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Solubility and Availability of Sorbed Sulfate in Hawaiian Soils[1]

S. M. HASAN, R. L. FOX, AND C. C. BOYD[2]

ABSTRACT

The capacity of volcanic ash soils to adsorb sulfate is related to rainfall, a major factor in weathering. Highly weathered and leached soils developed from ash may contain 7,000 μg/g of sulfate S in the subsoil. This sulfate has very low water solubility. Some less weathered ash soils are almost devoid of sulfates. Desorption of sulfate decreases the solubility of adsorbed sulfate remaining. Sulfate sorption-desorption isotherms plotted against log concentration of sulfate in equilibrium solutions give approximately straight lines up to 16 to 30 ppms S in solution. Indications are that "first phase" adsorption maxima were attained by highly weathered soil at about 16 ppm S in solution. For less weathered soils, "first phase" adsorption maxima were attained at about 33 ppm S in solution. Pot studies on soils which sorb much sulfate indicate that the soil solution should contain about 5 ppm SO_4-S for adequate nutrition of kikuyu grass. This corresponds to 60 to 80% of first phase adsorption maxima of the ash derived soils. Soils which have low capacity to adsorb sulfate need higher concentrations of sulfate in solution to compensate for low adsorption capacity. A soil which field experiments had shown to be very S deficient was only 10% sulfate saturated.

Additional Key Words for Indexing: sulfate adsorption, volcanic ash derived soils, anion exchange, kikuyu grass.

HAWAII is an excellent natural laboratory for studies in soil-plant relations. Great natural diversity is indicated by the complexity of the classification system for soil. There are 10 orders, 27 great groups, and 167 series. While this diversity has caused some frustration, it should permit the development of concepts and practices which extend beyond the narrow confines of the islands.

The parent materials for most Hawaiian soils are basaltic igneous materials: lavas, cinders, and ash. Time is an important factor in existing soil patterns. Some volcanoes are still active while others are ancient. Moisture is the overriding factor in soil formation. Rainfall varies greatly over short distances, for example, a range of 180 to 7,500 mm along a transect of 70 km.

Humid tropical conditions promote rapid alteration of ash and cinders. There is a heavy loss of silicon and bases and an accumulation of Fe and Al oxides. The resulting soils have little permanent negative charge and, if not too heavily limed, they will have considerable pH-dependent positive charge. Under these conditions cations leach readily (21) but some oxyanions such as sulfate and phosphate are strongly retained (14, 15).

The native S status of Hawaii soils is influenced by amounts of S in rain water. This depends on the amounts of rainfall received and S concentration. Both of these, in turn, are related to distance, direction, and elevation from the sea. On the island of Kauai, Fox et al. (15) recorded 5 ppm S in rainwater 0.5 km from the sea, but only 0.8 ppm 8 km inland.

Sorbed sulfate is abundant in many highly weathered soils, especially in acid subsoils, and is an important source of sulfur for plants (2, 3, 4, 12, 15, 16, 17, 20, 24) although Barrow (4) has concluded that adsorbed sulfate is lower in availability than soluble sulfate. Chao, Harward, and Fang in Oregon (7, 8, 9, 10, 11) and others (1, 3, 6) investigated many aspects of sulfate sorption and desorption, pointing out that sulfate retained by soils is in kinetic equilibrium with sulfate in solution and that sulfate retained is dependent on pH and on the concentration and nature of associated ions, both adsorbed and in the equilibrium solution. Soils which strongly retained sulfate were selected for special attention in the Oregon studies. These were leached soils—soils which are products of intense weathering associated with the temperate, high-rainfall climate of Western Oregon.

Sulfur deficiencies have been reported from several locations in the humid tropics (5, 14, 15, 22, 23) and there have been a few studies on soil sulfur there (6, 22), but much more needs to be done before accurate predictions can be made on the magnitude of the sulfur problem in the tropics or the probable requirements and effectiveness of sulfur fertilizers.

The sulfur status of soils derived from volcanic ash and cinders, soils which are very important in the Pacific Basin area, have been studied both in and out of the tropics (6, 9, 14, 15, 18, 19, 23). Very strong sulfate retention occurs on some soils but rapid leaching and deficiencies are reported, also. Most sulfate retention (sorption) by soils is associated with hydrated aluminum and iron oxides (17). Although work in New Zealand has indicated that some soils which contain allophane may retain little sulfate against leaching, older soils containing allophane retain sulfate strongly (18). Thus, sulfate sorption phenomena should be especially important in the highly weathered, fine-textured soils of the tropics. Mechanisms of sulfate retention have been discussed by Harward and Reisenauer (17), while Fields (13) considered anion retention in general.

Sulfate sorption and desorption reactions should be most relevant to plant nutrition at low solution concentrations. It is this range which seems to have been neglected most.

Although plants utilize sorbed sulfate, the concentration of sulfate in solution (the *intensity* of S nutrition) in relation to amount of sulfate sorbed (*capacity* for nutrition) has not been adequately studied. In this report we (i) document the magnitude of sorbed sulfate in some Hawaii Latosols, (ii) examine the relationship between sorbed sulfate and equilibrium solution concentration of sulfate and

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Table 1—Some properties of Hawaiian soils derived from volcanic ash, arranged according to rainfall and compared with three soils developed from basalt

Series name	Sub groups	Rainfall	pH*			dph	Reserve S	P† sorbed	S‡ sorbed	Sorbed§ S	Si¶ solubility
			H_2O	KCl	K_2SO_4						
		mm						μg/g			ppm
					Soils from Ash						
Kawaihae	Typic Vitrandept	200	6.5	6.40	6.97	0.57	180	600	30	60	0.9
Waimea	Typic Eutrandept	500	6.4	6.25	6.60	0.35	640	380	30	40	4.7
Hanipoe	Typic Dystrandept	1,100	5.6	5.58	5.91	0.33	520	600	110	130	2.7
Maile	Hydric Dystrandept	2,300	5.5	5.20	5.80	0.60	1,200	900	230	350	0.7
Ookala	Hydric Dystrandept	2,200	5.1	4.75	5.20	0.45	1,990	2,000	150	720	3.3
Akaka	Typic Hydrandept	4,500	5.9	5.20	6.14	0.94	2,200	2,000	160	1,100	1.1
					Soils from Basalt						
Molokai	Tropeptic Haplustox	600	6.8	6.62	6.75	0.13	360	60	20	30	3.1
Wahiawa	Tropeptic Eutrorthox	1,000	5.7	5.30	6.13	0.77	270	390	20	40	1.2
Kapar	Typic Gibbsihumox	2,500	4.4	4.40	4.90	0.50	600	1,100	160	250	0.6

* 0.1*N* salt solutions at 1:10 soil solution ratios, pH on supernatant solutions.
† Phosphate adsorbed at 0.2 ppm P in the equilibrated solution. Conditions were: 25C, 6 days, 0.01*M* $CaCl_2$.
‡ Sulfate-sulfur removed from solution by soil at 5 ppm S in the equilibrated solution. Conditions were 25C, 24 hours, 0.01*M* $CaCl_2$.
§ Total sulfate-S (initial labile S + sulfate removed from solution). Conditions as above.
¶ Si in solution after shaking a 1:10 suspension of soil in water for 4 hours.

(iii) present some preliminary data on the relationships between adjusted sulfate concentrations in soil solutions and plant response.

MATERIALS AND METHODS

Soil Samples—Soil profile samples were obtained in an area of volcanic ash parent material on the Island of Hawaii. This is the youngest and largest island of the Hawaiian chain. Rainfall varies greatly over short distances. Locations sampled for this study received from 200 to more than 6,000 mm mean annual precipitation. Highly hydrated volcanic ash soils dehydrate irreversibly, thus it is very important to prevent excess drying. In this case, large samples of the field-moist soils were double-bagged in polyethylene. Several of the soils are thixotropic so that screening these soils was impractical. Soil properties vary greatly with the amounts of rainfall, as can be seen from the data presented in Table 1. Values for *d*pH were generally high compared with data reported for some ash soils of Costa Rica (6) or Oregon (11) and phosphate and sulfate sorption were correspondingly high.

Laboratory Methods—Sulfate sorption isotherms were constructed from data collected as follows: Samples (3g) of soil were equilibrated with 30 ml solutions which were 0.01*M* with respect to $CaCl_2$ and contained variable amounts of Na_2SO_4. Equilibration was accomplished at 25C for 24 hours, which was more time than was required for obtaining constant sulfate in solution. Final solution concentrations of S were considerably less than the solubility of gypsum in 0.01*M* $CaCl_2$. Sulfate in solution was determined turbidimetrically. Sulfate sorbed was considered to be that which was not recovered. Allowance was made for dilution by water contained initially in the moist soil. All data on concentration were plotted as sulfate S remaining in solution after equilibrium had been attained.

Labile S was estimated by equilibrating soil with sulfate labeled with ^{35}S in 0.01*M* $CaCl_2$ as described above. Labeled sulfate in the equilibrated solutions was determined by liquid scintillation counting, and total sulfate in solution was determined turbidimetrically.

Sulfate was desorbed by equilibrating soil with 0.01*M* $CaCl_2$ in various soil:solution ratios, or by repeated extractions of the same soil sample with 0.01*M* $CaCl_2$. Continued sulfate desorption eventually resulted in very low concentrations of sulfate in solution. For sulfate determinations in this range of concentration (< 1 ppm), soils were equilibrated with ^{35}S. The ratio of sulfate to net counts was established, and then upon further dilution, net counts were used to assess sulfate in solution. Sulfate sorbed or desorbed was added algebraically to native sulfate (labile sulfate) to arrive at sorbed sulfate values for plotting the linear form of the Langmuir equation. The method of Bardsley and Lancaster (2) for "reserve" S was used to estimate total S in soils. Phosphate extraction of sulfate was with $Ca(H_2PO_4)_2$ as described by Fox et al. (16). The method for determining *d*pH is that of Chao et al. (11). Soils were equilibrated with 0.1*N* KCl and 0.1*N* K_2SO_4 and pH of the clear supernatant solutions was determined. The difference in pH, referred to as *d*pH, is believed to represent the extent OH^- is replaced by SO^{2-}_4.

Table 2—Total S in some Hawaii Latosols in relation to rainfall. Soils were developed from volcanic ash, Island of Hawaii. Data of Hough and Gile (19) are recalculated to give the weighted Mean for S in the 0 to 100 cm profile

Series name	Sub group	Rainfall, mm	Total S, μg/g
Waimea	Tropeptic Eutrorthox	500	880
Moalua	Hydric Dystrandept	1,200	960
Kaiwiki	Typic Hydrandept	4,400	5,570
Hilo	Typic Hydrandept	4,800	3,760
Kaiwiki	Typic Hydrandept	6,900	5,490

Greenhouse Methods—Sulfate adsorption isotherms were constructed by equilibrating soils with water containing varying amounts of sulfate. From these curves, amounts of sulfate required to adjust the soil solution to 2, 5, 10, 20, and 50 ppm sulfate S were determined and these amounts added to several soils. Kikuyu grass (*Pennisetum clandestinum*) was grown for two cuttings in 1-gallon pots. Phosphorus added was also based on adsorption isotherms. Other nutrients were added as constants. The S results reported are means for two phosphate levels in the adequate or near adequate range.

RESULTS AND DISCUSSION

Soils developed from highly weathered volcanic ash in Hawaii frequently contain large amounts of S, even though these soils may be subject to almost continuous leaching (15). In fact, it is the most highly leached soils which contain the greatest amounts of organic and sulfate S. Examples of the very large amount of total S which can accumulate in soils developed from volcanic ash are given in Tables 1 and 2, and the profile distribution of S in variously weathered and leached soils is presented for the chief experimental soils of this study in Table 3.

Sulfur accumulation and distribution in soil profiles is related to rainfall in three ways. (*i*) the capacity of soils to stabilize organic matter, which contains S, is a function of reactive products of weathering which, in turn, is related to rainfall; (*ii*) the capacity of soils to sorb sulfate is related to weathering products; and (*iii*) rainfall is a major source of sulfate. A comparison, in Table 3, of the Waimea (low rainfall) and Akaka (high rainfall) profiles demon-

Table 3—Sulfate sulfur removed by four successive extractions with $Ca(H_2PO_4)_2$ (16) from soils which are developed from volcanic ash. Soils are arranged according to increasing rainfall. Values are as ppm S in dry soil

Soil series	Depth increment, cm				
	0-15	15-30	30-60	60-90	90-120
Kawaihae	60	170	230	---	---
Waimea	13	11	4	4	5
Hanipoe	25	18	16	17	7
Maile	150	1,260	2,170	2,450	---
Ookala	1,070	780	960	1,170	1,140
Akaka	1,600	2,480	7,210	7,340	7,320

strates that for the Waimea soil sulfate decreased with depth, while large amounts of sulfate reside in the Akaka subsoil. This latter pattern is not unusual for Latosols and other highly weathered soils which contain hydrated aluminum and iron oxides. What is unusual is the magnitude of the values—over 7,000 ppm S.

Much of the sulfur of the highly weathered ash soils is sulfate, which can be displaced with phosphate but is not readily displaced with water. (Fox et al., 15). As many as four repeated extractions with phosphate are required to remove most of the sulfate (Table 4). This suggests that a wider soil/phosphate-extracting-solution ratio is needed to assess sorbed sulfate by a single extraction of these soils. If this sulfate had appreciable water solubility, it would have been leached from the profile long ago, since annual rainfall in excess of 300 inches (7,500 mm) is not unknown in some agricultural areas. In addition, for soils with greatly different capacities to sorb sulfate, sorbed sulfate (that which can be extracted with phosphate or will equilibrate with a S^{35} spike) will not correlate well with sulfate concentration in the soil solution.

Sulfate sorption-desorption isotherms constructed for two Latosols are presented in Fig. 1 and 2. Such plots are useful for predicting both the capacity for, and intensity of,

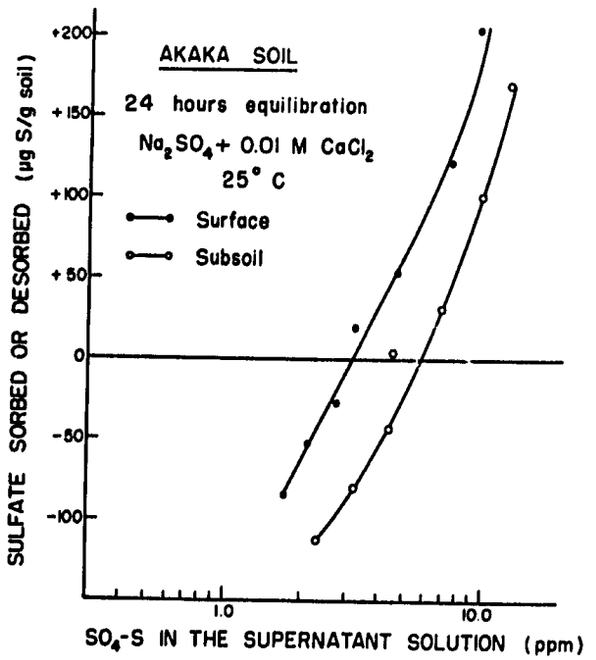

Fig. 1—Relationship between sulfate sorbed or desorbed and equilibrium solution concentrations for an Akaka surface and subsoil.

Table 4—Sulfate sulfur extracted from forested Akaka soil by four extractions with $Ca(H_2PO_4)_2$. Values on oven dry soil basis. Some field moist samples contained only 20% dry matter

Depth	SO_4-S extracted, µg/g				
	Extraction no.				Total extracted
	1	2	3	4	
0-6	165	11	0	40	216
6-12	628	317	214	50	1,209
12-18	1,500	1,095	450	246	3,291
18-24	1,930	1,304	574	365	4,173
24-30	2,283	1,648	774	304	5,009
30-36	2,605	2,000	995	365	5,965
36-42	1,860	1,447	727	256	4,290
42-48	1,946	1,760	800	233	4,740

Table 5—Estimates of the labile sulfate pool by three methods

Soil	Method of estimation		
	Radioisotope dilution	Extrapolation of the isotherm	Repeated phosphate extraction
Waimea	6	11	13
Hanipoe	17	29	25
Akaka	940	950	1,600
Wahiawa		110	115

plant nutrition. No knowledge is required of the initial labile sulfate pool, although the size of the labile pool certainly influences the position of the curves on the graph. In fact, an arithmetic plot of these data can be extrapolated to zero concentration to estimate the labile sulfate pool. Table 5 gives estimates of the sulfate pool by three methods for three Hawaii soils.

The highly leached and weathered Akaka soil is developed from ash. It has a high potential buffering capacity for sulfate, as is indicated by the steep slopes of the isotherm; the amount of original sorbed sulfate which the subsoil contains was sufficient to maintain more than 3 ppm sulfate in solution until almost 100 µg S/g soil had been withdrawn. The Wahiawa soil cannot match the Akaka in capacity or reserve supply of sulfate, but it is interesting to note that at zero sulfate sorption both surface soils were at equilibrium with 3 ppm SO_4-S in solution.

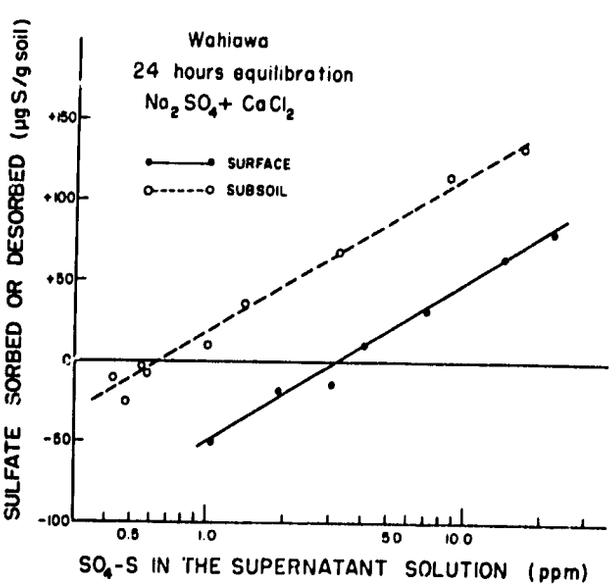

Fig. 2—Relationship between sulfate sorbed or desorbed and equilibrium solution concentrations for a Wahiawa surface soil and subsoil.

When initial labile S is included in plots of sulfate adsorption, curves like those presented in Fig. 3 are obtained. These curves are useful for predicting the intensity of S nutrition in relation to sorbed sulfate in soils.

Adsorption of sulfate fits the linear form of the Langmuir equation over a limited range of concentration, and adsorption maxima can be calculated (Fig. 4). The utility of these maxima has been questioned (9). Perhaps "first phase adsorption maxima" would be a more appropriate term to use. If adsorption maxima are to be useful, care should be taken to avoid increasing solution concentrations until further adsorption mechanisms are activated (3). Chao et al. (9) found that calculated adsorption maxima were not particularly helpful in that the values obtained did not predict any possible maximum of sulfate adsorption that would be consistent with other data they obtained. Nevertheless, the calculated values for adsorption maxima given in Fig. 4 fell into a reasonable pattern: they predict that the adsorption maxima for the most weathered and acid soils will be reached when the equilibrium concentration is about 16 ppm (Fig. 3—vertical bars). These values agree with a value reported by Chao et al. (9), for departure from linearity by a latosol and are not very different from three other values reported (10–11 ppm) by them.

For the less highly weathered soils, adsorption maxima are attained with 30 to 35 ppm in solution. This may indicate a tendency for anionic repulsion by the more negatively charged surfaces of the less weathered soils.

The first phase adsorption maxima may give useful information even if the basic mechanisms involved are not clear. These values could be useful for predicting the amount of sorbed sulfate required to give a desired level of sulfate in solution. If, for example, the volcanic ash soils of Hawaii are to have 5 ppm sulfate S in solution, the first phase sulfate adsorption capacity should be 60 to 80% saturated. It must be pointed out, however, that adsorbed phosphate can greatly modify the sulfate solution-sorbed phase equilibria (3, 7, 20). An example of this for a Hawaii soil is presented in Fig. 5. In high rainfall conditions sulfate mobilized by lime and phosphate applications does leach into the subsoil.

From what has been said above, adequate S nutrition of plants should be attained if sorbed sulfate is adjusted to give an appropriate level of sulfate in the soil solution. This hypothesis was tested on seven ash soils with good results. Selected data are presented in Fig. 6.

For soils with large capacity to sorb sulfate, represented in Fig. 6 by the Akaka soil, 5 ppm S in the adjusted equilibrium solution was near optimum for kikuyu grass. For soils with intermediate capacity to adsorb sulfate (Moaula and Hanipoe) 5 to 20 ppm was adequate; but, for soils which had little capacity to adsorb sulfate (Naalehu and Kawaihae) yields increased even beyond 20 ppm. High concentrations of sulfate in solution were required for adequate sulfate supplying capacity in these soils with little adsorbed sulfate in reserve.

A requirement of 5 ppm S in solution probably is a little high since it was obtained from a pot study, although field data from Nebraska suggest that a soil containing 4 ppm sulfate-S and irrigated with water containing 3 ppm SO_4-S was borderline deficient for corn (Daigger, L. A., and R. L. Fox. Yields and N/S ratios of sweet corn in relation to

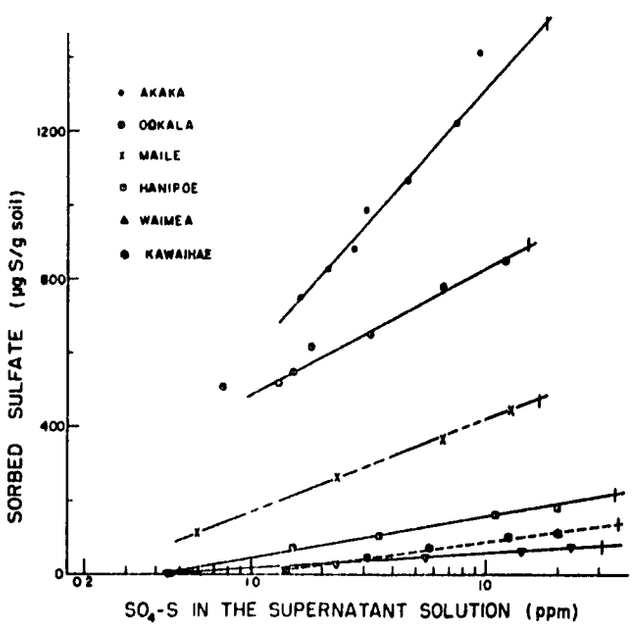

Fig. 3—Sulfate sorption isotherms for a series of six soils developed from volcanic ash, Island of Hawaii. The soils are listed in order of decreasing rainfall. The vertical bars near the end of each curve represent the calculated first phase adsorption maxima.

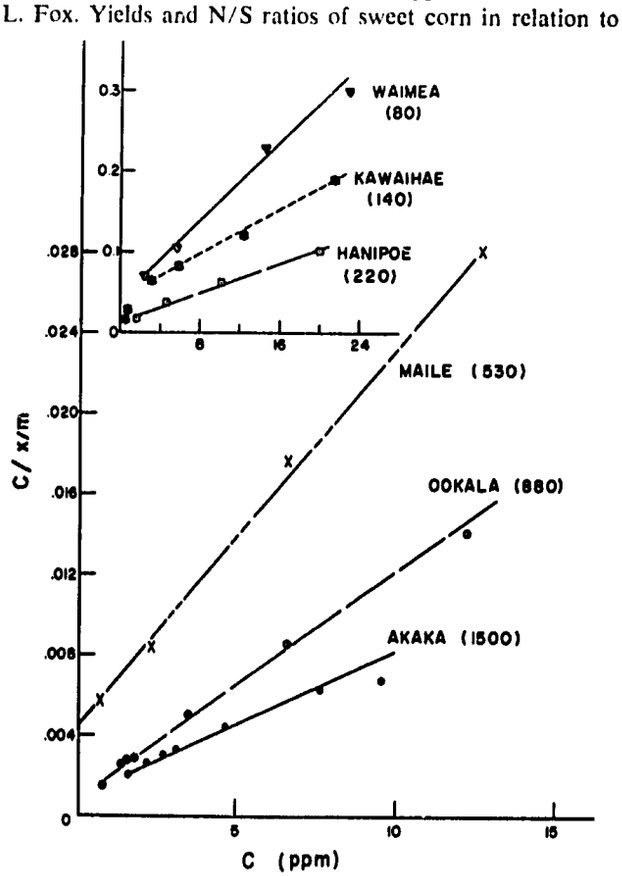

Fig. 4—Linear plots of the Langmuir isotherms giving the calculated adsorption maxima in parentheses. Equilibrium time was 24 hours at 25C in 0.01*M* $CaCl_2$.

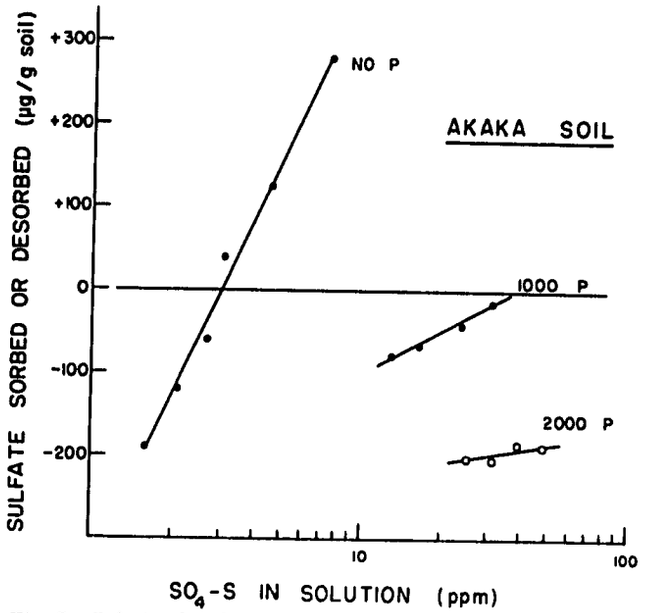

Fig. 5—Relationship between sulfate sorbed and sulfate in the equilibrium solution for an Akaka surface soil which has been treated with various amounts of phosphate. Values for phosphate added are µgP/g soil. The phosphate was added 6 days prior to the addition of sulfate after which both were equilibrated together with the soil for 24 hours.

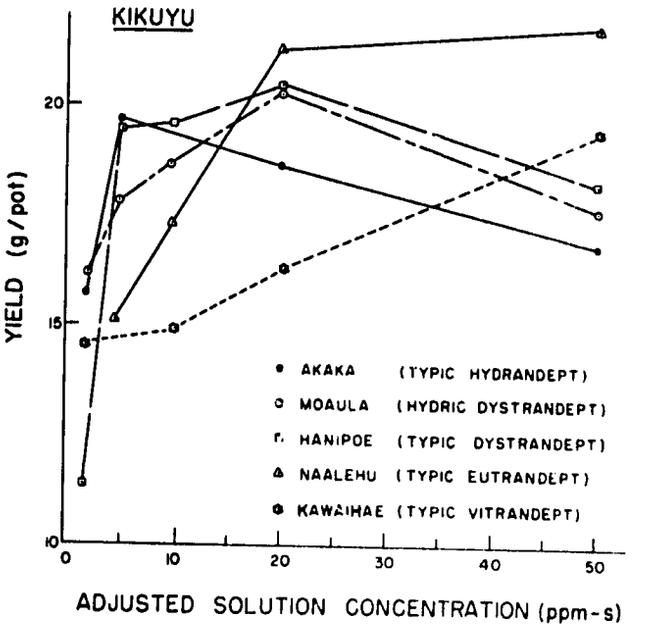

Fig. 6—Yield of kikuyu grass grown in pots in relation to adjusted sulfate concentrations in soil solutions.

fertilization and soil and water composition. Manuscript prepared for Agronomy Journal Note).

Fertilizer trials have been conducted in the field on three of the soils used in the pot studies. The Hanipoe was very sulfur deficient. On this soil S increased the yield of kikuyu 87% above the no-sulfur control (15). The data in Table 5 and Figs. 3 and 4 explain why. This soil has medium capacity to adsorb sulfate (first stage adsorption maximum = 220 µg/g) but native adsorbed sulfate was only about 20 µg g. This corresponds to 10% sulfate saturation which is far less than is required to maintain S in solution at an acceptable level.

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