# PHOSPHORUS FRACTIONS IN SOIL AMENDED WITH EMPTY FRUIT BUNCHES AND PHOSPHATE FERTILIZER – AN INCUBATION STUDY

ZULKIFLI, H\* and TARMIZI, A M\*

# ABSTRACT

In an incubation experiment, P was added to the Rengam Series soil through empty fruit bunches (EFB) alone (38 mg P kg<sup>-1</sup> soil), P fertilizer alone (900 mg P kg<sup>-1</sup> soil), and P fertilizer added together with EFB (938 mg P kg<sup>-1</sup> soil). The P fertilizers used were triple superphosphate (TSP), Gafsa (Tunisia) phosphate rock (GPR) and Christmas Island phosphate rock (CIPR). For each month during the six-month incubation, soil P fractions, namely iron oxide-coated strip P (Pi), Bray 2 P, NaOH P, occluded P, Ca P, organic P, microbial biomass P, total P, amount of P dissolved ( $\Delta$ P) from phosphate rock (PR), and exchangeable Ca were determined. The percentage dissolution of TSP, GPR and CIPR was 94.8%, 31.3% and 26.6% of added P, respectively, following the chemical reactivity of the materials. Addition of organic residues (EFB) only had a significant effect on the dissolution of CIPR. However, the results show a positive influence of organic residues added with P fertilizer which stimulated the formation of microbial biomass P (four times more than with P fertilizer added alone), organic P (about two times more than with P fertilizer added alone), and less movement into the inorganic P fractions. Addition of organic residues in dissolution of PR with time, over the six months of incubation, compared to when PR alone was added to the soil; this might be due to the additional sinks for P (biological transformation of P) and Ca (increase in Ca-exchange sites).

Keywords: phosphate rocks, oil palm, soil phosphorus fractions.

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# **INTRODUCTION**

Malaysian soils often have insufficient available P to permit significant cycling of soil P stocks, with the total P content being less than 250 mg P kg<sup>-1</sup> soil, depending on the soil type (Zaharah, 1979; Ng, 1986). Thus, the soils may not meet the P requirement of plants. In Malaysian Oxisols and Ultisols, soluble P may be strongly adsorbed onto the surfaces of Fe and Al oxides, and hydrous oxides.

P nutrition of oil palm is facilitated by applying phosphate rock (PR). When this is done, a large

amount of PR fertilizer is needed, i.e., 4 kg palm<sup>-1</sup> yr<sup>-1</sup> on inland soils and 2 kg palm<sup>-1</sup> yr<sup>-1</sup> on coastal soils, to produce a response, without the application of plant residues (Foster et al., 1989). Addition of organic residues to high P-sorbing soils is potentially attractive as it is a low-cost alternative to applying inorganic P fertilizer. It is generally accepted that the incorporation of organic materials, such as empty fruit bunches (EFB) and plant residues, improves nutrient conservation and availability in oil palm plantations. Iyamuremye et al. (1996a) demonstrated that the amount of P adsorbed by the soils was reduced when P fertilizer was amended with organic residues. Likewise, Hue (1990) reported that inorganic P fertilizers were utilized effectively by the crop when applied together with organic inputs, such as green manure and animal waste, probably by reducing P-sorption

<sup>\*</sup> Malaysian Palm Oil Board, P. O. Box 10620, 50720 Kuala Lumpur, Malaysia. E-mail: zulhashim@mpob.gov.my

capacity, hence increasing the P concentration in the soil solution. The use of organic materials such as farmyard manure, crop residues and green manures together with PR has also been shown to enhance PR dissolution and in turn plant uptake (Bah *et al.*, 2006).

Application of PR, together with organic residues, involves complex chemical and biological processes that influence the dissolution of PR materials and the distribution of various fractions of P in soil. This appears to be a most promising strategy for improving the efficiency of P use. The important factors that influence changes in the P fractions in soil are the chemical properties of the soil, the chemical and physical nature of PR, the presence of organic residues and climatic factors (Rajan *et al.*, 1996; Khalid, 1999).

Several researchers have evaluated the individual effects of soil-related factors, such as proton supply and Ca and P status, which influence the dissolution of PR in soil (Khasawneh and Doll, 1978; Chien et al., 1980; Wilson and Ellis, 1984; Robinson and Syers, 1991) and the interactive effects of these factors (Robinson and Syers, 1990). However, a good understanding of the interaction of these factors, with and without the addition of organic residues, is still lacking. Bolland et al. (2001) reported that no single soil property adequately predicted the agronomic effectiveness of North Carolina phosphate rock (NCPR) and Sechura PR relative to superphosphate, and concluded that the effectiveness of PR is a consequence of complex interactions of that PR with the soil.

Two PR materials of contrasting chemical reactivity and water-soluble triple super phosphate rock (TSP) were used in this incubation study. Thus, the main objective of the study was to investigate the effect of EFB, P sources, and combinations of EFB and P sources, on the partitioning and changes of the P fractions in soil.

#### MATERIALS AND METHODS

#### Soil

The soil type used was the Rengam Series soil. The soil samples were taken from the site (at the edge) of a field experiment evaluating P fertilizers (at MPOB Research Station in Kluang) and brought to the glasshouse for the incubation study. The Rengam Series is a member of the clayey kaolinitic isohyperthermic family of Typic Paleudults (Paramanthan, 1978).

#### **Incubation Study**

The soil samples (15 kg of air-dried soil) were transferred into pots made of PVC pipe with a

diameter of 25 cm, and filled to a depth of 30 cm. Each P fertilizer was applied at a rate equivalent to 900 mg P kg<sup>-1</sup> (180 kg ha<sup>-1</sup>), and thoroughly mixed to a depth of 10 cm, with and without the addition of EFB (38 mg P kg<sup>-1</sup> soil). The high P treatments used in the incubation experiment were greater than the rate normally applied in the field. This was in order to get a rapid dissolution of P fertilizer and distribution in the various soil P fractions. Some P was lost during the monthly soil sampling in the six-month incubation period. The experimental design was a randomized complete block design (RCBD) with three replications and three types of P fertilizers, tested in the presence and absence of EFB. The mixtures were incubated for about six months in the glasshouse, during which time the temperature ranged from 37°C to 40°C. Distilled water was added daily to bring the soil to field capacity.

#### **Chemical Analysis**

Duplicate samples of about 50 g soil, were removed from a depth of 0-15 cm from each pot, at 1, 2, 3, 4, 5 and 6 months after PR and EFB application. The samples were analyzed for the following fractions of P: total P, Bray 2 P, iron oxide-coated strip P (Pi), organic P, NaOH P, Ca P, microbial biomass P and microbial biomass C, exchangeable Ca, and amount of P dissolved ( $\Delta$ P). The absolute value of P added as EFB, P fertilizer and the combination of both P fertilizer and EFB was 12.7, 900 and 912.7 mg kg<sup>-1</sup> soil, respectively.

Total P was determined by digesting air-dried soil samples in concentrated sulphuric acid and 30% perchloric acid mixture (Anderson and Ingram, 1993). Inorganic P was determined colorimetrically by the molybdenum blue method with ascorbic acid as the reducing agent (Murphy and Riley, 1962).

Extractable P was measured by extracting an air-dried soil sample for 1 min with the Bray No. 2 extractant which is a mixture of 0.1 M HCl plus  $0.03 \text{ M NH}_4\text{F}$  (Bray and Kurtz, 1945).

Biomass C was measured by the fumigationextraction method (Vance *et al.*, 1987). The fumigation process was similar to the procedure for determining microbial biomass P.

The amount of P dissolved ( $\Delta P$  or the extent of dissolution) was determined from the increase in the amount of inorganic P, being the difference between the amount of P extracted from the soil alone and from the soil amended with PR or TSP, together with and without EFB (Mackay and Syers, 1986; Mackay *et al.*, 1986). The soil alone (control) and the treated soil (with PR or TSP, with and without EFB) were pre-washed with 1 M NaCl for 1 hr, to remove both solution and exchangeable Ca which otherwise may form Ca(OH)<sub>2</sub> during extraction with 0.5 M NaOH, and readsorb or coprecipitate with some of the dissolved P (Mackay *et al.*, 1986).

Organic P was estimated from the difference between acid-extractable P from non-ignited and ignited soil samples (Anderson and Ingram, 1993).

Phosphate sorption and desorption by soils were measured by determining the amount of P sorbed and desorbed from known amounts of P added as KH<sub>2</sub>PO<sub>4</sub> in 0.01 M CaCl<sub>2</sub> electrolyte (Dubus and Becquer, 2001). A linear form of the Langmuir equation, C/X = C/Smax + 1/KSmax, was used to evaluate the adsorption constants of the soils. In the equation, C is the P concentration at equilibrium condition (mg P l<sup>-1</sup>), X is the amount of P sorbed (mg P kg<sup>-1</sup>), Smax is the adsorption maximum, and *K* is a constant related to the binding energy. *Smax* is determined from the slope of the graph plot of C/X against C, and K is calculated from the intercept. The amount of P required by the soils to attain 0.2 mg P l<sup>-1</sup> was extrapolated from a graph of P adsorbed against P concentration at equilibrium. Phosphate desorption was determined by adding approximately 40 ml of 0.01 M CaCl to the soil by weighing the soil plus the solution to give the same weight as before the supernatant was removed following the sorption step. The P in solution was again analyzed using the Murphy and Riley (1962) procedure. The amount of desorbed P was measured by subtracting the weight of entrapped P from the weight of P desorbed (final P concentration in the solution during the desorption process). The percentage of desorbed P was calculated from the amount of P sorbed.

The forms of inorganic P (Ca P and occluded P) were determined using a sequential P fractionation technique (Chang and Jackson, 1957), as modified by Hedley *et al.* (1994).

Inorganic P (Pi) in the soils was extracted and analyzed using an iron oxide-coated strip (Menon *et al.*, 1989a, b).

Soil pH values were determined in water and 1 M KCl using an Orion combination pH electrode at a soil:solution ratio of 1:2.5 after equilibration for 30 min. Total C was analyzed by the Walkley and Black method (Walkley and Black, 1934). Exchangeable Ca was determined by a leaching method where 10 g of soil were leached with 100 ml of 1 M NH<sub>4</sub>OAc (pH 7) for 5 to 6 hr.

Exchangeable acidity (H and Al) was measured by shaking 10 g of soil with 50 ml 1 M KCl (Barnhisel and Bertsch, 1982) at 20 rpm for 1 hr. Total N was measured by a macro-Keldahl method, and particle size distribution was determined by the pipette method (Day, 1965).

The chemical and physical properties of the soil and its sorption and desorption characteristics are shown in *Tables 1* and 2, respectively.

TABLE 1. CHEMICAL AND PHYSICAL PROPERTIES OF RENGAM SERIES SOIL USED IN THE STUDY

Parameters	Value
Total P (mg kg <sup>-1</sup> )	72
Bray 2 P (mg kg <sup>-1</sup> )	8
Pi (mg kg <sup>-1</sup> )	4
NaOH P (mg kg <sup>-1</sup> )	18
Ca P (mg kg <sup>-1</sup> )	2
Occluded P (mg kg <sup>-1</sup> )	19
Organic P (mg kg <sup>-1</sup> )	10
Microbial biomass P (mg kg <sup>-1</sup> )	1.5
Microbial biomass C (mg kg <sup>-1</sup> )	305
pH (water)	4.5
pH (KCl)	4.0
Organic C (%)	1.30
Total N (%)	0.12
CEC (cmol + kg <sup>-1</sup> )	9.4
Exch. Ca [cmol (+) kg <sup>-1</sup> ]	0.74
Exch. Al [cmol (+) kg <sup>-1</sup> ]	1.6
Clay (%)	47
Silt (%)	4
Fine sand (%)	15
Coarse sand (%)	34

TABLE 2. SORPTION AND DESORPTION CHARACTERISTICS OF RENGAM SERIES SC	JIL
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Added P (mg kg <sup>-1</sup> soil)	Sorbed P (mg kg <sup>-1</sup> soil)	P eq (mg l-1)	Sorbed P <sup>1</sup> (%)	Desorbed P (mg kg <sup>-1</sup> soil)	P eq (mg 1-1)	Desorbed P <sup>2</sup> (%)
80	73.1	0.17	91.3	5.4	0.16	7.3
160	142	0.44	88.9	15.1	0.42	10.6
320	248	1.78	77.8	31.0	0.82	12.5
480	304	4.38	63.5	45.1	1.20	14.8
600	337	6.60	56.2	53.6	1.40	15.9

Note: 1Percentage of that added.

<sup>2</sup>Percentage of that sorbed.

#### **Empty Fruit Bunches and P Fertilizer Sources**

EFB were dried at 40°C overnight, cut into small pieces, before they were used in the incubation study at a rate of 38 mg P kg<sup>-1</sup> soil for each treatment, with and without P fertilizer. A partial chemical composition of EFB is shown in *Table 3*.

Characterization of the P sources involved the determination of the chemical and some of the mineralogical properties of PR and TSP. The chemical properties of the two PR sources, as well as TSP, were determined by digestion/dissolution in concentrated HNO<sub>3</sub>/HC1 (SIRIM, 1995), while Ca was measured by atomic absorption spectrophotometry (AAS). The solubility of the P sources in 2% formic acid (FA), 2% citric acid (CA) and neutral ammonium citrate (NAC) was also determined (Chien, 1993), and the results are shown in *Table 4*.

#### **Statistical Analysis**

The effect of adding PR and EFB, and of time, on the various fractions of P was analyzed using the general linear model (GLM). If the F-test was significant (p<0.05), the means were compared using Tukey's Studentized Range test. The same procedure was used to compare the time effects

#### TABLE 3. PARTIAL CHEMICAL COMPOSITION OF EMPTY FRUIT BUNCHES (EFB) USED IN THE STUDY

Parameter	% (w/w)
Ν	0.62
Р	0.19
Κ	1.42
Ca	0.25
Mg	0.12

# TABLE 4. SOME CHEMICAL CHARACTERISTICS AND SOLUBILITY OF THE PHOSPHATE SOURCES

Parameter	P Source				
ralameter	GPR	CIPR	TSP		
P (%)	13.4	14.5	20.4		
Ca (%)	31.8	34.2	16.2		
Sol. in FA (%)	20.8	11.6	43.3		
Sol. in FA (% of total P)	68.0	34.9	92.6		
Sol. in CA (%)	11.7	9.3	41.9		
Sol. in CA (% of total P)	38.2	27.9	90.0		
Sol. in NAC (%)	5.0	3.6	19.4		
Sol. in NAC (% of total P)	16.2	10.8	86.2		

Note: FA = formic acid; CA = citric acid, NAC = neutral ammonium citrate.

GPR - Gafsa phosphate rock.

CIPR – Christmas Island phosphate rock.

TSP - triple superphosphate.

within the treatments. All statistical analyzes were carried out using a SAS package (version 6.12, 2000).

# **RESULTS AND DISCUSSION**

The amounts of P dissolved ( $\Delta P$ ) and the values of the soil P fractions, namely readily-available P (Pi iron oxide-coated strip and Bray 2 P), other inorganic P (NaOH and occluded P fractions), undissolved PR (Ca P), organic P, and microbial biomass P in soil amended with PR or TSP alone, EFB alone, EFB with PR or TSP, and an untreated control, during a six-month incubation period are shown in Table 5. The untreated soil (control) contained a large amount of NaOH P (about 35% of the total P), followed by organic P (25% of total P), with a ratio of microbial biomass P to organic P of 1:14. A larger amount of NaOH P (21.4 mg P kg<sup>-1</sup> soil) than Pi (5 mg P kg<sup>-1</sup> soil) and Bray 2 P (11 mg P kg<sup>-1</sup> soil) confirms that the Rengam Series soil has a high P sorption as shown previously in Table 2. This property, together with low soil pH and Ca status, is expected to provide favourable conditions for PR use in Malaysian acid soils.

### Amount of P Dissolved

The amount of P dissolved ( $\Delta P$ ) and the percentage dissolution after six months in the incubation study are shown in Table 6. The results show that the  $\Delta P$  values for P materials followed their chemical reactivity; thus, water-soluble TSP dissolved significantly (p<0.05) more (82.3%) than the reactive GPR (27.4%), followed by the less reactive CIPR (23.6%). There was no significant effect (p<0.05) on  $\Delta P$  when EFB was added together with TSP or GPR (571 and 194 mg P kg<sup>-1</sup> soil, respectively), compared to that for TSP or GPR added alone (569 and 188 mg P kg-1 soil, respectively). However, adding EFB together with less reactive CIPR (174 mg P kg<sup>-1</sup> soil) significantly increased the amount of P dissolved compared to when CIPR was added alone (159 mg P kg<sup>-1</sup> soil). The results suggest that the reactivity or solubility of P materials, the amount of P added in EFB, and the interaction effect of adding P fertilizer together with EFB affect the dissolution of P materials and redistribution of the soil P fractions.

Adding a relatively small amount of P in EFB together with P fertilizer had some effects on the dissolution of P materials, especially CIPR compared to when P fertilizer was added alone. This suggests that organic residues can enhance the dissolution of PR materials. The results obtained in the present study are in agreement with those of several workers who have also reported a positive influence of organic materials on PR dissolution

	Treatment								
	Control		TSP		GPR		CIPR		
P fraction	Without EFB	With EFB	Without EFB	With EFB	Without EFB	With EFB	Without EFB	With EFB	
Pi	4.90g	7.40f	162a	142b	100c	74.5d	49.4e	51.7e	
Bray 2 P	11.0g	16.1f	272b	302a	244c	278b	172e	197d	
NaOH P	21.4g	22.2g	260a	227b	133e	120f	154c	145d	
Ca P	5.50g	6.50g	53.1f	82.5e	256b	233c	280a	212d	
Occluded P	10.2e	10.9e	125a	97.0b	96.4b	86.5c	86.1c	82.1d	
Organic P	15.5e	35.0d	75.0c	159a	86.5c	152a	71.3c	120b	
Microbial biomass P	1.10e	5.90d	7.70c	33.5a	7.10cd	31.2a	6.90cd	28.1b	
ΔΡ	-	6.40f	569a	571a	188b	194b	159d	174c	
Total P	63.5g	90.9f	678cd	694b	686c	703a	673d	690bc	

 TABLE 5. EFFECT OF ADDING EMPTY FRUIT BUNCHES (EFB) AND P FERTILIZER TO SOIL ON FRACTIONS (mg kg<sup>-1</sup> soil)

 OF SOIL P (for all sampling dates combined) AT 0-15 cm DEPTH

Note: Values followed by the same letter in the same row are not significantly different at the 5% probability level as determined by Tukey's Studentized Range test.

TSP – triple superphosphate.

GPR – Gafsa phosphate rock.

TABLE 6. AMOUNT OF P DISSOLVED (ΔP) AND PERCENT DISSOLUTION OF P FERTILIZER AFTER SIX MONTHS OF INCUBATION AT A SAMPLING DEPTH OF 0-15 cm

P (mg kg <sup>-1</sup> soil)	% dissolution*
569a	83.4
571a	82.3
188b	27.4
194b	27.6
159d	23.6
174c	25.2
	569a 571a 188b 194b 159d

Note: \*Percent dissolution of P materials = (P/Total P) × 100. Values followed by the same letter are not significantly different at the 5% probability level as determined by Tukey's Studentized Range test. GPR – Gafsa phosphate rock. EFB – empty fruit bunches.

TSP – triple superphosphate.

CIPR – Christmas Island phosphate rock.

(Iyamuremye *et al.*, 1996a, b) but only to a small extent, depending on the amount of P added with the organic residues. The factor that probably acts as a driving force for further dissolution of PR is the increase in the number of cation-exchange sites as a result of adding organic residues (Chien, 1979). Soil organic components may also supply organic functional groups or anions, such as citrate and oxalate, which can chelate Ca<sup>2+</sup> ions, and thus lower the Ca<sup>2+</sup> activity in the soil solution (Chien, 1979). These organic acids could also dissolve PR by supplying protons and by complexing the cations with P (Kpomblekou and Tatabai, 1994) which

would enhance the dissolution of PR. The results of the present study confirm that the dissolution of PR was enhanced when EFB was added together with PR, as shown by the decrease in the amount of Ca P compared with the treatment of adding PR alone.

The formation of metal complexes by organic acids with Al and Fe components which react with P from added P materials would increase plant-available P as reported by Hue et al. (1986), who found that organic acids reduced Al activity. It was also reported that carboxyl and hydroxyl functional groups in citrate can chelate Fe and Al, thereby releasing P (Huang and Violante, 1986). Competition between P and oxalate, citrate or malate for sorbing sites (Hue, 1992) could probably be another reason for the higher dissolution of PR in soil treated with both P fertilizer and EFB. The results are in agreement with the above mechanisms: NaOH P decreased by about 12%, 18% and 14% when EFB was added together TSP, GPR and CIPR, respectively, as compared to when P fertilizer was added alone.

The results also suggest that addition of organic residues with P fertilizer in the soil provides a sink for P, through an increase in the microbially-bound P (microbial biomass P) into dead or living cells (Chauhan *et al.*, 1979), which is directly correlated with microbial biomass C (Brookes *et al.*, 1984). Microbial biomass plays a key role in soil organic P mineralization (Brookes *et al.*, 1984) in releasing plant-available P. An increase in the P sink as shown in the present study is likely to be an important factor in enhancing the dissolution of PR at a later stage, compared with the soil treated with PR alone.

# Effects of Addition of P and EFB on Various Fractions of Soil P

Addition of PR and water-soluble TSP (at about 900 mg P kg<sup>-1</sup> soil) increased directly the amount of total P during the six-month incubation study. However, the amount of total P recovered was relatively low, *i.e.*, 678 mg kg<sup>-1</sup> soil from TSP, 686 mg kg<sup>-1</sup> soil from GPR, and 673 mg kg<sup>-1</sup> soil from CIPR treatments, compared to the amount added (900 mg P kg<sup>-1</sup> soil). The reason was due to a dilution effect as a result of mixing these P fertilizers into the top 10 cm of soil, while the soil samples were taken at the depth of 0-15 cm. The amount of total P at 10 cm depth was 960 mg P kg<sup>-1</sup> soil, with 900 mg P kg<sup>-1</sup> soil from PR and 63.5 mg P kg<sup>-1</sup> soil from the soil itself. In the upper 15 cm depth, where the soil samples were taken, the dilution effect would give a P value of around two-thirds of the total P, which is approximately 660 mg P kg<sup>-1</sup> soil. This value is comparable to the amounts determined by the chemical analyses (Table 5). The total P, when calculated by the sum of all the soil P fractions, was larger than that determined by chemical analyzes. This may be because some of the P extracted by the Pi procedure was also extracted by the Bray 2 method.

Addition of P fertilizer together with EFB increased the organic P during the six months of incubation (Table 5), significantly (p<0.05) more than when P fertilizer was added alone. The amounts of organic P when EFB was added together with TSP (159 mg P kg<sup>-1</sup> soil) and with GPR (152 mg P kg<sup>-1</sup> soil) were significantly larger (p<0.05) than that obtained when EFB was added with CIPR (120 mg P kg<sup>-1</sup> soil). Adding EFB together with P fertilizer increased the amount of organic P by 112%, 98% and 68% for TSP, GPR and CIPR, respectively, compared with that obtained for soil treated with P fertilizer only, following the reactivity or solubility of the P fertilizer. Smith et al. (1998) reported that dissolved organic P (DOP) ranged from 35.1% to 44.5% of the cumulative total P in leachates from organic matter (black spruce forest in central Quebec), similar in proportion of organic P to that reported from soil leachate (35% to 44% of total P) collected under a deciduous forest in North Carolina (Qualls et al., 2000). Therefore, the addition of organic P (expected to be around 40%-50% of total P in EFB), together with the immobilization of inorganic P, and solubilization of Ca P were possibly responsible for the increase in organic P.

Addition of EFB together with TSP increased organic P by 84 mg P kg<sup>-1</sup> soil, compared with that in soil treated with TSP alone. The interaction effect between GPR and organic residues also increased organic P (by 76 mg P kg<sup>-1</sup> soil) above that with GPR alone, whereas addition of CIPR together with organic residues increased organic P by 49 mg P kg<sup>-1</sup> soil, compared to that in soil treated with CIPR alone. The increase of organic P when EFB were added together with P materials was over and above that in EFB. Therefore, the results suggest that organic P is an important P pool accumulated during the dissolution of PR in soil amended with EFB and during the decomposition of EFB by microbial activity. Organic P compounds are thought to associate with positively-charged sites on organic matter and clay particles, or with cations in the soil solution, and could be a source of plant-available P (Cross and Schlesinger, 1995). The findings show that maximizing the build-up of organic matter can reduce the application of inorganic P fertilizers, especially with acid soils where there are distinct wet and dry seasons and higher temperatures, all of which can increase the amount of organic P mineralization (Sharpley, 1995), and thus could provide more plantavailable P.

Adding EFB increased microbial biomass P (4.8 mg P kg<sup>-1</sup> soil) and organic P (19.5 mg P kg<sup>-1</sup> soil) over the control. The organic P compounds in plants are nucleic acids, phospholipids and phytin (Stevenson, 1986). Therefore, it was expected that organic P increased by 70% in the soil treated with EFB, compared to the control, because probably organic P content of around 40%-50% of total P was added in EFB. The results suggest that, in the presence of organic residues, organic P becomes an important pool in the soil, and contributes to the potentially plant-available fraction through organic transformations and mineralization into inorganic P fractions. This is shown by the increase of Pi (2.5 mg P kg<sup>-1</sup> soil) and Bray 2 P (5.1 mg P kg<sup>-1</sup> soil). However, in the soil treated with EFB only, without inorganic P, immobilization of inorganic P to organic P was not as large as when P fertilizer was added together with EFB.

Addition of EFB, together with PR or TSP, increased the amount of microbial biomass P by about four times more than in the soil treated with PR or TSP alone. The results suggest that addition of EFB stimulated the formation of organic P and microbial biomass P, and caused less movement to NaOH P and occluded P. This is shown by the decrease in NaOH P (decrease by 12%, 10% and 6% for TSP, GPR, and CIPR, respectively) and occluded P (decrease by 22%, 10% and 5% for TSP, GPR, and CIPR, respectively) in the soil treated with both P fertilizer and EFB compared to the soil treated with P fertilizer alone. Microbial solubilization of inorganic P was possibly the mechanism involved in the decrease of NaOH P and occluded P, a mechanism which includes acidification and complexation by organic acids produced by microorganisms (Kucey et al., 1989).

Overall, the addition of P fertilizer increased the amount of Pi and Bray 2 P in the soil (Table 5) due to the reaction of added P with the soil (McCollum, 1991), and this varied substantially with the type of P fertilizer and with the time of contact (discussed in a later section). Bray 2 P is the most common extraction method used to indicate plant-available P in acid soils. The extractant used in this method is stronger than that used in the Pi method, and thus removed some of the adsorbed P or undissolved PR-P; it removed 17% to 22% more P than Pi (in terms of percentage of the total P), depending on the P materials added to the soil. Therefore, the iron-oxide strip (Pi method) is believed to indicate the most labile P (readily plant-available P) in the soil. The amounts of Pi and Bray 2 P in the soil treated with TSP (162 and 272 mg P kg<sup>-1</sup> soil, respectively) were significantly larger (p<0.05) than the amounts of these fractions in the soil treated with GPR (100 and 244 mg P kg<sup>-1</sup> soil, respectively) and CIPR (49.4 and 172 mg P kg<sup>-1</sup> soil, respectively). The reason is the reactivity of the materials, with TSP as a water-soluble source dissolving and supplying the largest amount of available P during the initial dissolution of TSP. Reactive GPR released 100% and 45% more Pi and Bray 2 P, respectively, compared to the less reactive CIPR. Reactive GPR dissolves more in 2% formic acid than CIPR, hence GPR is expected to supply more Pi and Bray 2 P in the soil, as was also reported by Chien (1993), and Hanafi and Syers (1994). However, with the high capacity of the soil to sorb the added P released, and rapid chemical reactions with Fe and Al oxides and hydrous oxides, these fractions are shortlived in the soil and are rapidly transformed into NaOH P and/or occluded P fractions to maintain equilibrium (Golden et al., 1991; Hedley et al., 1994) in the soil. As only one soil type was used, the direct relationship between the dissolution of PR and P-sorption capacity was not investigated. Nevertheless, several researchers (Khasawneh and Doll, 1978; Syers and Mackay, 1986; Bolan and Hedley, 1989; Hanafi et al., 1992a, b; Bolland et al., 2001) have reported that the dissolution of PR materials decreases as soil P-sorption capacity increases.

The amounts of Pi and Bray 2 P in the soil where P fertilizer and EFB were added together are shown in *Table 5*, and these ranged from 51.7 (Pi) and 197 (Bray 2 P) mg P kg<sup>-1</sup> soil for CIPR to 142 (Pi) and 302 (Bray 2 P) mg P kg<sup>-1</sup> soil for TSP. A larger amount of Pi and Bray 2 P is expected for the highly soluble TSP than the slowly soluble PR. It has been shown that the iron oxide-impregnated filter paper strips (Menon *et al.*, 1989a, b) estimate readily-available P in the soil. The P extracted using the iron-oxide impregnated filter paper closely approximated the amount of P extracted by an anion-exchange resin,

which is sometimes used in the fractionation of inorganic P in the soil (Sharpley, 1991).

Combining P fertilizer with EFB significantly decreased (p<0.05) the amount of Pi in the GPR (74.5 mg P kg<sup>-1</sup> soil) and TSP (142 mg P kg<sup>-1</sup> soil) treatments, when compared with the amount obtained for the soil treatments receiving GPR (100 mg P kg<sup>-1</sup> soil) and TSP (162 mg P kg<sup>-1</sup> soil) alone. However, the Pi value in the soil treated with CIPR together with EFB was slightly increased (51.7 mg P kg<sup>-1</sup> soil), compared with the soil treated with CIPR alone (49.4 mg P kg<sup>-1</sup> soil). This was probably because some of the P dissolved in the soil treated with water-soluble TSP (20 mg P kg<sup>-1</sup> soil) and reactive GPR (25.5 mg P kg-1 soil) was partly incorporated into the organic P. Therefore, the amount of Pi in the soil treated with TSP and GPR decreased, while the increase in Pi for CIPR was possibly due to the formation of metal complexes (Al and Fe compounds) by organic acids, which would release adsorbed P. Similar findings have been reported by Zhou et al. (1997), who found that organic matter-metal complexes tended to release P readily at low solution P, which was observed for the CIPR and EFB treatment. For GPR and TSP treatments, which already had larger amounts of Pi in the soil to which P was added, adding EFB possibly transformed the inorganic P into organic P, especially during the first two or three months of incubation (discussed in a later section), before mineralization took place.

The amount of NaOH P varied from 17.9% (GPR) to 30.5% (TSP) of the total P in the soils receiving P fertilizer compared with the control. The amount of NaOH P was significantly larger in the soil treated with TSP (260 mg P kg<sup>-1</sup> soil) than with CIPR (154 mg P kg<sup>-1</sup> soil) and GPR (133 mg P kg<sup>-1</sup> soil). The initial dissolution involves water moving into the TSP granules and dissolving monocalcium phosphate, forming a metastable triple-point solution containing dicalcium phosphate and phosphoric acid (Zoysa et al., 2001). In acid soils, in which Al and Fe are abundant, the phosphoric acid can react with soluble P to form relatively insoluble Fe and Al phosphates (Bolland et al., 2001). The amount of NaOH P was 17% and 20% (in terms of percentage of the total P), larger in the soil treated with TSP than with CIPR and GPR, respectively, while occluded P was 6.5% and 5% larger in the TSP-treated soil than CIPR- and GPRtreated soils, respectively. A large amount of P in the TSP-treated soil was present as NaOH P, occluded and plant-available P fractions, while Ca P fraction was the largest P fraction in the soil treated with PR. This was probably due to the large amount of undissolved PR (Ca P), *i.e.*, 256 mg P kg<sup>-1</sup> soil for GPR treatment and 280 mg P kg<sup>-1</sup> soil for CIPR, present in the soil during the six months of incubation. The results suggest that CIPR had a greater residual P than GPR, if the Ca P is considered as a measure for estimating the long-term apatite reserves of the soil. However, due to CIPR comprising several mineral groups that vary in their solubility, the amount of Ca P probably underestimated Ca-bound P, due to the fact that 1 M HCl was unable to solubilize the Ca-bound P from apatite. This was shown by the amount of P dissolved from PR, as measured by  $\Delta$ P, being smaller than that calculated from the difference from total P added and undissolved Ca P. The difference in the amount of dissolved P measured by these two methods probably indicated that the amount of Ca P was underestimated by 1 M HCl.

The amount of occluded P was significantly larger (p<0.05) in the soil treated with TSP (125 mg P kg<sup>-1</sup> soil) than with GPR (96.4 mg P kg<sup>-1</sup> soil) and CIPR (86.1 mg P kg<sup>-1</sup> soil). In this six-month incubation study, adsorbed P may have been converted from NaOH P to occluded P, as reported by Hagin *et al.* (1990).

About 75, 87 and 71 mg P kg<sup>-1</sup> soil was incorporated into organic P, when TSP, GPR and CIPR, respectively, was added to the soil, which was about 360% (CIPR), 384% (TSP) and 460% (GPR) larger than the control, but amounted to only about 10%-11% of the total P. The solubilization of Ca P was probably responsible for organic P accumulation during soil development as reported by Walker and Syers (1976). The result suggests that the solubilization of inorganic P resulted about 6.6%, 7.9% and 6.2% of added P were incorporated into the organic P for TSP, GPR and CIPR, respectively. There was no significant difference among the three P fertilizers, although GPR gave a slightly larger amount of organic P compared with TSP and CIPR. The amount of microbial biomass P, however, increased by only 1% of added P with all three P fertilizers. Microbial biomass P has been reported to play a role in the changes and distribution of soil inorganic and organic P fractions. It is a dynamic intermediary between the inorganic and organic fractions of soil P (Sharpley, 1995; Sharpley et al., 1995), but not to a great extent in soils with no added organic residues.

The amount of Ca P varied considerably when PR or TSP was added together with EFB, depending on the content of the P fertilizer. The Ca P increased when TSP and EFB were added together (82.5 mg P kg<sup>-1</sup> soil) compared to when TSP (53.1 mg P kg<sup>-1</sup> soil) was added alone. It was not clear why Ca P increased slightly in the soil treated with TSP and EFB, but could probably be because some of the Ca released from the EFB precipitated as Ca P, resulting in an increase of about 29 mg P kg<sup>-1</sup> soil when a large amount of P was added to the soil. In the case of GPR (233 mg P kg<sup>-1</sup> soil) and CIPR (212 mg P kg<sup>-1</sup> soil), the amount of Ca P decreased

significantly when added together with EFB, compared to the treatments without EFB (256 and 280 mg P kg<sup>-1</sup> soil for GPR and CIPR, respectively), suggesting that organic residues enhanced the dissolution of P materials. The amount of Ca P in soil was important for the continuous dissolution of PR in the soil over time, provided that the PR was sufficiently reactive. The residual effect (as shown by a larger amount of unreacted GPR and CIPR) was important in providing a longer-term supply of the plant-available P. As discussed earlier, more P was dissolved from water-soluble TSP and this caused a larger shift of dissolved P to adsorbed P (NaOH P) than with reactive GPR and less reactive CIPR.

# Effect of Time on the Amount of P Dissolved from Added P Fertilizer

The values of  $\Delta P$  in the soil to which TSP or PR was added, with and without EFB, during the six-month incubation period are shown in *Table 7*. Two different trends for PR dissolution with time were observed when P was added alone, and when added together with EFB. The rate of PR dissolution in the soil treated with PR fertilizer alone was higher than when PR fertilizer was added together with EFB.

When PR fertilizer was added alone to the soil,  $\Delta P$  values increased steadily, from 170 mg P kg<sup>-1</sup> soil (GPR) and 160 mg P kg<sup>-1</sup> soil (CIPR) at the beginning of the experiment until after four months (230 mg P kg<sup>-1</sup> soil for GPR) and five months (193 mg P kg<sup>-1</sup> soil for CIPR), when  $\Delta P$  values peaked, and decreased to 225 mg P kg<sup>-1</sup> soil for (GPR) and 188 mg P kg<sup>-1</sup> soil for (CIPR) after six months of incubation. However, the maximum amounts of P dissolved in the soils treated with GPR or CIPR alone did not change significantly with time, suggesting that the amount of P dissolved reached a plateau after approximately four months of incubation.

Addition of EFB together with PR fertilizer led to a steady increase in the  $\Delta P$  values, from 182 and 168 mg P kg<sup>-1</sup> soil to 242 and 203 mg P kg<sup>-1</sup> soil for GPR and CIPR, respectively, during the six months of incubation. This steady increase may be due to an increase in organic acid content resulting from microbial activity and chemical transformation of organic residues, which increased the dissolution of PR in the soil, as discussed earlier. Therefore, the result suggests that the addition of EFB together with PR was able to further increase the amount of P dissolved after six months of incubation compared with the soil treated with PR alone. The  $\Delta P$  values for soil treated with water-soluble TSP, with and without EFB, however, fluctuated with time and showed no specific trend. In the soil treated with TSP alone, more P was dissolved at the beginning of the experiment, largely because TSP

	Treatment								
Month	Control		TSP		GPR		CIPR		
	Without EFB	With EFB							
1	0	5.5d	625a	610a	170d	182c	160c	168c	
2	0	7.1bcd	589b	593a	191c	191c	180b	183b	
3	0	10.4ab	573bc	584a	206bc	213b	186ab	189b	
4	0	11.9a	589b	588a	227ab	230ab	193a	193b	
5	0	9.4abc	564c	592a	230a	236a	186ab	201a	
6	0	6.7cd	583bc	593a	225ab	242a	188ab	203a	

TABLE 7. EFFECT OF TIME ON THE AMOUNT OF ΔP (mg kg<sup>-1</sup> soil) WHEN TRIPLE SUPERPHOSPHATE (TSP) OR PHOSPHATE ROCK (PR) AND EMPTY FRUIT BUNCHES (EFB) WERE ADDED TO SOIL DURING A SIX-MONTH INCUBATION PERIOD

Note: Values followed by the same letter in the same column are not significantly different from one another at the 5% probability level, as determined by Tukey's Studentized Range test.

is a more soluble P source compared to GPR and CIPR. There was no significant effect of time on  $\Delta P$  in the soil treated with TSP and EFB. However, TSP always gave higher P values, with about 69% of the added P dissolving during the first month, but this decreased slightly to 68% in the presence of EFB. After six months of incubation, about 76% and 73% of GPR, without and with EFB, respectively, and 80% and 77% of CIPR, without and with EFB, respectively, remained undissolved in the soil. An insufficient sink for Ca was probably the reason why the PR materials dissolved slowly in the closed system (Hanafi *et al.*, 1992a).

For the soil treated with PR alone, the dissolution of P materials depended solely on their chemical reactivity, given adequate moisture. Adding organic residues together with P fertilizer, increased the dissolution of P materials, possibly due to the increase in P sink (through biological transformations of P and organic P fractions, as shown by the increase in microbial organic P and C) and Ca sink size (increase in soil CEC) as discussed earlier.

### CONCLUSION

The results suggest that organic P is an important P pool which accumulates during the dissolution of PR in soil amended with EFB and during the decomposition of EFB by microbial activity. Organic P compounds are thought to be associated with positively-charged sites on organic matter and clay particles, or with cations in the soil solution, and could be a source of plant-available P (Cross and Schlesinger, 1995). The results also suggest that the addition of EFB stimulates the formation of organic P and microbial biomass P, and causes less movement to NaOH P and occluded P. The organic residues have the potential of enhancing the dissolution of PR materials; this observation is in agreement with those of several workers,

who have also reported a positive influence of organic materials on PR dissolution (Chien, 1979; Iyamuremye et al., 1996a, b). Among the probable factors which act as a driving force for the further dissolution of PR is the increase in Ca sink (by increasing the number of cation-exchange sites which are able to adsorb Ca<sup>2+</sup>), and therefore more P is dissolved following the lowering of the Ca activity in the soil solution. Addition of organic residues possibly provides a P sink by increasing microbial biomass P, which plays an important role in the transformation of P from organic P to plantavailable P (Magid et al., 1996). Microorganisms can produce low molecular weight organic acids, such as citric acid and oxalic acid (Kucey et al., 1989), which will release P that is available to plants by complexing with Al and Fe that readily react with P. Moreover, a series of organic anions strongly compete with P for adsorbing sites on the surfaces of Fe and Al components (Violante et al., 1991), and the release of adsorbed P through ligand exchange reactions may be another mechanism of P being released into solution when PR materials are added together with organic residues.

Different trends for PR dissolution, as well as for plant-available P (Pi and Bray 2 P) with time, were observed when the P source was added alone and together with EFB. The rate of PR dissolution and the rate of release of plant-available P over time in the soil treated with PR fertilizer alone were faster than when PR fertilizer was added with EFB. These results show that the presence of organic residues continue to increase the amount of P dissolved over time, possibly due to the increase in P sink size (biological transformation and mineralization of organic P) and Ca sink size (increase in Ca-exchange sites) compared with when PR alone is added to the soil. This indicates that applying organic residues together with PR fertilizer continuously enhances the dissolution of PR in the soil during the six months of incubation.

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