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Suitability of different Extractants and Turbidimetric Reagents in the Quantitative Determination of Sulfate-Sulfur in Soils of South-Western Cameroon

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ABSTRACT

Routine soil characterisation in this active volcanic region is usually carried out without considering the sulfur (S) content. Considering the role of this element as a plant nutrient and as a danger to environmental and human health if in excess, there is need to have an extractant that could be used on a routine basis so as to reduce its content. Ten soil samples, from different locations in South-western Cameroon, were extracted to determine sulfate (SO_4^{2-}) by water (H_2O), calcium chloride ($CaCl_2$), calcium dihydrogen phosphate monohydrate ($Ca(H_2PO_4)_2.H_2O$), potassium dihydrogen phosphate (KH_2PO_4), and Morgan's solution (sodium acetate (CH_3COONa) and acetic acid (CH_3COOH)) methods. The SO_4^{2-} ion content of these extracts was determined by turbidimetry using barium chloride dihydrate ($BaCl_2.2H_2O$) in gelatine or in Tween 80. Correlation analyses showed that all the methods were highly correlated ($P<0.001$) with one another. Due to the ease of filtration, $Ca(H_2PO_4).H_2O$ has been recommended for the extraction of SO_4^{2-}-S in soils of this region. Barium chloride/Tween 80 reagent has also been recommended for quantitative analysis of SO_4^{2-}-S due to the presence of excess $BaCl_2$ for the formation of $BaSO_4$.

Key words: Sulfate, Extractants, Turbidimetric reagents, Soils

RÉSUMÉ

La charactérisation courante du sol dans cette région volcanique active est normallement faite sans tenir compte du niveau disponible de souffre. Eu égand au role de cet élément en tant qu'élément nutritive des plantes, et aussi un danger á la santé humaine et á l'environnement si en excès, il foudrait y avoir un extractant qui pourrait être utilize habituellement. Dix échantillons de sol de differentes emplacements dans le Sud Ouest du Cameroun ont été extraits pour determiner le sulfate (SO_4^{2-}) par les methods de l'eau (H_2O), du chlorure de calcium ($CaCl_2$), du phosphate dihydrogène de calcium monohydrate ($Ca(H_2PO_4)_2.H_2O$), du phosphate dihydrogène de potassium (KH_2PO_4) et de la solution de Morgan (acetate de sodium (CH_3COONa) et l'acide acétique (CH_3COOH)). Le SO_4^{2-} de ces extraits a été quantifié par turbimétrie en utilisant le barium de chlorure ($BaCl_2.2H_2O$) dans la gélatine ou $BaCl_2.2H_2O$ dans le Tween 80. L'analyse correlative a montré que toutes les methods étaient profondément liées les unes aux autres ($P<0.001$). En considerant la facilité de la filtration, $Ca(H_2PO_4)_2.H_2O$ était recommandé pour l'extraction de SO_4^{2-} dans les sol de cette prouvée quelque peu meilleure par rapport aux autres methods d'estimation du SO_4^{2-} dans les sols de cette sous-region. Le reactif de Chloure de Barium/Tween 80 était aussi recommandé pour les analyses quantitatives SO_4^{2-}-S grace à la presence en excés du $BaCl_2$ pour la formation de $BaSO_4$.

Mots clés: Sulfate, Extractants, Reactifs turbidimétriques, Sols

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INTRODUCTION

Sulfur, which is an essential element in plants, is a constituent of some amino acids and is involved in the synthesis of proteins (Mengel and Kirkby, 1982; Ewald, 1997). Many of the substances (eg glutenin and aliins), which are responsible for particular quality aspects of plant product are sulfur-containing compounds. Sulfur is the plant nutrient, which has perhaps the strongest impact on plant quality (Ewald, 1997). The baking qualities of wheat flour are directly related to the S status of the wheat (Ewald, 1997; Pederson et al., 1998). Plants absorb much of the S they need in the form of sulfate (SO_4^{2-}-S) from the soils (Chang and Thomas, 1963; Howard and Reisenauer, 1966; Sanchez, 1976; Brady, 1984). Deficiency of SO_4^{2-}-S in the soil leads to S deficiencies in plants. Sulfur deficiency in plants lowers the S containing amino acids (cysteine and methionine), which are essential building blocks of protein. Sulfur deficiencies have been reported in sub-saharan Africa and the sandy soils of central Africa (Kang et al., 1981; Acquaye and Kang, 1987). An insufficient supply of S can lower the utilization of nitrogen (N) from mineral and organic fertilizers leading to increased leaching of N (Haneklaus et al., 2000).

Due to its high solubility and hence less adsorption unto soil colloids (Kang et al., 1981), excess SO_4^{2-}-S may become an environmental pollutant. Sulfate-sulfur could be readily leached from the soils, especially in humid regions (Brady, 1984), into underground waters, which feed the aquifers of domestic water supply in this region.

Studies relating to the availability of soil S for plant growth are dependent on accurate methods for the extraction of small amounts of SO_4^{2-}-S, especially when most of the soil sulfur is in unavailable organic forms (Walker and Adams, 1958; Nelson, 1964; Robert, 1966; Freney and Stevenson, 1966). Many extractants for SO_4^{2-}-S and many methods for the determination of SO_4^{2-}-S in the extracts have been proposed and used (Chesnin and Yien, 1950; Bartlett and Neller, 1960; Ensminger and Freney, 1966; Barrow, 1967; Jones et al., 1972). Unfortunately, these extractants and methods were developed for temperate soils. There is need for them to be tested on tropical soils such as those of South-western Cameroon. The latter is an active volcanic region producing sulfur gases such as sulfur dioxide (SO_2) and hydrogen sulfide (H_2S). These gases are brought to the earth as SO_4^{2-} by mainly wet deposition (Brady, 1984). Determination of sulfur (S) in the form of soil sulfate (SO_4^{2-}-S) in routine soil analysis in South-western Cameroon is imperative considering the role of this element in plant nutrition and environmental pollution. The SO_4^{2-}-S determination requires a readily available extractant that combines efficiency of extraction with simplicity of the technique.

The objectives of this study therefore are to compare different methods of extracting SO_4^{2-}-S and to assess the suitability of different turbidimetric reagents for the quantitative determination of SO_4^{2-}-S from these extracts.

Table 1. Description of sampling sites.

Soil no.	Sampling depth (cm)	Site	Location	Land-use	Geology	Parent material	Classification (USDA Soil Taxonomy)
1	0 – 25	Molyko	4°7'N,9°15'E	Banana (*Musa* spp)	Young volcanic	Basalt	Andisol
2	0 – 25	Penja	4°37'N,9°39'E	Banana (*Musa* spp)	Young volcanic	Basalt	Andisol
3	40 – 50	Penja	4°37'N,9°39'E	Banana (*Musa* spp)	Young volcanic	Basalt	Andisol
4	0 – 25	Mundemba	5°9'N,8°49'E	Korup Forest Park	Basement complex	Granite	Ultisol
5	0 – 25	Ekona Research	4°19'N,9°18'E	Okra (*Abelmoschus caeil*)	Young volcanic	Basalt	Andisol
6	0 – 25	Obang Mamfe	5°37'N,9°21'E	Cassava (*Manihot*)	Sedimentary	Alluvium	Ultisol
7	30 – 50	Obang Mamfe	5°37'N,9°21'E	Cocoa (*Theobroma cacao*)	Sedimentary	Alluvium	Ultisol
8	30 – 50	Barombi Kang	4°35'N,9°26'E	Cocoyam (*colocasia*)	Old volcanic	Basalt	Ultisol
9	55 – 100	Southern Bakundu	5°6'N,9°25'E	Forest	Old volcanic	Basalt	Ultisol
10	100 – 140	Mbanga	4°31'N,9°33'E	Banana (*Musa* spp)	Old volcanic	Basalt	Ultisol

EXPERIMENTAL

Ten soil samples were collected from different locations in South-western Cameroon (latitudes 4°00'N and 5°47'N; longitudes 8°50'E and 9°42'E). Sampling was carried out on both uncultivated (forest) and cultivated sites (plantation and food crops). The location of the sampling sites is shown in Table 1. The soil samples were collected at depths less than 30 cm from the surface for top soils and greater than 30 cm from the surface for sub soils.

Each sample was air-dried, ground, and sieved using a 2-mm sieve. The soil pH was determined potentiometrically both in distilled water and in one normal potassium chloride (1N KCl) solution using a soil to extraction solution ratio of 1:2.5 (w/v) (IITA, 1979). Available phosphorus (P) was determined using the Bray II method (Bray and Kurtz, 1945), while total nitrogen (N) and organic carbon (C) contents were determined by the Kjeldahl and Walkley-Black wet oxidation methods, respectively (Jackson, 1958). Exchangeable sodium (Na), potassium (K), calcium (Ca) and magnesium (Mg) were extracted using one normal ammonium acetate (*1N* NH_4OAc) buffer, pH 7 in ratio 1:10 (w/v) (Jackson, 1958). Exchangeable acidity was determined by titrating against *1N* KCl as the extractant (McLean, 1965). Effective cation exchange capacity (ECEC) was calculated as the sum of the exchangeable bases (Ca, Mg, K and Na) and exchangeable acidity (Jackson, 1958).

$$ECEC = Ca + Mg + K + Na + Exch.\ acidity$$

Water-soluble SO_4^{2-}-S was extracted with distilled water in 1:5 soil to water suspension (Freney, 1958). The 0.15% $CaCl_2$-extractable SO_4^{2-}-S was estimated using an extraction ratio of 1:5 soil to salt solution (Ensminger and Freney, 1966). Calcium dihydrogen phosphate monohydrate-extractable SO_4^{2-}--S was achieved by using 1:5 soil to salt (0.01M $Ca(H_2PO_4)_2.H_2O$) solution (Fox et al., 1964). Potassium dihydrogen phosphate-extractable SO_4^{2-}-S was realised by preparing a KH_2PO_4 solution containing 500 mg kg^{-1} P at pH 6.5 and using the extractant ratio of 1:5 soil to salt solution (Fox et al., 1964). Morgan-extractable SO_4^{2-}-S was determined by adjusting the pH of the Morgan's solution to 4.8 and using a 1:5 soil to salt solution ratio (Howard and Reisenauer, 1966).

The sulfate ion content in the soil extracts was determined by turbidimetry. The two turbidimetric reagents used were $BaCl_2.2H_2O$ in Gelatine and $BaCl_2.2H_2O$ in Tween 80 (Chesnin and Yien, 1950; Bartlett and Neller, 1960).

RESULTS AND DISCUSSION

Some analytical results of the soils are shown on Table 2. All the soils studied were acidic, having a soil pH ranging from 4.3 to 5.7 and 3.7 to 4.8 in water and *1N* KCl, respectively. Based on the critical range of available P (10-15 mg P kg^{-1}) set by Tchuenteu (1994) for volcanic soils, available P was generally high in most of the soils, ranging from 8 to 78 mg P kg^{-1} soil. The high values of available P could partly be due to the residual levels from previous P fertilization. Total nitrogen was generally low in all the soils with a range of 0.09 % to 0.30 %. All the soils have varying amounts of organic carbon, exchangeable bases, exchangeable acidity and effective cation exchange capacity. The carbon-nitrogen

Table 2: Selected chemical properties of the soil used.

Soil	pH		Org. C	Tot. N	C/N	Avail. P	Exchangeable bases				Exch. acidity	ECEC
no.	H_2O	*1N* KCl					Ca	Mg	K	Na		
			%			mg kg^{-1}	cmol kg^{-1}					
1	4.3	3.7	2.32	0.22	11	78	6.97	2.93	2.78	0.30	0.22	13.20
2	5.6	4.7	2.90	0.30	10	59	6.20	2.25	1.49	0.16	0.16	10.26
3	5.2	4.3	0.58	0.11	5	26	3.10	0.90	0.84	0.11	0.14	5.09
4	4.9	4.0	1.74	0.15	12	16	3.10	0.90	0.22	0.03	1.66	5.91
5	5.5	4.4	1.74	0.17	10	32	6.20	2.14	0.82	0.09	0.24	9.49
6	5.0	3.9	2.03	0.12	17	14	5.42	1.35	0.12	0.02	0.48	7.39
7	4.8	4.0	1.74	0.13	13	8	3.87	1.13	0.14	0.02	0.70	5.86
8	5.7	4.7	1.16	0.11	11	22	4.65	1.24	0.10	0.02	0.08	6.09
9	5.7	4.8	1.16	0.09	13	11	3.87	1.01	0.22	0.02	1.08	6.20
10	5.4	4.4	0.58	0.11	5	25	3.10	1.13	0.14	0.02	0.68	5.07

Table 3. Sulfate content of soils as determined by two turbidimetric methods and five extractants.

Soil no.	Wat-G	Wat-T	Calchlo-G	Calchlo-T	Calpho-G	Calpho-T	Potpho-G	Potpho-T	Morgan-G	Morgan-T
					mg kg^{-1}					
1	89.0	192.5	76.3	160.3	173.3	374.0	207.8	420.5	256.0	521.3
2	35.3	75.0	18.8	40.5	53.0	108.0	61.0	123.0	73.5	153.3
3	19.8	42.3	14.3	35.0	86.0	171.3	106.8	216.8	126.0	262.5
4	5.8	11.5	4.0	9.8	8.5	18.5	9.3	18.8	11.8	25.3
5	4.5	10.5	3.5	8.5	9.3	18.3	13.3	28.3	14.0	25.5
6	2.0	4.8	2.5	5.0	4.5	10.5	9.5	23.5	10.3	24.5
7	4.5	10.0	4.5	4.0	8.0	17.3	10.3	21.0	11.3	23.3
8	10.5	22.8	9.0	20.3	18.5	37.5	28.5	58.3	30.3	64.3
9	1.5	4.3	3.3	7.8	4.5	11.5	8.3	17.3	10.0	23.3
10	70.8	140.8	65.8	140.0	125.5	265.0	144.5	290.5	156.8	313.3

Wat = H_2O; Calchlo = $CaCl_2$; Calpho = $Ca(H_2PO_4)_2.H_2O$; Potpho = KH_2PO_4; Morgan = CH_3COONa/CH_3COOH;
G = $BaCl_2.2H_2O$/Gelatin reagent; T = $BaCl_2.2H_2O$/Tween 80 reagent.

(C/N) ratio ranged from 5.27 to 16.92 with an average value of 10.63. This value is less than 32.00, implying that the soils are not rich in humus and hence immobilization of nitrogen will exceed mineralization, as noted by Olaitan et al. (1984).

With T80, the Morgan extractant gave the highest amount of SO_4^{2-} in all the soils while the H_2O extractant gave the lowest amount of SO_4^{2-} (Table 3). Low amounts of S were extracted from soils of food crop farms and forest while banana farm soils gave high amounts of S, irrespective of the extractant used. This could be due to the fact that food crop farms and forest receive little or no fertilizer while banana farms are accompanied by extensive use of agrochemicals such as single supper-phosphate (SSP) fertilizer, which contains some amount of S.

With $Ca(H_2PO_4)_2.H_2O$ and KH_2PO_4, the amount of SO_4^{2-} extracted increases with depth (Tables 1 and 3, soil samples 2 (0-25 cm) and 3 (40-50 cm)). This increase is not uncommon owing to the fact that the highly soluble SO_4^{2-}, which is also easily displaced from the soil colloids by phosphate (Kang et al., 1981), can be leached into the lower horizons (Russell, 1966). The reverse is true for phosphate where the amount of phosphorus extracted decreased with depth (Tables 1 and 2, soil samples 2 (0-25 cm) and 3 (40-50 cm)). Soil samples 1, 2, 3, and 10 had very high values for SO_4^{2-} compared to the other soil samples. These high values could be due to the fact that some of the fertilizers such as ammonium sulfate, $(NH_4)_2SO_4$ and single superphosphate, calcium dihydrogen phosphate - $Ca(H_2PO_4)_2$ and gypsum - $CaSO_4$, which are commonly applied to these plantations, have some amount of sulfur.

The SO_4^{2-}-S values for samples extracted with H_2O and $CaCl_2$ were lower than those with the other extractants. These low values could partly be attributed to the low ion-product constant (K_w) of water and the poor competitive nature of the chloride (Cl^-) ions for adsorption sites (Chang and Thomas, 1963; Chao et al., 1964) and thus exchangeable SO_4^{2-}-S (sulfate that is adsorbed unto the soil colloids) is not extracted but readily soluble SO_4^{2-}-S (sulfate that is in solution) is extracted by these extractants (Barrow, 1967). The high values of SO_4^{2-}-S extracted by $Ca(H_2PO_4)_2.H_2O$, KH_2PO_4 and Morgan (CH_3COONa/CH_3COOH) could be attributed to the fact that they extract both forms of SO_4^{2-}-S, the readily soluble and the exchangeable as established by Ensminger (1954); Fox et al. (1964); Howard and Reisenauer (1966). The anions, phosphate and acetate that are present in the respective salt solutions $Ca(H_2PO_4)_2.H_2O$ and KH_2PO_4, and Morgan (CH_3COONa/CH_3COOH) are stronger adsorbing anions than the sulfate ion (Howard and Reisenauer, 1966; Kang et al., 1981; Mengel and Kirkby, 1982). The potassium dihydrogen phosphate and Morgan's solution extracts had higher amounts of sulfate sulfur than calcium dihydrogen phosphate extract (Figure 1). The higher amounts of sulfate sulfur exhibited by potassium dihydrogen phosphate and Morgan's solution extracts could be attributed partly to the presence of K^+ and Na^+ ions, respectively, which have a dispersion tendency. Hence clear filtrates were difficult to obtain. Calcium dihydrogen phosphate

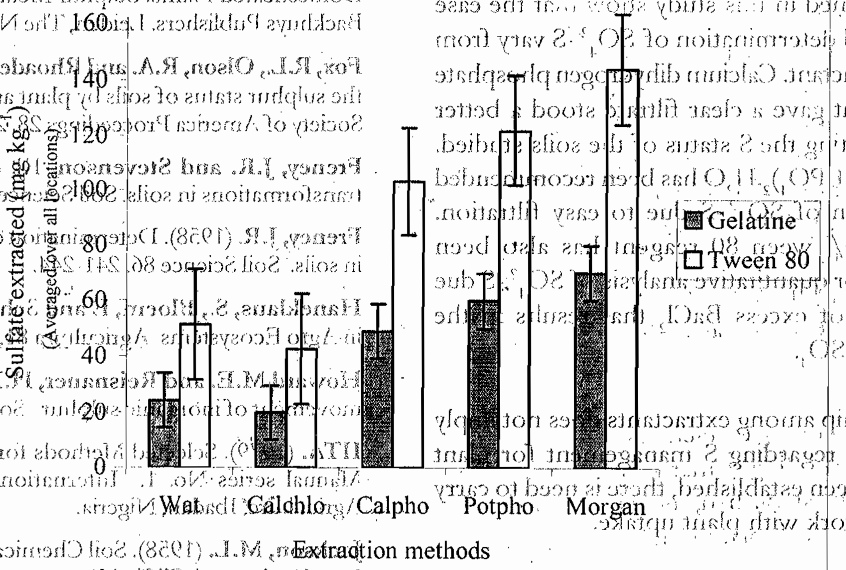

Fig 1: Sulfate content of soils as extracted and determined by some methods.
Wat = H_2O; Calchlo = $CaCl_2$; Calpho = $Ca(H_2PO_4)_2.H_2O$; Potpho = KH_2PO_4;
Morgan = CH_3COONa/CH_3COOH
⊥ = Standard Error of mean

monohydrate gave relatively clear extracts due to the added advantage that Ca^{2+} ions suppressed the extraction of organic matter by coagulating organic matter particles (Howard and Reisenauer, 1966; Barrow, 1967; Mengel and Kirkby, 1982; Brady, 1984).

For the turbidimetric reagents, T80 gave the highest amount of SO_4^{2-}-S for all the extractants and in all the soils (Table 3). The reason could be due to the presence of excess BaCl2 in T80 that is needed for the formation of $BaSO_4$. Barium chloride/Tween 80 reagent is therefore recommended for quantitative determination of extractable S in these soils.

There was a significant positive relationship ($p<0.001$) among all combinations of the different extractants and turbidimetric reagents (Table 4). A significant positive relationship implies that all the extractants and turbidimetric reagents gave the same trend in the level of SO_4^{2-}-S. On the basis of environmental pollution, the $Ca(H_2PO_4)_2.H_2O$ extractant is recommended for routine soil testing on soils of this agro-ecological zone because of its ability to suppress organic matter and produce clear filtrate.

Table 4. Linear correlation coefficient (r) between the extractants and tubidimetric reagents.

	Wat-G	Wat-T	Calchlo-G	Calchlo-T	Calpho-G	Calpho-T	Potpho-G	Potpho-T	Morgan-G
Wat-T	0.999	-							
Calchlo-G	0.988	0.984							
Calchlo-T	0.988	0.983	0.999	-					
Calpho-G	0.960	0.962	0.950	0.950					
Calpho-T	0.966	0.968	0.958	0.957	0.999				
Potpho-G	0.952	0.955	0.943	0.942	0.999	0.998	-		
Potpho-T	0.950	0.954	0.942	0.941	0.999	0.998	0.999		
Morgan-G	0.945	0.951	0.932	0.931	0.995	0.988	0.998	0.998	-
Morgan-T	0.940	0.947	0.926	0.924	0.994	0.994	0.997	0.997	0.999

Wat = H_2O; Calchlo = $CaCl_2$; Calpho = $Ca(H_2PO_4)_2.H_2O$; Potpho = KH_2PO_4; Morgan = CH_3COONa/CH_3COOH.
G = $BaCl_2.2H_2O$/Gelatine reagent; T = $BaCl_2.2H_2O$/Tween 80 reagent.
All values are significant at 0.1% level.

CONCLUSION

The results obtained in this study show that the ease of extraction and determination of SO_4^{2-}-S vary from extractant to extractant. Calcium dihydrogen phosphate monohydrate that gave a clear filtrate stood a better chance of estimating the S status of the soils studied. The reagent, $Ca(H_2PO_4)_2.H_2O$ has been recommended for the extraction of SO_4^{2-}-S due to easy filtration. Barium chloride/Tween 80 reagent has also been recommended for quantitative analysis of SO_4^{2-}-S due to the presence of excess $BaCl_2$ that results in the formation of $BaSO_4$.

Since a relationship among extractants does not imply that information regarding S management for plant production has been established, there is need to carry out calibration work with plant uptake.

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