A Method for Dissolving Agricultural Sulfur and Preparation of Nano-Sulfur Loaded on Nano-Cellulose

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ABSTRACT

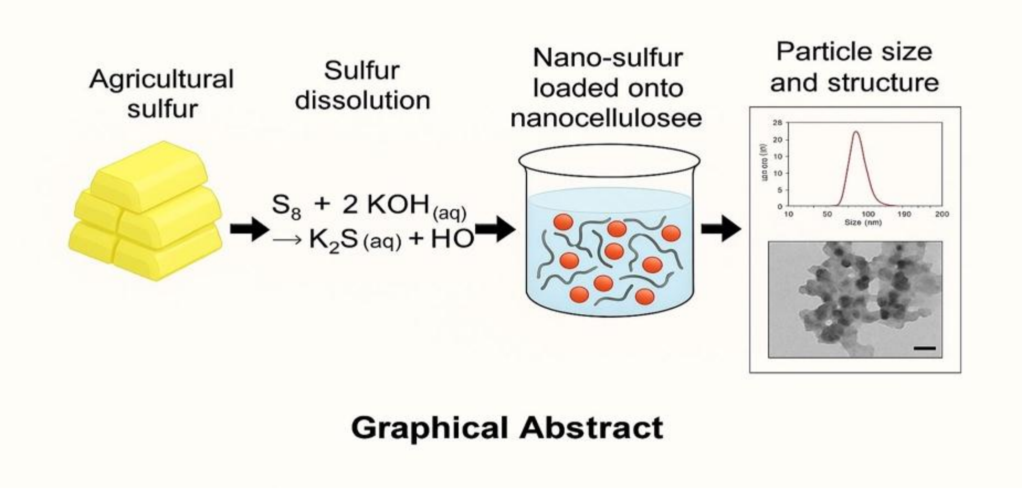
Sulfur (S_8) plays a vital role in plant nutrition, but its low water solubility limits its bioavailability and agricultural efficiency. This study presents a novel and mild chemical method to dissolve agricultural sulfur using potassium hydroxide, followed by the synthesis of nano-sulfur particles and their stabilization through loading onto nanocellulose. The formulation process includes stirring and gelation steps to ensure nanoscale dispersion and particle stability. Particle size analysis confirmed that the synthesized nano-sulfur particles ranged between 7–31 nm, while nano-cellulose fibers ranged from 30–50 nm. When combined, the nano-sulfur/nano-cellulose complex displayed enhanced uniformity (6.5–26.92 nm) and reduced agglomeration, as validated by transmission electron microscopy (TEM). This hybrid nanoformulation is highly promising for improving sulfur bioavailability, ensuring controlled release, and enhancing nutrient use efficiency in sustainable agricultural systems. The study demonstrates a green nanotechnological strategy for converting poorly soluble sulfur into an effective, scalable nano-fertilizer in the fields of nutrition and control of fungal diseases.

Keywords: Nano-sulfur; Nanocellulose; dissolving agricultural sulfur; Smart fertilizers; Particle size analysis; Transmission electron microscopy (TEM); Sustainable agriculture.

INTRODUCTION

Sulfur is an essential secondary macronutrient involved in plant metabolism such as amino acid synthesis (e.g., cysteine, methionine), coenzyme functions, and stress resistance. However, elemental sulfur (S_8) has extremely low water solubility (~20 µg/L at 25 °C), limiting its bioavailability and mobility in soil (Zhao *et al.,* 2019). There is only one way to dissolve agricultural sulfur, but it is expensive, which is to use an organic solvent (Carbon Disulfide) under high heat, as reported by Cui *et al.* (2022).

Nanotechnology, operating at the 1–100 nm scale, offers materials unique physicochemical properties such as a high specific surface area, enhanced reactivity, and tunable release kinetics. In agriculture, nanomaterials like nano-cellulose are widely applied in smart fertilizers, targeted nutrient delivery systems, and biodegradable agrochemicals. These innovations help improve nutrient use efficiency, reduce environmental losses, and support sustainable agricultural practices (Norizan *et al.*, 2022; Sherif *et al.*, 2022; Ding *et al.*, 2023 and Ahmed *et al.*, 2025).



DOI: 10.21608/asejaiqjsae.2025.450886
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Received, July 20, 2025, Accepted, August 31, 2025.

The use of nano-sulfur significantly enhances solubility, surface activity, and subsequent uptake efficiency by plants, leading to improved crop performance (Yazhini *et al.,* 2023 and El-Aziz *et al.,* 2025). Nano-sulfur improves soil chemistry by modifying pH, stimulating microbial activity, and enhancing nutrient cycling in alkaline or calcareous soils. Studies have shown that combining nano-sulfur with organic amendments significantly increases sulfur availability and improves plant growth under stress conditions, such as salinity or heavy metal stress (Cao *et al.,* 2023; Sharma *et al.,* 2023 and Yazhini *et al.,* 2023). These effects contribute to healthier crops, higher yields, and reduced chemical input dependency. Chelating or loading sulfur onto nano-cellulose enhances its solubility, dispersion, and stability. Nano-cellulose serves as a scaffold that prevents nanoparticle aggregation and supports controlled release. The synergy between sulfur and cellulose improves sulfur bioavailability, reduces nutrient loss through leaching or volatilization, and enhances nutrient uptake in plant roots (Yazhini *et al.,* 2023; Sun *et al.,* 2024 and El-Aziz *et al.,* 2025). Such systems are increasingly promising in precision agriculture and sustainable fertilization strategies.

Particle size analysis quantifies the distribution of particle sizes in a material and provides insights into homogeneity and performance. Key parameters such as D10, D50, and D90 help to determine the uniformity of a nanoformulation. Narrow distributions-characterized by low span and D90/D10 ratios-are favorable for improving solubility, controlled nutrient release, and uptake by plant systems (Kekeli *et al.*, 2025). In nano-sulfur systems, optimized Particle Size Distribution (PSD) ensures particle stability and prevents aggregation.

Transmission Electron Microscopy (TEM) is a critical imaging technique used to visualize nanomaterials at high resolution, allowing measurement of particle size, morphology, and dispersion. In sulfur–nano-cellulose composite, TEM provides evidence of nano-sulfur attachment to cellulose fibrils and confirms particle sizes below 100 nm (Mohaiyiddin *et al.,* 2020 and Kekeli *et al.,* 2025). This characterization step is essential to validate the uniformity and effectiveness of the nano formulation.

The objective of this study was to develop a novel method for dissolving elemental agricultural sulfur to establish a mild and efficient chemical or physical protocol that enhances the solubility and dispersion of elemental sulfur, which is naturally hydrophobic and poorly soluble in water; to synthesize and characterize nano-sulfur particles with controlled size and uniformity from agricultural sulfur; and to load and stabilize nano-sulfur onto nano-cellulose matrices. In addition to the nano-sulfur systems, optimized Particle Size Distribution (PSD) ensures particle stability and prevents aggregation.

MATERIALS AND METHODS

Dissolving of Agricultural Sulfur:

To study the optimum conditions for increasing sulfur solubility and availability in agricultural soils, two concentrations of potassium hydroxide (KOH) (1 *M* & 5 *M*) and three different temperatures (25 °C, 60 °C and 105 °C) were tested. After conducting many trials, the following procedures were found to be optimal for increasing the solubility of agricultural sulfur: To 25 grams of agricultural sulfur (S_8) powder, add 100 mL of 5 *M* KOH solution and heat the mixture at 105 °C under continuous stirring for 30 minutes. After the reaction, filter the solution to remove precipitated potassium sulfate and other insoluble residues. The suspension was nano-sulfur (NS).

Synthesis of Nanoscale sulfur-cellulose composite gel:

Nano-cellulose (NC) suspension was prepared according to the modified method from Sherif *et al.* (2022), at a concentration of 0.5% (w/v). This suspension was added to the dissolved sulfur solution, followed by stirring, form a stable hydrogel containing dispersed nano-sulfur-cellulose composite.

Physicochemical Characterization of nano-sulfur and nano-sulfur/nano-cellulose composites:

The viscosity and density of the prepared nano-sulfur and nano-sulfur/nano-cellulose composites were determined using a digital rotational viscometer (model NDJ-5S), according to Cheng (2008) and a pycnometer, respectively, at 25 ± 1 °C. Distilled water was used as the reference standard for calibration. The measurements were performed following the standard procedures outlined in ASTM D2196-20 (2020) for viscosity and ASTM D941-15 (2015) for density determination. Each sample was measured in triplicate, and the average values were reported.

Characterization of nanomaterials:

Transmission Electron Microscopy (TEM) analysis was performed using a JEOL JSM-1400PLUS (Japan) operating at 200 kV with a point-to-point resolution of 0.23 nm, according to the method of Jonoobi *et al.* (2015). Particle Size Distribution (PSD) were measured using a laser particle analyzer (Better size 2600 particle analyzer; Dandong Better size Scientific Ltd., Dandong, China) equipped with an automatic laser centering function. The particle size distribution was measured at

D10, D50, and D90 following the protocol of Chen *et al.* (2021).

RESULTS AND DISCUSSION

The optimum operating condition for dissolving agricultural sulfur:

After conducting many trials, the following procedures are recommended to be the optimum conditions to increase the solubility of agricultural sulfur: To a 250 g of agricultural sulfur, add 1000 ml of 5 *M* KOH solution at a temperature of 105°C with continuous stirring for 30 minutes, then filter the mixture and remove the precipitate consisting of potassium sulfate. The use of potassium hydroxide enhances sulfur solubility by facilitating its conversion into polysulfide forms.

Synthesis of nanoscale sulfur-cellulose composite:

To synthesize a composite of sulfur and nanocellulose, nano-cellulose was added at a concentration of 0.5% to the prepared sulfur solution, leading to the formation of a gel containing nano sulfur. Addition of cellulose can improve gel stability and enhance its applicability in various agricultural applications. Figure (1) shows sulfur solid before reaction, sulfur suspension after solubilization, and sulfur gel formation. This finding was in accordance with the study of Sherif *et al.* (2022) who found that cellulose-based carriers have been shown to enhance the controlled release and stability of micronutrients in soil applications.

Measurement of viscosity and density:

The physical characterization results (Table 1) showed a notable increase in both viscosity and density upon nanoformulation. Nano-sulfur suspension exhibited a viscosity of 5.214 mPa·s, which was more than 5-fold higher than that of distilled water (0.891 mPa·s), reflecting the increased internal friction due to dispersed nanoparticles. Incorporation of nano-cellulose led to a sharp increase in viscosity (39.642 mPa·s), consistent with gel network formation, indicating enhanced structural stability and potential for sustained release formulations (Klemm *et al.*, 2011 and Moon *et al.*, 2011).

As for density, nano-sulfur showed the highest value (1.300 g/cm³), while its combination with nano-cellulose resulted in a lower density (1.212 g/cm³), which may be attributed to the porous and lightweight nature of the cellulose matrix (Habibi *et al.*, 2010). These rheological changes confirm the effective dispersion of sulfur within the nano-cellulose scaffold, supporting its use in advanced agrochemical applications such as controlled-release fertilizers.

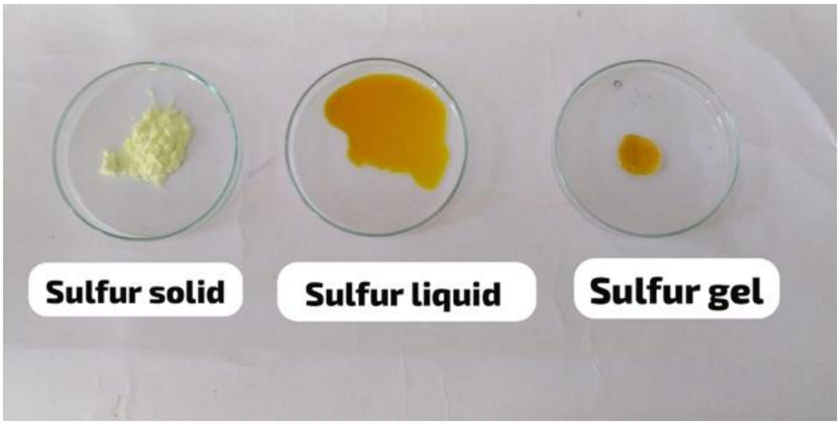


Fig. 1. Different photos of Sulfur solid, Sulfur liquid and Sulfur gel, respectively

Table 1. Density and viscosity of different formulations at 25 °C

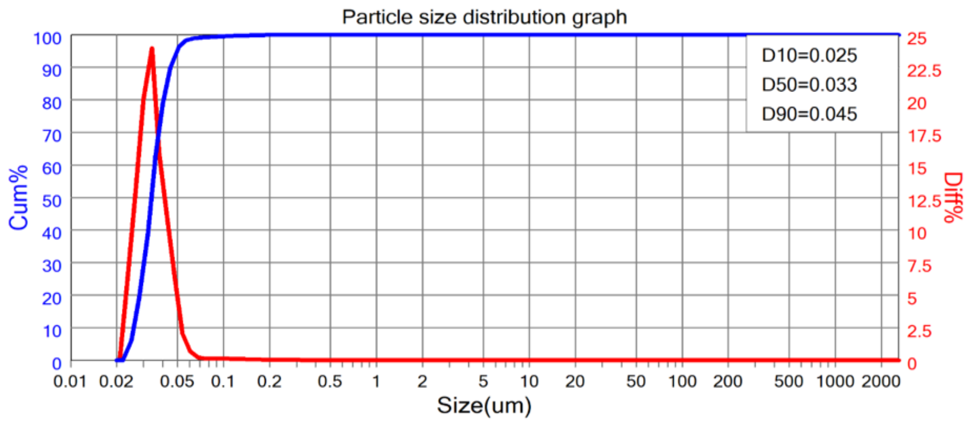
Sample	Density (g/cm³)	Viscosity (mPa·Sec)
Distilled Water (control)	1.000	0.891
Nano-sulfur (NS)	1.315	5.214
Nano-sulfur loaded on nano-cellulose (NS+NC)	1.212	39.642

Characterization of synthesized nanoscale particles: Particle Size Distribution (PSD) and morphology:

The PSD analysis shown in Figures (2, 3 & 4) indicated that synthesized nanoparticles were in the nanoscale particles range (<100 nm). The Particle size of nano-cellulose was in the range 30-50 nm, nano-sulfur size was in the range 7-31 nm, and nano-sulfur loaded on nano-cellulose was in the range 6.5-26.92 nm.

Data in Figure (2) presents the particle size distribution of nano cellulose. The graph includes both cumulative distribution (blue line) and differential distribution (red line). Approximately 99.92% of the particles are between 0.020 and 0.100 µm, demonstrating excellent control over particle size and distribution a critical factor for nano cellulose applications. The peak indicates that the majority of particles are centered around 0.03 µm. The curve is slightly right-skewed, indicating a minor presence of larger particles, but overall, the distribution is tightly packed and ideal for nano-scale applications. This uniform distribution and nano-range particle size make the material highly suitable for use in advanced applications such as smart membranes, controlled-release systems, carriers for agrochemicals or pharmaceuticals & reinforcement in biodegradable composites (Habibi *et al.,* 2010; Chen *et al.,* 2021and Girard *et al.*, 2021).

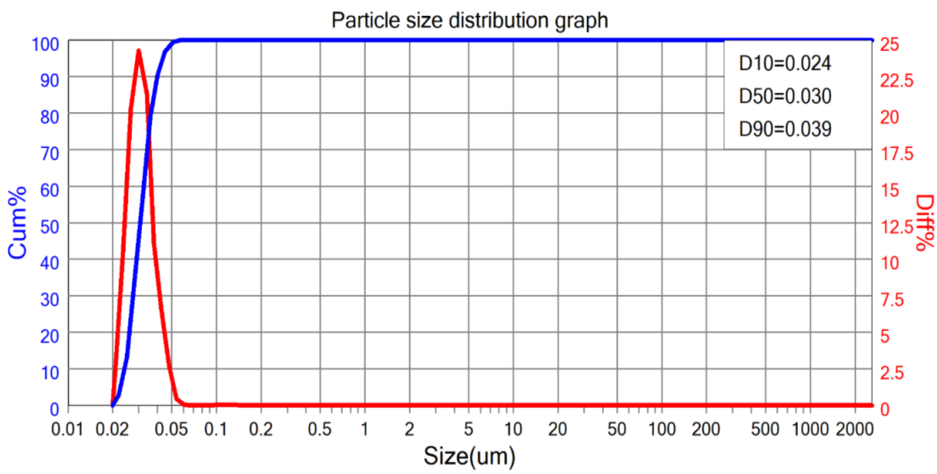
Data presented in Figure (3) demonstrates that the NS particles have a small and highly uniform size distribution, with most particles (over 99%) falling within the 0.020 to 0.050 µm range. The D10, D50, and D90 values further confirm the narrow particle size distribution (Chen *et al.*, 2021 and Sharma *et al.*, 2023).



Table

Diam um	Percent
0.000 - 0.020	0.00
0.020 - 0.050	95.56
0.050 - 0.100	3.94
0.100 - 0.200	0.42
0.200 - 0.500	0.08
0.500 - 1.000	0.00
1.000 - 2.000	0.00
2.000 - 5.000	0.00
5.000 - 10.00	0.00
10.00 - 20.00	0.00

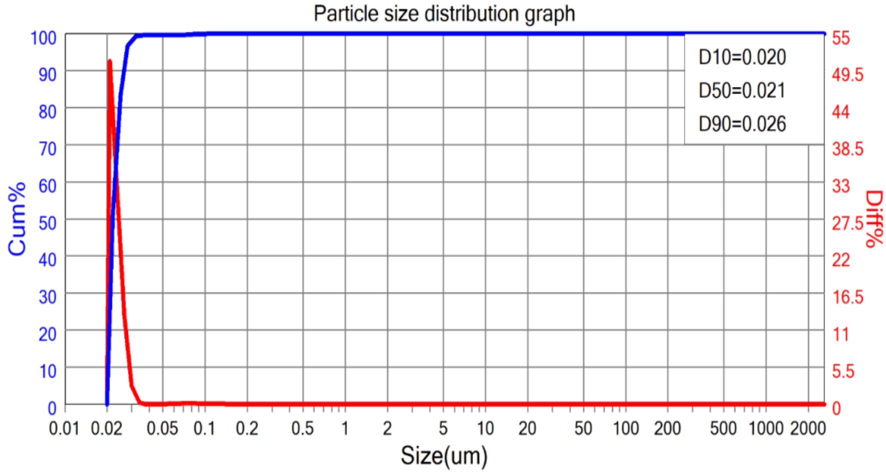
Fig. 2. Particle size of Nano-cellulose



Table

Diam um	Percent
0.000 - 0.020	0.00
0.020 - 0.050	99.26
0.050 - 0.100	0.68
0.100 - 0.200	0.06
0.200 - 0.500	0.00
0.500 - 1.000	0.00
1.000 - 2.000	0.00
2.000 - 5.000	0.00
5.000 - 10.00	0.00
10.00 - 20.00	0.00

Fig. 3. Particle size of Nano-sulfur



Table

Diam um	Percent
0.000 - 0.020	0.00
0.020 - 0.050	99.51
0.050 - 0.100	0.39
0.100 - 0.200	0.10
0.200 - 0.500	0.00
0.500 - 1.000	0.00
1.000 - 2.000	0.00
2.000 - 5.000	0.00
5.000 - 10.00	0.00
10.00 - 20.00	0.00

Fig.4. Particle size of Nano-sulfur loaded Nano-cellulose

Moreover, Figure (4) represents the particle size distribution of nano-sulfur-cellulose composite. It is shown that nanocomposite synthesized from sulfur and cellulose is highly uniform, with over 99% of particles under 0.05 µm. The narrow D-values (D10, D50, and D90) confirm that the nano-sulfur is well-integrated and evenly dispersed in the nano-cellulose support. This narrow distribution is beneficial for applications requiring consistent nanoscale properties such as in controlled release or enhanced reactivity and these values indicate a very narrow size distribution and that the particles are within the nanoscale range, suggesting effective dispersion of nano-sulfur in the nano- cellulose (Habibi *et al.*, 2010 and Norizan *et al.*, 2022).

In conclusion, the combined analysis of the three PSD of nanomaterials confirmed the formation of nanoscale particles of studied materials and indicating excellent uniformity in the nanoscale range, making it a compatible carrier (cellulose), and showing a synergistic effect between nano-sulfur and cellulose in stabilizing the size and preventing agglomeration of formed composite. This confirms that loading NS onto NC enhances the uniformity and dispersion of the active particles, which is critical for practical applications.

Transmission Electron Microscopy (TEM):

TEM images (Figures 5, 6, &7) also confirmed the synthesis of studied nanomaterials. These images confirm the uniform dispersion of sulfur nanoparticles and the effective interaction between sulfur and nano-cellulose fibers. According to previous research (Kumar *et al.*, 2021), such uniform dispersion is crucial for enhancing the bioavailability of sulfur in soil applications.

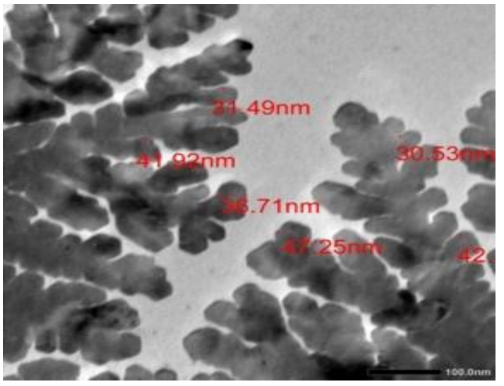


Fig. 5. TEM of nanoscale cellulose

Figure (5) presents a TEM image of nano-cellulose particles. TEM is a powerful characterization technique used to examine the morphological and structural features of nanomaterials due to its high resolution and magnification capabilities (Jonoobi *et al.*, 2015). The image reveals that the nano-cellulose particles exhibit an irregular, agglomerated morphology with particle sizes ranging approximately from 30 to 47 nm. This confirms that the synthesized material falls within the nanoscale range (less than 100 nm), which enhances its surface area, reactivity, and mechanical properties (Klemm *et al.*, 2011). Nano-cellulose is considered a promising nanomaterial due to its biodegradability, low density, high aspect ratio, and excellent thermal and mechanical properties. The relatively uniform particle size distribution observed in the TEM image indicates that the preparation method was effective in producing nano-cellulose with a controlled morphology, which is crucial for consistency and performance in various applications such as nanocomposites, drug delivery systems, membranes, and reinforcing agents in polymer matrices (Moon *et al.*, 2011).

Figure (6) presents a TEM image of nano-sized sulfur particles. The Figure consists of two contrasting views that illustrate different morphological characteristics. On the left side, the particles exhibit a well-defined spherical morphology with hollow or porous internal structures, while the right side shows smaller, more dispersed spherical nanoparticles (Chen *et al.,* 2020). The particle sizes vary significantly between the two regions. In the left image, the particles range from approximately 66 to 176 nm, indicating the formation of larger hollow sulfur nanospheres. Such morphologies can be advantageous in applications like drug delivery or energy storage, where internal voids can be used to encapsulate active substances. On the right, the particles are significantly smaller, ranging between approximately 5.89 and 18.27 nm. These smaller nanoparticles exhibit good dispersion, which enhances surface area and reactivity crucial for catalytic or antibacterial applications, nano-sulfur has received considerable attention due to its unique physicochemical properties, such as high reactivity, antimicrobial activity, and potential use in lithium–sulfur batteries, fertilizers, and pharmaceuticals. The observed variation in morphology and particle size may result from differences in synthesis methods or reaction conditions, which significantly influence the size distribution and final structure of nano-sulfur (Rahman, 2015 and Suleiman *et al.,* 2015).

Figure (7) shows a TEM image of nano-sulfur loaded onto nano-cellulose in the form of nanocomposite. This image illustrates the successful incorporation and dispersion of sulfur nanoparticles within the nano-cellulose matrix. The left panel reveals a relatively homogeneous distribution of sulfur nanoparticles across the cellulose network. The particles range in size from approximately 6.53 to 24.93 nm, indicating nanoscale confinement. This uniform dispersion suggests strong interaction between sulfur and the cellulose surface, possibly due to hydrogen bonding or surface functional groups (Rashid *et al.,* 2024). In the right panel, there is evidence of partial aggregation of sulfur particles, with particle sizes still within the nanoscale (approximately 11–16 nm). These clusters may be due to local overloading or insufficient surface area for dispersion, but they still reflect the compatibility between sulfur and the cellulose matrix.

In general, the combination of nano-cellulose with nano-sulfur results in a composite material with enhanced stability, dispensability, and potential functional performance. Nano-cellulose acts as an excellent support or carrier material, improving the structural integrity of sulfur particles and controlling their release or reactivity. Such composites are highly promising for applications such as slow-release fertilizers, antimicrobial coatings, energy storage devices (e.g., Li–S batteries), and environmental remediation.

Rationale of nanoscale -sulfur-cellulose composite:

The scientific importance of nano-sulfur-cellulose composite can be summarized as: improved Stability and Dispersion: nano-sulfur tends to agglomerate due to high surface energy, which reduces its effectiveness and nano cellulose acts as a natural, biodegradable carrier that prevents clumping and promotes stable dispersion of sulfur nanoparticles (Saikia and Lens, 2020). Enhanced bioavailability in agriculture: in agriculture, sulfur is essential for plant nutrition and disease resistance, when nano-sulfur is well-dispersed using nano cellulose, it can be more readily absorbed by plant tissues or soil microbes due to its small size and stable distribution and this results in higher efficiency and reduced environmental impact compared to conventional sulfur fertilizers or fungicides (Rahman, 2015 and Sun *et al.,* 2024). Controlled release potential: nano cellulose can act as a matrix for controlled release, gradually delivering sulfur over time, this minimizes the need for repeated application, saving cost and labor (Teo *et al.,* 2022 and Mahawar *et al.,* 2023). Nor & Tabatabai (1977) and Degryse *et al.* (2016) stated that sulfur in its elemental form (S_8) is not directly available to plants but undergoes microbial oxidation in the soil to form sulfate anions (SO_4^{2-}), which are readily absorbed by roots. The alkaline dissolution step employed in this study likely produces soluble polysulfide intermediates that oxidize to sulfate more rapidly than crystalline (S_8). This dual functionality nutrient provision and antimicrobial activity makes the nano-sulfur/nano-cellulose composite a promising material for both plant nutrition and pest management. In addition to this method is very cheap compared to using other organic solvents, as it is 125 times cheaper than it.

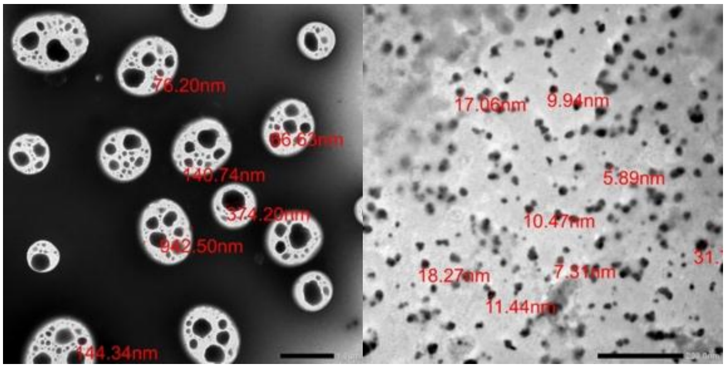


Fig.6. TEM of synthesized nanoscale sulfur

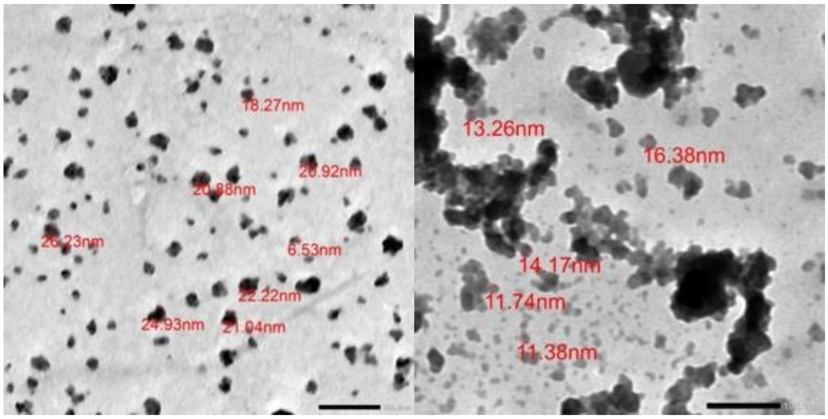


Fig.7. TEM of Nano- Sulfur-cellulose composite

CONCLUSION

An effective method was developed for dissolving agricultural sulfur with enhanced stability achieved by adding cellulose to form a gel. This formulation can be utilized to improve soil quality and enhance sulfur uptake by plants, contributing to more sustainable agricultural practices. That future work will include pot experiments to assess plant growth responses, nutrient uptake efficiency, and soil chemical changes under both controlled and field conditions. Significance of this new approach is concluded as the integration of nano-sulfur with nano- cellulose provides a new, efficient, and sustainable strategy to achieve homogeneous distribution of active nanomaterials, enhance stability, effectiveness, and safety, and support eco-friendly applications in agriculture and other industries. This technique opens new avenues for green nanotechnology and smart material formulation, where performance and environmental compatibility are both optimized.

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الملخص العربي

طريقة لإذابة الكبريت الزراعي وتحضير الكبريت النانوي المحمل على السليلوز النانوي

علي سعيد مصطفى

الكبريت العنصري (S_8) يلعب دورا حيويا في تغذية النبات، لكن قابليته المنخفضة للذوبان في الماء تحد من توافره البيولوجي وكفاءته الزراعية. تقدم هذه الدراسة طريقة كيميائية جديدة وبسيطة لإذابة الكبريت الزراعي باستخدام هيدروكسيد البوتاسيوم، يليها تخليق جزيئات الكبريت النانوية وتثبيتها من خلال التحميل على السليلوز النانوي. تتضمن عملية التحضير خطوات من الذوبان في وجود حرارة مع التحريك المستمر ومن ثم التحميل على السليلوز النانوي لضمان توزيع نانوي ثابت ومستقر للجسيمات. أكّد التحليل الحجمي للجسيمات أن الجسيمات النانوية الناتجة من الكبريت تراوحت بين 7 و31 نانومتر، في حين تراوحت ألياف السليلوز النانوي بين 30 و50 نانومتر. وعند دمجهما، أظهر مركب الكبريت النانوي–السليلوز النانوي تجانسًا دقيقا (6.5–26.92 نانومتر) وانخفاضًا في التكتل، كما تم توثيقه بواسطة المجهر الإلكتروني النافذ (TEM). تُعدّ هذه التركيبة النانوية الهجينة واعدة للغاية في تحسين التوافر الحيوي للكبريت، وضمان إطلاقه المتحكم به، وتعزيز كفاءة استخدام العناصر الغذائية ضمن أنظمة الزراعة المستدامة. وتُظهر الدراسة استراتيجية نانوية خضراء لتحويل الكبريت ضعيف الذوبان إلى سماد نانوي فعال وقابل للتطبيق على نطاق واسع في مجالات التغذية ومكافحة الأمراض الفطرية.

الكلمات المفتاحية: الكبريت النانوي؛ السليلوز النانوي؛ إذابة الكبريت الزراعي؛ الأسمدة الذكية؛ التحليل الحجمي للجسيمات، المجهر الإلكتروني النافذ (TEM)؛ الزراعة المستدامة.