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RESEARCH ARTICLE

Strategic differences in phosphorus stabilization by alum and dolomite amendments in calcareous and red soils

Bingqian Fan[1] · Jue Wang[1] · Owen Fenton[2] · Karen Daly[2] · Golnaz Ezzati[2] · Qing Chen[1,3]

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Abstract

Surplus phosphorus (P) above agronomic requirements can negatively affect the water status of connected surface and subsurface water bodies. The in situ stabilization of soil P through soil amendment has been recognized as an efficient way to reduce this environmental pressure. However, the mechanism of how P is stabilized during this process and how plant available P is affected are unknown. This can be achieved by sequential chemical extraction and synchrotron-based X-ray absorption near-edge structure (XANES) spectroscopy investigations. Therefore, in the present study, P-enriched calcareous and red soils were amended with alum, dolomite, and a 1:1 mixture of alum and dolomite (MAD) at a 20 g/kg soil rate, and soil properties and P fractions were measured after a 45-day period. Results showed that alum amendment significantly decreased $CaCl_2$-P and Olsen-P contents in calcareous and red soils when compared with dolomite. However, dolomite incorporation maintained relatively high P availability and even increased $CaCl_2$-P and Olsen-P contents by 1.32% and 40.5% in red soil, respectively, compared to control. Amendment with MAD was not as effectively as the alum in P stabilization. Sequential inorganic P extraction indicated that alum dominantly contributed labile P transformed to Al-P in both soils. P K-edge XANES spectroscopy measurements further explained that alum adsorbed phosphate in calcareous soil and precipitated phosphate as $AlPO_4$ in red soil. Results of P fractionation and Mehlich-3-extracted Ca showed that dolomite mainly adsorbed loosely bound P in calcareous soil and red soil. However, dolomite incorporation in red soil led to Al-P and Fe-P release. The P sorption isotherms showed that dolomite and alum increased soil P sorption maxima and decreased the degree of P saturation (DPS) in both soils, while dolomite declined the Langmuir bonding energy in red soil. Differences in P stabilization by alum and dolomite addition across soil types were closely related to their characteristics, and soil properties changed, especially soil pH.

Keywords Alum · Dolomite · P stabilization · P sorption · Calcareous soil · Red soil

Introduction

Phosphorus (P) underpins global food systems by ensuring soil fertility, maximizing crop yields, and supporting farmer livelihoods and ultimately food security. However, global legacy P accumulation in soil has increased in intensive farming systems (Yan et al. 2013; Sharpley et al. 2013, 2015; Powers et al. 2016). Such legacy stores of P are important untapped nutrient resources but also are of environmental concerns if not managed appropriately in the long-term (Carpenter 2005). Specifically in China, excessive fertilization, especially manure applications far in excess of crop requirements, is common in vegetable and orchard enterprises (Liang et al. 2015; Yang et al. 2016; Li et al. 2017), with corresponding P leaching and runoff being reported in some P-rich or coarse texture soils (Zhang et al. 2004; Yan et al. 2013).

Highlights 1. In P-enriched soils, alum was more efficient in stabilizing P than dolomite.
2. Amending dolomite increased P availability in red soil.
3. MAD amendment modulated soil $CaCl_2$-P and Olsen-P availability.
4. Soil solution P was precipitated by alum in red soil.

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✉ Qing Chen
qchen@cau.edu.cn

[1] Beijing Key Laboratory of Farmyard Soil Pollution Prevention-Control and Remediation, College of Resources and Environmental Sciences, China Agricultural University, Beijing 100193, China

[2] Teagasc, Environmental Research Centre, Johnstown Castle, Wexford, Ireland

[3] State Key Laboratory of Nutrition Resources Integrated Utilization, Linyi 276700, Shandong, China

P undergoes several geo-chemical processes in soil such as solubilization, complexation, adsorption, and precipitation that determine its form and content (Kruse et al. 2015). It is no doubt that P is fixed by high-energy sorption surfaces such as oxides and hydroxides of Fe and Al through the formation of insoluble Fe and Al phosphates by ligand exchange and precipitation reactions in acidic red soils (Haynes 1982). Whereas the high base status and pH of calcareous soils render P sparingly soluble due to the formation of metal complexes such as Ca-P and Mg-P (Westermann 1992). The soil P fractionation method proposed by Chang and Jackson (1957) is often used to characterize soil P forms and contents in which P fraction was extracted as loosely bound P, Al-P, Fe-P, occluded-P (O-P), and Ca-P. Jiang and Gu (1989) further separated the calcium phosphates into three types, including dicalcium phosphate, octacalcium phosphate, and apatite in the calcareous soil. The phosphate ion from fertilizer is added to a calcareous soil, the process of monocalcium phosphate converting to dicalcium phosphate, and then octocalcium phosphate occurred (Olsen and Flowerday 1971). However, the total amount of these three types of calcium phosphates approximately means the total amount of loosely bound P and calcium phosphate in the fractionation scheme of Chang and Jackson (Jiang and Gu 1989).

Prevention of P losses along surface and subsurface pathways in P-enriched soils with high soil test P (STP) and degree of P saturation (DPS) (Sattaria et al. 2012; Withers et al. 2015; Fischer et al. 2017) could be achieved through decreasing P loads and safeguarding P stores (Withers et al. 2017). In practice, many agricultural soils had already accumulated very high STP levels, and the eutrophication risk existed even partially omitting P inputs (Withers et al. 2015). Furthermore, the phyto-depletion of soil P from excessive to optimum agronomic P levels can take long period in different soils due to biogeochemical lag time and nutrient cycling rate (Schulte et al. 2010; Wall et al. 2013). While the timeline of P scarcity is contested, there is consensus that more efficient use and P recycling is required. Other potential strategies to prevent P loss to water typically occur by the interception of the pathway utilizing P sorbing materials (PSM) (e.g., Penn and Bowen 2018), as an organic fertilizer amendment (Huang et al. 2016) or as direct soil amendment (Novak and Watts 2005; Brennan et al. 2011; Fenton et al. 2012). Many studies have demonstrated that chemical amendments, i.e., clay minerals, iron/aluminum minerals, or calcium/magnesium minerals, were very efficient in reducing P loss across different soils (Xu et al. 2006; Miyittah et al. 2011; Gérard 2016). Among these chemical amendments, alum and dolomite were commonly considered to reduce P runoff or leaching due to the characteristics of efficient and easy to get (Brennan et al. 2011; Eslamian et al. 2018). However, the amending efficacy and related mechanisms may differ across soil and amendment types.

Alum, as an acidic material, added in P-enriched soil may transform soil labile P to Al-P of which pK_{sp} is higher to decrease the possibility of P loss. But, alum addition may also decrease the soil pH and then dissolve part of Ca-P, especially in calcareous soil. On the contrary, dolomite may increase the P adsorption or precipitation to reduce P loss, whereas it may increase the soil pH and break the ligand adsorption of Fe, Al. Thus, it is paramount to identify the P forms produced in the amended soil by using sequential chemical extraction as the effects on P availability and mobility closely related to the P forms. And, the technique of P K-edge X-ray absorption near-edge structure (XANES) spectroscopy can further reveal P forms to distinguish phosphate compounds through in situ identification. Besides, an ideal amendment should effectively remove labile P under prevailing soil conditions, including a fluctuating soil pH. Hence, there is a need to examine how mineral amendments perform across different soil chemistries.

Therefore, the objectives of the current soil amendment study were to investigate (1) the effectiveness of alum, dolomite, and dual alum and dolomite on reducing the solubility of P in calcareous and red soils with different physical and chemical characteristics; and (2) the mechanisms of alum and dolomite on soil P stabilization in view of agronomic and environmental aspects.

Materials and methods

Soil and amendments preparation

Two soils with Mehlich 3-extracted P that exceed 400 mg/kg were selected; both of which were representatives of the surface 0.2 m of the profile in the protected field but differed in terms of parameters as set out in Table 1. The calcareous soil that had a silty loam texture collected from Fangshan in Beijing has been used to plant tomato and leafy vegetables for 5 years before soil collecting in April 2017, whereas the acidic red soil used to plant tomato since 2008 that had a loamy texture was collected from Zhanjiang in Guangdong Province, China, in April 2017. Approximately 100 kg of each soil was transported to the laboratory. Coarse rocks and plant matter were removed by hand. Next, the soils were air-dried and passed through a 2-mm sieve. Each sieved soil was further homogenized via mixing using a shovel on a plastic tarpaulin.

The soil amendments used in the present study were (1) commercial grade alum (≥ 99.8% $KAl(SO_4)_2$ in dry basis) (pH = 2.83; *v*/*w* = 2.5/1; (2) commercial grade dolomite ($CaMg(CO_3)_2$) (pH = 9.88; *v*/*w* = 2.5/1); and (3) a mixture of alum and dolomite (1:1, *w*/*w*). Before incorporated into soil treatments, the amendments were dried and sieved through the nylon mesh with 100 mesh number (0.15 mm). The Brunauer–Emmett–Teller (BET) surface area of the sieved alum and dolomite determined by the nitrogen adsorption–

Table 1 Physical and chemical properties of the studied soil in the experiment

Parameters	Unit	Calcareous soil	Red soil
Soil texture		Silt loam	Loam
Sand content	%	27.1	34.0
Silt content	%	57.8	40.0
Clay content	%	15.1	26.0
Bulk density	g/cm	1.32	1.41
Organic matter	g/kg	11.4	23.9
pH ($w/v = 1{:}2.5$)		7.56	6.04
EC ($w/v = 1{:}5$)	μs/cm	207	130
Olsen-P	mg/kg	223	87.6
Mehlich 3 extractable P	mg/kg	414	472
Mehlich 3 extractable Al	mg/kg	240	482
Mehlich 3 extractable Fe	mg/kg	119	530
Mehlich 3 extractable Ca	mg/kg	5969	1852
Mehlich 3 extractable Mg	mg/kg	867	96

desorption measurements carried out at 77 K on a Quantachrome instruments were 0.463 and 2.833 m^2/g, respectively.

Experimental design

The 45-day batch experiment comprising eight treatments (four amendment treatments across two soil types) as designed as follows: (1) control, no any amendment; (2) alum; (3) dolomite; and (4) dual amendment of alum and dolomite (MAD). Each treatment consisted of three replicates.

The amendments were mixed well with 200-g air-dried soils passed through a 2-mm sieve at a rate of 20 g/kg, respectively. The soil treatments were placed into individual 0.7-L cylindrical containers (80-mm diameter × 88-mm height). Then, deionized water was added to each treatment to achieve approximately 70% of field water capacity. The containers were covered with parafilm and perforated for air circulation. Next, the containers were placed in a temperature-controlled incubating chamber at 20–25 °C and 70% relative humidity. During the incubation, the containers were weighted and soil water was compensated every 2–3 days to maintain ~ 70% of water field capacity. At the end of the batch experiment, soil samples were collected, sieved, and analyzed.

Soil available P

The $CaCl_2$-P and Olsen-P extracts from soils were determined using 0.01 M $CaCl_2$ (soil/solution ratio of 1:5) and 0.5 M $NaHCO_3$ (soil/solution ratio of 1:20), respectively (Olsen et al. 1954; Schofield 1955). P concentrations in extracts were measured colorimetrically using the ascorbic acid method (Murphy and Riley 1962).

Soil samples were extracted with Mehlich 3-extracting solution (0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.015 M NH_4F + 0.13 M HNO_3 + 0.001 M EDTA) by shaking for 5 min at a 1:10 soil to solution ratio to determine P (M3-P), Ca (M3-Ca), Mg (M3-Mg), Fe (M3-Fe), and Al (M3-Al) (Mehlich 1984). The exchangeable Al, Ca, Fe, and Mg extracted from soil were determined using 1 M KCl solution (soil/solution ratio of 1:10). And, the amorphous Al extracted from soil was determined using acid ammonium oxalate solution (0.2 M ammonium oxalate, 0.2 M oxalate, pH = 3, soil/solution ratio of 1:100) (Van Reeuwijk 1995). Elements in the Mehlich 3, KCl, and acid ammonium oxalate extracts were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES, ICAP6300, USA).

Soil pH was measured in 1 M KCl suspension with soil/solution ratio of 1:2.5 using a pH meter (MP522 version 3, SANXIN, China). The organic matter (OM) was determined using the potassium dichromate oxidation method (Bao 2010).

Sequential chemical extraction and synchrotron-based K-edge XANES spectroscopy

Soil samples from all treatments were dried and sieved manually. The P sequential fractionation of the calcareous soils used the method proposed by Jiang and Gu (1989) and modified by Adhami et al. (2006). P fraction was extracted as follows: 1.25 g of soil was weighed into a 50 ml polyethylene centrifuge tube. A volume of 25 ml $NaHCO_3$ at pH 7.5 as the first extractant was added to the centrifuge tube (Table 2). After shaking for 1 h, centrifugation was used at 4000 rpm for 8 min and the supernatant was removed and filtered. This extraction procedure was repeated sequentially with the seven extractants in calcareous soil listed in Table 2. The P sequential fractionation of the red soil was based on the Kuo (1996) modified procedure of Chang and Jackson (1957). Five extractants were used for the red soil (Table 2), and P concentrations in extracts were colorimetrically measured using the ascorbic acid method (Murphy and Riley 1962).

To elucidate the exact P species in the study treatments over time, the P K-edge XANES spectroscopy was performed at Beamline 4B7A of the Beijing Synchrotron Radiation Facility, Beijing, China (Zheng et al. 2014). The storage ring operated at 2.5 GeV and at a maximum beam current of 250 mA (Yan et al. 2018). The following standards were purchased from Sigma-Aldrich: aluminum phosphate ($AlPO_4$), iron phosphate dihydrate ($FePO_4{\cdot}2H_2O$), dibasic calcium phosphate dihydrate (DCP, $CaHPO_4{\cdot}2H_2O$), and hydroxyapatite (HAP, $Ca_5(PO_4)_3OH$). Before analysis, soil samples were air-dried and sieved (0.5-mm particle size). Ground P standards and soil samples were thinly spread over a P-free and double-sided carbon tape for the XANES data collection. The soil spectra were collected in partial fluorescence yield (PFY)

Table 2 A sequential fractionation method for inorganic-P based on the method proposed by Jiang and Gu (1989) and modified by Adhami (2006) in calcareous soil and Kuo (1999) modified Chang and Jackson (1957) method in red soil

Step	Inorganic P fraction	Extractant	pH	Shaking time
Calcareous soil				
1	Ca_2-P	0.25 M $NaHCO_3$	7.5	1 h
2	Ca_8-P	0.5 M NH_4AOc[a]	4.2	16 h stand, 1 h
3	Prevent re-adsorption	1 M $MgCl_2$	8.0	2 h
4	Al-P	0.5 M NH_4F	8.2	1 h
5	Fe-P	0.1 M NaOH-0.5 M Na_2CO_3	12	2 h, 16 h stand, 2 h
6	Occluded-P	0.3 M CDB[b]	13	16 h
7	Ca_{10}-P	0.5 M H_2SO_4	1.0	1 h
Red soil				
1	Loosely bound P	1 M NH_4Cl	5.5	30 min
2	Al-P	0.5 M NH_4F	8.2	1 h
3	Fe-P	0.1 M NaOH	12	17 h
4	Occluded-P	0.3 M CDB	13	16 h
5	Ca-P	0.25 H_2SO_4	1	1 h

[a] Ammonium acetate

[b] 0.3 M $Na_3C_6H_5O_7$ (20 ml)-$Na_2S_2O_4$ (1 g)-0.5 M NaOH (5 ml)

mode with a Si (Li) detector, while spectra of P standards were measured in total electron (TEY) mode, which is absent of self-absorption. Multiple spectra were collected and averaged. The energy range of the scans for P K-edge XANES spectra was set at 2130 to 2200 eV, and the steps of these scans were as follows: region 1 (2130–2140 eV), step 1 eV; region 2 (2140–2146 eV), step 0.50 eV; region 3 (2146–2149.2 eV), step 0.4 eV; region 4 (2149.2–2160 eV), step 0.2 eV; region 5 (2160–2170.5 eV), step 0.3 eV; and region 6 (2170.5–2200 eV), step 0.5 eV. All XANES spectra were analyzed by Athena (0.9.23). The absolute energy scale was calibrated to 2149 eV (E_0) as the maximum energy of the first peak in the first derivative spectrum of $AlPO_4$ (Beauchemin et al. 2003). Spectra were background corrected by a linear regression fit through the pre-edge region and normalized total K-edge intensity to one unit edge jump by defining the continuum regions (> 50 eV above absorption edge) as the post-edge region. Linear combination fitting (LCF) of the soil P spectra was conducted over the spectral region from 10 eV below E_0 to 15 eV above E_0 using all the spectra of standard P chemicals without E_0 fixation. Weights of all P standards used were forced to sum 1. The goodness-of-fit was judged by the chi-squared values and R values, and P standards yielding the best fit were considered as the most possible P species in the investigated soil samples.

Phosphorus sorption isotherms

To examine the P adsorption capacity of the amended treatments, P sorption isotherms were carried out using the method of Paulter and Sims (2000), where eight 30-ml solutions of P concentration were added to 2-g soil samples in 50-ml centrifuge tubes, in duplicate. Soil samples were equilibrated for 24 h in solutions containing different P concentrations in the form of KH_2PO_4, with 0.01 M $CaCl_2$ as the background solution. Solutions of different P concentrations (0, 1, 5, 10, 15, 20, 25, 50 mg P/L) were created by diluting KH_2PO_4 stock solution (500 mg P/L). After the equilibrated process, the samples were filtered to eliminate colloidal particles. Subsequently, the concentration of P in solution was measured according to Murphy and Riley (1962). P sorbed to the soil was calculated as the difference between initial concentration and P concentration measured at equilibrium. Adsorption data and affinity constants were determined by fitting sorption data to the Langmuir and Freundlich isotherm equations. The S values (in mg P/kg) versus P remaining in solutions (C in mg P/L) were plotted by the Langmuir equation which represents a straight line (Eq. 1):

$$C/S = 1/S_{\max} \times k + C/S_{\max} \tag{1}$$

where C is the P concentration after 24 h equilibration and S is the total amount of P retained with a slope $1/S_{\max}$ and intercept $1/S_{\max} \times k$. The sorption maximum ($S_{\max}$, mg P/kg) and k, a constant related to the binding energy (L/mg), were derived from the linear form of the Langmuir equation. P buffer capacity (PBC, l/kg) was calculated as the product of $S_{\max}$ and k (Indiati et al. 1999).

The Freundlich sorption isotherm model is expressed in Eq. 2 which represents a straight line between log$C$ and log$S$ with a slope n and intercept log$K$.

$$\mathrm{LogS} = \mathrm{nlogC} + \log \mathrm{K} \tag{2}$$

The maximum adsorption amount (K, mg/kg) and the empirical constant (n, L/kg) were calculated by the linear Freundlich model.

The DPS was calculated using M3-P and P sorption maxima ($S_{\max}$) derived from Langmuir equation as follows:

$$\mathrm{DPS}\% = \mathrm{M3}{-}P/S_{\max} \times 100 \qquad (3)$$

Statistical analysis

All statistical data were analyzed using Microsoft Excel 2016, and figures were made by Sigmaplot 12.5. Analysis of variance (ANOVA) was used to determine the statistical significance of the treatment effects based on a randomized complete block design. Multiple comparisons of mean values of $CaCl_2$-P, Olsen-P, M3-P, pH, and P contents in soil P fraction and P sorption parameters were performed using the Fisher's least significant difference (LSD, $p < 0.05$) among different treatments. IBM SPSS 19.0 software was used for all statistical tests.

Results

Changes in soil pH, $CaCl_2$-P, Olsen-P, and M3-P

Alum incorporation significantly decreased the soil pH from 6.29 to 4.16 and strengthened soil acidity in the red soil. Dolomite application statistically increased soil pH compared to the soil in the control treatment. However, MAD did not significantly change soil pH after 45 days incubation in the calcareous soil, due to the closely similar pH with soil (Fig. 1).

Alum incorporation strongly reduced $CaCl_2$-P in both soils after the 45 days incubation (Fig. 2a), which implied that alum was effective in terms of P stabilization thereby protecting water quality against dissolved P losses from surface or subsurface runoff (Moore and Miller 1994; Moore et al. 1998; Brennan et al. 2011, 2014), whereas alum amendment on the reduction of soil Olsen-P content was more efficiently in the calcareous soil than in the red soil (Fig. 2b). Results showed that alum amendment significantly decreased M3-P content in calcareous soil but slightly increased the M3-P in the red soil (Fig. 2c).

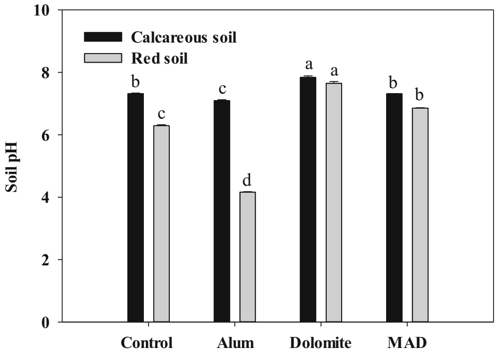

Fig. 1 One mole KCl extracted soil pH in a soil/solution ratio of 1:2.5 (*w*/*v*) in calcareous soil and red soil by adding alum, dolomite, and mixture of alum and dolomite (MAD) after 45 days incubation. Data are the means of three replicates. Bars with a different letter in different treatments in calcareous or red soil are significant at $P < 0.05$

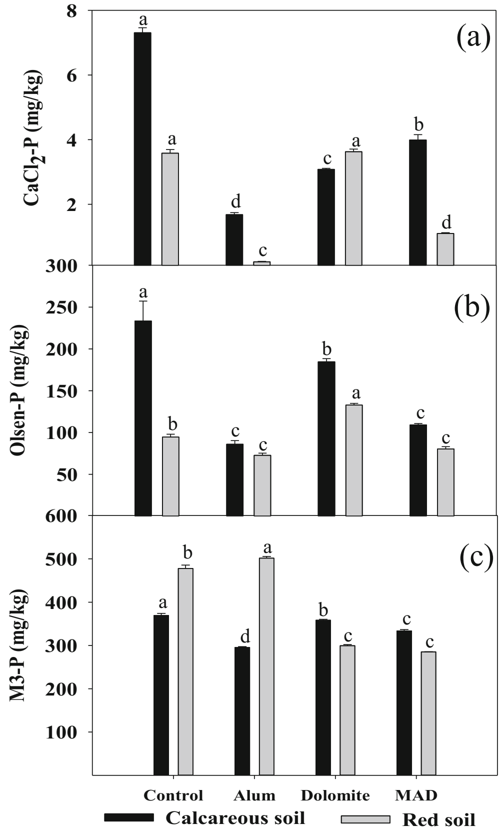

Fig. 2 Changes of (**a**) 0.01 M $CaCl_2$ extracted phosphorus, (**b**) 0.5 M $NaHCO_3$ extracted phosphorus, and (**c**) M3 reagent extracted phosphorus in calcareous and red soil treated with alum, dolomite, and mixture of alum and dolomite (MAD) after 45 days incubation. Data are the means of three replicates. Bars with a different letter in different treatments in calcareous or red soil are significant at $P < 0.05$

Compared to the amendment effects on the stabilization of soil $CaCl_2$-P, dolomite incorporation significantly decreased soil $CaCl_2$-P content by 57.8% in the calcareous soil, in contrast to the control treatment, whereas there was almost no stabilizing effect on soil $CaCl_2$-P content in the red soil (Fig. 2a). The effects of dolomite amendment decreased soil Olsen-P content by 20.8% in the calcareous soil but dolomite amendment increased the Olsen-P content in the red soil (Fig. 2b). There was no effects of dolomite amendment on M3-P in calcareous soil, but this was significantly reduced in the red soil (Fig. 2c).

The effects of dual amendment of alum and dolomite (MAD) on soil $CaCl_2$-P, Olsen-P, and M3-P varied for the two soils studied. MAD decreased soil $CaCl_2$-P by 45% and 69.2% in calcareous and red soil, respectively. MAD amendment decreased soil Olsen-P by 53.2% and 14.9% in calcareous and red soil, respectively (Fig. 2). The MAD amendment exhibited a stronger P binding effect on decreasing M3-P in red soil than that in calcareous soil. These results implied that dual amendment of alum and dolomite could not increase the stabilization effects of $CaCl_2$-P or Olsen-P in soil when compared with alum amendment alone. However, MAD was stronger in decreasing M3-P than alum or dolomite incorporation in the red soil (Fig. 2c).

Changes in the fractions of soil inorganic P

In calcareous soils, P forms included (1) readily soluble, labile, or exchangeable P such as Ca_2-P, (2) partly available P such as Ca_8-P, (3) Al-P and Fe-P, (4) occluded P (O-P), and (5) stable Ca-P such as Ca_{10}-P (Chang and Jackson 1957; Jiang and Gu 1989; Barbanti et al. 1994; Ruiz et al. 1997). The amendments did not alter the total inorganic P but the distribution of P in various fractions (Table 3). Alum incorporation decreased the fractions of Ca_2-P and Ca_8-P by 51.0% and 66.7%, respectively, whereas the fractions of Al-P and Fe-P increased by 318% and 40%, respectively (Table 3). There were no significant effects on the fractions of O-P and Ca_{10}-P in calcareous soil. On the other hand, alum incorporation decreased the fractions of loosely bound P (NH_4Cl-P), O-P, and Ca-P by 91.6%, 28.4%, and 40.5% in red soil, while the fractions of Al-P and Fe-P increased by 43.8% and 15.6%, respectively, compared to the control treatment (Table 3).

Dolomite addition increased Ca_2-P fraction by 9.4% and decreased the amount of Ca_8-P by 7.4% in the calcareous soil. However, the sum of Ca_2-P, Ca_8-P, and Ca_{10}-P in the dolomite treatments was similar to the control treatment (Table 3). On the contrary, dolomite addition increased the fraction of NH_4Cl extracted P by 3.78% and the fraction of Ca-P by 88.0% in the acidic red soil while decreased the Al-P and Fe-P contents by 19.4% and 19.9%, respectively (Table 3).

MAD had the same tendency relative to alum in the calcareous soil, which caused a 32.7% and 42.3% decline in Ca_2-P and Ca_8-P with the increase of 212% and 30.3% in Al-P and Fe-P. However, MAD reduced soil NH_4Cl extracted P by 42.8% as alum addition and increased soil Ca-P by 40.0% as dolomite added in red soil. More importantly, there were no significant changes of Al-P, Fe-P, and O-P contents due to MAD amending (Table 3).

Table 3 Contents of different fractions of inorganic phosphorus (P_i) in the calcareous and red soil treated with different amendments after 45 days incubation

Treatment	Contents (mg/kg)						
Calcareous soil							
	Ca_2-P[a]	Ca_8-P	Al-P	Fe-P	O-P	Ca_{10}-P	Total P_i
Control	137.3b[c]	292.7a	82.6c	39.6b	99.9a	426.5a	1079a
Alum	67.2d	97.5d	345.9a	55.4a	106.1a	430.6a	1103a
Dolomite	150.2a	271.0b	89.2c	37.9b	95.4a	410.4a	1054a
MAD[d]	92.3c	169.0c	257.7b	51.6a	95.8a	430.6a	1097a
Red soil							
	NH_4Cl-P[b]		Al-P	Fe-P	O-P	Ca-P	Total P_i
Control	45.4b		272.1b	420.4b	374.1a	133.7c	1246a
Alum	3.8d		391.4a	486.0a	268.0b	79.5d	1229a
Dolomite	62.6a		219.4c	336.7d	379.9a	251.3a	1250a
MAD	25.9c		271.2b	385.1c	407.6a	184.5b	1274a

[a] Ca_2-P, Ca_8-P, Al-P, Fe-P, occlude-P (O-P), and Ca_{10}-P were fractioned with modified Jiang and Gu method and were extracted with 25 ml 0.25 M $NaHCO_3$, 0.5 M NH_4AOc, 0.5 M NH_4F, 0.1 M NaOH-0.5 M Na_2CO_3, 0.3 M $Na_3C_6H_5O_7$-$Na_2S_2O_4$ (1 g)-0.5 M NaOH, and 0.5 M H_2SO_4, respectively, in calcareous soil

[b] NH_4Cl-P, Al-P, Fe-P, O-P, and Ca-P were fractioned with modified Chang and Jackson method and were extracted with 25 ml 1 M NH_4Cl, 0.5 M NH_4F, 0.1 M NaOH, 0.3 M $Na_3C_6H_5O_7$-$Na_2S_2O_4$ (1 g)-1 M $NaHCO_3$, 0.25 H_2SO_4, respectively, in red soil

[c] Data are the means of three replicates. Different letter in different treatments in calcareous or red soil are significantly at $P < 0.05$

[d] Mixture of alum and dolomite at the ratio of 1:1

Change of P speciation by K-edge XANES

The results of linear combination fitting (LCF) for soil samples under different treatments are presented in Table 4. All the soil samples had generally excellent chi-squared values and LCF fits < 0.5 and 0.005. The LCF results showed the proportion of total Ca-P (the sum of DCP and HAP) accounted for 61–76% of total phosphorus (TP) in the calcareous soil and $FePO_4$ accounted for 50–60% of the TP in red soil (Table 4). The results of the soil P fractionation also

Table 4 Phosphorus K-edge XANES fitting results showing the relative percent (%) of each phosphate species in the studied soil samples with different amendments after 45 days incubation

Treatment	HAP	DCP	$FePO_4·2H_2O$	$AlPO_4$	Total Ca-P
Calcareous soil					
Control	49.8a[a]	25.8c	24.4c	n.d	75.6a
Alum	22.0c	39.8a	38.2a	n.d	61.8c
Dolomite	49.4a	26.5c	24.1c	n.d	75.9a
MAD[b]	31.2b	34.6b	34.3b	n.d	65.7b
Red soil					
Control	17.5	26.5c	56.0b	n.d	44.0b
Alum	n.d	11.9d	71.1a	17.0	
Dolomite	15.2	33.9b	51.0c	n.d	49.1a
MAD	4.9	38.5a	56.6b	n.d	43.4b

HAP hydroxyapatite [$Ca_5(PO_4)_3OH$], *DCP* dibasic calcium phosphate dihydrate ($CaHPO_4·2H_2O$), *Total Ca-P* sum of HAP and DCP

[a] Data are the means of three replicates. Different letter in different treatments in calcareous or red soil are significant at $P < 0.05$

[b] Mixture of alum and dolomite at the ratio of 1:1

showed that the total Ca-P (Ca_2-P, Ca_8-P, and Ca_{10}-P) dominantly existed in the calcareous soil and Fe-P is the maximum fraction in the red soil.

Alum addition significantly decreased the total HAP and enhanced the $FePO_4$ in calcareous soil (Table 4). However, $AlPO_4$ was not detected which means that the increase of Al-P in the soil P fraction in the calcareous soil might be related to the $Al(OH)_3$ absorbed P. Conversely, a 17% increase in $AlPO_4$ was found in the red soil when compared with control treatment (Table 4).

Similar to the result of the soil P fraction, dolomite had almost no effect on the P speciation in the calcareous soil, whereas it significantly enhanced the proportion of $CaHPO_4$ and decreased the $FePO_4$ according to the LCF in red soil (Table 4). MAD significantly increased $FePO_4$ proportion, lowered HAP proportion and total Ca-P proportion as the alum addition in calcareous soil, and had no effect on $FePO_4$ in acidic red soil compared to the control treatment, similar to the results of soil inorganic P fractions (Table 4).

Characteristics of soil P sorption

The P sorption isotherms for all treatments are presented in Fig. 3. In the control treatment, the amount of soil adsorbed P showed a nonlinear relationship with the increasing equilibrium P solution (EPC) and was followed by a roughly steady value when the added P level was higher than 10 mg/L in both soils. All the amendment treatments produced a steeper curve compared with the control treatment. This means that both alum and dolomite increased the P sorption maxima whether the levels of $CaCl_2$-P, Olsen-P, or M3-P increased or decreased (Fig. 2). Generally, the sharpest P sorption curve was produced by dolomite addition in the calcareous soil and alum addition in red soil. This was in accordance with the results of soil $CaCl_2$-P change that alum decreased more $CaCl_2$-P in red soil than the calcareous soil, while dolomite bound more $CaCl_2$-P in calcareous soil. The alum-treated calcareous and red soils showed a poor adsorption amount when the amount of added P was higher than a certain threshold (25 mg/L).

The parameters of the sorption isotherms of phosphate on soils described by Langmuir and Freundlich models are listed in Table 5. Both the Langmuir model and Freundlich model could fit the sorption properties of the amendments treated soil ($R^2 > 0.88$). Dolomite addition produced a maximum P sorption (S_{max}) amount and P buffer capacity (PBC) in the calcareous soil but decreased the PBC in red soil. Alum addition produced the highest S_{max}, k, and PBC in red soil and the highest binding energy in calcareous soil.

The k values of the MAD treatment were lower than the control treatment which meant that it decreased the P absorbed capacity in red soil. But, MAD amending increased both the Langmuir S_{max} and k values in calcareous soil.

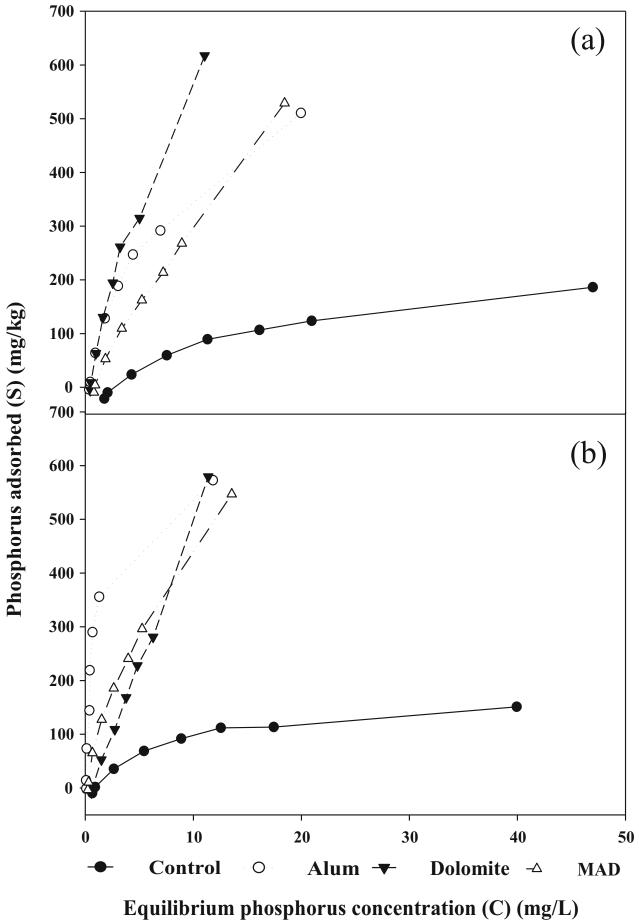

Fig. 3 The phosphorus soption isotherms in (**a**) calcareous soil and (**b**) red soil by adding alum, dolomite, and mixture of alum and dolomite (MAD) after 45 days incubation

Discussion

Effect of alum or dolomite on phosphorus stabilization

Adhami et al. (2006) reported that the abundance of different fractions of P was in the order of Ca_2-P < Fe-P < Al-P < O-P < Ca_8-P < Ca_{10}-P based on the 21 calcareous soils of Iran. Our results showed the content of the Ca_2-P increased rapidly in the high P accumulated soil, and its proportion has exceeded O-P (Table 3). In red soils, approximately 85 to 90% of added inorganic P adsorbed or precipitated with Fe, Al, and Ca in the year of application (Gichangi et al. 2009; Khan and Joergensen 2009; Yang et al. 2012); consequently, NH_4Cl extracted P which is defined as loosely bound P (Chang and Jackson 1957) is generally low in quantity. However, the excessive application of P fertilizer increased the NH_4Cl extracted P when strong P-bound sites were occupied. Wang et al. (2014) reported that the NH_4Cl extracted P in soil significantly increased from 1.58 to 16.1 mg/kg with 22-year pig manure application. The NH_4Cl extracted P of the red soil in our study

Table 5 Phosphorus sorption data represented by Langmuir S_{max}, k, and Freundlich K, n with R^2 values derived from data fitted to both model, M3 extracted elements and DPS in calcareous, and red soil treated with different amendments at 45 days incubation

Treatments	Langmuir				Freundlich								
	S_{max} (mg/kg)	k (L/mg)	PBC (L/kg)	R^2	K (mg/kg)	n	R^2	Al_{ox} (mg/kg)	M3-Al (mg/kg)	M3-Fe (mg/kg)	M3-Ca (g/kg)	M3-Mg (mg/kg)	DPS[a] (%)
Calcareous soil													
Control	570	0.56	322	0.99	331	0.14	0.99	373c	213c[b]	111b	5.85c	806d	64.8
Alum	840	0.77	645	0.98	393	0.25	0.99	1287a	877a	85d	10.12a	832c	35.2
Dolomite	1053	0.74	775	0.96	465	0.31	0.98	385c	191c	115a	5.27d	1843a	34.0
MAD[c]	917	0.41	375	0.93	353	0.28	0.96	825b	550b	105c	6.53b	1429b	36.4
Red soil													
Control	633	1.48	935	0.99	483	0.07	0.99	597c	474c	538a	1.82d	87d	75.5
Alum	1090	5.28	5750	0.99	784	0.15	0.95	1745a	1300a	473b	2.40c	168c	46.0
Dolomite	971	0.35	342	0.88	323	0.34	0.93	570c	299d	435c	2.85b	1586a	30.9
MAD	870	0.68	595	0.96	390	0.25	0.98	1068b	761b	394d	3.64a	997b	32.8

Al_{ox} aluminum extracted by the acid ammonium oxalate, *DPS* degree of phosphorus saturation

[a] DPS% = M3-P/S_{max} × 100

[b] Data are the means of three replicates. Different letter in different treatments in calcareous or red soil are significant at $P < 0.05$

[c] Mixture of alum and dolomite at the ratio of 1:1

had reached 50.5 mg/kg that represents the high risk for P loss (Table 3).

Addition of Al salts has been used for decades to restore eutrophic lakes (Lewandowski et al. 2003). Similar treating strategies were also found in manure and soil (e.g., Brennan et al. 2014), and in our study, alum addition greatly decreased the $CaCl_2$-P by 77.0% in calcareous soil and 93.8% in red soil, respectively (Fig. 2a). The main labile P fraction in soil was transformed to a more stable Al-P pool (Table 3). However, the mechanisms forming stable Al-P and the transformation of soil labile P to stable P were different in the calcareous and red soil. Lewandowski et al. (2003) reported that P precipitation removed phosphates by different mechanisms with alum addition, depending on the pH. In our experiment, soil pH changed from 7.31 to 7.10 in the calcareous soil and 6.29 to 4.16 in the red soil with alum addition (Fig. 1). Cooke et al. (1993) suggested that under circumneutral conditions (pH 6.0–8.0), $Al(OH)_3$ is formed. The result was supported by Delhaize and Ryan (1995) who demonstrated that Al hydrolyzes in solution such that the trivalent Al species, Al^{3+}, dominated in acid conditions ($pH < 5$), whereas the $Al(OH)^{2+}$ and $Al(OH)_2^+$ species form as the pH increased. At near-neutral pH, the solid phase $Al(OH)_3$ occurs, whereas $Al(OH)_4^-$ or aluminate dominated in alkaline conditions. On the other hand, the P K-edge XANES results showed that $AlPO_4$ was detected in the red soil with alum addition, indicating that part of labile P was precipitated by Al^{3+}. However, considering that the soil pH approached 7 with the alum addition and the K-edge XANES did not detect any $AlPO_4$ in the calcareous soil (Table 4), we speculated that the poorly crystallized hydroxy aluminum might be dominantly formed to adsorb labile P in calcareous soil with alum amendment addition. These speculation were supported by the significantly increased acid ammonium oxalate extracted Al content by 241% in the alum-amended calcareous soil compared to the control treatment (Table 5), which has a rather good relationship with the formation of the poorly crystallized hydroxy aluminum in soil (Devau et al. 2011; Li 1994). However, studies that focus on the interaction of phosphate with aluminum demonstrated the rate of phosphate removal strongly increased as pH decreased from 8.5 to 3.5. And, no further phosphate was removed after 1 day of reaction time with aluminum when pH = 8.5 (Chen et al. 1973). Therefore, it can be interpreted that the less efficiency in P stabilization in the calcareous soil with alum addition might as the contents of the poorly crystallized hydroxy aluminum which has a higher P sorption capacity was less than that in the red soil (Jenkins et al. 1971) (Table 5).

Dolomite addition could stabilize labile P in calcareous soils through surface adsorption or precipitation induced by Ca^{2+} ions (Tunesi et al. 1999). Eslamian et al. (2018) found that the dolomite application dosages at 2% decreased P loss in a clay loam soil, and the reason was that the elevated soil pH increased the stable Ca-P compounds, which was also found in the present study. Soil pH increased from 7.21 (control) to 7.84 after dolomite addition in the calcareous soil; correspondingly, the exchangeable Ca and M3-Ca declined from 1.72 and 5.81 to 1.37 and 5.27 g/kg in the calcareous soil, respectively (Tables 5 and 6). On the other hand, Drever (1982) noted that dolomite is usually formed by alteration of aragonite or calcite as follows:

Table 6 Contents of exchangeable Al, Fe, Ca, and Mg in the calcareous and red soil treated with different amendments after 45 days incubation

Treatment					
Calcareous soil	Ex-Al[a] (mg/kg)	Ex-Ca (mg/kg)	Ex-Fe (mg/kg)	Ex-Mg (mg/kg)	Ex-Ca/Ex-Al (molar ratio)
Control	1.87ab	1722c	1.08a	479.6b	645c
Alum	2.33a	3952a	1.00a	475.7b	1155a
Dolomite	1.62b	1370b	0.96a	682.4a	582c
MAD[b]	2.04ab	2587d	0.96a	660.1a	858b
Red soil					
Control	3.6b	1154c	1.39b	74.0d	232.3b
Alum	158.9a	2096a	3.90a	126.1c	8.9c
Dolomite	3.3b	819b	1.09b	330.3a	168.7b
MAD	3.0b	1885d	0.84b	313.1b	425.3a

[a] Exchangeable Al, extracted by 1 M KCl solution

[b] Mixture of alum and dolomite at the ratio of 1:1

$$CaMg(CO_3)_2 + Ca^{2+} = 2CaCO_3 + Mg^{2+}$$

Therefore, the decline in the $CaCl_2$-P, Olsen-P, and M3-P contents was from the adsorption of the dolomite rather than through Ca^{2+} precipitation in the calcareous soil. Results of XANES also showed that there was no significant difference in the $CaHPO_4$ content between the control and dolomite-treated calcareous soil (Table 4). However, the P transformation influenced by dolomite addition was quite different in red soil. The percentage of $CaHPO_4$ in the P K-edge XANES and stable Ca-P in the soil P sequential fractionation was significantly increased in the dolomite-treated red soil, and $FePO_4$ content was declined when compared to the control (Tables 3 and 4). It was no doubt that hydrated Al and Fe oxides were the dominant phosphates adsorbing surfaces in acidic red soils and maximum adsorption on such substance occurred at slightly below pH 4.74 (Juo and Fox 1977; Haynes 1982). Dolomite significantly increased the pH of the red soil (Fig. 1), which broke the ligand exchange onto the amphoteric metal hydrous oxide surfaces. Consequently, the decline of the Al-P and Fe-P in the P fraction (Table 3) and the enhancing effects on soil available P was observed in our experiment (Fig. 2). Simonsson et al. (2018) also reported that liming enhanced the solubility of P when spread in parallel with chemical fertilizers. On the other hand, the consequently increased soil Ca-P level in the red soil meant the new formed Ca^{2+} bound P (Table 3). The decreased soil exchangeable Ca in the red soil also interpreted that Ca^{2+} might be bound with other anions, i.e., phosphate (Table 6). Therefore, dolomite addition in the red soil to stabilize P might be through dolomite surface adsorption and Ca^{2+} precipitation. However, phosphate was mainly adsorbed by dolomite rather than the precipitation with Ca^{2+} because the red soil pH increased from 6.29 to 7.65 after dolomite incorporation (Fig. 1).

Soil P sorption capacity affected by soil amendments addition

The red soil exhibited greater values of S_{max}, k, and PBC derived from Langmiur model, which were supported by Daly et al. (2015) who demonstrated the greater values of S_{max}, k, and PBC of non-calcareous soil resulted from the greater amount of extractable Al. However, higher phosphorus sorption capacity in red soil as Fe- and Al-organic matter complexes could play an important role in controlling P sorption potential through cation bridges, altering surface charges, boosting competition with other anions for adsorption sites. This was related to high soil OM, high extractable Fe in red soil (Borggaard et al. 2005; Fink et al. 2016).

Alum addition in the calcareous soil increased the values of Langmuir sorption parameters in this study. Villapando and Graetz (2001) demonstrated Al was strongly correlated with P sorption and Ballard and Fiskell (1974) concluded that dominant nature of extractable Al provided the best indicator of P retention in soils. Therefore, the increased Langmuir sorption parameters in our study might be related to the increased amount of extractable Al (Table 5). Alum addition also significantly decreased the soil pH (Fig. 1), and the M3-Ca was significantly enhanced from 5.85 g/kg in the control treatment to 10.12 g/kg in the alum-treated calcareous soil (Table 5). However, the results of the P fractionation showed that there was no freshly formed Ca-P. It could be implied that P adsorbed by Al hydroxide was stronger than the Ca^{2+}, in line with the results reported by Zak et al. (2004) who indicated that the binding capacity of Al by adsorption is greater than that of Ca with P at weak alkaline conditions. Supposed high P concentration was added into the alum-treated soil and occupied the P adsorbed site of Al hydroxide, the Ca^{2+} might be precipitated with phosphate.

In the red soil, alum addition showed a rather higher Langmuir k and PBC values. Alum addition in the red soil caused a lower pH of 4.17 (Fig. 1). Consequently, the enhanced adsorption capacity of the Al and Fe oxides and the active Al^{3+} showed a rather strong bonding energy when alum was incorporated to the red soil.

Dolomite-treated soil showed a highest S_{max} (1053 mg/kg) derived from the Langmuir model in the calcareous soil (Table 5). However, the Jiang and Gu (1989) soil P fraction of the dolomite-treated calcareous soil implied that dolomite addition had almost no effect on the inorganic P transformation (Table 3). Dolomite amending significantly increased the P adsorption amount, whereas it decreased the adsorbed capacity compared to alum amending based on the significant decrease of the $CaCl_2$-P, highest P adsorption amount, and slightly fluctuation of the inorganic P transformation in calcareous soil. Emil (2000) also reported that P adsorbed by Ca type amendments was relatively weaker.

Dolomite addition in red soil also showed a rather high P adsorption amount (971 mg/kg), but less than that in calcareous soil. However, the increased soil pH inhibited the adsorption of the Fe, Al oxides, and the weaker adsorption of the Ca caused a lower binding energy and PBC (Table 5).

Phosphorus sorption curves showed a linear relationship between the increasing P adsorption of dolomite-treated soil with the increasing P concentration, while the adsorption amount of the alum-treated soil was limited when the P concentration exceeded the threshold of 50 mg/L, and the phosphate fixation declined sharply (Fig. 3). We concluded that the P adsorption or precipitation capacity of alum-treated soil might be strong, while the total P adsorbed amount might not be so high as the dolomite when high P concentration solution was added.

Agronomic and environmental impacts of P stabilization

P transportation from soil to water is controlled by (i) the total amount of desorbed P containing solution P and the continuously released P derived from the active or fixed P in the long-term (Kruse et al. 2015); (ii) the partition of P in the soil solid phase between adsorbed and precipitated forms, e.g., addition of fertilizer will overwhelm the balance between P in solution and solid phase, with time, the readily available P forms transformed into more recalcitrant P forms, but surplus P applications eventually convert soils from sinks to sources of P loss (Kleinman et al. 2011); and (iii) the kinetics of phosphate desorption from various adsorption sites influenced by pH, the concentration of Al, Fe, Ca, and Mg, and the behavior and surface area of soil particles and soil moisture (Mcdowell et al. 2001; Mulqueen et al. 2004). In the present study, high enriched P soils had adequate labile P sources to support crop growth, which increased the risk of P loss to water. Generally, Olsen-P and $CaCl_2$-P levels are commonly used to describe the agronomic and environmental thresholds. Olsen-P proved a reasonably good predictor of the amount of P released to the soil solution as a response to depletion through uptake by plants. However, $CaCl_2$-P level was used to tentatively evaluate some environmental risk.

Jiang and Gu (1989) found that the solubility of synthetic Ca_2-P increased steeply in $NaHCO_3$ (pH 8.5) with a concentration from 0.1 to 0.5 M at 20 °C after shaking for 1 h. It can be concluded that the content of $CaCl_2$-P that all belongs to Ca_2-P and Ca_2-P is the main part of Olsen-P which was extracted by 0.5 M $NaHCO_3$ at pH 8.5. Adhami et al. (2006) also showed a significant relationship between Ca_2-P and Olsen-P, and assumed Ca_2-P suggested by Jiang and Gu (1989) and Olsen-P extract the same pool of P. However, Olsen-P is extracting the available, readily desorbable P which includes a large proportion of Ca_2-P and a little Ca_8-P, Al-P, and Fe-P described in the soil P fraction. In our study, amending alum significantly decreased the Olsen-P and $CaCl_2$-P mainly due to the increasing transforming proportions Ca_2-P and Ca_8-P to Al-P (Table 3). Khsawneh (1980) described that the pK_{sp} of $AlPO_4$ is almost four times than the $CaHPO_4$ which means that alum addition would decrease the P solubility and followed by P loss. On the other hand, dolomite mainly decreased a large proportion of $CaCl_2$-P and a little Olsen-P in calcareous soil (Fig. 2). However, when considering the agronomic aspects, it is known that Al^{3+} reduces the uptake of Ca and Mg by replacing them in the root apoplast (Scholl et al. 2004). It was found that the dosage of alum and MAD in the 2% of the soil dry weight did not significantly increase the exchangeable Al contents in the calcareous soil. In the red soil, it was not suggested to add alum, as the soil pH would decrease significantly. Simultaneously, the soluble Al increased from 3.6 to 158.9 mg/kg with the 2% dosage alum addition. However, dolomite or MAD addition can decrease the soil exchangeable Al and increase the exchangeable Ca and Mg which benefits to plant growth in the red soil (Table 6). Dolomite addition also increased the P sorption maximum and decreased the degree of P saturation (Table 5), indicating the decline risk of P loss (Fischer et al. 2017) in calcareous and red soil. Considering the subsequent P fertilizer application after dolomite amending, it was implied that dolomite amending performed more flexibly in controlling soil test P when compromising the environmental and agronomic P management. Besides the functions of added metal ions from the amendments, the pH change of treated soils (Fig. 1) will influence P transformation and plant growth, which was also described by Novak and Watts (2005) and Eslamian et al. (2018). The decline of $CaCl_2$-P in the MAD treatment was not so effective as dolomite or alum addition independently, possibly because of the interaction between dolomite and alum which influenced the function of both materials (Table 5). However, the dual amendments only slightly changed the soil pH which means that it has less effect on the agronomy (Fig. 1).

Conclusions

The present work showed that alum, dolomite, or dual amendment reduced soil P loss risk when added to the soil. However, when using alum amendment alone, P was mainly adsorbed by Al hydroxide in calcareous soil and precipitated as $AlPO_4$ in red soil. When dolomite was amended alone, the labile P decreased through adsorption in calcareous soil, while it increased the loosely bound P due to mitigating the specific P adsorption by Fe-Al oxide in red soil. However, P sorption isotherms demonstrated dolomite addition in red soil increased the maximum P sorption amount and decreased the DPS. The dual amendments showed the similar tendency on P stabilization as the alum in calcareous soil and dolomite in red soil. However, MAD performed many agronomic advantages due to no influence on soil pH change and the risk of Al toxicity. Considering the higher P sorption amount and the agronomic and environmental aspects by soil amendments addition, it was concluded that amending MAD in calcareous soil and dolomite in red soil could efficiently stabilize P-enriched soils and regulate the balance of the environmental and agronomic aspects.

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