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Original Research

Speciation of Phosphorus in Phosphorus-Amended and Leached Calcareous Soils Using Chemical Fractionation

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Abstract

Phosphorus (P) application and leaching of P in soils affect P distribution and can significantly influence P losses. The effect of P addition and long-term P leaching on P fractionation in 11 calcareous soils of varying properties was investigated. The soils were spiked with 200 mg P·kg^{-1} and the soil columns were leached with distilled water over 40 days. In order to maximize P leaching and simulate the long-term leaching of P, we added more water to the columns than the mean annual rainfall of the study area. Phosphorus in native and amended soils were fractionated before and after leaching by sequential extraction procedure, in which the P fractions were experimentally defined as exchangeable ($NaHCO_3$-P), Fe- and Al-bound (NaOH-P), Ca-bound (HCl-P), and residual P (Res-P) fractions. The results showed that in native soils the major proportion of P was associated with Ca. Among the P fractions, the percentage of $NaHCO_3$-P and NaOH-P increased, whereas that of Res-P and HCl-P decreased with applied P, indicating former fractions served as a primary sink for P fertilizers added to soil. There were changes in the proportional distribution of P in all the soils, and P was redistributed among the various fractions during leaching of P. In general the proportions of P associated with the NaOH-P and Res-P tended to decrease, with corresponding increases in the HCl-P fractions after leaching, resulting in a minimal amount of P being leached from these calcareous soils. In contrast, there was little decrease in $NaHCO_3$-P fraction with leaching. The moderately labile P forms (NaOH-P and Res-P pools) seem to act as slow-release P sources contributing to long-term P release. The amounts of P extracted in the $NaHCO_3$ and NaOH fractions in native soils were good predictors of cumulative P leached during the 40 days of leaching in these calcareous soils.

Keywords: leaching, calcareous soils, phosphorus, fractionation

Introduction

Continued long-term application of fertilizers can lead to phosphorus (P) accumulation in surface horizons and its availability may increase in soils, which can have negative impacts on the environment as accumulated soil P in the topsoil is a major source of soluble and particulate P in surface runoff [1]. Fertilizer application in the form of animal manure, mineral fertilizers, and sewage sludge leads to significant input of P into soil [2, 3].

Phosphorus in soil is considered to be distributed among several geochemical forms that include soil solution and exchangeable phase, organic matter phase, Ca-bound phase, and Fe- Al-bound phase [4, 5]. Different forms of P exist in different amounts and proportions, depending on soil type and management. These P fractions have remark-

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able differences in mobility, bioavailability and chemical behaviors in soil and can be transformed under certain conditions [6]. Phosphorus fractionation provides an effective approach for investigating soil P availability and P transformation in soil, and the likelihood of its transport [4, 5, 7]. A number of studies on acid soils have examined relative distribution of soils P in various forms in soils that received an application of P and were leached with water using soil columns [8, 9]. Li et al. [8] found that most applied P was in the form of Fe and Al non-occluded P, whereas Silveira et al. [9] indicated that Ca compounds were the dominant P form in surface soil not receiving water treatment residual.

Phosphorus characteristics and fractionation have been studied in many soils under different farming systems [e.g. 10-13]. By contrast, little information is available about P fractions and P transformation under extensive P leaching in calcareous soils, a soil type that is widely distributed in arid and semi-arid regions. The use of P fertilizer increases the risks of P losses in these soils and should be considered for better management of soils. Thus, the present study used 11 calcareous soils aimed to investigate fractionation of P in fertilized soils after long-term leaching.

Materials and Methods

Sampling and P Leaching

The soil samples were taken from the 0-30 cm layer of agricultural soils in Hamedan province, western Iran. The soil samples were air-dried and passed through a 2-mm sieve for laboratory experiments. Soil physical and chemical properties were determined by routine methods [14]. Olsen-P was determined using a soil-to-solution ratio of 1:20 and 30 min shaking [15]. Phosphorus was added to the soil samples at a rate of 200 mg P·kg^{-1} soil as potassium dihydrogen phosphate and incubated for 24 h at 25ºC. The appropriate amount of distilled water was added to bring the soil to the estimated field capacity. After incubation time samples were taken and air dried and used for leaching and fractionation experiments. In order to maximize P leaching and simulate the long-term leaching of P, we added more water to the columns than the mean annual rainfall of the study area (300 mm). A total of 5,122.4 to 7,576 ml (3,083-4,561 mm) was added through each soil column during the 40-day study period, which corresponds to 10.2-15.2 years of precipitation (rain) in the study area. The effluents from the columns displaced at atmospheric pressure were collected and analyzed for P by the Murphy and Riley method [16]. The quantity of leached P was calculated for each soil from the P concentration and the volume of leachate fraction.

Sequential Extraction of P

Sequestration extraction of P was done on native soils and amended soils before and after leaching. After leaching experiments, the soil in the columns was allowed to be freely drained, air-dried, mixed, and subjected to sequential P fractionation using a modified version of the Hedley et al. [4] procedure as the modified procedure outlined by [17]. One gram of each soil sample was weighed into a 50 ml centrifuge tube and different fractions were extracted by the sequential fractionation procedure:

(1) soluble and exchangeable P ($NaHCO_3$-P), soil extracted with 25 ml 0.5 M $NaHCO_3$ for 16 hours at room temperature with continues agitation
(2) Fe- and Al-bound P (NaOH-P), residue from the first fraction extracted with 25 ml 0.1 M NaOH for 16 hours with continuous agitation
(3) Ca-bound P (HCl-P), residue from second fraction extracted with 1 M HCl for 16 hours with continuous agitation
(4) residual P (Res-P), residue from the last fraction extracted with a 5:2 mixture of concentrated H_2SO_4 and $HClO_4$ and determining P from the digest [4].

The amount of P in all extractions was determined using the colorimetric ascorbic acid method [16].

Results and Discussion

Soils

The soils were sandy loam to clay in texture, and were neutral to alkaline, and different in soluble salts content (EC 0.12 to 0.20 dS·m^{-1}) (Table 1). Clay contents ranged from 28 to 488 g·kg^{-1}, CEC ranged from 14.3 to 31.3 cmol$_c$·kg^{-1}, and organic carbon contents were low in all samples, ranging from 6.4 to 38.8 g·kg^{-1}. The Olsen extractable P ranged from 3.7 to 42.6 mg·kg^{-1}.

Phosphorus Fractionation

Phosphorus Fractionation in Native Soils

Concentrations of different P fractions in the native soils are shown in Fig. 1a. Native soils had a higher concentration of P in HCl-P and Res-P fractions. The $NaHCO_3$-P varied greatly, ranging from 22.1 to 44.2 mg·kg^{-1}. NaOH-P, HCl-P and Res-P ranged from 55.1 to 92.0 mg·kg^{-1}, from 314.4 to 1,110.2 mg·kg^{-1}, and from 161.1 to 345.5 mg·kg^{-1}, respectively (Fig. 1a). Sequential P fractionation indicated different P distribution in the native soils. The HCl fraction of P accounted for a higher percentage of the total (45.3 to 70.5%) than Res-P (21.0 to 40.9%) and NaOH-P (5.7 to 12.0%). This suggests that the dominant fraction of P in the native soil was HCl-P, with very low amounts in the EXCH fraction, indicating that P had a preference for Ca-bound at the expense of other fractions and that background P was mainly immobilized within the crystalline and secondary minerals, and P is essentially non-labile. The average distributions obtained were as follows: HCl-P (59.1%), Res-P (29.6%), NaOH-P (8.4%), and $NaHCO_3$-P (2.9%). These results were similar to the results observed by Alvarez-Rogel et al. [18] and Jalali and Ranjbar [13], who found that P was mainly bounded to Ca compounds (HCl-P).

Table 1. Some chemical and physical properties of studied soils.

Soil No.	Classification	pH	EC	CCE	CEC	OM	Clay	Silt	Sand	Olsen-P	Cumulative P leached	Land use
			$dS \cdot m^{-1}$	$g \cdot kg^{-1}$	$cmol_c\ kg^{-1}$	$g \cdot kg^{-1}$				$mg \cdot kg^{-1}$	$mg \cdot kg^{-1}$	
1	Typic xerorthents-Typic xerofluvents	7.21	0.147	121	22.78	6.4	68	214	718	20.7	96.1	Rangeland
2	Typic xerorthents-Typic xerofluvents	7.13	0.140	106	17.60	12.9	108	194	698	22.2	197.8	Leafy vegetables
3	Typic xerorthents-Typic xerofluvents	6.80	0.140	57	15.73	38.8	28	254	718	4.3	118.7	Orchard
4	Typic xerorthents-Typic xerofluvents	7.05	0.130	88	20.94	11.3	48	274	678	3.7	156.5	Rangeland
5	Typic xerorthents-Typic xerofluvents	7.02	0.163	41	14.28	35.2	28	274	698	6.5	164.1	Rangeland
6	Typic xerorthents-Typic xerofluvents	7.15	0.127	75	21.17	35.0	88	414	498	42.6	163.6	Orchard
7	Typic calcixerepts	7.54	0.134	185	25.04	9.5	208	354	438	5.2	174.8	Wheat
8	Typic xerorthents-Typic xerofluvents	7.36	0.205	145	22.75	15.3	288	274	438	17.5	134.3	Wheat
9	Typic calcixerepts	7.50	0.179	216	28.04	9.8	308	368	324	10.5	124.5	Potato
10	Typic xerorthents-Typic xerofluvents	7.43	0.130	172	26.77	6.6	248	428	324	11.6	117.8	Garlic
11	Typic xerorthents-Typic xerofluvents	7.37	0.116	245	31.35	6.7	488	308	204	14.5	146.2	Garlic

Vepraskas and Faulkner [19] indicated that P mainly precipitates as calcium phosphates or co-precipitates with carbonates in soils with pH>7. The NaOH-P fraction had been reported to be the largest fraction for P by many researchers in most tropical soils under continuous cultivation [20-22], whereas Aulakh and Parsricha [23] and Aulakh et al. [10] reported that in less weathered and subtropical soils, the excess P from fertilizer tends to accumulate as Ca-P. Dobermann et al. [11] and Kuo et al. [24] found that NaOH-P fraction was higher than the other fractions of P in acid upland soils and less weathered soil.

Less than 8.4% of total P associated with the NaOH pools. Because this fraction is assumed to represent primarily Al- or Fe-bound P [25], the effect of Al hydrous oxides in these soils is low.

Phosphorus Fractionation in Amended Soils

Sequential P fractionation revealed distinctly different P concentrations and distributions in the native soils receiving 200 $mgP \cdot kg^{-1}$ at the beginning of the leaching study (Fig. 1b). When P was added to the soils, the concentration of P bound to all fractions (except HCl-P) increased. For all soils, $NaHCO_3$-extractable P (labile P) was considerably increased, particularly in soils 2, 4 and 11, whereas NaOH-extractable P was increased two- to four fold compare with native samples. The $NaHCO_3$-P concentration varied greatly, ranging from 159.2 $mg \cdot kg^{-1}$ (soil 1) to 391.4 $mg \cdot kg^{-1}$ (soil 11). The P association in $NaHCO_3$-P fraction ranged from 17% to 29.7% in soils 4 and 6, respectively. These results are in line to laboratory experiments reported by other works [26, 27] in which the P application increased labile-P content in the soil. The shifts in the $NaHCO_3$-P fraction are particularly important because this fraction is thought to represent the labile pool of inorganic P [28], and is typically well correlated with P losses [29].

The applied P entered in $NaHCO_3$-P, NaOH-P, and Res-P fraction was decreased, but there were little changes in the percentage of Res-P between native and amended soils. For all soils, HCl-P was the dominant P form, and there is high variation between soils (13.7-51.5%), whereas its percentage was decreased, suggesting that some of the HCl-P fraction had been transferred to $NaHCO_3$-P and NaOH-P fractions. These results are in line with laboratory experiments reported by Halajnia et al. [12], in which P application in some calcareous soils increased P concentrations in all fractions with the exception of HCl-P.

Song et al. [30] studied P fractions in Udic Mollisols under different ecosystems. They found that with increasing P inputs, Ca-P and Al-P increased, whereas Fe-P and occluded P decreased, suggesting that the applied P had been transferred to these sparingly soluble forms. They also concluded that an excessive application of chemical P fertilizers over crop demands could thus decrease the use efficiency of P fertilizers and should be avoided. Some authors have concluded that the NaOH-P served as a primary sink for P fertilizers added to soil [20-22, 24], whereas others have reported involvement of NaOH-P, HCl-P, and Res-P [31, 32].

In some less weathered and subtropical soils, the excess P from fertilizer tends to accumulate as Ca-P [10, 23, 33]. Kuo et al. [24] indicated that when the amount of applied P was below that removed by corn, indigenous soil NaOH-P acted as a source of available P for the plant.

Phosphorus Fractionation in Amended Soils after Leaching

Concentrations of different P fractions in the amended soils after leaching are shown in Fig. 1c. For all studied soils it was observed that after leaching, the P concentration in all fractions (except HCl-P) was decreased. Phosphorus concentration in each fraction varied greatly (NaHCO$_3$-P, 130.8 to 378.2 mg·kg^{-1}, NaOH-P, 74.3 to 128.5 mg·kg^{-1}, HCl-P, 323.8 to 1,220.1 mg·kg^{-1}, and Res-P, 67.1 to 331.3 mg·kg^{-1}). The P concentration in HCl-P fraction was increased during leaching in all soils. The greater P concentration in HCl extracts could be attributed to the abundant Ca, Mg, and CaCO$_3$ present in soils.

After leaching, distribution of P in amended soils was changed and P was associated mainly with HCl-P fraction (37.2 to 75.7%), Res-P (4.4 to 25.3%), NaOH-P (5.2 to 14.3%), and NaHCO$_3$-P (13.2 to 32.2%). Our findings are different from other works performed in acid soils [8] that studied relative distribution of soil P in various forms in the surface, and Spodic horizon of a Spodosol that received an application of P and leached with eight pore volumes of water. They found that 67-77% of total P was in the form of Fe and Al non-occluded P, indicating that applied P either precipitated with or was adsorbed on Al and Fe oxides.

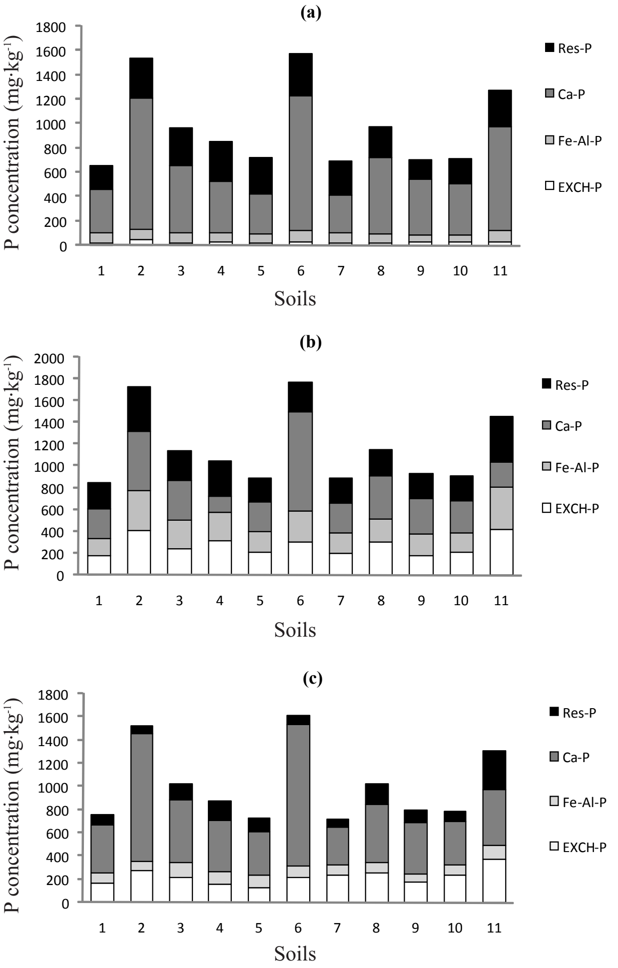

Fig. 1. Concentration of different P fractions in (a) native soils, (b) amended soils before leaching, (c) amended soils after leaching.

Overall, total P decreased with time, especially for soils 7 and 5. For all soil samples, Res-P and NaOH-P fractions became smaller with leaching, while HCl-P became larger. As suggested by Silveira et al. [9], P was redistributed among the various fractions with leaching. Although P concentration in NaHCO$_3$-P fraction decreased for the soil samples, no substantial changes in the NaHCO$_3$-P fraction were observed and the relative P distribution after the leaching trial (an average of 22.5%) remained similar to that at the beginning of the experiment (an average of 23.1%). The average distributions obtained were as follows: HCl-P (53.9%), Res-P (13.3%), NaOH-P (10.4%), and NaHCO$_3$-P (22.5%). Our findings are in agreement with Silveira et al. [9], which reported a decrease of Res-P fraction by the end of the leaching experiment. They also are in agreement with Zhang et al. [29], who suggested that the NaOH-P pool are potentially labile and subject to P loss [29].

While several studies have reported discrete P fraction native P leaching [34, 35], our results suggest that the more labile P fractions (NaHCO$_3$ and, perhaps, NaOH fractions) could be buffering P in solution, followed by redistribution of the resistant P forms into the more labile solid phases [9]. In this case, leaching affects the overall P distribution in the soils. It is suggested that P compounds with low solubility can be transformed to more soluble forms, or insoluble compounds are dissolved under depletion of soil P [30]. The moderately labile P forms (HCl-P and Res-P pools) seem to act as slow-release P sources, contributing to long-term P release. Silveira et al. [9] suggested that after each leaching event, the various P forms re-establish a new quasi-equilibrium, which remains relatively stable until the solution is depleted due to subsequent leaching events.

A significant fraction of applied P was retained by the soils, resulting in a minimal amount of P being leached from these calcareous soils.

Correlations between P Fractions with Soil Properties

The concentration of P in four fractions in native soils and amended soils before and after leaching was correlated with soil variables and cumulative P leached. The Olsen-P was positively correlated with HCl-P fraction in native (r=0.751; $P<0.01$) and amended (r=0.873; $P<0.01$) soils before and after leaching, and NaHCO$_3$-P fraction in native soils was positively correlated with cumulative P leached (r=0.595; $P<0.05$) from soils. The role of Ca-bound P in removing labile P (Olsen-P) from the solution through an adsorption or precipitation mechanism may be seen from the significant correlation between Olsen-P and Ca-bound P (HCl-P fraction). The Res-P fraction was correlated with

$CaCO_3$, but not significantly. The clay content was positively correlated with $NaHCO_3$-P (r=0.719; $P<0.05$) and Res-P (r=0.630; $P<0.05$) fractions in soils after leaching. This may imply that the clay content in these soils could retain P and prevent P leaching from soils. Several studies have reported good correlation between P losses and soil tests [36, 37].

Conclusions

Most of the native and applied P was extracted using HCl, indicating that P is mainly bounded to Ca compounds. The $NaHCO_3$-P fraction, which was considered an indication of the soluble form in soil, were increased in amended soils and did not change during leaching. The NaOH and Res-extractable P reduced during leaching, while HCl-P increased, indicating that a significant fraction of applied P was retained by the Ca compounds, resulting in a minimal amount of P being leached from these calcareous soils. This study clearly demonstrated that after each leaching event, there may be some internal transformations among the various soil P forms, establishing a new equilibrium that remains relatively stable until the solution is depleted due to subsequent leaching events.

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