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CHARACTERISTICS OF PHOSPHATE ROCK MATERIALS FROM CHINA, INDONESIA AND TUNISIA AND THEIR DISSOLUTION IN INDONESIAN ACID SOILS

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ABSTRACT

Dissolution of phosphate rock (PR) in soils is a primary concern for P in the PR to be available for plant. The dissolution of three PR materials, China (CPR), Ciamis (IPR) and Gafsa (GPR), in eight acid Indonesian soils (pH in water 4.1-5.7) was tested in a closed incubation system. Experiment was conducted in Soil Chemical Laboratory, Universiti Putra Malaysia and Indonesian Center for Agricultural Land Resources Research and Development from January to April 2002. The dissolution was determined from the increase in either 0.5 M NaOH extractable P (ΔP) or 1 M $BaCl_2$-triethanolamine (TEA)-extractable Ca (ΔCa) in soils amended with PR compared with control soil. Dissolution of the IPR was the highest (30-100%) followed by GPR (17-69%) and then by CPR (20-54%). The maximum dissolution followed the order: Bogor Ultisols > Bogor Oxisols > Subang Inceptisols > Bogor Inceptisols > Sukabumi Oxisols > Lebak Ultisols > Sukabumi Inceptisols > Lampung Ultisols. PR dissolution indicated a positive correlation with P retention capacity. The results implied that the extent of PR dissolution for the three PR sources (China, Indonesia and Tunisia) increased with increasing P retention capacity of the soils. PR dissolution can be based on a calibration curve of ΔCa meaning that if ΔP is high then the amount of PR dissolution measured by ΔCa in PR materials is also high.

[***Keywords***: Phosphate rock, acid soils, chemicophysical properties, dissolving]

INTRODUCTION

Dissolution of phosphate rock (PR) in soil is prerequisite for the P in PR to become available to plants. Several factors which influence the dissolution of PR in soils are chemical composition, particle size of the PR, and soil characteristics, such as pH, $H_2PO_4^-$ and Ca^{2+} (Mackay *et al*. 1986; Hanafi *et al*. 1992).

PR dissolution in acid soils can be represented as follows (Khasawneh and Doli 1978):

$$Ca_{10}(PO_4)_6F_2 + 12H^+ \Rightarrow 10Ca^{2+} + 6H_2PO_4^- + 2F^- \quad [1]$$

This equation indicates that the rate of dissolution is determined by the concentration of protons (H^+) and the concentration of reaction products of Ca^{2+} and $H_2PO_4^-$.

Mackay *et al*. (1986) using contrasting soils found that PR dissolution increases as exchangeable Ca decreases and as the P sorption capacity of the soil increases. Several authors (Chien *et al*. 1980; Smyth and Sanchez 1982; Kanabo and Gilkes 1987) have also reported that PR dissolution increases with P sorption capacity of the soil, thus maintaining a low soil P level and favoring reaction of PR dissolution. Kumar and Mishra (1986) and Hanafi *et al*. (1992) suggested that P fixation or P retention capacity of soil strongly influences PR dissolution in soils. Organic carbon, exchangeable Ca, and soil pH (negatively correlated) were found to be subsidiary factors (Khasawneh and Doll 1978; Kanabo and Gilkes 1987). In addition, soil Ca, soil P, soil texture, and soil organic matter content are also factors affecting PR dissolution in soils. Furthermore, in the laboratory incubation study Hanafi *et al*. (1992) found that P retention capacity, and P and Ca sink sizes are important variables in influencing the dissolution of Gafsa PR (GPR) and Christmast Islands PR (CIPR).

Indonesia is a large importer and consumer of PR, however information on chemical and physical properties of PR and soils as well as the reaction of PR with acid soils is lacking. PR is usually used as a raw material in the superphosphate fertilizer industries or for direct application on food and estate crops. The objective of this study was to compare the dissolution of three PR materials on eight Indonesian acid soils with different P retention capacities in a closed incubation system.

MATERIALS AND METHODS

This experiment was conducted in Soil Chemical Laboratory of Universiti Putra Malaysia and Indonesian Center for Agricultural Land Resources Research and Development from January to April 2002.

Phosphate Sources

Three PR materials were used, i.e. CPR from China, GPR from Tunisia, and IPR from Ciamis Indonesia. These materials were ground to pass the 149 mm sieve. The total P and Ca were determined by a tri-acid digestion method (O'Connor and Syers 1975). Solubility of PR in water, in 2% (w/w) citric, and in 2% formic acids were assessed according to the method of Association of Official Analitical Chemists (1980) and method developed by Robinson and Syers (1991), respectively.

Soils and Incubation System

Eight soils, i.e. Lampung Ultisols, Bogor Ultisols, Bogor Oxisols, Bogor Inceptisols, Sukabumi Inceptisols, Sukabumi Oxisols, Lebak Ultisols, and Subang Inceptisols having various P-retention capacities were used (Table 1). Surface soil samples (0-20 cm depth) were collected from various locations in Lampung, Banten, and West Java provinces of Indonesia. The soils were air dried, sieved (< 2 mm) and taken to the soil laboratory before use. The chemical properties of soil determined were organic carbon content (Walkey and Black 1934), pH in water with a soil/ solution ratio of 1:5, P retention capacity (Saunders 1965), Ca-exchange capacity by extraction with 0.10 M KNO_3 of the Ca sorbed from 0.025 M $CaCl_2$ (Mackay *et al.* 1986), estimation of plant available P by extraction with 0.50 M $NaHCO_3$ (Olsen *et al*. 1954), and pH buffering capacity by pH titration (Kanabo and Gilkes 1987).

The CPR, GPR, and IPR were added to separate plastic containers containing 200 g of air dried soil at a rate of 500 mg P kg^{-1} soil, thoroughly mixed and incubated at 25°C. Duplicate samples of each P fertilized soil were removed for analysis at 0, 1, 4, 10, 20, 35, 55 and 90 day after P addition. Analysis was conducted on moist soil at 90% of field capacity. The extent of dissolution of CPR, GPR, and IPR was measured from the change of P dissolution (ΔP) (Mackay *et al*. 1986) and change in Ca dissolution (ΔCa) (Bascomb 1964) in soil amended with PR compared with untreated soil controls (Hanafi *et al*. 1992). The ΔP or ΔCa is the difference in P or Ca content between PR treatment and control (without PR soil). Inorganic P in NaOH extracts was determined using the procedure of Murphy and Rilay (1962); Ca in 1 M $BaCl_2$-TEA extracts were determined by atomic absorption spectrophotometry after the addition of 1000 mg Sr L^{-1} to suppress chemical and ionization interferences in the flame.

RESULTS AND DISCUSSION

Chemical Characteristics of Eight Selected Soils

The soils taken from different locations had different physical and chemical characteristics (Table 1). The texture was clayey, except for Lampung Ultisols which was loamy clay. There was considerable variation in the clay content, i.e. Bogor Oxisols had the greatest clay content (93%), and Lampung Ultisols had the lowest (43%).

Most soils were low in organic C (< 20 g kg^{-1}), except the Sukabumi Inceptisols (26 g kg^{-1}). The soil pH (H_2O) ranged from 4.1 (Bogor Inceptisols) to 5.7 (Lampung Ultisols). With the exception of Sukabumi Inceptisols (pH = 5.6) and Lampung Ultisols (pH = 5.7), the soils were considerably strongly acidic soils (pH < 5.0). Soils that have pH < 6.0 are generally less suitable for field crops (Prasad and Power 1997).

Table 1. Selected physical and chemical properties of the Indonesian soils.

Soils	Subgroup	Clay (%)	Texture	pH in H_2O	pH-buffering capacity (mmol OH kg^{-1} pH^{-1})	Org. C (g kg^{-1})	Ca-EC ($mmol_c$ kg^{-1})	Exch. Ca* ($mmol_c$ kg^{-1})	P-retention capacity (%)	Olsen P (mg kg^{-1})
Bogor Ultisols	Typic hapludults	77	Clay	4.40	20.50	6.90	156.60	21.60	82	3.50
Sukabumi Oxisols	Typic hapludox	83	Clay	5.00	19.30	14.70	88.00	44.50	65	6.60
Bogor Oxisols	Typic hapludox	93	Clay	4.90	30.90	15.00	65.20	21.00	61	13.60
Bogor Inceptisols	Typic dystropept	57	Clay	4.10	64.20	7.00	61.30	6.70	60	6.10
Lebak Ultisols	Typic hapludults	65	Clay	4.40	137.60	2.70	75.00	15.00	55	10.30
Sukabumi Inceptisols	Typic dystropept	87	Clay	5.60	17.20	26.10	98.90	91.10	59	14.90
Subang Inceptisols	Typic dystropept	67	Clay	4.50	29.20	12.10	98.50	50.00	49	18.90
Lampung Ultisols	Typic hapludults	43	Loamy clay	5.70	24.90	6.90	96.20	81.40	24	5.70

*1M NH_4O Ac pH 7 extractable (Piper 1947).

Soil pH buffering capacity is the soil systems that maintain pH within a narrow range to resist a change in pH upon the addition of a small amount of acid or base. Among the tested soils, Lebak Ultisols (138 mmol OH kg^{-1} pH^{-1}), Bogor Inceptisols (64 mmol OH kg^{-1} pH^{-1}), and Bogor Oxisols (31 mmol OH kg^{-1} pH^{-1}) had a high pH buffering capacity, while Sukabumi Inceptisols (17 mmol OH kg^{-1} pH^{-1}) and Sukabumi Oxisols (19 mmol OH kg^{-1} pH^{-1}) had a low pH buffering capacity.

Bogor Ultisols exhibited the highest calcium exchange capacity (Ca-EC) (157 $mmol_c$ kg^{-1} soil), followed by Sukabumi Inceptisols (99 $mmol_c$ kg^{-1} soil), Subang Inceptisols (98 $mmol_c$ kg^{-1} soil), and Lampung Ultisols (96 $mmol_c$ kg^{-1} soil). However, both Sukabumi Inceptisols (91 $mmol_c$ kg^{-1} soil) and Lampung Ultisols (81 $mmol_c$ kg^{-1} soil) had a high exchangeable Ca. This may contribute to the values for Ca sink size (Table 1). In combination with high pH, low Ca sink size may create unfavorable conditions for PR dissolution.

The P retention capacity measures the amount of vacant P sorption sites in soils and controls the concentration of P in soil solution (Mackay *et al*. 1986). The P retention capacity varied greatly with soil order. Bogor Ultisols had the highest P retention capacity (82%) while Lampung Ultisols had the lowest (24%). Available (Olsen extractable) P of most of these soils were low (< 10 ppm P) except for Bogor Oxisols (14 mg P kg^{-1}), Sukabumi Inceptisols (15 mg P kg^{-1}), and Subang Inceptisols (19 mg P kg^{-1}) which were moderate.

Chemical Characteristics of Phosphate Rock

Results for total P and Ca determined by a tri-acid digestion method (O'Connor and Syers 1975), and for P solubility in water and 2% citric and formic acids (Robinson and Syers 1991) are given in Table 2. PR materials from Ciamis Indonesia (IPR) had the highest total P and Ca content and the greatest solubility in 2% citric acid and 2% formic acid. Total P content of GPR was not significantly different from CPR (128 c.f 130 g kg^{-1}), however, the solubility of GPR in citric and formic acids was much higher. According to Hammond *et al*. (1986), IPR and GPR corresponded to a category of high reactive PR exhibiting high P solubility in citric acid (> 67 g kg^{-1}), while CPR exhibited medium reactivity (34-54 g kg^{-1}). The reactivity of these PR materials in order from highest to lowest is IPR > GPR > CPR.

Mineralogy of Phosphate Rock

Apatite was the main mineral in all PR used. Minerals of CPR, GPR, and IPR were substituted by francolite (carbonate apatite). The GPR and CPR deposits were derived from marine sedimentary phosphate, while IPR was mostly derived from guano phosphate. The mineralogical model analysis indicated that GPR is composed of about 83.8 wt w/w % francolite. The most abundant gangue mineral was calcite (5.5 wt w/w). Quartz (1.8 wt w/w) was the only other gangue mineral that could be detected (IFDC 1995). The IPR showed a peak of 2.85-39.80Åsuggesting the dominance of fluorapatite mineral, while CPR showed a sharp peak of 2.80-40.40Å and 3.33-33.70Å indicating that this PR material contained not only fluorapatite but also quartz minerals (Fig. 1).

Morphology of Indonesian Phosphate Rock

Microscopic (petrography) analysis indicated that IPR in the research area belonged to the clastic limestones group, which are formed under marine conditions. The IPR material may originate from the dissolution of guano phosphate or from volcanic materials as shown from the micrograph (Fig. 2). The figure indicated that IPR material is formed by oolitic and physolitic textured, concentric layered, white and pale grey dahllite mineral (60%), that is banded with a brown fibrous, radial structure, and a dark brown, isotropic collophane (40%), some of which has formed a matric mass.

Table 2. Selected characteristics of the phosphate rock materials from China, Indonesia, and Tunisia.

Phosphate rock materials	Total (g kg^{-1})		P extracted by (mg kg^{-1})		
	P	Ca	2% citric acid	2% formic acid	Water
China (CPR)	130	286	47	49	0.06
Gafsa Tunisia (GPR)	128	332	68	117	0.06
Ciamis Indonesia (IPR)	153	356	119	123	0.09

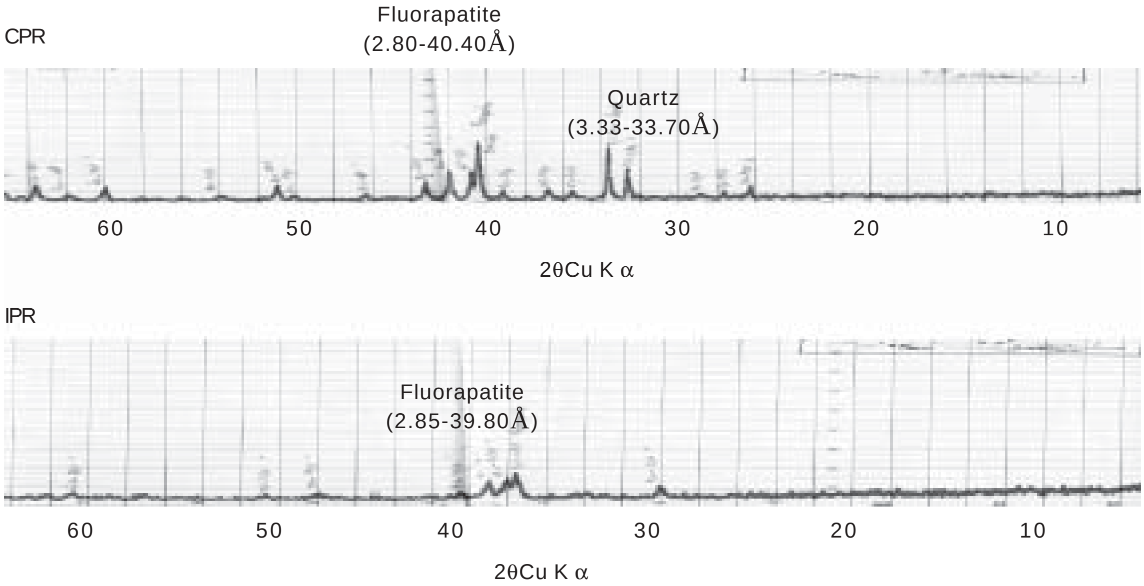

Fig. 1. The X-ray diffractogram of phosphate rock from China (CPR) and Indonesia (IPR); d spacing in Å.

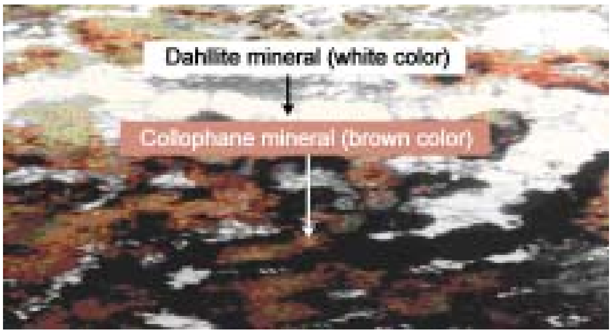

Fig. 2. Micrograph of the dahllite and collophane minerals in the phosphate rock materials from Ciamis Indonesia (IPR).

Changes in Phosphorus and Calcium in a Closed Incubation System

The change in P (ΔP), measured by NaOH, and Ca (ΔCa), measured by $BaCl_2$-TEA extractions (Fig. 3), initially increased with incubation time in all eight soils. In general, most dissolution occurred within few days up to 55 days, and afterwards it occurred at lower rate. The extent of PR dissolution for day one ranged from 3 to 23% for CPR, 20 to 74% for IPR, and 5 to 21% for GPR. After 90 days of incubation, PR dissolution ranged from 20 to 54% for CPR, 30 to 100% for IPR, and 17 to 69% for GPR.

The value of PR dissolution at 90 days observed in Bogor Ultisols, Bogor Oxisols, and Subang Inceptisols amended with IPR were much higher (reach 100% or 500 mg P kg^{-1} soil or more) than those of other treatments. These values may be due to: (1) much higher P retention capacity (82% for Bogor Ultisols and 61% for Bogor Oxisols), high soil acidity (pH = 4.4 for Bogor Ultisols, pH = 4.9 for Bogor Oxisols and pH = 4.5 for Subang Inceptisols), and high value of Ca sink size (for Subang Inceptisols); (2) the failure of the NaOH used in this study to act on all P originating from dissolved PR, as has been reported by earlier workers (Anderson *et al*. 1985; Samid 1990); and (3) the IPR used in this study being much more reactive than GPR and CPR (Table 2). Another possibility is that the moisture present in the soil was lower than the targetted 90% of field capacity, leading to overestimation of soil weight and P dissolution during extraction.

The functions of NaOH in this analysis were (1) to extract plant-available P which has been released from a PR, and (2) to minimize dissolution of the PR during extraction (Mackay *et al*. 1986). The dissolution data for PR indicated that both ΔP and ΔCa increased with incubation time. For CPR and GPR, the ΔCa method indicated a higher rate of dissolution than did the ΔP method (Fig. 3). This seemed to be related to the higher solubility of Ca compared to P and the presence of free $CaCO_3$ (H ≈ 5.30% in GPR) (Syers and Mackay 1986; Hanafi *et al*. 1992). It has been reported that Ca in free $CaCO_3$ is preferentially extracted by 0.50 M $BaCl_2$ -TEA during the ΔCa measurement and thus leads to overestimation of PR dissolution (Bolan and Hedley 1990).

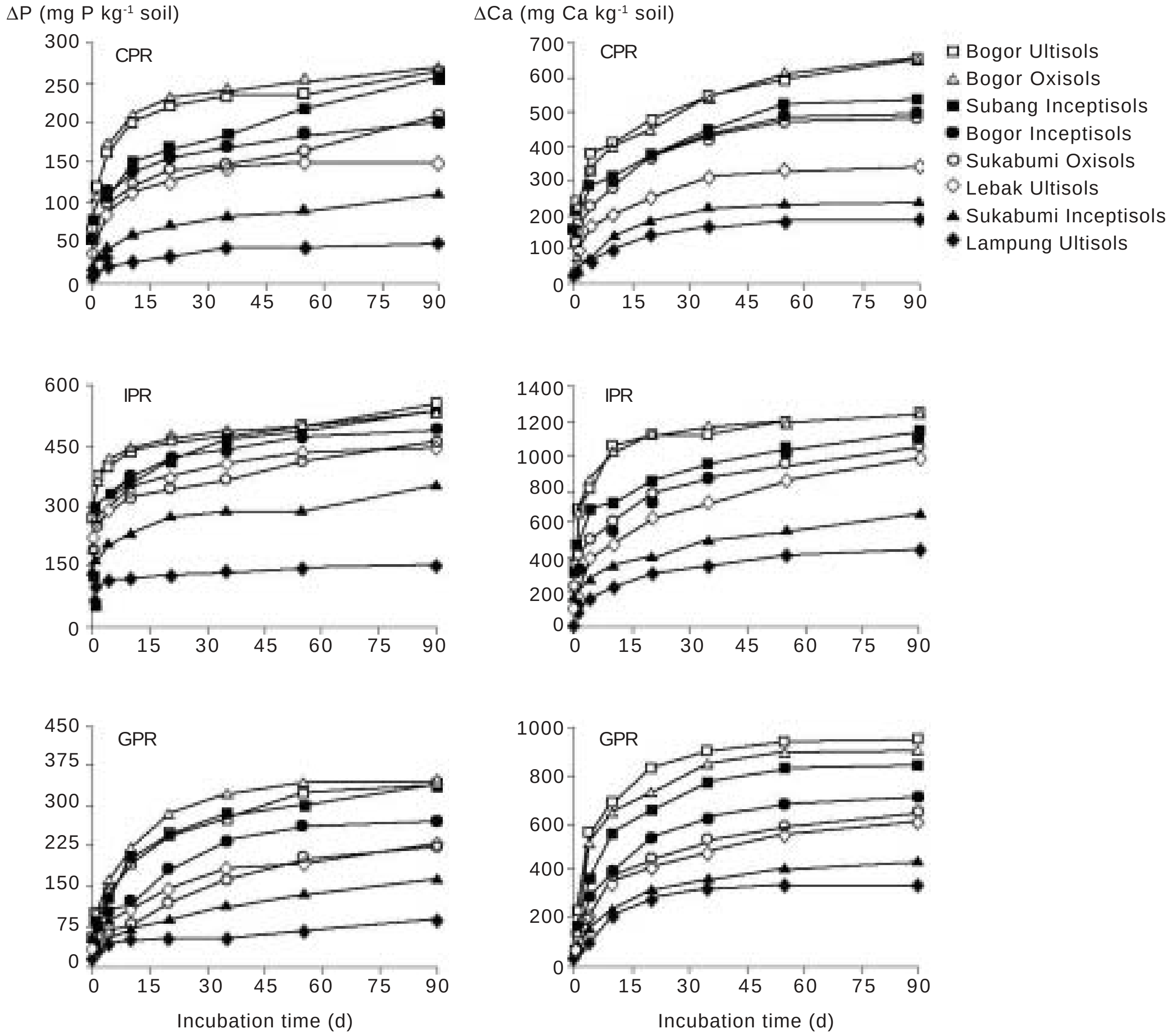

Fig. 3. Changes in P (ΔP) and Ca (ΔCa) in soils incubated with phosphate rock materials from China (CPR), Indonesia (IPR), and Tunisia (GPR) at 500 mg kg^{-1} soil in a closed incubation system.

Highly significant linear correlations ($P < 0.01$) were found between CPR ($R^2 = 0.49^{**}$), IPR ($R^2 = 0.63^{**}$) and GPR ($R^2 = 0.43^{**}$) dissolutions and P retention capacity (Fig. 4). This indicates that the rate of PR dissolution is dependent not only on the chemical solubility materials, but also on soil factors, such as P retention capacity. Soils with a high P retention capacity have been shown to provide a sink for P (Chien *et al*. 1980; Smyth and Sanchez 1982; Syers and Mackay 1986; Hanafi *et al*. 1992; Hanafi and Syers 1994). A highly positive relationship was also found between ΔP and ΔCa dissolution methods (Fig. 5), meaning that the PR dissolution measured by ΔP was also successfully predicted by ΔCa method, as indicated by the significant correlation coefficient for the regression equations.

No significant interaction between PR materials and soil type on the PR dissolution measured by ΔP method (Table 3). Independently, there is significantly different among PR materials on PR dissolution measured by ΔP methods. The highest (average) PR dissolution ($P < 0.01$) occurred with the addition of IPR (88%), followed by GPR (50%) and CPR (38%) (Table 3). These are consistent with the solubility or reactivity of these three PR materials (Table 2).

Independently, there was significant difference among soil type on PR dissolution measured by ΔP method. The average PR dissolution (ΔP) ($P < 0.01$) followed

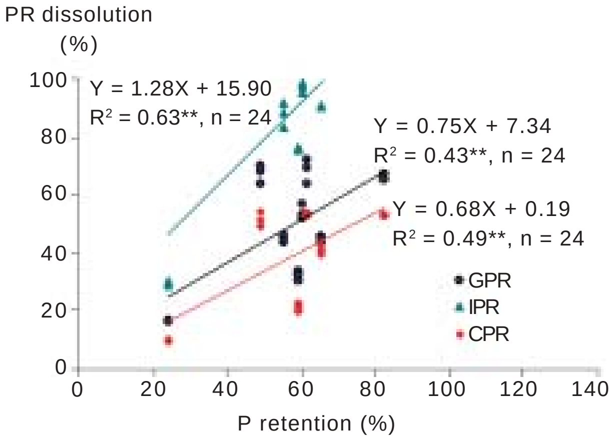

Fig. 4. Relationship between dissolution of phosphate rock materials from China (CPR), Indonesia (IPR), and Tunisia (GPR) and soil P retention capacity in a closed incubation system.

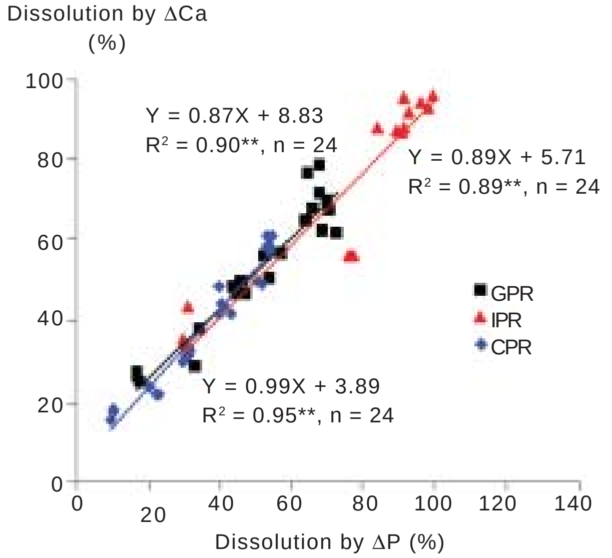

Fig. 5. Relationship between dissolution of phosphate rock materials from China (CPR), Indonesia (IPR), and Tunisia (GPR) estimated from ΔP and ΔCa in a closed incubation system.

the sequence: Bogor Oxisols > Bogor Ultisols > Subang Inceptisols > Bogor Inceptisols > Sukabumi Oxisols > Lebak Ultisols > Sukabumi Inceptisols > Lampung Ultisols (Table 3). This sequence is almost consistent with the P retention capacity of the soils (Table 1). Subang Inceptisols had a lower P retention capacity than Sukabumi Inceptisols or Sukabumi Oxisols. A lower pH of 4.50 (c.f. 5.60 or 5.00) and greater Ca-sink size/Ca-exchange capacity (48.50 c.f. 7.90 or 43.50 mmol_c kg^{-1}) contributed to high PR dissolution of the former compared to the later (Table 1). This suggests that besides P-retention capacity, acidity or pH and Ca-sink size had an interactive effect in influencing PR dissolution (Khasawneh and Doll 1978; Chien *et al*. 1980; Wilson and Ellis 1984; Robinson and Syers 1991).

Table 3. The mean value of ΔP and phosphate rock (PR) dissolution of eight acid Indonesian soils as affected by the addition of 500 mg P kg^{-1} soil PR materials at 90 days in a closed incubation system.

Treatment	ΔP (mg kg^{-1})	PR dissolution (% of total P added)
PR materials		
China (CPR)	189c	38
Indonesia (IPR)	439a	88
Tunisia (GPR)	248b	50
Soil type		
Lampung Ultisols	95f	19
Bogor Ultisols	384a	77
Bogor Oxisols	384a	77
Bogor Inceptisols	315b	63
Sukabumi Inceptisols	208e	42
Sukabumi Oxisols	296c	59
Subang Inceptisols	379a	76
Lebak Ultisols	274d	55

Mean values with different letters are significantly different ($P < 0.01$) by HSD or Tukey's test.

Proton supply is one of the factors that prevents the continuation of PR dissolution. Congruent dissolution of PR indicates that 2 mol of proton (H^+) are consumed by one mol of P released. As shown in equation 1, the rate of dissolution is determined by the concentration of protons (H^+) and the concentration of reaction products of Ca^{2+} and $H_2PO_4^-$. Based on this equation and laboratory analysis on IPR, between 9.74 and 35.46 mmol of H^+ are consumed to achieve the maximum dissolution (30-109.89%) observed with the eight soils (Table 4).

Phosphate rocks have more advantage to be applied in Indonesian acid soils (Santoso 1991) with high P fixing capacity compared to water soluble fertilizers because this materials could release P and Ca. The use of soluble P fertilizers such as TSP and SP-36 in these soils is inefficient and expensive due to low soil available P content and its capacity to fix P. The direct application of PR which can be of low grade and which often available locally can be suggested as a means to overcome the cost of water soluble fertilizers. The results obtained in this work implied that PR dissolution is related to soil properties such as P retention capacity and soil type.

Table 4. Amount of protons consumed and Ca exchange sites occupied by Ca at the maximum rate of Indonesian phosphate rock (IPR) dissolution in a closed incubation systems.

Soil	Maximum P dissolved (ΔP) (%)	H$^+$ consumed (mmol kg^{-1})	pH buffering capacity (mmol OH kg^{-1} pH^{-1})	Ca-sink size (CaEC-Exch. Ca) (mmol$_c$ kg^{-1})	Maximum Ca dissolved (ΔCa) (mmol$_c$ kg^{-1})	Ca-sink occupied (%)
Bogor Ultisols	109.89	35.46	20.50	135.00	31.59	23.40
Sukabumi Oxisols	91.20	29.42	19.30	43.50	26.22	60.28
Bogor Oxisols	108.07	34.86	30.90	44.20	31.07	70.29
Sukabumi Inceptisols	76.53	24.69	17.20	7.90	22.00	100.00
Subang Inceptisols	107.93	34.82	29.20	48.50	31.03	63.98
Lampung Ultisols	30.20	9.74	24.90	14.80	8.68	58.58
Bogor Inceptisols	97.73	30.89	64.20	54.60	28.10	50.40
Lebak Ultisols	88.67	28.60	137.60	60.10	25.47	42.41

CONCLUSION

The ΔP and ΔCa in soils incubated with PR were shown to be significant in most soils except in the Lampung Ultisols and Sukabumi Inceptisols. A high correlation was found between P retention capacity and PR dissolution (r = 0.66** to 0.80**) and between dissolution measured by ΔP and ΔCa methods (r = 0.94** to 0.97**).

Phosphate rock from Ciamis Indonesia (IPR) was found to be the most reactive PR with dissolution capacity of 30-100%. The dissolution capacity of the soils followed the sequence: Bogor Ultisols > Bogor Oxisols > Subang Inceptisols > Bogor Inceptisols > Sukabumi Oxisols > Lebak Ultisols > Sukabumi Inceptisols > Lampung Ultisols.

Information on P and Ca dissolution of PR as complementary studies would enhance the efficient use of the phosphate fertilizers in Indonesia. Dissolution of PR materials in acid soils requires further evaluation to determine their suitability as direct application fertilizers. Information on plant availability of P, residual effects of P derived from PR and its relative agronomic effectiveness (RAE) compared to TSP or SP-36 should be investigated in more detail.

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