Reduction of phosphorus release from high phosphorus soil by red mud

Zhen Liang · Xianjia Peng · Zhaokun Luan ·
Wenjun Li · Ying Zhao

Received: 1 August 2010 / Accepted: 9 May 2011 / Published online: 26 May 2011
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Abstract The high phosphorus levels cause the release of phosphorus from soils, thereby increasing the potential for phosphorus export to adjacent water bodies. The loss of phosphorus from soils to surface waters is a major source of water quality impairment. Therefore, soil phosphorus immobilization seems necessary. In this study, red mud (RM) was employed to immobilize phosphorus in a typical agricultural soil. It was found that phosphorus was effectively immobilized by RM. Batch leaching experiments showed that RM reduced phosphorus release from 14.38 to 2.56 mg/kg when soil was amended with 1% RM. Column leaching experiments showed that RM reduced the total amount of phosphorus released from 36.73 to 18.79 mg/kg during the investigated period. Sequential chemical extraction results indicated that RM amendment transformed H_2O -P into more stable fractions. The results suggested that application of RM amendment to soils could significantly immobilize soluble phosphorus, reducing phosphorus release to the environment.

Keywords Red mud · Immobilization · Phosphorus · Clay soil · Release

Introduction

Phosphorus is an essential element for all forms of life on the earth, which is considered as one of the major nutrients limiting the productivity of crops. To meet global needs for food, high ratios of the fertilizer or manure were applied on the soils. This leads to excessive phosphorus in soils, which makes these soils vulnerable to phosphorus loss. Phosphorus loss from agricultural soils has been reported to be one of the major non-point sources for pollution of surface water and contributes to the eutrophication of nearby water bodies (McDowell et al. 2008; Klienman et al. 2004; Udeigwe et al. 2009; Daniel et al. 1998; Parry 1998). Phosphorus pollution caused by soil release has attracted attention in recent years due to continuing trends of increasing phosphorus levels in soils. Especially, in East China, long-term fertilizer or intensive manure applications have resulted in far more phosphorus than the crops need in the majority of agricultural soils (Cao 2003; Wang et al. 2004; Liang et al. 2007). The accumulation of excessive phosphorus in surface soils could be released by the rainfall leaching and surface runoff and, therefore, contributes to the impairment of surface water quality (Sharpley 2003; Sims et al. 1998). Phosphorus release from soils is of increasing concern and soil phosphorus immobilization seems necessary.

The conventional options for minimizing agricultural soil phosphorus release focused on phosphorus input reduction, soil amendment, conservation tillage, and buffer zone building (Sharpley and Rekolainen 1997). Among them, a promising method is the addition of soil amendment, which can improve soil retention and reduce phosphorus release. Soil amendment is an in situ remediation method, where materials are added to contaminated soil to reduce phosphorus release. Soluble phosphorus in soil

Z. Liang · X. Peng (✉) · Z. Luan · Y. Zhao
State Key Laboratory of Environmental Aquatic Chemistry,
Research Center for Eco-Environmental Sciences,
Chinese Academy of Sciences, P.O. Box 2871,
Beijing 100085, People's Republic of China
e-mail: xjpeng@rcees.ac.cn

W. Li
College of City of Construction,
Hebei University of Engineering,
Handan 056038, People's Republic of China

solution can react with aluminum, iron, and calcium to form insoluble phosphorus. Previous studies have shown that added materials containing aluminum, iron, and calcium oxides or hydroxides can reduce phosphorus release. Various soil amendments including calcium oxides, calcium hydroxide, calcium carbonate, calcium chloride, magnetite nanoparticles, iron chloride, iron sulfate, and aluminum sulfate have been employed for reduction of phosphorus solubility and release to the environment (Moore and Miller 1994; Ann et al. 1999; Yang et al. 2007; Pan et al. 2010; Sherwood and Qualls 2001; Dou et al. 2003; Staats et al. 2004).

The feasibility of employing amendments to reduce soil phosphorus release will depend on the cost and availability of the materials. It seems interesting to search for cost-effective alternative amendments for reduction of soil phosphorus release. Red mud (RM) is the waste material of aluminum industry. Because of its relatively large surface area and high content of iron, aluminum, calcium oxides and hydroxides, RM can be considered as a cost-effective material for phosphorus adsorption and immobilization. A number of studies have shown that RM can be used for phosphorus removal from aqueous solutions (Roberge et al. 1999; Li et al. 2006, 2007; Huang et al. 2008a; Zhao et al. 2009; Yue et al. 2010). Otherwise, previous study has shown that RM can be an effective amendment for phosphorus immobilization in barren and sandy soils (Summers et al. 1993, 1996a, b, 2001, 2002; Vlahos et al. 1989; Ward and Summers 1993).

Clay soils are widespread in East China. Long-term fertilizer or manure applications have led to excessive phosphorus accumulation. There have been few reports on the feasibility of employing RM for immobilization of pre-existing phosphorus in clay soils. In this study, the feasibility of employing RM for phosphorus immobilization in clay soils was investigated. Soil samples were incubated with RM. Batch and column leaching experiments were conducted to evaluate the phosphorus immobilization efficiency. In addition, phosphorus speciation in the amended soil was analyzed to explain the mechanisms of immobilization of phosphorus.

Materials and methods

Materials

Soil samples were collected from the high phosphorus soil near the Taihu Lake, which is located in East China. Representative top-layer soil samples (depth: 0–10 cm) were collected using a mechanical scoop. Collected soil samples were air dried, milled to pass a 2-mm sieve and stored in closed dark bottles.

RM used in the present study was obtained from Shandong Branch, Aluminum Corporation of China, which was air dried, crushed and sieved to pass a 2-mm sieve prior to use. The composition and properties of RM were listed in Table 1.

Adsorption isotherm investigation methods

Prior to the immobilization investigation, phosphorus adsorption capacity of RM from phosphorus solution was studied in order to assess the immobilization potential of RM for phosphorus in soils. Adsorption studies were carried out by batch experiments, 0.04 g of RM was equilibrated with 40 ml of phosphorus solution of known concentration (0, 1, 2, 5, 10, 20, 50, 100, 200, 250, 300, 350 mg/L) at 25°C for 24 h. After equilibrium, the solutions were centrifuged at 5,000 rpm for 10 min. The supernatant was filtered through 0.45-μm membrane filter and collected for phosphorus analysis. The amount of adsorbed phosphorus was calculated from the difference of concentrations before and after adsorption. Phosphorus adsorbed was calculated as follows:

$$q = \frac{(C_o - C_e)V}{m} \quad (1)$$

where q is the amount of the phosphorus adsorbed (mg/g), C_o and C_e are the initial and final concentrations of the phosphorus in solution, respectively (mg/L), V is the solution volume (L), m is the mass of adsorbent (g). The results were graphically presented as a plot of the amount adsorbed versus equilibrium concentration in the solution.

Incubation methods

In the incubation experiments, soil samples were thoroughly mixed with RM at prescribed mass ratios. The mixtures were weighed into polypropylene bottles and then de-ionized water was added at a water/mixture ratio of 30% to maintain soil moisture at field holding capacity. The mixtures were sealed and incubated in the dark at room temperature for 7 days (Dou et al. 2003; Moore and Miller 1994; Felton et al. 2004). After the incubation process was completed, the incubated samples were air dried, milled to pass a 2-mm sieve and stored in closed dark bottles for further investigation.

Leaching investigation methods

Batch leaching experiments were carried out to optimize the mass ratio of RM to soil and assess the immobilization efficiency of RM amended soil. 2.0 g of incubated sample was added into a 50-ml centrifuge tube, and then 20-ml de-ionized water was added. Each sample was equilibrated

Table 1 Composition and properties of RM (Liang et al. 2010)

Composition	RM
SiO ₂ % (w/w)	12.45
Fe ₂ O ₃ % (w/w)	25.68
Al ₂ O ₃ % (w/w)	10.98
CaO% + MgO% (w/w)	13.88
Na ₂ O% (w/w)	5.76
K ₂ O% (w/w)	0.33
TiO ₂ % (w/w)	12.94
LOI% (w/w)	15.41
pH	12.55
BET (m ² /g)	16.48
Crystalline phases	Hematite, Bayerite, Calcite, Cancrinite, Goethite, Rutile and Quartz

for 1 h on a shaker and centrifuged at 5,000 rpm for 10 min. Unfiltered samples were collected for pH measurement. Filtered samples (0.45-μm membrane filter) were collected for phosphorus analysis and the immobilization efficiency was calculated as follows:

$$\eta = \left(1 - \frac{C_e}{C_o}\right) \times 100\% \quad (2)$$

where η is the immobilization efficiency, C_o is the phosphorus concentration of un-amended soil, C_e is the phosphorus concentration of amended soil.

Column leaching experiments were carried out to investigate the immobilization efficiency of RM in the field. A schematic diagram of the column used for this study is given in Fig. 1. Polyvinyl chloride (PVC) columns with inner diameter of 4.0 cm and length of 50.0 cm were used. Small amounts of incubated samples were homogeneously packed in the column by a spatula, which was repeated until the weight of the incubated samples reached 300 g. The column was operated in the downward flow configuration. De-ionized water was fed to the columns by a peristaltic pump. The average flow ratio was 250-ml/day, corresponding to 1.0 pore volume per day. Prior to the column leaching experiments, each column was saturated with de-ionized water at room temperature for 3 days to allow soil natural compact and chemical reaction equilibrium.

Analytical methods

The pH and electrical conductivity (EC) values of the soil were measured in slurry at a solid/solution ratio of 1.0:2.5 according to the method suggested by International Soil Association. Particle size distribution was determined using a Malvern Mastersizer 2000 laser particle size analyzer (Malvern Co., the UK) with an ultrasonic deconcentrator. The moisture content of the soil was calculated

Fig. 1 A schematic diagram of the column study

according to the mass difference before and after the soil being dried in an oven at 105°C for 24 h (Zupancic et al. 2004). Organic matter content of the soil sample was determined using the loss-on-ignition method (Ben-Dor and Banin 1989). Total phosphorus concentration (TP) was analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES, Prodigy, USA) following acid-peroxide digestion according to the EPA Method 3050A (1986). Soluble reactive phosphorus (SRP) concentration of the soil was measured in 0.01 M KCl solution at a solid/solution ratio of 1:10 (Agyin-Birikorang et al. 2007). Bray-P concentration of the soil was determined by reacting the soil sample with 0.025 M HCl in 0.03 M NH₄F solution at a solid/solution ratio of 1:10 (Brown 1998). For the total element analysis, the soil was digested using concentrated nitric acid, hydrofluoric and perchloric acid according to the method by Sterckeman et al. (2006), then the total element concentrations were analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES, Prodigy, USA).

Phosphorus concentration in the leachate was analyzed by the phosphomolybdate blue method of Murphy and Riley (1962) and Walter et al. (1997). Phosphorus speciation of the amended soil was determined according to Hedley et al. (1982). For the phosphorus speciation determination, the extraction was carried out in 50-ml centrifuge tubes. To each tube, 0.5 g of incubated soil samples was added. Briefly, five steps were included in the extraction. Step 1, 30-ml de-ionized water was added to the samples. The samples were shaken for 16 h at room temperature and centrifuged (H₂O-P). Step 2, after decanting, 30-ml 0.5 M NaHCO₃ (pH 8.2) was added to the residue from step 1. The residue was shaken for 16 h at room temperature and centrifuged (NaHCO₃-P). Step 3, after decanting, 30-ml 0.1 M NaOH was added to the residue from step 2. The residue was shaken for 16 h at room temperature and centrifuged (NaOH-P). Step 4, after decanting, 30-ml 1 M HCl was added to the residue from step 3. The residue was shaken for 16 h at room temperature and centrifuged

(HCl-P). Step 5, after decanting, the residue was digested with $\text{H}_2\text{SO}_4\text{--H}_2\text{O}_2$ by microwave accelerated reaction system (CEM, USA) (Residual-P). Total phosphorus was the sum of all above phosphorus.

Mineralogical constituents of RM were determined using X-ray diffractometry. The analysis was performed in a Rikaku TTR-III diffractometer at room temperature, with Cu $K\alpha$ radiation at a scan speed of $0.2^\circ/\text{s}$. The XRD patterns were recorded in the 2θ range of $10^\circ\text{--}90^\circ$. Loss of ignition was determined at 750°C according to the method suggested by ASTM D5142-02a (Arthur 2004). The specific surface area was determined by surface area analyzer (Micromeritics ASAP 2000, USA).

Results and discussion

Soil characteristics

Selected physical and chemical characteristics of the soil used in this study are shown in Table 2. The soil was acidic with pH value of 4.79 ± 0.03 . The EC value was $440 \pm 7 \mu\text{S}/\text{cm}$. The organic matter was $78.71 \pm 0.16 \text{ mg}/\text{kg}$. The soil presented a high phosphorus content, with TP reaching up to $2.48 \pm 0.03 \text{ mg}/\text{g}$. The SRP content was $24.12 \pm 0.32 \text{ mg}/\text{kg}$, which accounted for 0.97% of the total phosphorus content. The high SRP concentration suggests that without proper management, this soil can contribute to runoff phosphorus loss from this soil and pose a potential threat to the water quality of the nearby water bodies. The Bray-P content was $328 \pm 14 \text{ mg}/\text{kg}$. The Bray-P was used to evaluate the phosphate status of the soil for plant. The content of Bray-P in the soil can give farmers some advice on soil management and fertilization. The high Bray-P content reflects the long-term fertilizer or manure applications to the field.

Sand, clay, and silt fractions in the investigated soil were 34, 52, and 14%, respectively. The main fraction was clay, so the investigated soil was referred as clay soil. This soil is different from the previous investigated soil by Summers et al. (1993, 1996a, b, 2002), who used RM as the amendment to immobilize phosphorus on sandy soil. The sandy soil was barren and has low water holding capacity. So the soil phosphorus was hard to accumulate on the soil and was easy to loss when it was rainy. But the clay soil has the relative high absorption capacity for phosphorus; the phosphorus loss in clay soil was due to the excessive fertilization or manure applications in the soil.

Adsorption isotherm for phosphorus

RM is the residual of bauxite processing for aluminum extraction, the main constituents of RM are Fe_2O_3

Table 2 Physical and chemical properties of the soil

Parameter	Soil
pH	4.79 ± 0.03
EC ($\mu\text{S}/\text{cm}$)	440 ± 7
Moisture (%)	12.16 ± 0.23
Organic matter (mg/kg)	78.71 ± 0.16
SRP (mg/kg)	24.12 ± 0.32
Bray-P (mg/kg)	328 ± 14
TP (mg/g)	2.48 ± 0.03
Fe (mg/g)	32.77 ± 0.40
Al (mg/g)	38.65 ± 0.83
Ca (mg/g)	16.22 ± 0.25

(25.68%), A_2O_3 (10.98%), SiO_2 (12.45%), $\text{CaO} + \text{MgO}$ (13.88%), and Na_2O (5.76%). As several active constituents such as iron and aluminum oxides present in RM, RM has been one of the suitable materials for phosphate adsorption. To assess the immobilization potential of RM for phosphorus in soils, phosphorus adsorption capacity of RM from phosphorus solution was studied. Adsorption isotherm study was carried out by varying phosphorus concentrations from 1 to 350 mg/L . The adsorption data were fitted to Langmuir isotherm model. Adsorption parameters were calculated using the Langmuir equation:

$$q = \frac{q_{\max}}{1 + bC_e} bC_e \quad (3)$$

where q is the amount of the phosphorus adsorbed (mg/g), C_e is the final concentration of the phosphorus in solution (mg/L), $q_{\max}$ is the maximum adsorption capacity of adsorbent (mg/g), b is a constant. The values of the parameters of the Langmuir isotherm model and the correlation coefficients (R^2) were listed in Fig. 2. From the Fig. 2, it can be seen that the maximum adsorption capacity ($q_{\max}$) reached 14.47 mg/g .

Batch leaching experiments

To optimize the mass ratio of RM to soil and evaluate the RM amendment on soil phosphorus immobilization, soils amended with RM at mass ratio of 1, 3, 5, 7, and 10% were investigated by batch leaching experiments. The results were shown in Fig. 3. It is clearly shown in Fig. 3 that RM amendment significantly reduced phosphorus release from the soil samples. In the un-amended soil sample, the phosphorus release was 14.38 mg/kg , whereas when the soil samples were amended with 1, 3, 5, 7, and 10% RM, the phosphorus release was reduced to 2.56, 1.82, 1.40, 0.45, and 0.23 mg/kg , respectively. There was a general trend of decreasing phosphorus release with increasing amendment ratio. However, the increasing RM dosage will

Fig. 2 Langmuir isotherm

Fig. 4 Effect of amendment dosages on soil pH

Fig. 3 Effect of amendment dosages on phosphorus release

result in an increase in soil pH due to the alkalinity of red mud. The pH of amended soil should be considered. The pH values of amended soil were shown in Fig. 4. It was found in Fig. 4 that pH of un-amended soil was 4.79, whereas the pH of amended soil increased. The pH of amended soil was 7.04 at RM dosage of 1%. When RM dosage was 3%, the pH of amended soil reached 8.33, which is not within the accepted limit (between 6.5 and 7.5) for plants growth. As a compromise between the immobilization efficiency and plants growth, an application ratio of 1% was employed. When RM amendment dosage is 1%, RM amendment reduced the phosphorus release from 14.38 to 2.56 mg/kg. The immobilization efficiency of RM was 75%. The reduction in soluble phosphorus from the RM amended soil is possibly due to the following two reasons: the first one is that RM has the large specific surface area, which could absorb phosphorus onto the surface. The second one is that RM contains high iron,

aluminum, and calcium content, which specifically adsorbed phosphorus onto the surface of iron and aluminum oxides and formed precipitation of iron, aluminum, calcium, and phosphorus compounds (Laiti et al. 1996; Khare et al. 2005). So the phosphorus release from the RM amended soil was reduced. Similar results have been reported by Summers et al. (1993, 1996a, b, 2001, 2002, Vlahos et al. (1989), Ward and Summers (1993) and Snars et al. (1993), who found that RM can reduce phosphorus leaching from sandy soils.

Column leaching test

Based on the batch leaching experiment results, a RM/soil ratio of 1% was used in the following column leaching experiments. The use of column leaching experiments, usually performed to better approximate field conditions, may provide information that is not available from batch leaching experiments and test approximate natural conditions closer than any other laboratory tested (Miretzky et al. 2006; Kalbe et al. 2007). The phosphorus concentrations in the leachate of un-amended and amended soil columns were shown in Fig. 5. It can be seen from Fig. 5 that phosphorus concentration in the leachate from amended soil was lower than that from the un-amended soil. The phosphorus concentration in the leachate from un-amended soil was 2.32 mg/L after 1.0 pore volume and corresponding phosphorus concentrations after 2.0, 3.0, 4.0 and 16.0 pore volumes were 3.74, 4.06, 3.18, and 2.29 mg/L, respectively, while the phosphorus concentration in the leachate from RM amended soil was reduced to 1.39 mg/L after 1.0 pore volume and corresponding phosphorus concentrations after 2.0, 3.0, 4.0, and 16.0 pore volumes were reduced to 2.03, 2.23, 1.18, and 1.32 mg/L, respectively. The total amount of phosphorus released from un-amended

Fig. 5 Phosphorus concentration in the leachate during the column leaching of amended and un-amended soil

soil after 16.0 pore volumes was 36.73 mg/kg, whereas the total amount of phosphorus released from amended soil after 16.0 pore volumes was only 18.79 mg/kg. Furthermore, the variation of phosphorus concentration in the leachate as a function of leaching events was similar. For the un-amended and RM amended soils, the concentrations of phosphorus in the first 3.0 pore volumes were increased and decreased as leaching events increased. These results indicated that the release of loosely adsorbed phosphorus was a fast reaction. However, less soluble phosphorus were more slowly but steadily released. During the investigated period, the concentration of phosphorus in the leachate of RM amended soil was rather lower than that of the un-amended soil, which indicated that RM can effectively reduce phosphorus leaching. According to Snars et al. (2003), Summers et al. (1996b), Peters and Basta (1996) and Parry (1998), the mechanism for phosphorus immobilization is due to the fact that RM contains large amounts of iron and aluminum oxides and other microcrystalline minerals which have a high capacity to adsorb phosphorus. The high adsorption capacity of RM has been recognized as a potential management tool for reducing phosphorus leaching loss from soils. The characteristics of low mobility and strong retention in RM amended soil suggest that phosphorus may be considered not susceptible to leaching from the soil and thus have low risk to cause eutrophication of nearby water bodies.

Effect of RM immobilization on soil phosphorus speciation

To identify the changes of phosphorus speciation resulting from RM immobilization, the phosphorus speciation of un-amended and amended soil samples was analyzed by

sequential chemical extraction experiments, which provided useful evidence for explaining the phosphorus immobilization. The phosphorus speciation in the sequential extraction experiments was referred to as H_2O -P, $NaHCO_3$ -P, $NaOH$ -P, HCl -P, and Residual-P. Among them, H_2O -P is designated as loosely bound phosphorus, the sum of H_2O -P and $NaHCO_3$ -P is designated as labile phosphorus (Huang et al. 2008b). $NaOH$ -P is considered to be amorphous and crystalline iron and aluminum phosphates (Sharpley 1996). HCl -P is considered to be calcium phosphate. Previous study has proved that phosphorus extracted in H_2O , $NaHCO_3$, $NaOH$, and HCl is increasingly stable or decreasingly susceptible to potential environmental loss (Dou et al. 2003).

The main phosphorus speciation in the un-amended soil was $NaOH$ -P (45.8%), followed by $NaHCO_3$ -P (22.3%), Residual-P (17.8%), HCl -P (7.3%) and H_2O -P (6.8%). The percentage of calcium bound phosphorus was only a minor constituent in the investigated soil. The reason is that under the acidic conditions, calcium bound phosphorus is unstable, so the percentage of calcium bound phosphorus was low. Most soil phosphorus was combined with iron and aluminum. This result agreed with the work of several other authors who found iron and aluminum to be the dominant soil components involved in phosphorus retention in acid soils (Williams et al. 1971; Mozaffari and Sims 1996).

With regard to RM amended soil, the phosphorus fraction pattern changed (Fig. 6). The percentage of $NaOH$ -P, Residual-P and HCl -P increased from 45.8, 17.8 and 7.3% to 51.4, 19.7 and 7.9%, respectively, whereas the percentage of H_2O -P and $NaHCO_3$ -P decreased from 6.8 and 22.3% to 3.4 and 17.7%, respectively. Based on these results, it can be concluded that loosely bound phosphorus was reduced and more stable bound phosphorus increased. The increase in percentage of $NaOH$ -P indicated that

Fig. 6 Phosphorus speciation in un-amended and amended soils

H₂O-P was retained by adsorption to form iron and aluminum bound phosphorus. Generally, it can be suggested that phosphorus immobilization by RM is due to the fact that RM transformed the labile phosphorus into more stable forms.

Conclusions

1. RM is an effective adsorbent for removal of phosphorus and the maximum adsorption capacity of RM was 14.47 mg/g.
2. RM amendment significantly reduced phosphorus release from the clay soil with excessive phosphorus. The mass ratio of RM to soil was optimized to be 1%. At this dosage, RM amendment reduced phosphorus release from 14.38 to 2.56 mg/kg and the immobilization efficiency of RM was 82%. Column leaching experiments showed that RM could reduce phosphorus leaching. During the investigated period, total phosphorus release was reduced from 36.73 to 18.79 mg/kg.
3. RM amendment changed phosphorus speciation of amended soil and transformed the labile phosphorus into more stable forms.

Acknowledgments Financial support provided by National Water Pollution Control and Management Technology Major Projects (under Grant No. 2009ZX07104-005), National High Technology R&D Program (863) from Ministry of Science Technology of China (under Grant No. 2009AA062906) the Public Industry Research for National Environmental Protection (under Grant No. 201009037) is gratefully acknowledged. The provision of red mud by Research Institute of Shandong Branch, Aluminum Corporation of China was greatly appreciated.

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