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Influence of Soil Texture on the Mineralization of Sulphur Nanoparticles under Incubation

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Background: Nanotechnology has a great potential for achieving sustainable agriculture. Nano sulphur is highly homogeneous in the distribution to the ground with an average particle size of within 100 nm.

Study Design & Place: An incubation study was conducted at the laboratory, Micronutrient Research Centre, AAU, Anand, to study the effect of sulphur nanoparticles over conventional sulphur source on sulphur availability up to 60 days.

Methodology: Incubated soils were studied with the different levels of sulphur NPs in loamy sand and clayey soil at an interval of time *i.e.,* 10, 20, 40 and 60 days. The soils were treated with four levels of sulphur nanoparticles (0, 2, 4 and 8 mg S/kg soil) and one level of 8 mg S/kg soil through Elemental S. The each set (40) was completely withdrawn after 10, 20, 40 and 60 days and was analyzed for 0.15% $CaCl_2$-extractable sulphur.

Results: This lab study was stated that availability of sulphur up to 60 days in loamy sand soil was found higher with T_5 (8 mg S/kg soil through ES) was 20.30 ppm at 40 days. In case of clay soil higher with T_4 (8 mg S/kg soil through SNPs) was 64.34 ppm at 40 days. In case of fractionation at 60 days of incubated loamy sand soil, all forms of sulphur was found higher with T_4 (8 mg S/kg soil through SNPs) except sulphate sulphur and non-sulphate sulphur. In case of clay soil fractionation all forms of sulphur was found with T_4 (8 mg S/kg soil through SNPs) except non-sulphate sulphur. All forms of sulphur was found higher in SNPs treatments except non-sulphate sulphur in both loamy sand and clay soils. Under incubation study, the periodical availability of sulphate sulphur was found increasing pattern up to 40 days after application in soil and thereafter in declining trend in both soils and source of sulphur. Hence, looking to the overall observations, the sulphur nanoparticles proved better in increasing the periodical availability of sulphur as compared to elemental sulphur in loamy sand and clay soils.

Keywords: Elemental Sulphur; fractionation; Sulphur nanoparticles; sulphate.

ABBREVIATIONS

SNPs : Sulphur Nano-particles
ES : Elemental Sulphur

1. INTRODUCTION

The word nano-means "one-billionth of a meter (nm) size" ranges from 1 to 100 nm. Due to small size, nanoparticles have many unique properties that are being explored for new opportunities in agriculture comprises atom-by-atom manipulation and products developed are quite precise (Mikkelesen, 2018). Nano-fertilizer having higher surface area and auspicious picking for improving the quality and quantity of plants and seeds grown for consumption, to minimize production cost as well as eco-friendly to sustainable food production (Mani and Mondal, 2016). Nano-fertilizers could be more soluble or more reactive than bulk fertilizers and they can exactly release their active ingredients in responding to environmental triggers (Mastronardi *et al*., 2015). Nano-fertilizer have high surface area, increased solubility, small particle size <100 nm, controlled release of nutrients due to encapsulation and increased nutrient efficiency (Yuvaraj and Subramanian, 2015). However, some of the reports and patents strongly suggest that there is a vast scope for the formulation of nano-fertilizers (Tarafdar *et al*., 2012). Subramanian and Rahale (2013) stated that nano-fertilizers are capable of releasing nutrients, especially nitrate nitrogen for more than 50 days while nutrient release from conventional fertilizer (urea) ceased to exist beyond 10-12 days and also suggested that nano-fertilizers may be used as a strategy to regulate the smart release of nutrients that commensurate with crop requirement. Subramanian *et al.* (2015) reported that nano-fertilizers and nanocomposites can be used to control the release of nutrients from the fertilizer granules so as to improve the nutrient use efficiency while preventing the nutrient ions either get fixed or lost to the environment. Nano-fertilizers have high use efficiency and can be delivered in a timely manner to a rhizospheric target (Liu *et al.,* 2005). Sulphur is an essential macronutrient in plant growth and development. Sulphur is now recognized as the fourth major plant nutrient (Tandon *et al*., 2004; Oakley *et al.,* 2007). Sulphur plays important role in formation of three amino acids (cysteine, cystinine, methionine), activation of enzymes, winter hardiness, quality nutrient in oilseed (Prasad and

Shivay, 2017) (Mengel *et al*., 2001). Besides it is involved in various metabolic and enzymatic process including photosynthesis, respiration and legume rhizobium symbiotic nitrogen fixation (Rao*,* 2001). Sulphur is a basic element of nature and is one of the most abundant elements in the earth's crust. Sulphur is considered as quality nutrient as its application not only influences crop yield, but also quality, owing to its influence on protein metabolism and oil synthesis (Patel *et al.,* 2009; Priyadharshan et al., 2024). Sulphur nanoparticles has a great potential as fertilizer carrier to control release of sulphate by the slow release mechanism. Despite several agronomic strategies tested for improving the use S efficiency, it proved less success due to complex soil environmental factors. Sulphur use efficiency hardly exceeds 25%. Smaller size of nano-sulphur and its coating will help resist unwanted environmental processes associated with conventional fertilizer, *i.e.,* leaching, evaporation, and photolytic, hydrolytic and microbial degradation. Nano-sulphur particle are 1000 times smaller than elemental sulphur applied as soil application releases sulphur in 3-4 weeks compared to 4-6 month in elemental. Depending on the surface size, sulphur bacteria work very quickly. So it can be converted into forms that can be taken by plants within 1-2 days. The use of Nano S prevents the use of excess fertilizer and negatively affects the environment and human health of fertilizers. As a result, nearly 100% efficiency is obtained (Kaya *et al.,* 2018). The objective of this study provides critical insights into the role of soil texture in the transformation and bioavailability of sulphur nanoparticles. Understanding these dynamics is essential for optimizing the application of nanotechnology in agriculture, ensuring effective nutrient delivery, and minimizing potential environmental risks. This study bridges a significant knowledge gap in nanoparticle-soil interactions, offering valuable data for sustainable agricultural practices and soil fertility management. The findings could also guide future research and policy-making in the field of nanotechnology-driven agriculture.

2. MATERIALS AND METHODS

Collection and preparation of soil sample: The incubation study was conducted at laboratory of Micronutrient Research Center (ICAR), Anand Agricultural University, Anand (Gujarat) to study the periodical availability of sulphur in soils. Two types of soil were collected 1) Loamy sand soil was collected from Agronomy farm, BACA, Anand Agricultural University, Anand and 2) Clay soil was collected from Narmada Irrigation Research Project Farm, AAU, Khandha, Ta. Karjan, Dist. Vadodara

Methodology and Observation for determination of sulphur fractions: An incubation study was conducted at the laboratory, Micronutrient Research Centre, AAU, Anand, to study the effect of sulphur nanoparticles over conventional sulphur source on sulphur availability up to 60 days. The 100 g soil filled in the beaker and soil moisture was maintained at field capacity. Incubated soils were studied with the different levels of sulphur NPs in two type of soils (loamy sand and clayey) at an interval of time *i.e.,* 10, 20, 40 and 60 days. The soils were treated with four levels of sulphur nanoparticles (0, 2, 4 and 8 mg S/kg soil) and one level of 8 mg S/kg soil through Elemental S. The treatments was repeated four time adopting CRD design. The soil moisture was maintained at field capacity (FC) *i.e.,* 50% MWHC (Maximum water holding capacity) throughout the incubation period. The each set of 40 cup (total 160 cup) was completely withdrawn after 10, 20, 40 and 60 days and was analyzed for 0.15% $CaCl_2$-extractable sulphur. Soils was observed for moisture content, which is maintained at field capacity (FC) *i.e.,* 50% of MWHC throughout the incubation period. A 40-beaker set (20 loamy + 20 clay) was withdrawn after 10, 20, 40, 60 days and analyzed for 0.15% $CaCl_2$ extractable sulphur. Before analysis samples was air dried completely before 2-3 days for avoiding analysis error. Soils were analyzed for different forms of sulphur at the end of incubation adopting standard procedure as given in the Figs. 1 and 2.

Water soluble sulphur by Williams and Steinbergs (1959): Take 5 g soil and add 33 ml of 1% NaCl solution then shake it for 30 min on mechanical shaker and filter it with 42 no. filter paper. Then take 25 ml aliquot in to silica basin and evaporate to dryness with 2 ml of 3% H_2O_2. Then basin heat in an oven at 102 °C C for 1 h to ensure the removal of excess peroxide. After cooling, the residue takes with 25 ml of DW and transferred to centrifuge tube to remove suspended matter or filter it with 42 no. filter paper. After that sulphur determine as per heat soluble sulphur procedure.

$$\text{Calculation} = \frac{(\text{S} - \text{B}) \times \text{Extration added} \times \text{Final volume made}}{\text{Weight of sample} \times \text{Aliquot taken}}$$

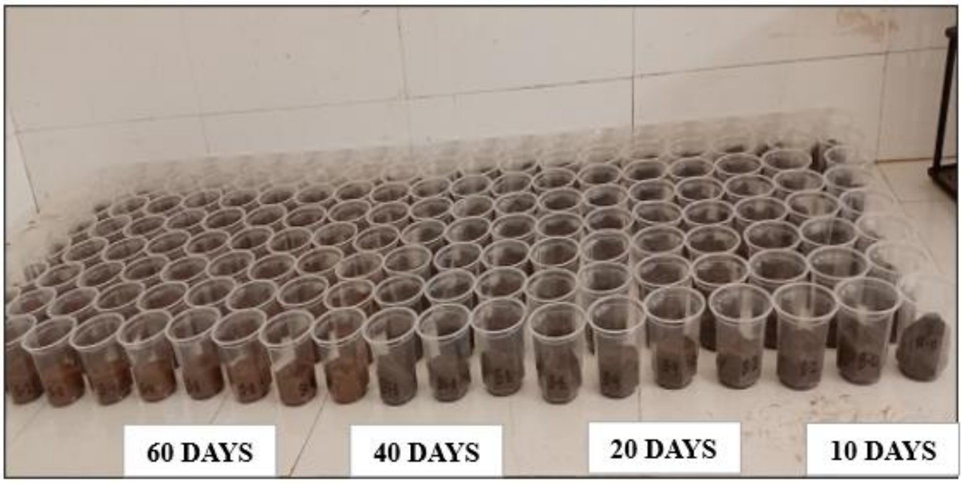

Fig. 1. Periodical availability of Sulphur in loamy sand soil

Fig. 2. Periodical availability of Sulphur in clay soil

Organic sulphur by Bardsley and Lancaster (1965): Take 10 g soil + 50 ml 1 N HCl in flask, shake it for 30 min on mechanical shaker. Filtered it and transferred entire soil with distilled water and washed with 100 ml 1 N calcium acetate. Soils dried in oven and pass through 20 mesh sieve and take 2.5 g of this soil in silica basin and add 0.5 g sodium bicarbonate mixed well with soil. Another 0.5 g sodium bicarbonate spread on the soil as surface layer, then ignited at 500 °C in furnace for 3 h. Then crucible cool and add 25 ml of sodium dihydrogen phosphate (extracting solution) and shake for 30 min on mechanical shaker. Then filter it with 42 no. filter paper. Take 10 ml filtrate in 50 ml volumetric flask, add 1 ml gum acacia solution + 1 ml of 6 N HCl swirled flask and add 0.5 g $BaCl_2$ powder allowed to stand for 1 min and then shake until the crystal is dissolved. Then make final volume and take reading within 2-3 min on 430 mµ wavelength.

$$\text{Calculation} = \frac{(\text{S} - \text{B}) \times \text{Extration added} \times \text{Final volume made}}{\text{Weight of sample} \times \text{Aliquot taken}}$$

Sulphate sulphur by Williams and Steinbergs (1959): Take 10 g soil (2 mm sieved) + 50 ml 0.15% $CaCl_2$ solution shake it for 30 min on mechanical shaker and then filtered it with 42 no. filter paper. Take 10 ml aliquot in 25 ml volumetric flask and add 10 ml morgan’s reagent + 1 g BACl2 +1 ml gum acacia solution and take reading on 430 mu wavelength.

$$\text{Calculation} = \frac{(\text{S} - \text{B}) \times \text{Extration added} \times \text{Final volume made}}{\text{Weight of sample} \times \text{Aliquot taken}}$$

Available (Heat soluble) sulphur by Williams and Steinbergs (1959): The most of sulphur is present in organic forms in soil. The fraction of this organic sulphur is being released through hydrolysis on gentle heating and become easily soluble in water. To examine this possibility, water was added to soils and then evaporated to dryness on a boiling water bath and finally dried in a hot air own, and then soil is extracted with sodium chloride. Further fraction of the soil sulphur is released by this treatment. Heat is the main factor leading to the release of additional sulphur in this treatment.

Procedure: Weigh 5 g of soil and put it in to a silica basin and add 20 ml of distilled water. The basin is then placed on a gently boiling water bath and evaporated to dryness. It is then heated for 60 min, in a hot air oven at 102 °C. After cooling, the soil is transferred to 150 ml conical flask and add 33 ml 1% NaCl. Shake it for half an hour. Filter it for half an hour. Filter it through what man no. 42 filter paper. Take suitable aliquot (10 mL) in 25 mL volumetric flask and add 10 mL morgan's reagent and 1 ml gum acacia. Shake the flask for one min and add 0.5 g $BaCl_2$ crystal and final volume is made. Shake the flak immediately for 3 min. and allow standing for 30 min. Take reading on colorimeter using blue filter or on spectrophotometer with 430 mµ wavelength.

$$\mathrm{S\ (ppm)} = \frac{(\mathrm{S} - \mathrm{B}) \times \text{final volume} \times \text{volume of extractant}}{\text{Aliquot} \times \text{Soil taken}}$$

Total sulphur by Chaudhary and Cornfield (1966), and modification of the method by Butters and Chenery (1959): Take 1 g air dried soil (0.5 mm sieved) in silica basin, add 10 mL digestion solution then evaporate to dryness on steam bath. Covered with watch glass, place in electric furnace and heat at 550 °C for 3 h. Cooled and add 5 ml of 25% nitric acid, the contents digested for an hour without loss of nitric acid on steam bath. Then covered watch glass washed with distilled water in to silica basin then filtrate it with soil through distilled water to collect 40 ml in 50 ml volumetric flask. Then add 5 ml 50% acetic acid + 1 ml orthophosphoric acid and dilute to about 45 ml + 1 g $BaCl_2$ add to each flask. Without disturbing the content flak keep as such for 10 min. Then inverted twice, the inversion was repeated after an interval of 5 min and this repeated for 10 times. Then add 1 ml gum acacia solution and dilute to 50 ml with DW and inverted twice. The flask keep as such for 90 min, then turbidity measure at 430 mµ wavelength after inverting the content for 10 times.

$$\mathrm{ppm\ S} = \frac{\mathrm{R\ (ppm)} \times 40 \times 50}{1 \times \text{aliquot taken}}$$

Non- Sulphate sulphur: Difference between total sulphur and sum of organic and sulphate sulphur.

Calculation = Total sulphur- (Organic sulphur + Sulphate sulphur)

3. RESULTS AND DISSCUSSION

Periodical availability of Sulphur in loamy sand and clay soils: The incubation study was conducted to study the availability 0.15% $CaCl_2$ extractable sulphur in loamy sand and black soils at the period of 10, 20, 40 and 60 days after incubation. The set of total 160 plastic cups were filled with soils (80 loamy sand soil and 80 black soil) and treated with different levels of SNPs and elemental sulphur and incubated in controlled conditions and a set of 40 sample was withdrawn periodically and analyzed for available sulphur in Figs. 3 & 4.

Periodical availability of Sulphur in loamy sand soil: The 0.15% $CaCl_2$ extractable S was determined in loamy sand soil (Initial Sulphur 5.01 ppm) at 10, 20, 40 and 60 days after incubation and presented in the Fig 3. The availability of sulphur up to 60 days in loamy sand soil was found higher with T_5 (8 mg S/kg soil through ES) from 13.60 ppm to 20.30 ppm from 10 to 60 days showed highest increase at 40 days followed by T_4 (8 mg S/kg soil through SNPs) from 12.14 ppm to 16.98 ppm up to 60 days showed highest peak at 40 days and then decline 14.94 ppm. The results of loamy sand soil indicated that the minimum changes were showed in treatment T_1 (control) which was within 1 ppm from 10 to 60 days of incubation and the maximum fluctuation was showed in SNPs treatment T_3 (4 mg S/kg soil through SNPs), wherein available S increase around 10 ppm with respect to initial value, while in elemental S treated soil, it was around 7 ppm. So to get the continuous supply of available sulphur, the application of sulphur through SNPs will be beneficial as compared to elemental.

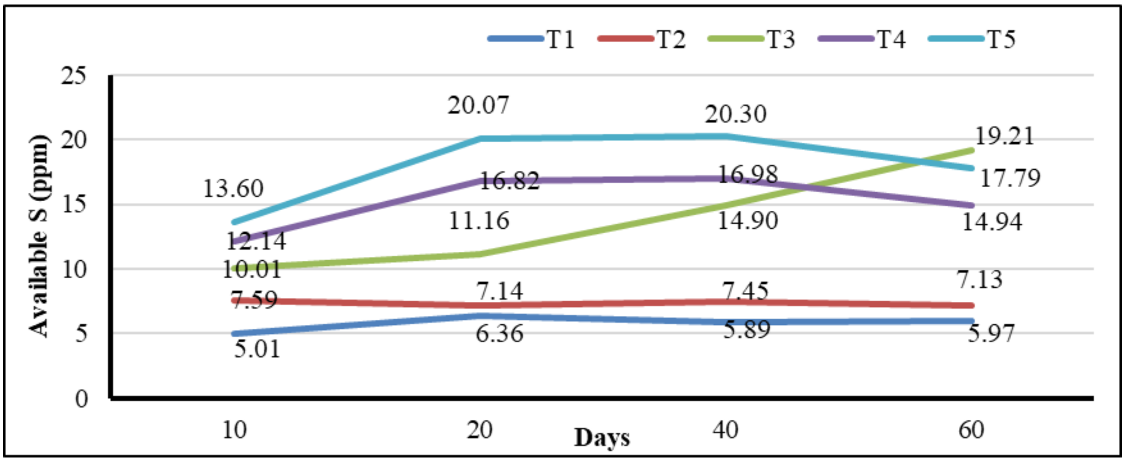

Fig. 3. Periodical availability of sulphur in loamy sand soil up to 60 days

Periodical availability of Sulphur in clay soil: Availability of sulphate sulphur (0.15% $CaCl_2$ extractable) was determined in clay soil (Initial Sulphur 36.63 ppm) at 10, 20, 40 and 60 days after incubation and presented in the Fig 4. The periodical availability of sulphur up to 60 days was found higher with T_4 (8 mg S/kg soil through SNPs) from 55.05 ppm to 64.34 ppm from 10 to 60 days showed highest increase at 40 days then declining 63.01 ppm at 60 days followed by T_5 (8 mg S/kg soil through ES) from 57.74 ppm to 62.21 ppm up to 60 days showed highest peak at 40 days and then decline 57.81 ppm at 60 days. The minimum release of S in clay soil towards application of sulphur nanoparticles showed by T_3 (4 mg S/kg soil through sulphur nanoparticles) within 3 to 4 ppm from 10 to 60 days of incubation and the maximum fluctuation was showed by T_2 (2 mg S/kg soil through sulphur nanoparticles) & T_4 (8 mg S/kg soil through sulphur nanoparticles) up to 10 ppm due to slow release behaviour of sulphur nanoparticles from 10 to 60 days.

Effect of Sulphur nano particles on different fractions of Sulphur in incubated soils: The different fractions of sulphur viz total sulphur, organic sulphur, sulphate sulphur, heat soluble sulphur, water soluble sulphur and non-sulphate sulphur was determined in incubated soils after 60 days of incubation.

Available (Heat soluble) sulphur: In loamy sand soil, the heat soluble sulphur content after 60 days was recorded significantly highest in treatment T_4 (8 mg S/kg soil through SNPs) (11.43 ppm) over T_1 control (3.78 ppm) and T_2 & T_3, but it was statistically at par with T_5 (8 mg S/kg soil through ES). Similarly, In clay type soil, heat soluble sulphur content after 60 days was also observed highest in T_4 (23.85 ppm) and value was 87.35% higher in comparison to control (12.73 ppm) in Fig 5.

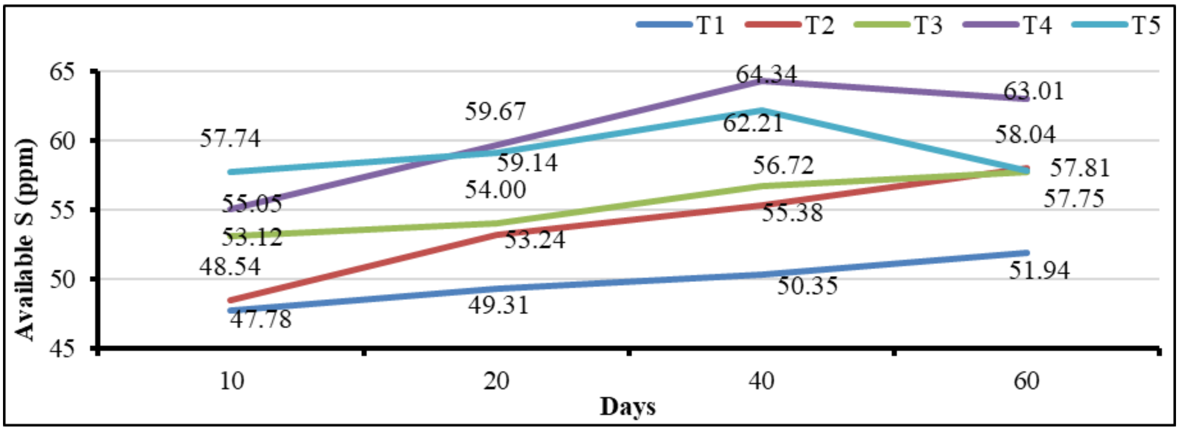

Fig. 4. Periodical availability of Sulphur in clay soil upto 60 days

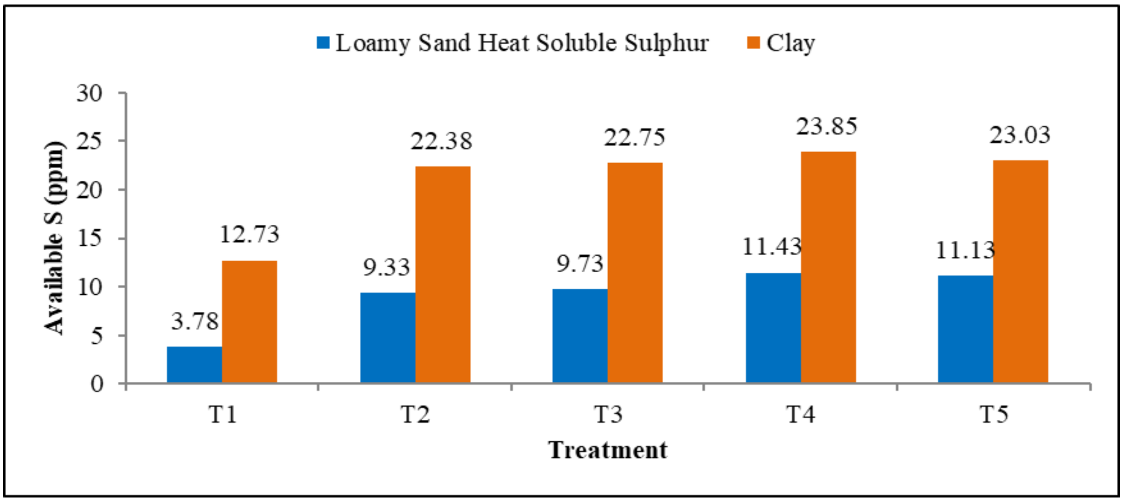

Fig. 5. Heat soluble sulphur content after 60 days in loamy sand and clay soils

Sulphate Sulphur: In loamy sand soil, availability of sulphate sulphur content after 60 days was significantly highest in T_3 (4 mg S/kg soil through SNPs) (19.21 ppm) over T_1 (Control) (5.97 ppm), while in case of clay soil, availability of sulphate sulphur content was recorded in T_4 (8 mg S/kg soil through SNPs) (63.01 ppm) over T_1 (Control) (51.94 ppm) in Fig. 6.

Total Sulphur: In loamy sand soil, the total sulphur content after 60 days was found highest in treatment T_4 (8 mg S/kg soil through SNPs) (249.18 ppm) over control (236.28 ppm), which was statistically at par with T_5 (8 mg S/kg soil through ES) (249.13 ppm). In case of clay soil, total sulphur content was found significantly highest in T_4 (8 mg S/kg soil through SNPs) (290.28 ppm) in comparison to control (276.58 ppm) in Fig. 7.

Organic Sulphur: In loamy sand soil, the organic sulphur content form after 60 days was observed highest in T_4 (8 mg S/kg soil through SNPs) (172.33 ppm) over control (171.03 ppm). In clay type soil, the organic sulphur content after 60 days was highest in T_4 (8 mg S/kg soil through SNPs) (211.43 ppm) over control (210.10 ppm) in Fig. 8.

Water-soluble Sulphur: In loamy sand soil, the water-soluble sulphur content after 60 days was found highest in T_4 (8 mg S/kg soil through SNPs) (8.48 ppm), which was statistically at par with T_5 (8 mg S/kg soil through ES) over to control (2.66 ppm). In clay soil, water soluble sulphur content after 60 days was found higher in T_4 (8 mg S/kg soil through SNPs) (19.78 ppm), in comparison to control (10.53 ppm), which was 87.84 % higher than control in Fig. 9.

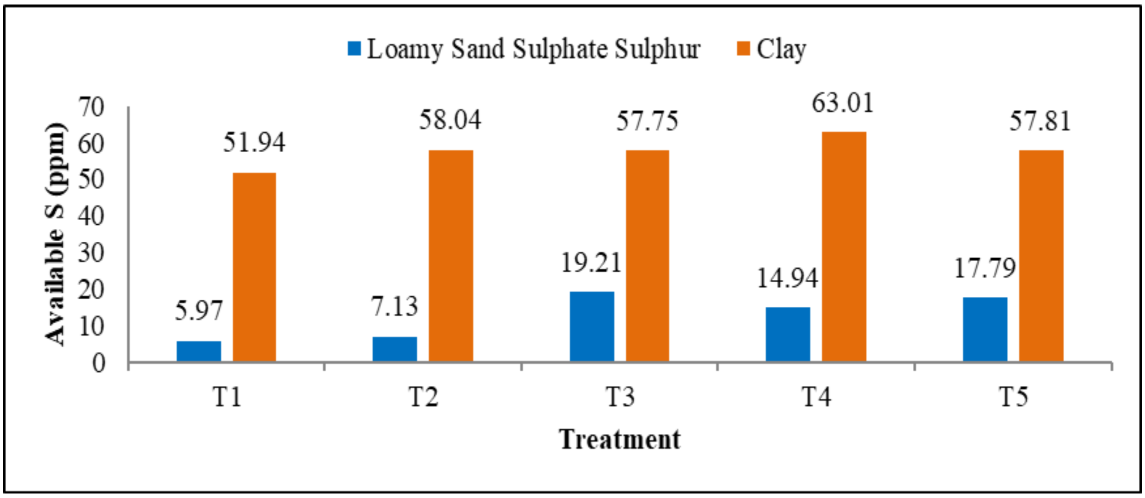

Fig. 6. Sulphate sulphur content after 60 days in loamy sand and clay soils

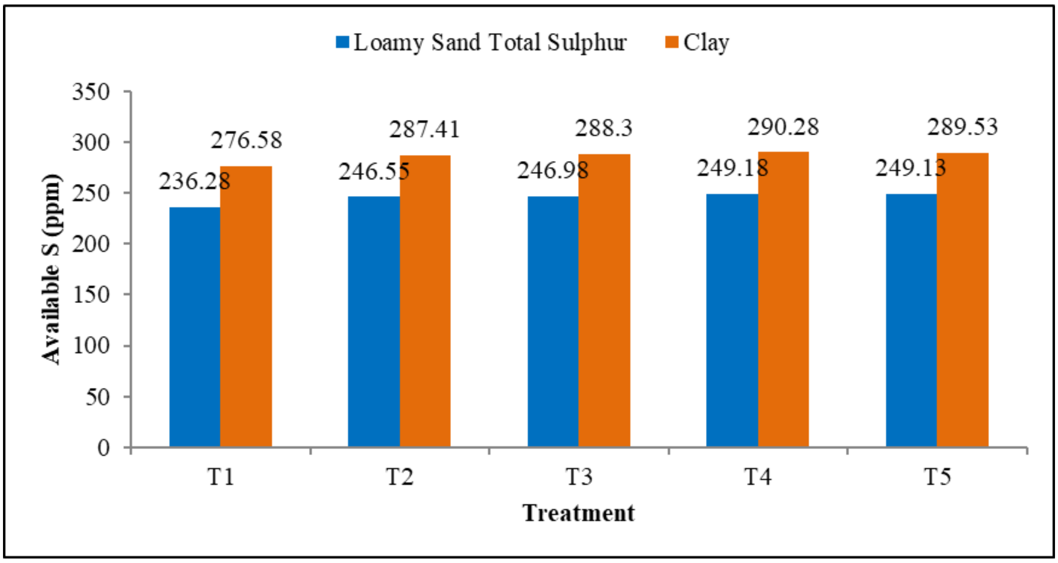

Fig. 7. Total sulphur content after 60 days in loamy sand and clay soils

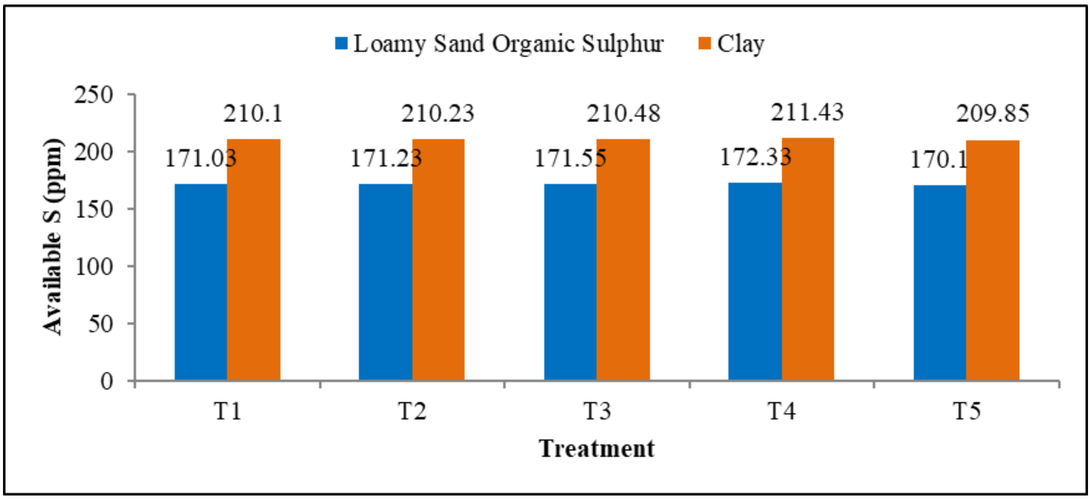

Fig. 8. Organic sulphur content after 60 days in loamy sand and clay soils

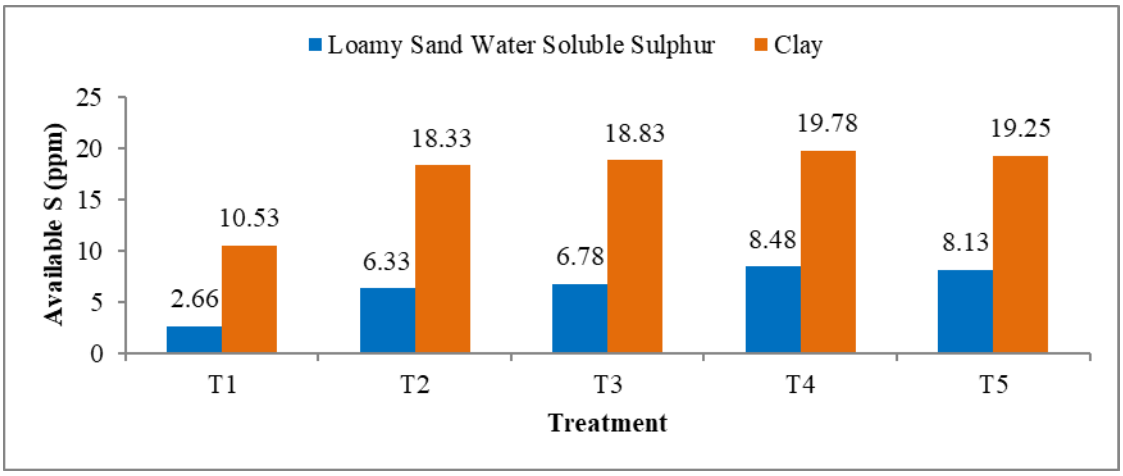

Fig. 9. Water-soluble sulphur content after 60 days in loamy sand and clay soils

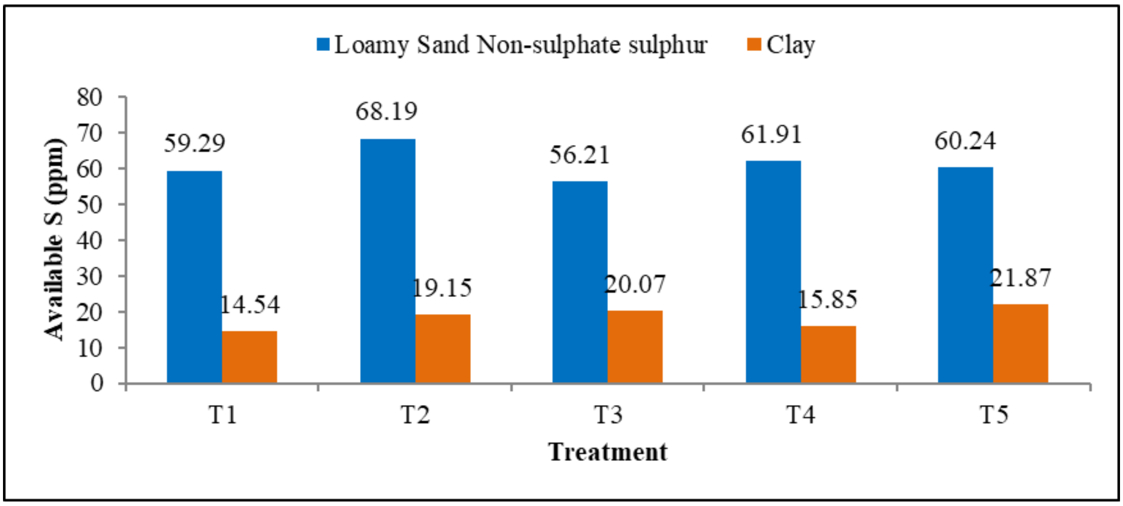

Fig. 10. Non-sulphate sulphur content after 60 days in loamy sand and clay soils

Non-sulphate Sulphur: In loamy sand soil, the non-sulphate sulphur content after 60 days was recorded in T_2 (2 mg S/kg soil through SNPs) (68.19 ppm), which was statistically at par with T_1 (control). In clay soil, the non-sulphate sulphur content after 60 days was found highest in T_3 (4 mg S/kg soil through SNPs) (21.87 ppm), which was statistically at par with T_5 (8 mg S/kg soil through Elemental Sulphur) in comparison to control (14.54 ppm) in Fig. 10.

After 60 days of incubation, in loamy sand soil, the highest heat soluble sulphur, organic sulphur, total sulphur and water-soluble sulphur content was found in T_4 (8 mg S/kg soil through SNPs), while sulphate sulphur in T_3 (4 mg S/kg soil through SNPs) and non-sulphate sulphur in T_1 (control). In clay soil, the highest heat soluble sulphur, organic sulphur, total sulphur, sulphate sulphur and water-soluble sulphur was observed in T_4 (8 mg S/kg soil through SNPs), while non-sulphate sulphur in T_5 (8 mg S/kg soil through ES). Similar results were also observed by Das and Datta (1973), they stated that heat soluble S, sulphate sulphur, reducible-S, organic S, carbon bonded S and elemental S increased with the increase in time under incubation. The water soluble, sulphate sulphur and heat soluble sulphur fraction was increased due to nanoparticle application in soil and it is mainly due to slow and steady release behaviour of the nano- based particles. Whereas non-sulphate sulphur was registered the highest in the sulphur applied through conventional particles which might be due to conventional particles are readily available nature thus it was fixed in the clay minerals in the soil, while sulphur applied as nano, it was slowly soluble thereby slow release of nutrients takes place. These results are in close agreement with Balanagoudar and Satyanarayana (1990). The organic sulphur was the dominant form of fraction in the soil. The variation in the organic sulphur is mainly due to mineralization and oxidation of this sulphur and also by varied based on organic carbon content and finer fraction of the soil. These findings corroborate the results of Jat and Yadav (2006).

In case of fractionation of incubated loamy sand soil, total sulphur, organic sulphur, water soluble sulphur and heat soluble sulphur was found higher with T_4 (8 mg S/kg soil through SNPs), while sulphate sulphur was found higher with T_3 (4 mg S/kg soil through SNPs) and non-sulphate sulphur was found higher in T_1 (control) (Yadav et al., 2023). In case of clay soil fractionation of incubated soil total sulphur, organic sulphur, water soluble sulphur, sulphate sulphur and heat soluble sulphur was found higher with T_4 (8 mg S/kg soil through SNPs), while non-sulphate sulphur was found higher in T_5 (8 mg S/kg soil through ES).

4. CONCLUSION

On the basis of results, it can be concluded that the application of sulphur nano-particles @ 8 mg S/kg soil significantly increased all forms of sulphur except non-sulphate sulphur in SNPs treated loamy sand and clay soils. Under incubation study, the periodical availability of sulphate sulphur was found increasing pattern up to 40 days after application in soil and thereafter in declining trend in both soils and source of sulphur. The sulphur use efficiency of sulphur nanoparticles was comparatively found superior as compared to elemental sulphur. Hence, looking to the overall results, the sulphur nanoparticles proved better in increasing the periodical availability of sulphur as compared to elemental sulphur in loamy sand and clay soils.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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