CHANGES OF INORGANIC FRACTIONS OF PHOSPHORUS AND AVAILABLE PHOSPHORUS WITH EQUILIBRATION TIME IN FLOODED AND MOIST SOILS[1]

Wilfredo G. Espada[1]

The application of phosphate fertilizers to the soil seldom results in the recovery of more than 10 to 20 percent of the phosphorus. Fixation occurs simultaneously with the dissolution of phosphate from the fertilizer especially when the soil environment contains ions such as Fe^{+3}, Al^{+3}, Ca^{+2}, K^{+}, NH_4^{+}, and others. Several discrete mineral species or phosphate compounds were identified in solution supporting both phosphate ions and one or more of these ions [12, 13, 14]. The major portion of inorganic phosphorus in the soil is bound by calcium, aluminum, and iron; the relative abundance of each being controlled by soil reaction.

Several investigators [4, 6, 16, 22] have determined the chemical status of the different fractions and the fate of applied phosphorus under widely different soil conditions. Chang and Jackson [5] observed the formation of more aluminum phosphate (Al-P) than iron phosphate (Fe-P) upon addition of phosphate fertilizer. But as time elapsed, Fe-P content increased at the expense of Ca-P and Al-P [5, 6, 16, 18]. Volk and McLean [22] noted a tendency for applied phosphates to occur predominantly as ferric phosphate in soils of high fixing capacity and as aluminum phosphate in soils of low fixing capacity. The relative extent of fixation under identical soil conditions except for moisture status has not been examined.

Available phosphorus refers to the amounts of P which would be removed from soil by plans over a long period of

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[1]Part of a thesis presented in partial fulfillment of the requirements for the Ph.D. degree, Ohio State University, Columbus, Ohio.

[2]Former International Rice Research Institute Fellow. Dr. Espada is professor of soil chemistry at the College of Agriculture, Central Philippine University, Iloilo City, Philippines.

time, i.e., an index of the capacity of a soil to release substantial amounts of P for crop growth [17]. A number of studies [2, 3, 21] have shown that it could be approximated by use of extracting reagents, but no one has shown universal application. So far these procedures have been mostly related to responses in crops grown under upland conditions. A more accurate prediction of available phosphate was obtained by Baker and Hall [2] using the Bray No. 1 reagent [3].

No method has been proposed to determine adequately the phosporus status of flooded soils. Air-dry soil analysis does not reflect the actual phosphorus supplying capacity of submerged soils [6, 7]. De Datta, *et. al* [7] found that air-dry soil with 2.03 ppm 0.02 N H_2 SO_4 extractable phosphorus yielded 10 to 20 times more extractable phosphorus after a crop of rice was harvested than before. This suggests conversion of a large amount of phosphorus into acid extractable form during the cropping period.

In view of the observations above and of inadequate information applicable to flooded soils, this study was conducted to examine the relationship between the inorganic fractions of phosphorus and the transformations of applied phosphorus in soils under moist and flooded conditions.

MATERIALS AND METHODS

Phosphorus fractionatin studies. The experiment consisted of applying 90 parts of phosphorus to 2,000,000 parts of soil (90 pp2m) in four soils: Wooster silt loam (pH 6.3), Ripley silt loam (pH 5.75 and 6.15) and Crowley silt loam (pH 6.3, Arkansas paddy soil). Phosphorus was derived from ground rock phosphate, 20 and 100 percent acidulated rock phosphate, aluminum phosphate ($AlPO_4.2H_2O$) and iron phosphate ($FePO_4.2H_2O$)[3]. Each phosphate treatment was replicated three times and arranged in completely randomized block design within soil. Twenty grams of soil and the appropriate amount of each fertilizer

[3]The aluminum and iron phosphates were prepared in the laboratory according to the procedure described by Jackson (11).

were weighed into 125-ml Erlenmeyer flask and mixed thoroughly. Two moisture conditions were imposed on the phosphate treatments, i.e, flooded and most (field capacity). The flasks were stoppered with cotton and incubated at room temperature from 0 to 16 weeks. Distilled water was added as necessary to maintain the moisture conditions. The incubation was arranged such that for any date of sampling, the entire sample of incubated soil was analyzed. At the end of the incubation period, more distilled water was added to give a total volume of 50 ml in both flooded and moist conditions. The flasks were shaken in a reciprocating shaker at moderate speed for one hour, the contents transferred to 50 ml centrifuge tubes and centrifuged until the solution was free of colloidal particles. The soil residues were analyzed for the different fractions of phosphorus according to the procedure of Chang and Jackson [4].

Determination of available phosphorus. Pint-size waxed cups were filled with 100 grams of soils noted above, to which were added various phosphate materials at 90 pp2m of phosphorus. The phosphate treated soils were incubated for 1 and 8 weeks under moist and flooded conditions. The incubation scheme was arranged so that soil sampling was done on the same day with both times of incubation. Individual soil samples were taken from the 100-gram incubated soils for determination of available phosphorus by Bray No. 1 reagent (0.03 N NH_4F + 0.025 N HCl) using a 5-minute shaking period in accordance with a procedure described by Jackson [11]. Approximately one gram dry weight was used for the analysis and the results were corrected on oven-dry weight basis from moisture content determined from each sample.

RESULTS AND DISCUSSION

Fixation of applied phosphate. The relative concentration of applied phosphate recovered was calculated by substracting the value of the check from the value of the phosphate treatments and expressing as percent of the total amount applied. The fractionation values for each two successive dates of incubation showed little variation justifying the grouping of the incubation dates into three periods: the 0 to 1 week incubation as the initial period; the 4 to 8 weeks as the intermediate period; and the 12 to 16 weeks as the final period.

The percent distribution of inorganic fractions from various sources are tabulated in Tables 1-3. Perusal of the data shows that the initial percent distribution generally reflects the relative concentration of constituent phosphates in the original materials. However, the 100 percent acidulated material was an exception. Evidently the soluble monocalcium phosphate in this material was imme-

Table 1. Percent Distribution of Inorganic Phosphorus from Different Sources in Limed Ripley Silt Loam Kept Moist or Flooded for the Indicated Periods.

Treatments	Periods of Observations								
	0 to 1 week			4 to 8 weeks			12 to 16 weeks		
	Ca-P	Al-P	Fe-P	Ca-P	Al-P	Fe-P	Ca-P	Al-P	Fe-P
				Flooded					
RP[a]	48	16	14	39	12	20	42	14	9
20%[b]	38	18	18	30	11	19	27	24	28
100%	7	40	36	2	25	54	2	24	49
$AlPO_4$	6	45	14	0	39	31	2	36	33
$FePO_4$	3	11	58	0	28	51	0	28	45
Ave.	20	26	27	14	23	35	22	25	33
				Moist					
RP	52	16	19	46	10	25	48	12	21
20%	38	21	13	38	10	21	36	11	24
100%	8	44	42	4	23	53	9	24	41
$AlPO_4$	10	44	21	0	43	43	3	44	34
$FePO_4$	2	13	59	0	13	53	2	8	64
Ave.	22	28	31	18	20	39	19	20	27

[a]Ground rock phosphate.
[b]Percent acidulation of rock phosphate.

diately tied up chiefly as Al-P and Pe-P leaving an insignificant amount of P as Ca-P. This continued to be true even after 16 weeks of incubation. The phosphorus from rock phosphate remained largely as Ca-P throughout the experimental period in Wooster and unlimed Ripley soils. The 20 percent acidulated material behaved somewhat intermediate between the rock phosphate and the 100 percent acidulated material. The pure $AlPO_4.2H_2O$ and $FePO_4.2H_2O$ compounds were predominantly present in the initial period as Al-P and Fe-P, respectively. With time of incubation, more Fe-P formed from $AlPO_4.2H_2O$ treatment in soil originally relatively rich in reactive iron (unlimed Repley soil, and more Al-P formed from

Table 2. Percent Distribution of the Inorganic Fractions of Applied Phosphorus from Different Sources in Unlimed Ripley Silt Loam Kept Moist or Flooded for the Indicated Periods.

Treat-ments	Periods of Observations								
	0 to 1 week			4 to 8 weeks			12 to 16 weeks		
	Ca-P	Al-P	Fe-P	Ca-P	Al-P	Fe-P	Ca-P	Al-P	Fe-P
				Flooded					
RP[a]	61	7	0	50	7	2	57	0	12
20%[b]	36	11	16	40	9	11	27	11	37
100%	2	29	35	7	18	37	16	27	53
$AlPO_4$	3	53	19	7	39	27	4	37	37
$FePO_4$	0	19	44	3	20	40	1	22	54
Ave.	20	24	23	21	19	23	21	19	39
				Moist					
RP	51	8	0	42	11	7	57	0	11
20%	27	20	14	42	14	16	27	11	29
100%	3	36	33	17	26	44	6	32	50
$AlPO_4$	6	44	11	10	38	30	3	31	41
$FePO_4$	1	29	42	5	26	43	2	19	44
Ave.	18	27	20	23	21	28	19	20	35

[a]Ground rock phosphate.
[b]Percent acidulation of rock phosphate.

$FePO_4.2H_2O$ treatment in soil originally relatively rich in reactive aluminum (Wooster soil). Liming the acid Ripley soil on the average caused the initial percent distribution of P fractions from these compounds to be maintained relatively constant after 16 weeks. However, the Fe-P increased somewhat where 20 to 100 percent acidulated and $AlP_4.2H_2O$ materials were added. The results indicate that the direction of the fixation reaction is controlled by the ion dominating the soil system, but all ions that are present which have affinity for phosphate exert a common ion effect [13]. In Wooster throughout the incubation periods and to a lesser extent in the Ripley soils initially, aluminum tied up more P than the iron both under moist and flooded conditions. This result agrees with the findings of Chiang [6] that P is initially fixed mostly as Al-P.

Table 3. Percent Distribution of the Inorganic Fractions of Phosphorus from Different Sources in Wooster Silt Loam Kept Moist or Flooded for the Indicated Periods.

Treatments	Periods of Observations								
	0 to 1 week			4 to 8 weeks			12 to 16 weeks		
	Ca-P	Al-P	Fe-P	Ca-P	Al-P	Fe-P	Ca-P	Al-P	Fe-P
				Flooded					
RP[a]	80	0	0	52	7	0	57	2	0
20%[b]	37	20	17	20	32	48	31	33	21
100%	2	40	37	0	42	53	4	40	27
$AlPO_4$	1	52	8	0	64	24	1	57	20
$FePO_4$	1	24	32	5	49	53	0	44	34
Ave.	24	34	23	14	29	36	19	35	20
				Moist					
RP	67	0	0	59	0	11	53	0	1
20%	35	34	0	36	33	13	31	29	17
100%	4	42	18	0	39	28	3	51	31
$AlPO_4$	4	55	7	0	48	22	8	43	25
$FePO_4$	2	33	41	0	41	28	6	48	33
Ave.	22	33	14	19	30	20	18	36	21

[a]Ground rock phosphate.
[b]Percent acidulation of rock phosphate.

in flooded soil. In unlimed Ripley soil, there was a tendency for applied phosphate to be fixed as Al-P initially but for it to be fixed chiefly as Fe-P after 12 to 16 weeks of incubation.

Although there were differences with the various phosphates added, the average percent distributions of inorganic phosphorus fractions were little different whether flooded or moist. The average initial trends Fe-P > Al-P > Ca-P in limed Ripley soil did not change greatly after 12 to 16 weeks of incubation. However, the Fe-P increased noticeably with 20 and 100 percent acidulated and $AlPO_4 .2H_2O$ treatments. In the unlimed Ripley soil the average initial distribution as Al-P > Fe-P > Ca-P and the final distribution Fe-P $\gg$ Al-P > Ca-P. In Wooster soil the trend was Al-P $\gg$ Ca-P > Fe-P with no perceptible change after 12 to 16 weeks. It is evident from these results that iron ultimately bound mostly the applied phosphate in Ripley soils while aluminum phosphate accounted for most of the phosphate applied in Wooster soil. The data for Crowley silt loam soil which is not reported here showed the same trends as the unlimed Ripley soil.

From the results above and in light of the findings of other workers [4, 5, 6], it can be concluded that aluminum and iron play a major role in controlling the concentration of phosphate ions in the soil even at pH as high as 6.3, the approximate pH of Wooster and Crowley soils used in this experiment.

Dynamics of inorganic fractions. The patterns of formation and distribution of the three discrete phosphate compounds in the soil are illustrated in Fig. 1-3. It is clear from the figures that the transformation caused decreases in concentration of one particular species of phosphate compound due to the formation of another species. The effect of moisture conditions is very evident in the case of Al-P and Fe-P fractions in Wooster soil. More Al-P fractions formed under moist condition while more Fe-P formed under flooded conditions. The Ripley soils exhibit the same trend although not significantly differ-

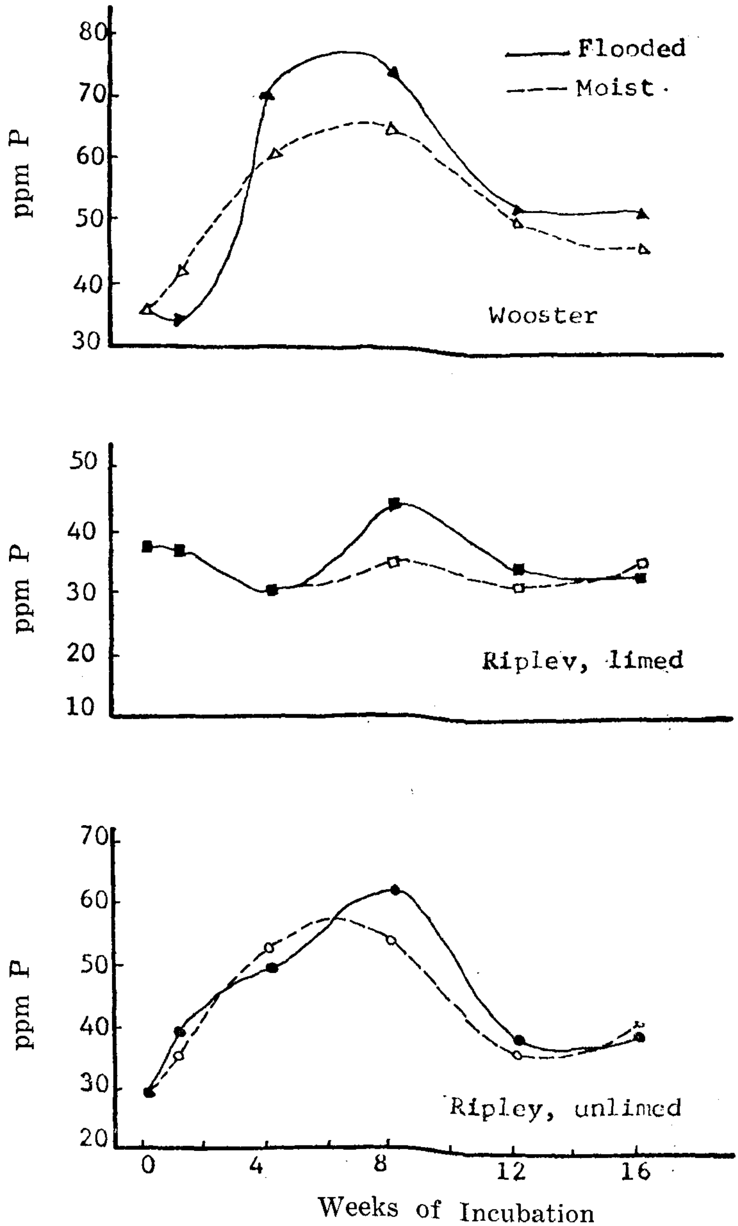


Fig. 1. The concentration of Ca-P fraction in three soils kept flooded or moist for various incubation periods.

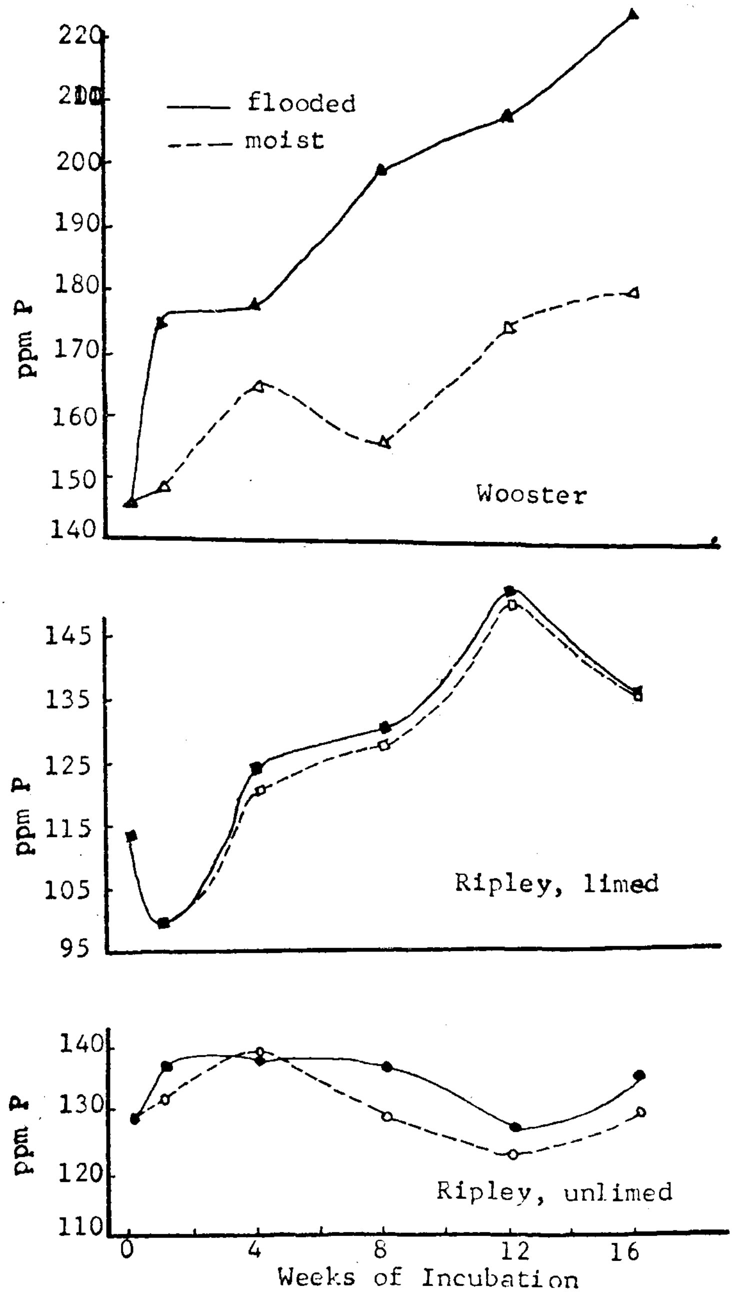


Fig. 2. The concentrations of Fe-P fraction in three soils kept flooded or moist for various incubation periods.

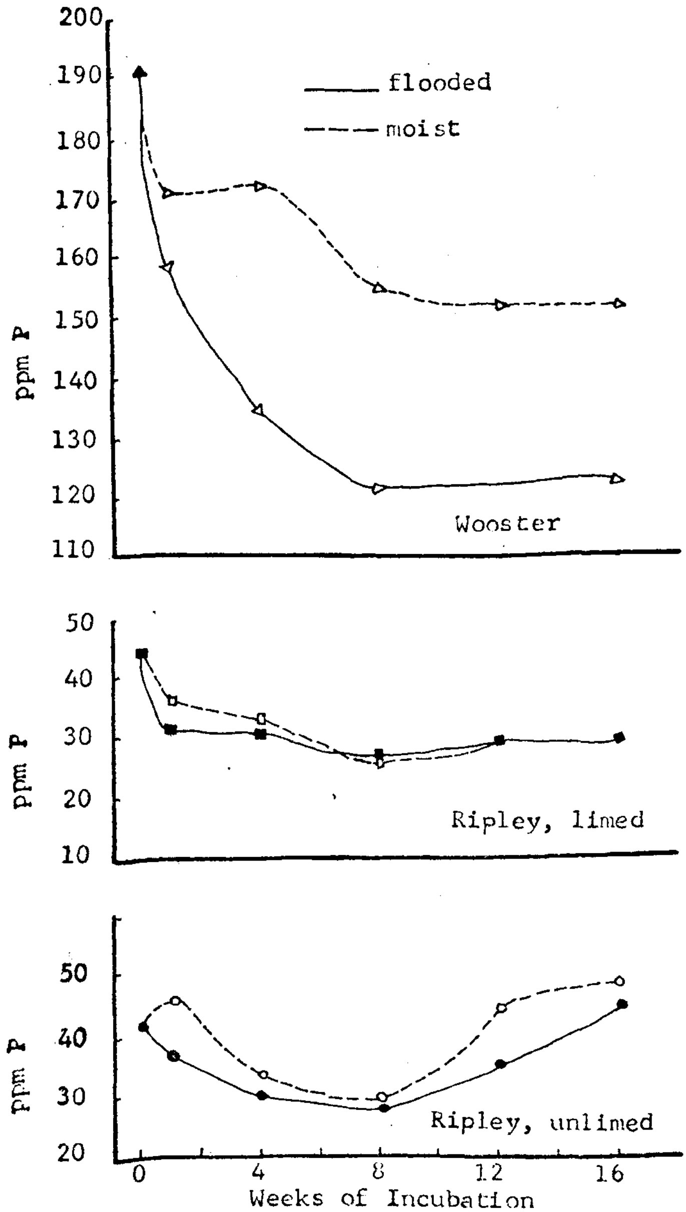


Fig. 3. The concentration of Al-P fraction in three soils kept flooded or moist for various incubation periods.

ent. As seen from Fig. 1, Ca-P fractions increased and reached maximum at the twelfth week of incubation which then attained quasi-equillibrium. The Fe-P fraction increasingly formed in Wooster and limed Ripley soils with time of incubation (Fig. 2). The Fe-P fraction in unlimed Ripley soil was not much affected by prolonged incubation. It is observed that Fe-P tended to decrease after 4 weeks and reached minimum at the 12th week, then picked up in concentration again at the 16th week. The Al-P fraction decreased with time of incubation in Wooster and limed Ripley soils, the extent of the change being more marked in the former (Fig. 3). In the case of the unlimed Ripley soil, the concentration reached minimum at the 8th week and increased thereafter until the 16th week.

In order to understand the kinetics of the transformation of these different fractions, the changes in concentration of the different species were examined. The results show that changes of inorganic fractions from phosphate treated soils paralleled those of the untreated (check). This indicates that the ratio of the different fractions is independent of the amount of soluble phosphate applied for any given period. The average concentration of all the treatments are illustrated in Fig. 4 and 5. The Al-P decreased significantly at the intermediate period. This was followed by increase in the Fe-P fraction in all soils and in Ca-P in Wooster and unlimed Ripley soils. The Ca-P in Wooster and unlimed Ripley soils then decreased after the intermediate stage while Al-P remained more or less constant. The Fe-P fractions continued to increase in all soils with time. It will be noted that Wooster and unlimed Ripley soils showed similar tendencies for change in Ca-P with time. On the other hand, the Wooster and limed Ripley soils showed more similar tendencies for change in Al-P and Fe-P with time. The Wooster soil was limed to pH 6.3 (original soil had a pH 4.5[4]), but with

[4]Data reported by V.V. Volk and E. O. McLean (22)

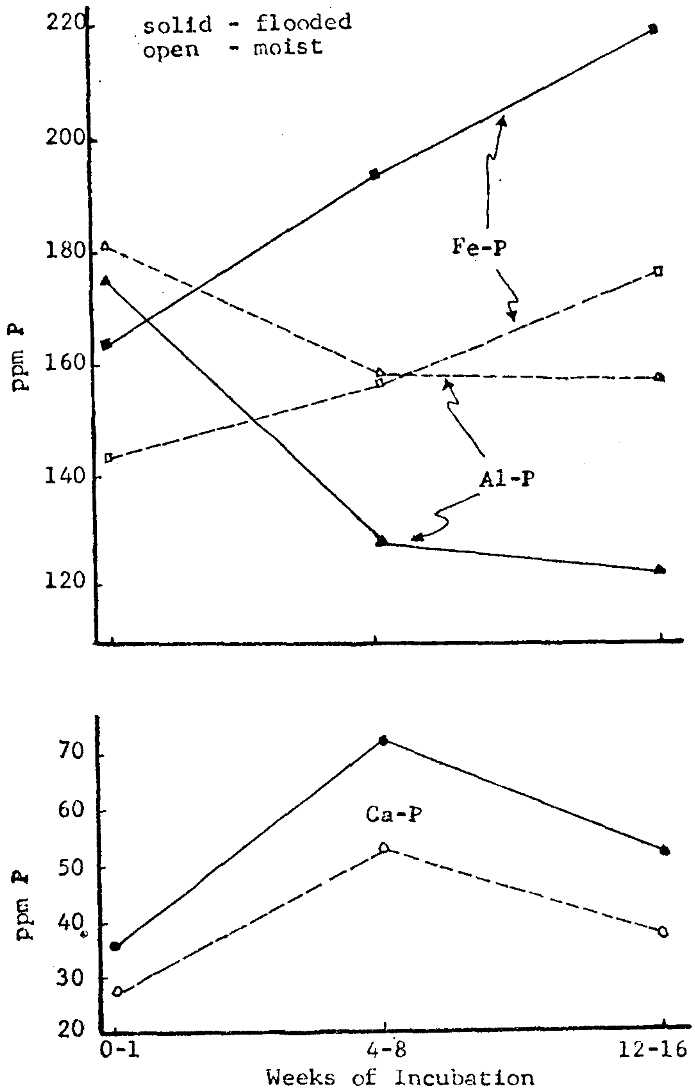


Fig. 4. Average concentrations of the inorganic fractions of phosphorus in Wooster silt loam kept flooded or moist for various incubation periods.

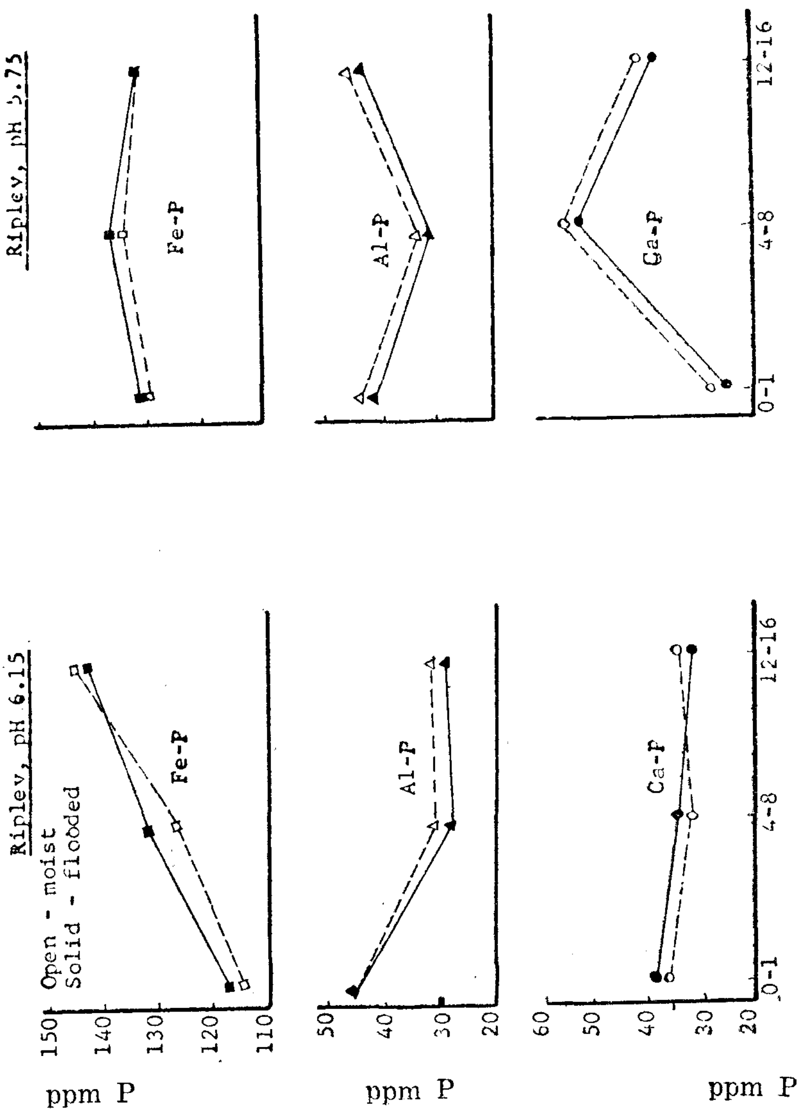


Fig. 5. Average concentrations of the inorganic fraction of phosphorus in limed and unlimed Ripley soils kept flooded or moist for various periods of incubation.

respect to changes in Ca-P, it behaved more like the acid Ripley soil. It should be also recalled from the previous discussion that the trend in initial percent distribution of the applied P bound by the different cations were similar in both soils. The initial concentration of Al-P (and presumably aluminum ion reactivity) in Wooster soil was high. Furthermore, aluminum ion was shown to be associated with acidity in the soil [15]. It appears that Wooster soil still retained some of the characteristics of an acid soil even if it was limed to approximately pH 6.3.

Further examination of the figures shows that Ca-P and Fe-P fractions were formed at the expense of Al-P fractions in Wooster soil and unlimed Ripley soils after 4 to 8 weeks. After this period Ca-P was mostly converted to Al-P in unlimed Ripley soil, the Fe-P fraction increased while Ca-P and Al-P fractions decreased after 4 to 8 weeks. The concentration of Ca-P and Al-P then remained more or less constant, but the Fe-P fraction continued to increase. No explanation could be given in the apparent increase in Fe-P fraction without concomittant change in Ca-P and Al-P fractions. It has been reported that the quantity of reductant soluble and concluded P in the soil could be significant [4, 16]. No report has been made under the conditions of this experiment about the changes of these forms with time of incubation. Glean, *et al* [8], however, reported that liming increased concluded phosphate at the expense of Al-P and Fe-P[5]. It is probable that the concluded or reluctant soluble P was formed to a form extractable by 0.1 Na-OH after the intermediate stage in this study, hence the higher rise in values of Fe-P in limed Ripley soil.

The final concentrations of the different inorganic fractions are summarized in Table 4. Regardless of soil properties, the discrete phosphate species that predominated in the soils at the end of the experiment is the Fe-P fraction both in flooded and moist conditions. Similar results were reported elsewhere [5, 9, 16] for the upland

[5]Unpublished data by K. Wada and M. L. Jackson reported by K. Wada[23].

Table 4. The Average Concentration in (ppm) of the Inorganic Fractions of Phosphorus in Four Soils Kept Flooded or Moist for Sixteen Weeks.

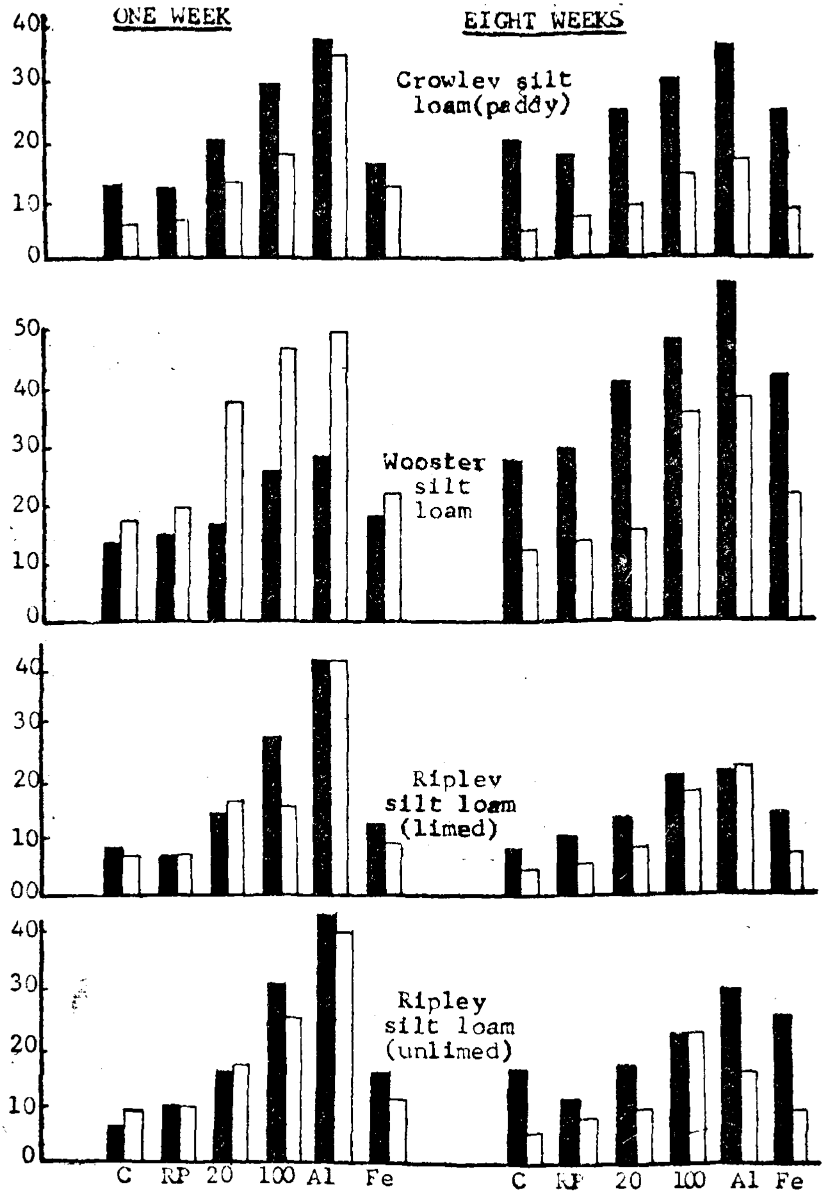
Soils	Ca-P		Al-P		Fe-P	
	Flooded	Moist	Flooded	Moist	Flooded	Moist
Wooster	51.5	44.0	121.3	175.0	218.0	174.0
Crowley[a]	43.7	37.5	29.7	38.0	122.5	113.0
Ripley (u)[b]	39.0	38.3	46.0	45.0	118.0	134.0
Ripley (1)	33.0	31.3	30.3	29.7	141.5	143.7

[a]Concentration at eight weeks of incubation
[b](u) — unlimed and (1) — limed

conditions. Hsu and Jackson [9] reported that reducing conditions promoted formation of Al-P instead of Fe-P, but Chiang [6] had shown that Al-P initially formed in paddy soils gradually converts to Fe-P with time of incubation which is in agreement with the results obtained in the present study. It should be mentioned that the moist treatment in his experiment was not allowed the alternate drying and waiting procedure as is usually done in P fixation studies. Whether this procedure affected the ultimate trends in Al-P and Fe-P fractions and masked the effect of flooded conditions needs some further investigations.

Based on the results obtained, the transformation of inorganic P favors the formation of Fe-P fractions both in flooded and moist soil.

Available phosphorus. The values of available P determined from four soils fertilized with various phosphates are graphed in Fig. 6. As seen from the figure, available P increased after eight weeks of flooding in both the check and phosphate treated soils. However, the available P in the 100 percent acidulated and $AlPO_4.2H_2O$ treatments decreased significantly in unlimed Ripley soils. Under moist condition the available P decreased at the end of the eighth week except in 100 percent acidulation in limed Ripley soils. The amount of P increased with increasing acidula-



Sources of Phosphorus

Fig. 6. Bray No. 1 extractable P in four soils as affected by source of phosphorus and time of moist or flooded incubation. C — Check; RP — rock phosphate; 20% acidulated; 100% acidulated; $AlPO_4$ $.2H_2O$; and $FePO_4.2H_2O$.

tion (increasing solubility) and with $AlPO_4 .2H_2O$ treatments. Since the Bray No. 1 reagent extracts mostly Al-P [19], the results obtained in this study are to be expected. The fact that significant amounts of P were also extracted from the $FePO_4.2H_2O$ treatment indicates that the inclusion of acid results in the dissolution of some other forms of phosphate.

The effect of moisture on the concentration of available P varied initially and appeared to be related to the properties of the soils before incubation. Wooster soil showed more available P under moist conditions while tendency for available P to increase in the Crowley soil upon flooding is typical of a paddy soil [7]. In the case of Wooster soil, the difference in available P appeared to be the consequence of higher initial concentration or Al-P fraction in moist than flooded conditions. The initial effect of moisture condition was not evident in Ripley soils except in the 100 percent acidulation which tended to be higher under flooded than moist condition. Examination of the values of the various phosphate fractions for these soils did not reveal any difference in both moisture conditions. With prolonged incubation, however, available P increased under flooded conditions. This increased availability of P under reduced conditions has been associated with decrease in redox potential [6] and reduction of $FePO_4.2H_2O$ to the more soluble $Fe_3(PO_1)_2.8H_2O$ and increase in solubility of $FePO_4 .2H_2O$ and $AlPO_4 .2H_2O$ cause by the increase in pH accompanying the reduction of acid soils [10]. It has been demonstrated that with water extraction (a condition approximating flooding), iron and aluminum phosphates are slowly hydrolyzed with the release of soluble phosphorus [20].

It appears from the data that Bray No. 1 reagent extracted forms of F other than Al-P fractions. Thus, the increased availability of P with prolonged is not governed solely by the Al-P fractions but by several other factors.

Simple correlation studies relating various fractions of phosphorus to Bray No. 1 extractable phosphorus show a highly positive correlation among the Bray-P, Al-P and Fe-P fractions especially at the eight week of moist and flooded incubations. Ca-P fraction is negatively correlated with Al-P in one week flooded and with Fe-P in one week moist incubation. Multiple correlations further show that all three forms of phosphorus contribute significantly to the amount of Bray-P with Ca-P exerting a negative effect. The results indicate that available phosphorus might be more accurately predicted with longer incubation period and that both Al-P and FeP fractions are good sources of phosphorus for plants growing under the conditions of the experiment.

SUMMARY

The fixation and transformation of phosphorus from ground rock phosphate, 20— and 100 percent acidulated rock phosphate, $AlPO_4.2H_2O$ and $FePO_4.2H_2O$ applied in four soils were investigated under moist and flooded conditions. Applied phosphate was tied up mostly as Al-P in Wooster silt loam and as Fe-P in Ripley silt loam soils and Crowley paddy soil. Phosphorus from rock phosphate remained largely as Ca-P while the soluble monocalcium phosphate in 100 percent acidulation was immediately tied up chiefly as Al-P and Fe-P after 16 weeks of incubation. Iron-P predominated in the untreated soils. Although there were differences with the various phosphates added, the average percent distribution of inorganic phosphorus fractions were little different, whether flooded or moist incubated. The distribution of the total inorganic fractions showed Fe-P to predominate in all soils at the end of 16 weeks. Fe-P formed at the expense of both Al-P and Ca-P. To some extent Al-P formed at the expense of Ca-P with prolongd period of incubation. More Al-P formed under moist condition and more Fe-P formed under flooded condition in Wooster and Crowely paddy soils. These changes were not evident in Ripley soils.

The Bray-P increased under flooded but decreased under moist condition with time of incubation. Correlation

studies showed strong positive association of Al-P and Fe-P with Bray-P especially at longer periods of incubation. It was suggested that Bray-P may be of more value in predicting P availability in flooded soils as long as the soil is subject to reducing conditions before effecting chemical analysis.

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