Article

Influence of Incidental Application of Sulfur Impurity in Triple-Super Phosphate Fertilizer on Wheat Yield

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Abstract: This research was specifically aimed at assessing the influence of sulfur in triple-super phosphate (TSP) on wheat yield. From the results, wheat showed response to sulfur (S) from gypsum (in 67%); and nitrogen (N) from urea in about 100% (of 24 sites). Based on this N was found to be the most limiting element to wheat production followed by sulfur, and then by phosphorus. TSP is tested to contain agronomically up to 2-6% by weight of S. However, wheat didn't show response to S impurity supplied in the form of TSP. Though, not statistically significant, it is observed that there have always been yield increments by certain percent due to S from TSP in 8 out of 10 target sites, which is depicted in the increasing trends of yield response curves. From this it is learnt that, the benefits of the accidental/incidental application of such high analysis fertilizers can be many-folds in the quality attributes of wheat, if the soils of such investigation at the same time would contain significant amount of organic matter (OM). Indeed, such analysis would be vital in varietal specific nutrient requirement studies in precision-farming and/or in categorizing soils into fertility gradients and fertilizer recommendation domains.

Keywords: Wheat; yield; triple-super-phosphate; sulfur; nitrogen; micro-dosing, precision-farming

1. Introduction

Soil fertility decline in Ethiopia has been well documented with most of the attention that has been given to nitrogen (N), phosphorus (P) and potassium (K). Sulfur (S) is a macro-nutrient that is reported to be taken-up by grain crops in amounts similar to those of P, 10–30 kg/ha [1,2], with its content in plants approximately the same as that of P [3]. However, it is forgotten element in African agriculture, though its adequate level is considered to be important not only for satisfactory plant growth, but also known for ensuring optimum levels of S-containing essential amino-acids in grains; and oils, vitamins and flavored compounds in plants [4]. The essential amino-acids methionine (21% S), cysteine (26% S) and cystine (27% S), which are the building blocks of proteins that impact the nutritional-value of human food and feed contain S [5]. It is also known to enhance other nutrients use efficiency, and ranks second only to N in importance for optimum crop yield and quality produce [6]. For example, wheat protein is rich in non-essential glutamic-acid and proline, whereas deficient in most essential-amino acids, such as lysine, tryptophan, threonine, methionine and histidine [7]. These essential amino-acids contain sulfur; and without modest and/or significant supply of sulfur crop-plants can neither express their full genetic potential in terms of yield nor can complete their biological life cycle. These essential amino-acids are reported to have significant positive correlation with protein in wheat [7]. From this it follows that fertilizing even small amounts of S can bring about very important improvements in quality of wheat for humans nutrition, and enhance yields. Furthermore, the S status of wheat grain is an important parameter for the baking quality and nutritional values [4,5].

It is well recognized that plant species and varieties differ their nutritional requirement ranging from micro-dose to macro-dose depending on their degree of essentiality. In this regard, in terms of essentiality elements like S lie between the macro-nutrients (NPK), and the micro-nutrients; and in terms of demand, cereals lie between legumes and oil crops for S. Generally, cereals, are reported to remove about 10–15 kg S/ha [8]. [9,10], reported that wheat has a relatively low S requirement amounting to about 20 kg S/ha for producing grain yields of 8 Mg/ha. Similarly, [11] reported a modest amount of S, 15–35 kg S/ha for better quality and optimum yields of wheat. According to [4] crops like wheat and maize can respond to sulfur rating between 5–10 kg S/ha. A study by [12,13], reported 5 to over 20 kg S/ha even less for wheat depending on the soils and/or sites. This is in fact, an amount of S that could be obtained from accidental applications of fertilizers like TSP and di-ammonium phosphate (DAP), which should be account in using fertilizers in both in economic terms and environmental concerns. But, such responses and/or benefits are easily overlooked where basal dressing of P is applied as TSP, a high-analysis fertilizer which is commonly assumed to be free of S [14]. TSP, however, contains agronomically significant amount of S, 2–6% by weight [4,15]. This is a micro-dose of S that can significantly increase small holder farmers' yields in precision farming, thereby increasing the profitability and productivity of fertilizers, or will avoid unnecessary use of external inputs.

In the view of the above background, therefore, this paper sought to (1), see the response of wheat to incidental application of S from TSP; (2), to compare wheat response to S supplied as TSP and mineral gypsum; (3), to see the trend of change of wheat grain yield due to the interaction effect of S, N and P; and (4) to infer the most limiting nutrient element crops production in the studied locations from nutrient response curves.

2. Results and Discussions

2.1. Selected Physico-Chemical Properties of Soils of the Study areas

Table 3a and b presents the overall soil variables that are much related to the supply of S to plants in two seasons. As showed the studied soils varied widely in their physico-chemical properties, which is specific to the areas, in a way that can affect nutrients availability to plants, particularly sulfur. Soil pH is a chemical property that significantly influences the availability of plant nutrients and varied in wide ranges. In WS zone it was ranged strongly to moderately acidic 5.1 to 6.7. It was strongly acidic to near neutral pH (5.3 to 7.0) in Arsi zone. Whilst in ES zone it ranged from neutral to moderately alkaline (7.1 to 8.2) with the majority of soils containing visible nodules of fragmented calcium carbonate ($CaCO_3$) i.e., calcareous in nature. In general, in all studied areas, pH values fall within a range 4.5 to 8.5 reported by [29] for agricultural soils, with the values of 5.5 to 7.0 or 8.5 preferred by most grain crops and pastures.

Nitrogen (N) content ranged from 0.06–0.25%. This falls in a range that is considered to be very low to low based on the criteria developed by [29,30] for tropical soils. The low N content can be attributed to alarmingly low levels of SOC in the studied soils.

Available phosphorus (P) content for ES zone ranged between 7.55–10.99 mg/kg. This falls far below, the 10–20 mg P/kg, a range considered low; and less than 20 mg P/kg low [31]. Similarly, the Bray-I P for soils from Arsi and WS zones ranging from 0.22 to 5.12 mg/kg are far below the low level Bray-I P < 20 mg P/kg [31]. Though fertilizer responses are most likely expected in such low P soils, in the calcareous soils, its availability can be limited, because of P fixation reactions with Ca. This can also be true for strongly acidic soils from WS zone, because P would be fixed by Al.

Sulfate sulfur (SO_4-S) ranged between 1.30–24.18 mg/kg. Based on 10 - 13 mg/kg $CaCl_2$ extractable SO_4-S as a critical limit (CLs) for S deficiency reported by [32], over 50% of soils were below this and may be S limiting. In such soils, the responses to S are most likely in crops like wheat, maize, oil rapeseed etc. When the CL for the soils SO_4-S can be to stretch a bit higher, for example to 11.3 mg/kg, about 72% of the soils can be regarded as S limiting for wheat production.

Organic carbon (OC) content of the soils in first season ranged between 0.90–2.99% (Table 3a), falling in a range considered to be very low to low and/or marginal. The OC of most studied sites, 78% are far below the CLs being cited in literature, 2.0%

The properties of the soils in season-II are presented in Table 3b, but didn't show much variation with that in season-I. Overall, for example, in this set of experiments the N content which ranged between 0.05–0.21% falls within a range considered to be very low and low for tropical soils, signifying that the studied soils are inherently low in the contents of N, which is expected to be affecting the sustainability of crop production, thereby negatively affecting food security. Available phosphorus (Av.P) in season-II, for ES zone ranged between 9.02–12.01 mg/kg, falling within a range considered to be low to marginal [30]. Similarly, for soils from Ar and WS zones, P values ranged between 0.50–3.01 mg/kg was very low. The sulfate sulfur (SO_4-S) in this season ranged between 4.03–35.83 mg/kg (Table 3b). Based on [32] rating for $CaCl_2$ extractable SO_4^{-2}-S as CL for S deficiency for most crop species 66.7% of the studied sites were found be S deficient. But, two sites (WG and BT) were found to be adequate in S. The OC contents of soils ranged between 0.96–2.71% (Table 3b), falling in a range considered to be very low to low or marginal. Most importantly, the OC contents of most studied soils were far below the CLs suggested by various workers [31] for sustaining soil health. In general, from the present study in about 83.3% of soils had very low levels of OC, which can be regarded as the major cause for the alarmingly low levels of plant available N, S and/or P. This is thought to be negatively affecting soil health or its quality. So, restorative measures are needed to rectify the problem, especially at smallholding farmers' level for sustainably building the soil OC to be an acceptable minimum level.

2.2. Wheat Response to Sulfur from Triple-Super-Phosphate

Ethiopia needs to increase agricultural production in order to feed an ever increasing population estimated at 102 million in 2017 [33]. To ensure cost-effective and quality produce, however, healthy soils are needed. In this regard, sulfur is essential not only for plant growth and quality produce, but also enhances other nutrients' use efficiency and ranks second only to N in importance for optimum crop performance [6,11]. The forthcoming sections discuss wheat response to S supplied as TSP (S + P), gypsum (S + Ca), N and/or P. For this purpose the sites which showed no response to P (P sufficient) and/or both extremities of soil pH conditions (high or low) are subjects of interest.

Wheat showed significant response to S supplied as gypsum (S + Ca) (Figures 1 through 6). Particularly, wheat showed highly significant response (***$p \leq 0.001$) to N in all sites. The sites which didn't reveal response to P in relation to next lower level treatment (i.e., NS) were AA_1, Do_1 and BL_1 (Ar zone) which makes 16.7%; and Ud_1 and Ki_1 (ES zone) 5.6%. But, those with marginal response to P were Do_1, Ud_1, DL_1 and NK_1 making 22% of the sites. With respect to S about 50% of sites gave highly significant response (***$p \leq 0.001$) to S from gypsum as related to soil-test levels; and 22% had marginal response (*$p \leq 0.05$), but 30% didn't show response. Figures 7 through 10 present wheat response to S-gypsum, N and/or P at G/Silingo$_2$ (GS_2); Keteba (Ke_2) and Bekejo (Bk_2); and Nano Suba (N/S_2) sites in season-II, (2015-16). These are sites which showed response to S-gypsum, but not to P from TSP. Here too, wheat showed significant response to S, N and/or P. Particularly, wheat showed highly significant response (***$p \leq 0.001$) to N; and significant response to S, either (**$p \leq 0.01$) or (*$p \leq 0.05$).

Taking specific locations, for example GS$_2$ site had better correlation of wheat yield with soil-test levels of (particularly N and S) than its neighbor WG/Do$_2$ (data not shown). Similarly, in ES zone S response was more pronounced in Ke$_2$ site than Bk$_2$. The low wheat yield in Bk$_2$ compared with Ke$_2$ may be due to the slightly lower soil-test SO_4-S levels. The sandy-clay nature of soils in Bk$_2$ might have also caused the loss of available S below the rhizosphere before it can be absorbed by the test-crop. It is widely reported that S deficient soils are often low in OM, coarse-textured, well-drained and subject to leaching [34]. Sandy soils are also reported to adsorb less (SO_4^{2-}) than both clay and silt-sized particles [35,36]. In this ES zone wheat response follows similar trend of change as Arsi zone. From this it is observed that ES had better consistency of response at all treatments, correlating better with soil-test levels compared with Ar and WS. Furthermore, in ES there was better positive synergy between S, N or P as there has always been increasing yield advantage with the type and level of nutrients supplied. However, the interaction effect of S with N is more pronounced than with S and P. [37] made a similar observation, in which case P has no such a big influence as N on protein content other than supporting the results in better assimilation and metabolism of absorbed N forms. In final analysis, the 10 figures depict that in all sites/fields with applied N there were sharp-rises in yield curves including the non-P and non-S responsive sites like Boneya Edo (BE$_1$). Furthermore, weather it is statistically significant or not the graphs are always rising at certain percent with the type and/or level of fertilizer added, (except in between NS$_3$-NP at Bekejo$_2$ and N/Suba$_2$) sites, indicate a strong positive synergy of (N, S and/or P) one over the other in producing the combined effects. But, the observed yield increments due to S in treatments up to or between 4th and 5th, (NS$_2$ and NS$_3$); and the discontinuing between 5th and 6th (NS$_3$ and NP); but again the response between, 6th and 7th (NP & NPS$_1$) and above may further affirm a strong positive interaction effect of N and S in increasing yield one over the other than N and P only.

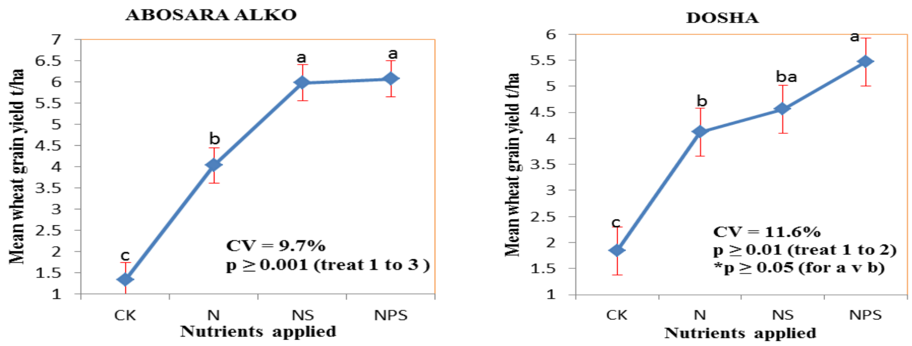
With applied N in in terms of yield gains, for example, 72.2% to 148.7% grain yield advantages over control in season-II alone were obtained. This collectively affirms that N is the most limiting element followed by S and/or P for wheat or other crops production in Ethiopian agriculture. This implies that to benefit from any kind of added essential element, saturating soils first with N is of paramount importance; because one can't harvest any photosynthetic produce, from any other added nutrient, if the soils are not supplied first with adequate amounts of this the most limiting element. This in turn has a strong connection with the low contents of OM vis-à-vis the dynamics of N in tropical climate and soils. This indicates that N management followed by S and/or P might be of at most importance in sustaining crop production in the studied areas.

Overall, in all 24 sites, maximum grain yield obtained was 6.6 t/ha, (i.e., even in only in one site, TH$_1$). As was reported in UK, the average grain yield of wheat, however, can reach on average $\geq$ 8.5 t/ha when all conditions are optimized [38]; indicating that the potential attainable yield can still be higher, if other factors are non-limiting. Only two sites had very slight negative yield advantages i.e., in treatments between NP and NS$_3$ (Bekejo$_2$ and Nano Suba$_2$) sites. All the rest had positive yield gains in certain percentages. For example, in Bk$_2$ site the grain yield obtained from treatment, NS$_3$ (3.9 t/ha) was slightly greater than that with treatment NP, (3.8 t/ha). At N/Suba$_2$ also the yield obtained from treatment, NS$_3$ (4.18 t/ha) was greater than that from treatment, NP (4.06 t/ha). This may indicate that in highly S-limiting soils, the test-crop's tendency in absorbing/assimilating more S than P. Indeed, the soils from these sites are tested very low in S compared with others showing better interaction of N with S in impacting wheat yield under S limiting soils than that with P. Such strong interaction effect of N with S on yield and quality attributes has been reported by other workers [39,40,41].

In fact, the responses to S are easily overlooked where a basal dressing of P is applied as triple-super phosphate (TSP), a high analysis compound fertilizer which typically does contain agronomic-ally significant quantities of S (2–6% by weight) [4,15]; in addition to P (45% P_2O_5) and Ca (15.0%). Sulfur as an impurity is associated with TSP during its manufacturing from rock phosphate and sulfuric acid (H_2SO_4). To see such effects, pair-wise orthogonal comparisons among treatments using SAS contrast statements were done on sites that showed response to applied S from gypsum (significantly or marginally), but not to P. The major target sites for such analysis were: AA_1, Do_1, BL_1, GS_2 (Ar zone); Ud_1, Ki_1, Ke_2 and Bk_2 (ES); and NK_1/(DL_1) (WS) in first$_{(1)}$ and second$_{(2)}$ seasons (Tables 5 through 9). The effect can be seen from yield gap between NPS_3 and NS_3. It is well noted that wheat cultivar showed significant response (***$p \leq 0.001$) to S-gypsum on average, 67% of sites in two seasons. However, the responses of wheat to S from TSP were not statistically significant, or the ANOVA didn't reveal such responses. This might be due to the inherently low levels of available P in soils, which can be seen by looking at wheat yield responses due to P, the differences between NP and N. As depicted in the tables and figures, the comparisons showed non-significant negative limits at 95% indicating that there was no wheat grain yield response to S from TSP (i.e., beyond 20 kg S/ha, which is the amount of S supplied as mineral gypsum). If a site is responsive to S from gypsum, but not to P from TSP, then any yield increments beyond 20 kg S/ha can be regarded as the response which has come due to S from TSP when keeping the other factors constant. The major reason is that responses of wheat to S that is expected from TSP might have been obscured due to the inherently low levels of P in soils (Table 3a and b). As supportive information, this can be seen by looking at the yield differences between NS_2 and NS_3 (r); NS_3 and NP (nr); and NP and NPS (r) in the x-axis of Figs. 1 through 10; and Tables 4 through 8. The observed difference in the first two, and last two; and the lack of response between the second two treatments may suggest that the amount of S from TSP (2–6 kg S/ha) also might not be sufficient enough to bring the intended yield difference as it was seen from lower treatment levels, for example (at 5 kg S/ha).

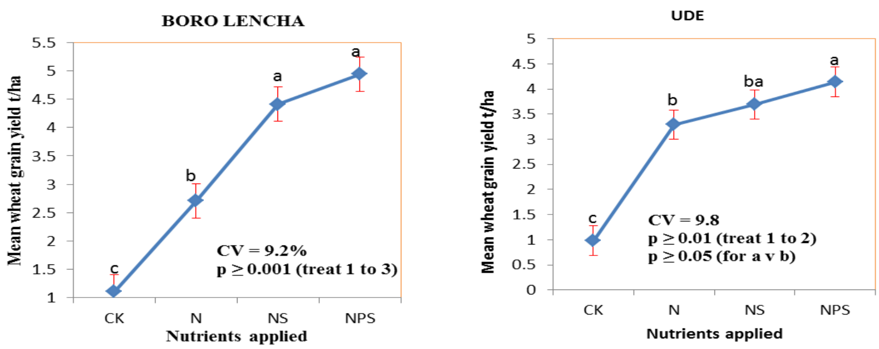
Though, the responses of wheat to S from TSP were not expressed statistically, there are yield increments between NS_3 and NP (nr) treatments for data that may or may not be significant. Such, yield increments, however, can be expressed in the quality attributes of wheat or other crop plants. So this shouldn't be overlooked as it has become evident from the overall progressive yield increments at any given treatment level. For example, at GS_2 there was wheat yield response due to N and S at all levels, except for the S that might be expected from TSP, (the yield difference between NPS_3-NS_3). But, it is noted that yield increments at this stage can also be attributable to P as there was P response in this site, which is obtained as the treatment difference between N and NP. As the initial soils tested low in P, the suggested amount of S in TSP, 2–6% might also be not adequate enough to bring the intended significant yield change. This reason can be noticeable from the differences between lower treatments (N and NS_1) as there was no yield response due to S from gypsum, at 5 kg S/ha; but, the S treatment levels above 5 kg/ha had significant yield increments throughout at GS_2 site suggesting, the existence of better positive synergy between N, N and/or P. However, it should be worth mentioning that though not significant statistically, this can be significant enough to be expressed in the quality attributes of the harvested produce. Therefore, it should be noted that if one accounts for the nutrient concentration in the form of impurities in such concentered fertilizers it will enable us to avoid unnecessary fertilizer bending and at the same time will bring the agricultural practices something like 25-50% organic, if such materials could be blended with the locally available materials like gypsum, which naturally contains 13 to 18.5% by weight SO_4-S.

2.3. Figures, Tables and Schemes



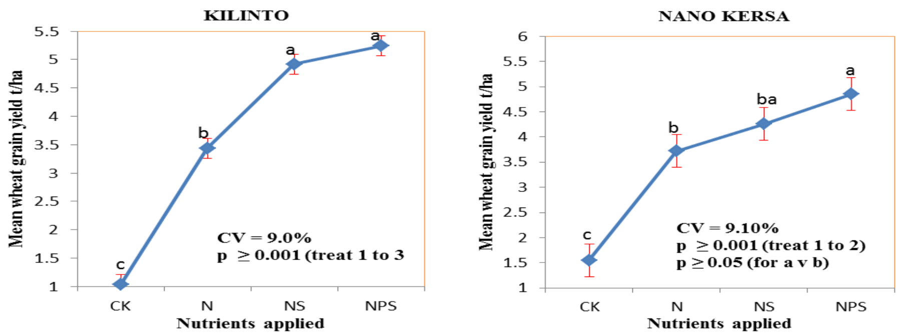
Figures 1 and 2: Wheat grain yield at AA1 and Do1 (Arsi zone) in response to the applied S, N and P (season-I)

*Means bearing the same letter(s) on bars within a field-site are not significantly different at (**$p \leq 0.05$) probability level analyzed by t-test.* ***Key:*** *highly significant at (***$p \leq 0.001$); significant at (**$p \leq 0.01$); marginally significant at (*$p \leq 0.05$); and ns = not significant otherwise. Percentage grain yield increase due to S from TSP at: AA_1 = 1.5%; Do_1 = 20.0%. The Error bars show the standard error of mean for each treatment.*



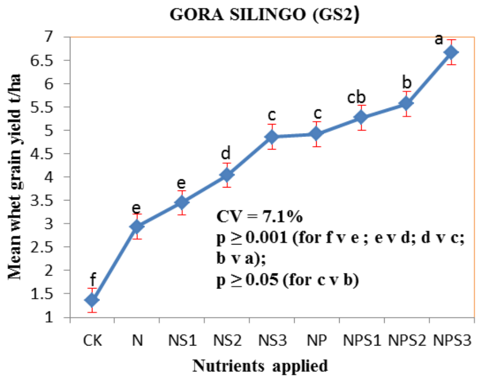
Figures 3 and 4: Wheat grain yield response at BL_1 and Ud_1 to applied S, N, and P (season-I).

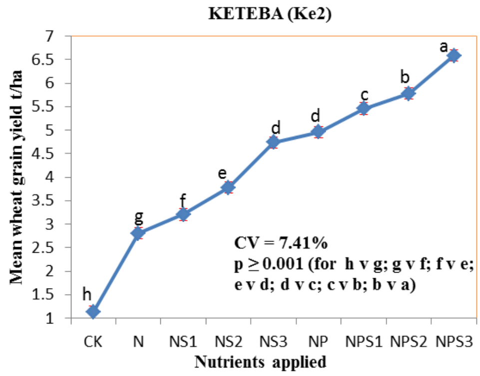
Means bearing the same letter(s) on bars within a field/site are not significantly different at ($p \leq 0.05$) probability level analyzed by t-test.*



Figures 5 and 6: Wheat grain yield response at Ki and NK/DL to S, N, and P (season-I).

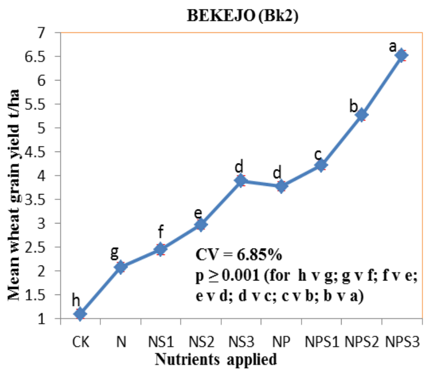
Means bearing the same letter(s) on bars within a field/site are not significantly different at (*$p \leq 0.05$) probability level analyzed by t-test. Percentage grain yield increase due to S from TSP at: BL_1 = 12.0%, Ud_1 = 12.2% and Ki_1 = 6.5%; NK_1 = 13.8%, and/or similar DL_1 = 13.9%.

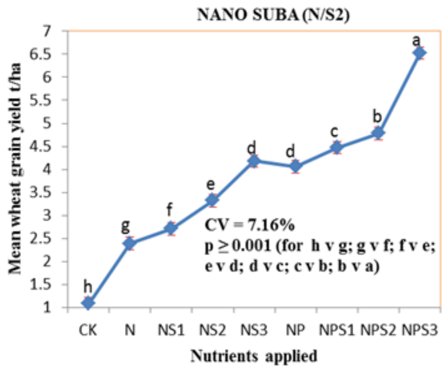




Figures 7 and 8: Wheat grain yield at GS_2 and Ke_2 sites in response to N, S and P nutrition (season-II)

Means bearing the same letter(s) within a group are not significantly different statistically at the probability level analyzed by t-test. Percentage grain yield increase due to S from TSP at: GS_2 = 1.23%; and Ke_2 = 4.64%.





Figures 9 and 10: Wheat grain yield response to applied N, S and P nutrients at Bk_2 and N/S_2 sites (season-II). Means bearing the same letter(s) within a group are not significantly different statistically at the probability level analyzed by t-test. Percentage grain yield increase due to S from TSP at: Bk_2 = -3.08%; N/S_2 = -2.87%.

2.4. Tables

Table 1. Locations of the selected study sites in the central highlands of Ethiopia.

	District	Farmer field/site	Latitude (N)		Longitude (E)		Altitude	Soil
Location/Zone			Degree	mm.mm	Degree	mm.mm	(m)	
Arsi (Ar)	Tiyo	Abosara Alko1 (AA1)	7	49.454	39	1.661	2297.02	TC
Arsi (Ar)	Tiyo	Dosha1 (Do1)	7	53.813	39	6.176	2418.32	Ult
Arsi (Ar)	Hitossa	Boro Lencha1 (BL1)	8	7.476	39	17.722	2186.37	An
East Shewa (ES)	Akaki	Kilinto (Ki)	8	54.099	38	49.133	2204.00	TP
West Shewa (WS)	Welmera	Nano Kersa1 (NK1)	8	55.605	38	31.062	2123.74	TC

West Shewa (WS)	Welmera	Dawa Lafto1 (DL1)	8	59.147	38	26.92	2173.60	Ult

TP = Typic Pellusterts (are heavy black clays or Vertisols); and TC = Typic Chromosterts (are light black clays or Vertisols); Ult = Ultisols; An = Andosols.

Table 2. Extraction or Analytical Method Used for the Studied Soils.

Variables considered	Unit(s) of measurement	Extraction/analytical method by:	References
pH	na	Potentiometrically,1:2.5 soil:water solution	[18]
Total Exchangeable Acidity (H^+ & Al^{3+})	$cmol_c/kg$	1.0M KCl and titration by 0.01M NaOH (@ pH:7.0)	[19]
Electrical Conductivity (EC)	mS/cm	1:5 soil:water suspension	[20]
Exchangeable Bases (Na^+ & K^+)	$cmol_c/kg$	1*M* NH4OAc solution, pH =7.00	[21]
Exchangeable Bases (Ca^{2+} & Mg^{2+})	$cmol_c/kg$	1*M* NH4OAc solution, pH =7.00	[22]
Cation Exchangeable Capacity (CEC)	$cmol_c/kg$	1*M* NH4OAc solution, pH =7.00	[22]
Saturation percent (SP)	%	Calculation from exch. bases	[22]
Calcium Saturation Percent (Ca-SP)	%	Calculated from exch. Ca^{2+}	[22]
Cation exchange capacity (CEC)	$cmol_c/kg$	1 *M* NH_4OAc solution at pH =7.00	[22]
Exchangeable Al^{3+}	$cmol_c/kg$	The difference between exch. acidity and H^+	[23]
Total nitrogen (TN)	%	Kjeldahl as described in	[24]
Organic carbon (OC)	%	Walkley-Black as described in	[25]
Available P	mg/kg	Bray-I, (pH < 7.00), for soils that came from Ar/WS.	[26]
Available P	mg/kg	Olsen, (pH > 7.00), for soils that came from ES.	[27]
Sulfate sulfur (SO_4-S)	mg/kg	Calcium phosphate, turbidimetric method	[21]
Soil texture	na	Hydrometer method	[28]

Key: Saturation percent (SP); total nitrogen (TN); organic carbon (OC); plant available phosphorus (P); plant available sulfate sulfur (SO_4-S); Electrical conductivity (EC); Exchangeable (Exch.); and Cation exchange capacity (CEC); na = not applicable.

Table 3. Selected Physico-chemical variables of soils of the study sites before planting.

Study Area/Zone	Farmer field /site	pH Soil:H_2O	EC (mS/cm)	Exchangeable Base Cations (cmol$_c$/kg) Ca^{2+}	Mg^{2+}	Na$^+$	K$^+$	CEC (cmol$_c$/kg)	SP (%)	TN (%)	OC (%)	Av.P (mg/kg)	SO_4-S (mg/kg)	Texture
				Season-I										
Ar	Abosara Alko (AA_1)	6.00	0.10	10.74	2.70	0.04	1.56	23.8	63.20	0.13	1.11	5.12	6.94	SC
Ar	Dosha (Do_1)	5.30	0.10	7.55	1.44	0.23	1.10	24.3	42.48	0.25	2.04	1.84	10.44	C
Ar	Boru Lencha (BL_1)	7.00	0.07	13.94	4.62	0.27	1.78	29.8	69.19	0.11	1.07	3.29	4.32	SC
ES	Ude (Ud_1)	7.10	0.06	26.10	6.06	0.29	3.32	39.4	90.80	0.10	1.23	9.53	12.37	C
ES	Kilinto (Ki_1)	8.00	0.24	32.48	8.53	0.32	4.18	47.8	95.23	0.06	1.39	8.17	8.27	C
OL	Nano Kersa (NK_1)	6.70	0.07	11.45	3.85	0.29	2.09	26.4	66.98	0.07	1.41	0.22	11.89	C
OL	Dawa Lafto (DL_1)	5.9	0.05	5.96	1.39	0.30	2.19	18.6	52.91	0.14	1.71	0.28	10.83	CL
				Season-II										
Ar	Gora Silingo (GS_2)	6.24	0.11	8.79	4.20	0.34	4.14	26.8	65.24	0.17	2.18	3.01	12.11	CL
ES	Bekejo (Bk_2)	7.15	0.10	19.72	5.22	0.34	2.50	33.4	83.19	0.08	1.17	12.01	4.03	SC

WS	Nano Suba (NS_2)	5.85	0.07	4.01	1.27	0.24	2.09	13.8	55.16	0.14	0.96	0.89	4.58	C
WS	Berfeta Tokofa (BT_2)	4.85	0.21	7.73	2.89	0.44	2.50	36.2	37.45	0.15	2.03	0.50	35.83	C

Key:- Study areas/zones/locations: Arsi (Ar), East Shewa (ES), West Shewa (WS); Districts: Tiyo (Ti), Hitossa (Hi), Ada'a (Ad) and Welmera (We); Soil types: indicated Table 1 above; and Soil texture: Sandy clay loam (SCL), Clay (C), Sandy clay (SC), and Clay loam(CL); Study sites: Abosara Alko$_1$ (AA_1), Dosha$_1$ (Do_1), Boro Lencha$_1$ (BL_1), Ude$_1$ (Ud_1), Kilinto$_1$ (Ki_1), Nano Kersa$_1$ (NK_1), Dawa Lafto$_1$ (DL_1), Gora Silingo$_2$ (GS_2), Bekejo$_2$ (Bk_2), Nano Suba$_2$ (NS_2), and Berfeta Tokoffa$_2$ (BT_2). Subscripts 1 and 2 indicate the two cropping seasons-I and II.

Table 4. Orthogonal comparisons among treatments in Arsi zone, at A/Alko, Dosha and G/Silingo sites (Season-I).

	A/Alko site				Dosha site				B/Lencha site			
Treatment comparisons	**DBM**	**95 % CL**			**DBM**	**95 % CL**			**DBM**	**95 % CL**		
NPS-NS	0.09	-0.75	0.93	Ns	0.92	-0.01	1.84	ns	0.55	-0.07	1.14	ns
NPS-N	2.04	1.20	2.88	***	1.35	0.43	2.27	***	2.24	1.63	2.84	***
NPS-CK	4.73	3.89	5.57	***	3.63	2.71	4.56	***	3.83	3.23	4.44	***
NS-N	1.95	1.11	2.79	***	0.43	-0.49	1.36	ns	1.70	1.10	2.30	***
NS-CK	4.65	3.81	5.49	***	2.72	1.79	3.64	***	3.30	2.69	3.90	***
N-CK	2.69	1.85	3.53	***	2.28	1.36	3.21	***	1.60	0.99	2.20	***

Key: DBM = difference between means; CL = confidence limits; N = nitrogen; P = phosphorus; S = sulfur and; CK = Check (without any fertilize); ns = not significant at 95% CL. Comparisons significant at 0.001 levels are indicated by ***. Percentage grain yield increase due to S from TSP at: AA_1 = 1.5%; Do_1 = 20.0%; BL_1 = 12.0%.

Table 5. Orthogonal comparisons among treatments in E/Shewa zone, C/Donsa, Keteba and Ude sites (Season-I).

	Ude site				Kilinto site				N/Kersa site			
Treatment comparisons	**DBM**	**95 % CL**			**DBM**	**95 % CL**			**DBM**	**95 % CL**		
NPS-NS	0.44	-0.15	1.04	Ns	0.32	-0.02	0.67	ns	0.59	-0.06	1.25	ns
NPS-N	0.84	0.25	1.44	***	1.80	1.46	2.15	***	1.13	0.48	1.79	***
NPS-CK	3.15	2.56	3.75	***	4.21	3.87	4.56	***	3.30	2.65	3.96	***
NS-N	0.40	-0.19	0.99	Ns	1.48	1.13	1.83	***	0.54	-0.11	1.19	ns
NS-CK	2.71	2.12	3.30	***	3.89	3.54	4.24	***	2.71	2.06	3.36	***
N-CK	2.31	1.72	2.90	***	2.41	2.06	2.76	***	2.17	1.52	2.82	***

Key: DBM = difference between means; CL = confidence limits; N = nitrogen; P = phosphorus; S = sulfur and; CK = Check (without any fertilize); ns = not significant at 95% CL. Comparisons significant at 0.001 levels are indicated by ***. Percentage grain yield increase due to S from TSP at: Ud_1 = 12.2% and Ki_1 = 6.5%; NK_1 = 13.8%; and/or similar DL_1 = 13.9%.

Table 6. Orthogonal comparisons among treatments for wheat gain yield, at the Gora Silingo site in the Arsi zone (Season-II).

	G/Silingo site			
Treatment Comparisons	**DBM**	**95% CL**		
NPS_3 - NPS_2	1.0933	0.5639	1.6228	***
NPS_3 - NPS_1	1.3933	0.8639	1.9228	***
NPS_3 – NP	1.7433	1.2139	2.2728	***
NPS_3 - NS_3	1.8100	1.2806	2.3394	***
NPS_3 - NS_2	2.6267	2.0972	3.1561	***
NPS_3 - NS_1	3.2200	2.6906	3.7494	***
NPS_3 – N	3.7233	3.1939	4.2528	***
NPS_3 – CK	5.3033	4.7739	5.8328	***
NPS_2 - NPS_1	0.3000	-0.2294	0.8294	ns

NPS_2 – NP	0.6500	0.1206	1.1794	***
NPS_2 - NS_3	0.7167	0.1872	1.2461	***
NPS_2 - NS_2	1.5333	1.0039	2.0628	***
NPS_2 - NS_1	2.1267	1.5972	2.6561	***
NPS_2 – N	2.6300	2.1006	3.1594	***
NPS_2 – CK	4.2100	3.6806	4.7394	***
NPS_1 – NP	0.3500	-0.1794	0.8794	ns
NPS_1 - NS_3	0.4167	-0.1128	0.9461	ns
NPS_1 - NS_2	1.2333	0.7039	1.7628	***
NPS_1 - NS_1	1.8267	1.2972	2.3561	***
NPS_1 – N	2.3300	1.8006	2.8594	***
NPS_1 – CK	3.9100	3.3806	4.4394	***
NP - NS_3	0.0667	-0.4628	0.5961	ns
NP - NS_2	0.8833	0.3539	1.4128	***
NP - NS_1	1.4767	0.9472	2.0061	***
NP – N	1.9800	1.4506	2.5094	***
NP – CK	3.5600	3.0306	4.0894	***
NS_3 – NP	-0.0667	-0.5961	0.4628	ns
NS_3 - NS_2	0.8167	0.2872	1.3461	***
NS_3 - NS_1	1.4100	0.8806	1.9394	***
NS_3 – N	1.9133	1.3839	2.4428	***
NS_3 – CK	3.4933	2.9639	4.0228	***
NS_2 - NS_1	0.5933	0.0639	1.1228	***
NS_2 – N	1.0967	0.5672	1.6261	***
NS_2 – CK	2.6767	2.1472	3.2061	***
NS_1 – N	0.5033	-0.0261	1.0328	ns
NS_1 – CK	2.0833	1.5539	2.6128	***
N – CK	1.5800	1.0506	2.1094	***

Key: Trt = treatment; DBM = difference between means; CL = confidence Limits; N = nitrogen; P = phosphorus; S = Sulfur and; CK = Check; ns = not significant. The treatments used were four levels of S (S_0 = 0 = CK, S_1 = 5, S_2 = 10 and S_3 = 20 kg S/ha); two levels of N (N_0 = 0 = CK, N = 69 kg N/ha); and two levels of P (P_0 = 0 = CK and P = 20 kg P/ha; CK means check/control, treatment without any fertilizer). Comparisons significant at the 0.001 level are indicated by ***. r = response, nr = no response.

Table 7. Orthogonal comparisons among treatments for wheat gain yield, at the Bekejo site in the E/Shewa zone (Season-II).

	Bekejo site			
Treatment comparisons	**DBM**	**95% CL**		
NPS_3 - NPS_2	1.25000	1.04998	1.45002	***
NPS_3 - NPS_1	2.29333	2.09331	2.49336	***
NPS_3 - NS_3	2.61667	2.41664	2.81669	***
NPS_3 – NP	2.73667	2.53664	2.93669	***
NPS_3 - NS_2	3.53667	3.33664	3.73669	***
NPS_3 - NS_1	4.05667	3.85664	4.25669	***
NPS_3 – N	4.43333	4.23331	4.63336	***

NPS_3 – CK	5.40667	5.20664	5.60669	***
NPS_2 - NPS_1	1.04333	0.84331	1.24336	***
NPS_2 - NS_3	1.36667	1.16664	1.56669	***
NPS_2 – NP	1.48667	1.28664	1.68669	***
NPS_2 - NS_2	2.28667	2.08664	2.48669	***
NPS_2 - NS_1	2.80667	2.60664	3.00669	***
NPS_2 – N	3.18333	2.98331	3.38336	***
NPS_2 – CK	4.15667	3.95664	4.35669	***
NPS_1 - NS_3	0.32333	0.12331	0.52336	***
NPS_1 – NP	0.44333	0.24331	0.64336	***
NPS_1 - NS_2	1.24333	1.04331	1.44336	***
NPS_1 - NS_1	1.76333	1.56331	1.96336	***
NPS_1 – N	2.14000	1.93998	2.34002	***
NPS_1 – CK	3.11333	2.91331	3.31336	***
NS_3 – NP	0.12000	-0.08002	0.32002	ns
NS_3 - NS_2	0.92000	0.71998	1.12002	***
NS_3 - NS_1	1.44000	1.23998	1.64002	***
NS_3 – N	1.81667	1.61664	2.01669	***
NS_3 – CK	2.79000	2.58998	2.99002	***
NP - NS_3	-0.12000	-0.32002	0.08002	ns
NP - NS_2	0.80000	0.59998	1.00002	***
NP - NS_1	1.32000	1.11998	1.52002	***
NP – N	1.69667	1.49664	1.89669	***
NP – CK	2.67000	2.46998	2.87002	***
NS_2 - NS_1	0.52000	0.31998	0.72002	***
NS_2 – N	0.89667	0.69664	1.09669	***
NS_2 – CK	1.87000	1.66998	2.07002	***
NS_1 – N	0.37667	0.17664	0.57669	***
NS_1 – CK	1.35000	1.14998	1.55002	***
N – CK	0.97333	0.77331	1.17336	***

Key: Trt = treatment; DBM = difference between means; CL = confidence Limits; N = Nitrogen; P = phosphorus; S = Sulfur and; CK = Check; ns = not significant. The treatments used were four levels of S (S_0 = 0 = CK, S_1 = 5, S_2 = 10 and S_3 = 20 kg S/ha); two levels of N (N_0 = 0 = CK, N = 69 kg N/ha); and two levels of P (P_0 = 0 = CK and P = 20 kg P/ha; CK means check/control, treatment without any fertilizer). Comparisons significant at the 0.001 level are indicated by ***.

Table 8. Orthogonal comparisons among treatments for wheat gain yield, at the Nano Suba site in the O/Liyuu zone (Season-II).

	$(N/Suba)_2$ site	
Treatment combinations	**DBM**	**95 % CL**

NPS_3 - NPS_2	1.7433	1.4751	2.0116	***
NPS_3 - NPS_1	2.0533	1.7851	2.3216	***
NPS_3 - NS_3	2.3500	2.0817	2.6183	***
NPS_3 – NP	2.4667	2.1984	2.7349	***
NPS_3 - NS_2	3.2033	2.9351	3.4716	***
NPS_3 - NS_1	3.8133	3.5451	4.0816	***
NPS_3 – N	4.1333	3.8651	4.4016	***
NPS_3 – CK	5.4433	5.1751	5.7116	***
NPS_2 - NPS_1	0.3100	0.0417	0.5783	***
NPS_2 - NS_3	0.6067	0.3384	0.8749	***
NPS_2 – NP	0.7233	0.4551	0.9916	***
NPS_2 - NS_2	1.4600	1.1917	1.7283	***
NPS_2 - NS_1	2.0700	1.8017	2.3383	***
NPS_2 – N	2.3900	2.1217	2.6583	***
NPS_2 – CK	3.7000	3.4317	3.9683	***
NPS_1 - NS_3	0.2967	0.0284	0.5649	***
NPS_1 – NP	0.4133	0.1451	0.6816	***
NPS_1 - NS_2	1.1500	0.8817	1.4183	***
NPS_1 - NS_1	1.7600	1.4917	2.0283	***
NPS_1 – N	2.0800	1.8117	2.3483	***
NPS_1 – CK	3.3900	3.1217	3.6583	***
NS_3 – NP	0.1167	-0.1516	0.3849	ns
NS_3 - NS_2	0.8533	0.5851	1.1216	***
NS_3 - NS_1	1.4633	1.1951	1.7316	***
NS_3 – N	1.7833	1.5151	2.0516	***
NS_3 – CK	3.0933	2.8251	3.3616	***
NP - NS_3	-0.1167	-0.3849	0.1516	ns
NP - NS_2	0.7367	0.4684	1.0049	***
NP - NS_1	1.3467	1.0784	1.6149	***
NP – N	1.6667	1.3984	1.9349	***
NP – CK	2.9767	2.7084	3.2449	***
NS_2 - NS_1	0.6100	0.3417	0.8783	***
NS_2 – N	0.9300	0.6617	1.1983	***
NS_2 – CK	2.2400	1.9717	2.5083	***
NS_1 – N	0.3200	0.0517	0.5883	***
NS_1 – CK	1.6300	1.3617	1.8983	***
N – CK	1.3100	1.0417	1.5783	***

Key: - Trt = treatment; DBM = difference between means; CL = confidence Limits; N = nitrogen; P = phosphorus; S = Sulfur and; CK = Check; ns = not significant. The treatments used were four levels of S (S_0 = 0 = CK, S_1 = 5, S_2 = 10 and S_3 = 20 kg S/ha); two levels of N (N_0 = 0 = CK, N = 69 kg N/ha); and two levels of P (P_0 = 0 = CK and P = 20 kg P/ha; CK means check/control, treatment without any fertilizer). Comparisons significant at the 0.001 level are indicated by***

3. Materials and Methods

3.1. Site Description

The study was made in three locations: Arsi (Ar), East Shewa (ES) and West Shewa (WS) zones in central Ethiopian agricultural soils. The geology of Ar zone consists of pyroclastic rocks mainly tuffs and ignimbrites of the recent volcanic eruptions. The upper soil layer consists of tephritic materials, whereas the substratum consists of calcareous material enriched through secondary precipitation over bedrock [16]. The soils are dominantly Mollisols, and Ultisols, with considerable proportions of Vertisols. In ES, the dominant soil-types considered were Vertisols and majorities are calcareous with nodules of fragmented $CaCO_3$ occurring in the first 0–60 cm soil profile-depths. In WS, the major soil-types were Mollisols, Ultisols, Ultisols and some Vertisols also observed. The major agro-ecological zones (AEZs) of study districts extend from extremely cool highlands to mid-highlands, 61 and 39% respectively.

3.2. Experimental Materials, Treatments and Design

The purity of gypsum depends on trace materials like boron and heavy metals; and it may need some kind of soil-testing and treatments. So, samples were collected from six curie sites or their shops and analyzed for SO_4-S contents. In the analyzed samples the S contents ranged between 13.5–18.0%, from which the samples with 18.0% SO_4-S where chosen as experimental material. The use of such locally available materials is important both in terms of reducing cost of production, and at the same time encourage organic forming. A recently developed bread wheat variety known as "Kekeba" was used as a test-crop. In the process of investigation nine treatments containing four nutrients N, gypsum (S + Ca) and P were considered. Ca was more or less incidental.

Overall 24 extensive field experiments each of which represent a site or farmer were conducted in two seasons. These sites are geo-referenced using Global Positioning System (GPS) GARMIN model number GPS-60 assisted by Google Earth (2011) and were classified by elevation and soil type when known. Some additional information including the specific locations and salient features of the selected sites are presented in Table 1.

In season-I (2013/14), 18 on-farm experiments were conducted, six in each administrative zone or location covering wide range of agro-ecological zones (AEZs). In this season, four treatments: an absolute control (CK) = ($N_0P_0S_0$), N alone or N = N_1 = ($N_1P_0S_0$), nitrogen plus sulfur or NS = N_1S_1 = ($N_1P_0S_1$), and nitrogen plus phosphorus plus sulfur or NPS = ($N_1P_1S_1$) were tested. The evaluated nutrients were two levels of S (0 and 20 kg/ha), two levels of P (0 and 20 kg/ha); and two levels N (0 and 69 kg/ha).

In season-II (2015/16), another six experiments were conducted (two in each location). Three study sites: Gora Silingo$_2$ (GS2), Keteba$_2$ (Ke2), and Nano Suba$_2$ (NS2) were selected because they were found to be responsive to S supplied as gypsum. Whereas, Wonji Gora1/ (Dosha2) or (Do$_2$), Bekejo2 (Bk$_2$) and Berfeta Tokofa2 (BT$_2$) were selected randomly without pre-soil testing, but on areas some 0.5 to 1.5 miles away from the last season S responsive sites, namely Do$_1$, Bekejo1 (Bk$_1$) and Berfeta Tokofa1 (BT$_1$) respectively. In this set of experiment, nine treatments: CK (without any fertilizer) = ($N_0P_0S_0$); nitrogen alone or N = N_1 = ($N_1P_0S_0$); nitrogen and sulfur or NS$_1$ = ($N_1P_0S_1$); nitrogen and sulfur or NS$_2$ = ($N_1P_0S_2$); nitrogen and sulfur or NS$_3$ = ($N_1P_0S_3$); nitrogen and phosphorus or NP = N_1P_1 = ($N_1P_1S_0$); nitrogen, phosphorus and sulfur or NPS$_1$ = ($N_1P_1S_1$); nitrogen, phosphorus and sulfur or NPS$_2$ = ($N_1P_1S_2$); and nitrogen, phosphorus and sulfur or NPS$_3$ = ($N_1P_1S_3$) were tested in three locations. The nutrient levels used were four levels of sulfur (S) (S_0 = 0 = CK, S_1 = 5, S_2 = 10 and S_3 = 20 kg S/ha); two levels of nitrogen (N) (N_0 = 0 = CK, N = 69 kg N/ha); and two levels of phosphorus (P) (P_0 = 0 = CK and P_1 = 20 kg P/ha). In both seasons, randomized complete block (RCBD) was used as experimental design, and this was replicated three times. Each replicate was sub-divided into 3 x 5 (15m^2) experimental units/plots. The agronomic spacing for wheat between rows and plants (25cm x 5cm) was used; and in total there were 12 rows of wheat per plot in both sets of experiments. There were two border rows in each side, and another one row was used for plant tissue sampling. The central rows (4 x 1.5 = 6m²) were used for yield data and plant sampling. Nitrogen was split applied. One third of N source was incorporated into soils before seeding, and the remaining 2/3 was top-dressed at the stage of tillering. Whereas, the entire sources of S and P were incorporated into soils just before seeding. Nitrogen supplied as urea (46% N); SP as TSP (2-6% S; and 46% P_2O_5); and another form of S as gypsum (18.0% S, and it also contains approximately 20-23% Ca). A wheat cultivar, namely "Kekeba" was used as a test-crop. Harvesting was commenced when the grain and stover samples were reached average grain moisture content of about 13.5%. In this paper out of the many agronomic data collected only grain yield data was considered.

3.3. Soil Sampling, Preparation and Analysis

Totally, 24 soil samples were collected in two seasons or sets. The first set, 18 six surface samples (0–20cm depth) were collected before planting each of the 18 six gypsum (S + Ca) response experiments in season(s)-I. Whereas the second set, six soils samples were collected before planting each of the six rate experiments in season(s)-II. In doing so, before planting, surface soil samples representing each block were taken from 10-spots (0–20cm) using auger and bulked together in preparations for drying and analysis. Then the soil samples representing each block were further composted to make one sample per farmer field or per experiment. Soil samples then were air dried immediately in dry-rooms to avoid sulfate formation from organic matter (OM) in the transit. The dried samples were ground and sieved to pass through < 2 mm sieve, and analyzed for the variables as indicated in Tables 3a and b as per the methods shown in Table 2. The gypsum samples were collected from the different curie-sites and/or shops and analyzed for the S contents. The contents of S in the analyzed samples ranged between 13-18 %. But we used the sampled with 18% S for this particular experiment.

3.4. Yield Data Analysis

Yield and yield components data were analyzed using SAS version-9 [17]. PROC UNIVARIATE in SAS-GLM was used to test normality assumptions to evaluated grain yield variable besides analyzing residual distributions. The analysis of variance (ANOVA) was done using PROC-MIXED in SAS protocols to evaluate treatment differences. The SAS linear model statement for RCBD considers replications and treatments as fixed effects. When differences between treatments were significant least significant difference (LSD) was used to separate the means at the significant levels of 5%, 1% or 0.1%. More specifically, pre-planned pair-wise orthogonal comparisons among treatments using SAS contrast statements were made to determine the significance of treatments at each level and to determine the effect of applied S as an impurity from TSP (S-TSP), together with N-urea and P-TSP on wheat yield.

4. Conclusions and Recommendations

From the results of the study (the nutrient response curves), in all studied sites (100%) wheat showed highly significant response to N as depicted by sharp-rises in yield graphs indicating that N is the most limiting element in wheat or other crops production followed by S and/or P. However, statistically there was no wheat response to S impurity supplied in the form of TSP, beyond 20 kg S/ha (maximum amount of S supplies as mineral gypsum). However, though not statistically significant, in eight 8 of the 10 target sites, there was always grain yield increments by certain percent (indicated in the captions of the figures) due to S impurity from TSP. Indeed, such percent yield increments can be big enough to be expressed in quality attributes of harvested produce, if measured precisely. Such concentration of nutrients in concentrated compound fertilizers like TSP/DAP can be vital in varietal specific nutrient requirement or micro-dosing studies and/or in categorizing soils into fertility gradients and fertilizer recommendation domains. It should be noted that such small amount of S is important to sustain crop production in precision-farming as smallholding farmers can afford 5–10 kg bag of fertilizers. In summary, the importance of S in materials like TSP in plant nutrition, the results should be further investigated; and/or verified through optimized conditions on different wheat cultivars.

Author Contributions: This work was carried out in collaboration between both authors. Author AM designed the study, performed field experiments, managed laboratory/statistical analysis, and wrote the first draft of the manuscript. Author JH, wrote the protocol, reviewed and edited the manuscript and managed the literature searches. Both authors read and approved the final manuscript.

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