



# Effect of sulfur content in a sulfur-activated carbon composite on the electrochemical properties of a lithium/sulfur battery



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## ABSTRACT

The content of sulfur in sulfur/activated carbon composite is controlled from 32.37 wt.% to 55.33 wt.% by a one-step solution-based process. When the sulfur content is limited to 41.21 wt.%, it can be loaded into the pores of an activated carbon matrix in a highly dispersed state. On the contrary, when the sulfur content is 55.33 wt.%, crystalline sulfur can be detected on the surface of the activated carbon matrix. The best electrochemical performance can be obtained for a sulfur electrode with the lowest sulfur content. The sulfur/activated carbon composite with 32.37 wt.% sulfur afforded the highest first discharge capacity of 1360 mAh g<sup>-1</sup> at 1 C rate and a large reversible capacity of 702 mAh g<sup>-1</sup> at 10 C (16.75 A/g).

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## 1. Introduction

Rapid advancement of portable electronic devices and electric vehicle technologies demands next-generation batteries with high capacities, high rate capabilities, and long cycle lives with a low cost [1,2]. The lithium/sulfur (Li/S) battery has attracted attention as one such next-generation batteries. The Li/S battery has a high theoretical specific energy density of 2600 Wh kg<sup>-1</sup>, assuming the complete reaction of sulfur with lithium to Li<sub>2</sub>S [2–4]. Sulfur has many advantages which include abundance in nature, low cost, and environmental friendliness [1,2,5,6]. However, Li/S batteries have serious problems, including low utilization of active material and poor cycle life because of the insulating property of sulfur and high solubility of lithium polysulfides in organic electrolytes [4,7]. To overcome the aforementioned problems, many methods have been attempted, such as wrapping sulfur with CNTs [8–16] or graphene [17–20], and combining sulfur with a conducting polymer [3,21–23]. One especially promising method for the improvement of Li/S batteries was the infiltration of sulfur into nanopores of mesoporous carbon [24–50]. The sulfur/carbon composite showed high sulfur utilization and a long cycle life due to the blocking of the dissolution of lithium polysulfides from

carbon mesopores and good conductivity of carbon [18,34,36,45,46,50].

The sulfur content in mesoporous carbon may affect the electrochemical properties of sulfur/carbon composite electrodes [27,29,30,35,37,40–42,46–50]. The theoretical maximum content of sulfur inside each pore can be calculated by multiplying the pore volume with the density of sulfur. When the sulfur content in the sulfur/carbon composite was higher than the maximum content infiltrated in the mesopore, the Li/S cell showed very poor cycling properties [27,41,46–48]. In general, the sulfur/mesoporous carbon composite with the lowest sulfur content had the highest first discharge capacity [27,30,35,37,40–42,46–50] and the highest rate capability [46]. However, Chen et al. [29] prepared sulfur/order mesoporous carbon (OMC) composites with sulfur contents ranging 50–75 wt.%. When the sulfur content was 55 wt.%, the S/OMC composite had a higher first discharge capacity of ca. 1190 mAh g<sup>-1</sup> compared to that of the 50 wt.% composite. Thus, the optimum content of sulfur for higher first discharge capacity is not yet clear.

Mesoporous carbon matrixes have been prepared through a series of complex processes with high cost [38,43]. Recently, our group employed a commercially available and inexpensive carbon matrix of activated carbon, and infiltrated sulfur into activated carbon by a one-step solution process [43].

In this study, we controlled the sulfur content in a sulfur/activated carbon (S/AC) composite using a simple solution process.

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The effects of sulfur content on the electrochemical properties were investigated through charge–discharge curve analysis and rate capability tests. Additionally, we discuss the physical properties of the composite and the optimum sulfur content in activated carbon.

## 2. Experimental

### 2.1. Preparation of the S/AC composite

The specific surface area (SSA) and pore volume of the activated carbon powder (phenol resin-based carbon, Kuraray Chemical Co.) were  $1696 \text{ m}^2 \text{ g}^{-1}$  and  $0.7932 \text{ cm}^3 \text{ g}^{-1}$ , respectively. From the value calculated on the basis of the pore volume of the AC, assuming the density of sulfur of  $2.07 \text{ g cm}^{-3}$ , the maximum sulfur content was determined to be 62.2 wt.% of the S/AC composite. The S/AC composite was prepared with various sulfur loading ratio such as high (H), middle (M) and low (L) by changing the carbon content. In the first step for preparation of the S/AC composite, 1 g of sulfur powder was dissolved in a dimethyl sulfoxide (DMSO) solvent heated by a mantle to  $90^\circ \text{C}$ . In the next step, the activated carbon powder was added into the solution and dispersed by stirring for 3 h. The weight of carbon varied from 0.33, 0.64, and 1.23 g. In other words, the weight ratio of sulfur (Aldrich Co.) to AC carbon powder were 3.00:1 (75.00 wt.%), 1.55:1 (60.78 wt.%), and 0.77:1 (43.50 wt.%), denoted as S/AC- $x$  ( $x$ =H, M, and L, respectively), wherein  $x$  represents the sulfur content.

The color of solution changed from yellow to black during the preparation of the S/AC composite. It is believed that this color change was evidence of the sulfur impregnation into the AC matrix. The solution was cooled to room temperature and became transparent, which meant recrystallization of sulfur in activated carbon. The S/AC composite powder was collected by a centrifuge and washed with ethanol several times to eliminate the residual DMSO. The composite was then dried in vacuum at room temperature to evaporate ethanol and finally heat in an oven at  $100^\circ \text{C}$  for 24 h.

### 2.2. Electrochemical measurements

A sulfur electrode consisted of an active material (S/AC composite), a conductive agent (Super-P), and a binder (poly(vinylidene fluoride)) with a weight ratio of 60:20:20. First, these three components were homogeneously mixed in an *N*-methyl-1,2-pyrrolidone (NMP) solvent using a planetary ball milling apparatus. The mixed slurry was coated on an aluminum foil and dried in an oven at  $60^\circ \text{C}$  for 24 h to remove the solvent. Lithium metal was used as a counter electrode and a Celgard 2400 microporous membrane was used as a separator. The electrolyte used was 0.5 M of dissolved bis(trifluoromethane) sulfonamide lithium (LiTFSI, Aldrich Corp.) in 1,2-dimethoxyethane (DME) and 1,3-dioxolane (DOXL) (4:1 by volume ratio). The Swagelok-type cells were assembled in an argon-filled glove box. The S/AC composite was cycled at C rates varying from 1 C to 10 C through a battery test system (WBCS 300 L, WonA Tech). The cut-off potentials for discharge and charge were 1.5 V and 2.8 V, respectively.

### 2.3. Materials characterization

The morphology of the S/AC composites was observed using a scanning electron microscope (SEM, Jeol, JSM-6380LV). The sulfur content of the S/AC composites was determined by thermogravimetric analysis (TGA, TA Instruments, Q50) performed in a nitrogen atmosphere with a heating rate of  $10^\circ \text{C min}^{-1}$ . The XRD patterns of the S/AC composites in a  $2\theta$  range from  $20^\circ$  to  $70^\circ$  were collected by X-ray diffractometry (XRD, Bruker, D8 Advance). Specific surface areas of the S/AC composites and the AC carbon powder were calculated using the Brunauer–Emmett–Teller (BET, Micromeritics, ASAP-2020M). The thermal stability of the S/AC composites was evaluated with a differential scanning calorimeter (DSC, TA Instruments, Q20) at a heating rate of  $5^\circ \text{C min}^{-1}$  from 50 to  $140^\circ \text{C}$ .

## 3. Results and discussion

Fig. 1 presents the SEM images of the raw activated carbon powder and S/AC composite powders with various sulfur contents.

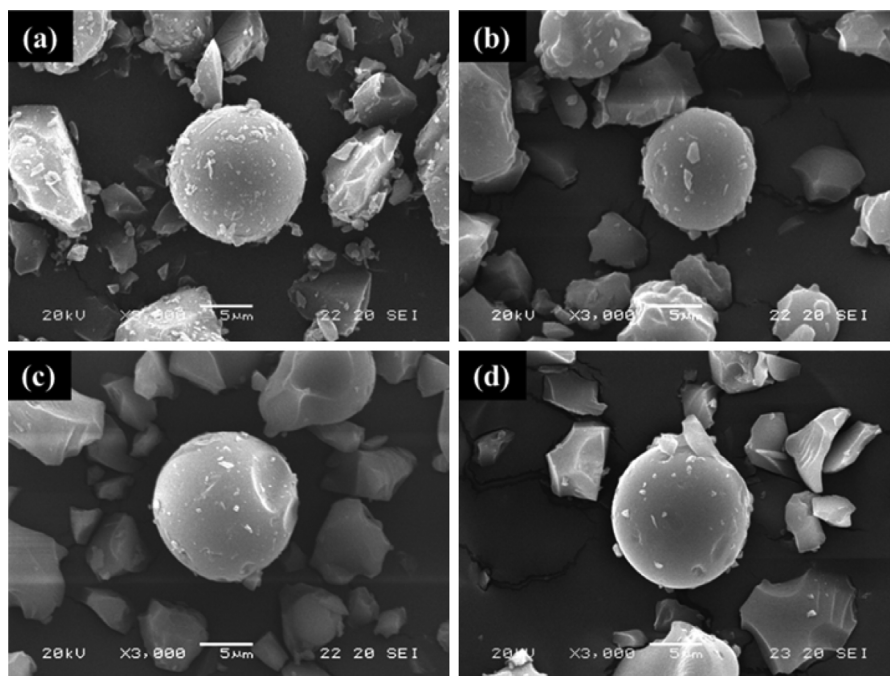


Fig. 1. SEM images of the AC powder and S/AC composite powder with different sulfur contents. (a) AC powder, (b) S/AC-L powder, (c) S/AC-M powder, and (d) S/AC-H powder.

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