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PREPARATION AND PHYSICAL CHARACTERIZATION OF SULFUR/CARBON BLACK COMPOSITE CATHODE MATERIAL FOR Li-S BATTERY

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Abstract - *Lithium sulfur batteries have been extensively nominated as one of the most capable candidate for next generation energy storage systems. However, it is still restricted owing to the low electronic conductivity of sulfur, volume expansion and polysulfide during shuttling. Herein, Sulur/Carbon black composite cathode material successfully prepared by ball milling method followed by heat treatment. The obtained composite cathode material was characterized for physical analyses using XRD, FTIR, RAMAN and SEM. From XRD analysis, the diffraction peak of the as-prepared sample displays the presence of elemental sulfur, which indicates the low content of carbon black has not been affected in its structure. SEM results reveal that the uniform distribution of sulfur in the S/CB composite. Hence, the uniform morphology of the prepared composite cathode material will enhance the electrochemical properties when it is used in the fabrication of the lithium sulfur battery.*

Key Words: **Lithium sulfur battery, cathode material, ball milling, composite, carbon black**

1. INTRODUCTION

A favorable alternative for the next generation energy storage device, lithium sulfur battery has fascinated more and more attentions over the past few years. Multi-electron redox reaction provides it with a high theoretical specific capacity of 1675 mAh g^{-1}, which is five times higher than those of traditional lithium ion batteries. Moreover, its low cost and eco-friendliness, compared by other cathode materials for secondary lithium batteries, are additional advantage to be considered [1-4]. In spite of these advantages, the practical applications of Li-S batteries are still hindered by the low conductivity of sulfur, dissolution of lithium polysulfide intermediates and volume expansion of active material during cycling process. All these disadvantages mainly cause the low Coulombic efficiency, low consumption of sulfur and quick capacity decay [5].

In order to overcome the dissolution of intermediate lithium polysulfides, numerous efforts have been devised, such as adding highly conducting carbons, conductive polymers and metal oxides [6, 7]. Among them, carbonaceous materials are considered as the ideal choice for the loading sulfur, such as porous carbons, carbon black, hollow carbon spheres, carbon nanotube/fibers, etc. which can not only growth the conductivity of sulfur, but also can improve the dissolution of lithium polysulfides into organic liquid electrolytes [5]. For example, Su et al. reported a sulfur/carbon black composite cathode synthesized by a facile in situ sulfur deposition method to deliver a high initial discharge capacity of 1116 mAh g^{-1} and a reversible discharge capacity of 777 mAh g^{-1} after 50 cycles. Because the carbon materials effectively increase the electrical conductivity of sulfur, thereby improve the utilization of active materials [8, 9].

Herein, the sulfur/carbon black composite material prepared via ball milling method. The carbon material in the composite can physically confine sulfur, accommodate volume expansion and improve the electronic conductivity of the cathode. The prepared composite material has been characterized for its structural and morphological analyses using XRD, FTIR, Raman and SEM.

2. EXPERIMENTAL WORK

The sulfur/carbon black composite was synthesized via ball milling process followed by heat treatment method. Sulfur and carbon black were taken in 7:3 ratios and manually mixed using mortar and pestle. The mixed powders were heated for 155 °C at 20 h using muffle furnace. Finally the sulfur/carbon black composite has been obtained and labeled as S/CB.

MATERIALS CHARACTERIZATION

A structural and morphological analysis of the as-prepared S/CB composite was identified by powder X-

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Ray diffraction (PANalytical XPERT-PRO with Cu Kα radiation), FTIR spectrophotometer (Thermo Nicolet 380 Instrument Corporation), Raman spectrometer (STR 500 Laser Raman spectrometer, SEKI, Japan) and Scanning Electron Microscopy (ZEISS).

3. RESULTS AND DISCUSSION

Structural studies

The crystal structures of the standard sulfur and as-prepared S/CB composite, was characterized by X-ray diffraction (XRD). As shown in Fig. 1, S/CB composite exhibits all the diffraction peaks well matched with the standard values of the F_{ddd} orthorhombic phase of sulfur (JCPDS no. 08-0247). From the XRD patterns, no new phase has formed in the S/CB composite during the synthesis process, because the high loading of sulfur in the composite.

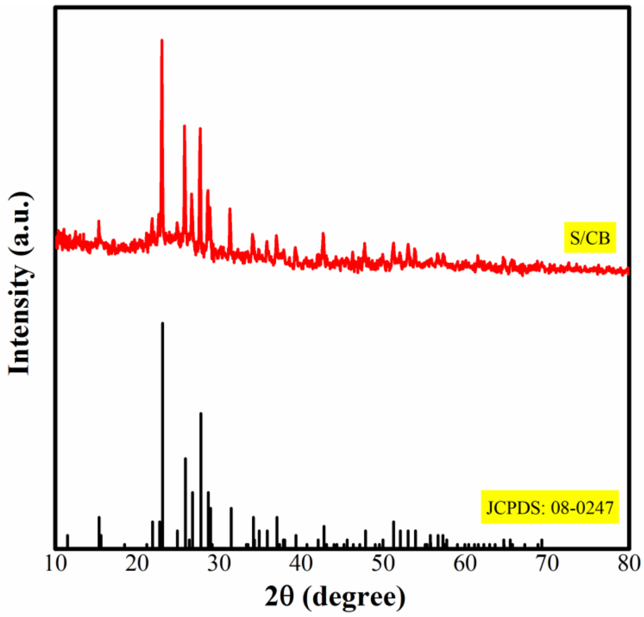

Fig -3.1: XRD patterns of Sulfur (JCPDS: 08-0247) and S/CB composite

Fig. 2 shows the Fourier transform-infrared spectra of the as-prepared S/CB composite in the wavenumber region of 4000 – 400 cm^{-1}. The vibrational bands at 1196 and 1634 cm^{-1} corresponds to the C-O and C=O stretching vibrations. The C-H bending vibration exist in the range of 643 cm^{-1} and the band at 2357 cm^{-1} belongs to ν (CH) stretching vibration.

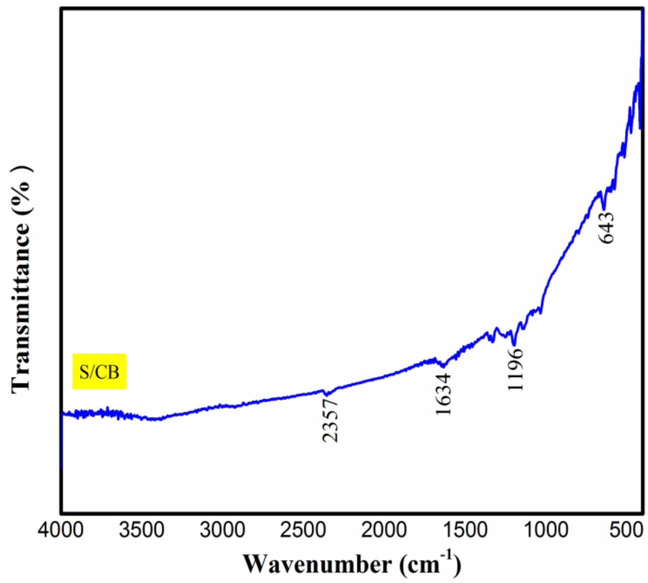

Fig -3.2: FTIR spectra of as-prepared S/CB composite

Raman spectroscopy is a powerful analytical tool for carbon materials to evaluate the degree of graphitic order. Fig. 2b shows two main peaks corresponding to the D and G bands in the range of 1336 and 1571 cm^{-1}, which could be originated from CB. The band at 475 cm^{-1} represents the Raman band of sulfur in the composite. From the Raman spectrum, it confirms the presence of sulfur and carbon in the as-prepared S/CB composite.

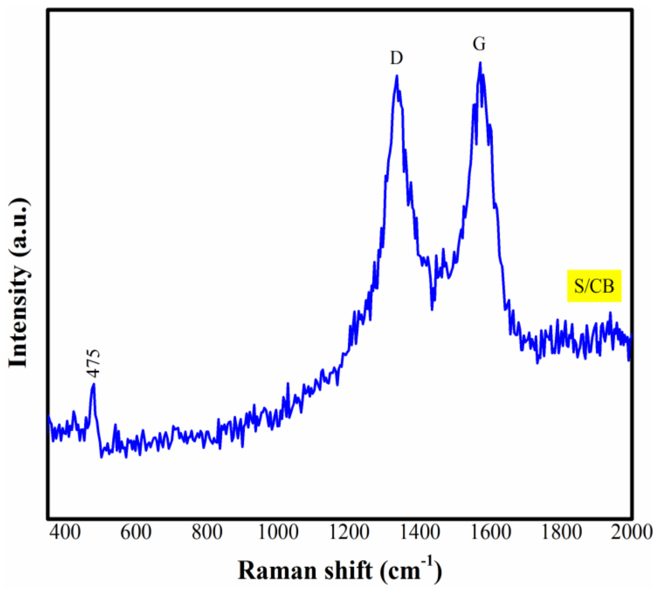

Fig -3.3: RAMAN spectra of as-prepared S/CB composite

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Scanning Electron Microscopy

Scanning electron microscopy is used to determine the morphology of the as-prepared S/CB composite shown in fig. 4. From SEM images, it reveals that most of the sulfur species were completely wrapped by the CB which may enhance the conductivity and the porous nature would allow the pathway of Li ions. From these, it can be clearly seen that morphology of the binary composite very well consistent with the Raman result.

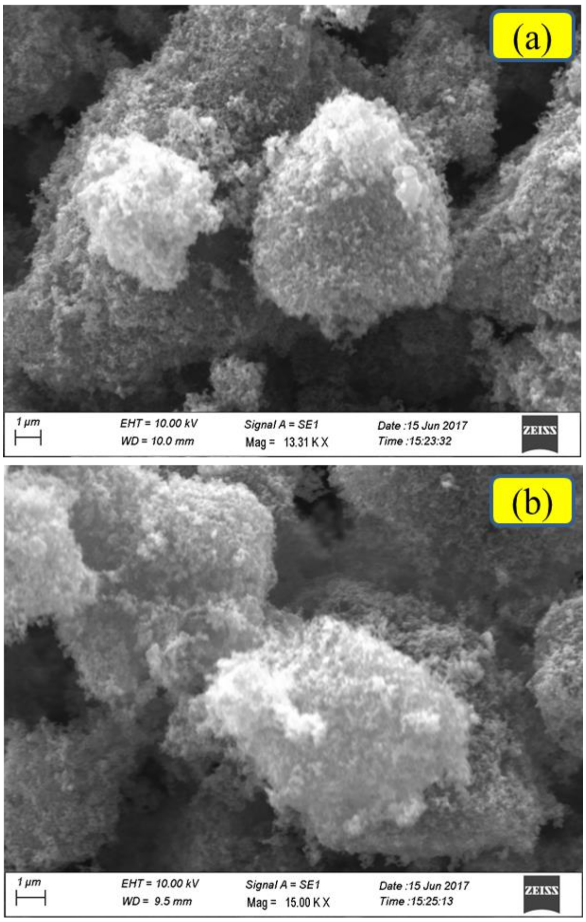

Fig : SEM images of as-prepared S/CB composite

4. CONCLUSIONS

The Sulfur/Carbon black composite material successfully prepared via ball milling process followed by heat treatment method. From the structural analysis, the diffraction pattern of the prepared composite material exhibits crystalline phase of orthorhombic sulfur. FTIR and Raman analysis confirms the presence of carbon content in the as-prepared composite. From SEM results, the composite cathode material attributed to the good dispersion of sulfur into the CB particles. From the above investigations, it could be concluded that the carbon material in the S/CB composite effectively enhance the electrochemical performance of the Li-S batteries for used as a cathode material.

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