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# Arsenic removal from aqueous solutions by adsorption on red mud

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## Abstract

Use of red mud, which is a waste product from bauxite processing, has been explored as an alternate adsorbent for arsenic in this study. The tests showed that the alkaline aqueous medium (pH 9.5) favored the removal of As(III), whereas the pH range from 1.1 to 3.2 was effective for As(V) removal. The process of arsenic adsorption follows a first-order rate expression and obeys the Langmuir's model. It was found that the adsorption of As(III) was exothermic, whereas As(V) adsorption was endothermic. It would be advantageous to use this residue as an adsorbent replacing polyvalent metal salts. © 2000 Elsevier Science Ltd. All rights reserved.

*Keywords:* Arsenic adsorption; Red mud; Langmuir isotherm

## 1. Introduction

Although environmental restrictions and regulations have limited the production and uses of arsenic and its compounds, they are still extensively used in metallurgy, agriculture, forestry, electronics, pharmaceuticals and glass and ceramic industry, etc. Arsenic, being one of the more toxic pollutants, is introduced into the environment through weathering of rocks and mine tailings, industrial wastes discharges, fertilizers, agricultural employments of pesticides, smelting of metals and burning of fossil fuels.

Arsenic occurs in  $-3$ ,  $0$ ,  $+3$  and  $+5$  oxidation states in aquatic systems. The elemental state is extremely rare whereas  $-3$  oxidation state is found only at extremely reducing conditions. Arsenate species (pentavalent state) are stable in oxygenated waters. Under mildly reducing conditions, arsenites (trivalent state) predominate [1]. Arsenic combines strongly with carbon in arsenical organic compounds which are used as pesticides, chemotherapeutic agents and chemical warfare agents.

The presence of arsenic in water causes toxic and carcinogenic effects on human beings. It has been reported that long-term uptake of arsenic contaminated drinking water has produced gastrointestinal, skin, liver and nerve tissue injuries. The toxicity of arsenic firmly depends on its oxidation state and trivalent arsenic has

been reported to be more toxic than pentavalent and organic arsenicals [2].

The wastewaters from some industrial source such as gold, copper and zinc ore extraction, acid mine drainage and wood product preservation contain up to  $130 \text{ mg l}^{-1}$  soluble arsenic [3,4]. Also, potable waters in some parts of the world have been found to contain  $0.1\text{--}2 \text{ mg l}^{-1}$  arsenic [5,6]. The presence of arsenic in drinking water has been restricted to  $0.05 \text{ mg l}^{-1}$  [2].

Arsenic is commonly removed from aqueous solutions by coprecipitation with polyvalent metal hydroxide flocs such as iron(III) [7] and aluminum hydroxides [8,9].

The use of solid adsorbents in removing such pollutants from wastewater compares favorably with conventional precipitation or flocculation methods. For example, in some flocculation treatments, a large amount of salt must be added which introduces pollutants such as sulfate ions into the water. Moreover, the cost of the chemical reagents used in such treatments can limit their commercial application. Activated carbon [10], activated bauxite [10], activated alumina [10,11], amorphous aluminum hydroxide [12], amorphous iron (III) hydroxide [13], iron(III) hydroxide loaded coral limestone [14] and hematite [15] can be mentioned among the adsorbents studied for arsenic removal from aqueous solution.

Red mud is formed during the digestion in the Bayer Process which is practised for alumina production from bauxite. Mineralogically, red mud consists mainly of

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different forms of iron and aluminum oxide minerals, calcium and sodium aluminum silicates, various titanium compounds, etc. Oxidic constituents are the undissolved part of bauxite whereas silicates are formed from dissolved silica and alumina during desilication of aluminate liquors [16,17].

The purpose of the present study was to test red mud waste from alumina production as an alternate arsenic adsorbent. In this paper, the results of an investigation on the arsenic removing characteristics of red mud are described.

## 2. Experimental

Red mud used in the present study was obtained from Etibank Seydişehir Aluminium Plant, Konya, Turkey. Red mud slurry was taken from the outlet of washing thickeners. Wet sieve analysis showed that more than 95% of the solid particles are less than 53 µm.

The suspension was wet sieved through a 200 mesh screen. A little amount remained on the sieve and was discarded. Suspension having <math>-200</math> mesh particles was allowed to settle and decanted, the liquid fraction was discarded. The solid fraction was washed five times with distilled water by following the sequence of mixing, settling and decanting. The last suspension was filtered and the residual solid was then dried at 105°C, ground in a mortar and sieved through a 200 mesh sieve. The product was used in the study.

A sample was subjected to wet chemical analyses [18]. Mineralogical analyses was done by a Siemens D-5000 XRD equipment. The chemical and mineralogical compositions are given in Table 1.

Stock solutions containing 1 g As(III) l<sup>-1</sup> were prepared by dissolving 1.320 g As<sub>2</sub>O<sub>3</sub> (Merck 123) in 10 ml of 5 M NaOH and making up to 1 l with distilled water. Na<sub>2</sub>HAsO<sub>4</sub>·7H<sub>2</sub>O salt (Merck 6284) was dissolved in water for 1 g l<sup>-1</sup> As(V) stock solution. These stock

solutions were used to prepare experimental solutions of specified concentrations.

One gram of red mud powder was placed in a conical flask. Solutions containing 125 to 1500 µg arsenic and 5 ml of 0.1 M NaCl solution were made up to 50 ml using distilled water. Thus, solutions representing wastewaters in the concentration range 2.5–30 mg l<sup>-1</sup> were obtained. The solution was added to the powder in a flask. The initial pH value of the solutions was adjusted with either HCl or NaOH solutions the concentration of which are 0.01, 0.1 and 1.0 M. The flasks containing mixtures were capped tightly and immersed into the temperature controlled water bath and then shaken at the rate of 800±50 cycle/min with a mechanical shaker. At the end of the contact period, the mixture was then centrifuged for 10 min at 10 000 rpm and the final pH of the supernatants was measured. The solutions were analyzed spectrophotometrically, using silver diethyldithiocarbamate method [19] which is used to determine both arsenic species.

All chemicals used were of analytical reagent grade. All labware used in the experiments was soaked in diluted HCl solution for 12 h, washed and then rinsed four times with distilled water.

The experiments were performed in duplicate and the mean values were considered. In order to ascertain the reproducibility of results, a group of experiments were repeated a number of times and the results were found to vary within ±5%. The blank experiments showed no detectable As(III) and As(V) adsorbed on the walls of the flask.

## 3. Results and discussion

### 3.1. Effect of pH

Preliminary studies carried out at the original pH of mixtures (without acid or base addition) showed that the

Table 1  
Chemical and mineralogical compositions of the red mud

| Chemical composition           |         | Mineralogical composition |   |         |
|--------------------------------|---------|---------------------------|---|---------|
| Constituent                    | % (w/w) | Minerals                  | Formula   | % (w/w) |
| Al <sub>2</sub> O <sub>3</sub> | 20.39   | Sodalite                  | Na <sub>2</sub> O·Al <sub>2</sub> O <sub>3</sub> ·1.68 SiO <sub>2</sub> ·1.73H <sub>2</sub> O           | 32.30   |
| CaO                            | 2.23    | Cancriinite               | 3NaAlSiO <sub>4</sub> ·NaOH   | 4.60    |
| Fe <sub>2</sub> O <sub>3</sub> | 36.94   | Hematite                  | Fe <sub>2</sub> O <sub>3</sub>  | 34.90   |
| Na <sub>2</sub> O              | 10.10   | Diaspore                  | AlO(OH)   | 2.50    |
| SiO <sub>2</sub>               | 15.74   | Rutile                    | TiO <sub>2</sub>  | 1.50    |
| TiO <sub>2</sub>               | 4.98    | Calcite                   | CaCO <sub>3</sub>   | 1.20    |
| P <sub>2</sub> O <sub>5</sub>  | 0.50    |                           |   |         |
| V <sub>2</sub> O <sub>5</sub>  | 0.05    | Minor minerals:           | Bayerite: Al(OH) <sub>3</sub> ; Boehmite: AlOOH; Quartz: α-SiO <sub>2</sub> ;                           |         |
| CO <sub>2</sub>                | 2.04    |                           | Anatase: TiO <sub>2</sub> ; Kaolinite: Al <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub> |         |
| S                              | 0.08    |                           |   |         |
| L.O.I. (900°C)                 | 8.19    |                           |   |         |



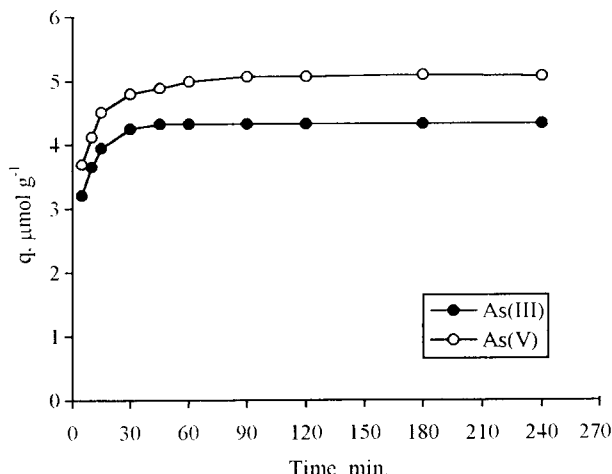


Fig. 2. Effect of contact time on the adsorption of As(III) and As(V) by red mud (initial concn.:  $133.5 \mu\text{mol l}^{-1}$ ; pH: 9.5 for As(III) and 3.2 for As(V); red mud dosage:  $20 \text{ g l}^{-1}$ ; temperature:  $25^\circ\text{C}$ ).

within 45 and 90 min, respectively. Data obtained in this study were fitted in the following first order rate expression of Lagergren (Fig. 3):

$$\log(q_e - q) = \log q_e - (K_{\text{ad}} \cdot t) / 2.303 \quad (2)$$

where  $q_e$  and  $q$  are the amounts of arsenic adsorbed at the equilibrium and at any time  $t$  and  $k_{\text{ad}}$  is adsorption rate constant. Linear plots of  $\log(q_e - q)$  vs  $t$  indicate the applicability of Eq. (2).

The  $k_{\text{ad}}$  values, calculated from the slopes of the lines in Fig. 3, are  $0.109$  and  $0.049 \text{ min}^{-1}$  for As(III) and As(V), respectively.

### 3.3. Adsorption isotherms and thermodynamic parameters

The adsorptions of As(III) and As(V) were found to be concentration dependent. It can be calculated from isotherm data that the amount adsorbed increased from  $1.35$  to  $7.46 \mu\text{mol g}^{-1}$  for As(III) and from  $1.54$  to  $6.41 \mu\text{mol g}^{-1}$  for As(V) in the initial concentration range of  $33.4$ – $400.4 \mu\text{mol l}^{-1}$  at  $25^\circ\text{C}$ . The removal percentages calculated were  $80.6$ – $37.3$  and  $92.2$ – $32.0$  for As(III) and As(V), respectively. The experimental data obtained under these conditions were applied to linearized forms of Langmuir, Freundlich, Frumkin and Temkin isotherms [Eqs. (3)–(6), respectively] which are suitable for evaluation of adsorption.

$$C_e/q_e = 1/(bQ^\circ) + C_e/Q^\circ \quad (3)$$

$$\ln q_e = \ln b + n \ln C_e \quad (4)$$

$$q_e = (Q^\circ/2D) \ln[b(Q^\circ - 1)] + (Q^\circ/2D) \ln(C_e/q_e) \quad (5)$$

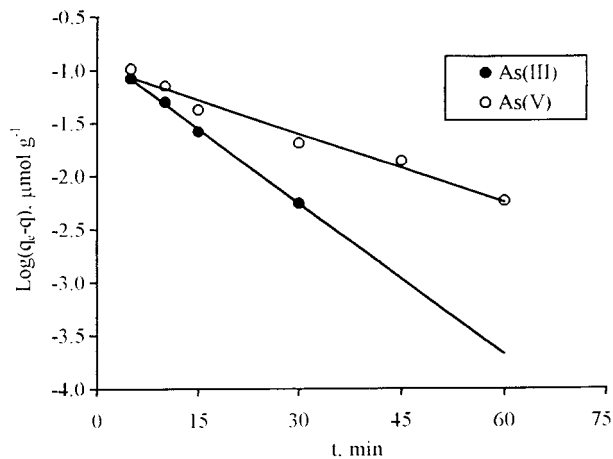


Fig. 3. Lagergren plots for As(III) and As(V) adsorption by red mud (initial concn.:  $133.5 \mu\text{mol l}^{-1}$ ; equilibration time: 45 min for As(III) and 90 min for As(V); pH: 9.5 for As(III) and 3.2 for As(V); red mud dosage:  $20 \text{ g l}^{-1}$ ; temperature:  $25^\circ\text{C}$ ).

$$q_e = n \ln b + n \ln C_e \quad (6)$$

where  $C_e$  is equilibrium concentration ( $\mu\text{mol l}^{-1}$ ),  $q_e$  is amount adsorbed at equilibrium ( $\mu\text{mol g}^{-1}$ ),  $Q^\circ$ ,  $b$ ,  $n$  and  $D$  are isotherm constants. The values of  $Q^\circ$ , which is adsorption maxima or adsorption capacity ( $\mu\text{mol g}^{-1}$ ) in Eqs. (3) and (5), can be compared with each other, whereas the definitions of  $b$ ,  $n$  and  $D$  are different for the various models.

All these isotherms were fitted to the adsorption data obtained. Calculated correlation coefficients for these isotherms by using linear regression procedure for As(III) and As(V) adsorption at different temperature are shown in Table 2. As seen, The Langmuir isotherm yielded best fits to the experimental data. Langmuir plots for the adsorption of As(III) and As(V) on red mud are shown in Fig. 4. The values of the Langmuir constants were calculated from slopes and intercepts of plots (Table 3).

It has been reported that the adsorption of As(III) by hematite [15], As(III) and As(V) by activated carbon, activated bauxite, activated alumina [10] and amorphous iron hydroxide [9], As(V) by amorphous aluminum hydroxide [12] follows Langmuir isotherm. Langmuir isotherm which leads the adsorption process indicates that the reaction is a reversible phenomenon [10] and the coverage is monolayer [10,15].

The remarkable removal of arsenic could not be achieved by red mud when compared with other separation techniques such as coprecipitation with aluminum and iron salts and adsorption by preformed aluminum and iron hydroxides. On the other hand, it can compete against the adsorbents such as hematite, activated bauxite, activated alumina and iron(III) hydroxide loaded coral lime stone (Fe-coral) which have limited effectivity. It has been reported that the maximum As(III) adsorp-

Table 2  
Comparison of adsorption isotherms for As(III) and As(V) adsorption by red mud at various temperatures

| Arsenic species | Temperature (°C) | Correlation coefficient for different isotherms, $r^2$ (%) |            |        |         |
|-----------------|------------------|--|------------|--------|---------|
|                 |                  | Langmuir   | Freundlich | Temkin | Frumkin |
| As(III)         | 25               | 99.43  | 95.86      | 97.06  | 91.94   |
|                 | 40               | 99.21  | 94.53      | 97.94  | 89.70   |
|                 | 55               | 98.84  | 95.23      | 97.23  | 89.27   |
|                 | 70               | 98.27  | 94.13      | 96.32  | 86.05   |
| As(V)           | 25               | 99.49  | 89.14      | 93.17  | 84.92   |
|                 | 40               | 99.89  | 86.18      | 94.79  | 85.34   |
|                 | 55               | 99.55  | 87.24      | 94.81  | 84.12   |
|                 | 70               | 99.84  | 84.96      | 94.80  | 81.90   |

tion capacity of these adsorbents are 2.63, 16, 14 and 0.17  $\mu\text{mol g}^{-1}$ , respectively. For As(V) adsorption by activated bauxite, activated alumina, activated carbon and the Fe-coral, the calculated corresponding values are 52, 67, 10 and 0.2  $\mu\text{mol g}^{-1}$  [10,14,15]. In the present study, As(III) and As(V) adsorption capacities of red mud at 25°C, estimated from Langmuir isotherm, are 8.86 and 6.86  $\mu\text{mol g}^{-1}$ , respectively. It is evident that red mud is more effective than hematite and Fe-coral. However, Fe-coral has an advantage compared to other adsorbents because it is effective in a wide pH range. In this connection, activated bauxite and activated alumina seem to be more advantageous adsorbents but the red mud is an attractive material in view of being inexpensive and a very fine material.

To determine if the arsenic adsorption process by red mud is favourable or unfavourable, for the Langmuir type adsorption process, the isotherm shape can be classified by a term “ $r$ ”, a dimensionless constant separation factor, which is defined as below [22–24].

$$r = 1/(1 + bC_0) \quad (7)$$

where  $r$  is a dimensionless separation factor,  $C_0$  is initial concentration ( $\mu\text{mol l}^{-1}$ ) and  $b$  is Langmuir constant ( $\text{l } \mu\text{mol}^{-1}$ ). The parameter  $r$  indicates the shape of the isotherm accordingly:

|             |              |
|-------------|--------------|
| $r > 1$     | Unfavorable  |
| $r = 1$     | Linear       |
| $0 < r < 1$ | Favorable    |
| $r = 0$     | Irreversible |

The  $r$  values for As(III) and As(V) adsorption can be calculated from Langmuir constants which are given in Table 3. For example, at 25°C and an initial concentration of 133.5  $\mu\text{mol l}^{-1}$ ,  $r$  values were calculated as 0.232 and 0.057 for As(III) and As(V), respectively. All calculated  $r$  values indicate that adsorption of As(III) and As(V) on red mud are favorable at all concentrations and temperatures studied. Also, it can be stated that the reversibility of As(V) adsorption is lower than that of As(III). The lower reversibility of As(V) adsorption by red mud suggests that the mechanism governing the process may be chemical adsorption.

Standard Gibbs free energy ( $\Delta G^\circ$ ), standard enthalpy ( $\Delta H^\circ$ ) and standard entropy changes ( $\Delta S^\circ$ ) for the adsorption process have been calculated from the Eqs. (8)–(10), respectively.

$$\ln(1/b) = \Delta G^\circ/RT \quad (8)$$

$$\ln b = \ln b_0 - \Delta H^\circ/RT \quad (9)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (10)$$

where  $b$  is Langmuir constant which is related to the energy of adsorption,  $b_0$  is a constant,  $R$  is an ideal gas constant (4.187 J mol $^{-1}$  K $^{-1}$ ) and  $T$  is temperature (K).

Calculated values of Langmuir parameters  $b$  and  $Q^\circ$  and the energy parameters  $\Delta G^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  are given

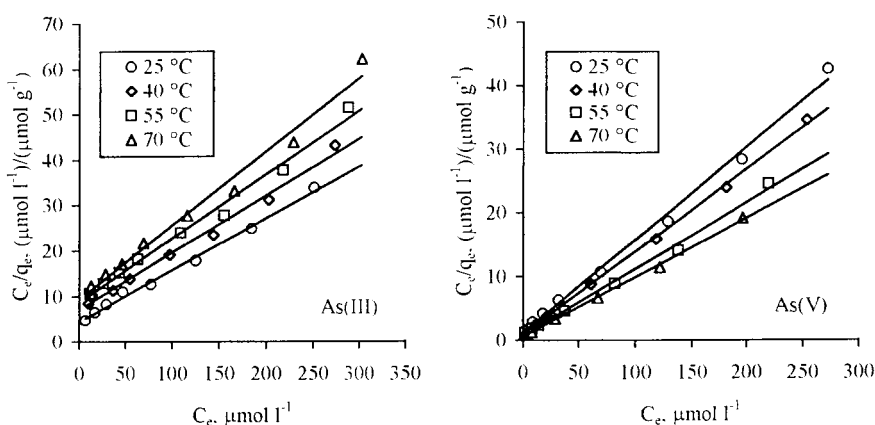


Fig. 4. Langmuir plots for As(III) and As(V) adsorption by red mud (initial concn.: varied from 33.4 to 400.4  $\mu\text{mol l}^{-1}$ ; contact time: 60 min for As(III) and 120 min for As(V); pH: 9.5 for As(III) and 3.2 for As(V); red mud dosage: 20 g  $\text{l}^{-1}$ ).

Table 3  
Calculated Langmuir constants and thermodynamic parameters at various temperatures for As(III) and As(V) adsorption by red mud

| Arsenic species | Temperature (°C) | Langmuir constants             |                                      | Thermodynamic parameters            |                                      |   |
|-----------------|------------------|--------------------------------|--------------------------------------|-------------------------------------|--------------------------------------|---|
|                 |                  | $b$ ( $l \mu\text{mol}^{-1}$ ) | $Q^\circ$ ( $\mu\text{mol g}^{-1}$ ) | $\Delta H$ ( $\text{kJ mol}^{-1}$ ) | $-\Delta G$ ( $\text{kJ mol}^{-1}$ ) | $\Delta S$ ( $\text{kJ mol}^{-1} \text{K}^{-1}$ ) |
| As(III)         | 25               | 0.025                          | 8.86                                 | -12.83                              | 25.11                                | 0.0412  |
|                 | 40               | 0.018                          | 7.93                                 |                                     | 25.58                                | 0.0407  |
|                 | 55               | 0.016                          | 7.17                                 |                                     | 26.41                                | 0.0415  |
|                 | 70               | 0.017                          | 6.18                                 |                                     | 27.81                                | 0.0438  |
| As(V)           | 25               | 0.123                          | 6.86                                 | 1.85                                | 29.06                                | 0.1037  |
|                 | 40               | 0.128                          | 7.73                                 |                                     | 30.62                                | 0.1034  |
|                 | 55               | 0.134                          | 9.60                                 |                                     | 32.22                                | 0.1038  |
|                 | 70               | 0.135                          | 10.80                                |                                     | 33.71                                | 0.1034  |

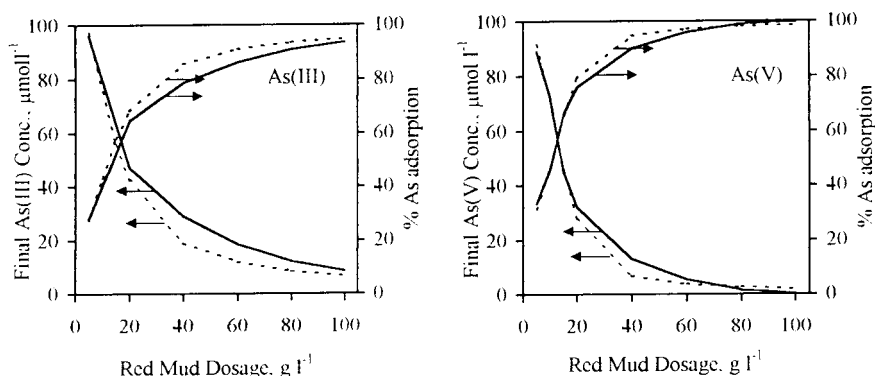


Fig. 5. Effect of red mud dosage on the As(III) and As(V) adsorption (initial concn.:  $133.5 \mu\text{mol l}^{-1}$ ; contact time: 60 min for As(III) and 120 min for As(V); pH: 9.5 for As(III) and 3.2 for As(V); temperature:  $25^\circ\text{C}$ ).

in Table 3. The estimated value of  $Q^\circ$  for As(III) adsorption decreases with rise in temperature while it increases for As(V) adsorption. The other Langmuir parameter  $b$  exhibits similar trends. It can be stated that As(III) adsorption is exothermic whereas the adsorption of As(V) is endothermic. These results can also be seen from calculated  $\Delta H$  values (Table 3). Hence, it can be concluded that the nature of As(III) adsorption is physical and that of As(V) is chemical. The negative Gibbs' free energy values indicate the adsorption of both arsenic types are spontaneous. The decrease in free energy change with the rise in temperature shows an increase in feasibility of adsorption at higher temperatures [25]. The positive values of entropy change suggest some structural changes in adsorbate and adsorbent.

### 3.4. Effect of adsorbent dosage

Fig. 5 shows the effect of red mud dosage on the removal of arsenic. The arsenic removal efficiency is increased with the amount of red mud. Final arsenic concentrations and removal efficiencies were also calculated from the isotherms in Fig. 4. In Fig. 5, results from dosage study and values extracted from isotherms are given with solid and dashed lines, respectively. It can be

stated that there is an acceptable fit between the results of isotherm and dosage studies.

Final arsenic concentration can be reduced below the regulation limits by increasing the adsorbent dosage. In general, the red mud adsorbed As(V) effectively more than the As(III). About a  $100 \text{ g l}^{-1}$  red mud dosage is sufficient for a final arsenic concentration below the regulation values of potable waters for As(V) while more is needed to adequately remove As(III).

## 4. Conclusion

The solid fraction of red mud was tested to find out its As(III) and As(V) adsorption characteristics. Batch experiments show that red mud is capable of removing arsenic from aqueous solutions.

As(III) and As(V) adsorptions are equilibrated within 45 and 90 min respectively, at  $25^\circ\text{C}$ ,  $133.5 \mu\text{mol l}^{-1}$  ( $10 \text{ mg l}^{-1}$ ) concentration and  $20 \text{ g l}^{-1}$  red mud dosage. For As(III) and As(V), favorable adsorptions take places at pH 9.5 and 3.2, respectively. It should be noted that the adsorption densities at these conditions are  $4.31$  and  $5.07 \mu\text{mol g}^{-1}$  for As(III) and As(V), respectively. Data obtained from equilibration time study fit Lagergren

equation for both arsenic species. Isotherm studies show that the Langmuir equation fits the experimental data reasonably well. Thermodynamic calculations based on the data from the study on temperature indicate that As(III) adsorption reaction is exothermic and that of As(V) is endothermic.

A practically usable adsorbent should be readily separated from the liquid, effective in a wide range of pH, inexpensive and able to be reutilized. The difficulties in solid-liquid separation and its being effective in a narrow pH range decrease the usability of red mud as an adsorbent. However, red mud is a very economical material since it is a waste product and is very fine grained. In addition, arsenic adsorbed red mud may be reused in some red mud usable metallurgical processes which are recommended to utilize red mud as an iron source [16].

In conclusion, since red mud is a waste, is fine grained and inexpensive it can be economically used for the removal of arsenic from wastewaters. Its adsorption capacity may be increased by activation. On the other hand, liquid phase of red mud constituting a weak alkaline aluminate solution may be utilized for arsenic removal by coagulation. Forthcoming studies based on developing the arsenic adsorption capacity of red mud by activation and utilizing the liquid phase of red mud in the removal of arsenic by coagulation are in progress.

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