

Sorption of phosphorus in calcareous paddy soils of Iran: effects of soil/solution ratio and pH

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Abstract The sorption of phosphorus (P) was measured in calcareous paddy soils of Iran at room temperature as a function of pH and soil/solution ratio using batch experiments. Seven soils of different properties and five soil/solution ratios, 1:10, 1:25, 1:50, 1:100, 1:150, and eight pHs, 2, 3, 4, 5, 6, 7, 8, 9 were employed for this study. The pH value of these seven soil samples was adjusted to cover a range of 2–9 by addition of 1 M HCl or 1 M NaOH. The sorption of P was affected by the soil solution pH; at low pH the sorption of P was high. The soil/solution ratio was found to have significant effect on the sorption of P. A larger soil/solution ratio, i.e., more soil for a constant mass of solution, resulted in a smaller maximum P sorption value. This result also held true for Freundlich distribution coefficient at most studied pHs. There were significant correlations between P sorption and Freundlich distribution coefficient and pH and soil/solution ratio. The equations obtained can be used to predict the solubility of P and Freundlich distribution coefficient as a function of pH and soil/solution ratio in calcareous paddy soils.

Keywords Paddy soils · Calcareous soils · Phosphorus sorption · pH · Soil/solution ratio

Introduction

Phosphorus (P) is one of the essential nutrients required for the growth of plants and animals, but long-term applications

of organic and inorganic fertilizers have resulted in increase in the P status of soil. Many soils are now considered to be a potential diffuse source of P to surface waters. The high P concentration in water affects plants and can lead to eutrophication (Zhou et al. 2005). The major P sorbents are Fe (hydr) oxides and clay minerals (Hinsinger 2001; Hiemstra and Van Riemsdijk 1999; Gustafsson 2001). In addition, P sorption is influenced by the chemistry of soil solutions, such as pH, ionic strength, concentration of organic ligands and redox potential (e.g., Sah and Mikkelsen 1986; Hinsinger 2001; Kirk 2002; Spiteri et al. 2008). Gustafsson et al. (2012), Jehangir et al. (2012) and Zhou and Zhu (2003), recently showed that the pH and soil/solution ratio can affect P sorption and indicated that the pH is one of the most important factors affecting sorption of P in soils. Aqueous P speciation changes with pH, affecting P sorption on soil surfaces. Gustafsson et al. (2012) stated that the pH value may vary with time and agricultural practices. In addition, sorption of P may be varied during irrigation/drainage and floods (Zhou and Zhu 2003).

In Iran long-term applications of P fertilizers and animal manures have resulted in accumulation of P in the soils (Jalali 2007), increasing the risk of P losses to aquatic ecosystems (Jalali 2009). There have been fewer studies on the P sorption in the paddy soils of Isfahan Province in central Iran. This is an important rice production area, covering 46,000 km² and soils are calcareous type (i.e., more than 50 % of the soils have a content of total carbonate >10 %) derived from dolomite parent materials. In recent years, the application of P fertilizers to paddy soils in Isfahan has been increasing in order to enhance the per unit area yield of rice. The increasing input of P into agricultural soils in this area has become an important environmental problem.

While many published papers have studied acidification of paddy soils (e.g., Zhang et al. 2013; Wen et al. 2013),

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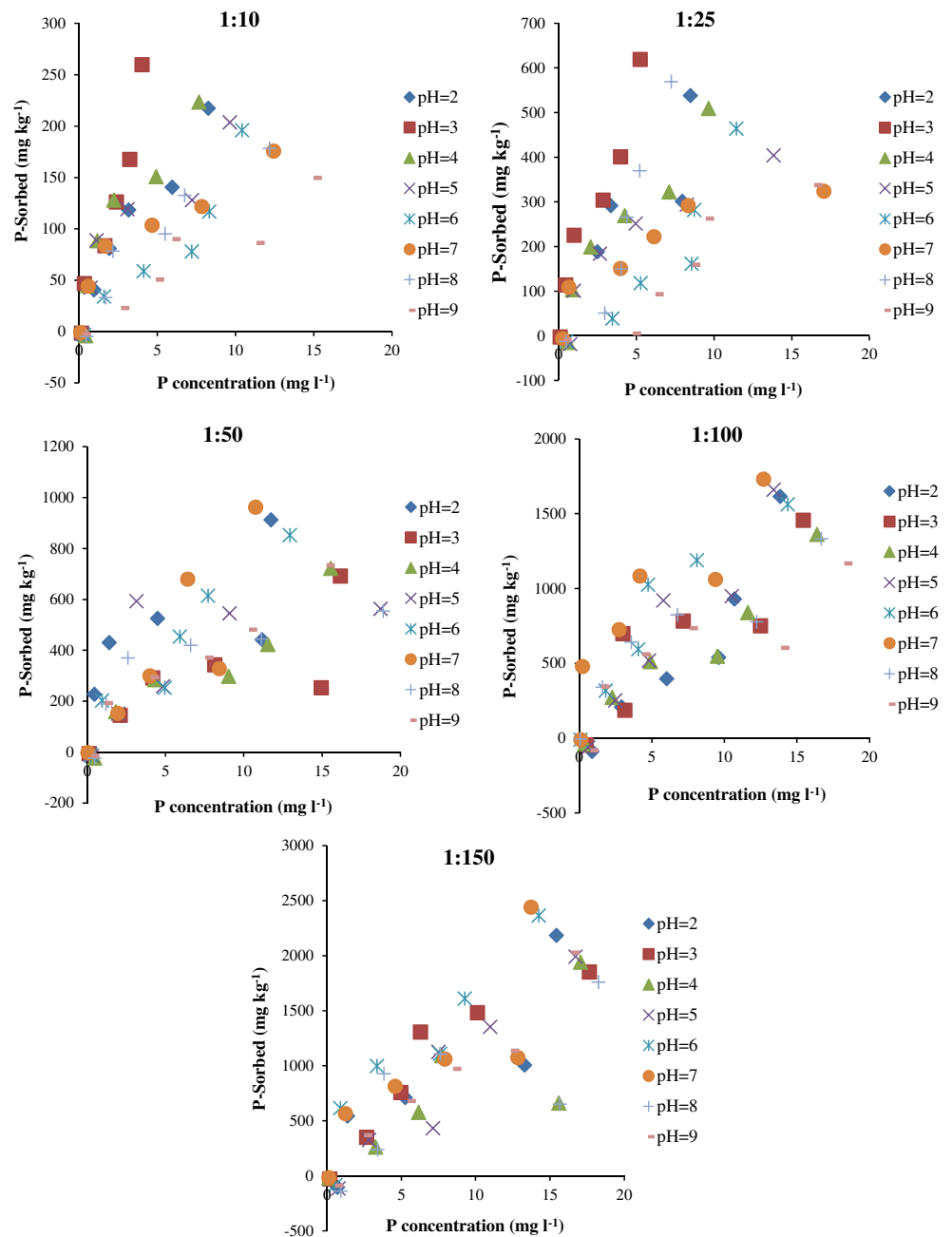
Table 1 Physical and chemical properties of studied soils

Soil no	pH (1:5) ^a	EC (dS m ⁻¹) ^a	OM (%) ^a	CaCO ₃ (%) ^a	Clay (%) ^a	Silt (%) ^a	Sand (%) ^a	CEC (cmol _c kg ⁻¹) ^a	Olsen-P (mg kg ⁻¹) ^a	Mg (1:5) (mg kg ⁻¹) ^b	Oxalate-extractable Al (mg kg ⁻¹) ^b	Oxalate-extractable Fe (mg kg ⁻¹) ^b	Oxalate-extractable P (mg kg ⁻¹) ^b	PSI (%) [†]
1	8.4	0.286	2.2	24.8	42	48	10	18.6	70.0	5.3	8.5	407.5	7.7	3.3
SD	0.01	0.01	0.21	0.04	2.83	7.07	2.69	2.04	1.41	0.82	5.37	28.75	0.06	0.01
2	7.9	0.427	1.5	24.7	36	46	18	16.3	30.0	15.5	14.6	1,223.5	7.5	1.1
SD	0.07	0.01	0.17	0.88	3.82	3.25	8.49	1.81	1.84	0.41	3.82	452.66	0.77	0.07
3	9.4	0.171	2.2	24.8	30	52	18	15.3	30.0	3.2	4.4	886.6	21.8	4.4
SD	0.54	0.04	0.10	0.04	1.84	5.66	1.41	5.22	4.38	1.24	0.42	54.35	10.93	0.54
4	8.7	0.672	2.3	24.9	37	48	15	21.3	35.0	8.8	13.0	912.5	13.6	2.5
SD	0.01	0.00	0.07	0.07	1.41	1.13	3.82	1.50	0.14	0.82	0.35	63.76	12.82	0.01
5	7.6	0.282	0.6	24.8	22	46	32	13.0	35.0	9.9	27.5	740.3	12.5	2.9
SD	0.05	0.01	0.29	0.07	2.83	7.07	8.49	1.73	0.28	0.00	3.32	20.82	1.61	0.05
6	9.3	0.202	0.6	24.6	14	24	62	11.0	30.0	6.1	6.6	658.7	16.9	4.2
SD	0.71	0.00	0.00	0.00	4.10	2.12	3.54	2.28	0.57	1.24	1.48	170.73	13.37	0.71
7	7.9	1.472	1.2	24.8	26	32	42	23.7	30.0	8.8	29.0	1,248.1	20.5	2.8
SD	0.07	0.00	0.05	0.04	8.49	5.09	4.95	3.66	2.4	0.82	4.10	67.03	21.76	0.07

† Phosphorus saturation index

^a Jalali and Hemati (2013)^b This study

Fig. 1 Phosphorus sorption as a function of pH and soil/solution ratio for the studied soils



accumulation and mobilization of trace elements (Tu et al. 2013; Hundal et al. 2013; Li et al. 2013; Zhong et al. 2012; Rogan et al. 2010; Wu and Zhang 2010), there is limited information on the behavior and availability of applied P in calcareous paddy soils especially when pH and soil/solution ratio changes occur (von Wandruszka 2006; Jalali 2007; Ige et al. 2008; Curtin and Syers 2001; McDowell and Sharpley 2001; McDowell et al. 2003; Zhou et al. 2005; Devau et al. 2011). Understanding the sorption of P in calcareous paddy soils receiving continuous different P forms is essential in order to develop appropriate P management strategies for sustainable agricultural production and environmental

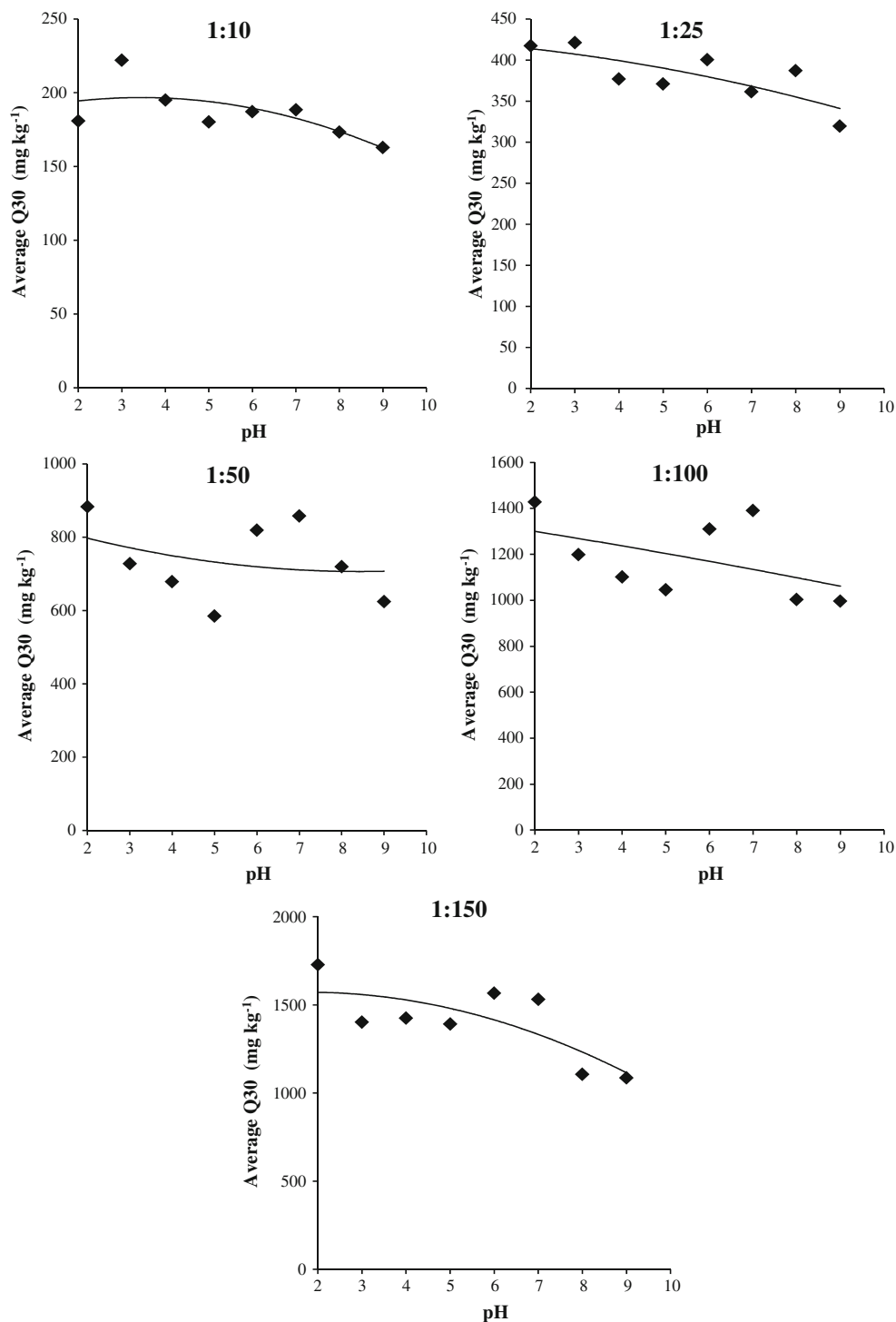
protection. At present, only a few studies have incorporated the effect of pH and soil/solution ratio on P sorption predictions. Thus, the objectives of our study were to determine P sorption under different pHs and soil/solution ratios.

Materials and methods

Soil sampling

Twenty-eight surface paddy soil samples were collected from three different localities widely distributed in the

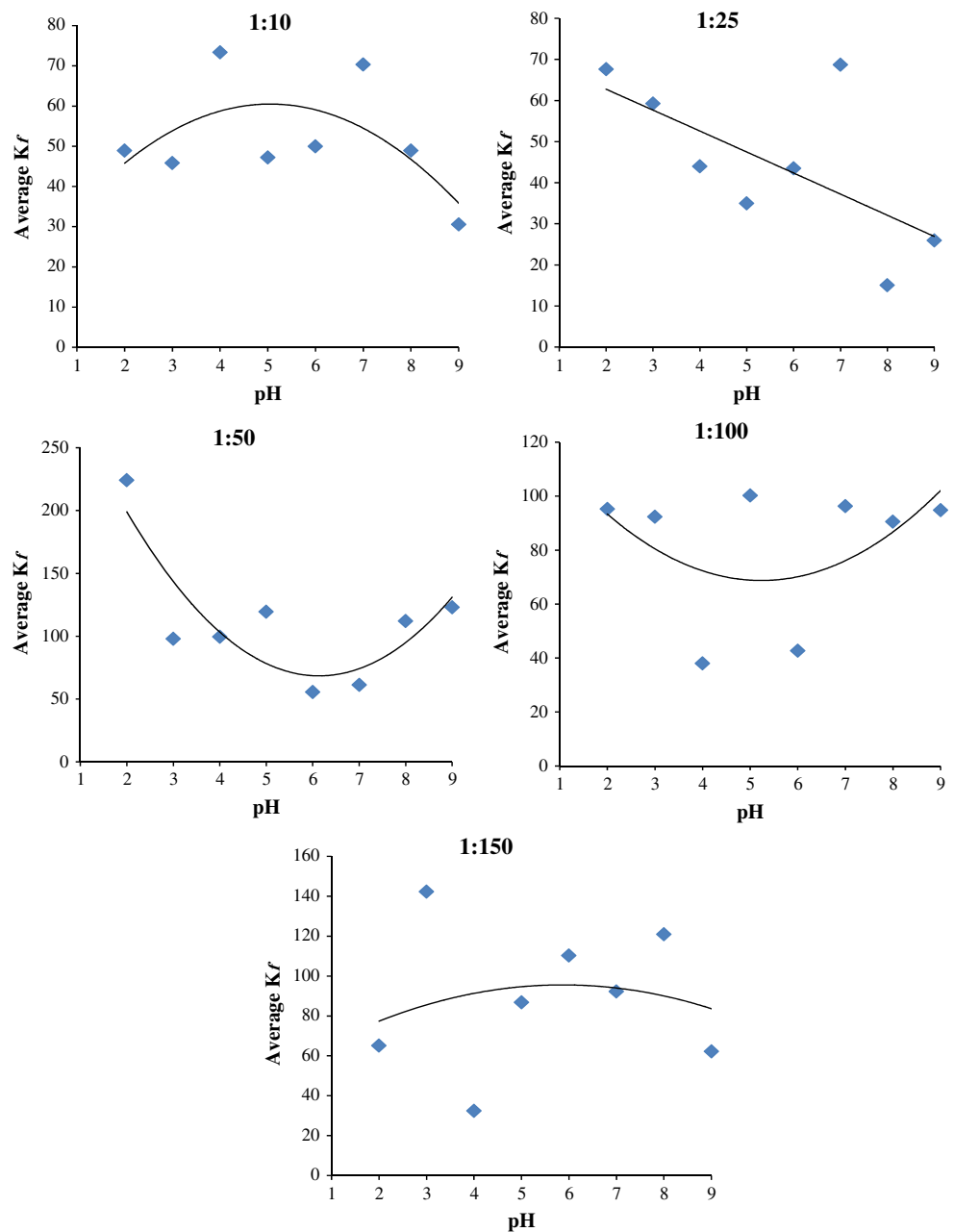
Fig. 2 The relationship between average maximum P sorption and pH under different soil/solution ratios



Isfahan Province in central Iran (between longitudes 51°20'0.5''–51°30'45.3''E and latitudes 32°21'24.0''–32°32'57.6''N). Information on the study area and soil sampling were provided by Jalali and Hemati (2013) and Jalali and Hemati Matin (2014). The major soil types are Aridisols belonging to great groups Calcicargids, Haplocambids, Haplogypsids and Haplosalids (Pirzadeh et al. 2010). From

these soil samples, seven soils with different physical and chemical properties were selected (Table 1). Oxalate-extractable P, Fe and Al were determined by 0.5 g of soil after its pH reached 5.5 after shaking with 30 ml of 1 M ammonium acetate (pH 5.5) for 1 h. In some samples, shaking with ammonium acetate was repeated several times until the solution was reached the referred pH. Then the

Fig. 3 The relationship between average Freundlich distribution coefficient and pH under different soil/solution ratios



solution was decanted and the soils were dried. After drying, the samples were shaken with 30 ml of 0.175 M ammonium oxalate (pH 3) for 2 h (in darkness). Iron and aluminum concentration were measured with atomic adsorption spectrophotometer (model Varian spectra 220), UV-vis spectrophotometer (model Varian Cary 100), respectively (Mehra and Jackson 1960). Concentration of magnesium in soil solution was measured by titration method (Bower and Wilcox 1965). A P saturation index (PSI) was calculated by dividing the molar concentration of oxalate-extractable P by the sum of the molar concentrations of oxalate-extractable Fe and Al, and multiplying by 100 to convert to percent.

Batch sorption experiments

Batch sorption experiments were performed at room temperature to determine soil sorption capacity under different pHs and soil/solution ratios. In order to obtain the soil/solution ratios of 1:10, 1:25, 1:50, 1:100 and 1:150, a 10 mM CaCl₂ solution (25 ml) containing 5, 10, 15, 20 and 30 mg l⁻¹ of P were inserted into a series of 50-ml centrifuge tubes with 2.5, 1, 0.5, 0.25 and 0.16 g of soil. After that the sorption mixtures were agitated for a pre-determined time (24 h) period using horizontal shaker. The 24-h equilibration period was selected based on batch extraction studies

by Zhou and Zhu (2003). They found that batch equilibrium was attained at 10–12 h for various extracting solutions. The influence of the aqueous phase pH on P sorption was studied by adjusting the reaction mixture to different initial pH values (2, 3, 4, 5, 6, 7, 8 and 9) and analyzed for residual P after equilibrium contact time. We acidified or alkalized the aqueous phase by adding either acid (1 M HCl) or alkali (1M NaOH). This resulted in a matrix of 7 soils \times 5 soil/solution ratios \times 8 pH values for 280 unique conditions. There were two samples for each of these conditions resulting in a total of 560 samples. After equilibration, soils were separated from solution by centrifugation followed by filtration. The residual P in the aqueous phase was analyzed colorimetrically using ascorbic acid method of Murphey and Riley (1962). Both Langmuir and Freundlich models were used. While the Freundlich model was very well fitted to the sorption data, the Langmuir model was not fitted well with all data (data not shown). The computer program Visual MINTEQ version 2.30 (Allison et al. 1991) was used to predict saturation indices (SI) and P species in the equilibrium solution concentration when 5 and 30 mg P l⁻¹ under varying pH solution and two soil/solution ratio were applied in the sorption experiments.

Results and discussion

Soil characteristics

Table 1 shows the chemical and physical properties of the soil samples. The average CaCO₃ content is about 25 %,

while the pH values varied from 7.6 to 9.3. The CEC values from 11.0 to 23.7 cmol_c kg⁻¹. Soil organic matter varied from 0.6 to 2.2 %. Based on the texture, soils can be classified as loam, silty clay, silty clay loam and sandy loam.

Influence of pH

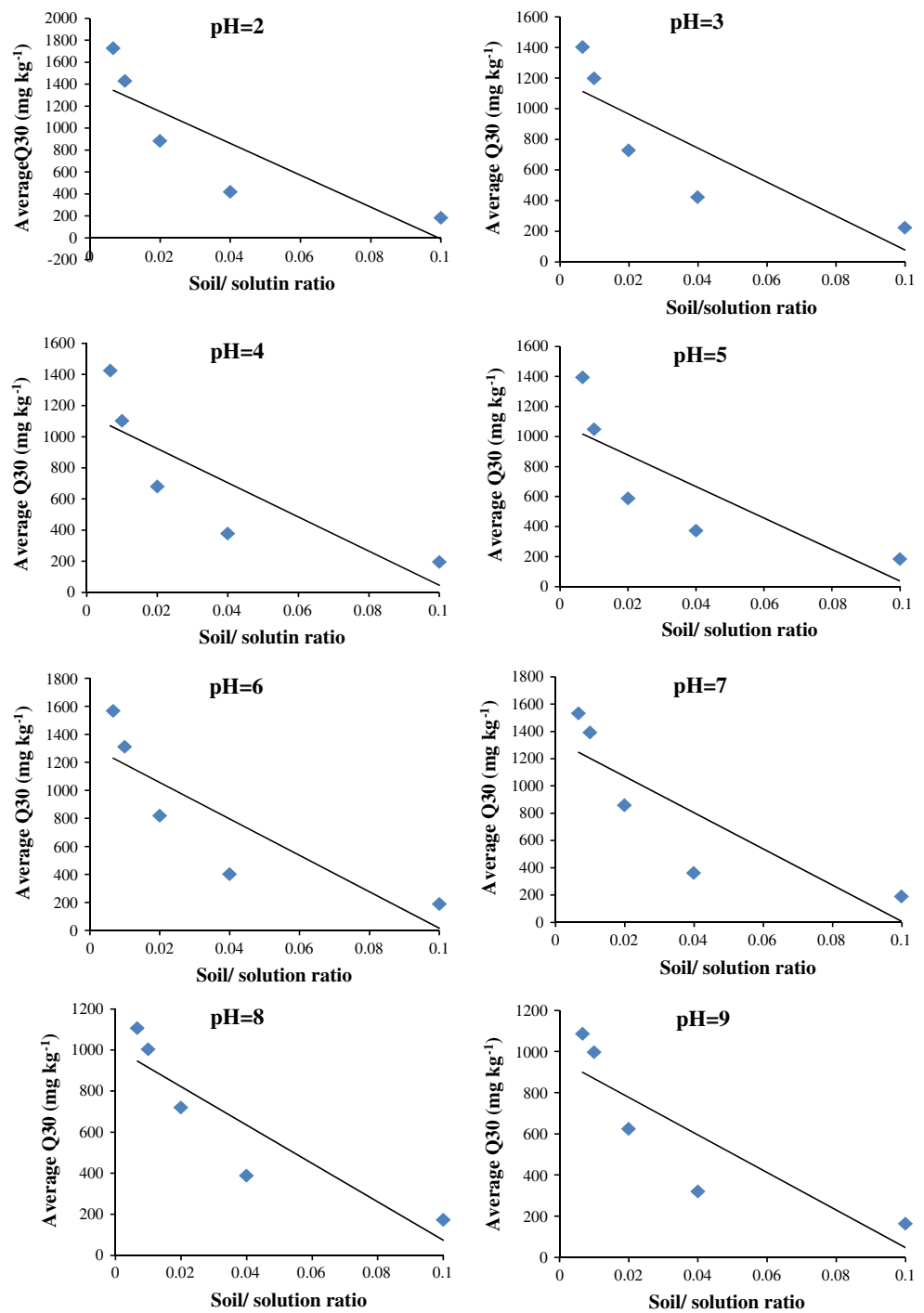
The effect of pH on the sorption of P under different soil/solution ratio is depicted in Fig. 1. In general, the extent of P sorption on the soil showed a decrease as the pH of the aqueous solution increased from 2.0 to 9.0. The sorption of P when 30 mg P l⁻¹ was applied to the soil was used to calculate maximum P sorption (Q_{30}) and the effect of pH on the mean maximum sorption (mean of seven soils) of P under different soil/solution ratio is indicated in Fig. 2. The average Q_{30} decreased with increasing pH and reached a maximum in a pH range of 6.0–7.0 and then decreased with further increases in pH under all different soil/solution ratios (Fig. 2). The effect of pH on the mean Freundlich distribution coefficient under different soil/solution ratio is indicated in Fig. 3. Table 2 presents the results of regression equations. In general K_f decreased with increasing pH and less significant correlation was found in comparison with Q_{30} . This could be attributed to the anionic sorption. Németh et al. (1998) stated that the P sorption on soil is determined by the surface charge and the protonation state of P in the bulk solution. As pH increases and surface charge becomes more negative, the sorption of anions like P decreases.

The metal Fe and Al content has been considered to be the main factor that determines sorption capacity, because of the high specific surface of the iron/aluminum (hydr)

Table 2 Parameters of linear and polynomial equations used to describe maximum P sorption and Freundlich distribution coefficient under different pHs and soil/solution ratios

pH	Equation	<i>r</i>	Equation	<i>r</i>
2	Average $Q_{30} = -14,496 \text{ soil/solution} + 1,439.4$	0.852	Average $K_f = -718.14 \text{ soil/solution} + 125.6$	0.387
3	Average $Q_{30} = -11,078 \text{ soil/solution} + 1,185.7$	0.849	Average $K_f = -793.54 \text{ soil/solution} + 115.6$	0.809
4	Average $Q_{30} = -10,972 \text{ soil/solution} + 1,142.9$	0.831	Average $K_f = 259.30 \text{ soil/solution} + 48.3$	0.351
5	Average $Q_{30} = -10,488 \text{ soil/solution} + 1,084.8$	0.811	Average $K_f = -617.14 \text{ soil/solution} + 99.5$	0.665
6	Average $Q_{30} = -12,991 \text{ soil/solution} + 1,315.6$	0.854	Average $K_f = -281.85 \text{ soil/solution} + 70.4$	0.382
7	Average $Q_{30} = -13,284 \text{ soil/solution} + 1,335.2$	0.853	Average $K_f = -195.66 \text{ soil/solution} + 84.7$	0.484
8	Average $Q_{30} = -9,336.7 \text{ soil/solution} + 1,007.5$	0.904	Average $K_f = -719.20 \text{ soil/solution} + 102.9$	0.619
9	Average $Q_{30} = -9,134.2 \text{ soil/solution} + 960.4$	0.866	Average $K_f = -673.08 \text{ soil/solution} + 91.1$	0.621
Soil/solution				
1:10	Average $Q_{30} = -1.108 \text{ pH}^2 + 7.630 \text{ pH} + 183.6$	0.710	Average $K_f = -1.582 \text{ pH}^2 + 15.98 \text{ pH} + 20.1$	0.615
1:25	Average $Q_{30} = -0.624 \text{ pH}^2 - 3.543 \text{ pH} + 423.5$	0.776	Average $K_f = -0.0062 \text{ pH}^2 - 5.052 \text{ pH} + 72.9$	0.646
1:50	Average $Q_{30} = 2.193 \text{ pH}^2 - 37.01 \text{ pH} + 862.5$	0.307	Average $K_f = 7.633 \text{ pH}^2 - 93.68 \text{ pH} + 356.0$	0.854
1:100	Average $Q_{30} = -0.502 \text{ pH}^2 - 28.56 \text{ pH} + 1,359.1$	0.480	Average $K_f = 2.353 \text{ pH}^2 - 24.63 \text{ pH} + 133.2$	0.468
1:150	Average $Q_{30} = -8.663 \text{ pH}^2 + 30.39 \text{ pH} + 1,546.2$	0.752	Average $K_f = -1.217 \text{ pH}^2 + 14.28 \text{ pH} + 53.7$	0.179

Fig. 4 The relationship between average maximum P sorption and soil/solution ratio under different pHs



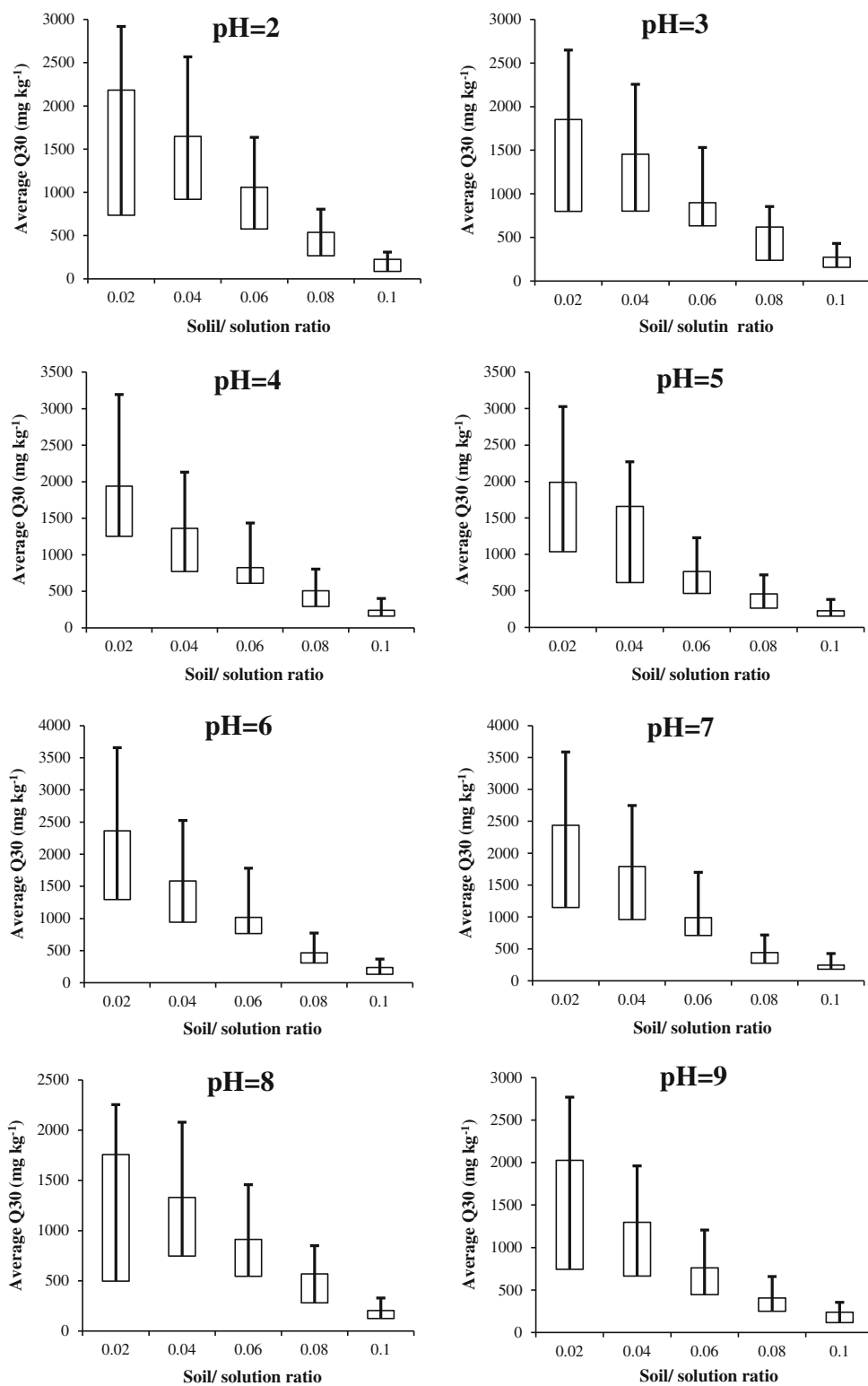
oxides. In addition, P in calcareous soils would be expected to not only adsorb on minerals, but also interact with the dissolved Ca to form phosphate minerals. Freeman and Rowell (1981) indicated that in calcareous soils, P sorption is greatly controlled by the adsorption and precipitation reactions on Ca-carbonate surfaces. However, the effect of increasing pH on soluble P is dependent on soil mineralogy as well as initial and final soil pH (Erich et al. 2002). In addition to adsorption/desorption processes, solubility and

dissolution of P is controlled by mineral equilibria, including the aging and transformation of P minerals over long periods (McDowell et al. 2003; Spiteri et al. 2007).

Soil/solution ratio effect on pH-dependent P sorption

The relationship between the mean *Q*₃₀ and the soil/solution ratio at different pHs is shown in Fig. 4. The change of the *Q*₃₀ as a function of soil/solution ratio was similar for the all

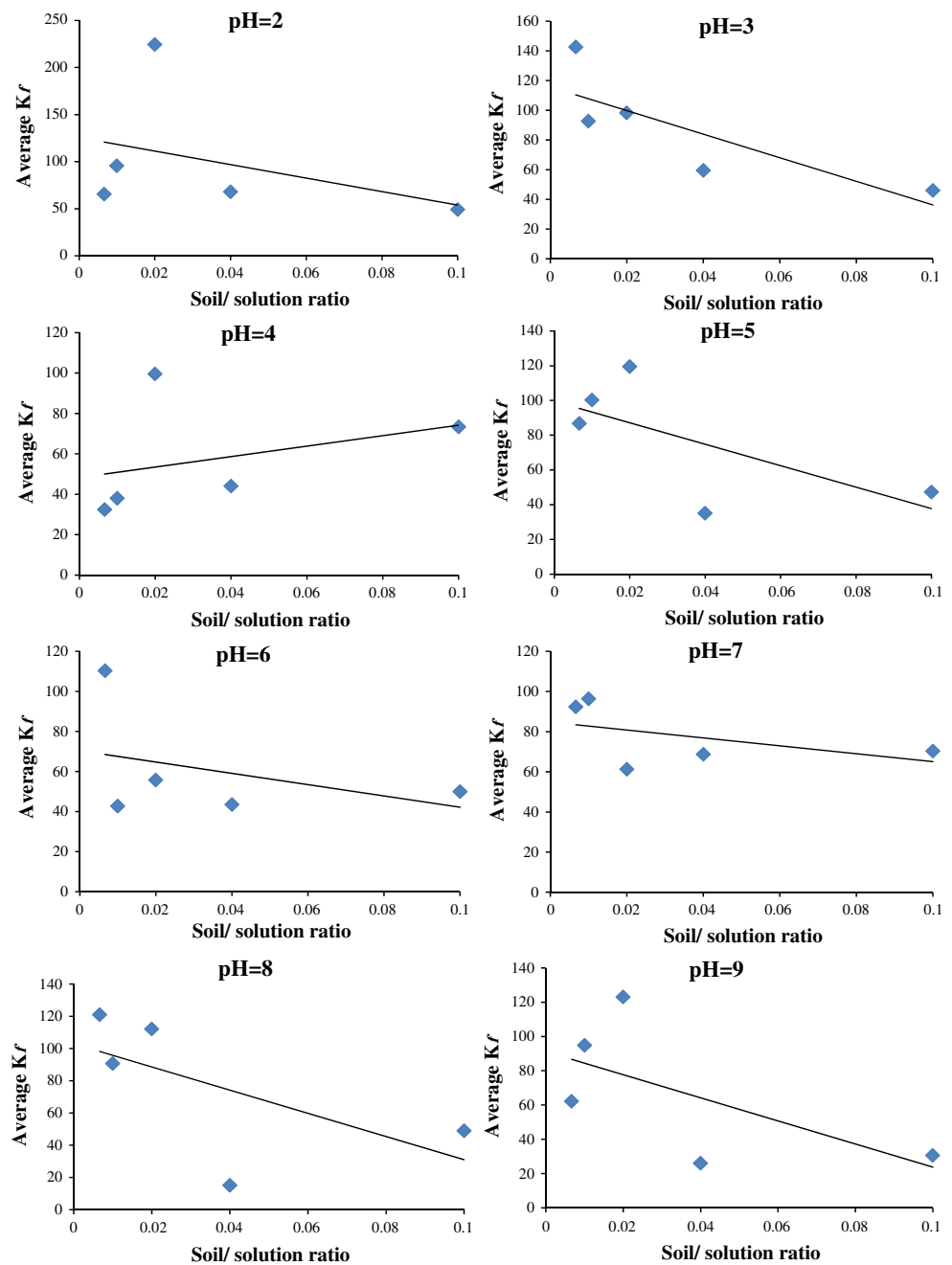
Fig. 5 Maximum phosphorus sorption range at different soil/solution ratios used in batch experiments in studied soils under different pHs. Bars the range of different values of maximum P sorption



soils and Fig. 5 indicate the maximum P sorption at different pHs and soil/solution ratios. As it can be seen at each pH, Q_{30} decreased with an increase in soil/solution ratios, which is in agreement with recent findings by Zhou and Zhu (2003) and Cucarella and Renman (2009). It has been indicated that P

sorption varies with soil/solution ratios (Hope and Syers 1976; Nair et al. 1984). The effect of soil/solution ratio on the mean Freundlich distribution coefficient under different pH is indicated in Fig. 6. In general K_f increased with decreasing soil/solution ratio in all studied pHs except at pHs 4. Several

Fig. 6 The relationship between average Freundlich distribution coefficient and soil/solution ratio under different pHs



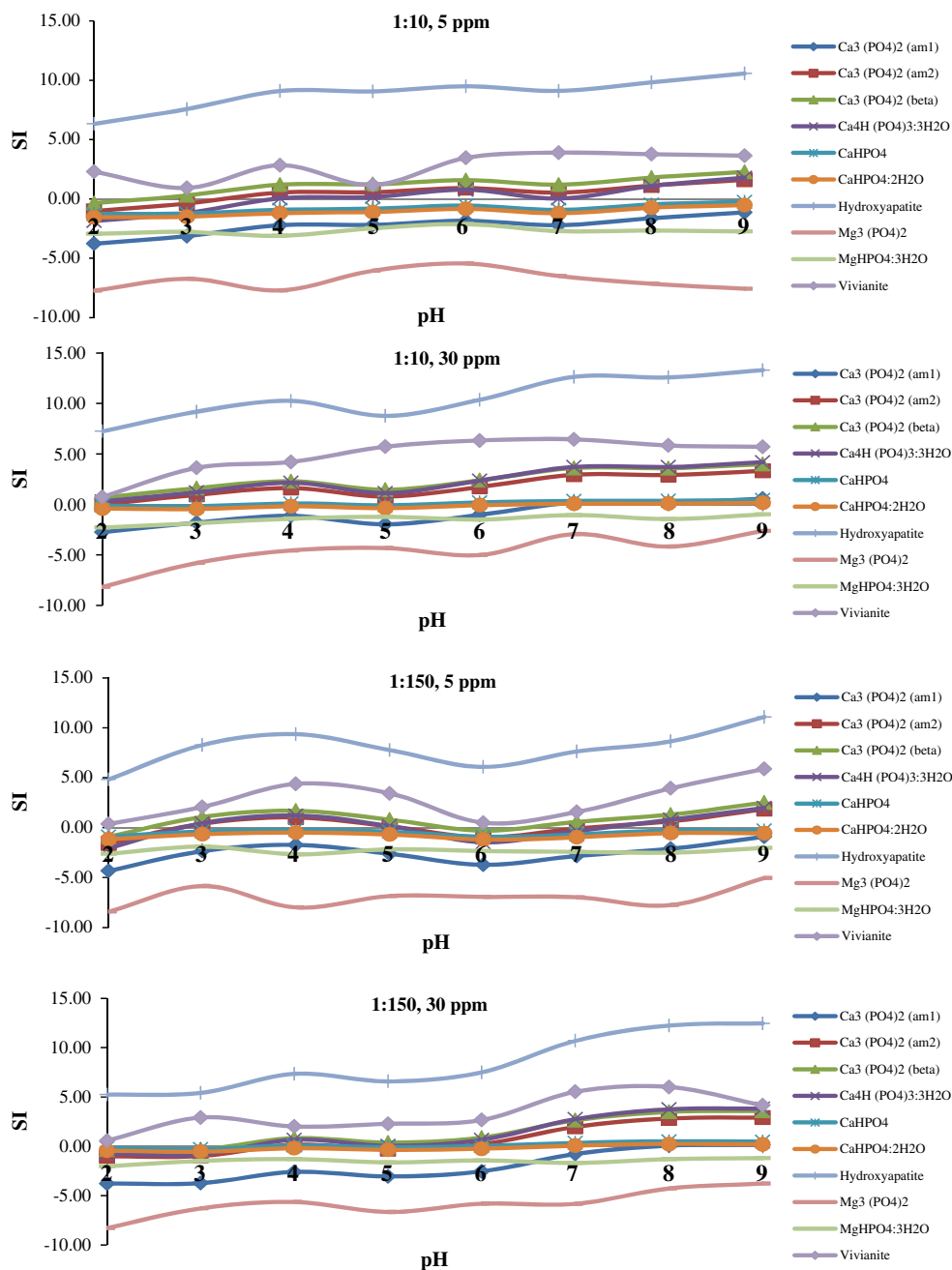
investigators have studied the relationship between K_f and soil/solution ratio, their results were similar to our observations (Puls et al. 1991; Chang et al. 2002). It was indicated when the soil sorption capacity is high in relation to the amount of sorbate present, increasing soil/solution ratio (higher soil concentration), cannot increase the amount of sorption (Harter and Naidu 2001). Correlation coefficients along with parameters of fitted equations were presented in Table 2.

Cucarella and Renman (2009) stated that a variation in the soil/solution ratio may alter either the equilibrium constant or the complex concentration by reaching a new

equilibrium. Smaller ratios may lead to higher concentrations of P sorbed to the material, that is, a higher percentage of P removal. Some authors indicated that the smaller the ratio, the longer the time needed to reach equilibrium (Søvik and Kløve 2005; Ádám et al. 2007). Cucarella and Renman (2009) stated that in the field more soil is in contact with the solution and using small soil/solution ratio may cause a big difference between batch tests and field conditions.

Zhou and Zhu (2003) concluded that agricultural irrigation/drainage and floods are disadvantageous to the sorption of P on the soil because it can result in the release

Fig. 7 Changes in saturation index of P minerals as a function of pH under different P added and soil/solution ratio in soil 1



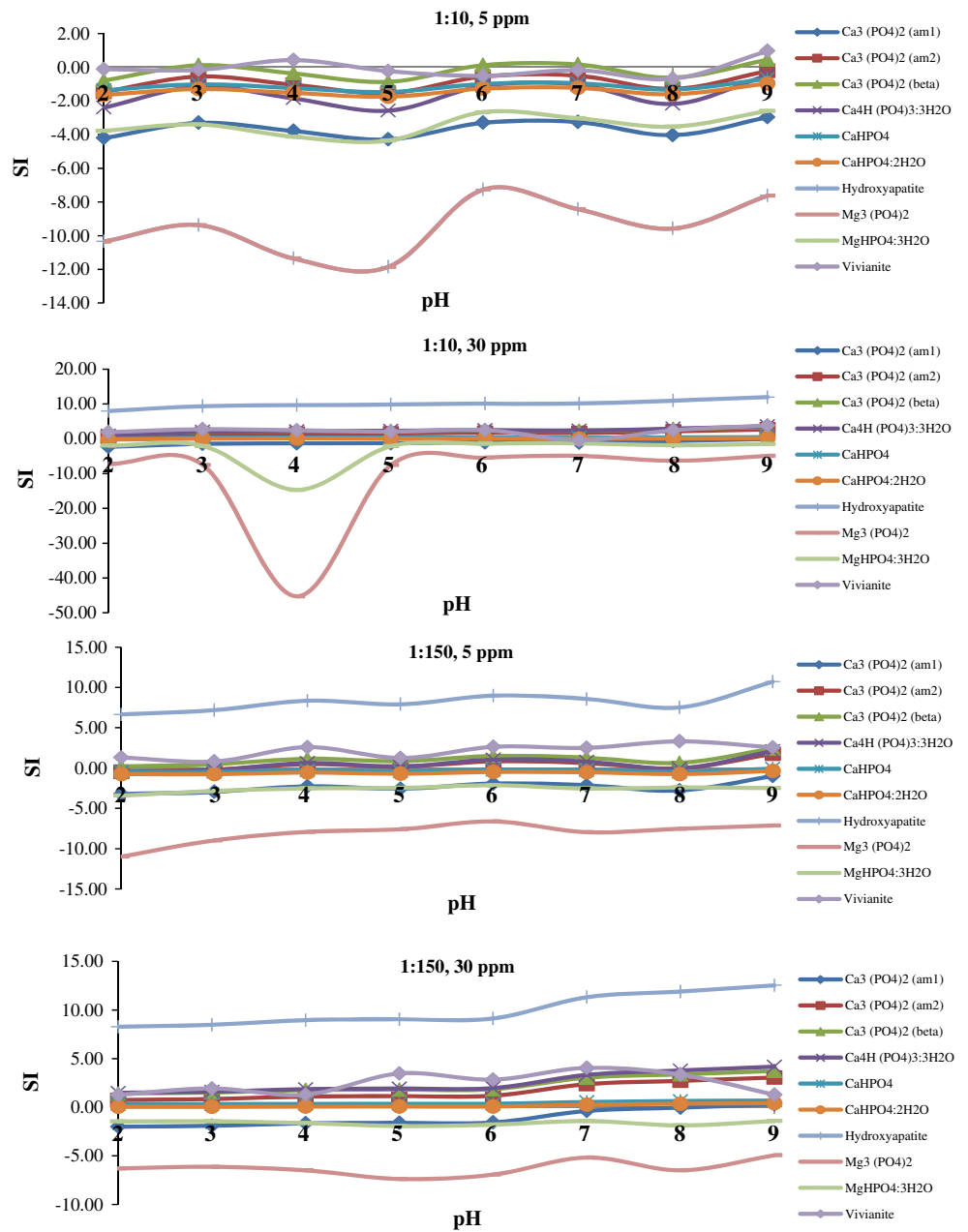
of soil P into water. Yin et al. (2002) concluded that due to the effect of soil/solution ratio, higher soil/solution ratios which are approximately close to the field conditions should be used.

Saturation indices and P speciation

Figures 7 and 8 show variation of SI with pH calculated for two soils (only soil 1 and soil 5 were chosen because these soils had maximum differences in physical and chemical properties among the studied soils) at low and

high added P and low and high soil/solution ratio. The geochemical speciation indicated that in general all solution samples in two soils were supersaturated with respect to hydroxyapatite (HA, $\text{Ca}_5(\text{PO}_4)_3\text{OH}$), octacalcium phosphate (OCP, $\text{Ca}_8(\text{HPO}_4)_2(\text{PO}_4)_6$), β -tricalcium phosphate (TCP, $\text{Ca}_3(\text{PO}_4)_2$), $\text{Ca}_3(\text{PO}_4)_2(\text{am}2)$, vivianite ($\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$) and undersaturated with respect to dicalcium phosphate dehydrate (DCPD, $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$), dicalcium phosphate (DCP, CaHPO_4), and magnesium phosphates ($\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$ and $\text{Mg}_3(\text{PO}_4)_2$). In general, SI increased with increasing pH and at high added P and

Fig. 8 Changes in saturation index of P minerals as a function of pH under different P added and soil/solution ratio in soil 5



low soil/solution ratio, more P minerals were saturated. Thus, the main mechanisms that control adsorption of P in these soils are precipitation of calcium and iron P minerals.

The contribution of different forms of P to the total P concentration in the soil solution differed between the various soil/solution ratio and soil solution pH (see supporting information figures S1 and S2). In general, at low pH most P was present in its H₂PO₄⁻ form; however, at higher pH most of the total P was present as HPO₄²⁻ and CaHPO₄ (aq). However, in soil 5 at low P added and low soil/solution ratio, most P was present as H₂PO₄⁻, while

for soil 1 CaHPO₄ (aq) and HPO₄²⁻ were the dominant species. The pH is one of the most important factors affecting the solubility of P in soils. The results indicated that aqueous P speciation changes with pH and soil/solution ratio which affect P sorption soils.

Most previous studies were conducted under conventional experimental conditions and did not consider variation in pH and soil/solution ratio. Characterization of P sorption under varying conditions in calcareous paddy soils provides information about mechanism of P retention and release into the overlying water. The results of present experiments can be used to predict soil P behavior.

Conclusion

The sorption behavior of P on seven paddy soils was investigated at varying soil/solution ratios (1:10, 1:25, 1:50, 1:100, 1:150) and different pH from 2 to 9 using a batch equilibration experiment. The results indicated that the sorption of P was influenced by the pH and soil/solution ratio. The sorption of P on calcareous paddy soils decreased with increasing pH. At low pH values (2–3), the sorption of P increased, while it was decreased with increasing pH. The P sorption was also influenced by soil/solution ratio and more P was sorbed as more water was available. Release and retention of P in paddy soil may affect the water quality and nutrient status of soils. Chemical equilibria with P-containing minerals can control the dissolved P concentration in soil solution and groundwater.

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