Using Laterite Materials to Remove Phosphate in Water

Nguyen Thi Hang Nga Department of Water Resources Engineering Thuy Loi University 175 Tay Son street, Dong Da district Ha Noi city, Viet Nam

Abstract: Batch experiments of phosphate adsorption onto laterite materials located in Viet Nam was conducted to assess phosphate removal capacity in water using these materials. Results revealed that laterite materials effectively removed phosphate from water. Phosphate adsorption was affected by pH in solution. At equilibrium behavior with pH from 6.5-6.7, phosphate adsorption reached 1.8-1.9 mg P g⁻¹. They were 1.3-1.4 mg P g⁻¹ at solution pH 3 and 1.0-1.1 mg P g⁻¹ at solution pH 9. The presence of other anions, e.g., sulfates and carbonate decreased phosphate adsorption efficiency. However, as adsorption competition occurred between phosphate, sulfate and carbonate in mixed solution, laterite preferred phosphate than selectivity The order of adsorption others. was phosphate>sulfate>carbonate.

Key words: Adsorption, phosphorous, laterite, water treatment

I. INTRODUCTION

Phosphorus (P) is an essential nutrient for the growth of organisms, but excessive amount of phosphorus in water bodies can also cause eutrophication resulting in the bloom of aquatic plants, growth of algae and depletion of dissolved oxygen in water and hence deteriorate water quality. Phosphorus is released into aquatic environments in many ways including industrial, agricultural, mining activities and mineralization of natural forest coverage. Although phosphorus contamination in aqueous environment has attracted increasing interests recently because of damages to ecosystem, loads of phosphorus discharged into environment has been increasing. Therefore, phosphorous removal from water is an urgent demand for water quality improvement and environmental protection.

In recent years, several techniques for phosphorus removal from wastewater have been developed and attracted much attention. The methods including adsorption, physical settling and filtration process, chemical precipitation with aluminum and iron compounds, and biological processes that rely on biomass growth of bacteria, algae, plants or intracellular bacterial polyphosphates accumulation are mentioned in a review by Bashana and Basan (2004). Finding advanced adsorbents to bring about high efficiency for treatment process is gaining increasing interest of scientists. Several low-cost and natural materials such as clays, waste materials and by-products such as montmorilonite, kaolinite, illite (Edzwald et al., 1976), palygorskite (Ye et al., 2005; Cheng and Sheng, 2006), La(III)-chelex resin (Wu et al., 2007), dry iron oxide tailings (Zeng, 2009) fly ash (Cheng et al., 2007), blast furnace slag (Oguz, 2004), hydroxides sludge (Golder et al., 2006), activated aluminum oxide and ferric hydroxide

(Genz *et al.*, 2004), have all been investigated as effective adsorbents to remove phosphorous in water. Despite the abundance of literature on phosphate adsorption capacity of clay minerals and natural materials, only few studies are available for the ability of laterite.

Laterite is formed by deep weathering in tropical and subtropical environments. The heavy rainfall in these regions leaches out all soluble weathering products in such soils, leaving behind clay minerals, rutile, and hydrated Al and Fe oxides which have been known for their strong interaction with arsenate and phosphate ions in solution (Cornell and Schwertman, 2003). So far various studies have focused on applications of laterite for water treatment. Frederick (2008) investigated removal of arsenic from drinking water sources of laterite thanks to metal oxy-hydroxides of aluminum and iron in composition. Udoevo et al. (2010) indicated high absorption capacity of laterite for aqueous heavy metals. Lateritic soils have been used as effective sorbents for arsenic removal from groundwater (Majiet et al., 2006; Rahman et al., 2008; Maiti et al., 2010). Nga et al. (2013) indicated good performance of laterite materials collected from Viet Nam in removal of suspended clay particles from water.

Instead of using synthetic compounds of high cost and toxicity to environment to remove pollutants from water, development of natural adsorbents of low cost should be paid attention. Laterite might be an alternative material which is effective and environmentally friendly. In Viet Nam, laterite is abundant and distributed in many regions due to strong lateritirization, but their applications to water treatment have been very few. The study aims demonstrate application of laterite materials from Viet Nam to water treatment.

II. MATERIALS AND METHODS

1. Materials

The present study used three laterite materials (1-TT, 2-HL and 3-QT) collected from mountain zones in Viet Nam as adsorbents. The 1-TT sample was collected from a layer at a depth of 1.5 m in Thach That town where completely weathered laterite distributes. The original sample is a hard block with dark yellow color. The 2-HL sample was found in excavated soil at a construction site. It is a hard block containing red solid gravels. The 3-QT sample was taken from a laterite profile at a depth of 0.5 m from the surface. The initial raw samples were washed with tap water and airdried at a room temperature. The fraction that passed through a 2-mm screen after gentle crushing was used as adsorbents.

The phosphate stock solution containing 10 mmol L^{-1} was prepared by dissolving pure chemical of sodium dihydrogen orthophosphate (NaH₂PO₄) in distilled water. Phosphate working solutions of different concentrations were prepared by diluting the phosphate stock solution in 0.005M MgCl₂ solution for adjusting ionic strength. The pH value of the phosphate working solution was measured before and after adsorption experiments.

Table 1. Some characteristics of laterite samples

N-		TOC	Clay	Silt	Fine sand	Coarse sand	ECEC
<u>No</u>	<u>рН</u> 5.0	(%)	(%)	(%)	(%)	(%)	(cmol _c /kg)
TT	3	0.18	67.42	6.29	7.71	18.58	1.36
2-							
Н	4.9						
L	5	0.13	57.03	6.24	9.89	26.83	1.00
3-							
Q	5.1				24.6		
Т	8	0.13	30.93	8.04	5	36.38	0.77

2. Mineralogy and surface charge analysis of laterite Mineralogy of the laterite samples were analyzed for four particle size fractions (clay, silt, fine sand and coarse sand) by X-ray diffraction (RINT-2000V, Rigaku) with a Cu K_{α} radiation at 40 kV and 20 mA following the procedure of Nga *et al.* (2013).

Surface charge of laterite samples was determined by serial potentiometric titration method. Five gram portion of the sample was equilibrated with NaCl solutions having different pHs for 4 h ationic strengths of 0.005 mol L⁻¹, 0.05 mol L⁻¹ or 0.01 mol L⁻¹. Proton and hydroxide ion adsorption by laterite was calculated from pHs before and after equilibration and plotted against pH. The pHpzse were determined from the intersection point of the curves for different ionic strengths. The net surface charge was set to zero at pHpzse.

3. Thermal analysis

Differential thermal and gravimetric analysis (DTA-TG) was carried out simultaneously using Rigaku thermo plus EVO. The samples were heated from room temperature to1000°C at a heating rate of 10 °C min⁻¹. The amount of sample used in each test was 10mg

4. Phosphorous adsorption experiments

To construct adsorption isotherm, 0.3 g of laterite and 50 mL of phosphate working solution at concentrations of 0; 0.1; 0.2; 0.3; 0.4; 0.5; 0.6; 0.8; 1.0 mmol L⁻¹were mixed in centrifuge tubes. The tubes were capped and placed on a shaker at 170 rpm for 24 h at room temperature to ensure equilibrium. The suspension was separated by centrifugation (2500 rpm, 15 min) and the supernatant was filtered through a 0.45 μ m membrane filter. The residual concentration of phosphate was determined by the molybdate blue spectrophotometric method. Each experiment was duplicated.

A similar procedure was adopted in the experiment on the effect of pH on phosphate adsorption. The fixed initial phosphate concentration of 0.5 mmol L^{-1} was employed and the pH was adjusted by 0.1M HCl and 0.1M NaOH to cover a range from 4 to 9. The pH measurement was implemented before shaking and after equilibration.

Phosphate adsorption kinetics was examined at 25°C and phosphate working solution at concentration of 0.5mmol L⁻¹. A 3 g portion of a laterite sample was added to 500 mL phosphate solution in a 1-L bottle and shaken at 170 rpm without pH adjustment. Several mL of equilibrium solution was sampled intervals between 0 and 24 h, immediately filtered through a 0.45 μ m membrane filter and then analyzed for P concentration.

The competitive adsorption experiments of phosphate was conducted with solutions containing 0.5 mmol L^{-1} sulfate, 0.5 mmol L^{-1} carbonate and 0.5 mmol L^{-1} phosphate, which were prepared from sodium sulfate, sodium carbonate and sodium dihydrogenate orthophosphate (NaH₂PO₄), respectively. Centrifuge tubes are used to contain 0.3 g of laterite and 50 mL of solution and then shaken for 24 h at room temperature. The solutions examined include single phosphate, complex of sulfate and phosphate and complex of phosphate, sulfate and carbonate.

The P concentration was measured after filtering through $0.045\mu m$. The analysis of phosphate was done by spectrophotometer using method described in standard methods of Kuo (1996). The experiments were done in triplicate and the results were averaged.

III. RESULTS AND DISCUSSION

1. Mineralogy and surface charge characteristics of laterite samples

X-ray diffraction patterns of the Mg-saturated and glycerolsolvated clay fractions and fine sand fractions of three laterite samples are shown in Fig 1.

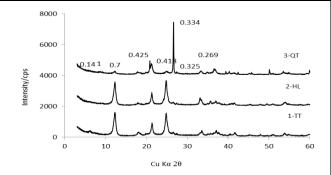


Fig 1. X-ray diffraction pattern of laterite samples.

The clay fractions of the 1-TT and 2-HL samples showed fairly strong peaks at 0.7, 0.418, 0.35 and 0.269 nm, indicating that these samples contain kaolinite and goethite and hematite. In addition, the 1-TT sample showed weak peak at 1.4 nm, indicating that it contain small amount of non-expandable 2:1 layer silicates. The weak peak at 0.487 nm indicates that all the samples contain gibbsite. The 3-QT sample showed a significantly different mineralogy in that it is dominated by quartz and contained much less amount of kaolinite and goethite. The dominance of 0.425 and 0.334 nm peaks in the X-ray diffraction patters for fine sand samples indicate that the major mineral in the fine sand fraction is quartz. The small but distinct peak at 0.418 and 2.69 nm shows that the fine sand fractions also contain goethite and hematite. The silt and coarse sand fractions exhibited similar X-ray patterns.

Basic chemical properties are summarized in Table 1. The pH_{H2O} was in a range from 5.3 to 5.6 and the pH_{KCI} was similar, indicating that the samples contained little exchangeable Al. The 1-TT sample showed the highest clay content of 67% followed by the 2-HL and 3-QT samples. The clay content of the 3-QT sample was only 30% and it had fairly high sand content. Although the clay content was high, the effective cation exchange capacity was very low. This is a reflection of the virtual lack in 2:1 type layer silicate minerals as indicated by X-ray diffraction (Fig. 1).

The results of thermal analyses are shown in Fig. 2. All the samples showed clear endothermic peaks centered at around 50, 320°C and corresponding weight losses. The 1-TT and 2-HL samples exhibited an additional large endothermic peak at 480°C but the 3-QT sample showed only a faint peak at that temperature. The endothermic peak at 50°C obviously came from desorption of adsorbed water. And the peaks at 320 and 480°C are assigned to dehydration reaction of hydroxide minerals and kaolinite, respectively. Since goethite dehydrates at lower temperature than gibbsite, the endothermic peak centered at around 320°C suggests that the contribution of gibbsite was larger. These results harmonize with X-ray diffraction patterns shown in Fig.1, particularly in that the 3-QT sample showed very weak diffraction peak at 0.7 nm. The 2-HL and 3-QT samples showed large endothermic peaks at around 840°C but they did not accompany weight loss. The origin of these endothermic peaks was not identified.

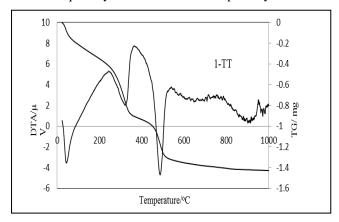
The dehydration reaction of gibbsite and kaolinite are expressed as

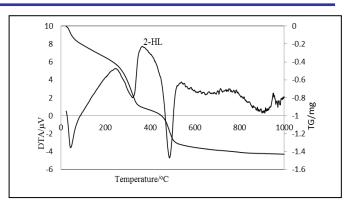
$$2Al(OH)_3 = Al_2O_3 + 3H_2O$$
 (1)

And

$$Si_4Al_4O_{10}(OH)_8 = SiO_2Al_2O_3 + 4H_2O$$
 (2)

The weight loss from 200 to 400°C was about 0.5 mg for all three samples. If this came from the dehydration of gibbsite, the stoichiometry in the reaction (1) gives the gibbsite weight of 1.4 mg in 10 mg sample. Similarly, the weight of kaolinite is estimated to be 1.4 mg for the 1-TT and 2-HL samples and 0.71 mg for the 3-QT sample. But actually the dehydration of both gibbsite and goethite occurs at around 300°C and it is difficult to quantify these two minerals separately.





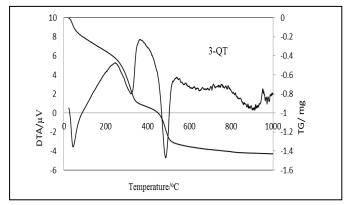


Fig.2. Results of differential thermal analysis and thermo gravimetric analysis.

Based on the mineralogical and chemical data, the approximate mineralogical composition of the laterite samples is summarized in Table 2.

Table 2. Mineralogy identification of laterite samples

Sample	Fraction	Identification				
1-TT	Clay	Mica; kaolinite; Geothite; feldspars				
	Silt	Kaolinite; Geothite; Quazt; Feldspars				
	Fine sand	Quazt; Geothite				
	Coarse sand	Kaolinite; Geothite; Quazt				
2-HL	Clay	Kaolinite; Geothite; Feldspars				
	Silt	Kaolinite; Geothite; Quazt; feldspars				
	Fine sand	Geothite; Quazt				
	Coarse sand	Kaolinite; Geothite; Quazt; hematite, Feldspars				
3-QT	Clay	Kaolinite; hematite; Quazt; Feldspars				
	Silt	Quazt				
	Fine sand	Quazt				
	Coarse sand	Geothite; Quazt				

2. Phosphate adsorption isotherm

The phosphate adsorption isotherms of the laterite samples are shown in Fig 3. The phosphate adsorption capacity increased with increasing equilibrium phosphate concentration from 0 to 1 mmol L^{-1} . The adsorption capacity is determined at equilibrium phosphate concentration of 0.5 mmol L^{-1} and at pH 5-6 which corresponding to

approximately 0.048 mmol g⁻¹, 0.045 mmol g⁻¹and 0.032 P mmol g⁻¹for 1-TT, 2-HL and 3-QT, respectively. The adsorption of phosphate onto laterite samples is believed to be complexation between surface hydroxyl groups of goethite, hematite and the phosphate ions (Seiki, *et al.*, 2003) as expressed, for example, by equation (3). With further increase of the phosphate concentration, the phosphate adsorption plateaued. As indicated by values of q_m (Table3), the 1-TT sample gave the highest adsorption efficiency followed by the 2-HL sample, and 3-QT sample. One possible reason for the observed lower phosphate adsorption is the low gibbsite content of the 3-QT sample. The Langmuir and Freundlich equations expressed in Eqs. (4) and (5) were used for evaluating the experimental data

Fe-OH+
$$HPO_4^{2-}$$
+H = Fe-OPO₃²⁻ +H₂O (3)

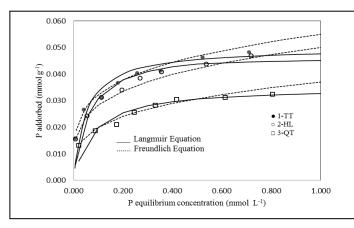
$$q_e = q_m b c_e / (1 + b c_e) \tag{4}$$

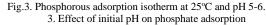
$$q_e = K_f c_e^{1/n} \tag{5}$$

where c_e (mg L⁻¹) and q_e (mg g⁻¹) are the equilibrium phosphorus concentrations in the aqueous and solid phases. Here, q_m (mg g⁻¹) is the maximum adsorption capacity and *b* is the affinity parameter. K_{fi} and 1/n are the Freundlich adsorption constant and *n* is interpreted as a measure of adsorption intensity. The constants determined by least squares method are summarized in Table 3. The regression coefficients (R^2) are also shown in Table 3. The R^2 values obtained for the Freundlich and Langmuir isotherms were both above 0.976, indicating that both models can be used to describe the adsorption of phosphate onto laterite. The Langmuir isotherm appears to be slightly better suited to describe the equilibrium data.

 Table 3. Estimated isotherm parameters for phosphate adsorption

Sample	Langmuir Equation			Freundlich Equation		
	$q_m (\text{mg g}^{-1})$	b	R^2	Κ	1/n	R^2
1-TT	1.01	20.2	0.99 1	0.05 5	0.233	0.993
2-HL	0.84	17.16	0.99 1	0.05	0.246	0.994
3-QT	0.46	13.04	0.99 2	0.03	0.266	0.976





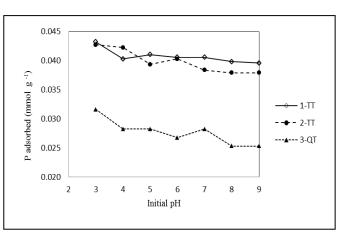


Fig 4. Effect of initial pH on phosphate adsorption by laterite materials. (P concentration 0.5mmolL⁻¹, contact time 24 h, adsorbent dose 6 g L⁻¹.)

At pH 3, phosphate adsorption was highest: approximately 0.034-0.045 mmol g⁻¹ at an equilibrium concentration of 0.5 mmol L⁻¹. The higher pH values lead to slight reduction in phosphate adsorption. Fig. 4 shows that phosphate adsorption by laterite is only slightly pH-dependent. Compared to the maximum amount of adsorbed phosphate at pH 3, less than 10% decrease was observed at pH 9. The adsorption is possibly hindered at high pHs, because both surface of laterite and phosphate carries more negative charges at higher pHs, which induces more intensive repulsion between the negatively charged surface and phosphate.

4 Adsorption kinetics

The phosphate adsorption kinetic was examined under conditions of room temperature 25° C, equilibrium concentration of 0.5 mmol L⁻¹ and pHs 5-6. The adsorption kinetic of phosphate on the three adsorbents performed a similar trend. Phosphate adsorbed onto laterite increased with time, significant increase was in first 8 h and reached equilibrium at about 24 h (Fig. 4).

Experimental data was evaluated by kinetic adsorption equations, Power function equation (6) and Elovich equation (7)

Power function equation:
$$q = at^{b}$$
 (6)

Simple Elovich equation: $q = a + b \ln t$ (7)

Where q is the amount adsorbed (mg g⁻¹) and t is the adsorption time. The other parameters are kinetic constants, which can be determined by regression of the experimental data. Since the power function gave low correlation coefficients (R^2) for the present experimental data (0.7) and this model was ruled out. On the other hand, the simple Elovich equation gave acceptable correlation coefficients (R^2) of 0.84; 0.82 and 0.7, for 1-TT, 2-HL and 3-QT, respectively. Therefore, the fitted curves by Elovich equation are overlain in Fig 4. The suitability of the simple Elovich equation was also reported by Ye*et al.* (2006) and Chen and Seng (2006).

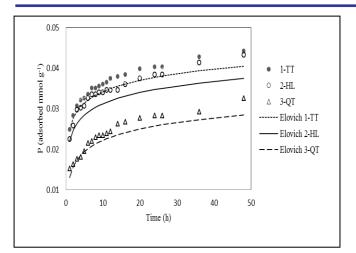


Fig 4. Phosphorous adsorption kinetic at 25°C and pH 5-6.

5. Competitive adsorption of phosphate

Table 5. Amount of Phosphorus adsorbed (mmol kg⁻¹) in mixed solution

mixed boldton								
	Samples			Comparison (%)				
Solution	1-TT	2-HL	3-QT	1-TT	2-HL	3-QT		
Phosphate only	40.32	38.38	28.23	100.00	100.00	100.00		
Phosphate and sulfate Phosphate, carbonate and	36.50	34.87	26.12	90.53	90.85	92.53		
sulfate	35.33	34.02	25.22	87.62	88.64	89.34		

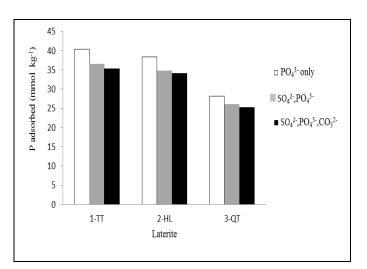


Fig. 5. Phosphorous adsorbed on laterite from single and complex solution. Single solution: NaH_2PO_4 (0.5 mmol). Complex solution: $NaHCO_3$ (0.5 mmol), Na_2SO_4 (0.5 mmol), NaH_2PO_4 (0.5 mmol)

Adsorption experiments were carried out separately with simple phosphate solutions, binary solutions of phosphate and sulfate, ternary solutions of phosphate, sulfate and carbonate. A fixed concentration of 0.5 mmol L^{-1} phosphate is mixed

with the same concentration of sulfate and carbonate anions. Amount of phosphate anion adsorbed onto laterite in the presence of sulfate and carbonate anions is presented in Table 5 and plotted in Fig.5. The adsorbed phosphate decreases when sulfate and carbonate anions presented in the solutions. The amount of phosphate adsorbed on laterite in mixed solutions was less than that in simple solutions (Table 5). However, the rate of phosphate adsorbed is still higher than the rate of sulfate and carbonate adsorption sonto laterite. Thus, the results of competitive adsorption experiments indicate that laterite could selectively adsorb phosphate ion and is suitable for the removal of phosphate from aqueous solution. In addition, experimental data shown in Table 5 also indicate the order of selectivity of adsorption of anions is phosphate > sulfat e > carbonate.

IV. CONCLUSIONS

In this study, three laterite samples distributed in different regions in Viet Nam were examined for the removal of phosphate ions from aqueous solutions. The adsorption isotherms, kinetics, pH effect, competitive adsorption and equilibrium time of phosphate onto laterite were investigated. The combined effects of surface group of goethite and zero point charge determined the adsorption capacity for phosphate. 1-TT performed the highest adsorption capacity $(0.045 \text{ mmol g}^{-1})$, followed by 2-HL $(0.04 \text{ mmol g}^{-1})$ and 3-QT (0.035 mmol g⁻¹). The adsorption was rapid in first 8 h and slowly reached adsorption equilibrium in 24 h. The results of equilibrium adsorption data fitted to both the Freundlich and Langmuir models. The simple Elovich equation fairly accurately described the phosphate adsorption kinetics onto laterite. Phosphate adsorption of laterite was less pH-dependent and laterite could selectively adsorb phosphate ion in mixed solutions containing sulfate and carbonate. The natural laterite is a low cost adsorbent to remove phosphorous in water. However, laterites containing smaller amount of goethite, e.g., 3-QT sample, is not recommended for water treatment at high pHs

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