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SULPHUR MINERALIZATION UNDER ORGANIC MATTER AND ELEMENTAL SULPHUR TREATMENT IN SOILS OF WUDIL AND GAYA LGAs OF KANO STATE, NIGERIA

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Abstract
A laboratory investigation was carried out to determine the effect of organic matter and elemental sulphur fertilizer on the mineralization of sulphur in soils of Wudil and Gaya local government of Kano state, Nigeria. Soil samples were collected along intensively cultivated farms in both Gaya and Wudil communities across 36 different farms (18 farms in each location). Soils were incubated with cow dung, elemental sulphur and a mixture of cow dung and elemental sulphur at a ratio of 1:2. Different forms of sulphur were determined weekly and changes in different fractions were monitored. Significant differences ($p < 0.05$) were observed in the concentration 420.10mgkg^{-1} - 498.30mgkg^{-1} of different forms of sulphur obtained. There was an increase in all forms of sulphur; total sulphur 420.10mgkg^{-1}, inorganic sulphur 36.89mgkg^{-1}, sulphate sulphur 35.83mgkg^{-1}, and organic sulphur 383.50mgkg^{-1} during the three weeks incubation period except for non-sulphate 0.74mgkg^{-1} and adsorbed sulphur 1.06mgkg^{-1}. The highest concentration of total sulphur (498.30mgkg^{-1}) was obtained when the soil was incubated with a mixture of cow dung and elemental sulphur while the lowest was observed with the control (420.10mgkg^{-1}). It can be concluded that a mixture of elemental sulphur and cow dung improved sulphur mineralization in soils of Wudil and Gaya local government area of Kano state, Nigeria.
Keywords: Total sulphur, organic sulphur, inorganic sulphur and sulphate sulphur.

Introduction
Among plants' essential nutrients, sulphur is ranked fourth and its role in plant metabolism is well documented. This includes structural component of proteins, catalyst in chlorophyll production, promotes nodulation in legumes, as well as resistance to pests and diseases attacks (Sutar *et al.,* 2017). Sulphur is similar to nitrogen in that both compounds play an important role in cycling but there is no large reservoir of sulphur compounds in the atmosphere. Also, there has been a negative sulphur balance in most parts of the world and most of the earlier works carried out in Nigeria has indicated sulphur deficiency (Raji, 2008). This deficiency is associated with the use of high analysis fertilizer, restriction on SO_2 emission due to pollution, decline in the use of sulphur containing fungicides, use of high nutrient demanding crops, poor use of organic matter (Scherer, 2001). Large amount of sulphur is found in organic matter and its mineralization into plants' available form is microbially driven. However, it is influenced by some factors including temperature, texture, manure characteristics and soil moisture. The sandy texture and low organic matter content of the soils of dry savanna zone of northern Nigeria have been linked to the deficiency of sulphur in the soil.

Thus, it is pertinent to understand some important soil characteristics such as pH, texture, moisture content, clay minerals and organic matter content of the soil as they may influence the mineralization process by controlling the retention and leaching of highly mobile sulphates in the soil (Biswas *et al.,* 2003). Similarly, the mineralization of sulphur in the soil and the factors controlling it are essential for predicting sulphur supply and management in the soil. The organic form of sulphur cannot be readily taken up by plants except it is converted to inorganic form. Organic sulphur generally becomes available to plants through mineralization to sulphate. The sulphate sulphur from organic matter as well as elementals sulphur fertilizer is the only form that can be held against leaching. All other sources from fertilizers are prone to leaching. However, mineralization is done by soil microbes and therefore requires soil conditions that are warm, moist and well drained to proceed rapidly. Mineralization of organic sulphur involves two processes: biochemical and enzymatic processes. Biological mineralization involves the breakdown of organic sulphur in to carbon bonded sulphur and subsequently into sulphate sulphur in the presence of *Pseudomonas putida.* Biochemical mineralization involves the breakdown of organic sulphur to ester sulphate in the presence of sulphatase (Hayes *et al.,* 2000). The rate of mineralization varies from soil to soil. Studies have shown that only *pseudomonas putida* is the only microbe involve in the mineralization and immobilization of sulphur. Its functions include increased mobilization, protection against pathogens and production of phytohormones such as auxins. The microbe makes use of carbon from organic matter as energy source and produces plant available sulphur (Yurembram *et al.,* 2015). When manure is used as a source of particular nutrient, knowledge of the mineralization rate under field condition is needed.

The present study was set out to assess the mineralization of sulphur in soils of Wudil and Gaya local government area of Kano state, Nigeria under controlled condition as well as improve sulphur mineralization by complimenting the sulphur in organic matter with elemental sulphur fertilizer to improve the sulphur supplying capacity and management in soils of dry savanna zone of northern Nigeria.

Materials and Methods

Soil Sampling and Preparation

Soil samples were collected along intensively cultivated farms in both Gaya and Wudil communities across 36 different farms (18 farms in each location). On each farm, four samples were collected and mixed to form a composite sample. Soil samples were air-dried. The samples were gently crushed and passed through a 2mm sieve size. Two different types of materials were used; cow dung and elemental sulphur. The organic residue used was cow dung which was collected from the Department of Animal Science Farm, Bayero University, Kano. Elemental sulphur used was produced by Shell Thiogro phosphate Fertilizer Company. The organic residue was dried and crushed gently into fine particles for analysis.

Soil Quality Analysis

Soil texture was determined using the Boyoucous hydrometer method as described by Gee and Bouder (1986). Soil pH was determined based on the IITA (1982) procedure and was measured using JENWAY 3520 glass electrode. Organic matter was determined using Walkley-Black wet oxidation method (Nelson and Sommers, 1982). Total nitrogen was determined by micro- Kjedhal digestion method (Bremmer and Mulvancy, 1982). Available phosphorus was determined using Bray and Kurt method (1945) and Olsen method (1945). Available S was determined turbidimetrically according to method described by Chesnin and Yien (1950). Exchangeable cations (Ca, Mg, K and Na) were extracted with 1N NH4OAC solution buffered at pH 7.0 as described by Anderson and Ingram (1998). Exchangeable acidity was determined with 1N KCl and titrated with 0.05N NaOH (Mclean, 1965). Available micronutrients were extracted using 1N HCl extraction solution (Mclean, 1965).

Sulphate sulphur was extracted with 30ml 0f 0.15 % $CaCl_2$ as suggested by Williams and Steinbergs (1959). Organic sulphur was calculated by subtracting 0.01M KH_2PO_4 extractable sulphur from total sulphur as described by Evans and Rost, (1945). Total sulphur was determined using diacid digest (USEPA, 1996). Adsorbed S was calculated by deducting the values obtained with 0.15% $CaCl_2$ extractant from those with KH_2PO_4 extractant as described by Fox *et al., (*1964). Non sulphate S was calculated by subtracting organic and inorganic S from total S as given by Virmani and Kanwar (1971). Inorganic S includes both $CaCl_2$ extractable S and adsorbed S. Sulphur content in all extracts was determined turbidimetrically as described by Chesnin and Yien (1951).

Incubation Experiment

Each of the sampled soil was incubated with cow dung, elemental sulphur and a mixture of cow dung and elemental sulphur. The ratio of soil sample to cow dung was 1:2 (5g of soil sample to 10g of cow dung), 5g of soil sample to 0.5g of elemental sulphur (1:10) and 5g of soil sample was mixed with 10g of cow dung and 0.5g of elemental sulphur. The treatments were thoroughly mixed and transferred in to a container. Water was added every three days to rewet the soil to maintain moisture above 75% field capacity by soil volume. This was done to stabilize the soil microbial population and avoid a sudden flush of sulphur mineralization.

Soil samples were analysed periodically during the first, second and third week of the incubation experiment to monitor changes in different fractions of sulphur. Sulphate sulphur was extracted with 30ml 0f 0.15 % $CaCl_2$ as suggested by Williams and Steinbergs (1959). Total sulphur was determined using diacid digest (USEPA, 1996). Adsorbed S was calculated by deducting the values obtained with 0.15% $CaCl_2$ extractant from those with KH_2PO_4 extractant as described by Fox *et al., (*1964). Non sulphate S was calculated by subtracting organic and inorganic S from total S as given by Virmani and Kanwar (1971). Inorganic S includes both $CaCl_2$ extractable S and adsorbed S. Sulphur content in all extracts was determined turbidimetrically as described by Chesnin and Yien (1951).

Data Analysis

Descriptive statistics were used to determine the physical and chemical properties. The results obtained from incubation analysis were subjected to analysis of variance and means with significant differences were done using the turkey test ≤ 0.05 P level.

Results and Discussion

Physical and Chemical Properties of Soils in Gaya

The results of particle size distribution for Gaya (table 1) showed that Gaya farm had an average sand content of 76.47 % and an average clay content of 11.41 %. All the sampling points fall within the Sandy Loam textural class (sand 76.47%, silt 12.11% and clay 11.41%). The sandy nature of Gaya farms could be attributed to the parent materials which were probably developed from pre-Cambrian basement complex rocks such as granitic sandstones (Shehu *et al.,* 2015). It was observed that the mean pH value across Gaya farm was 6.99 which were categorized as neutral, there was low variability in pH across the farm with a coefficient of variability (CV) of 15.73 %. The neutral pH observed in Gaya farm may be partly due to the management practices adopted by the farmers which are considered ideal for most cultivated crops (Brady and Weil, 2002). The average total nitrogen across Gaya farm was found to be 0.06 %, with a minimum value of 0.004 % and a maximum of 0.39 %. The average total nitrogen across all the sampling point was categorized as low with a moderate CV of 69.44 %.

The spatial distribution of nitrogen across the farm also reveals that the nitrogen was low to moderate across most sites, with some few boundaries having high nitrogen content. Organic carbon is an important component of the soil which serves as a store house for plant nutrient, the mean organic carbon in Gaya farm was 0.38 % which was categorized as very low. Similarly, the low content of organic matter could be related to the poor land cover in Gaya farm. The average sulphur content in Gaya farm was 0.14 %. The sulphur level in the soil was categorized sufficient (NSPFS, 2005) because the critical concentration of sulphur in soil is 0.06%, below which is considered deficient. Majority of the farms in Gaya are cereal based cropping system and legumes which are known to require low amount of sulphur compare to oil seed crops or other high yield crop varieties that will require large amount of sulphur. Sawyer *et al.* (2015) reported that high yield crops extract more S and also creates additional crop residue high in sulphur content. During decomposition process, inorganic S may be preferentially utilized by soil microbes making it unavailable to the crop (immobilization). This could be related to the sufficient level of sulphur observed in Gaya farm which may be associated with the cropping of low sulphur requiring crops. The available phosphorus in Gaya farm as shown in table 1 was in the range of 0.52 to 51.34 $mgkg^{-1}$ with a mean of 12.50 $mg^{-1}kg$ which falls under the moderate fertility class (NSPFS, 2005). The mean exchangeable potassium in Gaya farm was 0.14 $cmolkg^{-1}$ which was rated as low according to Esu (1991).

Table 1: Physical and Chemical Properties of Soils in Gaya

Soil parameter	Mean	Minimum	Maximum	CV
Sand (%)	76.47	73.80	78.00	1.14
Silt (%)	12.11	6.24	19.69	20.97
Clay (%)	11.41	5.14	16.22	16.54
pH (1:1)*	6.99	4.54	8.90	15.73
Nitrogen (%)	0.06	0.00	0.39	69.44
Organic C (%)	0.38	0.09	1.97	56.12
Sulphur (%)	0.14	0.04	0.66	50.88
P ($mgkg^{-1}$)	12.50	0.52	51.34	94.55
K ($cmolkg^{-1}$)	0.14	0.00	7.07	394.63
Ca ($cmolkg^{-1}$)	1.13	0.05	8.47	95.58
Na ($cmolkg^{-1}$)	0.44	0.01	8.37	154.66
Mg ($cmolkg^{-1}$)	0.54	0.00	5.89	136.25
Fe ($mgkg^{-1}$)	83.13	0.01	101.30	106.25
Zn ($mgkg^{-1}$)	2.75	0.012	128.50	342.61
Cu ($mgkg^{-1}$)	2.24	0.048	5.32	42.14
Mn ($mgkg^{-1}$)	0.14	0.002	3.58	213.83

***pH (1:1): Soil to water ratio**

Considering the spatial distribution of potassium across the farm, it was observed that a large portion of the land apparently have low potassium content (0$mgkg^{-1}$). These showed that the farm requires different potassium management strategies, such that places with high potassium levels be allocated for high potassium demanding crops like crop trees. The mean exchangeable calcium, magnesium and sodium across the farm as shown in table 1 were 1.13, 0.54 and 0.44 $cmolkg^{-1}$ respectively. Calcium and magnesium were rated as low and medium, respectively. While sodium was rated as high according to Esu (1991). The low contents of total nitrogen (N), organic C, available P and ECEC in Gaya farm could be related to the low inherent status, such as the parent materials which are mostly of aeolin origin with low weatherable mineral reserve and partly due to the complete crop removal by the farmers (Manu *et al.,* 1991).

Physical and Chemical Properties of Wudil Farms

Table 2 shows the physical and chemical properties of soils in Wudil. The result of the particle size distribution in Wudil indicates that majority of the textural class were Sandy Loam (sand 75.10%, silt 16.29% and clay 8.59%) with a high sand content of 75.10% across the farm. The sandy nature of the soil calls for proper soil management to reduce the effect of nutrient leaching during the cropping season. Malgwi *et al.,* (2000) and Voncir *et al.,* (2008) has suggested that the dominance of sand contents of soils in northern Nigeria is as a result of sorting of materials by clay eluviation and wind erosion. This could be associated with the high sand fraction observed in both locations. It is clear that Wudil has a slightly acidic pH of 6.24 (NSPFS, 2005). The slightly acidic pH could be related to the management practices adopted by the farmers which are considered ideal for most cultivated crops (Brady and Weil, 2002). The mean organic carbon was 0.24 % as shown in table 2 was categorized as very low (NSPFS, 2005). Savannah soils of northern Nigeria are also characterized with short wet season and prolong dry season (6-9months) with abundant short grasses and a few scattered trees (Sowunmi and Akintola, 2010). This could be attributed to the poor land cover in Wudil farms which resulted in low amount of organic matter. The organic carbon level across the farm ranged from 0.16 % to 0.30 %, with a CV of 15.60 % (table 2). The spatial distribution of soil organic carbon across the farm also showed that there was very low level of organic C across the study area. According to Shehu *et al.,* (2015), soil productivity declines when vegetation cover is lost and appropriate management practices are not adopted which could results in depletion of organic matter and reduced agricultural productivity. The average total nitrogen across the farm was found to be 0.03 %, with a minimum value of 0.018 % and a maximum of 0.05 %. The average total nitrogen across all the sampling point as shown in table 2 was categorized as very low (NSPFS 2005) with a low CV of 22.63 %. The spatial distribution of nitrogen across the farm also reveals that the nitrogen was very low across all sites. The average sulphur content was 0.09 % with a range of 0.066 % to 0.11 %. The sulphur level in the soil was categorized sufficient because the critical concentration of sulphur in soil is 0.06%, below which it is considered deficient. The low CV of 13.36 % showed that there is no much difference in spatial variability of sulphur across the farm, the available phosphorus in the study area was in the range of 0.94 to 11.92 $mgkg^{-1}$ with a mean of 5.98 $mgkg^{-1}$ which falls under the low category (NSPFS 2005).

The spatial distribution in phosphorus content observed across the field (CV of 55.23 %) falls between two fertility classes; low to moderate (table 2). Also, the low to moderate amount of P observed in Wudil farms could be related to the sufficient level of available S. Camberato *et al.,* (2012) reported that soils with high P level can displace available S contributing to deficiency of sulphur in the soil. This could be related with the sufficient level of sulphur in the soil. The low amount of exchangeable cation exchange capacity (ECEC) of the soil in Wudil farms implies that the soils were dominated by low activity clays and sesquioxides (Tan, 2000). The mean exchangeable potassium was 0.08 ($cmolkg^{-1}$) which was rated as low according to Esu (1991). Considering the spatial distribution of potassium across the farm, it could be observed that a large portion of the land have low potassium content, while some few areas are high in potassium. These showed that the farm requires different potassium management strategy. The mean exchangeable calcium, magnesium and sodium across the farm as shown on table 2 were 0.94, 0.21 and 0.19$cmolkg^{-1}$ respectively. Calcium and magnesium were rated as low while sodium was rated as moderate according to Esu (1991). The low contents of total nitrogen (N), organic C, available P and ECEC in both locations could be related to the low inherent status, such as the parent materials which are mostly of aeolin origin with low weatherable mineral reserve and partly due to the complete crop removal by the farmers (Manu *et al.,* 1991).

Table 2: Physical and Chemical Properties of Soils in Wudil

Soil parameter	Mean	Minimum	Maximum	CV
Sand (%)	75.10	74.50	75.70	0.35
Silt (%)	16.29	7.89	20.33	17.16
Clay (%)	8.59	4.93	17.22	31.79
pH (1:1)*	6.24	4.44	8.11	14.68
Nitrogen (%)	0.03	0.02	0.05	22.63
Organic C (%)	0.24	0.16	0.30	15.60
Sulphur (%)	0.09	0.07	0.11	13.36
P ($mgkg^{-1}$)	5.98	0.94	11.92	55.23
K ($cmolkg^{-1}$)	0.08	0.01	0.38	113.36
Ca ($cmolkg^{-1}$)	0.94	0.09	5.47	116.14
Na ($cmolkg^{-1}$)	0.19	0.03	0.33	42.85
Mg ($cmolkg^{-1}$)	0.21	0.02	1.31	119.42
Fe ($mgkg^{-1}$)	21.55	4.50	59.72	58.53
Zn ($mgkg^{-1}$)	1.62	0.084	11.59	145.14
Cu ($mgkg^{-1}$)	2.42	0.446	4.19	38.24
Mn ($mgkg^{-1}$)	2.58	0.221	17.46	122.29

***pH (1:1): Soil to water ratio**

Sulphur Mineralization in Gaya and Wudil farms

All forms of sulphur increased as a result of added nutrients. The increase in the forms of sulphur was more in soils treated with elemental sulphur and organic matter (total S 498.30mgkg^{-1}, organic S 452.70mgkg^{-1}, inorganic S 44.31mgkg^{-1}, sulphate S 43.44mgkg^{-1} and non-sulphate S 1.53mgkg^{-1}. High amount of sulphur was recorded on the first and third week of the incubation experiment (table 3) with week three having the highest amount of total S 485.30mgkg^{-1}, organic S 442.60mgkg^{-1}, inorganic S 42.99mgkg^{-1} and sulphate S 42.34mgkg^{-1}.

Table 3: Effect of Treatments, Time and Location on the Various Forms of Sulphur (mgkg^{-1})

Source	Total S	Inorganic S	Sulphate S	Organic S	Non sulphate S	Adsorbed S
C	420.10^{c}	36.89^{c}	35.83^{c}	383.50^{c}	0.74^{c}	1.06
S + Es	420.10^{c}	36.60^{c}	35.59^{c}	383.60^{c}	0.83^{c}	1.00
Cd + S	489.70^{b}	44.31^{b}	43.44^{b}	445.00^{b}	1.27^{b}	0.87
S + Cd + Es	498.30^{a}	44.99^{a}	44.09^{a}	452.70^{a}	1.53^{a}	0.90
SE	3.290	0.276	0.263	3.110	0.005	0.080
Time						
Week 1	452.70^{b}	40.70^{b}	38.93^{b}	411.70^{b}	2.06^{a}	1.77^{a}
Week 2	433.10^{c}	38.40^{c}	37.94^{c}	394.40^{c}	0.79^{b}	0.46^{c}
Week 3	485.30^{a}	42.99^{a}	42.34^{a}	442.60^{a}	0.42^{c}	0.65^{b}
SE	3.510	0.337	0.333	3.220	0.076	0.058
Location						
Wudil	443.00^{b}	40.65	39.76	402.20^{b}	1.13	0.89
Gaya	472.60^{a}	40.88	39.89	431.70^{a}	0.99	0.99
SE	3.000	0.297	0.211	2.740	0.041	0.042

^{a,b,c}: Means not connected by the same letters are significantly different using turkey HSD; C: Control; S + Es :Soil sample with elemental sulphur; S + Cd: Soil sample with cow dung only; S + Cd + Es: Soil sample, cow dung and elemental sulphur; Trt: Treatment.

The high mineralization of sulphur was linked to the carbon source in the organic matter which helps to enhance the proliferation of soil microbial activities thus, rendering a comparatively low amount of mineralizable sulphur in to the soil (Saren *et al.,* 2016).

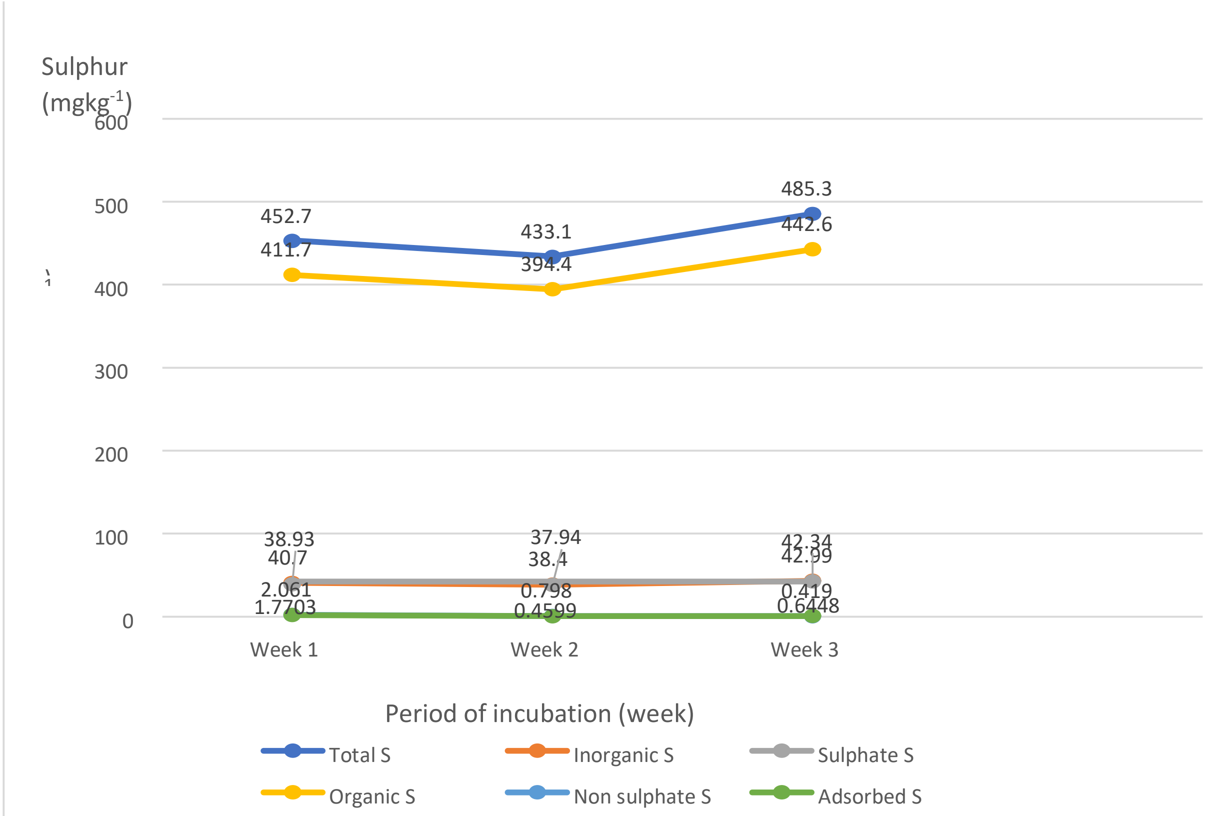

Figure 1: Effect of organic matter and elemental sulphur fertilizer on the mineralization of sulphur

This could also be attributed to the high amount of sulphur during the initial stage of the incubation which later gets mineralized and become reduced during the second week by sulphur oxidizing microbes. At week two total S was 433.10mgkg^{-1}, organic sulphur 394.40mgkg^{-1}, inorganic S 38.40mgkg^{-1}and sulphate S 37.94mgkg^{-1} (Fig 1). Dead sulphur oxidizing microbes and decomposed plants' residues contributed more to the amount of sulphur with time. However, a slow rate of mineralization was observed with soils treated with elemental sulphur alone during the period of incubation. Total sulphur 420.10mgkg^{-1}, organic S 383.50mgkg^{-1}, inorganic S 36.60mgkg^{-1}, sulphate S 35.59mgkg^{-1}non sulphate S 0.83mgkg^{-1} and adsorbed S 1.00mgkg^{-1} (Fig 1). This slow rate of mineralization could be attributed to the immobilization of sulphur by sulphur oxidizing microbes rendering a comparatively lower amount of sulphur in the soil (Saren *et al.,* 2016). With increase in the period of incubation, dead sulphur oxidizing microbes decomposes and sulphur is released in to the soil from the organic source (Das and Sah, 2003).

Conclusion

It could be concluded that addition of elemental sulphur and organic matter improved the mineralization of sulphur in soils of Gaya and Wudil farms. This was evidence by the increase in the concentration of different forms of sulphur during the period of investigation. The highest form of sulphur was recorded with soil samples treated with elemental sulphur and organic matter.

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