



# Effect of Continuous Application of Fertilizer and Manure on Distribution of Sulphur Fractions in a Vertisol

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## **Authors' contributions**

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

## **Article Information**

DOI: 10.9734/AJSSPN/2024/v10i2273

## **Open Peer Review History:**

This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: <https://www.sdiarticle5.com/review-history/114862>

**Original Research Article**

**Received: 05/02/2024**

**Accepted: 09/04/2024**

**Published: 15/04/2024**

## **ABSTRACT**

The fertilization experiment was started at Experimental field of Eklavya University, Damoh, Madhya Pradesh-India at a fixed location in the experimental field study was carried out. Under this study a randomized block design with eight treatments and four replications high used Urea, single

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super phosphate (SSP), muriate of potash, and DAP instead of SSP were employed as fertilizers in the sulphate-free treatment.

**Keywords:** Single super phosphate; muriate of potash and diammonium phosphate.

## 1. INTRODUCTION

The plant absorbs sulphur in its sulphate form from the soil solution ( $\text{SO}_4^{2-}$ ). Sulphur is found in plants as part of the amino acids methionine, cysteine and cystine, which are the building blocks of proteins. Sulphur is also needed to boost the oil content of oil seed crops. Sulphur is removed by crops in India in the amount of 1.26 million tonnes (Mt), whereas it is replenished by fertilizers in the amount of 0.76 Mt. [1]. Sulphur is a mobile nutritional element in the soil system, moving down to the lower soil layers as a result of numerous pedological as well as frequent irrigations and rains, which increased the extent of S movement and alteration in the soil system [2]. The sulphur could come from weathering of soil minerals, the environment, or already bonded sulphur. Microbial activity, which includes processes of mineralization, immobilization, oxidation, and reduction, was thought to be the primary cause of sulphur changes in soil [3]. The main source of sulphur nutrition for plants is soil, and its availability is determined by sulphur fractions. Sulphur in soil is divided into five types: accessible sulphur (S), water soluble sulphur (S), heat soluble sulphur (S), organic sulphur (S) and total sulphur (S). Organic-S influences the level of plant accessible sulphur the most among these sulphur pools. By providing physical protection within water-stable soil aggregates, soil structure helps shield soil organic matter from microbial attack. The long-term fertilizer experiment focused on crop yield performance when manure and fertilizer applications were balanced and imbalanced in a cropping sequence's soil fertility management [4].

## 2. MATERIALS AND METHODS

The experiment was conducted layout of Randomized Block Design experiment with ten treatments reproduced four times. The plots are 17 x 10.8, with 1 m between plots and 2 m between replications. Along the side of the primary experiment, an additional strip is kept as a no-crop control (Fallow strip). The soil test value was used to calculate the appropriate

fertilizer dose (100 per cent NPK) for each crop. The present investigation was undertaken with eight treatments marked as (\*) out of 10 treatment.

### List 1. List of treatments used for the study

Treatments	Dose
*T <sub>1</sub>	50%NPK
*T <sub>2</sub>	100%NPK
*T <sub>3</sub>	150%NPK
T <sub>4</sub>	100% NPK+ Hand weeding
T <sub>5</sub>	100% NPK + Zn (as $\text{ZnSO}_4$ ) #
*T <sub>6</sub>	100 % NP
*T <sub>7</sub>	100% N
*T <sub>8</sub>	100%NPK + FYM@ 5 ton ha <sup>-1</sup>
*T <sub>9</sub>	100%NPK-S (Sulphur free)
*T <sub>10</sub>	Control

# The use of Zn as  $\text{ZnSO}_4$  was stopped Since 1987 due to high buildup of Zn in this treatment

### 2.1 Soil Reaction (pH)

With the use of a glass electrode pH metre, the pH of a suspension of soil in water with a soil water ratio of (1:2.5) was determined (Piper, 1950).

### 2.2 Electrical Conductivity of Soil

The suspension of soil which was already used in pH observation was allowed to settle and the conductivity of the supernatant liquid was measured with a conductivity metre (Piper, 1950).

### 2.3 Organic Carbon of Soil

The rapid titration method of Walkley and Black (1934) was used to measure organic carbon (Piper, 1950).

### 2.4 Available Nitrogen of Soil

The alkaline permanganate method was used to measured available nitrogen in soil samples (Subbiah and Asija, 1956).

### 2.5 Available Phosphorus of Soil

The amount of available phosphorus in the soil sample was evaluated using the extraction method outlined.

## 2.6 Available Potassium of Soil

Muhrel *et al.*, used neutral normal ammonium acetate to extract the available potassium, which was then measured using a flame photometer (1965).

## 2.7 Available Sulphur of Soil

The available sulphur content of the soil was assessed using a 0.15 per cent chloride solution and sulphur was quantified using the turbidimetric method. At 420 nm, the spectrophotometer used to observe the solution's transmittance or absorbance.

## 2.8 Organic Sulphur of Soil

The amount of organic sulphur in the soil was determined by mixing 1g of air dried (20 mesh) soil with 1g of NaHCO<sub>3</sub> in a porcelain crucible. The mixture was heated with 3 hours at 500 °C temperature in an electric furnace (muffle). Then transfer the contents of the crucible to a 100 ml flask and add 25 ml of the extraction solution after it has cooled (Dissolve 46 g NaH<sub>2</sub>PO<sub>4</sub>.H<sub>2</sub>O 1 litre of 2N acetic acid). The contents of the flask was shaken for half an hour after the reaction had died down. The solution was then filtered using a Whatman No. 1 filter paper that was dry. Sulphate was assessed using a turbidimetric technique using an aliquot of the filtrate.

## 2.9 Water Soluble Sulphur of Soil

De ionized water was used to extract the water soluble sulphur from the soil and sulphur was measured using a Turbid metric method. On a spectrophotometer with a wavelength of 420 nm, the solution transmittance or absorbance was measured.

## 2.10 Heat Soluble Sulphur

Heat soluble sulphur was tested by pouring 20 ml of distilled water into a silica crucible containing 5 g of air dried soil. Then it was placed in a boiling water bath and evaporated until it was completely dry. After cooling, it was baked in a

hot air oven at 102 °C for 60 minutes. It was then transferred to a 50 ml centrifuge tube and extracted with 33 ml of 1 per cent NaCl after chilling. With the use of a pipette, a 25 ml aliquot was transferred to a silica basin and evaporated to dryness with 2 ml of 3 per cent H<sub>2</sub>O<sub>2</sub>. To assure the elimination of superfluous H<sub>2</sub>O<sub>2</sub>, the crucible was roasted in a hot air oven at 102 °C for 60 minutes. The residue was dissolved in 25 ml distilled water, transferred to a centrifuge tube and centrifuged to remove any suspended debris. Sulphur was determined turbid metrically by collecting an appropriate aliquot (Williams and Stein bergs, 1959).

## 2.11 Total Sulphur of Soil

In a 250 ml beaker, one gram of air dried soil (ground to pass a 0.5 mm filter) was taken and added 10 ml of digesting solution (100g AR grade KNO<sub>3</sub>+ 350 ml concentrated HNO<sub>3</sub> and dilute to 1 litre). On a steam bath, the contents were evaporated to a dry state. The beaker was then placed in an electric furnace and heated to 500 °C for three hours (Choudhary and Cornfield, 1996). After cooling, 5 ml of 25% HNO<sub>3</sub> was added and the contents were digested for another hour on a steam bath until dry. The solution was filtered using a Whatman filter paper No. 42 after the soluble salts were extracted with distilled water. The filtrate was then diluted to a volume that could be measured. Sulphur turbid metric analysis was performed on an aliquot (Chaudhary and Cornfield, 1966).

## 3. RESULTS AND DISCUSSION

### 3.1 Organic Carbon

The reaction of long-term manuring and fertilizer use underintensive cropping is depicted on organic carbon, which is a key barometer of soil health assessment. The control with no fertilizer had the lowest organic carbon content (4.70 g kg<sup>-1</sup>). However, at 50 per cent NPK (5.75 g kg<sup>-1</sup>) and 100 per cent NPK (7.32 g kg<sup>-1</sup>) and 150 per cent NPK (7.98 g kg<sup>-1</sup>) doses, the organic carbon levels improved dramatically with proportionate fertilizer addition. This findings appeared to be attributable to improved crop root development as a result of higher residue decomposition in intensive farming with constant fertilizer application. These findings corroborate those of Patel *et al.*, (2018). As a result, FYM addition had

a significant impact on soil organic carbon buildup (Mundheet *et al.*, 2018), indicating that combining chemical fertilizer with organic manure can help maintain soil health and crop yield [5].

### 3.2 Available N in Soil

The available N content as a function of treatment, indicating that higher N content was acquired from surface soil, possibly due to the presence of crop residues after harvest. When an imbalance fertilizer was administered, on the other hand, the reduced N content was noted. Similarly, Dwivedi *et al.* [6] observed that the available N status could only be maintained by combining fertilizer and manure to improve N use efficiency over unbalanced fertilizer consumption. The addition of 50 per cent NPK and 100 per cent NPK fertilizer doses, on the other hand, improved N content, demonstrating that fertilizer application had an impact on N pool enrichment.

Gupta *et al.* [7]. also found that higher N status was attained on the surface, while it gradually fell with depth, however the pace of depletion was more obvious from the surface to the subsurface. This could be due to increasing root biomass in the rhizosphere zone soil layer, which decreased when soil depth was increased.

### 3.3 Available P

The results on available P status demonstrated that continual fertilization and manuring significantly enhanced available P in practically all treatments receiving P annually when compared to fertilizer without P application. The huge difference in P content recorded from various fertility treatments getting 50 per cent NPK, 100 per cent NPK, and 150 per cent NPK dosages of nutrition, respectively, indicates increased P buildup. Furthermore, the highest content was found when integrated fertilizer application was done with FYM (37.86 kg ha<sup>-1</sup>) followed by 150 per cent NPK (36.08 kg ha<sup>-1</sup>) treatments, indicating that FYM has a greater beneficial effect on P mineralization in soil, according to Dwivedi *et al.* [8] Dwivedi and Dwivedi [5].

The doses also showed that above the surface, P accumulation was higher than at lower depths. This was attributed to the soil fixation of applied P and the restriction of its transport as a result.

Dubey *et al.* [9] are a group of researchers who have worked on same.

### 3.4 Available K

K is a vital plant nutrient that regulates a variety of physiological and metabolic activities. According to estimates, black soils are normally high in K, but intense agriculture and insufficient replacement may result in a constant depletion of K pools. The effects of continuous cropping and fertilizer application on available K status were depicted in (Table 1). The results also demonstrated that prolonged application of 100 per cent NPK and 150 per cent NPK doses resulted in depletion of available K, resulting in a negative balance and, as a result, a significant loss of K fertility Bhattacharya *et al.* [10]. There was also a drop in available K status to roughly 300 kg K ha<sup>-1</sup> in the soil, which was initially well supplied (370 kg ha<sup>-1</sup>). With a progressive deeper profile, a diminishing trend in available K status was seen, and maximum content was attained at a system that gradually fell to lower depths, as previously reported by Yadhuvanshi and Swarup, [11] Padharia *et al.*, (2021).

### 3.5 Available Sulphur

The greatest amount was detected in the balanced optimal dose administered along with FYM, which could be attributable to increased nutrient supplementation over imbalanced fertilizer addition Gokila *et al.*, (2017). With depth, the largest concentration of accessible S form was contained on the surface and rapidly fell down profile. This could be owing to the decreased mobility of available S, preventing additional travel to deeper depths later Misal *et al.*, (2017). There was a buildup of available S at surface soil in all treatments where P was applied and the degree of the buildup corresponded to the level of applied fertilizer. As a result, the highest levels of accumulation were seen in 150 per cent NPK and 100 per cent NPK+FYM. The highest value was found to be connected with the addition of FYM, which creates ideal conditions for improved nutrient use efficiency (Khandagle *et al.*, 2019). However, when only N was treated, the accessible S content at the surface soil was low, whereas available S content increased with increasing amounts of fertilizer application, reaching a peak value (35.84 kg ha<sup>-1</sup>) when 100 per cent NPK

+FYM was applied. Suman *et al.*, [12] obtained similar results. It was also discovered that the S content of the post-harvest soil sample differed somewhat from the pre-sowing soil status. Venugopal *et al.*, (2017) found a similar pattern in sulphur availability.

### 3.6 Organic Sulphur

In both the 100 per cent NPK+FYM and 150 per cent NPK treatments, organic sulphur concentration in subsurface soil was found to be comparable. The total to organic sulphur ratio in these treatments was around 30%, indicating that roughly 28-30% of sulphur was maintained in the organic form in the surface and subsurface soil layers. However, with different treatments at different depths, it ranged from 20 to 33 per cent. In cultivated temperate soil, organic sulphur is the primary binding form, accounting for up to 95 per cent of total sulphur. Similar findings were reported by Scherer *et al.*, [13] who determined that higher organic sulphur content was confined to the surface soil and decreased as the depth of the profile increased. As the subsequent addition of S with higher transformation resulted in lower content in control (39.78 kg ha<sup>-1</sup>) and 100 per cent NPK-S (42.65 kg ha<sup>-1</sup>) due to the absence of addition of S, increasing the content of organic-S form in the surface soil. Suman *et al.*, [12] found that soybean- wheat cropping with continuous use of 100% NPK without sulphur resulted in decrease in organic sulphur content above control. The organic-S concentration decreased with depth, with the maximum concentration at the surface. This statement conformity Rajput *et al.*, [14].

### 3.7 Water Soluble Sulphur

The content of water soluble sulphur form increased with application of fertilizer S over imbalance and/or without additions, *i.e.* in control

(11.04 kg ha<sup>-1</sup>) and 100 per cent NPK-S (11.10 kg ha<sup>-1</sup>) soil complexes, which could be due to the transformation of applied fertilizer S to available S pool in soil complex. On the other hand, using organic manure in combination with the recommended fertilizer dose resulted in a significant increase in available form. This could be due to the release of organic acids during the decomposition of organic matter, resulting in the conversion of soluble S compounds from both the applied and inherent S pools. As a result, it immediately represents the activity and concentration of accessible S status, which is beneficial in increasing S availability in soil (Birla *et al.*, 2015) It was also noted that with successively higher additions of S from 100 per cent NPK to 150 per cent NPK dose, a significantly higher content of available S was observed, which could be due to differential conversion of applied S in available S form as a result of varying transformation reactions operating in soil exchange complex Sen *et al.*, [15].

### 3.8 Heat Soluble Sulphur

Higher fertilizer application increases S content, which could be related to the transformation and translocation of the fertilizer S into heat soluble sulphur in the soil system. The largest level of heat soluble sulphur was discovered in 100 per cent NPK+FYM, whereas the lowest content was reported with unbalanced fertilizer addition or in control. Dalavi *et al.*, [16] came to the same conclusion. The heat soluble sulphur concentration was highest at the surface and gradually declined down profile due to microbial degradation of organic matter and increased organic carbon content, resulting in confined on the surface soil [17-19]. It's clear since this type of sulphur is linked to organic -S, which declined with depth, according to Suman *et al.*, [12]

**Table 1. Effect in treatments on distribution of available soil Organic Carbon, Nitrogen, Available P and Available K**

Treatments	Soil OC (g kg <sup>-1</sup> )		Available N (kg ha <sup>-1</sup> )		Available P (kg ha <sup>-1</sup> )		Available K (kg ha <sup>-1</sup> )	
	0-15 cm	15-30 cm	0-15 cm	0-15 cm	0-15 cm	15-30 cm	15-30 cm	15-30 cm
50% NPK	5.75	5.20	221	21.87	260	245	16.89	196
100% NPK	7.32	6.53	265	32.15	304	280	22.58	232
150% NPK	7.98	7.21	298	36.08	328	294	27.54	265

100% NP	6.58	5.75	235	28.04	251	218	19.26	210
100% N	5.05	4.39	210	9.12	24	216	8.72	189
100%NPK + FYM	8.65	7.86	316	37.86	337	301	28.13	284
100%NPK- S	7.21	6.10	262	31.68	301	278	22.14	230
Control	4.70	4.07	180	8.16	246	210	7.96	167
SEm±	0.31	0.27	13.26	2.26	13.58	9.58	1.38	10.35
CD (P=0.05)	0.91	0.79	39.01	6.64	39.94	28.19	4.07	30.44

**Table 2. Effect of treatments on distribution of available sulphur, Organic Sulphur, Water soluble Sulphur and heat soluble sulphur**

Treatments	Available S (kg ha <sup>-1</sup> )		Organic Sulphur (kg ha <sup>-1</sup> )		Water soluble Sulphur (kg ha <sup>-1</sup> )		Heat soluble Sulphur (kg ha <sup>-1</sup> )		Total S (kg ha <sup>-1</sup> )	
	0-15 cm	0-15 cm	0-15 cm	15-30 cm	15-30 cm	15-30 cm	0-15 cm	0-15 cm	15-30 cm	15-30 cm
50% NPK	22.58	63.27	18.10	12.91	50.10	21.44	34.00	167	144	31.00
100% NPK	31.23	71.01	27.04	20.15	57.45	28.20	36.50	183	168	33.50
150% NPK	34.01	73.54	32.85	25.14	65.40	30.56	40.80	201	186	36.25
100% NP	30.27	65.68	25.29	19.50	52.93	27.11	36.18	174	156	33.00
100% N	11.10	40.02	11.50	8.60	33.68	11.05	24.75	89	79	22.40
100% NPK + FYM	35.84	74.21	31.52	26.20	65.51	32.20	43.20	203	190	42.50
100%NPK- S	11.89	42.65	11.10	8.75	34.32	10.90	25.10	95	82	22.60
Control	11.55	39.78	11.04	8.10	32.56	10.60	24.14	85	78	22.05
SEm±	1.21	1.59	0.84	0.59	1.10	1.10	0.84	10.04	5.03	0.89
CD (P=0.05)	3.55	4.68	2.36	1.75	3.24	3.24	2.35	29.54	14.79	2.61

### 3.9 Total Sulphur

The impact of continuous cropping and fertilizer uses on Total- S status was observed lower content in control (85 kg ha<sup>-1</sup>) followed by 100 per cent NPK-S (95 kg ha<sup>-1</sup>) which could be due to the fact that without fertilizer application and/or S inclusion in the fertilizer schedule, it has obviously resulted in its lower content. On the other hand, successive increases of S from 50, 100, and 150 per cent NPK resulted in a corresponding increase in Total-S in soil. It could be attributed to a proportionately larger transformation of S into Total-S from inherent and applied sources. Sulphur availability increases when fertilizer doses rise from 50 per cent NPK to 150 per cent NPK. However, the highest level was detected when 100 per cent NPK was combined with FYM, demonstrating that FYM had a synergistic effect on speeding the rate of transformation into Total-S in soil for later use by crop plants [12]. The largest content of Total-S form limited to the surface and subsequently fell to lower depths, indicating a

falling tendency in Total-S form with depth [20,21]. This could be attributed to Total-slower S mobility, limiting movement to lower depths Das *et al.*, (2006).

### 4. CONCLUSION

The various form of sulphur fractions tended to increase with application of fertilizer containing sulphur nutrients in comparison to without sulphur application (imbalance fertilizer and DAP application). Sulphur fractions were found to be predominantly higher amount in 100% NPK+FYM with order of Total S > Organic S > Heat soluble S > Available S > Water soluble S in a Vertisol.

### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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