PHOSPHATE ADSORPTION AND IMMOBILIZATION CHARACTERISTICS
OF THREE SOILS FROM SOUTHERN BAHIA, BRAZIL[1]

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ABSTRACT - Measurements of the phosphate adsorption/desorption characteristic of three acid soils from Southern Bahia, Brazil, were compared with other soil properties and with the response of sorghum to a range of phosphate additions. One soil (Tropudult Camacã), containing smectite and with a high content of exalate-extractable (amorphus) Fe and Al oxides, showed high phosphate adsorption and provided the test plants with a good supply of phosphate. A contrasting soil (Haplorthox crystalino) showed lower phosphate adsorption, lower phosphate availability, and gave a high phosphate immobilization. It contained high amounts of Fe and Al extractable by dithionite; gibbsite and goethite were identified. The half-life of labile phosphate in this soil was 170, 210 and 275 days measured by phosphate uptake by sorghum plants, isotopic exchange, and anion-resin extraction, respectively. In the other two soils the half-life was over 400 days by all three methods. Anion-exchange resin extraction provided a convenient index of labile phosphate, and hence of immobilization of phosphate. The contrasting behaviour of the soils shows that adsorption and immobilization of phosphate need to be distinguished: a high adsorption coefficient does not necessarily mean high immobilization.

Index terms: adsorption/desorption, smectite, dithionite, phosphate.

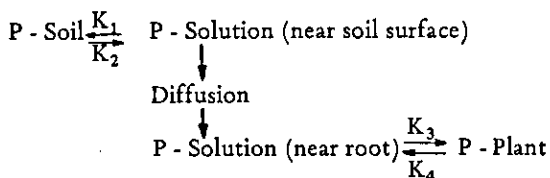## ADSORÇÃO E IMOBILIZAÇÃO DE FÓSFORO EM TRÊS SOLOS DO SUL DA BAHIA

RESUMO - Em amostras superficiais de três solos ácidos do sul da Bahia, as características de adsor-çãó/dessorção de fósforo foram relacionadas com outras propriedades do solo e com a resposta do sorgo (Sorghum vulgare) a doses crescentes de fósforo. O solo T. Camacã, contendo esmectita e altos teores de Fe e Al extraídos por oxalato, apresentou maior capacidade de adsorver fósforo e forneceu um adequado suprimento de fósforo para as plantas. O solo H. Cristalino, com altos conteúdos de Fe e Al extraídos por ditionito e apresentando gibsita e goetita na difractometria de raios X, evidenciou menor disponibilidade de fósforo e alta capacidade de imobilizar esse elemento. Este aspecto foi bem caracterizado pela meia-vida do fósforo lábil e que correspondeu nesse solo a, respectivamente, 170, 210 e 275 dias para as variáveis fósforo absorvido pelo sorgo, fósforo isotopicamente trocável e fósforo extraído pela resina de troca aniônica. Nos dois Tropudults, a meia-vida do fósforo lábil foi superior a 400 dias para as mesmas variávies, e, em todos os solos ensaiados, o fósforo extraído pela resina cons-titui um bom índice do fósforo lábil e permite avaliar de modo satisfátorio o grau de imobilização des-se elemento. O comportamento contrastante dos solos ensaiados indica que adsorção e imobilização de fósforo são processos que devem ser distinguidos, pois uma alta adsorção não implica necessariamente uma alta imobilização desse elemento.

Termos para indexação: adsorção/dessorção, esmectita, ditionito, fósforo

INTRODUCTION

The concepts of intensity and capacity have proved to be a helpful way of looking at the phosphate supply to plants (Nye & Tinker 1977), and can be incorporated into models of phosphate supply to plants such as that of Gunary & Sutton (1967):

$$P - Soil \underset{K_2}{\overset{K_1}{\rightleftarrows}} \quad P - Solution \text{ (near soil surface)}$$

Diffusion

$$P - Solution \text{ (near root)} \underset{K_4}{\overset{K_3}{\rightleftarrows}} P - Plant$$

Supply of phosphate to the plant can be restricted by any of four factors: Capacity (P-Soil), rate (K_1/K_2), Intensity (P-solution) and diffusion (the ratio of K_3/K_4 is assumed to be high and K_3 to be non-limiting). Capacity or Quantity (Q) describes the size of the labile pool (P-soil). The relationship

(Q/I) between Quantity and Intensity (I) gives the phosphate buffer capacity which describes the change in quantity per unit change in concentration of the solution (Holford & Mattingly 1976).

The Intensity factor (I) is usually determined by equilibrating the soil with $CaCl_2$ in the concentration range: 0.01M to 0.001M and containing ^{31}P at different concentrations. At $\Delta P = 0$ on a Q/I curve the concentration of phosphate is that of the equilibrium solution. The quantity factor (Q) can be evaluated either as the amount of phosphate which is adsorbed during equilibration, or as the amount which remains labile using (a) isotopic dilution with [32]P (Russell et al. 1957, Larsen 1967) or (b) an anion-exchange resin (Diest et al. 1960).

To describe adsorption curves several equations have been employed. The Freundlich equation describes adsorption curves well over a limited range of concentrations, but the Langmuir equation is some times preferred because of its theoretical basis. One of its advantages is that it permits calculation of the adsorption maximum (Olsen & Watanabe 1957) but it requires assumptions that are usually not valid (Larsen 1967). Also, the adsorption maximum calculated from low concentrations is generally exceeded at high concentrations (Barrow 1978). Modifications have been made to the Langmuir equation by including a square root term (Gunary 1970) and by inserting the condition that the energy of adsorption decreases linearly as the occupied adsorption sites increases, as in the Temkin equation (Bache & Williams 1971). Holford & Mattingly (1976) suggested a third equation which considered two types of surfaces with contrasting bonding energies.

Adsorption curves have also been used pragmatically (Barrow 1978) to determine the amount of P fertilizer required to bring the concentration of the equilibrium solution to that which gives maximum plant growth (Ozanne & Shaw 1968, Fox & Kamprath 1970). One difficulty is that the required concentration in the equilibrium solution varies not only between plant species (Fox et al. 1974), but also with environmental conditions such as light intensity (Fox & Kang 1978).

The influence of some soil properties on the magnitude and pattern of the adsorption curves has been also investigated, and a relationship with free Fe and Al (dithionite extraction), organic mattern and clay content were found in a large number of Latin America soils (Fassbender 1975). Later, the surface area and the Al and Fe fractions extracted by ammonium oxalate at pH3 (dark extraction, were investigated (Le Mare 1981).

Reviews of the Langmuir, Freundlich, Temkin and other equations used in describing phosphate adsorption curves have been published by Barrow (1978) and Parfitt (1978).

In soils of Southern Bahia, phosphate adsorption was measured in surface samples from eight profiles (Cabala-Rosand 1970). The samples were with and without lime, and the technique used was that of Fassbender & Igue (1967). On average, lime reduced the adsorption, and a positive correlation was found between adsorption and the content of free Fe and Al extracted by Na-dithionite. Comparison with P uptake by sorghum plants showed that the greatest adsorption took place in an Alfisol in which yields were high and where there was no response to P additions. There was less adsorption in an Oxisol in which the yields were low without addition of phosphate and increased gradually with successive increments of added phosphate.

Later, P adsorption was measured on soil samples from field experiments with cocoa. From the Lagmuir equation the maximum phosphate adsorption varied between 438 and 1041 kg P_2O_5 ha^{-1} (Morais et al. 1977). Yields during 3-4 years were close to the maximum with additions of 90 kg/ha^{-1} of P_2O_5. When that level was doubled only small increases in yield were obtained Cabala-Rosand et al. 1976). It appeared therefore, that the adsorption maximum has very limited value in predicting the response of cocoa to P additions.

This paper reports the adsorption properties of three Southern Bahia soils. These results are compared with uptake of phosphate by sorghum and also with other soil properties.

MATERIAL AND METHODS

Soils
    Samples were obtained of soils Tropudult Camacã, Haplorthox Crystalino and Tropudult Vargito described

by Silva et al. (1975). Some of their chemical properties are given in Table 1, and others are in Cabala-Rosand & Wild (Prelo).

Phosphate adsorption isotherms

The procedure of Fox & Kamprath (1970) was used unitially. In this method, 3-g soil samples are shaken intermittently at 25°C for six days with 30 cm³ 0.01M $CaCl_2$ containing various amounts of $Ca (H_2PO_4)_2$. The solution was labelled with ^{32}P in order to measure isotopically exchangeable phosphate as well as adsorbed phosphate. After equilibration, ^{32}P was measured by Cerenkov counting and isotopically exchangeable phosphate was calculated on the principle of isotopic dilution (Russell et al. 1957). Analysis for phosphate was by the colorimetric method of Murphy & Riley (1962).

In later experiments the method was modified. Samples of 2.5 g soil were shaken at 20°C for periods of one, two or four hours with 25 cm³ 0.01M $CaCl_2$ containing various concentrations of phosphate with a constant label of ^{32}P. Suspensions were allowed to stand for 20 hours before being centrifuged, filtered and analysed for phosphate and ^{32}P as described above.

Phosphate desorption with anion-exhange resin

After shaking samples of soil with solutions contai-ning different concentrations of ^{31}P, phosphate was desorbed using anion-exchange resin (Fig. 1) as follows: 4 g of air-dry soil (less than 2 mm) was weighed into 125 cm³ plastic bottles. To this was added 100 cm³ of a solution containing 0, 50, 100, 150, 200, 250, 300 and 400 μg P g^{-1} and 3 drops of chloroform. The bottles were shaken for 22 hours at 20°C and kept for another 22 hours for equilibration at the same temperature. 20 cm³ of the supernatant liquid were transferred to a weighed tube containing 0.5 g NaCl to flocculate the clay in the suspension. The clear solution was analysed for phosphate (P in equilibrium solution). To bring back to the initial volume (100 cm³), 20 cm³ of deonized water was added to each bottle. Resin bags were added to the bottles which were again shaken for 22 hours at 20°C. The resin bags were taken out, washed with deionized water until they became free of soil particles, and were placed in clean plastic bottles. Phosphate was recovered from the resin by extraction with 0.5M HCl and determined colorimetrically (P-resin). The phosphate extracted by anion-exchange resin was corrected for the amount of soil in the aliquot used to determine the P concentration in the equilibrium solution. The results were corrected to an oven-dry soil basis (105°C).

TABLE 1. Particle size measurement results of the X-ray diffraction analyses and percentages of iron and aluminium extracted by pyrophosphate, oxalate, dithionite and perchloric acid (dry basis at 105°C).

Parameter	Unit	Tropudult Camacã	Tropudult Vargito	Haplorthox Crystalino
2000 - 200 μm particle	%	1	1	28
200 - 50 μm particle	%	2	6	17
50 - 2 μm particle	%	22	60	10
2 μm particle	%	75	33	45
X-ray diff. clay	-	Skq (m)	lk (m)	K
X-ray diff. whole soil	-	QSK	QKV (mf?)	QKGbGo
Fe - Pyrophosphate	%	0,715	1.102	1.681
Al - Pyrophosphate	%	0,510	0.279	0.583
Fe - Oxalate	%	0.822	0.602	0.174
Al - Oxalate	%	0.416	0.198	0.178
Fe - Dithionite	%	3.780	2.150	7.300
Al - Dithionite	%	0.500	0.360	1.970
Fe - Perchloric acid	%	6.400	2.820	9.020
Al - Perchloric acid	%	9.220	6.430	9.220
Fe - Active fraction[1]	-	0.217	0.280	0.024
Al - Active fraction[1]	-	0.832	0.550	0.090
Al - Exchangeable	meq/100 g^{-1}	5.320	7.800	0.750
Al - Saturation	%	15.200	76.200	36.230

S = Smectite   M = Mica (m)   K = dominant mineral
K = Kaolinite   Q = Quartz (q)   k = minor mineral
I = Interstratified   Gb = Gibbsite   (k) = trace mineral
V = Vermiculite   Go = Goethite
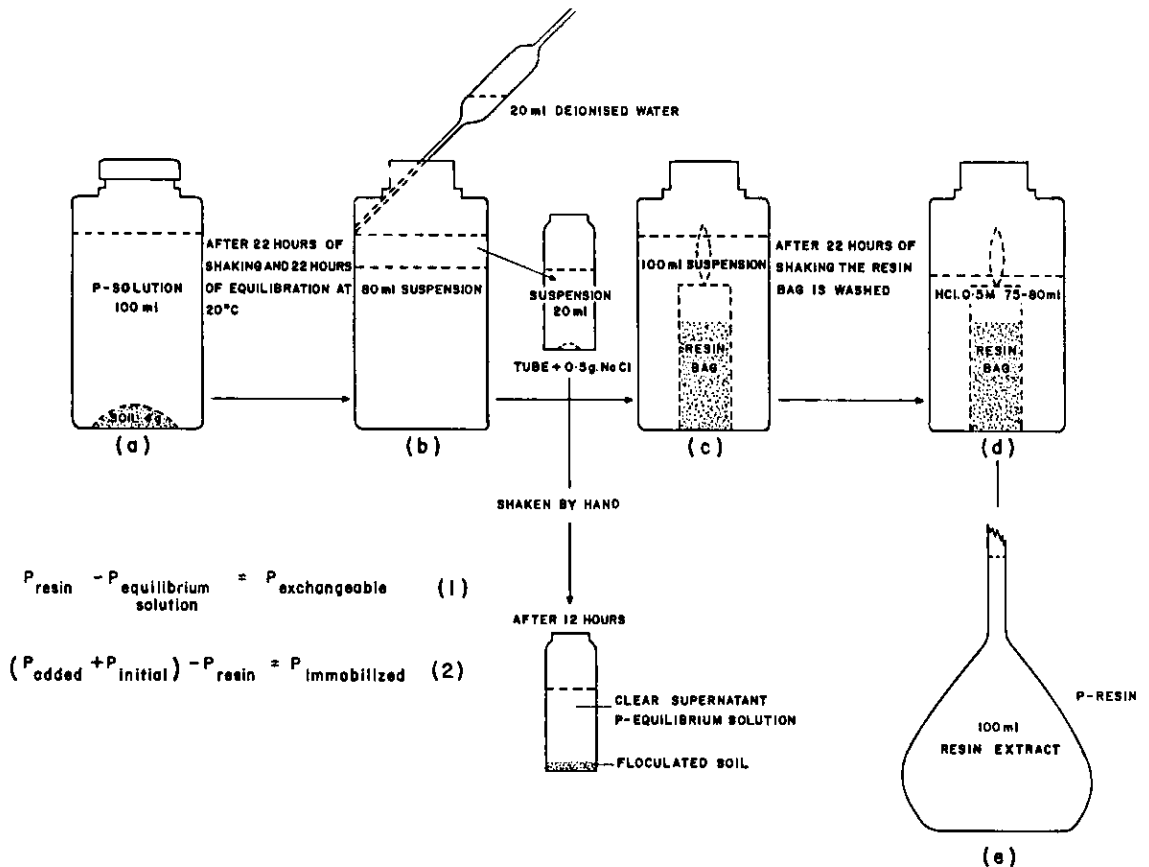F = Fieldspar (f)   1 = Oxalate/Dithionite

FIG. 1. Schematic representation of the method for measuring phosphate adsorption-desorption using anion-exchange resin in polystyrene netting-bags.

To calculate the exchangeable P and P immobilized, the following equations were used:

$$P_{exchangeable} = (P_{resin}) - P_{equilibrium\ solution} \qquad (1)$$

$$P_{immobilized} = (P_{initial} + P_{added}) - P_{resin} \qquad (2)$$

Response curve with sorghum

A response curve was obtained using the same soils with additions of 0, 50, 100, 150, 200, 250, 300 and 400 $\mu g P\ g^{-1}$ as monocalcium phosphate and growing sorghum for 25 days, as described by Cabala-Rosand & Wild (Prelo). The plants were grown in a greenhouse. Nitrogen, potassium and micronutrients were added and two replicates were randomized. The dry weights and the P content in the plants were considered as response variables.

RESULTS AND DISCUSSION

Soil properties

Some physical characteristics of the soils are given in Table 1. According to the USDA textural classification, the Tropudult Camacã and Vargito are respectively clay and silt clay loam soils. The Haplorthox Crystalino is a sandy clay. In the Tropudult Camacã soil, smectite is the dominant clay mineral with smaller amounts of kaolinite; quartz and mica were also detected. The Tropudult Vargito contains kaolinite and interstratified clays, the latter formed by layers of mica and vermiculute as verified by saturating the clay with magnesium and treating with glycerol. In the Haplorthox Crystalino soil, the only clay mineral detected was kaolinite (Table 1). Using unorientated samples os whole soil, gibbsite and goethite were also detected in the Haplorthox Crystalino soil.

The highest amounts of Al and Fe extracted by

pyrophosphate were in the Haplorthox Crystalino soil, but in both Tropudults the oxalate fractions were higher than in the Haplorthox. The amounts of Fe and Al extracted by dithionite and by perchloric acid were higher in the Haplorthox Crystalino than in the other soils (Table 1). These results indicate that both Tropudults contain high amounts of Fe and Al in the inorganic amorphus form (oxalate extraction). The Haplorthox Crystalino contains the highest amount of free oxides (dithionite extraction), and also Fe and Al in organic complexes (pyrophosphate extraction).

Phosphate adsorption

The order of phosphate adsorption was Tropudult Camacã (soil 1) - Haplorthox Crystalino (soil 2) - Tropudult Vargito (soil 3), and for all three soils adsorption increased with reaction time (Fig. 2). The data did not closely fit either the Langmuir or Freundlich equations but did fit the Temkin equation reasonably well (Table 2).

As shown in Table 1, soil 1 has the highest content of amorphous (oxalate-extractable) oxides of Fe and Al, supporting the mass of evidence (Parfitt 1978) that it is mainly the surface area of the oxides that determine the phosphate buffer capacity of soils. Compared with soil 3, soil 2 has a lower content of amorphous oxides of Fe and Al, and also of exchangeable Al (Table 1), but adsorbs more phosphate. Its high content of

gibbsite and goethite might be the cause. There are two few soils for generalizations to be made and regression analysis, such as that used by Le Mare (1981) on soils from Southern Nigeria, is needed to establish relationships between phosphate adsorption characteristics and soil properties.

Measurements of phosphate adsorption were used by Ozanne & Shaw (1968) and developed by Fox & Kamprath (1970) to assess the requirements for phosphate fertilizer. The assumption was made that phosphate additions should raise the phosphate concentration of the soil solution until it was non-limiting for crops. Concentrations regarded as non-limiting are in the range 0.01 to about 0.4 μg cm^{-3} (Fox & Kamprath 1970, Fox & Kang 1978). Fig. 3 shows the relationship between added phosphate and the concentration of phosphate in solution after a reaction time of six days. The order of the soils is again soil 1 > soil 2 > soil 3, although the order 1 and 2 is reversed at concentrations below about 0.04 μg cm^{-3}, implying some high energy adsorption sites in soil 2 to raise the concentration of phosphate in solution to 0.1 μg cm^{-3} the required amounts of P, under the conditions of the experiment, are 270, 210, 100 μg g^{-1} for soils 1, 2 and 3 respectively.

Labile phosphate

Phosphate which is adsorbed (plus the very small proportions that remains in solution) may

TABLE 2. Variance accounted for by Temkin equation applied to phosphate adsorption measurements made by three methods.

Soil	Method	Variance accounted for (%)
Tropudult Camacã	Fox & Kamprath[1]	97.4
	4 hours shaking[2]	93.9
	Resin[3]	95.5
Tropudult Vargito	Fox & Kamprath[1]	97.3
	4 hours shaking[2]	93.5
	Resin[3]	92.4
Haplorthox Crystalino	Fox & Kamprath[1]	96.1
	4 hours shaking[2]	95.3
	Resin[3]	97.0

Methods (1) Fox & Kamprath (1970)
(2) Modification of the Fox & Kamprath method (see text)
(3) Labile phosphate measured by anion-exchange (see text).

remain labile, that is isotopically exchangeable with ^{32}P or extractable by an anion-exchange resin. Phosphate which is adsorbed and is not exchangeable or extractable will be described as immobilized.

In the experiments from which the results shown in Fig. 2 and 3 were obtained, estimates were made of the phosphate which remained isotopically exchangeable and of that which was extractable with an anion-exchange resin. With increasing additions of phosphate, labile phosphate increased more in soils 1 and 3 than in soil 2 (Fig. 4). At each rate of addition, labile phosphate was always the lowest in soil 2 (Haplorthox Crystalino), implying the greatest immobilization of phosphate in this soil. The conditions of reaction were not the same in the three experiments, and the results cannot be compared, but in all three the reaction period was comparatively short.

Reaction times were up to 270 days in a fourth experiment in which the reaction between added phosphate and soil took place at a water potential of -33 k Pa. The experiment has been described elsewhere (Cabala-Rosand & Wild Prelo). A graphical representation is shown in Fig. 5 of labile phosphate measured as anion-resin extractable (P_R), isotopically exchangeable (P_E) and phosphate uptake by sorghum (P_U) in 25 days, all measured at the end of the reaction period and expressed as percentages of monocalcium phosphate at zero reaction time. The linear scales fit the data better than the exponential decay rate of labile phosphate reported by Larsen et al. (1965). The calculated half-life of labile phosphate depends on the mathematical expression used for the decay rate. On a linear basis the half-life of monocalcium phosphate by all three estimates is about 425 days and over 500 days in soils 1 and 3 respectively (both require extrapolation and the values are only approximate). In soil 2 (Haplorthox Crystalino) the values are 170, 210 and 275 days for P_U, P_E and P_R respectively. Although the values differ for soil 2 their order is the same over the time span of the experiment.

This longer experiment supports the conclusion from the three short experiments that phosphate immobilization in greatest in soil 2. From the data in Table 1, only the crystalline oxides of Fe and Al are present in greater amounts in soil 2 than in soils 1 and 3, which suggests that they might be the cause of immobilization; but more soils need to be investigated before this conclusion can be reached with any confidence.

It is of interest that immobilization in greatest in soil 2 but adsorption is greatest in soil 1. The two reactions are therefore not directly related. Adsorption of phosphate onto oxide surfaces is now fairly well understood (Parfitt 1978), but little is yet known of the chemistry of immobilization. It is a slow reaction, but it is uncertain whether the speed of the reaction and the change from labile to non-labile form of the phosphate are due to crystallization or to diffusion of phosphate beneath the oxide surface. P both adsorption and immobilization affect phosphate availability to plants. Adsorption determines the amounts of phosphate in the soil solution and also the rate of
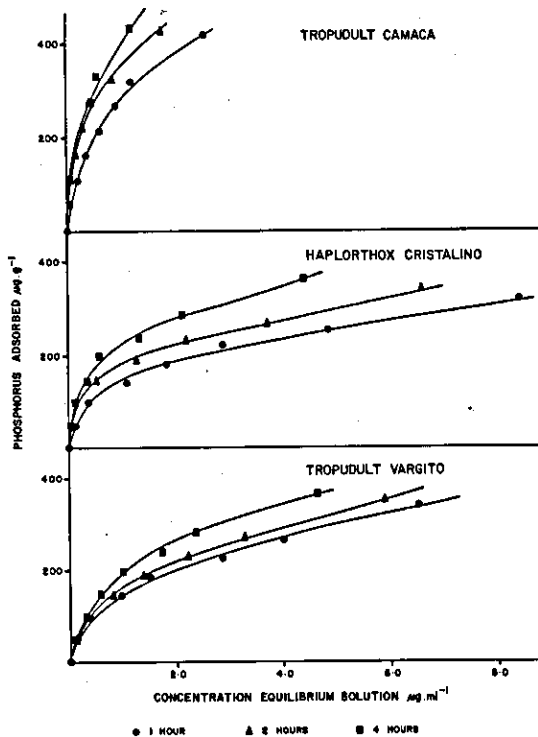

FIG. 2. Plot of phosphorus adsorbed against phosphorus concentration in the equilibrium solution after 1, 2 and 4 hours of shaking and 20 hours of equilibration.
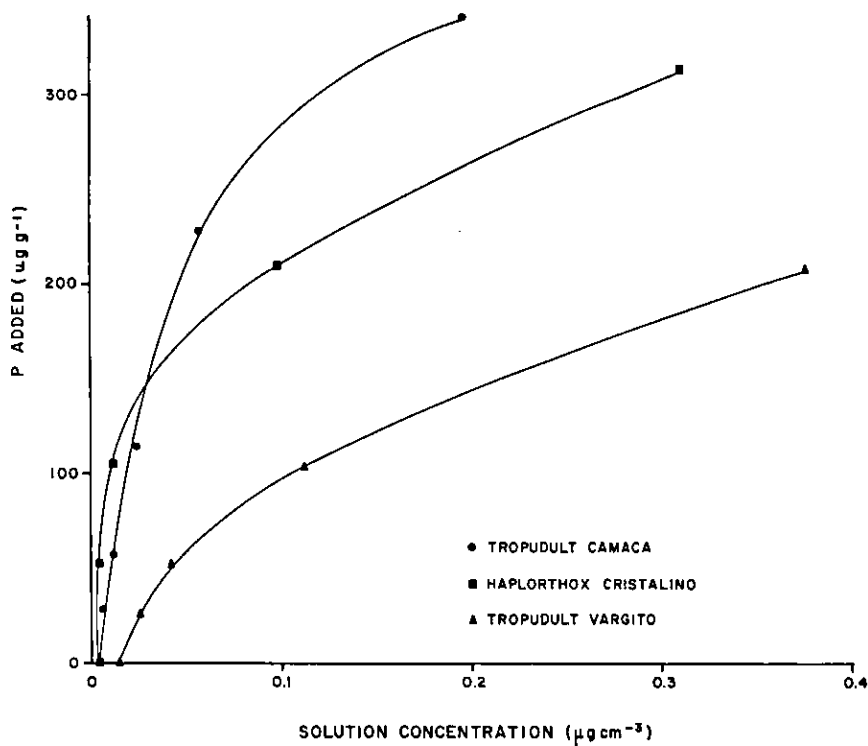
FIG. 3. Relationship between amount of phosphate added and concentration in solution (by method of Fox & Kamprath 1970).
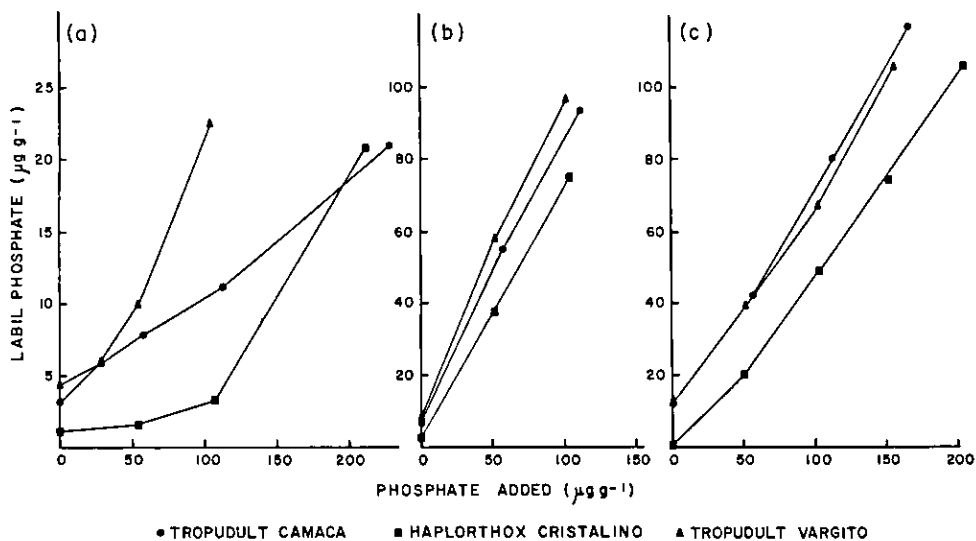

FIG. 4. Relationship between amount of phosphate added and labile phosphate measured; a. Isotopically exchangeable P using modified metnod of Fox & Kamprath (1970); b. Isotopically exchangeable P after 4 - h shaking period (see text) and c. Phosphate measured anion-exhange resin (see text).

diffusion of phosphate to the root. Immobilization reduces the amount of phosphate which is in the labile form, and the reaction appears to be reversible (Owusu-Bennoah & Wild 1980), but the factors determining the relative rates of the immobilization and re-mobilization in different soils are not understood. Because of the high amounts of phosphate required by most of the soils of the South American "cerrado", both reactions need further investigation.
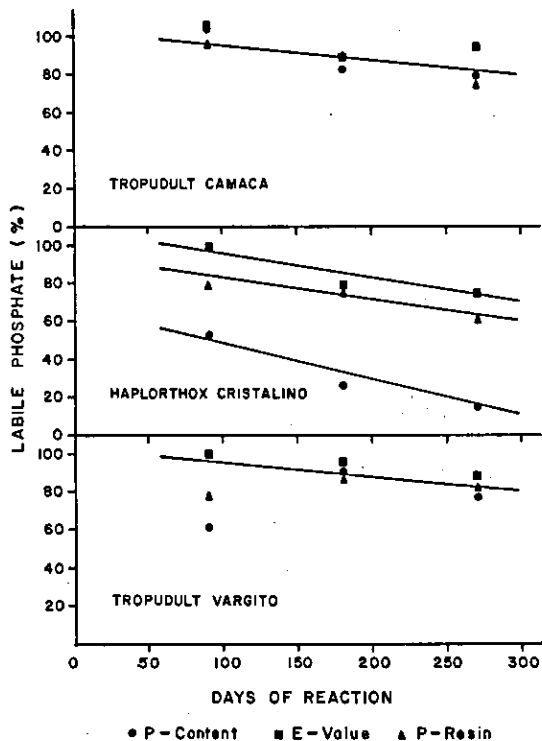

FIG. 5. Relationship between labile phosphate and time of reaction with added phosphate, measured by phosphate uptake by sorghum, phosphate extracted by anion-exchange resin and as isotopically exchangeable phosphate. Labile phosphate is expressed as a per cent of the value at time zero.

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