Ni foam with brushing method. The mass of slurry on the electrode was 3 mg cm^{-2} on 1 cm^{-2}. Finally, the fabricated electrodes dried at 100 °C for 150 min. Fabricated electrode can be seen in Fig.1. The following equations are used to determine the specific capacitance (Csp) [19] Fe_3O_4@C nanoparticles were synthesized by thermal decomposition. Then these synthesized nanoparticles, 20–30 nm in size were processed in a solution of glucose at 200 °C during 12 h, which led to an unexpected phenomenon—the nanoparticles self-assembled into large conglomerates of a regular shape of about 300 nm in size. The morphology and features of the magnetic properties of the obtained hybrid nanoparticles were characterized by transmission electron microscopy, differential thermo-gravimetric analysis, vibrating sample magnetometer, magnetic circular dichroism and Mössbauer spectroscopy. It was shown that the magnetic core of Fe_3O_4@C nanoparticles was nano-crystalline, corresponding to the Fe_3O_4 phase. The Fe_3O_4@C@C nanoparticles presumably contain Fe_3O_4 phase (80%, .

$$C_{sp} = \frac{I \times \Delta t}{m \times \Delta V} \quad (1)$$

3. Results and discussion

3.1. Structural Characterization

As can be seen in Fig.2 (a-c), based on the scanning electron micro-

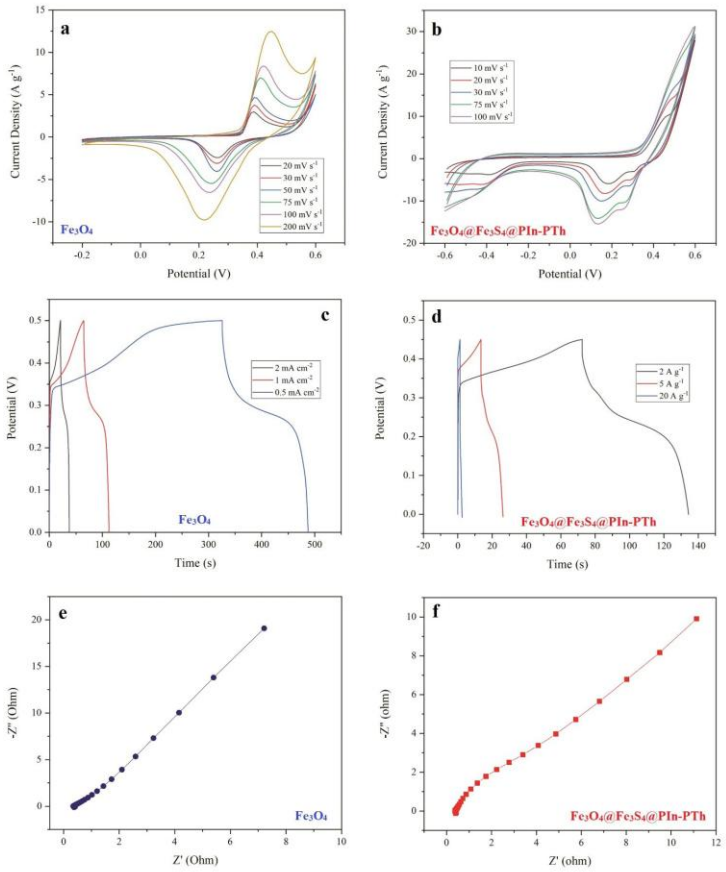

Fig. 3. (a-b) CV curves, (c-d) GCD curves, (e-f) EIS curves of Fe_3O_4 and Fe_3O_4@Fe_3S_4@PIn-PTh.

scope images of the synthesized nanoparticles, a spherical morphology with relatively homogeneous particle distribution is seen. One of the things that prevents a completely desirable morphology in these situations is the lack of complete interaction between the reacting materials. The dimensions of the synthesized particles are in nanometers, which causes a favorable interaction with the electrolyte in order to exchange electrons. According to previous researches, nanometer dimensions and spherical morphology for active materials, due to having a more specific area, will improve the electrochemical performance of these materials in supercapacitor applications [20–22]. In comparison with Fe_3O_4 sample, particles have more diameter because of having sulfide particle on their surface and a cover of PIn-PTh. X-ray diffraction test has been used for phase identification (Fig.2 (d-e)). By analyzing the diffraction pattern resulting from this test by X'Pert HighScore software, iron oxide (Fe_3O_4) in Fe_3O_4sample and a complex of Fe_3O_4 and Fe_3S_4 phases have been identified in Fe_3O_4@Fe_3S_4@PIn-PTh sample [15], [23–26]. As can be seen, the intensity of Fe_3O_4peaks were decreased by using sulfide element and the pattern looks more like amorphous phase which this causes more active cites for electron transition. Another benefits of turning oxide component as anion with sulfide is having lower electronegativity which enhances electrochemical performance [13].

Firstly, Fe_3O_4 and Fe_3O_4@Fe_3S_4@PIn-PTh were exanimated by the cyclic voltammetry (CV) curves. Fig.3 (a-b) illustrate that Fe_3O_4@Fe_3S_4@PIn-PTh possesses a higher CV area compared to another sample, which leads to high capacitance performance. Furthermore, the CV curve of Fe_3O_4@Fe_3S_4@PIn-PTh has one more peak which is belonged to PIn-PTh component and electrolyte reaction [27, 28]. Moreover, the potential range of Fe_3O_4@Fe_3S_4@PIn-PTh is more than Fe_3O_4 which leads to work that wider range of potential. The scan rate of both sample ranged from 5 to 200 mV s-1, and the corresponding current intensity of the peaks increased and shows the good electrochemical reversibility. Both samples were evaluated by the galvanostatic charge-discharge (GCD) technique in 2 M KOH solution at different range of current density (Fig.3 (c-d)). Both samples' curves show a nonlinear charge-discharge profile that confirmed that the pseudocapacitance type of mechanism. The performance of Fe_3O_4@Fe_3S_4@PIn-PTh is better than $Fe_3O_4$and there is little iR drop (easy electron transformation) and can work at higher current densities with more capacity [29–33] which is constructed with many intercrossed nanorods, is successfully synthesized via a facile hydrothermal method. The achieved Fe_3S_4 microspheres are evaluated as advanced anode materials for alkaline iron-based secondary batteries for the first time. Influences of hydrothermal temperature on the physical and electrochemical performances of Fe_3S_4 particles are investigated systematically. It is found that the Fe_3S_4 fabricated at 150 °C exhibits better electrochemical properties than the ones obtained at other temperatures, including higher discharge capacity, attractive high-rate capability and good cycling stability. At 0.2, 0.4, 1.0, 2.0, and 5.0 C, the Fe_3S_4(150 °C. The specific capacitance of the Fe_3O_4@Fe_3S_4@PIn-PTh electrode (100.83 F g^{-1} at 2 A g^{-1}) is larger than the Fe_3O_4 electrode (41.75 F g^{-1}).

Electrochemical impedance spectroscopy (EIS) was performed in a frequency range of 100 kHz-0.01 Hz with an AC voltage amplitude of 2 mV. In the EIS plots in Fig.3, for Fe_3O_4@Fe_3S_4@PIn-PTh sample, at high frequencies, a resistance of less than 1 ohm was observed for the sample, which indicates low internal resistance and good performance for electron exchange. At low frequencies, due to the approaching linear behavior and closeness to the vertical state, performance close to that of ideal supercapacitors can be seen. The results of EIS investigation for Fe_3O_4 is worse compared to Fe_3O_4@Fe_3S_4@PIn-PTh (Fig.3 (e-f)) [34–37].

4. Conclusions

In summary, the Fe_3O_4@Fe_3S_4 nanocomposites coated with polyindole-polythiophene have been synthesized by a simple chemical method. According to structural characterization and electrochemical investigation, the results present that using sulfide and polymer component improves electrochemical performance. It is believed that Fe_3O_4@Fe_3S_4@PIn-PTh can be used as active material in electrochemical storage applications.

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Conflict of Interest

All authors declare no conflicts of interest in this paper.

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