

# Lithium-Sulfur Battery: The Review Of Cathode Composite Fabrication Method

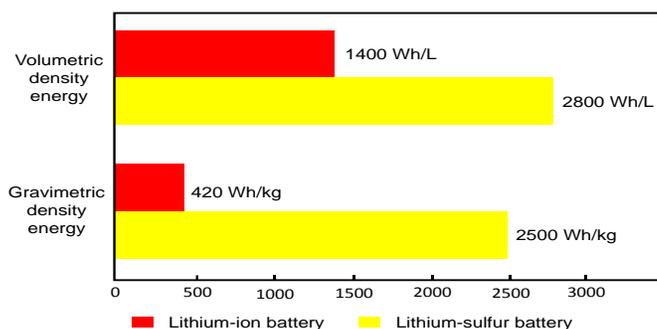
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**Abstract:** Nowadays, the energy demands increase along with the enhance lifestyle and the increase of population, so that alternative energy sources are needed to meet the demand. Several renewable energy has been developed, but the low capacity energy storage system makes the energy that has been produced is partially lost. Lithium-sulfur battery is one of a promising energy storage system due to its high energy density, inexpensive material, and abundant in nature. However, the current problem despite the high energy density is the low life cycles due to the dissolution of sulfur into the electrolyte. Research on lithium-sulfur batteries was carried out in 1962 by Hebert et al., but it has some disadvantages, such as a low discharge capacity and short cycle times. Various studies have been developed to solve these problems. In this review, we will discuss the electrochemical process on lithium-sulfur batteries and the method of a lithium-sulfur battery cathode manufacturing to give information to the researcher about the recent development of lithium-sulfur cathode manufacturing. The present review will discuss both physical and chemical manufacturing methods.

**Index Terms :** composite, method, Lithium-Sulfur, batteries

## 1. INTRODUCTION

Along with the increase in the population of the human make the high demand for energy sources, the renewable energy development such as solar and wind that is environmentally friendly and renewable is urgently needed to replace conventional energy sources [1]. But, with changes in uncertain natural conditions, an energy storage system is required to store the energy so it can be used at any time to supply the energy demand for both homes and industries. In this case, rechargeable batteries have an important role as one of the media that can be used for storing energy. But unfortunately, the lithium-ion battery used today has an energy density that is not too high [2]. These limitations become one of the obstacles to applying batteries for various applications [3]. Therefore it is necessary to develop battery technology that has a higher energy density than current lithium-ion batteries.



**Fig. 1.** Graph of the comparison energy density between lithium-sulfur vs. Batteries [4, 5].

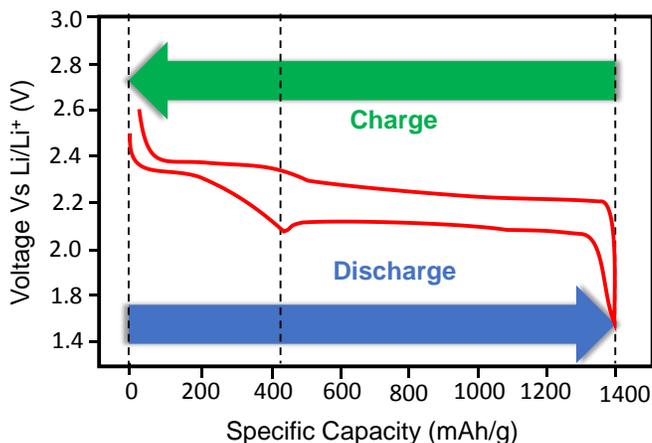
Lithium-sulfur battery is a kind of promising rechargeable battery type because it has not only a high theoretical energy density but also sulfur is a material that is easily found in nature, non-toxic, and inexpensive [6]. The difference between lithium-ion batteries and Lithium-sulfur battery is the difference in the energy storage mechanism. Lithium-ion batteries work by intercalation of lithium ions contained in the cathode material (graphite anode and lithium metal oxide cathode) so that the energy density is theoretically  $\sim 420 \text{ W h kg}^{-1}$  or  $1400 \text{ W h L}^{-1}$  (fig. 1) [4]. Whereas for lithium-sulfur batteries in addition to intercalating lithium ions, there is a reaction on the cathode (sulfur) side. The reaction that occurs at the cathode side results in a battery having a high energy density of  $\sim 2500 \text{ W h kg}^{-1}$  or  $2800 \text{ W h L}^{-1}$  (fig. 1) [5]. Research on lithium-sulfur batteries was carried out in 1962 by Hebert et al., but there are drawbacks to these batteries, such as low discharge capacity and short cycle times [7]. Research on lithium-sulfur batteries began to show an increase since 2009 when Nazar et al. succeeded in making carbon-sulfur mesoporous cathode materials that have high energy densities. Since then, research on lithium-sulfur batteries began to increase rapidly [8]. But the development of these batteries is still experiencing obstacles in the low life cycle [6]. Various studies have been developed to solve these problems. To support the development of this battery, the information about the research of this battery should be explored. In this review, we will discuss the electrochemical process on lithium-sulfur batteries and the methods of a lithium-sulfur battery cathode manufacturing to give information to the researcher about the development of lithium-sulfur cathode manufacturing. The present review will discuss both physical and chemical manufacturing methods.

## 2. ELECTROCHEMICAL PROCESS ON LITHIUM-SULFUR BATTERIES

Basically, a lithium-sulfur battery has a chemical reaction  $\text{S}_8 + 16\text{Li} \leftrightarrow 8\text{Li}_2\text{S}$ . Even though it looks simple, in reality, the chemical reactions that occur during the charge/discharge process on lithium-sulfur batteries are more complicated (fig. 2) [4]. During the discharge process, sulfur dissolved in the electrolyte will occur in a litigation process to form a long chain of lithium polysulfides ( $\text{S}_8 \rightarrow \text{Li}_2\text{S}_8 \rightarrow \text{Li}_2\text{S}_6 \rightarrow \text{S}_4$ ). This process contributed to increasing the theoretical

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battery capacity by  $\sim 420$  mAh/g (25%). Then, the chain will be broken into a short lithium sulfide chain ( $\text{Li}_2\text{S}_4 \rightarrow \text{Li}_2\text{S}_2 \rightarrow \text{Li}_2\text{S}$ ). This process contributes to increasing battery capacity by  $\sim 1260$  mAh/g (75%) [9].



**Fig. 2.** Chemical reaction on lithium-sulfur batteries during the charge/discharge process [4].

### 3. PHYSICAL METHODS

In general, sulfur material has the cyclic  $\alpha$ -octasulfur form ( $\alpha\text{-S}_8$ ) with an orthorhombic structure. In addition, sulfur has melting and boiling points around  $115^\circ\text{C}$  and  $444.6^\circ\text{C}$  [10]. Based on its physical properties, several methods can be used in making cathode materials for lithium-sulfur batteries.

#### 3.1. Ball-milling

The ball-milling method is a method used to smooth and mix material in a closed container. The ball-milling method is not suitable for making high-performance cathode materials. This is because the material produced has a weak bond between the active material and the host. Therefore, this method is usually only for mixing materials. Xi et al. reported their research about the fabrication  $\text{FeS}_2/\text{FeS}/\text{S}$  composites using the continuously ball-milling method. They used planetary ball mill at a speed of 350 rpm for three h to mix the sodium sulfide nonahydrate and sublimed sulfur, and then three h at the same speed to mix ferric trichloride hexahydrate and previous material. From the experimental results obtained that the battery has a capacity of 1044.7 mAh/g with Coulombic efficiency of around 100% until 30 cycles and reaches a discharge capacity of 941.3 mAh/g after 200 cycles [11]. Zhang et al. also reported their research about the fabrication of sulfur on carbon nanofiber and vertical graphene composite. The planetary ball mill at a speed of 300 rpm for 10 hours to mix the materials. This research showed remarkable rate-performance and excellent cycling stability with a specific capacity of 666 mAh/g after 300 cycles [12]. On the other hand, Soni et al. also reported their research. The graphite and sulfur were mixed with ball-milling in an open atmosphere at a speed of 500 rpm and different times (590, 1725, and 2880 minutes) [13].

#### 3.2. Melt-Diffusion

Melt-diffusion is a method that utilizes the physical properties of sulfur itself, which has a melting point of  $\sim 115^\circ\text{C}$  [10]. This method uses heat regulated around  $155^\circ\text{C}$

to mix sulfur with the host material. The entry of sulfur in the host pore is due to capillarity when the sulfur melts, which then forms nanocrystals, which have a strong bond to the host material during the cooling process. However, there is a problem in using this method, where the sulfur is still easily dissolved in electrolytes [14]. Rajkumar et al. reported their research about Sulfur/nitrogen-doped graphene using the melt-diffusion method. The composites were at a temperature of  $155^\circ\text{C}$  for 20 h without any special atmosphere. The result showed that the battery has a discharge capacity of 1135 mAh/g at the first cycle and 687 mAh/g over the 50<sup>th</sup> cycle at 0.1C [15]. Arie et al. also reported their research about the use of waste tea-sulfur composite using melt-diffusion. The porous carbon produced from waste tea was mixed with sulfur by the heating process at  $155^\circ\text{C}$  for ten h. The result showed that the porous carbon from waste tea could accommodate the sulfur until 60wt%. The specific capacity of the battery about 1048 mAh/g at 0.05C at the first cycle and 627 mAh/g after 100 cycles [16]. On the other hand, Huang et al. reported the synthesis of hollow  $\text{FePO}_4$  nanospheres, reduced graphene oxide, and sulfur composites. This composite was synthesized using the classic melt-diffusion method, and the process was done in a vacuum glass tube with the heating treatment at  $160^\circ\text{C}$  for 12 hours. The composite showed a good specific capacity of 1256 mAh/g at 0.1C for the first cycle and 582 mAh/g for 1000 cycles with a capacity decay rate of 0,037% per cycle [17].

#### 3.3. Precipitation

The precipitation method is one of the methods used to produce a precipitate derived from reactions that occur in a solution. Sulfur is a nonpolar material that can be dissolved in a nonpolar solution and can be re-precipitated into sulfur particles by adding a polar solution [18]. Wadekar et al. Used a precipitation method to produce sulfur graphene oxide. The graphene oxide solution was mixed with the DES solution (containing sulfur compound), and the reaction mixture was refluxed at  $90^\circ\text{C}$  for six h. The result showed that the cathode material had a maximum specific capacity of 1265 mAh/g at the initial cycle and 903 mAh/g after 100 cycles [19]. Sun et al. made a sulfur/Superaligned carbon nanotube (S-SACNTs) composite by utilizing precipitation method. Sun dissolves sulfur in ethanol (nonpolar solvent) and is sonicated. After that, DI water (polar solvent) is added so that the sulfur settles particles and is deposited on the surface of SACNT. From the experimental results, it was found that the S-SACNTs composite had a high discharge capacity of 1071 mAh  $\text{g}^{-1}$  and good performance (1006 mAh  $\text{g}^{-1}$  at 2C, 879 mAh  $\text{g}^{-1}$  at 10C with Coulombic efficiency reaching  $\sim 100\%$  [18].

#### 3.4. Vapor-phase Deposition

The vapor-phase infiltration method is a method that utilizes the vapor phase of the active material to be inserted in the host material. In this case, sulfur is a material that is easily made in the gas phase because it has a low enthalpy of evaporation ( $\sim 2.5$  kcal  $\text{mol}^{-1}$ ) and sublimation ( $\sim 2.9$  kcal  $\text{mol}^{-1}$ ) [20]. This method produces a strong bond between sulfur and host material. In addition, because the process takes place in the vapor phase, sulfur more easily enters the pore than the host material itself. Wu et al. made carbon /  $\text{Co}_3\text{O}_4$  hollow microspheres/sulfur composite using this

method. The composite was gotten by mixing the C/Co<sub>3</sub>O<sub>4</sub> and sublimed sulfur, and the mixture was given ultrasonication treatment to achieve complete volatilization. The sulfur content of this composite up to 67.3%. The result showed that the specific discharge capacity of 1171.6 mAh/g at 0.2C and excellent longterm stability over 1000 cycles with low decay rates of 0.076% [21]. Li et al. reported on their research on the use of the vapor-phase infiltration method. They placed the sulfur material and 3D CNT foam in a sealed small vessel. Then the infiltration process is carried out by heating the vessel at 1750C so that when the pressure reaches saturation, the sulfur will turn into a vapor phase. With this process, a CNT sulfur-3D cathode battery has a specific capacity of 1039 mA h/g at 0.1C [22]. On the other hand, Zheng et al. reported their research on the use of sulfur inserts in cracks in the graphene oxide layer. Zheng mixed sulfur with GO powder and heated it to a high temperature of 600<sup>o</sup>C under vacuum. The purpose of the heating is to reduce GO and break the S<sub>8</sub> molecule into S<sub>2</sub>. The S<sub>2</sub> molecule will enter between the rGO layer. With this process, the rGO-S cathode has a capacity of around 880mAh/g for more than 220 cycles [23].

### 3.5. Dissolution-Crystallization

In general, the Dissolution-crystallization method utilizes a shaft or gap in the host material to insert the active material. The difference with other methods is that the active material, in this case, sulfur is first dissolved in a solvent. Based on the theory, sulfur can be dissolved in nonpolar solutions such as carbon disulfide (CS<sub>2</sub>), dimethyl sulfoxide (DMSO), tetrahydrofuran (THF) and toluene [14]. Vimal et al. reported this method in their research about the production of sulfur-co-polymer porous long carbon nanotube composite. In their research, the sulfur powder was dissolved in DMSO at 120<sup>o</sup>C for 1 hour, and then the DEB was added in sulfur and DMSO solution at 165<sup>o</sup>C for 2 hours to get sulfur-copolymer. The result showed that the composite had a high discharge capacity of 1040 mAh/g at 0.5C in the first cycle and 610 mAh/g after 200 cycles [24]. Iskandar et al. also used this method to synthesis sulfur, PVP, graphene oxide composite. The sulfur powder was dissolved in Sodium Hydroxide solution at 80<sup>o</sup>C for 1 hour. After that, the solution was precipitated by L-ascorbic acid solution in the graphene oxide solution. This method could accommodate up to 50% sulfur in cathode materials [6]. Chen et al. reported research on the manufacture of monodispersed sulfur nanoparticles on the surface of reduced graphene oxide (rGO). Chen added sulfur to ethylenediamine (EDA) to form the S-EDA precursor. Then the S-EDA precursor is dripped slowly on the rGO dispersion. The experiments show that sulfur particle size is around 5 nm, and the batteries using the rGO-S composite have a capacity of 1672 mA h/g at 0.1C [25].

## 4. CHEMICAL METHODS

sulfur is a material that can not be dissolved in water. Sulfur can only dissolve in some organic solvents such as carbon disulfide (CS<sub>2</sub>), dimethyl sulfoxide (DMSO), and toluene. Therefore the compounds containing sulfur were used, which are easily dissolved in various solvents in these chemical manufacturing methods. Based on its chemical

properties, several methods can be used to make sulfur-based cathodes.

### 4.1. Chemical Deposition

The chemical deposition is one method that can be used to obtain sulfur particles. This method is done by adding hydrochloric acid (HCl), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), or another acid to produce the compounds containing sulfur. In this case, the compounds that are often used to become sources of sulfur are sodium sulfide (Na<sub>2</sub>S) and sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>). Whang et al. reported in their research about the use of hydrochloric acid (HCl) to get the sulfur compound. The raw material for the sulfur source was sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) that was dissolved into deionized water. The HCl was added drop by drop for 2 hours. In this research produced sulfur/CoOOH composite that had an ultrahigh sulfur content of 91.8wt%. On the other hand, the composite also had a high specific discharge capacity of 115.1 mAh/g for the initial charge and 858.8 mAh/g for 100 cycles [26]. Guo et al. also used sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) as a source of the sulfur compound. Hydrochloric acid (HCl) was used to break down the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> to obtain the element sulfur. In this case, Guo added PVP as a sulfur coating. From the experimental results obtained that the battery has a stable capacity of 600 mAh/g at 2C for 150 cycles [27]. On the other hand, Jeong et al., in their research, also reacted to the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and acid solution to obtain sulfur compound. Similar to Guo, their research made a composite that sulfur was coated by PVP in the form of a yolk-shell with nano size. The difference of this research was used sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) to breakdown the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. From the experimental results, it was found that the sulfur added to PVP has a homogeneous size around 300 nm and has a capacity of 765 mAh/g at 0.2C [28].

### 4.2. Electrochemical Deposition

Electrochemical deposition is one method of synthesis in which the material is obtained by precipitating an ion solution on the surface of a conductive plate (electrode). Electrochemical deposition is an interesting method to be developed because it is inexpensive, fast, has high purity, and produces many substrate deposits. This method requires two electrodes and an electrolyte solution. Zhang et al. reported their research on making mesoporous carbon (MC) / sulfur by electrochemical deposition synthesis methods. Zhang used three electrodes with a size of 2 x 2 cm. Pt flakes as working electrodes, cathode connecting Pt wire, and saturated calomel electrode (SCE) as reference electrodes. The electrolyte solution that was used was a mixture of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), potassium hydroxide (KOH), Sulfourea (as a source of elemental sulfur), and DI water. Composites were obtained from adding MC to the electrolyte, which is then given a constant voltage of 1.0 V. During the electrodeposition process, an infiltration of Sulfourea molecules occurs in the MC, and then in-situ turns into sulfur particles. Batteries with mesoporous carbon / sulfur (MCS) composite electrodes have a specific capacity of 1160 mAh/g at 0.1C and 857 mAh/g after 200 cycles at 0.5C [29].

### 4.3. Oxidation Routes

The oxidation routes method is one method that utilizes the oxidation process. In this method, sulfur is obtained from the oxidation process of H<sub>2</sub>S gas. H<sub>2</sub>S is rarely used as a source of sulfur because it is a type of toxic gas. But in reality, H<sub>2</sub>S can be used as a source of sulfur with a purity that reaches 94% [14]. Fei et al. used H<sub>2</sub>S gas to produce sulfur-graphene-sulfur nanosheet sandwiches. H<sub>2</sub>S gas was obtained from the hydrolysis of thioacetamide (TAA) (CH<sub>3</sub>C(S)NH<sub>2</sub>). They conducted mixing Graphene oxide and TAA powder (1:20 wt%) in a closed Teflon vessel at 200°C for 20 hours. From the experimental results obtained, a hybrid G/S composite that had a strong C-S covalent bond. So that it could effectively reduce the dissolution of polysulfide in electrolytes. This battery also had a high capacity reaching 1047 mAh/g and 700 mAh/g after 70 cycles at 0.5C [30].

### 4.4. Copolymerization Strategy

Copolymerization is a process in which there are two or more polymerizing monomers. In general, the copolymerization method is at a temperature of 159°C, where sulfur is in the liquid phase. In this phase, the S<sub>8</sub> monomer releases its ring-shaped bonds into linear chain polysulfide with the chain ends becoming radical. Because it becomes radical, the end of the chain will bond with other monomers to form copolymer compounds. Zeng et al. reported on their research on sulfur copolymer manufacturing. Copolymerized (sulfur 3-butylthiophene) (CP (S3BT)) was obtained by mixing liquid sulfur (sulfur was obtained from heating sulfur powder at 170°C) with 3-butylthiophene. In this case, when sulfur was heated, the ring-shaped S<sub>8</sub> chain breaks off, forming polysulfides, which then bind to 3-butylthiophene. The mixing process was carried out at 170°C for 5 hours. With a hybrid polymer composite as a cathode, the Li-S battery has a discharge capacity of 1362 mAh/g at 0.1C and 682 mAh/g after 500 cycles at 1C [31]. Ghosh et al., in their study, also used sulfur powder, which was heated at 185°C in an oil bath to break the ring chain structure of sulfur powder. Then Cardanol benzoxazine (C-a) was added to liquid sulfur and heated at 185°C for 10 minutes. From the reaction, sulfur-random-cardanol benzoxazine (S90) copolymer was obtained, and then the step continued by mixing it with rGO to form S90-rGO composite. Batteries with S90-rGO composite cathodes have a specific capacity of ~ 975 mAh/g after 200 cycles [32].

## 5. CONCLUSION

In this review, the fabrication of the cathode composite method for the lithium-sulfur battery has been presented. Both physical and chemical manufacturing methods have been discussed. All studies focused on the effect of the method that was used to the specific discharge capacity and cycle life of batteries. Although there are still many challenges, especially in the cycle life of the battery, the lithium-sulfur battery could become a promising energy storage system due to its high energy capacity.

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