

EFFECT OF MINERAL AND MANURE FERTILIZERS ON PHOSPHORUS LEACHING UNDER DIFFERENT LAND USES IN CALCAREOUS SOILS

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Abstract

A study on soil columns was performed to investigate P leaching from calcareous soils under different land uses treated with 200 mg P kg⁻¹ soil as superphosphate (S) or 30 g kg⁻¹ soil as cattle manure (A). The soil samples were incubated for 17 days before the leaching process. Overall, comparison of the five leaching pore volumes showed that the concentration of P in leachates increased in the final pore volumes (PVs) and was higher for soil under vegetable use treated with manure for both locations. The lowest values were recorded for soils amended with superphosphate and uncultivated soils. On comparing the various concentrations of CaCl₂, the highest P concentration was found in the leachate of 10 mM of CaCl₂ solution. Derabin's P concentration in the leachate was nearly twice that of Batil. The differences in soil properties are the main reasons for the different behaviours of P leaching from both soils. The results showed that all the leachates of all CaCl₂ concentrations and different soils were undersaturated with respect to dicalcium phosphate dihydrate (DCPD), dicalcium phosphate (DCP), octacalcium phosphate (OCP), tricalcium phosphate (TCP), and hydroxyapatite (HA) through the leaching process. The saturation index (SI) values for soil treated with superphosphate and uncultivated soil were lower than those cultivated with manure.

Keywords: Phosphorus; incubation; pore volume; saturation index; fertilizers.

Introduction

Understanding soil phosphorus behaviour is important to ensure appropriate plant nutrition because soil generally has a very low concentration of phosphorus (P). In addition, P in soils becomes unavailable to plants for many reasons. In calcareous soils that contain significant amounts of CaCO₃ (Thompson 2007), fixation of P results in conversion to stable forms (Al-Rohily et al. 2013). When P is added to the soil as fertilizer, 90% of the inorganic P is fixed by the soil (Sharpley and Beegle 2001; Grant 2003) which is the major factors that affect P availability in calcareous soils. In alkaline soils, phosphorus chemically binds with Ca to form insoluble Ca-phosphates (Brady et al. 2008) and with time, the applied P tightly binds to adsorption sites of CaCO₃ (Rajput et al. 2014). Poor P availability in calcareous soils represents a major constraint to crop production (Ahmed et al. 2009). But excessive P fertilization results in an increase in P concentration in agricultural soils and leads to the build-up of soil P and increased risk for P losses. The risk of nutrient loss from soils treated with animal manure has been reported in many studies (Pizzeghello et al. 2016).

Long-term and continuous fertilization of medium to coarse-textured soils leads to increased P mobility downward (Mozaffari and Sims 1994; Zhang et al. 1995), because of annual applications of phosphate fertilizers, which make microbial immobilization and plant uptake unable to remove the entire P from the soil solution. Surface water nutrient enrichment can occur environmentally at very low phosphorous concentrations. In some cases, low phosphorous concentrations that reach $20 \mu\text{g l}^{-1}$ are adequate to induce eutrophication and algal growth (Powlson 1998). Koopmans et al. (2007) and Sui et al. (1999) stated that application of P manure in large amounts exceeding crop needs can be a source of downward movement of P through the soil. Because of their high capacity to adsorb P, calcareous soils were thought to have less leaching of P into ground water (Olson et al. 2010). Similarly, James et al. (1996) stated that for irrigated calcareous soils, the risk of ground water P contamination due to manure application was negligible because the soil had a large capacity to retain P. It was also reported that P sorption increased with increasing P concentration in calcareous and non-calcareous soils (Jalali 2007). In contrast, Eghball et al. (1996) reported that despite the high P sorption capacity, leaching of P may be an issue in irrigated calcareous soil. When the P sorption capacity of soil approaches saturation, the risk of P loss to drainage and ground water increases (Schoumans and Groenendijk 2000). Many studies demonstrate very high P concentrations in surface and subsurface runoff after fertilizer or manure application (Liu et al. 2012). They stated that such incidental P losses could be reduced by limiting the application rates of P and incorporating fertilizers or manure into the soil soon after the application of fertilizers. Uusi-Kämppä and Heinonen-Tanski (2008) showed that slurry incorporation and injection can reduce P mobility in soil and total P losses in runoff by up to 80–90 %.

Phosphorus stability and retention are affected by factors that vary between land uses (Sharpley and Beegle 2001). Jalali and Ahmadi Mohammad Zinli (2011) found out that significantly different quantities of P were released under different land uses.

Because of water scarcity, there was an increase in the irrigation of soil by ground water containing large quantities of calcium ions, which encouraged P retention in the Ca-bond fraction. Jalali and Kolahchi (2009) indicated that P leaching is initially rapid, it then decreases with successive leaching. After 20 pore volumes, the amount of total P leached ranged from 1.7 to 1.8 kg ha^{-1} . Sharpley and Smith (1989) showed that CaCO_3 and clay content controlled the rate of phosphorous release in calcareous soils. In spite of the fact that there are many P minerals present in the soil along with Ca-P minerals, as evidenced by the high concentration of Ca^{+2} and the low solubility of Ca-P in the applied CaCl_2 solutions, Hansen and Strawn (2003) indicated that Ca-P minerals were the most dominant minerals in the soil.

Column experiments are usually used to study the chemistry of soil and leachates, as well as to provide information about the mobility and release of elements in the soil (Voegelin et al. 2003; Qureshi et al. 2004). In Iraq, the available P status is poor and P availability to the crops is a problem, especially in calcareous soils. Therefore, more studies on this problem are required to conserve P resources. So, it is worthwhile to (1) study the influence of mineral and manure

fertilizers on P status under different land uses, (2) test the risk of P leaching using different concentrations of Ca solutions and (3) determine different forms of P in the leachates.

Materials and Methods

Sampling collection

Samples from Derabin (D) (Lat. 37°04'56.60" N, Long. 42° 24' 44.56 "E) and Batil (B) (Lat. 36°57'44.87" N, Long. 42°40'15.55" E) in Duhok-Kurdistan Region-Iraq were collected from (0–30 cm) depth on the basis of a composite sampling method (i.e. ten single samples were collected from each site, mixed well to prepare one composite sample representing the site). The study soils covered different land uses with a wide range of calcium carbonate content. For each location, samples were taken after harvest of wheat (W), harvest of vegetables (V) and uncultivated area (Un) as a control. The soil samples were air-dried, ground, sieved through a 2-mm sieve, and exposed to different chemical and physical properties. A summary of some important properties is given in Table 1.

Laboratory Analyses

According to Anderson and Ingram (1994), the soil reaction (pH) and electrical conductivity (EC) of soil were measured in a 1:2 soil-water ratio. Standard procedures outlined by Black (1965), were followed to determine the soluble cations (Ca^{++} , Mg^{++} , Na^+ , K^+) and anions (HCO_3^- , CO_3^- , Cl^- , and $\text{SO}_4^{=}$). Cation exchange capacity (CEC) was measured by using ammonia acetate (1N) NH_4OAC extraction (Black 1965). The Kjeldahl digestion method was used to determine total nitrogen (TN) (Bremner 1965). By extracting the soil with 0.5 M NaHCO_3 at pH 8.5, available P (A.v P) was measured according to Olsen and Sommers (1982). Soluble P in the soil leachate was measured according to Murphy and Riley (1962). The total P (TP) was determined following wet digestion with H_2SO_4 (Cresser and Parsons 1979), and the P was determined according to Murphy and Riley (1962). The particle size distribution was determined using the hydrometer method, which was carried out in accordance with Gee et al. (1986).

Table 1. Some physiochemical properties of the used soils.

Sample sites		Derabin			Batil		
Land use		W	V	Un	W	V	Un
pH		8.42	8.19	8.3	8.42	8.28	8.47
EC dSm ⁻¹		0.56	1.01	0.59	0.63	0.7	0.53
Ca^{+2}	mmol L ⁻¹	4.4	8	4.3	4.2	4.4	4.4
Mg^{+2}		2.4	2.4	2.3	2	2.8	1.6
Na^+		0.28	0.48	0.48	0.32	0.77	0.36
K^+		0.14	0.16	0.23	0.25	0.23	0.5
CO_3^-		0	0	0	0	0	0
HCO_3^-		4.8	2.8	4.6	4.6	3.6	4.4
Cl^-		2.4	2.4	2.6	2	2	2

SO ₄ ⁻²		0.02	5.84	0.11	0.15	2.87	0.12
CaCO ₃		354.3	276	350.3	216.3	151.7	214.7
A-CaCO ₃	g kg ⁻¹	110	70	150	100	45	90
Gypsum		38.49	24.29	37.48	65.46	45.75	61.78
TN		1.7	1.5	1.7	1.7	2.2	1.1
OM		17.73	10.37	14.38	20.07	25.76	20.07
A.v P mg l ⁻¹		14.85	8.7	7.51	8.02	27.14	6.14
CEC Cmolc kg ⁻¹		28.3	19.32	28.88	30.86	23.25	30.01
Clay		495	345	520	537	362	520
Silt	g kg ⁻¹	250	175	250	348	373	350
Sand		255	480	230	115	265	130
Texture		C	SaCL	C	C	CL	C
SaCL: Sandy Clay Loam; CL: Clay loam; C= Clay.							
W: wheat; V: vegetables; Un: uncultivated							

The content of organic matter (OM) was determined by using the method outlined by Walkley and Black (1934). Total calcium carbonate was measured with 1N HCl using a Calcimeter apparatus. Titrimetrically, the active calcium carbonate (A-CaCO₃) was measured using the Droulinean method (Kozhekov and Yakovleva 1977). The content of gypsum was measured using the method described by Omran (2016). Cattle manure was analysed as shown in Table 2 following the same procedure mentioned earlier.

Incubation period

Six hundred grams of air-dry soil were taken from each location under different land uses and treated with either inorganic P (200 mg P kg⁻¹ soil) as tri-superphosphate (S) or cattle manure (30 g kg⁻¹ soil). The soil and the fertilizers were mixed well, then each treatment placed in plastic bag and incubated for 17 days at 25 °C in laboratory. An appropriate amount of tap water was added to bring the soil to the estimated field capacity. The samples were kept moist by adding tap water as needed. After the given time, the soil was air-dried before being used for leaching experiments, and sub-samples were taken and analysed for Olsen P.

Table 2. Some characteristics of cattle manure.

pH (1:5)	EC dSm ⁻¹ (1:5)	TN g kg ⁻¹	TP g kg ⁻¹	C: N ratio	C: P ratio	OM g kg ⁻¹	OC g kg ⁻¹
7.54	10.13	16.32	4.63	17.85	62.85	501.8	291.04

Column leaching experiment

Six plastic (PVC) sewage pipes (6.7 cm in diameter and 20 cm long) were used for each soil site, giving two replicates of each P fertilization treatment and uncultivated soil (i.e. 2

replicates for soil treated with mineral fertilizer, 2 replicates for soil treated with manure and 2 replicates as control for uncultivated soil). The bottom of each column was drilled; the filter paper was fitted, and a small amount of fine gravel was placed above the filter for easy water movement inside the column. Then, each soil was placed into the columns at a height of 15 cm by uniform tapping with a wooden rod to achieve a uniform bulk density. After packing, the top soil was covered with filter paper to protect the surface from disturbance.

The columns were positioned vertically in a wooden stand. A leaching experiment was conducted under saturation conditions at room temperature. Soils were leached with CaCl_2 solutions of various concentrations (5, 10, and 15 mM CaCl_2). About 200 ml of each solution was added to the top of each column daily. Effluents from each leaching stage were collected for five days in an individual plastic bottle for each column and analysed for pH, EC, Ca, and $\text{CaCl}_2\text{-P}$. In addition, the volume of leachate was recorded. The time interval was enough for most of the applied solution to drain and be collected just before the next solution addition.

Saturation indices (SI)

The geochemical speciation model Visual MINTEQ version 2.30 was used to compute the saturation indices (SI) for P in leachates. To calculate the saturation indices (SI) for P in leachates, the following equation was used:

$$\text{SI} = \log \left(\frac{\text{IAP}}{\text{K}_{\text{eq}}} \right) \quad (1)$$

Where IAP denotes the ion activity product and K_{eq} represents the solid phase equilibrium constant, (McDowell et al. 2003). For the input of Visual MINTEQ, pH, EC, and P were measured in the leachates. In addition, the ionic strength (I) was calculated using the following equation (Griffin and Jurinak 1973):

$$I = 0.013 \text{ EC} \quad (2)$$

Statistical Analysis and Software Package

The saturation index of leachates was carried out using the Visual MINTEQ 2.30 program (Gustafsson 2005). Microsoft Excel software was used to determine descriptive statistics of soil properties between some selected variables.

Results and Discussion

Phosphorus Leaching

The data regarding the physio-chemical properties of both soils presented in Table 1 reveal that the soils were clayey to clay loam, with a high pH, low in available P and organic matter (OM), and highly calcareous. concerning of texture, many studies have found out that clay content has a significant impact on P sorption (Kaloi et al. 2011). Additionally, the silty clay and silt soil fraction also have a great influence on P sorption, as reported by Awasthi and Pathak (1971) and Yadav et al. (2017).

The results in Figure 1 for Batil soil cultivated with wheat and amended with cattle manure showed a slight difference in P concentrations in the leachates throughout different pore volumes (PVs) and at all CaCl_2 solutions. The lowest P concentrations in the leachates recorded from the third pore volumes were 0.28, 0.20, and 0.18 mg l⁻¹, while the highest P concentrations were 0.34

mg l^{-1} from the first pore volume, 0.25, and 0.28 mg l^{-1} from the final pore volumes with 5 mM, 10 mM, and 15 mM CaCl_2 , respectively. In the soil cultivated with vegetables and amended with cattle manure, in a 5 mM CaCl_2 solution, there was an increase in P concentrations in the leachates starting from the initial pore volume to the final pore volume. The solutions of 10 mM and 15 mM CaCl_2 had the same trend, with the exception of pore volume three. The highest P values were 0.73, 0.79, and 0.58 mg l^{-1} in the final pore volumes for 5, 10, and 15 mM CaCl_2 , respectively. The results revealed very low P concentrations in the leachates of the soils cultivated with wheat and vegetables amended with superphosphate (S) and uncultivated soil. One reason behind superphosphate application decreasing of P leaching could be that the application rate was not high enough to promote the concentration of P in leachate to a significant extent. Bolan et al. (1994) and Eghball et al. (1996) stated that the addition of organic acids can increase the availability of P in the soils by decreasing the adsorption of P and increasing solubilization of P compounds. This may even increase the P leaching potential compared with mineral P fertilizer (Tarkalson and Leytem 2009). Similar results were reported by Siddique and Robinson (2003). The addition of organic material to mineral soils increased the P availability. Liu et al. (2012) also reported that P leaching from treatment with manure application was greater than where mineral P had been used. In general, leachates from the wheat (W) soil were lower than those from the vegetable (V) soil when both were amended with cattle manure. This may be attributed to the high release of P and the high concentration of P initially found in soil (V) of 0.8 mg l^{-1} compared to 0.42 mg l^{-1} for (W) soil. The soil (V) received a large amount of fertilizer through the rotations of many vegetables grown yearly. In addition, soil (W) had higher CaCO_3 and clay content compared to soil (V) (Table 1). Soil phase dicalcium phosphate may control P solubility in soils with high CaCO_3 content (Von Wandruszka 2006). Kaloi et al. (2011) pointed out that the P release increased where there was less clay content.

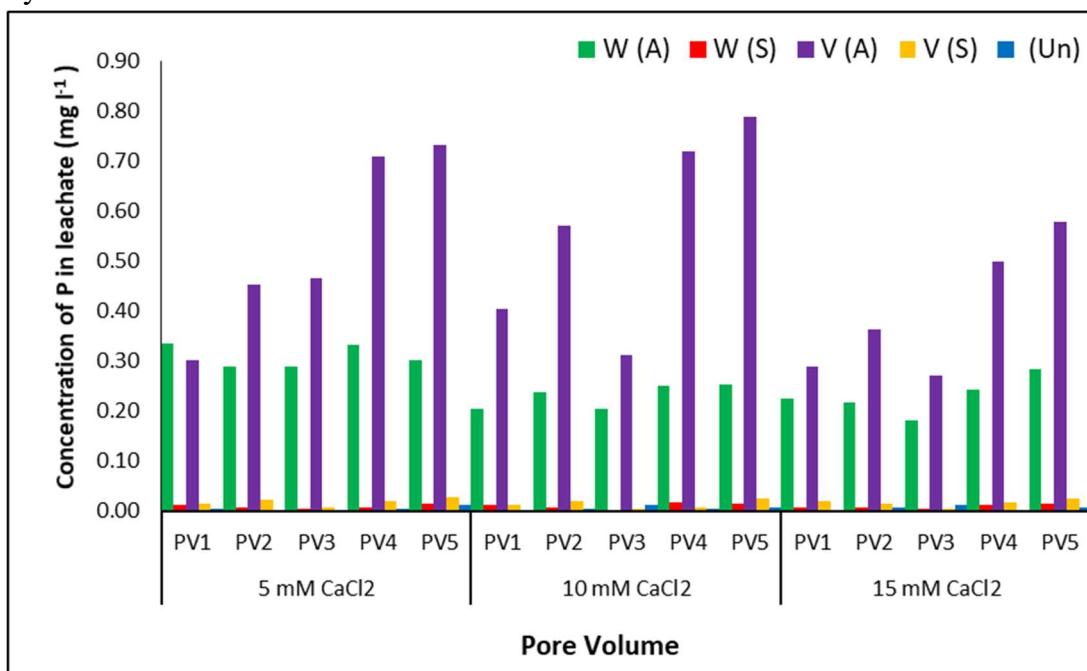


Figure 1. Phosphorus in leachates of the Batil soil leached with different CaCl_2 solutions.

(W (A): wheat amended with manure; W (S): wheat amended with tri-superphosphate; V (A): vegetables amended with manure; V (S): vegetables amended with tri-superphosphate).

Regardless the concentrations of the CaCl_2 solutions, P leaching generally increases with an increase in pore volume. On comparing the various concentrations of CaCl_2 , the highest P concentration was found in leachates of 10 mM CaCl_2 solution. McDowell et al. (2001) stated that extractable CaCl_2 -P has previously shown to be a useful indicator for P leaching. The 10 mM CaCl_2 has comparable ionic strength to natural soil solution (Robbins et al. 1999). The relatively low leaching of P in 15 mM CaCl_2 solutions was likely to be due to the high concentration of soluble Ca used in the solution; which enhanced the formation of Ca-P mineral and decreased available P. Similar results were found out by Jalali and Kolahchi (2009).

In Figure 2, for Derabin soils, there were also differences between the soils cultivated with wheat and those with vegetables amended with cattle manure. The P concentrations in the leachates of these soils were much higher than those of the same soils but amended with superphosphate (S). Similar results were mentioned by Sui et al. (1999), Sui and Thompson (2000) and Halajnia et al. (2009) who reported that p sorption is rapid in the first few hours or days, then it slows down over time. They reported that manure addition generally has significant dynamic impacts on phosphorus fractions because P is gradually released from manure and converted by the time to unavailable forms. In the soils cultivated with wheat and vegetables amended with cattle manure and treated with 5 mM CaCl_2 , there was an increase in P concentrations in the leachates starting from the initial pore volume of about 0.44 and 0.58 mg l⁻¹ up to the highest values of about 1.13 and 1.73 mg l⁻¹ in the fourth pore volume and then decreased in the final pore volume, respectively.

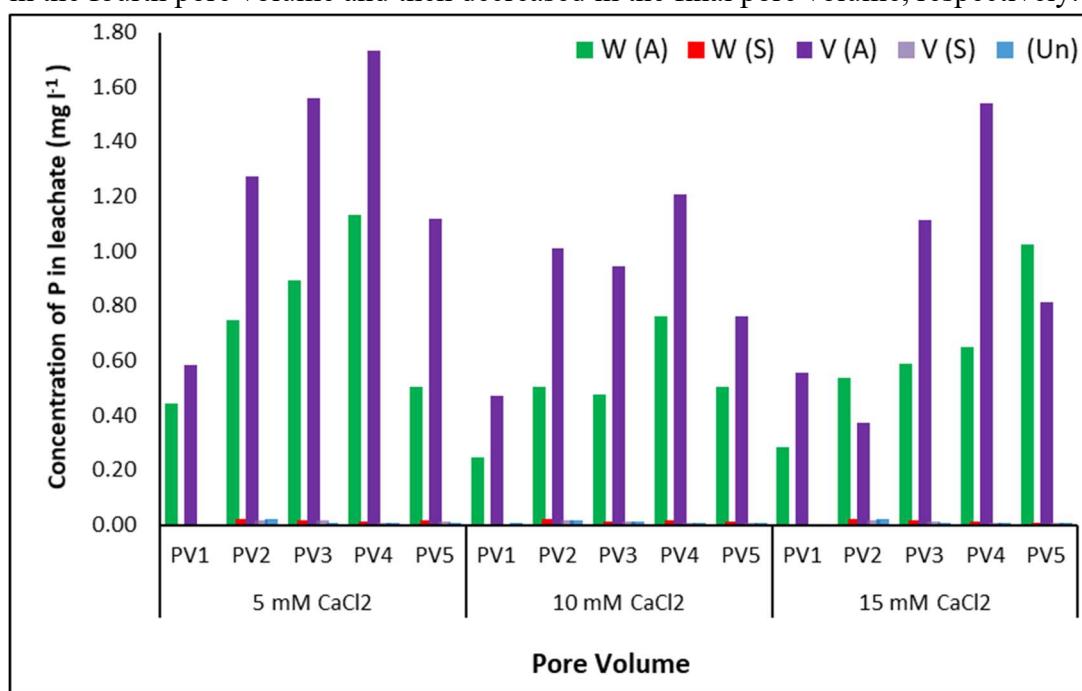


Figure 2. Phosphorus in leachates of the Derbun soil leached with different CaCl_2 solutions.
(W (A): wheat amended with manure; W (S): wheat amended with tri-superphosphate;
V (A): vegetables amended with manure; V (S): vegetables amended with tri-superphosphate).

The P concentration did not show a consistent pattern when comparing two treatments in 10 and 15 mM solutions. The lowest P concentrations were about 0.24 and 0.47 mg l⁻¹ at the initial pore volume, while the highest values were about 0.76 and 1.20 mg l⁻¹ at the fourth pore volume for (W) and (V). They then decreased at the final pore volume, respectively. The lowest P values were 0.28 and 0.37 mg l⁻¹ and the highest were 1.02 and 1.54 mg l⁻¹ for (W) and (V) cultivated soils under a 15 mM CaCl_2 solution, respectively. The low release of phosphorous in the initial pore volumes may be due to the P sorption with clay and manure, as well as its fixation on the surface of CaCO_3 itself until it reaches the saturated state. The released phosphorous in soils cultivated with vegetables was more than that in soils cultivated with wheat. This may be due to the seasonal and continuous fertilization of the soils cultivated with vegetables as compared to those cultivated with wheat. Similar results were obtained by Mozaffari and Sims (1994) and Zhang et al. (1995), who stated that long-term and continuous fertilization of medium to coarse-textured soils leads to increased P mobility downward. Grobbelaar and house (1995) stated that in surface water, 0.01 to 0.05 mg l⁻¹ total solution P may result in considerable phytoplankton production. The maximum contamination limit of P for the surface water quality standard is 0.05 mg l⁻¹ according to the USEPA (1986) surface water quality guidelines. The P concentrations in the leachates of the two locations in this study were greater than the recommended limit mentioned above. Similar to Batil location in the Derabin soil, P leaching increased with an increasing number of pore volumes and was more with (V) cultivated soil. It also increased in the soil that initially had more P (it was 0.79 and 1.19 mg l⁻¹ for (W) and (V) respectively). However, all values of P concentrations in the leachates of Derabin soil were higher (nearly twice) than those of Batil soil. This was probably due to the same reasons mentioned earlier (Table 1). These results are in line with Liu et al. (2012) who reported that the differences in soil properties were the main reasons behind the different behaviours of P leaching from the two soils and the response to the application of P. The results were also in agreement with Liu et al. (2012) who reported that loamy sand had a higher amount of P leachate than clay loam.

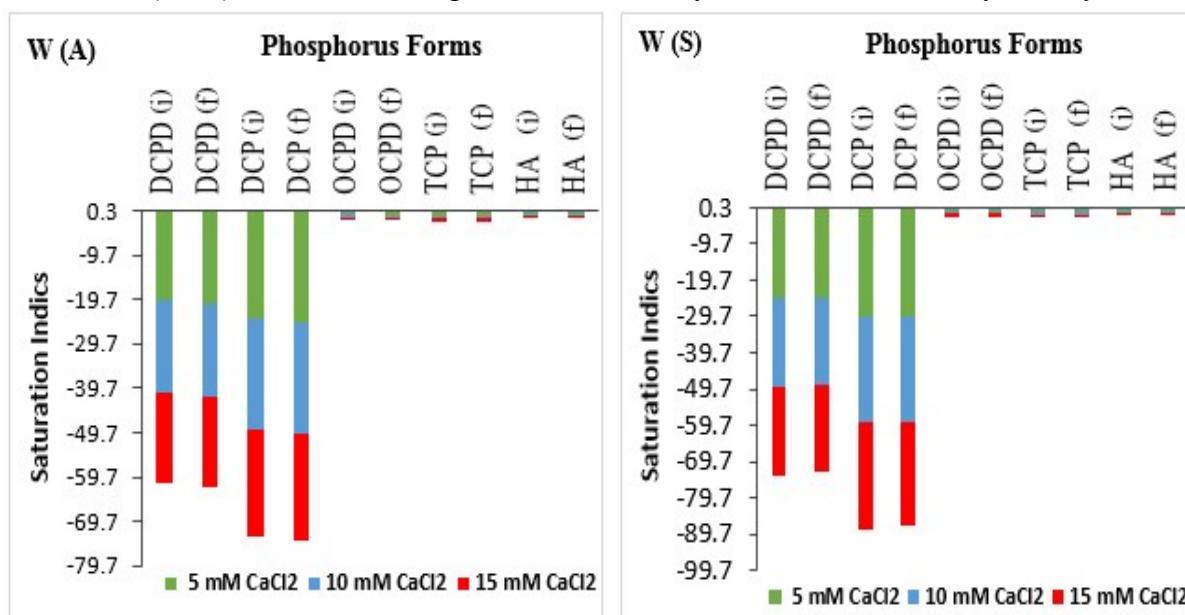
Saturation indices (SI)

The results in Figures 3, 4 show the values of the saturation index (SI). The benefit of this analysis lies in the fact that it identifies the ability of the liquid phase to dissolve or precipitate minerals using the Visual MINTEQ 2.30 program (Gustafsson 2005). By applying this program, the value of the saturation index can be obtained. If the SI values are positive, this indicates that the equilibrium solution with a solid phase is oversaturated for the mineral described by this value, indicating the possibility of precipitation; if the value is negative, this indicates that the equilibrium solution with a solid phase is undersaturated and the mineral is dissolving. If the SI value is zero, it means that an equilibrium state between the solution and solid phases has occurred. It was clear

from the results of Figure 3 that all the leachates of all the CaCl_2 solutions for different soils were negative. This means a state of undersaturation with respect to dicalcium phosphate dihydrate (DCPD), dicalcium phosphate (DCP), octacalcium phosphate (OCP), tricalcium phosphate (TCP), and hydroxyapatite (HA) throughout the leaching process. The dissolution of calcium phosphate phases is arranged as follows:

$$\text{HA} < \text{OCP} < \text{TCP} < \text{DCPD} < \text{DCP}$$

Solubility of P in soil rich in CaCO_3 can be controlled by solid phase dicalcium phosphate or P sorption on CaCO_3 , with the creation of the surface complexes of Ca-P (Afif et al. 1993). Overall, the comparison between the five saturation indices in Figures 3 and 4 showed a quite similar trend for all the different treatments and CaCl_2 solutions. However, on comparing the SI values, the soil treated with superphosphate and the uncultivated soil gave lower values than the soil cultivated with manure. These results were in line with the results in Figures 1 and 2. Adler and Sikora (2003) observed that organic fertilizers may increase the solubility of P by



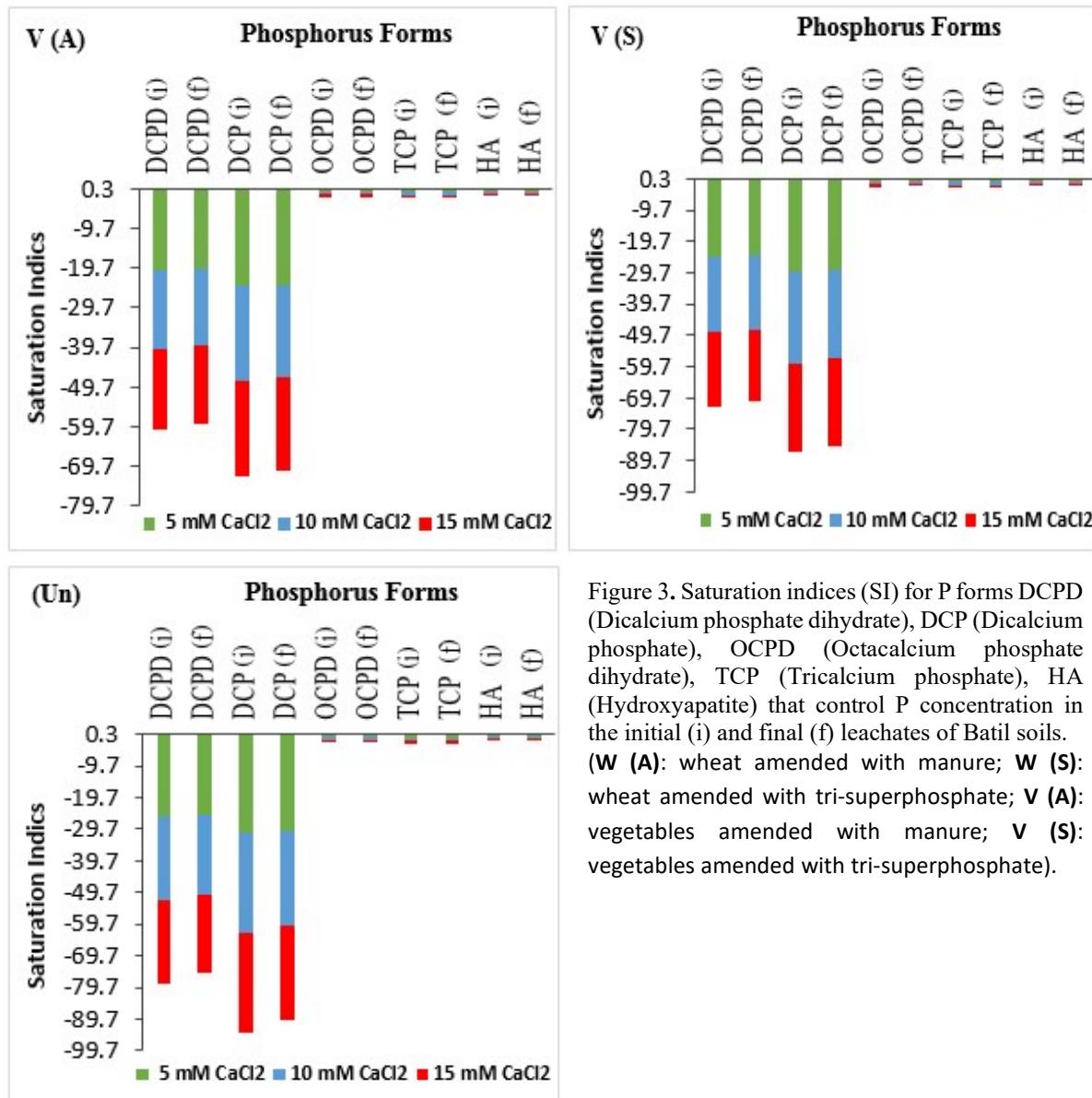
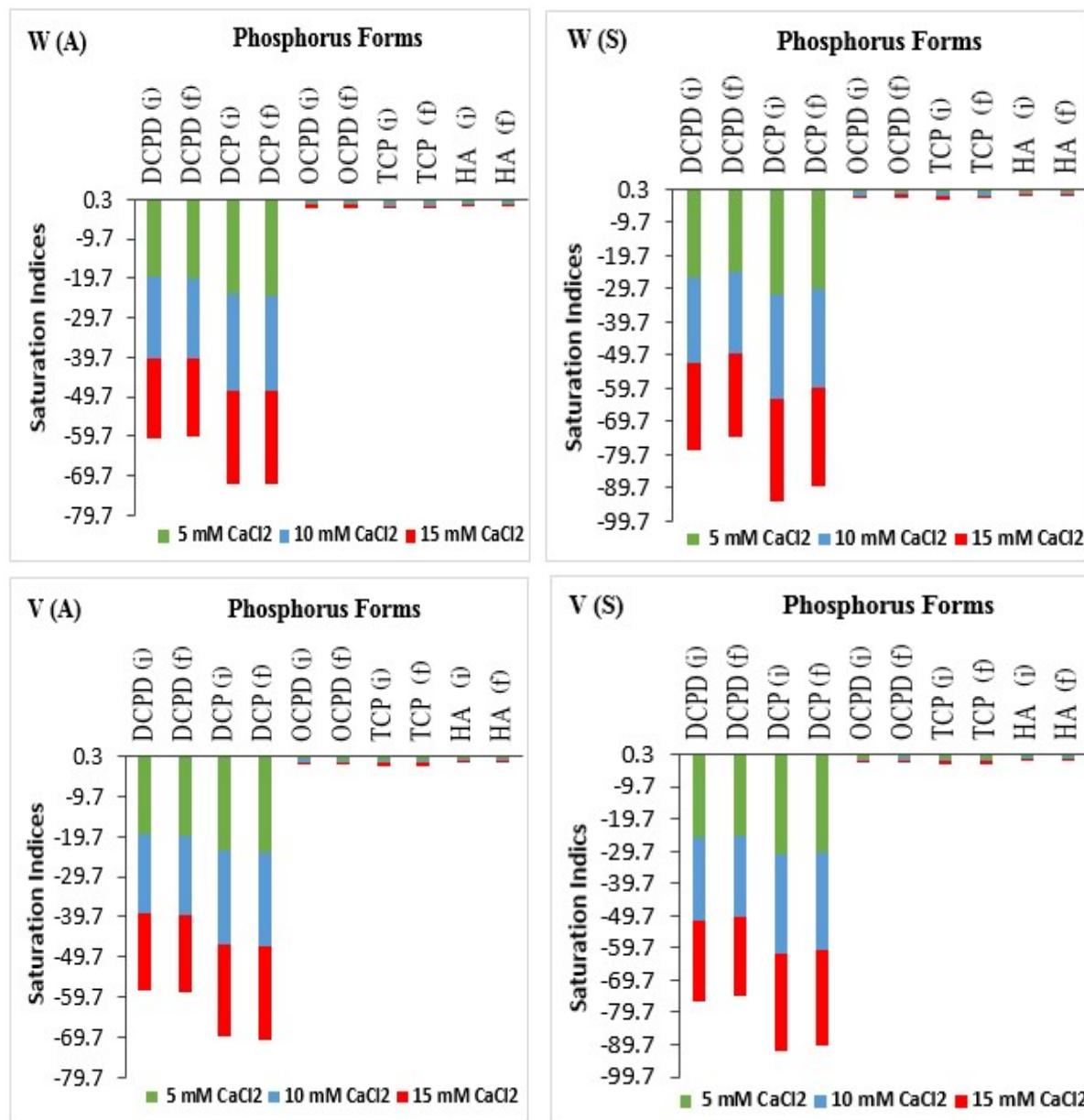


Figure 3. Saturation indices (SI) for P forms DCPD (Dicalcium phosphate dihydrate), DCP (Dicalcium phosphate), OCPD (Octacalcium phosphate dihydrate), TCP (Tricalcium phosphate), HA (Hydroxyapatite) that control P concentration in the initial (i) and final (f) leachates of Batil soils.
(W (A)): wheat amended with manure; **(W (S))**: wheat amended with tri-superphosphate; **(V (A))**: vegetables amended with manure; **(V (S))**: vegetables amended with tri-superphosphate).



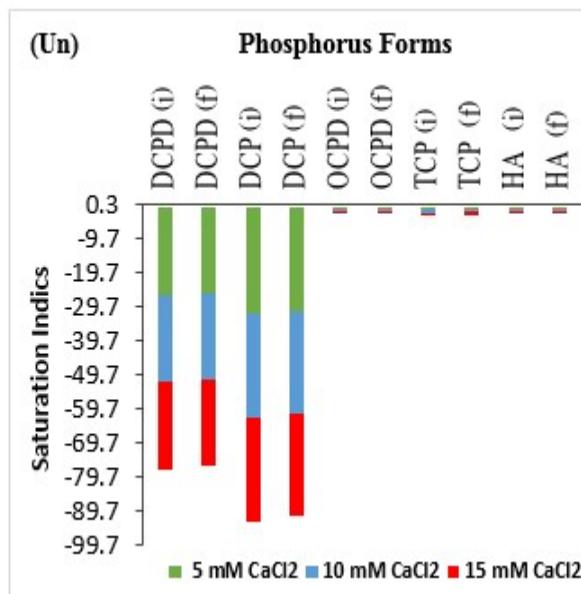


Figure 4. Saturation indices (SI) for P forms DCPD (Dicalcium phosphate dihydrate), DCP (Dicalcium phosphate), OCPD (Octacalcium phosphate dihydrate), TCP (Tricalcium phosphate), HA (Hydroxyapatite) that control P concentration in the initial (i) and final (f) leachates of Derbun soils.

(W (A)): wheat amended with manure; W (S): wheat amended with tri-superphosphate; V (A): vegetables amended with manure; V (S): vegetables amended with tri-superphosphate).

producing organic acids or humic compounds that compete with P sorption sites; thus increasing P concentrations in the soil solution.

Conclusion

The current study focuses on investigating P availability and leaching under different land uses when the soil is treated with mineral and manure fertilizers and leached with solutions of different Ca concentration. The results reveal that P leaching was affected by both land use and the type of fertilizer. Maximum leaching occurred in the soil cultivated with vegetables and amended with cattle manure. The values of P in the leachates of Derabin soil were nearly twice those of Batil soil. Leaching with different Ca solutions showed that P concentration increased with increasing pore volumes. Using more than 5 mM CaCl₂ concentration enhanced P retention in Ca-bond fraction and reduced the P leaching. The concentrations of P in the leachates of the two soils in this study were greater than the recommended limit (0.05 mg l^{-1}) mentioned by USEPA (1986). The dissolution of calcium phosphate phases followed the order of HA < OCP < TCP < DCPD < DCP.

It may be recommended to minimize the use of inorganic fertilizers and attention should be paid to the long history manure application to reduce build up and leaching of P to protect water quality. Finally, attention also should be paid to the Ca-rich irrigation water.

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