

Influence of Humic Substances on P-sorption in Some Malaysian Soils under Rubber

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Humic and fulvic acids of five soils under rubber were separated and purified. The humic substances were found to have a wide range of elemental composition. Treatment of humic acid with Al or Fe resulted in higher absorption of P by the acid. However, at higher levels of Al or Fe, the level of P associated with humic acid was somewhat lower suggesting that P in the humic acid could be held by physical adsorption forces of the organo-metallic complexes.

Sodium humate had a greater tendency to extract more P, Al and Fe from the soil than sodium fulvate. Treatment of oxidised soils with humic substances led to lower P-fixation by the soil.

The data showed that humic substances played an important role in decreasing the phosphate-fixing capacity of the highly weathered Malaysian soils.

Application of phosphorus fertiliser during the immature and mature phases of rubber has been known to lead to an accumulation of phosphorus in the soils^{1,2}. The soil-P can exist in two major fractions, viz. the organic or inorganic fractions. Lau *et al.*³ found that contribution to the total P 'reserve' in the soils by humic and fulvic acids amounted to about 32 kg P₂O₅/ha. Further studies showed that the phosphorus occurs as Fe or Al organo-metallic complexes which could affect the pool of labile P for plant uptake. Pushparajah *et al.*² assessed the residual effect of applied phosphates on performance of rubber and found that this was influenced by the presence of Al-, Fe- and Ca-bound phosphates in the soils.

Humic and fulvic acids are believed to play an important role in the phosphorus nutrition of plants. Sinha⁴ observed that organo-metallic phosphates of humic and fulvic acids possess varying degrees of solubility and mobility. The relatively mobile fulvic acid-metal phosphate compounds are more available to plants. Elsewhere, studies on the effect of organic matter on the sorption capacity of the soil

for phosphorus were reported by Perrott⁵, Appelt *et al.*⁶ and Leaver and Russell⁷. Lau *et al.*⁸ observed that organic matter generated by leguminous covers enhanced N and P status in the soil more significantly than those derived from non-leguminous covers. There seems therefore a strong basis to examine in greater detail the influence of the humic fraction of soils on P availability.

Consequently, this study investigates the effects of humic and fulvic acids, with Fe and Al on the fixation of phosphorus in five common soils under rubber of contrasting pedological characteristics and attempts to elucidate the practical significance of their relationships on the phosphorus nutrition of rubber.

EXPERIMENTAL

Soil Sampling

Surface soils (0-15 cm depth) sampled at ten random points along the inter-row areas of rubber which earlier were under various types of vegetative covers were bulked. Phosphorus

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as rock phosphate had been applied to the area at the rate of about 500 kg/ha over a period of one year during which the covers were established. The soils were air-dried (28°C) and ground to pass through a 2 mm sieve. Relevant physico-chemical properties of the soils studied (<2 mm) are given in *Table 1*.

Extraction and Purification of Humic Materials

Fulvic and humic acids in the five soils were extracted and purified according to the procedure reported by Lau *et al.*³ Approximately 1 kg of soil was extracted with 20 litres of 0.2 M NaOH at room temperature. Humic and fulvic acids in the extractant were separated, purified, freeze dried and kept for the P-sorption studies.

Effect of Fe and Al on the Absorption of P by Humic Acid

About 0.2 g purified humic acid in 50 ml of a solution of Al or Fe was agitated slowly. The solutions of Al and Fe contained approximately the range of Al and Fe contents found in the crude organic acids (3.4%–6.8% and 7.9%–15.8% by weight of HA for Al and Fe, respectively). After a period of about 72 h, the suspension was centrifuged and the supernatant discarded. The humic acid was washed once with distilled water and transferred to a dialysis bag with about 75 ml of distilled water. The dialysis bag, sealed at both ends, was suspended in a solution containing about 50 µg P/ml until there was no change in the concentration of P in the solution. After about four days when equilibrium was attained, the humic acid was separated as before and analysed for P, Al and Fe.

Dissolution of Soil Adsorbed P by Humic Substances

Organic matter in the five soils was removed by continuous hydrogen peroxide oxidation until no further reaction was observed. The oxidised soils after repeated washing with distilled water were then successively equilibrated with a solution of sodium phosphate containing 50 µg P/ml at pH 4.5 until maximum P adsorp-

tion was attained. The soils were separated by centrifugation, washed with distilled water and air-dried at room temperature.

The influence of humic and fulvic acids (HA and FA) on the dissolution of adsorbed P in the soils were determined by gently shaking 10 g of the P-saturated soil with 50 ml of a solution of sodium humate (NaHA) or sodium fulvate (NaFA) containing 5–20 µg HA or FA/ml at pH 4.5. The amount of HA and FA in the solutions corresponded with the range of pure HA and FA associated with the soils. After 3 h, the soil solution mixture was separated by centrifugation. The concentration of P together with Al and Fe in the supernatant solution was determined. The amount of P dissolution from the soil corresponds to the difference between the initial and final P concentrations of NaHA or NaFA solutions. A total of three extractions was carried out and the total P, Fe and Al determined. The same procedure was repeated on unoxidised soils which were treated as before.

Absorption of P by Oxidised Soils after Pre-treatment with HA and FA

10 g of oxidised soil prepared in the manner described was successively shaken with 50 ml of a solution of NaHA or NaFA for about three days. Solutions containing 5 µg/ml and 20 µg/ml HA or FA and maintained at pH 4.5 were used. 2 g of the soil, which had been separated by centrifugation and washed with distilled water were then shaken with 50 ml of solution containing 20 µg P/ml. The amount of P adsorbed by the soil was determined by the loss of P from the solution.

Analysis of Soil and Solutions

Total nitrogen, organic carbon and phosphorus contents in the soil were determined by the normal micro-Kjedahl digestion method, Walkley and Black's titration method and HClO₄/H₂SO₄ acid digestion, respectively¹⁰. Soluble P was determined by Bray and Kurtz No. 2 method¹¹, while exchangeable Al was determined by M KCl extraction¹⁰. The amount of phosphorus, aluminium and iron in the

TABLE 1. RELEVANT PHYSICO-CHEMICAL PROPERTIES OF THE SOILS STUDIED

Soil series	Sub-group ^a (parent material)	Mechanical composition ^b				pH ^c	Organic carbon (%)	P ($\mu\text{g/g}$ soil)		Al [C _{mol} (+) kg ⁻¹ soil]	Humic ^d acid (g/100 g soil)	Fulvic ^d acid (g/100 g soil)
		Coarse sand	Fine sand	Silt	Clay			Soluble	Total			
Durian	Orthoxic tropudult (siliceous shale)	8.9	12.2	34.8	44.1	3.8	1.48	28	120	65 (74)	0.34	0.88
Munchong	Tropeptic haplorthox (shale)	15.1	11.3	16.7	56.9	4.1	2.42	24	605	48 (66)	0.26	0.97
Rengam	Typic paleudult (granite)	30.3	12.3	5.8	51.6	4.0	1.62	20	219	38 (90)	0.29	1.19
Serdang	Typic paleudult (sandstone)	42.5	37.3	4.8	15.4	4.2	1.13	32	160	42 (95)	0.30	0.93
Kuantan	Haplic acrothox (basalt)	8.4	8.8	24.4	58.4	4.5	2.63	42	341	42 (61)	0.25	1.23

Figures within brackets indicate exchangeable Al as percentage of cation-exchange capacity.

^aClassification according to USDA Soil taxonomy⁹ at sub-group level

^bExpressed as percentage of soil

^cIn 0.01M KCl at soil to solution ratio 2.5

^dEstimated

NaHA and NaFA extracts were determined after a suitable aliquot of the extract or amount of the organic acid was dry ashed, ignited and dissolved in dilute nitric acid.

RESULTS AND DISCUSSION

Mechanical composition, organic carbon content, soluble and total P in the five soils showed contrasting differences (Table 1). The Kuantan, Durian and Munchong series soils contained a large proportion of silt and clay while the other soils were texturally sandy in nature. Total P and organic carbon content were highest in Munchong and Kuantan series soils and lower in Durian, Serdang and Rengam series soils. pH of the soils fall within a narrow range of 3.8-4.5. The low pH and high exchangeable Al values indicate that the soils are highly weathered and acidic in characteristic. Owen^{12,13}, found that these soils have very high phosphate-fixing capacity and that a sizeable fraction of soil-P was not readily available for plant uptake. Generally, the soils have more fulvic acid (FA) than humic acid (HA). The high FA/HA ratios in the soils suggest that most of the organic matter in the soils had undergone a high degree of humification. Soong¹⁴ and Lau *et al.*⁸ observed that these actions affected the physical properties of the soils and the availability of soil nitrogen and phosphorus.

Elemental composition of humic and fulvic acids extracted from the five soils showed wide variability (Table 2). The crude organic products are highly dominated by Fe and Al. Upon purification, the P, Fe and Al contents were

considerably reduced and these reductions were much larger than those of C and N. The simultaneous reduction of P, Fe and Al during the purification processes suggested that P in HA and FA are closely correlated with its sesquioxide content. Using labelled P, Larsen *et al.*¹⁵ reported that the presence of Fe and Al in HA greatly increased P-fixation and there was no apparent interaction of Fe with Al on the fixed P.

Effect of Fe and Al on P Absorption by HA

The absorption of P by HA is shown in Table 3. Generally, the addition of Fe and Al resulted in higher absorption of P by HA. Of the two cations, higher absorption occurred in HA treated with Al. This trend was also reported by Chang and Chu¹⁶ who showed that application of soluble phosphates to soils favoured the fixation of P as Al-P rather than Fe-P. The exact manner in which the HA interacted with Al and P in the system could not be established in this study.

The amount of P, Fe and Al associated with the Al- and Fe-treated HA extracted from different soils appeared to be variable. Such variations could be attributed to the differing properties of HA found in the different soils which are under different types of vegetative covers. Norhayati¹⁷ attempted to characterise HA and FA obtained from some Malaysian soils and found that these humic substances have different complexation capacities. Mean complexation capacities were found to be in the order of $9.0 \times 10^{-4} M$ and 5.08×10^{-4} per

TABLE 2. MEAN NITROGEN, PHOSPHORUS, IRON AND ALUMINIUM CONTENTS OF HUMIC AND FULVIC ACIDS^a

Acid	C	N	P	Fe	Al
Humic acid					
Crude	42.3 ± 12.3	3.8 ± 1.6	1.80 ± 0.81	8.7 ± 5.2	9.6 ± 4.3
Purified	32.4 ± 8.1	2.7 ± 1.1	0.27 ± 0.06	0.29 ± 0.09	0.40 ± 0.06
Fulvic acid					
Crude	40.8 ± 10.7	5.5 ± 2.4	1.43 ± 0.61	7.2 ± 3.8	8.4 ± 3.9
Purified	38.1 ± 4.2	4.1 ± 1.7	0.19 ± 0.07	0.13 ± 0.11	0.18 ± 0.10

^aValues expressed as percentage of organic acid content

TABLE 3. EFFECT OF IRON AND ALUMINIUM ON THE ABSORPTION OF PHOSPHORUS BY HUMIC ACID

Treatment of HA	Total P	Fe Amount (mg/100 g HA)	Al	ΔP	ΔFe Amount (mg/100 g HA)	ΔAl
Durian series						
Untreated	190	167	385	—	—	—
Al added (%) : 3.4	530	—	410	340	—	25
6.8	560	—	527	370	—	142
Fe added (%) : 7.9	380	230	—	190	63	—
15.8	420	221	—	230	54	—
Munchong						
Untreated	200	263	340	—	—	—
Al added (%) : 3.4	760	—	436	560	—	96
6.8	740	—	487	540	—	147
Fe added (%) : 7.9	700	300	—	500	37	—
15.8	610	356	—	410	93	—
Rengam						
Untreated	260	210	437	—	—	—
Al added (%) : 3.4	960	—	518	700	—	81
6.8	920	—	589	660	—	152
Fe added (%) : 7.9	880	288	—	620	78	—
15.8	430	308	—	170	98	—
Serdang						
Untreated	330	300	521	—	—	—
Al added (%) : 3.4	820	—	568	490	—	47
6.8	820	—	600	490	—	79
Fe added (%) : 7.9	540	347	—	210	47	—
15.8	490	400	—	110	100	—
Kuantan						
Untreated	380	505	348	—	—	—
Al added (%) : 3.4	890	—	487	510	—	139
6.8	920	—	528	540	—	180
Fe added (%) : 7.9	840	520	—	460	15	—
15.8	800	528	—	420	23	—

ΔP Increase in P content

ΔAl Increase in Al content

ΔFe Increase in Fe content

(%) As percentage of HA used

gramme of acid for HA and FA, respectively. Lau *et al.*³ also reported that elemental compositions of humic substances from soils under different ground covers were different. Of the five soils studied, HA from the Durian series soil seemed to contain the least absorbed P, Fe and Al. In this instance, the types of ground covers established in the immature phase of rubber had a profound influence on the characteristics of the HA.

As the levels of Fe and Al added to HA are doubled, P associated with HA is not significantly increased. The mode of action of HA resulting in lower P absorption at higher Fe or Al contents is difficult to account for. However, it can be reasoned that the HA forms complexes with Al or Fe and the increase in P (ΔP) in the HA is attributed to the physical adsorption forces of the complexes. If such is the case, the amount of P in HA would be determined by the surfaces of the HA-Al or Fe complexes rather than the concentration of Fe or Al. These observations conformed with the work of Tschapek and Sakun¹⁸ who found that there was no loss of cation-exchange capacity when HA and clay suspension were mixed and interaction of cations and HA-clay suspensions were through physical attractive forces.

Dissolution of Soil-P

The effectiveness of NaHA and NaFA on the dissolution of soil-P was studied. The amount of soil-P extracted by two levels of NaHA and NaFA as well as by the conventional Bray and Kurtz No. 2 method ($\text{NH}_4\text{F}/\text{HCl}$) are shown in *Table 4*. For all soils, NaHA appeared to extract more P from the oxidised and unoxidised soils. The amounts of Al and Fe in the NaHA extracts were also higher. The tendency of NaHA to extract more P, Al and Fe over NaFA reflected the greater tendency of HA to form soil organo-metallic complexes and the consequent release of nutrient ions. The trends that occur in all the soils lend support to this explanation, although more data are required for its confirmation.

The Bray and Kurtz No. 2 method extracted much more P, Fe and Al when compared with the NaHA and NaFA extractions (*Table 4*). The ability of the Bray extractants to extract more P is due to the high affinity of the fluoride ions to complex with Fe^{3+} or Al^{3+} in an acid environment resulting in the release of phosphate ions into the solution. Although HA and FA form complexes with Fe and Al in the soil, its mode of action may not be similar to that of fluoride. Release of soil-P as a result of interaction of the organic materials with sesquioxides (Al and Fe) in the soil would thus be less effective. Oxidised soils generally have high $\text{NH}_4\text{F}/\text{HCl}$ soluble P suggesting that P is associated with the organic matter and is thus less available. Soil-P is released when the organic matter is mineralised or decomposed.

HA and FA on P-sorption

The effects of HA and FA on the absorption of P by the five soils were further studied. Oxidised soils pre-treated with NaHA and NaFA were shaken with solutions of phosphate. The amount of P absorbed by the soils are given in *Table 5*. At the higher level of treatment (L_2), all the soils showed negative absorption. The Durian series soils appeared to be the most affected and this was followed by the Munchong and Serdang series soils. The effects of HA and FA on soil chemical and physical properties have been established^{4,7,14}. Soong¹⁴ found that HA and FA together with sesquioxides had a profound effect on micro-aggregate formation. He further established the fact that aggregate formation by FA is more efficient than that by HA. Since aggregate formation would tend to lower the exposed surfaces of the soil, phosphate sorption by the soil would considerably be reduced also.

Despite its high silt and clay contents, the effect of HA and FA on Kuantan series soil is not so pronounced when compared with that on Durian series soil. This observation seemed to indicate that the oxidation process of using hydrogen peroxide may not be adequate to completely destroy all the organic matter present in Kuantan series soil. Compared with other soils, Soong¹⁴ noted that soil aggregates

TABLE 4. EXTRACTION OF PHOSPHORUS, IRON AND ALUMINIUM IN THE SOILS BY SODIUM HUMATE AND SODIUM FULVATE

Soil series	Treatment	NaHA			NaFA			NH ₄ F/HCl				
		P	Fe	Al	P	Fe	Al	P	Fe ^a	Al ^a		
Durian	Oxidised	L ₁	68.3	4.9	2.4	52.1	4.2	2.0	375	433	239	
		L ₂	75.7	5.2	2.9	60.9	4.4	2.2	418			
	Unoxidised	L ₁	30.6	2.9	2.6	29.3	2.4	2.4	288	420	207	
		L ₂	38.9	3.0	2.5	35.6	2.0	2.6	257			
	Munchong	Oxidised	L ₁	58.6	5.2	3.0	56.7	3.6	3.0	268	467	382
			L ₂	60.7	5.6	3.2	57.4	3.8	3.0	310		
Unoxidised		L ₁	20.4	3.0	2.8	34.5	3.2	2.6	243	405	400	
		L ₂	25.8	4.1	2.9	30.3	2.4	2.3	277			
Serdang		Oxidised	L ₁	50.2	3.7	4.1	42.2	2.6	3.5	270	391	481
			L ₂	47.1	3.6	4.8	38.8	3.1	3.6	326		
	Unoxidised	L ₁	13.0	2.5	2.6	13.0	1.7	3.8	281	342	456	
		L ₂	15.0	2.4	3.0	10.0	1.7	3.2	307			
	Rengam	Oxidised	L ₁	58.2	2.1	2.4	47.4	2.2	2.5	360	387	397
			L ₂	54.4	2.8	2.8	40.0	1.9	2.8	391		
Unoxidised		L ₁	9.7	1.7	2.6	11.0	1.1	2.4	259	221	410	
		L ₂	12.3	1.4	2.1	12.0	0.0	2.2	275			
Kuantan		Oxidised	L ₁	87.5	3.8	2.7	88.7	4.2	2.2	391	428	318
			L ₂	107.3	4.3	2.5	93.0	3.8	2.5	402		
	Unoxidised	L ₁	28.4	2.8	2.3	34.3	2.2	1.9	293	397	282	
		L ₂	39.0	3.0	1.9	29.7	2.3	1.8	333			
	Mean		43.5	3.4	2.8	40.8	2.7	2.6	314	379	179	

The amounts of P, Fe and Al are given in $\mu\text{g/g}$ soil.

^aValues given are means of L₁ and L₂.

L₁ : 5 μg HA or FA/ml

L₂ : 20 μg HA or FA/ml

from the Kuantan series soil suffered less destruction on treatment with H₂O₂. Humic materials in this soil may have characteristics that are quite different from those found in other soils.

CONCLUSION

The five soils studied contain variable amounts of humic and fulvic acids. The crude humic materials are high in Al and Fe contents.

TABLE 5. ABSORPTION OF PHOSPHORUS AFTER PRE-TREATMENT OF OXIDISED SOILS WITH SODIUM HUMATE AND SODIUM FULVATE

Soil series	Control	P in soil ($\mu\text{g/g}$ soil)				Percentage increase/decrease in P absorbed ^a			
		NaHA		NaFA		NaHA		NaFA	
		L ₁	L ₂	L ₁	L ₂	L ₁	L ₂	L ₁	L ₂
Durian	76.3	73.2	69.4	78.1	66.8	-4.0	-9.0	+2.4	-12.5
Munchong	52.2	50.8	48.7	49.7	46.6	-2.7	-6.7	-4.8	+10.7
Rengam	63.1	65.6	60.9	62.5	61.0	+4.0	-3.5	-1.0	-3.3
Serdang	83.5	80.9	78.4	84.5	74.3	-3.1	-6.1	+1.2	-11.0
Kuantan	58.6	60.1	56.7	58.3	56.0	+2.6	-3.2	-0.5	-4.4

^a + denotes gain in P in solution

- denotes loss in P from solution

Reduction in P content during the purification process corresponds with lower Al and Fe contents. The amount of P absorbed by humic acid increased with the amount of Al added to the acid. Compared with Fe, Al is more effective in increasing the retention of P by HA. It is observed that P in the humic acid-Al and Fe complex is held by the physical adsorption forces of the complexes as the amount absorbed is found to be independent of the amount of Al or Fe added. Soluble P determined by NaHA extractant is greater than that by NaFA suggesting the greater tendency of HA to form soil organic complexes resulting in release of P. Oxidised soils when treated with HA and FA appeared to adsorb less P.

The dominance of Al and Fe in highly weathered Malaysian soils has resulted in the very high phosphate-fixing capacity of the soils. The current investigations showed the importance of humic substances in determining the P-sorption characteristics of the soils. The establishment of good ground covers and the proper use of mulching techniques will enhance the generation of organic matter which on humification will have a beneficial effect on P availability in the soils. Particularly in soils with very high sesquioxide content, the formation of organo-metal phosphates and the subsequent release of P upon decomposition would be an added advantage.

ACKNOWLEDGEMENT

The author wishes to thank the Director of the Rubber Research Institute of Malaysia for permission to publish this paper and the Head of Soils and Crop Management Division, Dr Chan Heun Yin for his valuable comments and suggestions. Thanks are also due to my colleagues, Drs Norhayati Moris and Wong Chaw Bin for helpful discussion during the preparation of this manuscript. Appreciation is also extended to Encik Thomas Kovil Pillay for analytical work and Encik Thea Ah Kow for collecting the soil samples.

Date of receipt: November 1988

Date of acceptance: April 1989

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