Immobilization of phosphorus in sewage sludge using inorganic amendments

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Abstract Sewage sludge contains large amount of water-soluble phosphorus, which is prone to runoff loss when sewage sludge is land applied. Phosphorus export from sewage sludge is an important cause for water eutrophication. Immobilization of phosphorus before sewage sludge land application seems necessary. In this study, three amendments including red mud (RM), lime and mixture of RM and lime were employed to immobilize phosphorus in fresh sewage sludge. It was found that phosphorus was effectively controlled by RM, lime and mixture of RM and lime. Sequential chemical extraction results indicate that the RM amendment transformed H_2O -P in the sewage sludge into the $NaOH$ -P, while the lime amendment transformed H_2O -P in the sewage sludge into the HCl -P. Such transformations in phosphorus fractions would have little influence on phosphorus availability but would retard and reduce potential phosphorus loss following sewage sludge was land applied.

Keywords Sewage sludge · Red mud · Lime · Phosphorus

Introduction

Population growth and urbanization resulted in generation of large amount of sewage. Sewage sludge is an inevitable end byproduct of sewage treatment. Treatment and disposal of sewage sludge are the most costly phase of sewage treatment (Huang et al. 2008b). It is reported that over 1.47 million tons of dewatered sewage sludge was produced in China in 2007 (Wang W et al. 2010), and the production is predicted to increase to 3.60 million tons in 2010 (Lee et al. 2006). The accumulation of sewage sludge from sewage treatment plants is of increasing concern.

The disposal methods of sewage sludge include landfill, incineration, dumping and land application (Zheng et al. 2006; Matthews 1992). Landfill may cause ground water contamination due to its leaching. Incineration is unpopular because of the high cost and environmental hazards involved. Dumping at sea has been prohibited in many countries including China. Land application of sewage sludge has become more popular because of the relative low cost and potential benefits of recycling plant nutrients and organic matter, as well as improving the physical properties and agricultural productivity of soils (Clapp et al. 1994).

Despite the relative low cost and the potential benefits, there is still concern about land application of sewage sludge. Ratios of nitrogen to phosphorus in sewage sludge are usually narrower than that required for crop growth (O'Connor et al. 2004). Land application of sewage sludge based on the nitrogen requirements of crops generally results in excessive phosphorus accumulation in soils. The excessive phosphorus accumulation in soils is not harmful to crops but could lead to phosphorus loss to ground water and contribute to eutrophication of ground water (Peterson et al. 1994; Correll 1998).

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There are many methods to control phosphorus loss. A promising method is the addition of chemical amendments. Soluble phosphorus in sewage sludge solution can react with aluminum (Al), iron (Fe) and calcium (Ca) to form insoluble phosphorus. Previous studies have shown that addition of materials containing these elements can reduce phosphorus solubility (Dou et al. 2003; Stout et al. 2000; Kalbasi and Karthikeyan 2004). A large number of different amendments have been proposed and tested for immobilization of phosphorus in sewage sludge and manures. It has been confirmed that ferrous sulfate, aluminum sulfate, calcium oxide, calcium carbonate, gypsum and nanoscale zero-valent iron were effective for immobilization of phosphorus in sewage sludge and manures for land application (Huang et al. 2007, 2008b; Huang and Shenker 2004; Logan and Burnham 1995a, b; Moore and Miller 1994; Li et al. 2007; Ippolito et al. 2003; Mahdy et al. 2009). Although some of these materials and techniques have been studied in laboratory, greenhouse and field, their continual use could depend on the cost and availability of the materials. Thus, there is an increasing need for cost-effective alternative amendment to reduce phosphorus loss.

Red mud (RM) is an industrial byproduct of bauxite with sodium hydroxide during aluminum extraction. Because of its relatively large surface area and high content of iron, aluminum, calcium, silica, titanium oxides and hydroxides, RM can be considered as a cost-effective material for phosphorus adsorption. 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; Liu et al. 2007; Huang et al. 2008a; Zhao et al. 2009). Further, Summers et al. showed that application of RM to soils can be very effective in increasing phosphorus retention in sandy soil catchments, reducing phosphorus leaching from sandy soils and improving pasture growth on sandy soils (Summers et al. 1993, 1996a, b; Vlahos et al. 1989).

Although the immobilization of phosphorus using RM amendment in soils has been successfully demonstrated in many studies (Summers et al. 1993, 1996a, b; Vlahos et al. 1989; Ho et al. 1989; McPharlin et al. 1994; Udeigwe et al. 2009; Anderson et al. 1995), there have been few reports on the investigation of immobilization of phosphorus in sewage sludge by RM. The objective of this study was to investigate the efficacy of RM to immobilize phosphorus in sewage sludge compared with that in lime and mixture of RM and lime (RM/lime = 2:1, w/w) amended sewage sludge. The utilization of RM for sewage sludge immobilization may substitute lime and minimize the costs of lime purchasing and landfilling. In addition, leaching experiments were conducted to evaluate the efficiency of RM, lime and RM/lime to immobilize phosphorus in sewage

sludge. Furthermore, sequential chemical extraction experiments were conducted to explain the mechanisms of immobilization of phosphorus in sewage sludge.

Materials and methods

Materials

Sewage sludge was obtained from Xiao Hongmen sewage treatment plant, located at Chaoyang District, Beijing. Lime was of analytical reagent grade. RM used in this study was obtained from Shandong Branch, Aluminum Corporation of China, the composition and properties of which were listed in Table 1.

Analytical methods

The pH and electric conductivity (EC) values of the sewage sludge were measured in slurry at a solid/solution ratio of 1:2.5 in accordance with International Soil Association Method. The moisture content of the sewage sludge was calculated according to the mass difference before and after the sewage sludge being dried in an oven at 105°C for 24 h (Zupancic et al. 2004). Total organic matter was estimated using the loss-on-ignition method (Ben-Dor and Banin 1989). For the measurement of total phosphorus concentration, the sewage sludge was microwave-assisted acid digested according to Walter et al. (1997) and the phosphorus concentration was analyzed by the phosphomolybdate blue method of Murphy and Riley (1962) on a UV/visible spectrophotometer (DR/4000 HACH, USA) at a wavelength of 700 nm. For the total element analysis, the sewage sludge was digested using concentrated nitric acid, hydrofluoric and perchloric acid according to

Table 1 Composition and properties of RM

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

Table 2 Sewage sludge amended with RM, lime, and RM/lime

	Sewage sludge (g)	RM (g)	Lime (g)	RM/lime (g)	DI water (ml)
Test A	3	0	0	0	10
Test B	3	0.15, 0.3, 0.45, 0.9	0	0	10
Test C	3	0	0.15, 0.3, 0.45, 0.9	0	10
Test D	3	0	0	0.15, 0.3, 0.45, 0.9	10

Sterckeman et al. (2006), then the total element concentrations were analyzed using an inductively coupled plasma atomic emission spectroscopy (ICP-AES, Prodigy).

Mineralogical constituents of RM were determined using X-ray diffractometry (ShimadzuXRD-6000 Japan). The analysis was performed in a Rikaku TTR-III diffractometer at room temperature, with Cu K α radiation at a scan speed range of 0.2°/s. The XRD patterns were recorded in the 2θ range of 10°–90°. Loss of ignition was determined at 750°C in accordance with ASTM D5142-02a (Arthur 2004). The pH of RM was measured in slurry at a solid/water ratio of 1:2.5 in accordance with International Soil Association Method. The specific surface area was determined using the N₂ BET method (Micromeritics ASAP2000).

Immobilization experiments

As for the immobilization experiments, 1,000 g of sewage sludge samples was thoroughly mixed with amendments in 2,500-ml polypropylene bottles with mass ratios of 5, 10, 15 and 30%. Sewage sludge samples without amendment were also incubated as controls. Un-amended and amended sewage sludge samples were capped and incubated at 25°C for 3 days. The pH values were measured at the completion of the incubation process. After the incubation process was completed, the samples were dried and milled to pass a 0.4-mm sieve and stored in closed dark bottles for further investigation.

Batch leaching experiments

In order to evaluate the efficiency of RM, lime and RM/lime for immobilization of phosphorus, un-amended and amended sewage sludge samples were leached with de-ionized water. Samples and amendments were weighed into 50-ml centrifuge tube. The dosages of samples and amendments were presented in Table 2. Samples and amendments were treated as the above immobilization experiment. At the completion of incubation, samples were agitated for 1 h with 10-ml de-ionized water; the tubes were then centrifuged at 10,000 rpm for 10 min. Un-filtered samples were collected for measurement of pH and EC. Filtered samples were collected for measurement of phosphorus concentrations.

Sequential leaching experiments

In order to evaluate the long time immobilization efficiency of each amendment, un-amended and amended sewage sludge samples were subjected to sequential leaching experiments using de-ionized water at a solid/liquid ratio of 1:20. 1 g of incubated samples and 20-ml de-ionized water were placed in the tubes, capped and agitated on a shaker for 24 h at 25°C, then centrifuged for 10 min at 10,000 rpm. The supernatants were filtered through 0.45- μ m membrane filter and collected for phosphorus analysis. After removal of the supernatants, 20-ml de-ionized water was added to the centrifuge tubes again, the next steps were the same as above. This leaching process was repeated 10 times. The leachate was analyzed by the phosphomolybdate blue method of Murphy and Riley (1962) on a UV/visible spectrophotometer (DR/4000 HACH, USA) at a wavelength of 700 nm.

Column leaching experiments

Based on the batch leaching experiment results, a schematic diagram of the columns 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. The samples of the incubated sewage sludge were packed in the columns and the de-ionized water by a pump at a flow ratio of 10 ml/h. Leachates were collected every 24 h for the measurement of phosphorus concentrations and the leaching experiments lasted for 7 days.

Fig. 1 Schematic layout of a single unit of the experimental columns

Sequential chemical extraction experiments

In order to analyze phosphorus speciation after immobilization, the incubated samples were also dried at 60°C and milled to pass a 0.4-mm sieve. Soil phosphorus was sequentially chemical extracted according to the modified procedure of Hedley's method (1982). A diagrammatic representation of this scheme is given in Fig. 2. The extraction was carried out in 50-ml centrifuge tubes. To each tube, 0.5 g of incubated sewage sludge was added and sequentially extracted with 30-ml de-ionized water, 0.5 M NaHCO₃ (pH 8.2), 0.1 M NaOH, and 1.0 M HCl each for 16 h. After each extraction step, the tubes were centrifuged at 10,000 rpm for 10 min and the supernatants were filtered through 0.45-μm membrane filter. Residual phosphorus was digested with H₂SO₄–H₂O₂ by microwave accelerated reaction system (CEM, USA). Total phosphorus was the sum of all above phosphorus. The concentration of phosphorus was analyzed by the phosphomolybdate blue method according to Murphy and Riley (1962) on a UV/visible spectrophotometer (DR/4000 HACH, USA) at a wavelength of 700 nm.

Results and discussion

Sewage sludge characteristics

The main physical and chemical properties of sewage sludge are presented in Table 3. As shown in the table, the

Fig. 2 Sequential chemical extraction scheme

Table 3 Selected properties of the sewage sludge

Parameter	Sewage sludge
pH	7.46 ± 0.18
EC (μS/cm)	1,158 ± 21
Moisture content (%)	83 ± 0.90
Total organic matter (g/kg)	497 ± 0.40
Total P (g/kg)	15.41 ± 0.17
Cu (mg/kg)	281 ± 43
Zn (mg/kg)	978 ± 16
K (mg/kg)	1,128 ± 58
Na (mg/kg)	1,880 ± 32
Fe (mg/kg)	4,784 ± 44
Mn (mg/kg)	154 ± 4.00

pH and EC values of sewage sludge were 7.46 ± 0.18 and 1,158 ± 24 μS/cm, respectively. In addition, sewage sludge presented high moisture content, reaching up to 83.0 ± 0.90%, while the corresponding total solids consisted of nearly 50% organic matter and the total organic matter value was 497 ± 0.40 g/kg. Furthermore, the total phosphorus content in the sewage sludge was 15.41 ± 0.17 g/kg, implying that sewage sludge would be a rich source of phosphorus when it was land applied.

Batch leaching experiments

Figure 3a presents pH trend of the amended sewage sludge. Treatment effected on pH varied according to the type of amendments. The pH values of un-amended and amended sewage sludge are shown in Fig. 3a. It was found that RM, lime and RM/lime amendments all raised the pH of amended sewage sludge. The pH value of un-amended sewage sludge was 7.13 ± 0.05, whereas the pH values of RM amended sewage sludge increased from 8.50 ± 0.21 to 11.38 ± 0.06. The pH values of lime and RM/lime amended sewage sludge increased from 12.21 ± 0.16 and 8.54 ± 0.35 to 12.37 ± 0.02 and 12.25 ± 0.01, respectively.

Figure 3b presents EC trends of the amended sewage sludge. The EC increased as a result of amendments addition. EC is usually measured because it reflects the salinity of the mixture and its suitability for plant growth. The EC value of un-amended sewage sludge was 1,042 ± 68 μS/cm, whereas the EC values of RM amended sewage sludge ranged from 2,716 ± 192 to 4,077 ± 63 μS/cm. The EC values of lime and RM/lime amended sewage sludge increased from 6,943 ± 132 and 3,215 ± 251 μS/cm to 7,936 ± 12 and 7,653 ± 164 μS/cm, respectively.

There was a general trend of decreasing phosphorus leaching with increasing amendment ratios (Fig. 3c). Phosphorus leached from the un-amended sewage sludge was 237 ± 22 mg/kg, while phosphorus leached from the

Fig. 3 **a** pH, **b** EC, and **c** phosphorus in the leachates of the un-amended and amended sewage sludge

30% RM amended sewage sludge was only 5 ± 0.8 mg/kg. The reduction in phosphorus leaching from the RM amended sewage sludge is possibly due to the following two reasons. One is that iron and aluminum oxides contained in RM have a high capacity to adsorb phosphorus. The adsorption by the oxides reduced the phosphorus leaching. The other is that phosphorus was precipitated by iron, aluminum and calcium to form stable iron, aluminum and calcium phosphorus compounds.

Fig. 4 The cumulative phosphorus leaching as a function of time in un-amended and amended sewage sludge

As did the RM treatment, phosphorus leaching in the lime amended sewage sludge decreased from 237 ± 22 to 0 ± 0 mg/kg. It appears that calcium and pH are the main factors reducing phosphorus solubility and its leaching in the lime amended sewage sludge. Dou et al. (2003) concluded that pH above 7.0 and high calcium content was able to immobilize phosphorus effectively. The reduction in phosphorus leaching from lime amended sewage sludge is possibly due to precipitation of insoluble Ca-phosphate compounds under alkaline conditions.

Phosphorus leaching from the RM/lime amended sewage sludge decreased from 237 ± 22 to 7 ± 1.2 mg/kg. Both lime and RM/lime amendments can immobilize phosphorus in sewage sludge, but pH is not within the accepted limit (between 6.5 and 9.5) for plants growth, so from the agricultural view, RM would be a better choice for immobilization of phosphorus.

Sequential leaching experiments

Phosphorus leaching from the un-amended and amended sewage sludge as a function of leaching time is shown in Fig. 4. The amount of phosphorus leaching from un-amended and amended sewage sludge decreased with sequential leaching. As expected, compared with the un-amended sewage sludge sample, amended sewage sludge samples leached significantly less phosphorus. When sewage sludge was amended with 10% RM, lime and RM/lime, the cumulative amount of phosphorus leaching was reduced from 1,225 to 333, 18 and 40 mg/kg, respectively.

The mechanism by which the cumulative phosphorus leaching is reduced maybe was the result of precipitation and adsorption reactions represented by the following possible reactions:

Column leaching experiments

A column leaching test was set up to evaluate the phosphorus leaching from the amended sewage sludge for land application. Phosphorus concentrations in the leachate of un-amended and amended sewage sludge columns are presented in Fig. 5. Phosphorus concentrations in the leachate of the un-amended and amended sewage sludge decreased over time as the more soluble forms of the phosphorus were flushed out of the columns. Phosphorus concentrations in the leachate from the amended sewage sludge columns were less than that in the leachate from the un-amended sewage sludge column. After 7-day continuous leaching, phosphorus concentration in the leachate from un-amended sewage sludge decreased to 2.47 mg/l, while phosphorus concentrations in the leachate from RM, lime and RM/lime amended sewage sludge decreased to 0.88, 0.50, 0.82 mg/l, respectively. These results show that RM, lime and RM/lime had a high capacity for the immobilization of phosphorus.

Sequential chemical extraction experiments

To identify the changes of phosphorus speciation resulted from the amendments added, the un-amended and

amended sewage sludge were analyzed by sequential chemical extraction experiments. This method provided useful information for explaining the immobilization of phosphorus. This information is useful to predict the transport of phosphorus in soil as well as to predict the likelihood of its bioavailability. The main phosphorus speciation was $\text{H}_2\text{O-P}$, $\text{NaHCO}_3\text{-P}$, NaOH-P , HCl-P and Residual-P. Among them, $\text{H}_2\text{O-P}$ is designated as loosely bound phosphorus, the sum of loosely bound phosphorus and $\text{NaHCO}_3\text{-P}$ is designated as labile phosphorus (Huang et al. 2008b). In the un-amended sewage sludge, the percentage of the loosely bound phosphorus and labile phosphorus was 3.35 and 19.41%, respectively. The large amounts of these fractions indicate that phosphorus in the un-amended sewage sludge is easy to leach and is available to plants. All immobilization procedures changed the phosphorus concentration and distribution among fractions. There was an apparent trend of total phosphorus decrease with increasing amendments addition. This trend is partially explained by dilution and does not necessarily reflect the immobilization process. To better evaluate phosphorus immobilization process, the relative phosphorus distribution among fractions in Fig. 6 was discussed next.

In un-amended sewage sludge sample, the percentage of $\text{H}_2\text{O-P}$, $\text{NaHCO}_3\text{-P}$, NaOH-P and HCl-P was 3.35, 16.06, 37.36 and 18.11%, respectively. After RM immobilization, the phosphorus distribution among fractions changed. The percentage of $\text{H}_2\text{O-P}$ and HCl-P decreased from 3.35 and 18.11% to 1.42 and 5.28%, respectively, whereas the percentage of NaOH-P increased from 37.36 to 51.10%. The NaOH-P fraction is considered to be amorphous and crystalline Fe and Al phosphates (Hedley et al. 1982; Sharpley 1996). The above results indicate that loosely bound phosphorus was retained by absorption to newly formed Fe and Al hydroxides, while transformed $\text{H}_2\text{O-P}$

Fig. 5 Phosphorus concentrations in the leachate from the un-amended and amended sewage sludge

Fig. 6 Phosphorus speciation in the un-amended and amended sewage sludge. S1 $\text{H}_2\text{O-P}$, S2 $\text{NaHCO}_3\text{-P}$, S3 NaOH-P , S4 HCl-P , S5 Residual-P

into NaOH-P. Huang et al. (2008b) observed the similar result when the sewage sludge was amended with material containing Fe and Al elements. In the 10% lime amended sewage sludge, the percentage of H₂O-P and NaOH-P decreased from 3.35 and 37.36% to 0.036 and 8.18%, respectively, whereas the percentage of HCl-P increased from 18.11 to 37.70%. The HCl-P fraction is considered to be calcium phosphate. These phosphorus distribution changes indicate that loosely bound phosphorus and Fe and Al phosphate were transformed to calcium phosphate by addition of lime. Similar result has been reported by Huang and Shenker (2004), who confirmed this transformation by SEM-EDXS. In the 10% RM/lime amended sewage sludge sample, the percentage of H₂O-P, NaHCO₃-P, NaOH-P and HCl-P was 0.055, 12.70, 38.28 and 16.58%, respectively.

From the results obtained, it indicates that all amendments significantly changed the phosphorus speciation in the amended sewage sludge and transformed H₂O-P into more stable fractions.

Conclusion

1. RM, lime and RM/lime amendments were successfully used for immobilization of phosphorus in sewage sludge. But pH of lime and RM/lime amended sewage sludge is not within the accepted limit (between 6.5 and 9.5) for plants growth. So from the agricultural view, RM would be a better choice for immobilization of phosphorus.
2. Phosphorus leaching from RM amended sewage sludge was reduced due to iron and aluminum oxides contained in RM that have a high capacity to adsorb phosphorus and phosphorus was precipitated by iron, aluminum and calcium to form stable iron, aluminum and calcium phosphorus compounds, while phosphorus leaching from the lime amended sewage sludge was reduced only due to precipitation of insoluble calcium phosphorus compound under alkaline conditions.
3. Sequential chemical extraction experiments showed that red mud amendment transformed H₂O-P in the sewage sludge into the NaOH-P, while the lime amendment transformed H₂O-P in the sewage sludge into the HCl-P.

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