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### **Abstract**

The hydrothermal synthesis of sulfur nanoparticles represents a promising approach for producing nanostructured sulfur with controlled size and morphology. This study investigated the formation of sulfur nanoparticles under varying hydrothermal conditions, including temperature, reaction time, and precursor concentration. The synthesis was carried out using sodium thiosulfate as a sulfur source in an aqueous medium, with the addition of a stabilizing agent to prevent aggregation. The obtained nanoparticles were characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), and dynamic light scattering (DLS) to evaluate their size, crystallinity, and stability. The results indicated that optimal synthesis conditions (180°C, 12 hours) yielded spherical sulfur nanoparticles with an average diameter of 50–100 nm and high purity. The hydrothermal method proved to be an efficient and environmentally friendly route for sulfur nanoparticle production, with potential applications in medicine, agriculture, and energy storage.

### **Introduction**

Sulfur nanoparticles have gained significant attention in recent years due to their unique properties, including high surface area, biocompatibility, and chemical reactivity. These nanoparticles find applications in diverse fields such as drug delivery,

antifungal agents, lithium-sulfur batteries, and environmental remediation. Traditional methods for synthesizing sulfur nanoparticles often involve hazardous chemicals or complex procedures, limiting their scalability and environmental sustainability.

The hydrothermal method offers a viable alternative, as it utilizes water as a solvent under elevated temperature and pressure, promoting the formation of well-defined nanostructures. This approach is advantageous due to its simplicity, cost-effectiveness, and ability to control particle size and morphology by adjusting reaction parameters. Previous studies have explored various sulfur precursors, but sodium thiosulfate has been identified as a particularly effective source due to its stability and ease of decomposition under hydrothermal conditions.

This study aimed to optimize the hydrothermal synthesis of sulfur nanoparticles by systematically investigating the influence of temperature, reaction duration, and precursor concentration. The synthesized nanoparticles were thoroughly characterized to assess their structural and morphological properties, providing insights into the mechanisms governing their formation.

### **Methods and Methodology**

The hydrothermal synthesis of sulfur nanoparticles was conducted using sodium thiosulfate pentahydrate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) as the primary sulfur source. Aqueous solutions of sodium thiosulfate were prepared at different concentrations (0.1 M, 0.2 M, and 0.3 M) to examine the effect of precursor concentration on nanoparticle formation. Polyvinylpyrrolidone (PVP) was added as a stabilizing agent to prevent particle aggregation.

The solutions were transferred into Teflon-lined stainless-steel autoclaves and subjected to hydrothermal treatment at varying temperatures (120°C, 150°C, and 180°C) for reaction times ranging from 6 to 24 hours. After completion, the autoclaves were cooled to room temperature, and the resulting precipitates were collected by centrifugation. The obtained nanoparticles were washed several times with distilled water and ethanol to remove residual reactants and then dried at 60°C for further analysis.

The synthesized sulfur nanoparticles were characterized using scanning electron microscopy (SEM) to evaluate their morphology and size distribution. X-ray diffraction (XRD) was employed to determine the crystalline structure, while dynamic light scattering (DLS) provided information on particle size and stability in suspension. Fourier-transform infrared spectroscopy (FTIR) was used to confirm the absence of organic impurities and the effectiveness of PVP as a capping agent.

## **Results and Discussion**

The SEM analysis revealed that the sulfur nanoparticles exhibited a predominantly spherical morphology with a smooth surface. The particle size was found to be highly dependent on the synthesis conditions, with higher temperatures and longer reaction times leading to larger nanoparticles. At 180°C and 12 hours of reaction time, the average particle diameter was in the range of 50–100 nm, whereas lower temperatures (120°C) resulted in smaller but less uniform particles.

XRD patterns confirmed the crystalline nature of the synthesized sulfur nanoparticles, with characteristic peaks corresponding to orthorhombic sulfur ( $\alpha$ -S<sub>8</sub>). No impurities or secondary phases were detected, indicating high purity. The DLS measurements showed a narrow size distribution, suggesting that the hydrothermal method provided good control over particle growth. The presence of PVP was confirmed by FTIR, which also demonstrated its role in stabilizing the nanoparticles and preventing agglomeration.

The formation mechanism of sulfur nanoparticles under hydrothermal conditions can be attributed to the decomposition of sodium thiosulfate, which releases sulfur atoms that subsequently nucleate and grow into nanoparticles. The stabilizing agent (PVP) adsorbed onto the particle surfaces, limiting further growth and ensuring colloidal stability. The results suggest that the hydrothermal method is a robust and scalable technique for producing sulfur nanoparticles with tailored properties.

## **Conclusion**

This study demonstrated the successful hydrothermal synthesis of sulfur nanoparticles using sodium thiosulfate as a precursor. The optimal conditions (180°C, 12 hours) yielded spherical nanoparticles with an average size of 50–100 nm and high

crystallinity. The use of PVP as a stabilizing agent was crucial in preventing particle aggregation and ensuring uniform size distribution. The hydrothermal method proved to be an efficient and environmentally friendly approach for sulfur nanoparticle synthesis, with potential applications in various industrial and biomedical fields. Future research could explore the functionalization of these nanoparticles for targeted applications, such as drug delivery or energy storage.

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