Journal of Applied and Natural Science
15(3), 937 - 944 (2023)
ISSN : 0974-9411 (Print), 2231-5209 (Online)
journals.ansfoundation.org

Research Article

Synthesis and characterization of Nano sulphur: Exploring its potential as slow release fertilizer

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Article Info
https://doi.org/10.31018/jans.v15i3.4665
Received: May 4, 2023
Revised: July 28, 2023
Accepted: August 3, 2023

How to Cite

Yazhini, R. I. *et al.* (2023). Synthesis and characterization of Nano sulphur: Exploring its potential as slow release fertilizer. *Journal of Applied and Natural Science*, 15(3), 937 - 944. https://doi.org/10.31018/jans.v15i3.4665

Abstract
Sulphur is rapidly being recognized as the fourth key nutrient for plants after nitrogen, phosphorus, and potassium. It functions in several critical metabolic and physiological processes, such as Chlorophyll synthesis, Protein synthesis, Activation of enzymes, Stress tolerance and Seed production. In this background, an attempt was made to synthesize nano sulphur fertilizers for slow release using the reverse microemulsion (water-in-oil microemulsion) technique. Cyclohexane was used as oil phase. Tween-80 and ethanol were used as surfactant and co-surfactant, respectively. Hydrochloric acid and sodium polysulfide solution acted as an aqueous phase. This technique resulted in the successful synthesis of nano sulphur fertilizer. The sulphur nano fertilizer was characterized using X-ray diffraction (XRD), Fourier-Transform infrared spectroscopy (FT-IR), Scanning electron microscope (SEM) and Thermogravimetric analysis (TGA). The XRD pattern revealed the orthorhombic nature of nano sulphur and the lattice face-centred. The FTIR spectra at 1406 cm^{-1} confirmed the sulphur vibrations. The microemulsion method yielded stable, uniform, spherical nano sulphur particles with dimensions ranging from 25 to 47 nm. The thermal disintegration between 117°C to 122°C in TGA graph was attributed to the sublimation of sulphur in orthorhombic crystalline form, indicating the successful synthesis of nano sulphur. A laboratory study on nano sulphur fertilizer and conventional sulphur fertilizer was studied with a Percolator reaction system to evaluate the slow release of sulphur from both fertilizers at ambient temperature. Percolation reactor experiment indicated that sulphate release from nano sulphur was longer for 42 days than gypsum amended soil which exhausted within 35 days. Hence, synthesized nano sulphur fertilizer maximizes nutrient retention, eliminates environmental nutrient loses and decreases fertilizer requirements.

Keywords: FT-IR, Microemulsion, Nano sulphur, Percolation reactor, SEM, Slow release of sulphate, XRD

INTRODUCTION

Sulphur is rapidly being recognized as the fourth key nutrient for plants after nitrogen, phosphorus, and potassium. While phosphorus and sulphur are comparable and critical nutrients for plant growth, sulphur got less attention for a long time because fertilizers and atmospheric inputs provided adequate soil quality. Sulphur is a naturally occurring element that ranks thirteenth in terms of abundance in the earth's crust

(Verma *et al.*, 2020). Deficiency of sulphur in Indian agriculture was acquainted as a foremost problem after the green revolution owing to the application of high-analysis fertilizers which are deficient in sulphur, low sulphur returns from farmyard manure, high-yielding crops, and intensive agriculture, decreased usage of sulphur fungicides, and reductions in atmospheric inputs as a result of tighter emission regulations (Ghosh, 2022). Areas of sulphur deficiency have recently become common across the globe (Zenda *et al.*, 2021) (Scherer, 2001).

India suffers from severe sulphur shortage, especially in Tamil Nadu, and it is worse in coarse-textured soils. The deficiency of sulphur worldwide is assessed to be about 20% in North America, 50–70% in Western Europe, 40–45% in India, and 30% in China. Moreover, mild to moderate deficiencies in arable lands exist globally (Dhaliwal *et al.*, 2022). A deficiency of sulphur can harm crop yield and quality because it is essential for the synthesis of proteins and enzymes, metabolism of carbohydrates, oil content, and protein contentand because it is a component of major three amino acids methionine, cystine and cysteine, chlorophyll, vitamins B, biotin, and thiamine. As each fertilizer unit produces between three and five units of edible oil, sulphur is considered a primary nutrient in oilseeds (Nazar *et al.*, 2011).

It is well known that sulphur fertilizer increases the yield and quality of crops, including safflower (Debnath and Basu, 2013), sesame (Shah *et al.*, 2013), and groundnut (Rao *et al.*, 2013). The most affordable and effective sulphur source among several sulphur fertilizers is gypsum. The low sulphur utilisation efficiency in typical field soil conditions is between 8% and 12%. The majority of sulphur is found in organic forms. Bacteria and mycorrhizae play a crucial role in their conversion to water-soluble ions and transportation into the plant, which is the main cause of sulphur's low nutrient utilisation efficiency.

The potential for nanotechnology as a developing field of research in agriculture is enormous. Nano fertilizers may have advantages over their inorganic equivalents in terms of high efficacy, low ecological risks, and low cost. The main disadvantage of using standard inorganic fertilizers is significant nutrient loss through volatilization and leaching, which might be significantly reduced by using nano fertilizers. Depending on the nutrient needs of plants, nano fertilizers can be created. The production of nano fertilizers and their formulation includes a wide variety of elements and compounds (Muhammad *et al.*, 2020).

When tested for its effectiveness in preventing the powdery mildew of *bhendi* caused by *Erysiphe cichoracearum*, nano sulphur of size 50-90 nm showed significantly superior fungicide effects when compared to conventional sulphur against the fungus *Aspergillus niger*. It was also tested for its performance in reducing the powdery mildew by better reducing conidial germination in the applied treatments as compared to those equivalent. These results illustrate the tremendous perspective of sulphur nanoparticles as an environmentally friendly and long-term defence against crop damage method (Gogoi *et al.*, 2013).

Several chemical techniques have been used for producing sulphur nanoparticles of specific sizes and shapes, including the wet chemical precipitation method (Shankar *et al.*, 2018) (Shankar and Rhim, 2018), microemulsion method (Soleimani *et al.*, 2013), electrochemical method (Anandgaonker *et al.*, 2019), template method using an eggshell membrane (Roy *et al.*, 2021). Microorganisms, enzymes, and plant extracts can all be used in biological processes known as "green synthesis" to synthesize nanoparticles. Sophora japonica pod extracts were employed as capping, dispersion, and stabilising agents for bio-synthesised sulphur nanoparticles (Joshi, 2022). Natural clays and zeolites were reduced to nano-dimension for the synthesis of nano-S fertilisers (1–100 nm). Compared to conventional fertilizers, these fertilizers deliver nutrients over a longer period (Subramanian *et al.*, 2015). Although these sorts of fertilisers are capable of controlled release, S usage efficiency could yet be increased. Numerous processes were used to create nano sulphur in order to boost its effectiveness in use. In this background, the present study aimed to synthesize nano sulphur by reverse micro emulsion technique for slow release and steady availability of sulphur nutrition to crops.

MATERIALS AND METHODS

Preparation of sodium polysulfide solution

Sulphur powder was taken in a mortar and grounded thoroughly. 40g of grounded sulphur powder was taken in a beaker filled with 100 mL of 6M sodium sulphide solution. The reaction occurred at room temperature for 60 minutes under constant stirring. As the reaction continued, colour of the solution slowly changed to orange with the dissolving of sulphur and sodium polysulfide (Na_2S_x) solution was prepared.

$$(x-1)S + Na_2S \rightarrow Na_2S_x \qquad \text{Eq. 1}$$

Synthesis of sulphur nanoparticles

Nano sulphur synthesis involves stirring and mixing two reverse microemulsions. In a reverse microemulsion, the aqueous phase was dispersed as tiny droplets in the oil phase. Here, the oil phase acts as the continuous phase, and the water phase acts as the dispersed phase.

Stable reverse microemulsions were obtained by mixing cyclohexane as oil phase, ethanol as co-surfactant, the non-ionic surfactant tween-80 and 6 mol/L sodium

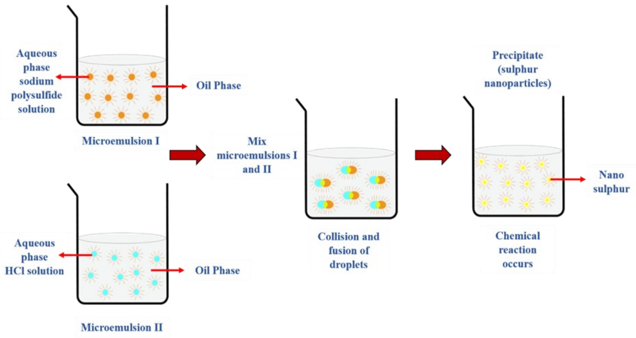

Fig. 1. *Synthesis of sulphur nanoparticles by microemulsion technique*

polysulfide solution as aqueous phase (microemulsion I) or 6 mol/L hydrochloric acid solution as aqueous phase (microemulsion II). Cyclohexane, tween-80, and ethanol were mixed under continuous stirring till the blend turned out to be transparent. Then the appropriate amount of 6 mol/L sodium polysulfide solution (microemulsion I) or 6 mol/L hydrochloric acid solution (microemulsion II) was added dropwise under strong stirring till the blend turned out to be transparent.

The microemulsion II was added dropwise to microemulsion I under stirring at room temperature. At the end of the reaction, acetone was combined with microemulsion to source precipitation of sulphur nanoparticles synthesized in microemulsion. The precipitate was removed by centrifugation and repeatedly washed with acetone, methanol, and water to eliminate residual organic constituents and salt formed (NaCl) from the product to make it pure. Finally, the product was dried in an oven (Fig. 1) (Soleimani *et al.*, 2013)

Equation depicts the reaction that resulted from the blending of these two microemulsions.

$$Na_2S_x + 2HCl \rightarrow 2NaCl + H_2S + (x - 1)\,S \qquad \text{Eq. 2.}$$

Characterization of nano sulphur:

Scanning Electron Microscope (SEM)

The surface morphology of synthesized nano sulphur was characterized using a scanning electron microscope (SEM) (Quanta 240, FEI Techni Sprit, Netherlands). The synthesized sulphur powder was placed in a vacuum chamber for sputter coating with gold (Au), involving a sputter coater (EMITECH SC7620 sputter coater). After sputter coating, the material remained inside the SEM's sample holder and was imaged to identify the area of interest (Cheng *et al.*, 2023).

Transmission Electron Microscope (TEM)

Using TEM (Quanta 240, FEI Techni Sprit, Netherlands), the morphology of nano sulphur was analyzed. The transmission electron micrographs were taken with a W-source and an ultra-high resolution pole piece (Laue, 2023).

X-Ray Diffractometer (XRD)

The lattice structure, crystalline size and d spacing of the synthesized nano sulphur were characterized using a pANALYTICAL-Xpert pro X-Ray Diffractometer with Cu K line as incident radiation and a filter at a scanning rate of 5° s^{-1}. The powdered sample of 0.5g was used for XRD measurement (Shalaby *et al.*, 2022).

Fourier Transform Infrared Spectroscopy (FT-IR)

The functional group of nano sulphur fertilizer was observed using FT-IR. A Bio-Rad Excalibur 3000 MX FT-IR spectrometer and a helium-purged MTEC 300 photo-acoustic cell were used to gather the spectral data (Subramanian *et al.*, 2022). The most useful infrared region used for characterization of nano sulphur lies between 4000 and 500 cm^{-1}. The term infrared covers the range of electromagnetic spectrum between 0.78 and 1000 µm. In Infrared spectroscopy, the wavelength is usually measured in wavenumbers cm^{-1} (wavenumber = 1/wavelength in centimetres).

Thermogravimetric analysis (TGA)

TGA analysis is a technique for thermal analysis in which the mass of a sample is determined as the temperature rises with time. The TGA was performed using TG/DTA – EXSTAR/6300 thermogravimetric analyser (Sugumaran *et al.*, 2023). The thermal stability and composition of materials, as well as their decomposition mechanisms, kinetics, and thermal properties were determined.

Percolation reactor experiment for nutrient release

A percolation reactor was built to provide a consistent solution flow through a test soil column. The experimental setup was basically the same as reported by (Thirunavukkarasu and Subramanian, 2014). The percolation reactor consisted of a glass column (internal diameter = 1.5 cm; height = 25 cm) through the top, of which ultrapure water was continuously supplied at a flow rate of 20 mL day^{-1}. It was filled with 10 g of soil, and then 1.0 g of nano sulphur was dispensed on top. Leachates were gathered in order to figure out sulphur concentration. The temperature was 25°C on average throughout the experiment. A 25 ml volumetric flask was filled with 5 ml filtrate, 10 ml sodium acetate acetic acid buffer (pH 4.8), and 1 ml gum acacia. In a 25 ml volumetric flask with 10 ml of sodium acetate acetic acid buffer and 1 ml of gum acacia, potassium sulphate standard solution concentrations of 1, 2, 3, 4, and 5 ppm were prepared simultaneously. At the time of UV-VIS Spectrophotometer reading, 1 g of barium chloride was added to each volumetric flask, and the solution was made up to the mark. The absorbance of each solution was read at 420 nm on UV VIS Spectrophotometer after calibrating the absorbance to zero with blank. The sulphur standard curve calculated the sam-

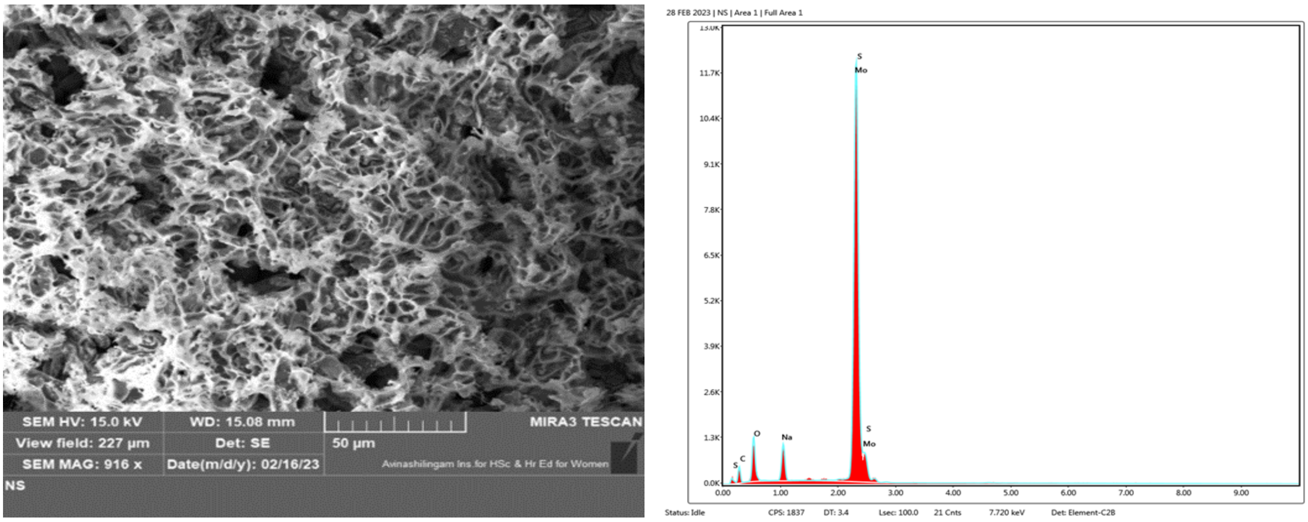

Fig. 2. *Characterization of nano sulphur synthesized by microemulsion by (a) SEM, (b) EDAX to study the homogeneity and chemical makeup*

ple's sulphate concentration (Chesnin and Yien, 1951).

RESULTS AND DISCUSSION

SEM and TEM analysis

The surface morphology of sulphur nanoparticles was determined using Field Emission Scanning Electron Microscopy (FESEM). Nano sulphur was made homogeneous by sonicating and the SEM image was attained (Fig. 2a). This image demonstrated that nanoparticles made using this approach were spherical in shape. The SEM results were similar to the findings of (Soleimani *et al.*, 2013) that the aggregation of sulphur crystals was prevented during synthesis because the chemical interaction of sodium polysulfide and hydrochloric acid was space-confined in the water droplets of microemulsions. The size of the sulphur nanoparticles was only affected by the diameter of the water droplets. Micelles that entrapped reactive elements could restrict particle growth.

The chemical makeup and purity of the synthesised nanoparticles were confirmed using energy-dispersive X-ray analysis (EDAX). The product's EDAX spectrum is shown in Fig. 2b, and the data show that it was entirely free of impurities and contained only the sulphur element. Weight % and atomic % of sulphur were quantified as 54.34 % and 42.83 %, respectively, in the ZAF Matrix (correction matrix used to compensate for the atomic number (Z), absorption (A), and fluorescence (F) effects) (Fig. 2b). Based on the data provided. It is evident that the product consists solely of sulfur and contains no impurities.

TEM was used to analyse nanostructured materials with atomic-scale resolution. TEM image revealed an average particle size between 25-47nm (Fig. 3). Its particle distribution and surface morphology supported the homogenous nature of nano sulphur.

XRD analysis

Using X-ray diffraction technique, synthesised nano sulphur's phase purity and crystallinity showed that the diffraction peaks reached different angles and the corresponding lattice planes indicated the synthesis of pure sulphur nanoparticles. The XRD pattern displayed the orthorhombic nature of sulphur nanoparticles and lattice face-centred. The study results were similar to the findings of Radhika *et al.* (2018) ; Paralikar and Rai (2018) who confirmed the crystalline nature of sulphur nanoparticles based on the position and intensity of diffraction peaks. For sulphur nanoparticles, Bragg's reflections peaks at 2θ values of 23.124, 26.404, 27.758, 31.466, 37.171 and 43.533° were referred to crystal planes of sulphur at 222, 224, 206, 044, 317, and 426, respectively, representing crystalline nature of sulphur nano particles (Fig. 4). These values agreed with Joint Committee on Powder Diffraction Standards (JCPDS card No. 65-1436).

FT-IR analysis

Nanosulphur was characterized using FT-IR. The FT-IR spectra produced strong, broad bands at 2972 cm-1 and 2902 cm-1. These bands were associated with O-H stretching and N-H stretching of carboxylic acid, alcohol, water, phenols, and amine salt compound class, mostly intramolecular bonded at 3000 cm-1. The O-H stretching of alcohols was responsible for sharp peaks at 3666 cm-1. The broad band at 3381 cm-1 was due to N-H stretching of aliphatic primary amines. C-O stretching in aromatic rings elucidate a peak at 1253 cm-1 due to aromatic ester and alkyl aryl ether presence. The peak at 868 cm-1 relates to C-H bending of 1,2,4- trisubstituted compounds. The strong appearance of spectra peaks at 1406 cm^{-1} was responsible for S=O stretching of sulphate and sulfonyl chloride compounds (related to sulphur vibrations). The sulfoxide group ap-

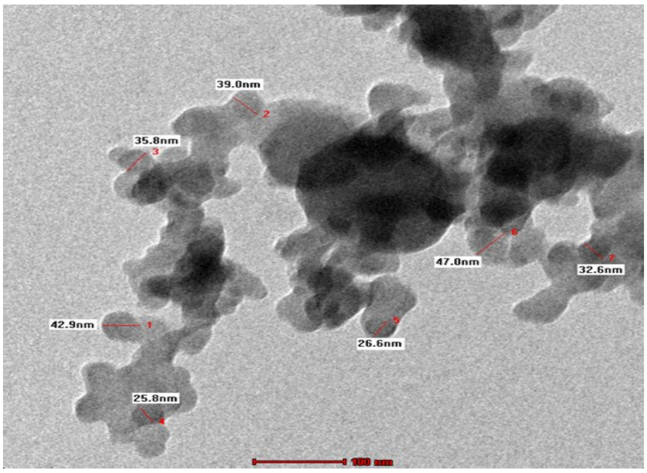

Fig. 3. *Characterization of nano sulphur synthesized by microemulsion by TEM*

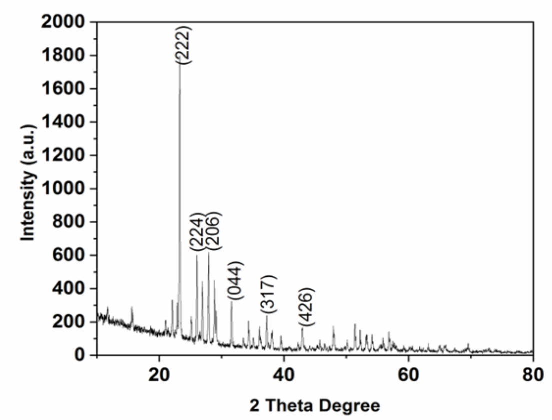

Fig. 4. *XRD spectra of nanosulphur*

peared as strong S=O stretching at 1053 cm-1 (Fig. 5). The functional groups related to sulphur vibrations occurred in the fingerprint region of FT-IR spectra, which confirmed the successful synthesis and characterization of nano sulphur. The peaks in the spectrum of the nano sulphur exhibit identical shapes to those observed by Thirunavukkarasu and Subramanian (2014), confirming the synthesis of sulphur.

TGA analysis

TGA analysis showed that the slight decline in weight at 100°C, as seen in TGA curve, was attributed to the loss of free water. Weight loss up to 250°C has been attributed to loss of constitution water paralleled with alteration of sulphur's crystalline character. There were two allotropic sulphur forms: α-form (Orthorhombic crystal structure) and β-form (Monoclinic crystal structure). Both allotropes were yellow, with the α-form a brighter yellow and the β-form a paler, whitish-yellow. Both rhombic and monoclinic crystalline forms of sulphur existed. The thermal disintegration after 100°C between 117°C and 122°C was also observed by Momoniat and Rahmanian (2022) due to the melting and sublimation of sulphur in rhombic crystalline form. The graph's trough above 170°C confirms the transition of sulphur into a new allotrope and the colour changed from yellow to red. In the DTG graph, the peak was also shown at this range as the rate of material weight changed after gradual heating.

There were four types of sulphur: rhombic sulphur, monoclinic sulphur, liquid sulphur, and gaseous sulphur. The transition of sulphur from its rhombic to monoclinic forms resulted in the first peak of DTG analysis, which had a temperature range of 103°C to 107°C. Thereafter, the monoclinic sulphur sublimed into liquid sulphur, transforming into a deeper colour at the second peak, which ranged in temperature from 117 to 122°C. The TGA graph obtained with the thermal breakdown peaked after 300°C was the same as obtained by Momoniat and Rahmanian (2022); and Shankar and Rhim (2018) due to the result of liquid sulphur turning into gas. Weight loss in TG% was 44.6% between 300 – 310°C, 48.6% between 330 – 530°C and a total of 98.7% was recorded as a thermal breakdown in the TGA analysis (Fig. 6).

Release kinetics of nanosulphur

Over seven weeks, periodic assessment of sulphate release from soil applied with conventional sulphur (gypsum) and nano sulphur showed that leachate sulphur content gradually rose while the experiment progressed until the seventh week. In contrast, as the experiment further progressed, sulphur release from gypsum-amended soil dropped. On the last day of the experiment, the soil treated with nano sulphur had significantly more sulphur (9.5 mg kg^{-1}) than the soil supplemented with gypsum (7.4 mg kg^{-1}), and the percentage increase was 22.1%. Throughout the investigation, sulphur content in control was recorded the least as 0.5 mg kg^{-1}. The use of nano sulphur produced by microemulsion as a source of slow-release fertiliser is being investigated in this preliminary study.

The average amount of SO_4^{2-} emitted by gypsum as a source of sulphur, Nano sulphur, and control were

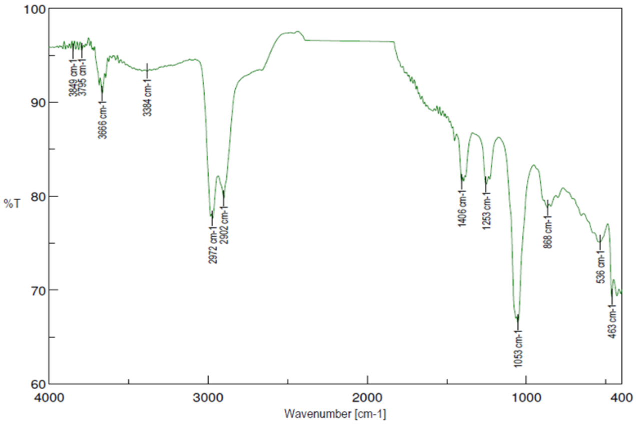

Fig. 5. *FT-IR spectra of nanosulphur*

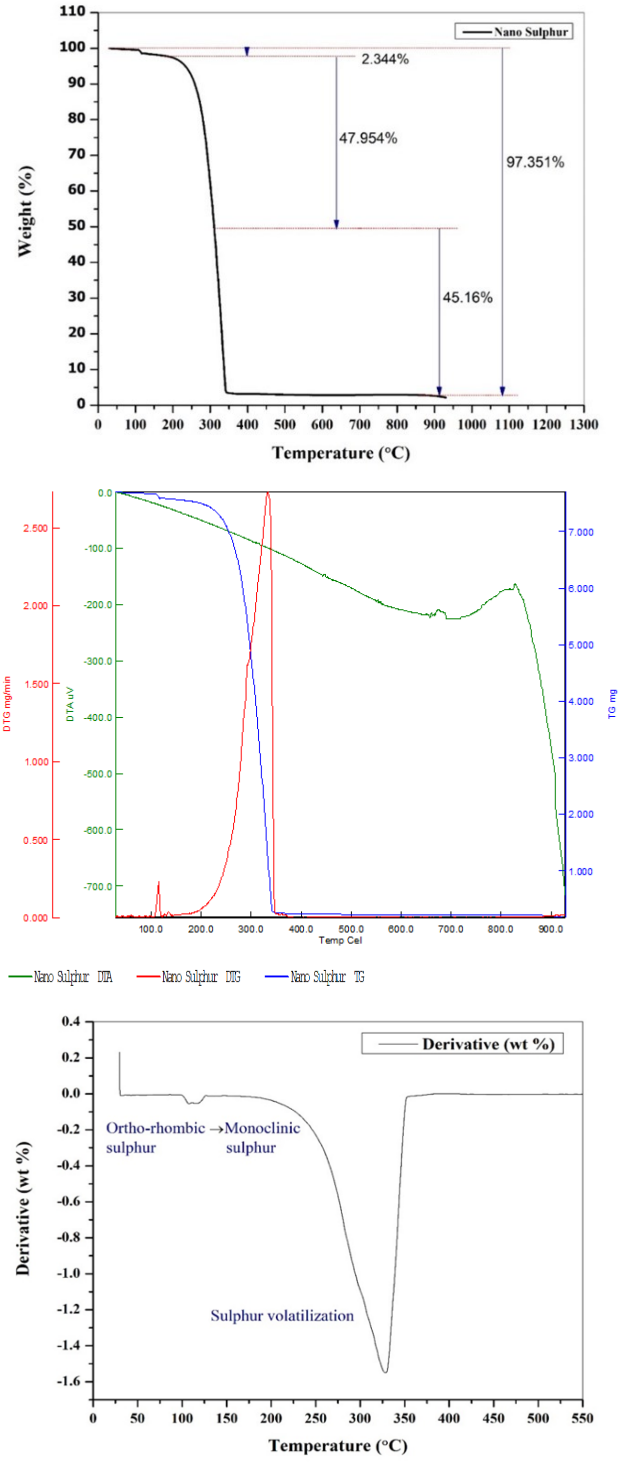

Fig. 6. *TGA analysis curves for nanosulphur*

11.63 mg kg^{-1}, 8.59 mg kg^{-1}, and 1.5mg kg^{-1}, correspondingly. Gypsum's rise over control was 87.1%, whereas nano sulphur was 82.54% (Fig. 7). The Nano sulphur functioned as a slow release of sulphur nutrients. Hence a modest increase was observed throughout the experiment. Similar results were obtained by Lateef *et al.* (2019), which might be due to the small size (nanometer) range of nano sulphur fertilizer. The reduced particle size increases the surface area, allowing for a larger contact area with the surrounding environment. This increased surface area facilitates a slower release of nutrients as the sulphur particles gradually dissolve or react with the soil components.Carmona *et al.* (2022) reported that nano sulphur particles exhibit higher reactivity than conventional sulphur fertilizers. This increased reactivity allows for gradual chemical reactions with the soil moisture and components, leading to a controlled release of nutrients over an extended period. By maintaining a renewable and sustainable nutrient supply in the rhizosphere region of the crop, the nano sulphur maximises nutrient retention, eliminates environmental nutrient losses, and decreases fertilizer requirements.

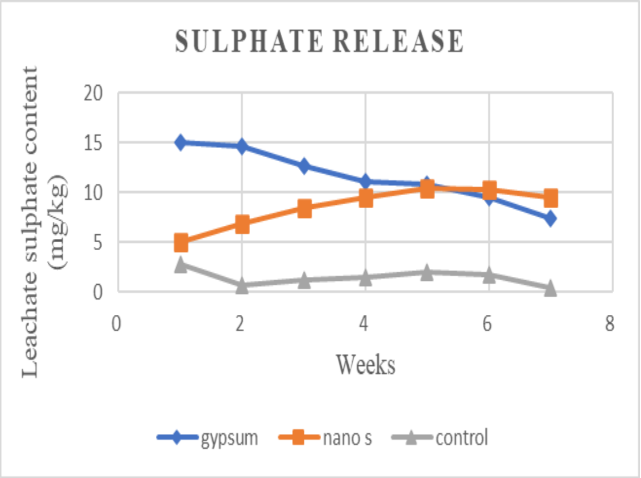

Fig. 7. *Comparison of sulphate release from control, soil applied with gypsum and nano sulphur.*

Conclusion

The microemulsion method worked well to produce nano sulphur. The nano sulphur is stable, spherical, and uniform with a dimension of 25-47nm as imaged by SEM and TEM. Besides, XRD showed orthorhombic crystalline nature of sulphur. FT-IR, TGA and SEM-EDAX confirmed the successful synthesis of sulphur nanoparticles. The nano sulphur synthesized could be used as a viable option for the slow release of nutrients to crops. The release kinetics of nutrients suggest that sulphate release in nano sulphur-amended soil was seven days longer than in conventional fertilizer-amended soil. These results encourage that nano sulphur has great potential as fertilizer for the slow release of nutrients.

ACKNOWLEDGEMENTS

The authors thank the CANT- Centre for Agricultural Nanotechnology, Tamil Nadu Agricultural University for providing facilities to carry out the research.

Conflict of interest

The authors declare that they have no conflict of interest.

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