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FRACTIONATION STUDIES OF SULPHUR IN SOILS OF INDIA: A REVIEW

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ABSTRACT

Sulphur is an essential secondary plant nutrient, is required by plants (especially oilseeds) for growth and development as well as by animals. Sulphur is involved in the metabolic and enzymic processes of all living organism. Sulphur is present in the soil as its various forms viz.; organic and inorganic fractions. Bulk of the total sulphur exist as organic sulphur. The fractions of sulphur found in the soil are organic sulphur, available sulphur, water soluble sulphur and total sulphur. Sulphur is recognized as fourth major nutrient after N,P and K in terms of extent and deficiency in the country. Of the total geographic area in India, 41% of the area is deficient in sulphur. Both highly leached acidic and alkaline coarse textured soils with low organic matter suffer from S deficiencies. Sulphur fractionation plays a crucial role in understanding the sulphur dynamics in soils. This study investigates the influence of different levels of sulphur on sulphur fractionation, effect of long term fertilizer experiment on sulphur fractions, focusing on organic and inorganic sulphur forms. Hence knowledge about the fractions contribute to a better understanding of sulphur dynamics in soils and its implications for agricultural productivity and environmental sustainability.

Key words : Sulphur fractions, Oilseeds, Long term fertilizer experiment, Sulphur, NPK.

Introduction

Sulphur is a vital nutrient for plants and animals, ranking fourth in importance after nitrogen, phosphorus and potassium for agricultural crop production. Its significance in Indian agriculture is growing due to its role in boosting crop yields, not only for oilseeds, pulses, legumes and forages but also for many cereals. Sulphur is known for its role in the synthesis of proteins with the formation of amino acids such as methionine (21% S) and cysteine (27% S), chlorophyll, oil content of the seeds and nutritive quality of forages. Sulphur in the soil is majorly taken up by the plant roots in Sulphate (SO_4^{2-}) form which is usually available in the water-soluble form or adsorbed form and also it depending upon mainly characteristics of soil that may be the availability of iron oxides/hydroxide and pH. Sulphur is found in various oxidation states which readily undergo transformation by chemical and microbiological processes. Sulphur in soils occurs in many distinct forms such as water soluble, available, inorganic, organic and total sulphur. The nature and amount of

various forms of S depends on soil texture, pH, calcium carbonate, organic matter and other characteristics.

Total soil sulphur which comprises inorganic and organic binding forms ranging from 250 to 2500kg ha⁻¹ in most top soils used for farming. The readily available form of sulphur for plants is inorganic sulphur, which includes water soluble and adsorbed sulphate is generally believed to be immediate source of plants. Generally, it accounts for less than 5% of total soil S. In soil solution, SO_4 is present only in small quantity which varies continuously and its concentration at a particular time depends on the balance between plant uptake, fertilizer input, mineralization and immobilization. In order to use S fertilizer more efficiently, an understanding of the S transformations in relation to its availability in soil for crops is necessary.

Available forms of Sulphur in soil

Organic Sulphur : Sulphur in soil exists in two forms: organic and inorganic. However, the majority of sulphur is present in the organic form which is combined with

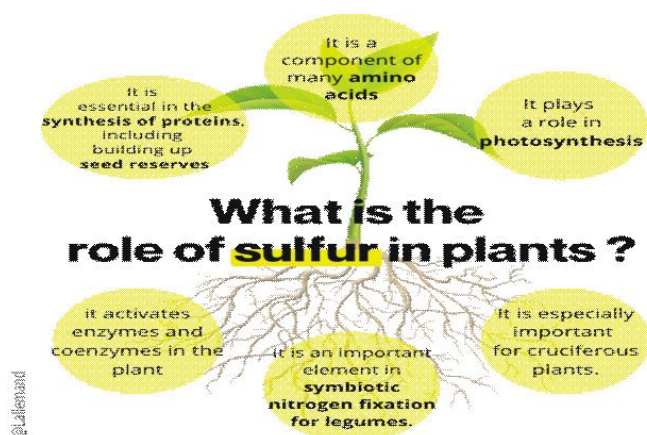


Fig. 1 : Role of sulphur in plants.

nitrogen and carbon. Notably, peat soil contains 100% of its available sulphur in organic form.

Again, Organic Sulphur is divided into 2 forms.

1. Carbon bounded 2. e.g : Sulphur of amino acid
2. Non Carbon bounded - Phenolic Sulphates, Lipids , Choline Sulphates etc.

Inorganic Sulphur : The available form of inorganic sulphur in soil is less but it is the immediate up taking form in the roots of the plants. e.g:- Sulphate form SO_4^{2-} .

Fractionations of Sulphur

A) Water-soluble sulphur- Taking off 5 gm extracted soil in distilled water of 25ml in a ratio of 1:5 ratio and then shake it for 10 minutes, centrifuge and finally filtered.

B) Available sulphur- The residue of soil that has been extracted from above water-soluble sulphur has been treated with 1% NaCl solution of 25 ml and then shaken immediately for 30 minutes and then centrifuge it and again filter the solution.

C) Inorganic sulphur- has been extracted by the addition of 1% HCl solution of 25 ml to the previously extracted soil solution and then immediately shake it for about 10 minutes and centrifuge and finally filtered the solution. Then make the soil free of chloride by addition of distilled water and leaching it.

D) Organic sulphur- Take the soil residue from

above inorganic sulphur of HCl extracted and of this again take the 2-gram soil and oven dried it and treat it with H_2O_2 up to the effervescence stops and again centrifuge and filter it.

E) Total sulphur- Total sulphur in soil was estimated by the acid digestion method. Take 5-gram fine ground soil and mix it with 3 ml 69% HNO_3 and then heated it in the steam bath. Then again add the 3 ml of 60% HClO_4 and 7 ml of H_3PO_4 and heat it in a sand bath at 190-210°C up to the visible fumes and cool it and add 2 ml of 37% HCl and then again heat it to the white fumes visible. The digested mixture was transferred quantitatively and adjusted the volume to 100 ml using 1N HCl.

Fractionation studies of Sulphur in soil

Fractionation studies on long term fertilizer experiment

Setia *et al.* (2005) stated that the application of higher doses of single superphosphate in long-term fertilized plots under a maize–wheat cropping system led to a significant increase in available sulphur content in the soil. This suggests that continuous phosphorus fertilization can also contribute to sulphur buildup due to the sulphate content in single superphosphate.

Scherer *et al.* (2012) reported that the long-term effects of applying inorganic fertilizers, farmyard manure (FYM), compost, sewage sludge and found that FYM and compost had a more positive impact on various soil sulphur fractions compared to the use of inorganic fertilizers alone.

The initial decrease in available sulphur (S) content was attributed to the higher removal of native sulphur by crops, as biomass production was reported to be higher during that period (Anonymous, 1992). In contrast, the later gradual increase in available S was due to reduced biomass production, which led to lower uptake of native S compared to the rate of S mineralization from the soil (Anonymous, 2017).

Setia *et al.* (2005) reported that application of 100% nitrogen (N) alone led to a greater accumulation of heat-soluble sulphur (S) than the 100% NPK treatment without added sulphur (-S) in a long-term fertilizer experiment.

Table 1 : Extraction of fractions of sulphur.

Sulphur Fraction	Extractant / Reagent	Key Steps
Water-soluble S	Distilled water	Shake 10 min → Centrifuge → Filter
Available S	1% NaCl	Shake 30 min → Centrifuge → Filter
Inorganic S	1% HCl	Shake 10 min → Centrifuge → Filter → Leach with water
Organic S	H_2O_2	Effervescence → Centrifuge → Filter
Total S	$\text{HNO}_3 + \text{HClO}_4 + \text{H}_3\text{PO}_4 + \text{HCl}$	Acid digestion → Fume until white → Dilute to 100 ml

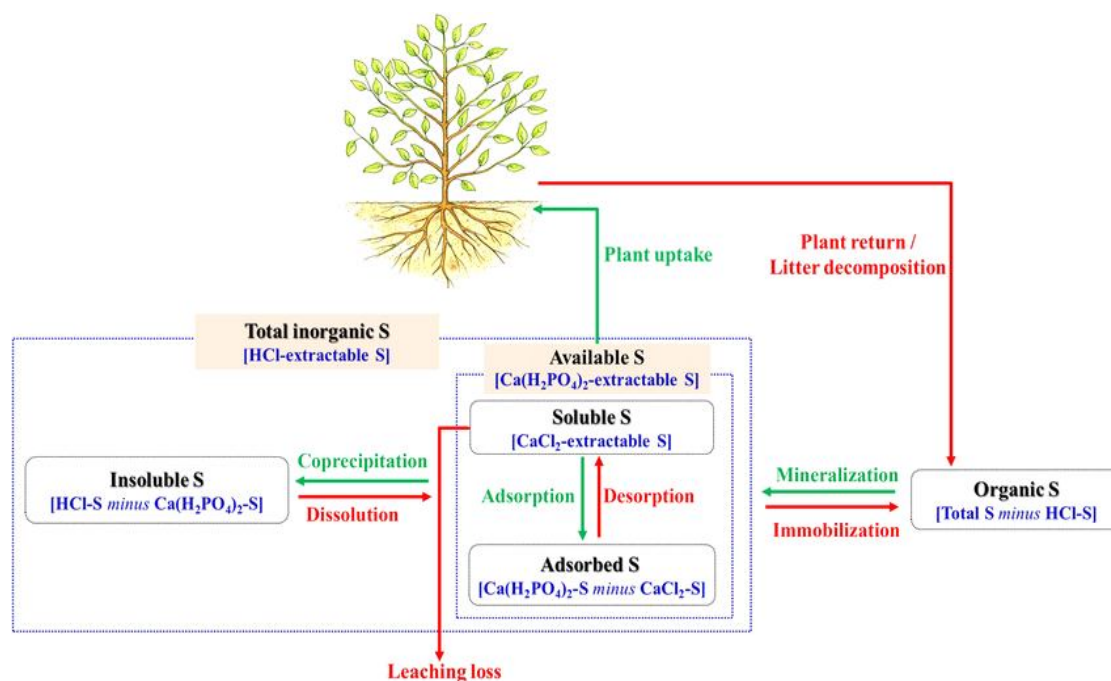


Fig. 2 : Different fractions of sulphur in soils.

McLare and Cameron (2004) observed that organic sulphur content was found to be lower in treatments that received sulphur-free or imbalanced nutrient applications. The minimal increase observed in these treatments could be attributed to the conversion of organic sulphur to available forms through the mineralization of sulphur from soil organic matter, as well as reduced addition of plant root biomass.

Lavanya *et al.* (2019) analysed that the total sulphur content in soil over the years as influenced by long term fertilizer and manure application varied significantly (Table 2). As expected, like other fractions the total sulphur content in soil showed increasing trend over years in all the treatments. However, in T_9 , T_7 and T_{11} treatments, there was decrease in T-S content initially (1991) and then increased gradually over the years and maintained slightly higher over the initial soil T-S content.

Fractionation studies for different levels of sulphur

Basumatary *et al.* (2018) conducted an experiment to know the impact of sulphur fertilization on sulfur fractions distribution and use efficiency in black gram in subtropical acidic soil of Assam, India by applying different doses of 0, 10, 20, 30, 40 Kg S ha⁻¹ and recommended dose of N, P, K as he reported that available 'S' fractions were decreased with crop growth in control and recommended fertilizer treatment and he finds better Sulphur fractions in 10, 20, 30, 40 kg S treatments and among these 20 kg S ha⁻¹ shows the better result in fractionation and better yield.

Diwakar *et al.* (2014) explained the transformation

of S fractions under field conditions with Groundnut in *Alfisols* of Karnataka by applying four levels of sulphur at 0, 15, 30, 45 Kg S ha⁻¹ and 3 sources of elemental S, gypsum and ammonium sulfate and reported that S fractions were increased in all treatments except no application of S treatment among these ammonium sulfate application shows the greater result of S fractions in comparison with both elemental S and gypsum. The lowest sulphur fractions were noticed in the treatments of elemental S.

Wani (2000), Dhananjaya and Basavaraj (2002) and Dutta *et al.* (2013) also observed that increased level of sulphur containing fertilizers significantly increased total sulphur in soil. Among sulphur sources, the total sulphur was observed numerically maximum when sulphur was applied through Sartaj gypsum followed by single super phosphate, local gypsum and elemental sulphur, respectively, though the differences were not significant.

Kumar and Trivedi (2012) have also reported an increase in mustard seed yield with the application of S levels. With an increasing supply of sulfur, the process of tissue differentiation from somatic meristematic to reproductive and developmental activity primordial flower could have grown, resulting in more flowers and siliqua, a longer siliqua and a higher seed yield.

Skwierawska *et al.* (2008) performed a field experiment with each year three sulphur fertilization rates applied: @ 40, 80 and 120 kg ha⁻¹ in the form of sulphate (S-SO₄²⁻) and pure (S-S₀) sulphur during the years from 2000 to 2002 in North-East Poland. They reported that

Table 2 : Effect of long term manuring on total sulphur content in soil over the years from 1986 to 2016 at five years interval.

Treatments	1986	1991	1996	2001	2006	2011	2016
	mgkg ⁻¹						
T ₁ :50%NPK	273.69	294.43	312.59	312.44	320.95	328.02	337.71
T ₂ :100%NPK	285.88	314.73	322.32	338.26	341.95	350.87	353.78
T ₃ :150%NPK	277.11	332.56	347.18	352.02	355.88	370.82	373.63
T ₄ :100%NPK+HW	282.10	317.09	325.15	339.86	345.92	352.39	353.67
T ₅ :100%NPK+lime	271.05	324.71	343.04	344.24	350.73	352.78	365.73
T ₆ :100%NP	286.01	314.75	322.70	337.06	344.30	345.77	356.55
T ₇ :100%N	287.17	276.90	276.45	286.04	289.17	297.95	296.12
T ₈ :100%NPK +FYM	275.30	327.03	346.64	362.18	367.64	375.53	382.82
T ₉ :100%NPK (S-free)	286.33	270.81	271.32	278.67	282.03	288.12	290.11
T ₁₀ :100%NPK+FYM+lime	289.40	330.02	334.85	363.39	371.89	375.53	384.04
T ₁₁ : Control	273.78	283.19	296.66	292.01	296.09	303.63	307.30
SEm ±	9.81	10.80	11.20	10.81	9.82	12.66	11.67
CD @ 5%	NS	31.85	33.04	31.90	28.96	37.36	34.42

as the sulphur rates increased and the duration of the experiment progressed, sulphates accumulated in soil. In the 0-40 cm soil layer, the increasing rates of sulphur tended to increase the content of N-NH₄⁺. Mostly NPK + S fertilization and especially the single S-SO₄²⁻ treatment caused an increase in N-NO₃⁻ in both soil layers compared with the NPK fertilized. The dose of 120 kg/ha S-SO₄²⁻ caused a significant increase in the concentration of available phosphorus in soil in the 0-40 and 40-80 cm layer.

Nathan *et al.* (2018) observed that the effect of nitrogen 0, 40 and 60 kg N ha⁻¹ and sulphur (0, 25 and 45 kg S ha⁻¹) levels on yield and economics of safflower. They reported the highest gross returns (Rs 55408 ha⁻¹) and net returns (Rs 36777 ha⁻¹) with applied 60 kg N ha⁻¹ and 45 kg S ha⁻¹ and the B:C ratio (2.97).

Diwakar *et al.* (2014) conducted field experiment with four levels of sulphur (S; 0, 15, 30 and 45 kg ha⁻¹) and three sources (elemental S, gypsum and ammonium sulfate) on groundnut crop. They reported significant increase in contents of all the soil S fractions compared to control and decreased with crop growth and maximum values obtained in RDF with applied S @ 45 kg ha⁻¹ treatment. Among sources of S, ammonium sulfate recorded highest values of S fractions compared to gypsum and elemental S treatments. Water-soluble and available soil S fractions significantly decreased between flowering and harvest as contributed to the pool of plant-available S, residual S fraction significantly increased due to levels of applied S even after harvest and obtained significant positive correlations between the S fractions.

Singh *et al.* (2023) conducted an experiment in mustard and revealed that application of 60 kg S ha⁻¹

produced higher number of siliquae (340 plant⁻¹), length of siliquae (5.03 cm), number of seeds (16.93 siliqua⁻¹), stem girth (6.25 cm), test weight (5.38 g), seed yield (2.71 t ha⁻¹), stover yield (5.11 t ha⁻¹) and biological yield (7.82 t ha⁻¹), which were significantly higher over control.

Verma *et al.* (2018) performed an experiment and reviewed that progressive increase in doses of sulphur significantly increased number of days taken to 50% flowering. Significantly highest days taken to 50% flowering (61.18) were recorded with the application of 50 kg S ha⁻¹ followed by 40 kg S ha⁻¹ (59.99), 30 kg S ha⁻¹ (59.05), 20 kg S ha⁻¹ (58.50) and 10 kg S ha⁻¹ (57.74). The lowest number of days taken to 50% flowering (57.48) of Indian mustard were recorded in control plot.

Singh *et al.* (2023) observed that application of 60 kg S ha⁻¹ produced maximum plant height (203.3 cm at 90 DAS), leaf area index (3.78 at 90 DAS), No. of leaves/plant (45.75 at 90 DAS), primary branches at harvest/plant (6.13), secondary branches at harvest/plant (17.55) in mustard, which were significantly higher over control.

Yadav *et al.* (2020) analysed that increasing the levels of sulphur up to 60 kg/ha progressively increased total uptake of N (117.31 kg/ha), P (15.50 kg ha⁻¹), K (107.55 kg ha⁻¹) and S (8.64 kg ha⁻¹), which were significantly higher over control by 120, 109, 99 and 95 per cent, respectively in groundnut.

Nagar *et al.* (2017) asserted that application of 60 kg S/ha significantly increased protein content (39.27%), protein yield (524.40 kg ha⁻¹), oil content (22.00%) and oil yield (292.40 kg ha⁻¹) in soybean, which were significantly higher over control.

Yadav *et al.* (2019) stated that the highest net return

Table 3 : Comparison of Conventional Sulphur (S-level) and Nano Sulphur (NS) on Groundnut Yield.

Sulphur Level	Type	Pod Yield	Haulm Yield	Trend/Observation
S ₀ (0 kg/ha)	Field S-level	22.3 q/ha	31.1 q/ha	Baseline yield
S ₂ O	Field S-level	24.2 q/ha	33.3 q/ha	Moderate increase
S ₄ O	Field S-level	26.1 q/ha	36.0 q/ha	Max field yield
10 kg NS	Nano S	10.1 g/plant	18.4 g/plant	Small increase from baseline
20 kg NS	Nano S	11.0 g/plant	21.1 g/plant	Moderate increase
30 kg NS	Nano S	12.2 g/plant	23.9 g/plant	High increase
40 kg NS	Nano S	13.0 g/plant	25.7 g/plant	Maximum yield in pot study

Thirunavukkarasu *et al.* (2018) and Patel *et al.* (2023).

(Rs. 55,374 ha⁻¹) and benefit cost ratio (1.60) obtained at sulphur level 60 kg ha⁻¹ in groundnut. However, further increase in Sulphur level up to 75 kg ha⁻¹ non significantly increased economic returns but both these treatments gave significantly higher returns over 45, 30 and 15 kg S ha⁻¹.

Scherer (2001) stated that high seed yield and quality of oilseeds are possible when they have access to the optimum amount of S.

Sreemannarayan and Raju (1995) observed that the application of S @ 20, 40 and 60 kg ha⁻¹ led to an increase in uptake of 33.5, 68.7 and 95.0% S over control, respectively.

Dileep *et al.* (2021) conducted a field experiment on groundnut and reported that the application of 40 kg S/ha significantly enhanced plant growth parameters compared to 30 kg and 20 kg S/ha. At harvest, the 40 kg S/ha treatment recorded maximum plant height (59.31 cm), dry matter accumulation (56.73 g/plant), number of nodules (47.53/plant), crop growth rate (13.82 g/m²/day), and relative growth rate (0.008 g/g/day).

Fractionation studies on Sulphur with different doses of N, P, K

Dutta *et al.* (2013) reported that increasing fertilizer application rates from 50% to 100% of the recommended NPK levels led to a significant increase in heat-soluble sulphur content. This suggests that adequate and balanced nutrient supply not only supports better crop growth, but also enhances sulphur availability in the soil, possibly due to improved microbial activity and mineralization processes.

Sharma *et al.* (2014) reported that application of chemical fertilizers, either alone or in combination with organic amendments, significantly increased the organic sulphur content of the soil compared to the control. This enhancement with higher levels of NPK application may be attributed to the sulphur contributed by phosphorus fertilizers and various organic sources. These findings

are in agreement with those reported by Dutta *et al.* (2013).

Setia and Sharma (2005) reported that the application of phosphorus through superphosphate significantly enhanced the water-soluble sulphur content in both 50% and 100% NPK treatments. This increase can be attributed to the sulphur content (12%) present in superphosphate, alongside its phosphorus contribution.

Warjri *et al.* (2017) and Sharma *et al.* (2014) observed that the application of 100% NPK significantly increased the water-soluble sulphur content compared to treatments with imbalanced or no fertilizer inputs. This increase in available sulphur under balanced nutrient regimes can be attributed to improved nutrient interactions and enhanced microbial activity, which facilitate the mineralization of organic sulphur into plant-available forms. These findings underscore the importance of integrated nutrient management in sustaining soil fertility and optimizing crop nutrient uptake.

Conclusion

Fractionation studies of sulphur in soils of India have significantly advanced our understanding of sulphur dynamics in diverse soil types and under various management practices. These studies have highlighted the importance of sulphur fractionation in determining sulphur availability, soil fertility and crop productivity. It is concluded that by increasing the level of sulphur there is a significant increase in growth and yield parameters of the plants and even in long term experiments continued application of sulphur leads to an increase in all fractions of sulphur in soils. It also shows that different levels of N, P, K also affect the different fractions of sulphur in soil.

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