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COMPARATIVE STUDY ON NATURAL SULFUR PURIFICATION FROM INDONESIAN SOURCES AND ITS SUITABILITY FOR LITHIUM-SULFUR BATTERY APPLICATIONS

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Abstract. Sulfur has great potential as a battery cathode material due to its high energy storage capacity. However, impurities in natural sulfur can negatively impact battery performance and stability. This study aims to enhance the purity and crystallinity of natural sulfur from Kawah Ijen and Tangkuban Perahu using the dissolution-recrystallization method with toluene at 100 °C. By dissolving sulfur in toluene, impurities are separated, allowing pure sulfur to recrystallize. X-ray diffraction (XRD) analysis showed that recrystallized sulfur from Tangkuban Perahu achieved a higher crystallinity of 88.4%, compared to 75.62% for Kawah Ijen sulfur. In contrast, scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) revealed that sulfur from Kawah Ijen had a higher elemental purity, with 91.159% sulfur and 8.841% carbon, whereas Tangkuban Perahu sulfur contained more impurities. These findings demonstrate that the geological source of sulfur significantly impacts the effectiveness of the purification process. Although sulfur from Tangkuban Perahu exhibits greater crystallinity, the higher elemental purity achieved from Kawah Ijen makes it a more promising candidate for lithium–sulfur battery applications. This comparative analysis offers valuable insights for optimizing domestic sulfur processing and advancing the development of high-performance Li-S battery materials

Keywords: sulfur purification, dissolution-recrystallization, Li-S battery, sulfur purity, Crystallinity

Abstrak. *Sulfur merupakan material yang menjanjikan sebagai katoda baterai karena memiliki kapasitas penyimpanan energi yang tinggi. Namun, keberadaan pengotor dalam sulfur alam dapat menurunkan kinerja dan stabilitas baterai. Penelitian ini bertujuan untuk meningkatkan kemurnian dan kristalinitas sulfur alam yang berasal dari Kawah Ijen dan Tangkuban Perahu melalui metode disolusi–rekristalisasi menggunakan pelarut toluena pada suhu 100 °C. Proses pelarutan sulfur dalam toluena memungkinkan pemisahan pengotor dan pembentukan kembali kristal sulfur murni. Hasil analisis X-ray diffraction (XRD) menunjukkan bahwa sulfur hasil rekristalisasi dari Tangkuban Perahu memiliki tingkat kristalinitas lebih tinggi, yaitu sebesar 88.4%, dibandingkan dengan sulfur dari Kawah Ijen sebesar 75.62%. Sebaliknya, hasil karakterisasi menggunakan scanning electron microscopy dan energy-dispersive X-ray*

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spectroscopy (SEM-EDS) menunjukkan bahwa sulfur dari Kawah Ijen memiliki kemurnian unsur yang lebih tinggi, dengan komposisi 91.159% sulfur dan 8.841% karbon, sedangkan sulfur dari tangkuban perahu mengandung lebih banyak pengotor. Temuan ini mengindikasikan bahwa asal geologis sulfur memengaruhi efektivitas proses pemurnian. Meskipun sulfur dari Tangkuban Perahu menunjukkan kristalinitas yang lebih tinggi, tingkat kemurnian unsur yang lebih tinggi pada sulfur dari Kawah Ijen menjadikannya kandidat yang lebih potensial untuk aplikasi baterai lithium–sulfur. Analisis komparatif ini memberikan kontribusi penting dalam optimalisasi pemrosesan sulfur domestik dan pengembangan material katoda baterai Li–S berkinerja tinggi.

Kata kunci: *pemurnian sulfur, disolusi-rekristalisasi, baterai Li-S, kemurnian sulfur, kristalinitas*

1. Introduction

Advancing battery technology is essential to meet the growing energy demands,especially in portable electronic devices and electric vehicles. For several years, Li-Ion batteries have been the industry standard [1]. Despite two decades of development, Li-ion batteries have not surpassed a capacity of 250 mAh/g and an energy density of 800 Wh/kg. These capacityand energy density levels do not meet the criteria for a 500 km range on a single charge forelectric vehicles. Additionally, Li-ion batteries have several other drawbacks, including poor high-temperature tolerance, short lifetime, and relatively high cost [2]. Therefore, it is necessary to increase battery capacity for large-scale applications.

Modifying the battery cathode structure using alternative materials is one approach to addressing these issues. The most promising material is sulfur, which is abundant on Earth. Sulfur also has excellent electrochemical properties, as it can accept two electrons per atom. Sulfur has a theoretical capacity of 1675 mAh/g [2], while lithium has a theoretical capacity of 3861 mAh/g [3]. Lithium batteries with sulfur cathodes (Li-S) have a theoretical energy density of 2600 Wh/kg [1]. This energy density is 3-5 times higher than conventional lithium batteries with $LiCoO_2$ and $LiFePO_4$ cathodes [1, 3]. Replacing conventional lithium battery cathodes with sulfur offers several advantages, including a relatively low operating voltage of 2.15 V [4], which enhances safety. Moreover, sulfur is inexpensive due to its abundance and low extraction cost. Sulfur is also non-toxic [5], making it more environmentally friendly than cobalt or other transition metals [2].

Using sulfur as a cathode material for lithium-ion batteries still presents many challenges. It has extremely low conductivity ($5x10^{-30}$ S/m at 25°C), significantly reduces cell capacity and electrochemical performance. Volumetric changes during discharge and charging cause the cathode volume to increase by about 80% after complete discharge, resulting in sulfur coming out of the cathode and corroding the damaged lithium anode. Polysulfides dissolved in the electrolyte lead to a reduction in sulfur mass in the cathode. The presence of polysulfides (PS) in long chains (Li_2Sx; $3<x<8$) formed in the cathode causes a "shuttle effect" that inhibits ion mobility [2, 6].

Nonetheless, many studies show high potential to overcome such problems and significantly improve battery performance through several methods, such as fabricating sulfur composite cathodes with porous carbon. The study used commercial sulfur produced by Sigma Aldrich with a purity of 99.98%. Natural sulfur contains impurities that must be eliminated to ensure the purity and quality of the final product.

Indonesia has several sulfur mines, such as those at Kawah Ijen (Ijen Crater), Mount Merapi, and Mount Bromo in East Java, Mount Sibayak and Mount Sinabung in North Sumatra. Additionally, there are sulfur mines at Kawah Putih (White Crater) and Mount Tangkuban Perahu in West Java, which are known as tourist destinations and sulfur sources. The sulfur reserves at Kawah Ijen are claimed to be the largest in Indonesia, with at least 14 tons of sulfur mined daily [7]. However, domestic sulfur production cannot meet the demand, and the lack of exploration in sulfur production processes has resulted in Indonesia having high levels of sulfur imports. In 2021, Indonesia imported sulfur valued at $13.1 million, making it the 4^{th} largest sulfur importer in the world. In the same year, sulfur ranked as the 728th most imported product in Indonesia [8]. Mastering sulfur processing for commercial purposes is essential to fulfill domestic sulfur requirements and decrease dependence on imports. Additionally, sulfur purification steps are necessary, especially for specialized applications such as its use as a cathode in lithium-sulfur (Li-S) batteries.

Despite the promising applications of sulfur, existing sulfur purification methods face several limitations, such as incomplete removal of impurities, high costs, and the need for complex equipment. Previous studies, like the use of modified Frasch process with steam [9], and sulfur purification using steam autoclave with CO2 pre-injection [10], have shown improvements but still present significant challenges. This research involves studying sulfur purification using the dissolution-recrystallization method. This method purifies organic solids by dissolving the material to be purified (solute) in a suitable hot solvent. As the solvent cools, the solution becomes saturated with the solute, which then crystallizes (reforms into a solid). As the crystals grow, impurities are excluded from the crystal lattice, completing the purification process. [11, 12]

The novelty of this study lies in its application of the dissolution-recrystallization method to natural sulfur sources from Kawah Ijen and Tangkuban Perahu, which has not been extensively explored before. By comparing these two natural sulfur sources, this study aims to provide new insights into their purity and crystallinity, highlighting significant differences that could impact local sulfur processing industries. Additionally, the dissolution-recrystallization method is considered more effective and cost-efficient compared to other methods due to its ability to achieve high purity with relatively simple equipment and lower operational costs.

Moreover, producing high-purity sulfur will reduce our dependence on imported materials for technological applications. Additionally, processing sulfur from domestic natural resources could provide economic benefits to local communities, particularly sulfur miners. This research focuses on purifying natural sulfur to achieve high purity for use as a cathode material in Li-Sulfur batteries. Obtaining high-purity sulfur powder with a high yield will determine the success of this research.

2. Research Methods

2.1 Materials

This research utilizes sulfur obtained from Kawah Ijen and Kawah Tangkuban Perahu. Additionally, toluene (Merck, with a purity of >99%) is used as the solvent.

2.2 Sulfur Purification using Dissolution-Recrystallization

Figure 1 shows the flow diagram of the process used to dissolve the samples with toluene (C_7H_8).

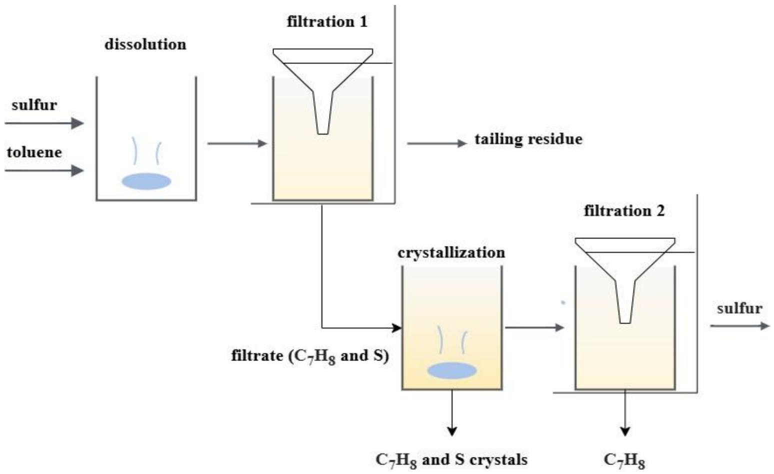


Figure 1. Sulfur purification by dissolution-recrystallization using toluene as solvent.

The purification procedure involves heating the sulfur components and solvent, followed by crystallization upon cooling. The heating temperature of 100 °C was selected because it is slightly below the boiling point of toluene (110.6 °C), allowing the solvent to remain liquid and effectively dissolve sulfur. This temperature was also found to be ideal for producing high-purity sulfur, as evidenced by a study that achieved approximately 96.3% purity at room temperature following dissolution recrystallization process [13]. Natural sulfur is first ground into a fine powder. 200 mL of toluene is poured into a beaker and placed on a hotplate magnetic stirrer to be heated to 100°C. Next, 40 g of sulfur powder is added to the beaker containing toluene, with the solution's temperature continuously monitored by a thermometer and stirred at a speed of 290 rpm. The first filtration is then carried out to separate the sulfur dissolved in toluene from the solid impurities in the solution. Subsequently, the solution is gradually cooled by placing the beaker on a hotplate magnetic stirrer and decreasing the hotplate temperature by 5°C every 10 minutes until the solution reaches 30°C, without stirring. Once the solution's temperature reaches 30°C or room temperature, it is left at room temperature for 60-120 minutes to ensure full recrystallization of the sulfur. Sulfur was subsequently extracted from the solution. The solution was slowly poured into another container to remove the liquid toluene, leaving sulfur crystals in the beaker. The sulfur crystals were then air-dried. Next, the sulfur crystals are finely ground with a mortar and labeled STP (Sulfur Tangkuban Perahu) and SKI (Sulfur Kawah Ijen).

2.3 Yield Measurement

The yield analysis of purified Sulfur (S) is conducted using a mass comparison method involving the precise weighing of Sulfur samples before and after the purification process. Subsequently, the calculation of the purified Sulfur sample is performed by comparing the initial and final mass of the sample.

$$Yield\ (\%) = \frac{Mass\ of\ purified\ sulfur}{Initial\ mass\ of\ sulfur} \times 100 \quad (1)$$

2.4 Sulfur Testing with SEM-EDS and XRD

Characterization of the purified sulfur was conducted using Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) and X-Ray Diffraction (XRD). XRD testing, using a Cu radiation source with a wavelength of 1.5406 Å and 2θ ranging from 10° to 90°, was employed to confirm the removal of impurities by analyzing the diffraction patterns. These patterns, interpreted using the HighScore Plus software with the Rietveld refinement method. SEM provided detailed observations of the surface and particle morphology of the purified sulfur. Integrated with SEM, EDS identified the elemental composition, ensuring that unwanted elements and impurities were effectively removed. These characterization techniques validated the purity and structural integrity of the sulfur post-purification.

3. Result and Discussion

The sample results in Figure 2 show a difference in the color of all the types of samples, where commercial sulfur has a slightly pale yellow color, sulfur from Tangkuban Perahu has a greenish-yellow color, refined sulfur from Tangkuban Perahu has a bright yellow color, sulfur from Ijen has a bright yellow color, and refined sulfur from Ijen shows the palest yellow color among all.

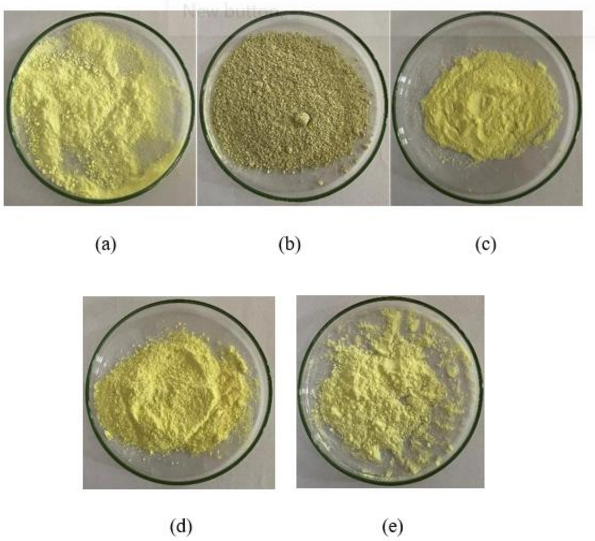


Figure 2. Sample results showing (a) commercial sulfur (b) natural STP, (c) purified STP, (d) natural SKI, and (e) purified SKI.

This variation is due to several factors. When sulfur is reduced to a fine powder, the increase in surface area of the sulfur particles allows for greater interaction with light. Consequently, smaller sulfur particles can scatter light differently compared to larger particles, resulting in variations in color perception. This phenomenon may be attributed to the refined Kawah Ijen sulfur sample being filtered using a 400 mesh sieve, which has an opening of 0.037 mm, whereas commercial powdered sulfur is generally filtered using a 325 mesh sieve, and other sulfur samples were not filtered through a mesh. Additionally, the color transformation in sulfur powder can be observed under various lighting conditions or viewed from different angles. For instance, sulfur powder appears pale yellow under direct light. Conversely, when light hits sulfur powder at an oblique angle, the smaller particles can cause diffraction, resulting in a captivating yellow appearance.

Table 1 provides information about the yield of purified sulfur, calculated using the formula yield (%) = (mass of sulfur product / mass of initial sample) × 100. Sulfur from Tangkuban Perahu shows a yield of 46%, while sulfur from Ijen results in a yield of 19%. This suggests that sulfur from Tangkuban Perahu exhibits greater solubility in toluene, facilitating a higher purification yield.

Table 1. Comparison of initial mass, final mass, and yield amount of sulfur purification for STP

Sample Type	**Initial Mass (g)**	**Final Mass (g)**	**Yield (%)**
STP	40	18.59	46%
SKI	40	7.48	19%

The XRD results for the STP and SKI samples are shown in Figure 3. Sample peaks were analyzed using the Rietveld Refinement method and matched to COD database no. 96-901-1363. Table 2 displays the structural parameters of the sulfur samples. The XRD results indicate that both samples conform to the Orthorhombic crystal structure ($a \neq b \neq c$), characteristic of S8 (octa-Sulfur) crystals. This Orthorhombic structure, defined by space group 70/fddd, showcases the symmetry and lattice pattern of sulfur, demonstrating a well-organized crystal structure within this space group. Lattice parameters and crystal structures are primarily influenced by atomic interactions and their specific arrangements within the crystal lattice. Impurities can distort the lattice by introducing irregularities or defects. During purification, reducing impurities results in a more regular crystal structure, which enhances the accuracy and definition of lattice parameters. Additionally, heating during the purification process can provide atoms with sufficient energy to rearrange into a more stable or lower-energy configuration, potentially refining the crystal structure [14, 15]

Table 2. Structural parameters of STP and SKI sulfur samples, including space group and lattice parameters (a, b, and c).

Sulfur samples	**STP**	**SKI**
Space group	70/fddd	70/fddd
Structure	Orthorhombic	Orthorhombic
Lattice Parameters		
a (Å)	10.465	10.467
b (Å)	12.866	12.870
c (Å)	24.486	24.493

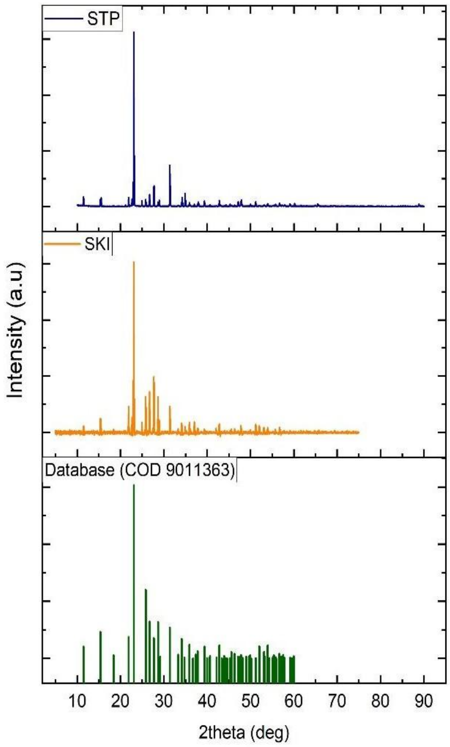
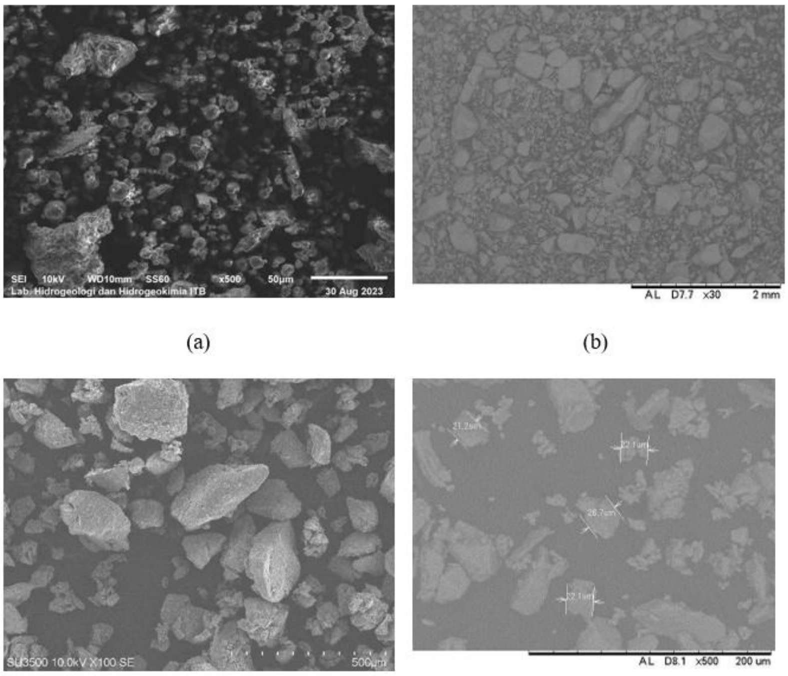


Figure 3. XRD patterns of purified sulfur samples showing orthorhombic structure and crystallinity differences

The degree of crystallinity is determined by comparing the crystal peak area (Reduced Area) with the total peak area (Global Area). Based on the XRD analysis, the global area for the SKI sample is 1482.686 cts and the reduced area is 1121.21 cts, resulting in a crystallinity degree of 75.620%. For the STP sample, the global area is 141.28 cts and the reduced area is 124.93 cts, yielding a crystallinity degree of 88.4%. The substantial crystallinity indicates a high level of atomic order, which facilitates efficient charge and ion transport within the crystal lattice. This results in enhanced electrochemical performance of the batteries.

The composition of sulfur samples was analyzed to determine their purity using energy dispersive spectroscopy (EDS) in conjunction with a Scanning Electron Microscope (SEM). Due to the varying particle sizes, SEM mapping was performed at multiple magnifications to examine the distribution and morphology of the samples. The results of the SEM EDS analysis for both refined and natural sulfur samples are presented in Figure 4.



(a) (b)

(c) (d)

Figure 4. SEM results displaying the morphology of (a) natural STP, (b) natural SKI, (c) purified STP, and (d) purified SKI.

Table 3. Elemental content of EDS testing on sulfur before and after purification.

Sample Type	Elements	Weight (%)	Atomic (%)
Natural STP	Carbon (C)	49.27	70.21
	Oxygen (O)	5.06	5.41
	Sulfur (S)	45.67	24.38
Natural SKI	Carbon (C)	45.807	69.291
	Sulfur (S)	54.193	30.709
Purified STP	Carbon (C)	46.2	68.83
	Oxygen (O)	2.04	2.28
	Sulfur (S)	51.76	28.88
Purified SKI	Carbon (C)	8.841	20.567
	Sulfur (S)	91.159	79.433

The EDS spectra were used to assess the purification levels for all elements, as shown in Table 3. The EDS results indicate that carbon is the highest contaminant. Carbon can bind to sulfur as solid particles or in the form of organic compounds. Carbon contamination can occur if sulfur is exposed to air containing carbon dioxide (CO_2) or environmental contaminants such as dust or organic particles. Toluene, being an organic solvent, can also contribute to the presence of organic carbon compounds in sulfur if separation between sulfur and toluene is not complete. However, the carbon contaminant content decreases after the purification process, leading to increased sulfur content. The purification results for both samples showed an increase in sulfur content, with the highest purity achieved in the Ijen sulfur sample, containing 91.159% sulfur and 8.841% carbon compounds. The differences in sulfur sources are significant, as sulfur from Kawah Ijen exhibited higher purity compared to sulfur from Tangkuban Perahu. In Kawah Ijen, sulfur extraction involves a well-organized process where miners use metal pipes to channel sulfur dioxide gas from within the crater. This gas condenses into liquid sulfur as it cools and then hardens into yellow solid sulfur upon contact with the air. The miners manually collect these sulfur blocks, often transporting them in baskets. This intensive extraction process results in high-purity sulfur [16]. In contrast, at Tangkuban Perahu, the sulfur collection is less intensive. Workers gather sulfur that has naturally condensed around fumaroles, forming crystals or solid deposits. These deposits are then cleaned by washing with water or drying to remove impurities. The sulfur collected here is often used for creating souvenirs rather than large-scale commercial use [17].

The high-purity sulfur obtained via the dissolution–recrystallization method exhibits strong crystallinity, as confirmed by XRD analysis. Sulfur with crystallinity level of 96% has been reported to reduce voltage drop, suppress polysulfide shuttle effects, and improve long-term cycling stability [18]. Additionally, in-situ crystallization during charging has been shown to regulate polysulfide concentrations, thereby enhancing cell durability [19]. On the other hand, sulfur impurities and amorphous structures can promote side reactions and degrade battery performance [20]. Our findings support these observations by showing that sulfur purified from both Tangkuban Perahu and Kawah Ijen possesses enhanced crystallinity and purity. While Tangkuban Perahu sulfur exhibits slightly higher crystallinity, the significantly greater elemental purity of Kawah Ijen sulfur (91.159%) is likely more impactful for electrochemical applications, as it reduces internal resistance and suppresses unwanted redox reactions. Therefore, despite its lower crystallinity, Ijen sulfur emerges as the more promising candidate for Li–S battery cathodes.

Although the dissolution–recrystallization method has proven effective in improving the purity and crystallinity of sulfur, there are several limitations that need to be considered. One major concern is the potential loss of sulfur mass during the filtration and solvent evaporation stages, which can reduce the overall process efficiency. In addition, the use of organic solvents such as toluene necessitates an efficient solvent recovery system to ensure that the process remains both environmentally friendly and economically viable. These challenges must be addressed, especially if the method is to be scaled up for industrial applications.

4. Conclusions

This study demonstrates that sulfur purification from Tangkuban Perahu and Kawah Ijen using the dissolution-recrystallization method with toluene solvent at 100 °C produces different results in terms of crystal structure and chemical purity. XRD analysis reveals that sulfur from Tangkuban Perahu has a higher degree of crystallinity (88.4%) compared to sulfur from Kawah Ijen (75.620%), while SEM-EDS results show that sulfur from Kawah Ijen has higher elemental purity, with sulfur content of 91.159% and carbon content at 8.841%. The EDS technique is more effective in determining the chemical purity of sulfur as it can directly identify and measure the elements in the sample, whereas XRD provides insights into the crystal structure and degree of crystallinity. Therefore, for Li-S battery cathode applications, the purity of sulfur is crucial for optimal battery performance, making sulfur from Kawah Ijen more suitable for use as a Li-S battery cathode compared to sulfur from Tangkuban Perahu. The findings of this study illustrate the quality and characteristics of high-purity sulfur, highlighting the success of the dissolution-recrystallization purification technique as a critical step in creating high-quality sulfur cathodes for advanced Li-S batteries. Future research should involve FTIR testing to determine the purity of sulfur through indicators of the absence of sulfur bonds with carbon, oxygen, or other light elements such as hydrogen and nitrogen. Additionally, testing sulfur in prototype Li-S batteries is recommended to validate its suitability for commercial battery applications and to identify areas for improvement in battery design and materials. Further studies should also focus on enhancing the efficiency and scalability of the purification process.

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