__Original Research Article__

Different Fractions of Sulphur, their correlations with soil properties and Yield of Soybean and Safflower Influenced by long Term fertilizers Experiment under Typic Haplusterts

ABSTRACT

Sulphur is an important element in Indian soils, and it has been found as a major limitation for increasing the oil seed productivity. The results emerged out from experimental field showed that 100% NPK + FYM @ 5 t ha^{-1} recorded significantly maximum fractions of sulphur, including organic sulphur (37.13 mg kg^{-1}), non-sulphate sulphur (118.28 mg kg^{-1}), water soluble sulphur (12.28 mg kg^{-1}), heat soluble sulphur (1.45 mg kg^{-1}) and total sulphur (153.65 mg kg^{-1}) followed by only application of organic manuring as FYM @ 10 t ha^{-1}. Different sulphur fractions exhibited negative correlation with pH and EC while, found positive significant with organic carbon, available N, P, K and S. Similarly, soybean and safflower both crops produced higher yields of grain (18.30 and 8.66 q ha^{-1}) and straw (28.70 and 27.73 q ha^{-1}), respectively. However, higher N (131.80 kg ha^{-1}), P (26.27 kg ha^{-1}), K (56.44 kg ha^{-1}), S (15.43 kg ha^{-1}) and Zn (253.90 g ha^{-1}) uptake were observed by soybean whereas, N (54.43 kg ha^{-1}), P(16.47 kg ha^{-1}), K(69.90 kg ha^{-1}), S (11.29 kg ha^{-1}) and Zn (162.19 g ha^{-1}) were observed by safflower in application of 100 % NPK + FYM @ 5 t ha^{-1}.

(Keywords: Yield, Fractions of sulphur, organic, nutrient uptake)

INTRODUCTION

Sulphur is one of the essential plant nutrients classified as a secondary nutrient; it is essential for all plants and is indispensable for growth and metabolism oil seed crop. The concentration was found to be the highest in oilseed (1.1 -1.7 %), intermediate in pulses (0.24 - 0.32%) and the lowest (0.12 – 0.20 %) in cereals. These crops contribute more than 70 % of the total oil seed production in India and thus form the backbone of food security (Lathwal et al., 2010). Sulphur induces chlorophyll concentration in leaf, grain and protein content in soybean (Chatterjee *et al.*, 1992). Sulphur occurs in a wide variety of organic and inorganic combinations. The transfer of sulphur between the inorganic and organic pool is entirely caused by the activity of the soil biota, particularly the soil microbial biomass, which has the greatest potential for both mineralization's and also for subsequent transformation of the oxidation state of sulphur (Tiwari and Gupta 2006). Sulphur in Indian agriculture is now gaining more importance because of the recognition of its role in increasing crop production, not only of oil seeds, pulses, legumes and forages but also of many kinds of cereal, plantation crops and forest vegetations (Singh et al., 2000). The content and forms of sulphur in soils is an integrated index that reveals the sulphur supplying capacity of the soil. Urkurkar et al., (2010) observed the influence of long-term use of inorganic and organic manures on soil fertility and sustainable productivity of rice -wheat cropping system. The present study was undertaken to evaluate different fractions of sulphur and their association with soil properties and effect on grain and straw yield of soybean – safflower cropping sequence..

MATERIAL AND METHODS

Experimental site

The field experiment was conducted for two consecutive years during kharif and Rabi season of 2017-2018 and 2018-2019 at the research farm of the Department of Soil Science and Agricultural Chemistry, College of Agriculture, Vasantrao Naik Marathwada Krishi Vidyapeeth, Parbhani. The sowing of soybean (cv. MAUS- 162) in Kharif season was carried out on 18 June during 2017-18 and 20 June during 2018-19 using 65 kg ha^{-1} seed rate and sowing of safflower (cv. PBNS- 12) in Rabi season was carried out on 15 November during 2017-18 and 18 June November 2018-19 using 7 kg ha^{-1} seed rate. The both crops were fertilized with nitrogen through urea, phosphorous through single super phosphate and potassium through muriate of potash as per treatments. The full dose of N, P and K was applied to soybean crop at the time of sowing and half dose N and full dose of P and K and remaining half dose of N after one month of sowing was applied to safflower crop. The recommended agronomic practices were followed through the experiment for raising the crop (Singh and Singh 2003)

Nutrient uptake and yield

The soybean and safflower were harvested from net area of 13 x 10 M^2 of each plot at 26 and 18 October during 2017-18 and 2018-19 and 15 and 18 April during 2017-2018 and 2018 - 2019, respectively. The harvested produce was sun dried for 4-5 days and weighed for recording biological yield. The grains were separated from the produce with mechanical thresher, cleaned, sun dried to approximately 12.0% moisture and weighed for recording grain yield. The straw yield was recorded by subtracting the grain yield from the biological yield. Total N, P and K concentrations in grain and straw samples of soybean and safflower were determined after finely grinding as described by Page (1982) and N, P and K uptake by grain and straw was computed on oven dry weight basis.

Physiography of experimental site and climate

Geographically, Parbhani district is situated in the Godavari drainage basin in the central part of the India between 76^0 46' East longitude and $19^0$16' North latitude having elevation of 410 m above the mean sea level in Marathwada Division of Maharashtra state. The region has semi-arid climate. It is under assured monsoon rainfall agro climatic zone with an average annual precipitation of 835 mm. The major portion of precipitation (75 per cent) being received through South-West monsoon from June to March. During 2017-2018 the crop growth period of annual rainfall was 999.3 mm. The mean maximum temperature varies from 13.7^0 C in winter to 40.6^0 C in summer and the mean minimum temperature varies from 6.1^0 C to 27.0^0C (Fig. 1.)

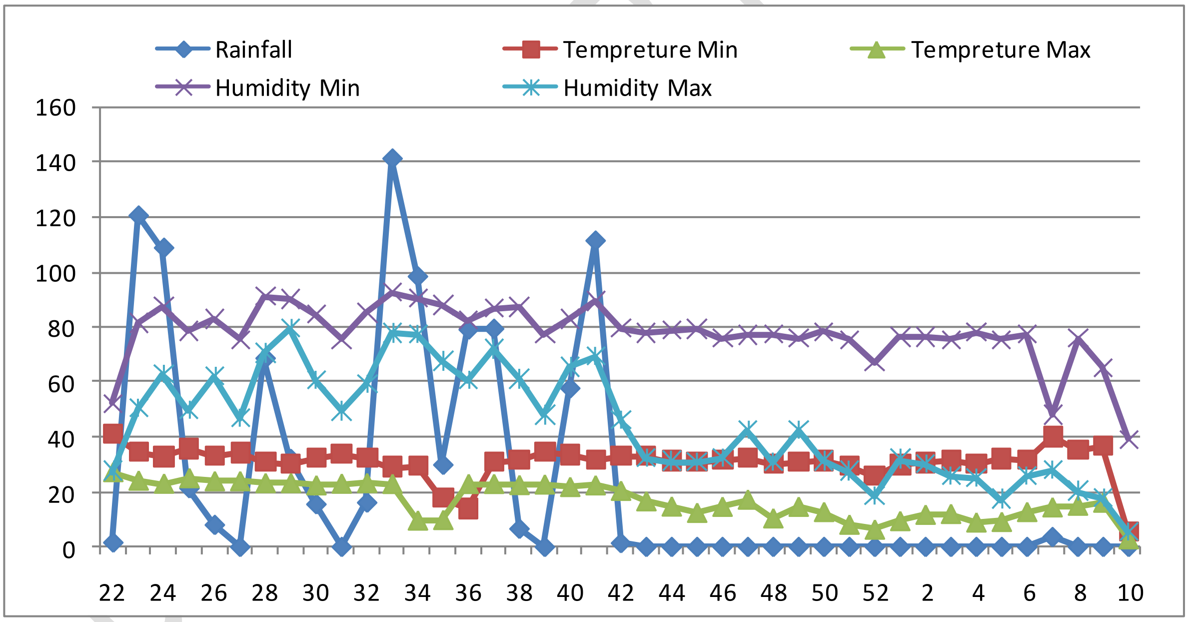

Fig. 1. Weather data recorded during experimental period 2017-18 under long term fertilizer experiment

soil sampling and analysis

The surface (0-15 cm) soil samples were collected from individual plots after harvesting of safflower crop during both the years. The collected soil samples were thoroughly mixed and brought to the laboratory, air-dried, ground with wooden mortar and pestle and sieved through a 2 mm sieve, for analyzing sulphur fractions (total sulphur, water soluble sulphur, heat soluble sulphur, organic sulphur, non-sulphate sulphur.)

The approach outlined by Evans and Roost (1945) was used to estimate organic sulphur. 5 gm of finely powdered soil was added to the beaker and exposed to hydrogen peroxide in order to estimate the amount of organic S. The samples were filtered through filter paper, and then the sulphur was precipitated with $BaCl_2$ and shaken for one minute. 1 ml of 0.25 % gum acacia solution was added. A spectrophotometer was used to measure the turbidity at a wavelength of 440 nm. By subtracting organic sulphur from total sulphur as reported by Chesnin and Yein (1951), non-sulphate sulphur was obtained. The approach outlined by Jackson (1973) was used to estimate the amount of water-soluble sulphate sulphur. The water-soluble sulphate sulphur was extracted by shaking the soil with water (1:5) for 30 min. The suspension was filtered to remove suspended matter. Then added 1 gm $BaCl_2$ and 1ml of 0.25% gum acacia solution and the volume make up to 25ml. Measure the turbidity on a spectrophotometer at 440 nm wavelengths. Heat Soluble Sulphur is estimated by Cottenie, (1973) method. Total sulphur was determined by turbidity method (Chopman and Pratt, 1961).

Organic Sulphur (Turbidity method, Evans and Roost, 1945).

5 gm soil → 250 ml conical flask

↓

Oxidize with ml hydrogen peroxide (H_2O_2)

↓

1gm of $BaCl_2$

↓

1 ml of 0.25 % gum acacia powder.

↓ → Filter with filter paper Whatman No. 42

Precipited sulphur with Barium Chloride ($BaCl_2$)

↓

Shake for one minute.

↓

Measure the turbidity in Spectrophotometer at 440 nm wavelength.

Water Soluble Sulphur (Turbidity method, Jackson, 1973)

1 gm soil 250 ml conical flask

↓

Add 5 ml of distilled water and Filter the sample

↓

Add 1 ml of gum acacia + 1 gm of $BaCl_2$ powder.

↓

Take the turbidity reading after 5-20 minutes on the spectrophotometer at 440 nm wavelength.

Heat Soluble Sulphur (Turbidity method, Cottenie, 1973)

5 gm of soil into a silica basin

↓

Add 20 ml of distilled water.

↓

Place the basin on boiling water bath and evaporate to dryness.

↓

Heat it in hot oven at 102^0 C for 60 min.

↓

After cooling, transfer soil to a 50 ml centrifuge tube and extract it with 30 ml of 1 % Nacl.

↓

Pipette out 25 ml of aliquot into Silica Basin and evaporate to dryness with 25 ml of 3% H_2O_2

↓

Heat it in hot air oven at 102^0 C for 60 min.

↓

After cooling the residue is taken up in 25 ml water transferred to a centrifuged tube

↓

Take 10 ml of extract (Aliquot) in 150 ml conical flask

↓

Add 2.5 ml of stabling solution and 0.2-0.3 gm $BaCl_2$ crystal

↓

Shake flask for 1 minute at constant rate

↓

Measure the turbidity in spectrophotometer at 340 nm wavelength

Total Sulphur (Turbidity Method, Chopman and Pratt, 1961

2 gm soil in 100 ml conical flask

↓

Add 20 ml of di-acidic (HNO_3:$HCLO_4$) mixture in 10:4 ratio

↓

Heat for an hour's continuous heating white residue obtained

↓

Cool and filter in 100 ml volumetric flask, wash with the residues and make up volume

↓

Take 10 ml of extract in 25 ml volumetric flask and add 1 gm of $BaCl_2$ crystal or powder form

↓

Shake well for one minute and add 1 ml gum acacia solution

↓

Take the turbidity reading after 5-20 min on the spectrophotometer at 440 nm wavelength.

In order to determine the amounts of N, P, K, S, Zn, and Fe, oven-dried plant samples were ground to the necessary fineness. Vanadomolybdate phosphoric yellow colour method

(Jackson, 1973) was used to measure phosphorus, while Microkjelddhl's method was used to estimate nitrogen. Tri-acid extract on a flame photometer was used to estimate potassium (Piper, 1966). Sulphur content in the plant was determined spectrophotometrically using the method described by Palaskar *et al.* (1981). In accordance with the methodology described by Dhyan Singh *et al.* (2005), the zinc and iron content of plant digestion was measured using an atomic absorption spectrophotometer.

Statistical analysis

The data obtained on soybean and safflower crop and different fractions of sulphur in soil for both the years were pooled and subjected to standard analysis of variance (ANOVA) followed standard procedures for Randomized Block Design (Gomez and Gomez 1984). The f test was used to find out comparable significant differences between treatment means with the least significant differences (LSD) at 5 % level.

RESULT AND DISCUSSION

Long-term effect of nutrient management on sulphur fractions (Pooled mean of 2017-18 and 2018-19)

Organic Sulphur

Mean of organic form of sulphur is the reserve source of sulphur for growth and development of plants, but it undergoes mineralization to sulphate form before availability to crops. Results (Fig. 2) indicated that long-term fertilisation had a substantial impact on form forms of sulphur. Organic form of sulphur varied from 28.25 to 37.13 mg kg^{-1} in in soybean-safflower cropping sequence under Vertisol. The application of 100% NPK + FYM @ 5 t ha^{-1} treatment resulted in significantly maximum content of organic sulphur (37.13 mg kg^{-1}), followed by solely FYM @ 10 t ha^{-1}, and both treatments are at par with each other. Whereas, significantly lower soil organic form of sulphur was observed (28.25 mg kg^{-1}) in absolute control. The soil organic form of sulphur was also influenced by zinc application (34.15 mg kg^{-1}) recorded by 100% NPK + Zn and lowest organic form of sulphur was found with 100 % NPK- sulphur and 100 % NPK after harvest of safflower crop.

Combining chemical and organic fertilisers resulted in a noticeably higher level of soil organic sulphur than the absolute control. This could be attributed due to applied chemical fertilisers combined with FYM as organic source. These results are consistent with those of Dutta *et al.* (2013), who found that continuous cropping with 100 % NPK(-S) led to the depletion of organic -S. The minimal amount of organic S in this treatment may be the result of continuous cropping without S addition Rongzhong *et al*. (2010), which increased the demand on native soil S to meet our crop requirements. According to Rajani *et al.* (2010) found that the application of FYM along with chemical fertiliser produced the highest level of organic sulphur, but it is only natural that the addition of organic material increases their bound nutrient in the specific soil which was followed by treatment with 100 % NPK + $ZnSO_4$.

Non-sulphate sulphur

Mean of non-sulphate form of sulphur accounts about 2-4 % of total sulphur content in soil in terms of the proportion of total sulphur, this fraction is the opposite of organic carbon.

The non-sulphate form of sulphur (Fig-2) was ranged from 107.85 to 118.28 mg kg^{-1} in soil. Application of 100 % NPK + FYM @ 5 t ha^{-1}, significantly recorded more non-sulphate form of sulphur (118.28 mg kg^{-1}) followed by only FYM @ of 10 t ha^{-1} (117.63 mg kg^{-1}) and 150 % NPK (116.05 mg kg^{-1}) and are on par with each other. However, significantly lower soil non-sulphate form of sulphur was recorded in absolute control. The increase or decrease in non-sulphate form of sulphur depends on the organic sulphur and sulphate sulphur in soils (Sutaria *et al*., 2016). Borkotoki and Das (2008) Non-sulphate form of sulphur is mostly made up of SO_4^{2-} occluded in and adsorbed on carbonates or insoluble S compounds of Fe and Al in soil which remains unextractable after removal of organic carbon and SO_4^{2-}—S

Water-soluble sulphur

Mean of water-soluble form of sulphur (Fig.2) recorded significantly maximum by the treatment of 100% NPK + FYM @ 5 t ha^{-1} (12.28 mg kg^{-1}) followed by 150 % NPK ha^{-1} at harvest in soil. Whereas, lowest water-soluble form of sulphur (10.15 mg kg^{-1}) was observed in fallow treatment. The water-soluble sulphur content was enhanced by the constant application of FYM and single super phosphate as P source. This could be as a result of SSP, which is soluble in water, and the release of sulphur from organic sources (Setia and Sharma, 2005). More microbial activity under various organics led to the mineralization of organic sulphur to accessible sulphur may be responsible for the noticeably greater content of water-soluble sulphur component with the application of organics along with chemical fertilisers (Dutta et al., 2013).

Heat soluble sulphur

Mean of heat soluble sulphur (Fig.2) commonly referred as the mineralizable form of sulphur in soil. The long-term fertilization had significant impact on heat soluble sulphur and it was ranged between 19.25 – 46.50 mg kg^{-1} at harvest of safflower in Vertisol. Significantly higher heat soluble sulphur (46.50 mg kg^{-1}) found in 100% NPK + FYM @ 5 t ha^{-1} followed by only FYM @ of 10 t ha^{-1}. Whereas, significantly lower soil heat soluble form of sulphur was showed (19.25 mg kg^{-1}) in absolute control. The addition of FYM in combination with RDF considerably boosted the heat soluble-sulphur content in soil. This might be because this treatment contained more organic materials and this proportion of S was linked to organic material that produced S when heated (Bediger *et al.,* 1985). Setia and Sharma (2005) found that the application of 100 % N alone resulted in a larger content of heat soluble-S as compared to 100 % NPK (-S) in long term fertilizers experiment

Total sulphur

Pooled data on total sulphur varied from 142.48 to 153.65 mg kg^{-1} (Fig. 2) and found maximum total sulphur (153.65 mg kg^{-1}) in 100% NPK + FYM @ 5 t ha^{-1} followed by only FYM @ of 10 t ha^{-1}. The outcome is consistent with findings of Rajani *et. al.,* (2010) which showed that the application of organic manures had the highest total S content at 100 % NPK + $ZnSO_4$.

The application of FYM along with 100 % NPK+S raises the total-S status of the soil over 100 % NPK+S, according to Reddy *et al.* (2004). Total sulphur may be attributed to the presence of organics, which can supply extra sulphur in the soil. The faster rate of application through a single super phosphate may be the cause of the 150 % NPK+S treated higher plots. The total S concentration of the soil decreased above control under continuous cropping with 100% NPK without S application, which may be related to the crop's removal of S. According to Dalvi *et al.* (2018) the soil total sulphur content was noticeably higher in the treatment when FYM was applied than in the absolute control plot. This may be because the treatment had more clay than the absolute control plot and the total sulphur levels were lower.

The different forms of sulphur were increasing with increasing inorganic fertilizers dose, results showed that 150% NPK fertilizer dose recorded higher sulphur forms as compared to 50% NPK and 100% NPK fertilizer dose. Increased in total sulphur may be ascribed to its addition through single super phosphate and organic manures (Datta et. al., 2013)

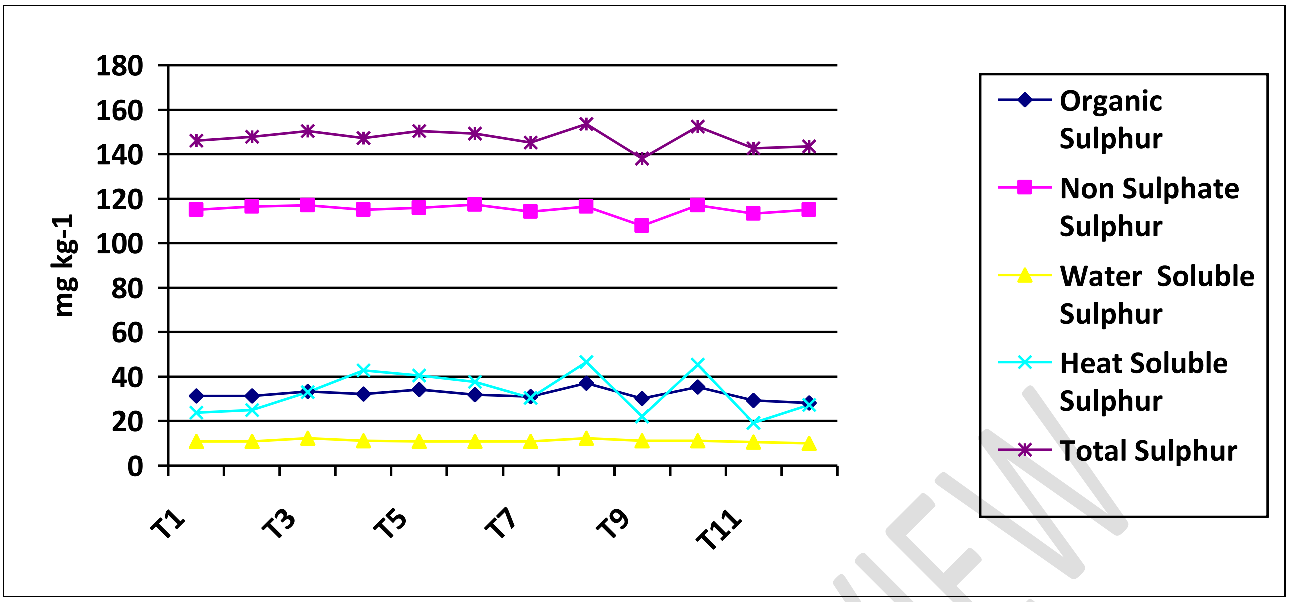

Fig. 2. Forms of soil sulphur as influenced by long-term use of organic manure and inorganic fertilizers under soybean-safflower cropping sequence (Pooled mean of two years)

Correlation of sulphur fractions with soil properties

The correlation coefficient of different fractions of sulphur was carried out with chemical properties of soil. It was illustrated from the table 1, the pH and EC were negatively correlated with all the sulphur forms of experimental soil. This might be due to presence of H^+ and OH^- ions on the soil complex, where H+ ions attract SO_4^{2-} ions under high salinity conditions SO_4^{2-} ions may be leached down because of the presence of salts in soluble forms.

Positively significant correlation of different forms of Sulphur were observed with organic carbon under Vertisols (r^2 =0.829**,0.470, 0.736**,0.663* and 0.742** organic Sulphur, non-sulphate Sulphur, water soluble Sulphur, heat soluble Sulphur and total Sulphur, respectively) and it was clearly indicated that the organic matter serves as a reservoir of Sulphur fractions. These results are in confirmatory with the results obtained by Bhatnagar *et al*., 2003. Similarly, Mali and Syed Ismail (2002) deliberated sulphate S, organic S, total S and non-sulphate Sulphur significantly affected by pH, EC, organic carbon content in soil. However, soil pH and EC showed negative and significant relationship with all the fractions of sulphur in Vertisols. Organic Sulphur and total Sulphur sustained by the significant positive correlation with available Nitrogen (r^2= 0.727** and 0.630*), P (r^2=0.760** and 0.742**), K (r^2= 0.769** and 0.677*), S (r^2= 0.905** and 0.684*) and DTPA – Zn (r^2=0.742** and 0.594*). These findings corroborate with the findings of Rajkonwar *et al.*, 2016. Water soluble Sulphur established positively significant relationship with available

Nitrogen (r^2= 0.802**), Phosphorous (r^2=0.814**) Potassium (r^2= 0.883**) and Sulphur (r^2= 0.876**). Water soluble Sulphur exhibited a positive and significant correlation with available Nitrogen indicating the influence of organic matter on Sulphur availability. Similar, observations have also been made by Borkotoki and Das (2008) in some Entisols of Assam observed the significant and positive correlation between water soluble Sulphur and available Nitrogen. The present investigation can be explained with the fact that Sulphur being an integral part of organic matter, the amount of organic carbon determines available Sulphur status in soil. Moreover, since Sulphur and Nitrogen are the essential constituents of proteins in organic matter, they maintain a definite N: S ratio. Hence, a significant and positive correlation of available Sulphur with available Nitrogen is imminent. Heat soluble Sulphur exhibited positively significant correlation with available Phosphorus (r^2= 0.696*), S(r^2=0.709**) and DTPA- Zn (r^2=0.682*).

Table 1. Relationship of different fractions of sulphur and chemical properties of soil.

	Organic S	**Non-sulphate S**	**Water soluble S**	**Heat soluble S**	**Total S**
pH	-0.797**	-0.318	-0.563	-0.629*	-0.633*
EC	-0.123	-0.123	0.340	-0.385	-0.144
Organic C	0.829**	0.470	0.736**	0.663*	0.742**
Available N	0.727**	0.384	0.802**	0.486	0.630*
Available P	0.760**	0.536	0.814**	0.696*	0.742**
Available K	0.769**	0.424	0.883**	0.494	0.677*
Available S	0.905**	0.302	0.876**	0.709**	0.684*
DTPA-Zn	0.742**	0.288	0.300	0.682*	0.594*

*Significant at 5% level: -0.576
**Significant at 1% level: -0.708

Grain and straw yield

Varying organic and inorganic treatments of long-term fertilization resulted in significant variations in soybean as well as safflower grain and straw yields. Mean grain and straw yield of both crops (Fig. 3) indicated that the 100% NPK + FYM @ 5t ha^{-1} gave significantly highest grain (18.30 q ha^{-1} and 8.70 q ha^{-1}) and straw yield (28.70 q ha^{-1} and 27.73 q ha^{-1}) of soybean and safflower, respectively and found to be significantly superior over all the treatments and statistically at par with 150% NPK, 100% NPK+ Zn, 100% NPK and 100% NPK + hand weeding. Even as, the absolute control followed by 100 percent nitrogen produced the lowest grain and straw yield soybean and safflower.

The increased yields with the application of NPK+FYM may be due to a boosted population of microorganisms that fix nitrogen and solubilize phosphate, increasing the nutrients' steady availability. The stimulated soil microbes are valuable not because they supply nutrients but also because they enhance the synchrony of plant nutrients demand with soil (Jain and Sharma, 2009). These results have been corroborated with the findings of Meshram *et al.*, 2019. Similarly, Sharma *et. al*., (2013) noted that the application of fertilizers alone or in combination with organic manures increased the grain and straw yield of the crops. Conjoint use of fertilizers and organic manures significantly increased grain and straw yield of crops over absolute control.

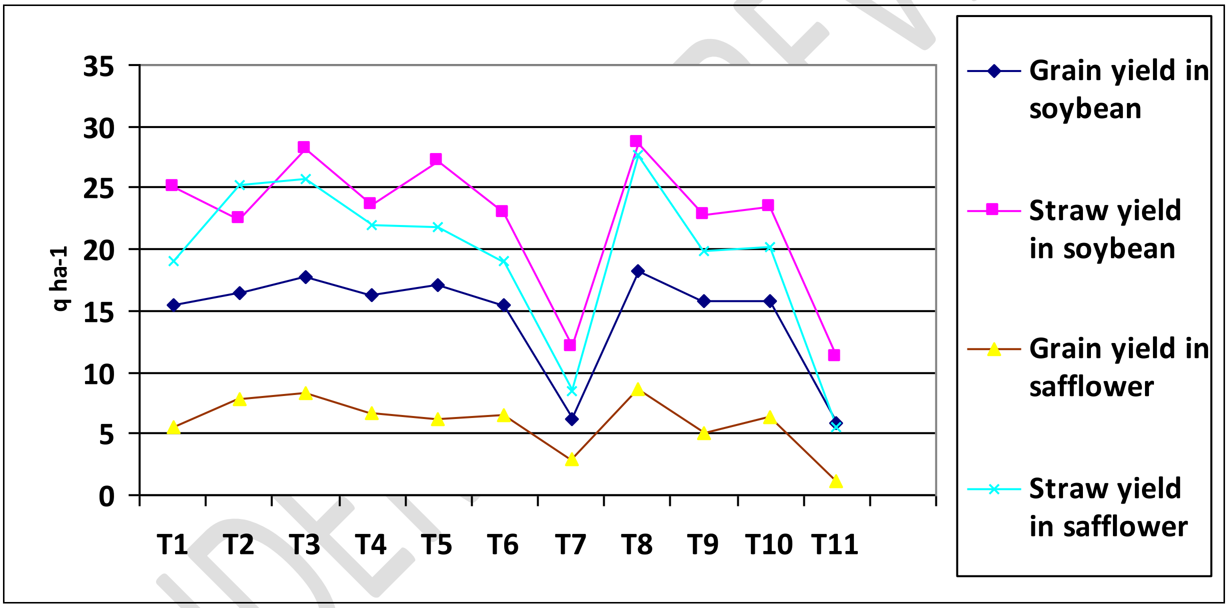

Fig. 3. Effect of manures and fertilizers on grain and straw yield of Soybean, safflower in Soybean – Safflower crop sequence (Pooled mean)

Nutrient uptake

The pooled mean uptake of **N**, P, K and S by grain and straw of soybean and safflower (Fig. 4) for the year 2017-2018 and 2018-2019 showed significant variations due to different treatments of long-term fertilization. The 100% NPK + FYM @5 t ha^{-1} noted significantly more total uptake of N, P, K and S (131.80, 26.27, 56.44 and 15.43 kg ha^{-1}, respectively) of soybean and uptake of N, P, K and S (54.43 kg ha^{-1},16.47 kg ha^{-}1, 69.90 kg ha^{-1} and sulphur 11.29 kg ha^{-1}, respectively) of safflower which was found statistically at par with 150% NPK in soybean safflower cropping sequence.

These results are in line with similar findings of Meshram *et al*. (2018) reported that treatment receiving 100% NPK + FYM @5 t ha^{-1} noted higher N, P, K, S and Zn uptake in soybean-safflower cropping sequence under Vertisol. Similarlly, Khandare *et al*. (2015 and 2020) observed that maximum N, P and K uptake by grain and straw of wheat were recorded with application of 100% NP. Arbad and Syed Ismail (2011) also reported a similar result for soybean under the soybean-safflower cropping system in Vertisol.

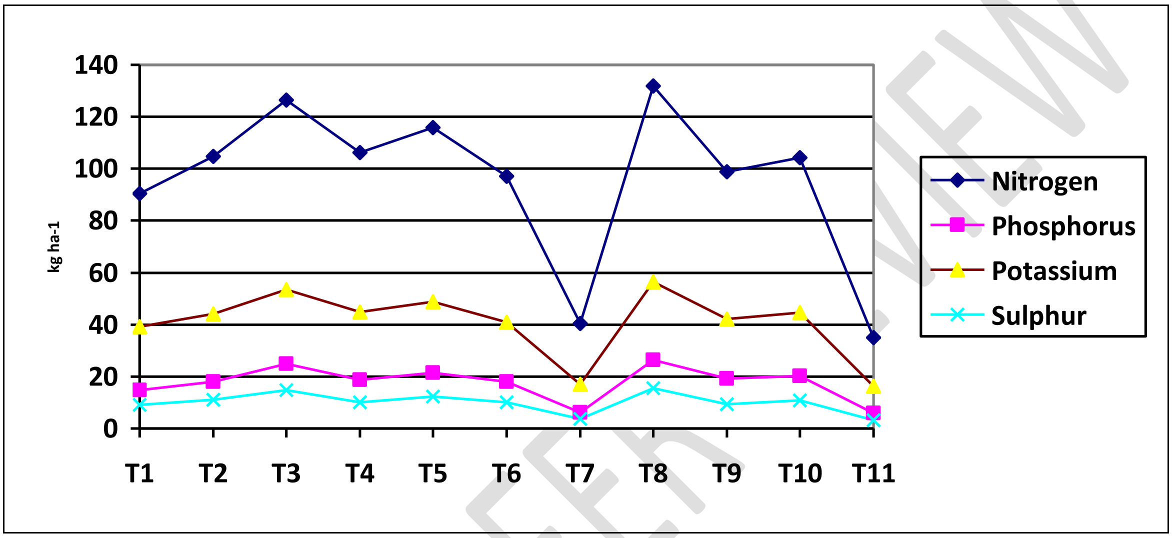

Fig. 4. Effect of different treatments on nutrient uptake (kg ha^{-1}) by Soybean crop in Soybean – Safflower crop sequence

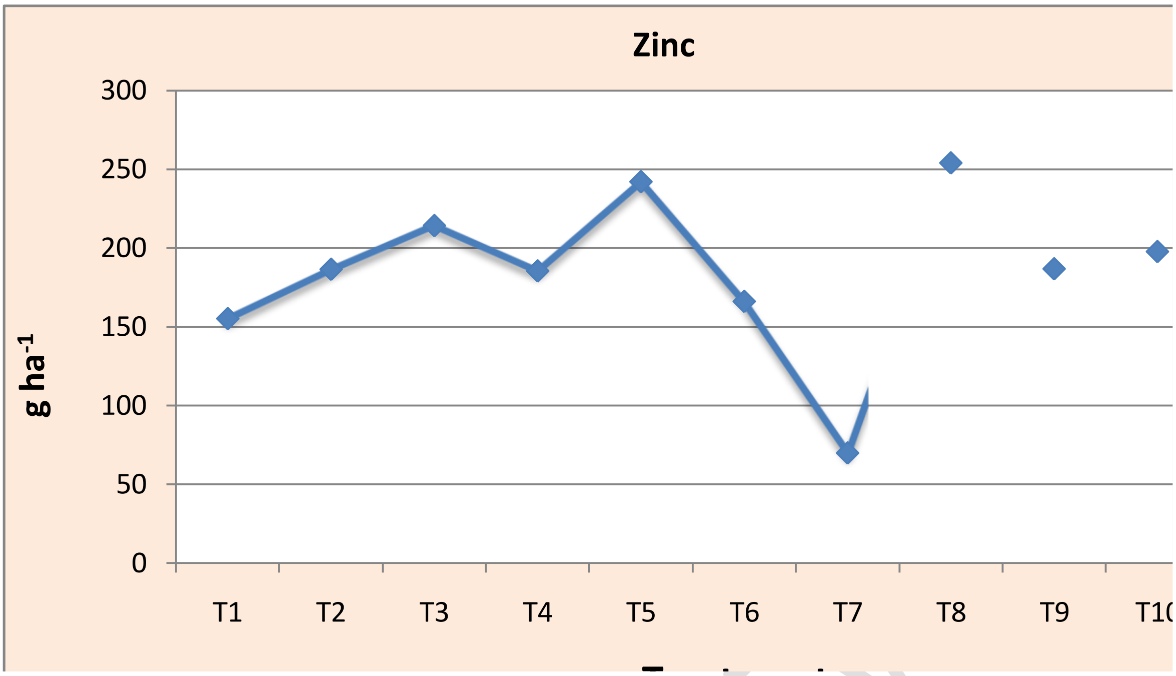

Fig. 5. Effect of different treatments on zinc uptake (g ha^{-1}) by Soybean crop in Soybean – Safflower crop sequence

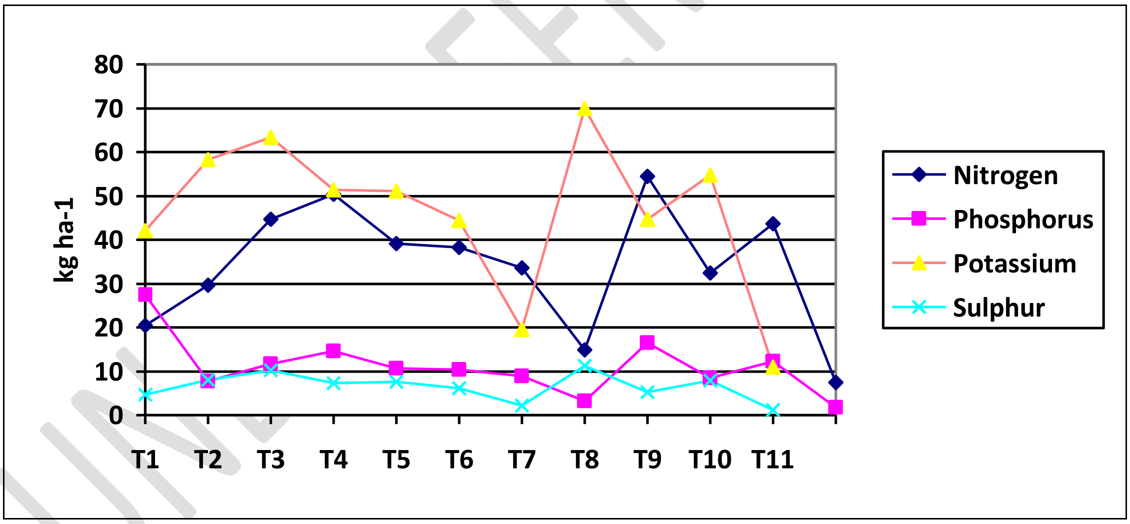

Fig. 6. Effect of different treatments on nutrient uptake (kg ha^{-1}) by Safflower crop in Soybean – Safflower crop sequence

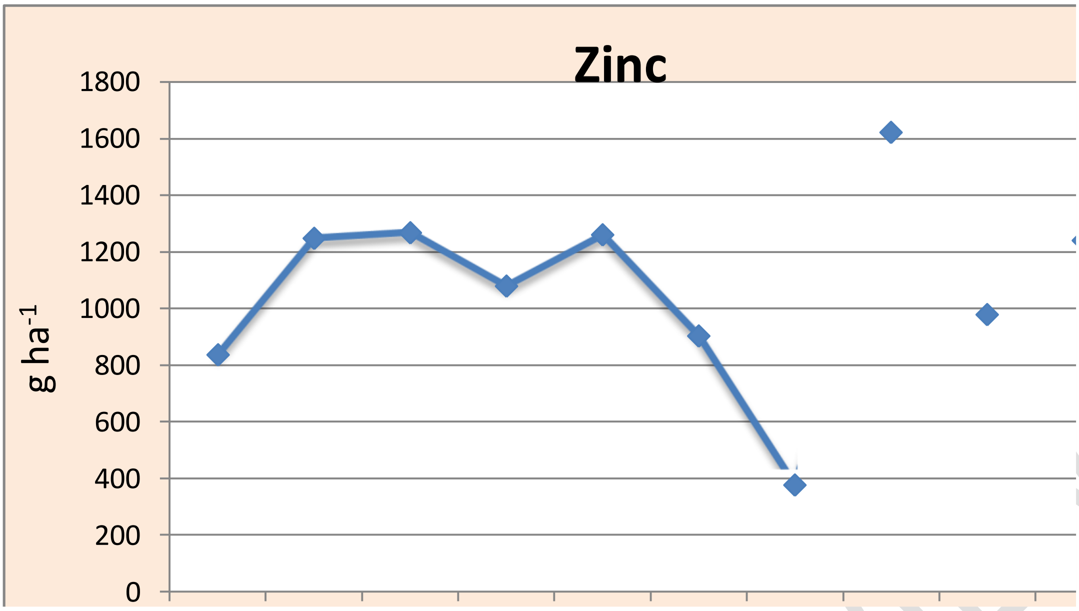

Fig. 7. Effect of different treatments on zinc uptake (g ha^{-1}) by Safflower crop in Soybean – Safflower crop sequence

CONCLUSION

It can be concluded from the above finding that sulphur fractions were recorded highest in the application of organic manure treated along with the inorganic fertilizers, such as 100% NPK + FYM @ 5 t ha^{-1} and Only FYM @ 10 t ha^{-1} at par with the 150% NPK. A significant impact on grain and straw yield as well as nutrient uptake (N, P, K, S and Zn) was noted highest by the treatment of 100% NPK + FYM @ 5 t ha^{-1} in soybean and safflower crop sequence under Vertisol.

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