

The influences on the Phosphate Removal from Aqueous Solution by using red mud

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Abstract: This paper highlights the result obtained in the study of phosphate removal by heat modified red mud at 400°C in 1 hour. These results pointed that under the conditions of pH 7.0, heat modified red mud dose 10 g/L, contact time 4 hours, initial phosphate concentration 25 mg/L, shaking speed at 150 rpm in the batch way. The phosphate removal and adsorption capacity of heat modified red mud was found to be over 90% and 10.2 mg/g, respectively. The results obtained in this study also pointed that the adsorption isotherm and kinetic of phosphate was best described by Freundlich model and Pseudo second order with relative coefficient $R^2 > 0.99$. Therefore, heat modified red mud can be used as a low - cost and environmentally friendly material for contaminants removal in general, phosphate removal from an aqueous solution in particular.

Keywords: Red mud modification, phosphate removal, modified red mud.

1. INTRODUCTION

In the Bayer process, red mud is known formally as bauxite residue and considered as a harmful factor to environment due to the chemical composition of red mud. It found that red mud containing various oxides such as Fe₂O₃, Al₂O₃, SiO₂, TiO₂, Na₂O, CaO, SO₃, P₂O₅, Cr₂O₃, etc (the percentage of Fe₂O₃ and Al₂O₃ are estimated about 78.5%). However, the previous researches were conducted showed that these compositions can play an important role in removal of metal ions from aqueous solution such as Zn²⁺ [1], Pb²⁺ [2], Cd²⁺ [3], Co²⁺ [4], As⁵⁺ [5], etc. Besides, these oxides also can remove some nutrient elements causing of eutrophication such as PO₄³⁻ [6,7,8,9]. However, the results obtained in the previous studies were found to different due to the oxides content in red mud are strongly dependence on the characteristics of precursor bauxite ore, as well as on applied technological process, and consequently fluctuates strongly among samples generated in the different parts of the world (Tab 1).

Table 1: Chemical composition of red mud

Nations Elements (%Wt)	Vietnam [10]	Italy [11]	Spain [11]	UAS [11]
SiO ₂	2	18.6	4.4	8.5
Fe ₂ O ₃	15.9	15.2	37.5	35.5
Al ₂ O ₃	-	24.7	21.2	18.4
Al(OH) ₃	13.6	-	-	-
TiO ₂	-	11.45	4.97	6.31
CaTiSiO ₅	9	-	-	-
CaO	-	5.51	2.33	7.73
MgO		0.46	0.08	0.09
FeO(OH)	50.8	-	-	-
γ-FeO(OH)	3.8	-	-	-
K ₂ O	-	1.1	0.26	0.47
Na ₂ O	-	11.7	0.36	6.1

Vietnam is considered as one of the richest bauxite ore resource countries in the world, the total of bauxite ore capacity estimated about 5.5 billion tons and concentrates mostly in the Central Highland (91.4%). In Dak Nong and Lam Dong provinces, the total of bauxite ore is investigated to 4.3 billion tons [10]. At the moment, the biggest alumina plants in Vietnam are Nhan Co alumina (Dak Nong province) and Tan Rai alumina (Lam Dong province) emit approximately 1.2 million tons of dry red mud annually. According to the mass balance, the production of two tons of alumina creates from 2 to 4 tons of red mud or bauxite residue with various different component compared to red mud generated in the other countries such as Al(OH)₃, FeO(OH), γFeO(OH). Therefore, in the next 50 years, about 1.15 billion tons of red mud will be generated from alumina production in the Central Highland. It is really a huge pressure to environmental management in Vietnam in the future. Therefore, finding an effective solution for handling the amount of red mud generated is one of the leading concerns of the authorities in Vietnam at the present. The use of red mud for contaminant removal is being considered as a good way for implementation. This paper will introduce results obtained in the study on the phosphate removal

efficiency of heat modified red mud under the optimal conditions such as initial pH, contact time, modified red mud dose, initial phosphate concentration, shaking speed.

2. CHEMICALS AND METHODS

1. Red mud

Red mud was collected from Tan Rai Alumina Company, is located in Lam Dong province, Central Highland of Vietnam. The pH and surface area of red mud were measured to be 12.8 and 86 m²/g, respectively. 92.3% particle sizes of red mud less than 600 μm. XRD analysis detected that red mud contains the following mineral components: FeO(OH) (50.8% Wt), Fe₂O₃ (15.9% Wt), Al(OH)₃ (13.6% Wt), CaTiSiO₅ (9.0% Wt), CaCO₃ (4.9% Wt), γ-FeO(OH) (3.8% Wt), and SiO₂ (2% Wt). After pH neutralization by deionized water, red mud was modified by heat at 400°C within 1 hour by using Lenton furnace (1.9KW). After modification, heat modified red mud was used directly for PO₄³⁻ adsorption experiments.

2. Chemicals

All chemicals used in this study (HNO₃, NaOH, KH₂PO₄) were of analytical grade and produced by Merck (Germany). The phosphate working solution with the concentration range of 25.0 - 200 mg/L was prepared from KH₂PO₄ salt and diluted by deionized water. The initial pH of phosphate solution was adjusted by using NaOH and HNO₃ solutions

3. Experimental procedure

The phosphate adsorption experiments were conducted by batch technique at the room temperature. A fixed amount of the heat modified red mud (except the adsorbent dose experiments) was added into a series of the 100 mL flask containing 25 mL of PO₄³⁻ solution with the different of concentrations, initial pH, and shaken at 150 rpm. After finishing each adsorption experiments and phases separation by Universal 320R centrifuge (Hettich Zentrifugen) at 6000rpm in 5 minutes, the pH of aqueous solution was measured again to determine the equilibrium pH by using Orion 4 star pH meter (Thermo Scientific). The PO₄³⁻ concentration remains in the adsorbed solution was analyzed by SMWW 4500 – E method [12] and Lambda 35 UV/VIS Spectrometer (Perkin Elmer), The PO₄³⁻ removal efficiency is calculated based on the mass balance equation as follows.

$$H(\%) = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

Where:

C_o: Initial phosphate concentration (mg/L)

C_e: phosphate concentration in the adsorbed solution at the equilibrium (mg/L)

Besides the adsorption experiments, the blank sample was also carried out to ensure that no adsorption was taking place on the wall of the flask used.

4. Analytical methods

The change in chemical compositions and surface of the modified red mud before and after adsorption will be determined by X-ray Powder Diffraction (Siemens D5005, Germany) and SEM analysis methods (Nova Namo Scanning Electron Microscopy NEP 199). The weight reduction of red mud before and after heat and acid modification was determined by GH 252 analytical balance (A&D Instrument Company, England).

3. RESULTS AND DISCUSSIONS

1. Effect of initial pH on phosphate removal

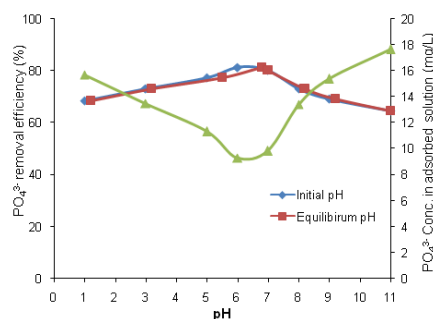


Fig 1: Effect of initial pH on phosphate adsorption (heat modified red mud dose 10g/L, temperature 25°C, shaking 150 rpm, initial phosphate concentration 50 mg/L, contact time 4 hours)

The effect of initial pH on phosphate removal by heat modified red mud is presented in Fig 1. It is easy to find the relation between initial pH and phosphate removal efficiency. When initial pH was 1; 3; 5; 6, the phosphate removal efficiency increased and reached maximum 82% at pH 6.0. After that phosphate decreased with the increase in pH. This trend similar behavior was also reported on phosphate from aqueous solution in the previous researches [6, 13, 14].

This trend occurred due to the competition for binding sites between cations and protons at low pH values. While at higher pH, solubility of complexes decreases

sufficiently allowing precipitation which may complicate the sorption process and do not bind to the sorption sites on the surface of the modified red mud [7].

As calculated and reported by **Kamiyango [15]**, the pH effects on both fraction compositions of phosphate species and charge development on the red mud surface. In the solution, phosphate exists under various forms such as $H_2PO_4^-$, H_3PO_4 , HPO_4^{2-} , and PO_4^{3-} (**Fig 2**), in the range of pH from 4.0 to 6.0, PO_4^{3-} is mainly exists in $H_2PO_4^-$, which form easy to metal oxides containing in the red mud with the binding energy is 135.2eV [16].

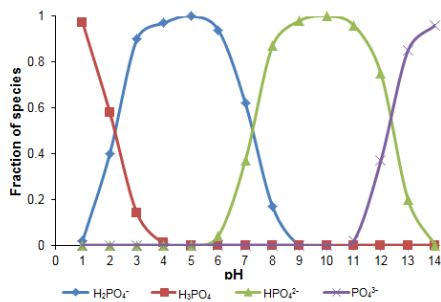
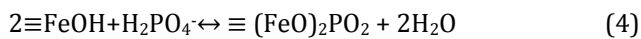
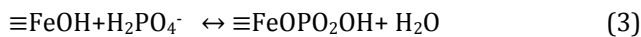
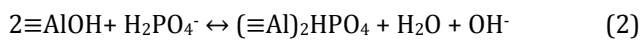


Fig 2: Fraction composition of phosphate species in the solution at different pH [15]

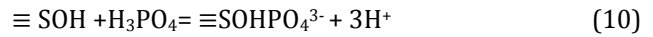
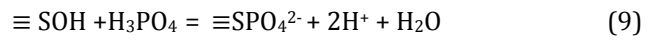
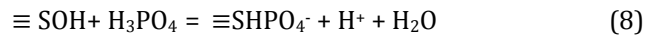
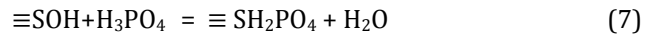
Kamiyango also indicated that the adsorption of phosphate on $Al(OH)_3$, $FeO(OH)$ is guided by the $\equiv AlOH$ and $\equiv FeOH$ group, located on crystal edges. When phosphate ion contact with $Al(OH)_3$ and $FeO(OH)$ will create the inner sphere complexes between phosphate and singly coordinated hydroxyl group. The ligand exchanged reaction mechanism is presented as equations following:



The same point with **Kamiyango, Liu [6]** also indicated that a ligand exchange mechanism took place in the sorption of phosphate onto the samples, and the sorption reaction may be written with the following ligand exchange reactions. When the red mud was added into the solution, it exists such surface hydrolysis reactions depending on pH of adsorbate solution.



When phosphate exists in the solution, then these sorption reactions take place depending on pH:



Where: $\equiv SOH$ represent sample surface of red mud.

When the pH increase, the amount of H_3PO_4 decreased and $H_2PO_4^-$ and HPO_4^{2-} were the predominant aqueous species. In the study of phosphate-metal precipitation reaction seems to be an alternative explanation for phosphate adsorption efficiency [17], which could be presented as the equation:



Where: "M" is the metal components, such as Al, Fe, Ca, and Si. Yang also pointed that monovalent dihydrogen phosphate ($H_2PO_4^-$) was well adsorbed by red mud, this idea is the same with [15].

2. Effect of contact time

Contact time is one of the most effective factors in batch adsorption process. Adsorption rate initially increased rapidly, and the optimal removal efficiency 82% was reached after 4 hours (**Fig 3**). Further, increase in contact time did not show significant change in equilibrium concentration; that is, the adsorption phase reached equilibrium due to the nature of adsorbent and the available adsorption sites affect the rate of adsorption of phosphate. The mechanism of solute transferred to the solid includes diffusion through the fluid film around the adsorbent particle and diffusion through the pores to the internal adsorption sites. In the initial stages of adsorption of phosphate, the concentration gradient between the film and the available pore sites is large, and hence the rate of adsorption is faster. The rate of adsorption decreases in the later stages of the adsorption probably due to the slow pore diffusion of the solute ion into the bulk of the adsorbent [18].

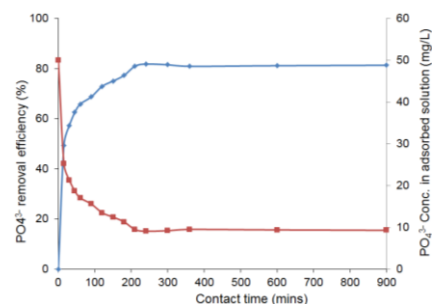


Fig 3: Effect of contact time on phosphate adsorption (Initial phosphate concentration 50mg/L, temperature 25°C, red mud dose 10g/L, initial pH 6.0, shaking 150rpm)

3. Effect of modified red mud dose

The effect of modified red mud dose was studied in the range of 2.0 - 20 g/L. It was observed that the adsorption percentage of phosphate ion onto the modified red mud increased rapidly with the increasing of adsorbent dose (Fig 4). This result is expected because the increase of adsorbent dose leads to greater surface area. When the adsorbent concentration was increased from 2 to 10 g/L, the percentage of phosphate ion adsorption increased from 42.2% to 82, respectively. At higher dosages, the equilibrium uptake of phosphate ion did not increase significantly with increasing modified red mud dosages. Such behavior is expected due to the saturation level attained during an adsorption process. This finding agrees with Koumanova [19]. For subsequent studies, a dose of 10 g/L of modified red mud was selected.

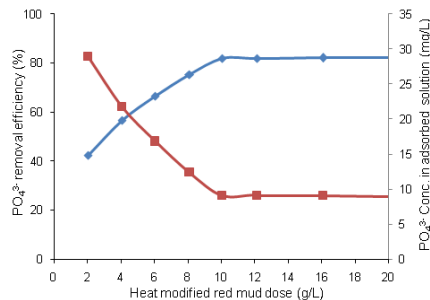


Fig 4: Effect of heat modified red mud dose on phosphate adsorption (initial pH 6.0, contact time 4h, temperature 25°C, shaking 150rpm, initial phosphate concentration 50mg/L)

4. Effect of initial phosphate concentration

The effect of initial adsorbate concentration on the adsorption was investigated by varying initial phosphate concentration from 25 to 200 mg/L. The experimental data were illustrated in Fig 5. The results indicated that the increase in the initial phosphate concentration led the removal efficiency of heat modified red mud decreased from 90.2% to 46.32%. The decrease can be explained by competition of the amount of phosphate to contact with the limited of adsorption sites on the surface of red mud.

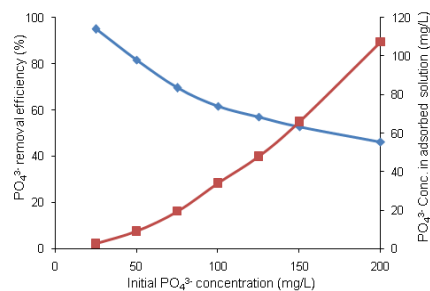


Fig 5: Effect of initial phosphate concentration (heat modified red mud dose 10g/L, initial pH 6.0, temperature 25°C, shaking 150rpm, contact time 4 hours)

5. Effect of shaking speed

In the batch model, the shaking speed directly effects on the phases movement between modified red mud and phosphate ion in the solution. The effect of shaking speed on the phosphate removal is given as Fig 6.

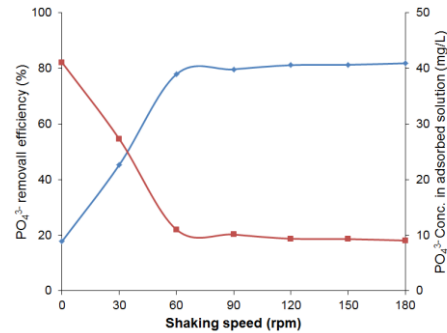


Fig 6: Effect of shaking speed on phosphate adsorption (initial pH 6.0, contact time 4h, temperature 25°C, shaking 150rpm, initial phosphate concentration 50mg/L, modified red mud dose 10g/L)

The results showed that the phosphate removal was not effective at low shaking speed (lower than 60rpm) due to only fine particle sizes of modified red mud suspended in the liquid phase, the large particle sizes sediment into the bottom. However, when shaking speed increased faster than 60rpm, both fine and large particle sizes of modified red mud will move together in the solution. Therefore, phosphate removal was constantly when shaking speed faster than 60rpm.

6. Adsorption isotherm

The linearized Langmuir adsorption isotherm equation which is valid for monolayer sorption onto a surface with finite number of identical sites is as follows:

$$\frac{t}{q_e} = \frac{1}{q_m} b C_e + \frac{t}{q_m} \quad (12)$$

Where:

C_e : the equilibrium concentration of phosphate in solution (mg/L),

q_e : the amount phosphate adsorbed at equilibrium (mg/g),

q_m : the theoretical maximum adsorption capacity (mg/g)

b : the Langmuir constant (L/mg).

The linear plot of $\frac{1}{C_e}$ versus $\frac{t}{q_e}$ with correlation coefficient R^2 over 0.98 for heat modified red mud

indicates the applicability of Langmuir adsorption isotherm (Fig 7). This indicates a monolayer sorption of phosphate onto the absorbent surface, the maximum adsorption capacity 'q_m' and binding energy constant 'b' of heat modified red mud for phosphate ion was 10.2 mg/g according to Langmuir model. This value is investigated higher than coir-pith activated carbon [20] or hydrotalcite-like compounds (HTLcs) [21] were 7.7 mg/g and 8.67 mg/g, respectively.

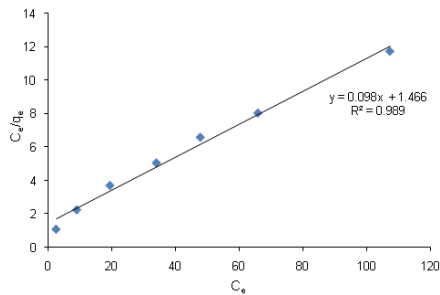


Fig 7: Langmuir isotherm of phosphate adsorption by heat modified red mud

Freundlich adsorption isotherm adopts multilayer adsorption on heterogeneous surfaces. Linearized form of the Freundlich equation is given by the following equation:

$$\ln q_e = \ln K_f + \frac{1}{n} C_e \quad (13)$$

Where:

q_e: the amount of phosphate ions adsorbed at equilibrium time (mg/g),

C_e: the equilibrium concentration of phosphate ions in the solution (mg/L),

K_f: the adsorption capacity (mg/g),

n: an empirical parameter,

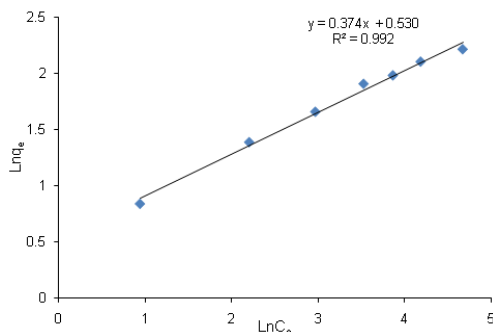


Fig 8: Freundlich isotherm of phosphate adsorption by heat modified red mud

The isotherm parameter of both Langmuir and Freundlich are shown in Table 2. According to

R² coefficient, it is easy to release that the phosphate adsorption followed both single and multi layer on the surface of red mud. However, the Freundlich isotherm provides a better fitting to isotherm than Langmuir isotherm due to the higher R². Therefore, it can be suggested that some heterogeneity in the surface of modified red mud play a role in the phosphate adsorption.

Table 2: Langmuir and Freundlich isotherm parameters

Langmuir isotherm		
q _{max} (mg g ⁻¹)	B	R ²
10.20	0.68	0,986
Freundlich isotherm		
N	K _f	R ²
2.67	1.45	0.992

7. Adsorption kinetics

The rate constant K₁ for the adsorption of phosphate was studied by Lagergren rate equation [21] for initial phosphate concentration of 50 mg/L.

Pseudo-first-order rate expression of Lagergren equation:

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.203} \quad (14)$$

Where:

q_e: The amount of phosphate adsorbed at the equilibrium (mg/g)

q_t: The amount of phosphate adsorbed at time 't' (min), respectively (mg/g)

K₁: The Pseudo-first-order rate constant (min⁻¹).

The K₁ and correlation coefficient R² were calculated from the slope of the linear plot of log(q_e - q_t) versus 't' at different time intervals. The K₁ and R² of heat modified red mud are shown in Table 3.

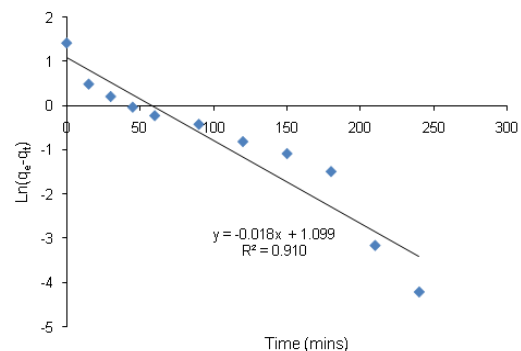


Fig 9: Pseudo first order of phosphate adsorption by heat modified red mud

The Pseudo-second-order rate expression is:

$$\frac{t}{q_t} = \frac{1}{K_2 * q_e^2} + \frac{t}{q_e} \quad (15)$$

Where: K_2 is the Pseudo-second-order rate constant (g/mg.min).

The calculation from the slope and intercept of the plot $\frac{t}{q_t}$ versus time 't' was almost linear shown in Fig 10.

The values of K_1 , K_2 and R^2 of heat modified red mud are shown in Table 3

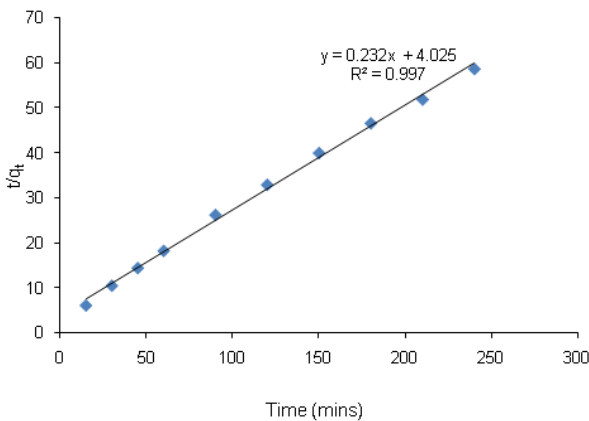


Fig 10: Pseudo second order of phosphate adsorption by heat modified red mud

Table 3: Kinetic paramter of phosphate adsorption by heat modified red mud

Pseudo first order			
K_1 (min ⁻¹)	q_e (mg/g)	R^2	q_e^* (mg/g)
0,018	3	0,91	4.09
Pseudo second order			
K_2 , g/mg.min	q_e , mg/g	R^2	q_e^* (mg/g)
0,014	4,31	0,997	4.09

The fitted curve and parameters of kinetic models are shown in Fig 9, Fig 10 and Table 3. It is clearly that the phosphate adsorption process on modified red mud is well described by the Pseudo second order model because of the higher correlation coefficient R^2 was 0.997 compared to R^2 was 0.91 by Pseudo first order. According to the results introduced above, it can be suggested that the phosphate adsorption by heat modified red mud due to the increase in area surface. After heat modification the area surface of red mud was measured about 100.68m²/g higher 13.73% compared to raw red mud (86.95m²/g), respectively. Besides, some chemical compositions of modified red mud were found to decrease in content.

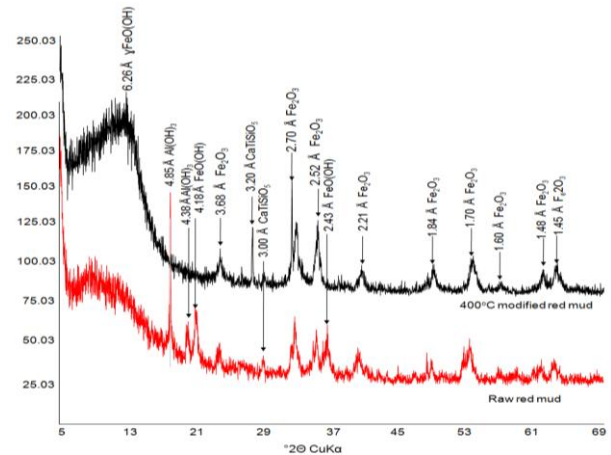


Fig 11: XRD analysis results of red mud before and after heat modification

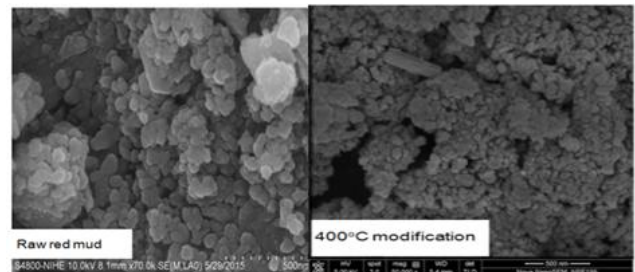


Fig 12: SEM images of red mud before and after heat modification

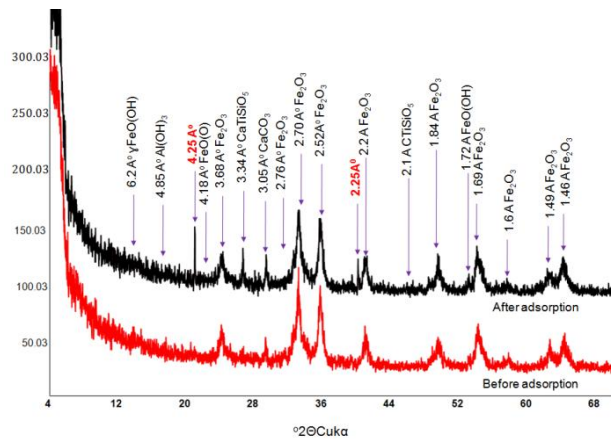


Fig 13: XRD analysis results of modified red mud before and after adsorption

As shown in Fig 13, γ FeO(OH), Al(OH)₃, FeO(OH), Fe₂O₃, CaTiSiO₅ were detected on the surface of modified red mud before and phosphate adsorption, two strange peaks (4.25A° and 2.25A°) appeared on the spectrum, which demonstrated that phosphate was successfully adsorbed onto the surface of modified red mud. Although the XRD's peak did not indicate exactly the form of phosphate appeared after adsorption, but based on Barbaux's report, phosphate could react with FeO⁺ and Al(OH)₃ by the precipitation and ion exchange

mechanism [22] or the formed phosphate complexes including $[\text{FeOPO}_4]_{\text{adsorption species}}$ and $[\text{Al}(\text{OH})_3\text{-PO}_4]_{\text{adsorption species}}$ could also adsorb additional H_3PO_4 analog by hydrogen bonding [23].

CONCLUSION

According to the results obtained in this study, some conclusions can be recommended as follows:

- Initial pH, initial phosphate concentration, contact time, modified red mud dose could influence the phosphate adsorption performance of 400°C modified red mud significantly.
- Heat modification enhances three times adsorption capacity of red mud compared to raw red mud.
- The kinetic and isotherm of phosphate adsorption onto the surface of 400°C modified red mud was well described by Pseudo second order and Langmuir–Freundlich isotherm, which demonstrated that the phosphate adsorption onto 400°C modified red mud could be governed by multiple mechanisms.

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