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Microwave-assisted hydrothermal synthesis of HCN@NiS composite electrodes for supercapacitors

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***Abstract* –** In recent decades, rapid urbanisation, industrialisation and global population growth have led to environmental degradation and the depletion of non-renewable energy sources. However, these green and environmentally friendly new energy sources are often affected by the climatic environment and region, so the energy produced has shortcomings such as intermittency and instability, requiring energy storage devices with high energy and power density. In this study, hollow carbon nanorods (HCN) were obtained in one step and HCN structures were coated with nickel sulphide (NiS) by microwave-assisted hydrothermal method. The obtained samples were characterised by SEM and EDX. Morphological properties show that NiS is coated on carbons with a complete nanorod structure. As a result, HCN@NiS obtained by microwave-assisted hydrothermal synthesis, which is a fast, easy and inexpensive method, provides a capacitance of 0.15 F/cm^2 at a current density of 1 mA/cm^2. Electrochemical test results show that it provides a large number of reactive sites and shows better electrochemical properties. In the light of these results, it is understood that microwave-assisted material synthesis may be suitable synthesis conditions for supercapacitors in the future.

Keywords – Supercapacitor, Microwave Synthesis, Carbon Nanorod, Nis

I. INTRODUCTION

Fossil fuels are still the primary source of energy to meet the ever-increasing energy demand with the increase in world population and economic development. Greenhouse gases resulting from the use of fossil fuels cause global warming and climate change. In order to ensure energy security and a sustainable future, the share of decarbonised energy sources, such as renewable solar, wind, wave, biomass, etc., in electricity generation is increasing rapidly. However, renewable resources provide energy in variable amounts and intervals depending on climatic conditions. For this reason, there are difficulties in integrating them into power transmission lines that require continuous energy, and energy storage systems with both high energy density and high-power density are needed. Chemical (batteries) and capacitive (supercapacitors) energy storage systems offer solutions for variable energy needs [1]. As an efficient electrochemical energy storage device, supercapacitors are environmentally friendly, have ultra-high power density, high energy density and excellent cycle stability, so they are expected to be widely used in future energy storage systems [2-4]. In energy storage systems, electrode material is the main component that determines the performance of the system. Improving the electrode interface by optimising parameters such as surface area, conductivity, porosity, etc. is key to the development of high-performance next generation energy storage devices. Activated carbons with large surface area and high electrical conductivity are the main electrode materials used in supercapacitors due to their low cost and easy availability [5]. Among many transition metal compounds, nickel-based materials are considered as promising positive materials for supercapacitors due to their economical, environmentally friendly and high theoretical specific capacitances [6,7]. It is well known that the morphology and structure of electroactive materials have a significant influence on the electrochemical behaviour. Therefore, many researchers want to develop different morphological NiS to design electrode materials with large surface area and rough surface to improve the wettability of active materials and short diffusion paths for electrolyte and charge transport [8,9]. In this study, NiS composite on hollow carbon nanorods was successfully synthesised by a one-step microwave-assisted hydrothermal method. During the synthesis process, hollow carbon nanorods were first obtained. Then, in the microwave synthesis system, a 1:1 ratio of Ni:S was synthesised in the presence of hollow carbon nanorods in a single step. When tested as electrode material for supercapacitors, the obtained HCN@NiS composite shows improved rate performance and impressive cycle stability.

II. Materials and Method

$Co(NO_3)_2.6H_2O$, $Ni(NO_3)_2.6H_2O$, $Na_2S.9H_2O$, ethanol and KOH were purchased from Sigma-Aldrich. All chemical reagents were of analytical grade and were used without further purification.

A. *Hollow carbon nanorod synthesis (HCN)*

$FeCl_3.6H_2O$ and urea were combined in 50 mL of water and heated at approximately 90-95°C for around 8 hours. Once the reaction was completed, the obtained material was washed three times with deionized water and dried. The product is β-FeOOH. A solution containing 0.63 g of β-FeOOH was added to tris buffer (10 mM; pH: 8.5) and mixed with dopamine. The mixture was stirred at 50°C for 24 hours. After the reaction, the product was obtained with centrifugation. The obtained material is polydopamine (PDA) coated iron oxide. To get hollow carbon material, the calcination process took place in a tube furnace under an Ar atmosphere in two stages: (i) with a heating rate of 1°C/min, raising the temperature to 400°C for 2 hours, and (ii) with a heating rate of 5°C/min, reaching 500°C for 2 hours. After cooling down to the room temperature, the product was etched with 2 M HCl to remove the core iron oxide. The last product is hollow carbon nanorods (HCNs) with large cavities.

B. *Synthesis of NiS on hollow carbon nanorod (HCN@NiS)*

Firstly, HCNs were coated PVP polymer. Then, PVP-coated HCNs were combined in 50 mL of water with $Ni(NO_3)_2.6H_2O$, hexametylenetetramine (HMT), and trisodium citrate. After 8 hours reaction at around 90-95°C, the mixture was cool down to the room temperature. $Na_2S.9H_2O$ was added to the mixture and it was placed in microwave for 20 min at 700 W. The final product was gained after centrifugation and named as HCS@NiS.

III. Results and Discussion

A. *Morphological Analysis*

SEM images of the samples at different magnifications are presented in Figure 1. From these SEM images, it is clearly seen that NiS materials are coated on the hollow carbon nanorods. The morphology of HCN@NiS material is rod-like nanostructure. The rod-like morphology of HCN@NiS allows the diffusion of electrolyte ions into the electrode more easily than other morphologies (flake and spherical), resulting in reduced ionic diffusion resistance.

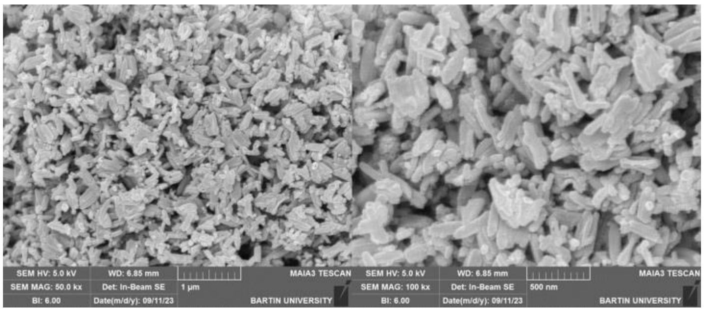

Figure 1. SEM images of HCN@NiS composite materials at different magnifications

Figure 2 shows the EDX spectrum of HCN@NiS nanocomposite. The EDX result confirms the presence of Ni and S in the HCN@NiS nanocomposite. The EDX spectrum shows that 8.9 wt% is Ni and 2.8 wt% is S.

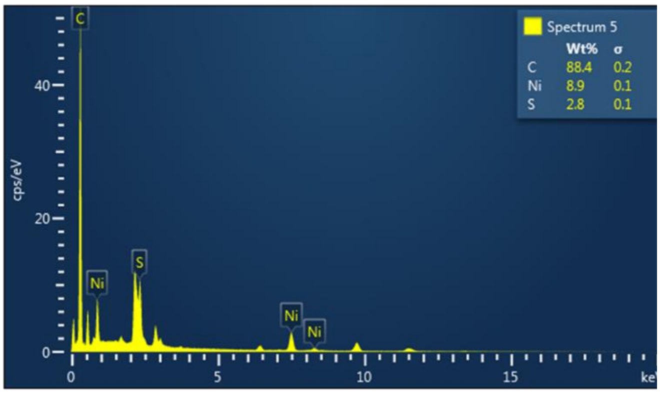

Figure 2. EDX spectrum of HCN@NiS composite material

B. *Electrochemical Analysis*

In order to obtain the best reaction time of the prepared composite materials, a three-electrode system with 3 mol L^{-1} KOH as electrolyte, platinum as counter electrode, Hg/HgO as reference electrode and HCN@NiS as working electrode was established. Firstly, the cyclic voltammetry test was performed under a voltage window of 0-0.4 V and different scan rates (10-100 mV s-1) as shown in Figure 3. All CV curves obtained have redox peaks. However, as the scan rate increases, the peak potential of the oxidation peak gradually increases while the peak potential of the reduction peak decreases. At high scan rates the CV curve shows a clear deformation, indicating the formation of electrode polarisation.

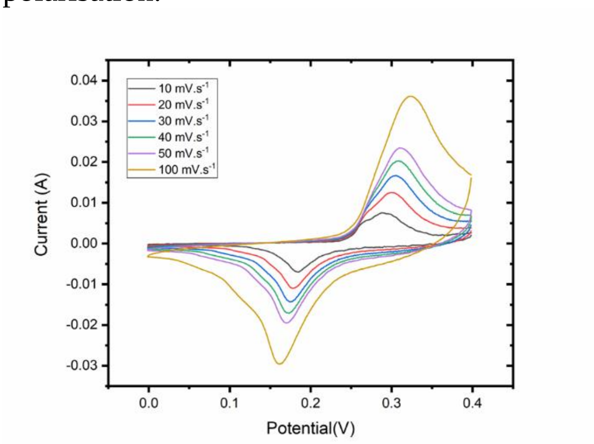

Figure 3. CV curves of HCN@NiS material at different scan rates (10-100 mV s^{-1})

The GCD test was performed on the HCN@NiS electrode at different current densities (1-10 mA cm^{-1}) as shown in Figure 4. Due to the Faraday reaction, all curves show a clear discharge platform, indicating the presence of capacitance.

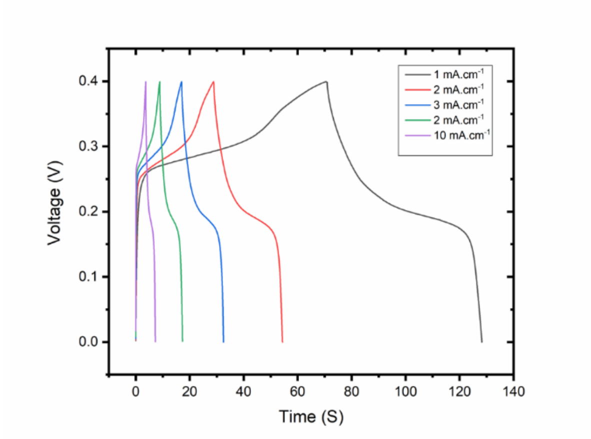

Figure 4. GCD curves of HCN@NiS at various current densities

IV. CONCLUSION

In this paper, hollow carbon nanorods were synthesised in a single step. Then, HCN@NiS material was synthesised by microwave-assisted hydrothermal synthesis in a fast, facile and inexpensive manner. Considering the defects of NiS (such as poor structural stability and poor specific surface area), an economical, efficient and binder-free HCN@NiS electrode was prepared. The results show that NiS was coated on the obtained hollow carbon nanorods. The obtained HCN@NiS material has a capacitance value of 0.15 F/cm^2 at a current density of 1 mA/cm^2. The HCN@NiS structure appears to be promising as a potential energy storage device.

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