

Porous Activating Carbon made from Enormous Coffee Waste

Zohaib Hassan Nawaz*

Department of Material Science, Nano Material Research Center, Pakistan

Abstract

Using a carbon-based material as an interlayer between the cathode and separator, a novel strategy has been proposed for enhancing the performance of lithium-sulfur batteries (LSBs). This technique reduces the cross-over of lithium polysulfides (LiPS) to the anode, thereby increasing the sulfur cathode's reutilization. Coffee waste was used as a source of carbon in this study, and potassium hydroxide (KOH) was used as an activating agent. The reaction temperatures ranged from 500 to 800°C. The AC samples' specific surface areas, micro-surface areas, and micro-pore volumes gradually increased with the heating temperature. In particular, the capacity and cycling performance of the AC sample, which was made at 800°C and used as a functional separator for LSB, were both improved, and the LiPS shuttle effect was reduced.

Keywords: Lithium sulfur batteries; Separator; Coffee waste; Activated carbon; Shuttle effect; Physical adsorption

Introduction

Due to their high theoretical capacity of 1675 mAh g⁻¹, high energy density of 2600 Wh kg⁻¹, and abundant sulfur resource as an active material, lithium-sulfur batteries (LSBs) are promising energy sources. They are capable of converting chemical energy into electrochemical energy and vice versa via electrochemistry [1]. However, the disadvantages of LSBs include the dissolution of lithium polysulfides (LiPS) during the conversion process and the electrical isolation of the sulfur cathode [2]. Sulfur cathodes also have a low electrical conductivity of 5 1030 S·cm⁻¹ at 25°C. LSB problems can cause high polarization, low Coulombic efficiency, or fast capacity decay [3]. Particularly, the extremely solvent-like LiPS (Li₂S_x) (4 x 8) that are produced during charging and releasing in electrolytes reduces the amount of dynamic materials at the cathode. The LSB's performance is affected when insoluble Li₂S or Li₂S₂ insulating layers form on the separator and anode's surface when LiPS covers them. Additionally, the dissolved LiPS is transported to the Li anode via the separator by the so-called "shuttle effect" of LiPS [4].

As a result, a novel strategy to boost LSB performance has been proposed that uses a carbon-based material as a practical cathode-to-separator separator [5]. It increases sulfur cathode reutilization and reduces LiPS cross-over to the anode. By demonstrating LiPS's high electrical conductivity and further developed actual adsorption properties, the physical adsorption capacity of carbon-based materials, which is based on van der Waals forces, may also be able to prevent the diffusion of LiPS during the initial cycle. Multi-walled carbon nanotubes and decreased graphene oxide are two examples of useful carbon-based separator materials. Carbons with high unambiguous surface regions and micropores and mesopores commonly help in Li⁺ particle dispersion. Template carbon and activated carbon's specific surface areas, pore volumes, and microsurface areas were compared [6]. In particular, various electrochemical processes have utilized carbon arranged from coffee grounds. Carbons made from coffee waste (Coffee extract, CAS 84650-00-0) have a number of advantages, including their low price, high electrical conductivity, clearly defined porous structure, and relatively large surface area. Shen and colleagues found that the Li-S cell made from activated corn-derived puffed corn performed better electrochemically [7]. When coconut shell carbon was applied to the Li-S cell, a high release limit was also observed. Enacted carbon (AC) tests were only prepared in this concentrate using potassium hydroxide (KOH) as an initiator and espresso waste as a carbon source

at response temperatures ranging from 500 to 800°C. The AC samples contained amorphous crystals with well-formed porous structures and high specific surface areas [8]. Due to its porous structure and large specific surface area, the 800°C AC sample, which was utilized as a functional separator material for LSBs, delivered the highest capacity and improved cycling performance [9].

Experimental

Synthesis of Activated Carbons (ACs) Derived from Coffee Waste

To arrange enacted carbon (AC), an initiation cycle was used with KOH (95%, SAMCHUN, Seoul, Korea) powder and espresso waste (Mixed Smooth Way, CAS 84650-00-0). In particular, a mortar was used to evenly mix 2 grams of coffee waste and 3 grams of KOH flakes. The mixed powder was heated for 30 minutes in an N₂ gas atmosphere in an alumina tube [10]. After being washed with 2 L deionized (DI) water to remove impurities like K and K₂CO₃, the heated samples were dried in a vacuum oven at 50°C for 12 hours to achieve a pH of 7.0.

MWCNT/S as a Cathode

The MWCNT/S cathode for a functional separator was made from a multi-wall carbon nanotube (MWCNT, GRAPHENE SUPERMARKET, Ronkonkoma, NY, USA) and sulfur (S, 99.98%, Sigma Aldrich, St. Louis, MI, USA). 2 g of MWCNT powder was acid-treated by stirring it in HCl (35 percent, 10 mL) and HNO₃ (60 percent, 30 mL), both. The acid-treated MWCNT was then dried for 12 hours in an oven at 50°C after being washed several times with DI water. MWCNT can use this to better adsorb polar polysulfides. MWCNT and sulfur were ground together to form a 2:8 weight mixture. In an autoclave lined with Teflon, the mixture was heated for 12 hours at 155°C [11].

*Corresponding author: Zohaib Hassan Nawaz, Department of Material Science, Nano Material Research Center, Pakistan, E-mail: mahrzohaib53@gmail.com

Received: 01-Nov-2022, Manuscript No: JMSN-22-80936; **Editor assigned:** 04-Nov-2022, Pre-QC No: JMSN-22-80936 (PQ); **Reviewed:** 18-Nov-2022, QC No: JMSN-22-80936; **Revised:** 25-Nov-2022, Manuscript No: JMSN-22-80936 (R); **Published:** 30-Nov-2022, DOI: 10.4172/jmsn.100057

Citation: Nawaz ZH (2022) Porous Activating Carbon made from Enormous Coffee Waste. J Mater Sci Nanomater 6: 057.

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Materials Characterization

The crystal structure was confirmed by X-ray diffraction (XRD, D2, USA) employing a Ni filter and a Cu K X-ray source ($\lambda = 0.154$ nm). A Nd:YAG laser with a wavenumber between 800 and 2000 cm^{-1} ($\lambda = 532$ nm) was used for Raman spectroscopy (Leica DM2700 M, Leica Microsystems, Wetzlar, Germany). Energy dispersive X-ray spectroscopy (EDX, XFlash Detector 6) and field emission scanning electron microscopy (FE-SEM, GeminiSEM 300, ZEISS, Oberkochen, Germany) were used. An N_2 adsorption/desorption analyzer (Micromeritics ASAP 2020) was used to characterize the specific surface areas and pore structures of the samples by utilizing the BET (Brunauer–Emmett–Teller) theory and the BJH (Barrett–Joyner–Halenda) method [12]. Thermal gravimetric analysis (TGA, TGA 2-XP1, METTLER TOLEDO, Columbus, OH, USA) at temperatures ranging from 25 to 800°C in an atmosphere of air confirmed the presence of impurities in the activated carbon samples. The degassing temperature in the air was 200°C. The samples' Li_2S_6 adsorption capacity was measured using UV/VIS spectroscopy (UV/VIS spectrophotometer, V-650, JASCO, Oklahoma City, OK, USA).

Electrochemical Measurements

To create a functional separator structure for LSB, the AC sample was mixed at a weight ratio of 9:1. A Celgard 2400 separator made of polypropylene (PP) was coated with N-methyl-2-pyrrolidone (NMP, Samchun), which contains polyvinylidene fluoride (PVDF, Alfa Aesar, Ward Hill, Massachusetts, USA) [13]. The coated separator was used after being cut to a diameter of 19 mm and dried for 12 hours at 50°C in a vacuum oven. The Air conditioner test had a mass stacking of 0.70 mg cm^{-2} on the covered separator. In addition, the LSB cathode was made by homogenizing MWCNT/S (80% by weight) in NMP with super P (100% by weight, Alfa Aesar) and PVDF (100% by weight) as the conducting agent and binder, respectively. The slurry was applied to aluminum foil using a doctor's blade. After 48 hours at 50°C in a vacuum oven, the foil was cut to 13 mm in diameter [14]. For LSBs, sample-coated polypropylene served as a functional separator, and coin cells (size 2032, Hohsen Corporation, Osaka, Japan) with a sulfur cathode on aluminum foil and a Li metal anode were assembled in an Ar-filled glove box. The load level of sulfur was between 2.0 and 2.1 mg cm^{-2} . The electrolyte was prepared by diluting 1,3-dioxolane (DOL, TCI chemical, Tokyo, Japan) and 1,2-dimethoxyethane (DME, TCI Chemical) in a volume ratio of 1:1 with 1.0 M lithium bis(trifluoromethanesulfonyl)imide (LiTFSI , Aldrich) and 0.2 M LiNO_3 (Alfa Aesar). A battery tester (WBSC3000Le, WonATech Co. Ltd., Seoul, Korea) was used to generate charge–discharge curves at a current density of 0.5 C in the range of 1.8–2.6 V vs. Li/Li^+ . The ratio of S to electrolyte was 15 L mg^{-1} [15].

Characteristic charge-discharge curves of Li/S cells with the AC samples serving as functional separators and a pristine PP separator were measured at a current density of 0.1 C in the potential range of 1.8–2.6 V vs. Li/Li^+ . At voltages of 2.0–2.1 and 2.2–2.4, all cells exhibit two plateaus during discharge. Electrochemical reduction of solid-state S8 to long chain polysulfide (4 x 8) results in the first plateau, which is 25% of the theoretical capacity of sulfur. In the second plateau, long-chain polysulfide (4 x 8) is electrochemically reduced to short-chain polysulfide (Li_2S_x (1 x 2)), which contains 75% of the theoretical capacity of sulfur. In contrast, during charging, the oxidation of Li_2S_2 and Li_2S in the first plateau results in the formation of soluble long-chain polysulfide (4 x 8), and the oxidation of soluble long-chain polysulfide (4 x 8) in the second plateau results in the formation of solid-state S8, both of which are identical to peaks in the CVs that are

associated with oxidation and reduction. Initial discharge capacities of 738, 897, 944, 977, and 992 mAh g^{-1} were found for the Li/S cells with PP, AC-500, AC-600, AC-700, and AC-800, respectively. When compared to the PP separator, the high electrical conductivity of AC samples as functional separators may result in increased capacities and redox reaction rates. Particularly, the porous structure of the AC samples has the potential to boost electrochemical performance by increasing the number of reaction sites and facilitating the movement of Li^+ ions. The capacity of the Li/S cells increased as the heating temperature increased. The enhanced electrochemical reaction that is associated with LiPS adsorbed in porous carbon structures may be caused by the increased specific surface areas, micro-surface areas, and micro-pore volumes with increasing heating temperature. As a result, both in terms of specific surface area and meso-surface area, AC-800 had the highest discharge capacity. The Li/S cells' cycling performance was measured after 100 cycles at 0.1 C. The discharge capacities of the Li/S cells equipped with AC-500, AC-600, AC-700, and AC-800 at 100 cycles were 610, 645, 698, and 767 mAh g^{-1} . The retentions of the Li/S cells equipped with AC-500, AC-600, AC-700, and AC-800 at 100 cycles were 68%, 68%, 71%, and 77%, respectively. At a higher current density of 0.5 C, the Li/S cells' cycling performance was recorded for 100 cycles with a Coulombic efficiency of less than 100%.

Conclusion

At various response temperatures, the air conditioner tests were planned with KOH as the actuation agent and espresso waste as the carbon source. The AC samples had amorphous crystals with large specific surface areas and well-defined porous structures. With increasing heating temperature, the specific surface areas, micro-surface areas, and micro-pore volumes of the AC samples gradually increased. The highest capacity and improved cycling performance of the AC sample, which was made at 800°C and used as a functional separator material for LSB, suppressed the LiPS shuttle effect due to its improved porous structure.

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