



## Research article

# Characterization studies of red mud modification processes as adsorbent for enhancing ferricyanide removal



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## ABSTRACT

In this study, waste solids of the alumina industry (red mud) have been modified and utilized as a ferricyanide adsorbent which is characterized by XRD, XRF, FTIR, SEM, EDX, ICP and BET analysis. The four modification methods including the processes of washing with seawater (B), acid treatment Bauxsol (ATB), activated ATB using ammonia (ABA), and activated Bauxsol using cetyltrimethylammonium bromide (CTAB) (ABC) were evaluated to increase the reactivity of red mud (RM) for ferricyanide removal. The ferricyanide adsorption capacity was obtained at 12.40, 6.84, 2.95, 2.50 and 0.44 (mg/g) for ABC, ATB, ABA, B and RM, respectively. The CTAB with a concentration above the critical micelle concentration changed the negative charge of red mud to positive charge. It decreased the negative charge repulsion force between the Bauxsol surface and ferricyanide ions. The adsorption capacity of ferricyanide was decreased from 12.40 to 2.75 (mg/g) with increasing the adsorbent amount from 0.5 to 5 (g). The results showed that the activated Bauxsol using CTAB could be effectively used as a new adsorbent for ferricyanide ions from wastewaters.

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## 1. Introduction

Cyanide is a very toxic compound and dangerous for humans and aquatic life. The cyanide reacts with the trivalent iron in the cytochrome oxidase for inhibition of electron transport that leads to the prevention of oxygen consumption of cells and impairment of vital functions (Gebresemati et al., 2017). There is cyanide in industrial wastewaters including ore extraction, manufacturing of synthetic organic and inorganic compounds, coal carbonization, coke-processing, plants metal plating, chemical synthesis (nylon, fibres, resins, herbicides), photographic processing, gold refining, steel industries, plating and surface finishing. The wastewaters of these industries contain different levels of free and metal complexes of cyanides. Thus, the cyanide removal is necessary to meet standard environmental requirements (below 0.02 mg/L) before discharge into the environment (Adhoum and Monser, 2002; Bushey and Dzombak, 2004; Noroozifar et al., 2009; Asgari et al., 2012; Hanela et al., 2015; Gebresemati et al., 2017).

Various methods including sedimentation, oxidation, chemical treatment, physical-chemical adsorption, biological and electrical

processes have been used for cyanide removal from wastewaters (Ali and Gupta, 2006; Ali, 2010, 2012, 2014; Mihai et al., 2016). The adsorption process is efficiently applied to remove cyanides, in which cyanide is concentrated at the adsorbent surfaces from its liquid. It has some advantages such as high efficiency, good operational conditions and low cost. In order to remove cyanide, the adsorbent of oxidic minerals, coffee husk, zeolite,  $\gamma$ -alumina, gibbsite, goethite, sand, gravel and activated carbon have been recently applied (Mihai et al., 2016). In order to improve the reactivity of adsorbent for removal of ions in the solution, various modification methods have been recently used (Genç-Fuhrman et al., 2004a,b; Grudić et al., 2006; Cengeloglu et al., 2007; Ha et al., 2011; de Souza et al., 2013). Pradhan et al. (1999) used the activation method of simple acid dissolution followed by ammonia precipitation to remove hexavalent chromium from aqueous solution by red mud. Altundogan et al. (2002) used the acid treatment method to increase the arsenic adsorption capability on red mud. They showed that the adsorptive capacity of red mud in removing arsenic increased with acid treatment (Altundogan et al., 2002). Tor and Cengeloglu (2006) also successfully used the acid activated red mud for removal of congo red from aqueous solution (Tor and Cengeloglu, 2006). Cao et al. (2016) used the cation surfactant of cetyltrimethylammonium bromide (CTAB) to improve the reactivity of iron oxide nanoparticles for the removal of phosphate.

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They showed that efficiency in removing phosphate increased using CTAB (Cao et al., 2016).

The industrial solid wastes such as red mud (RM) can be used to treat the wastewater of another industry (Ali et al., 2012). The RM is relatively toxic due to the high calcium and sodium hydroxide. It can create a serious pollution hazard. In recent years, numerous studies have investigated the application of RM for the treatment of wastewater containing toxic heavy metals. It is economical and also effective to solve the problem of solid waste disposal from environmental aspects (Wang et al., 2005). The RM is the waste solid of the alumina industry that is produced as a by-product from the Bayer process. Large amounts of RM (between 1 and 2 tons) are produced for every ton of alumina production, which is remarkable for wastewater treatment (Schwarz and Lalík, 2012). The application of RM as adsorbent strongly depends on its origin. In order to improve the adsorption property of RM, some activation methods are successfully used. However, studies on the application of activated RM to remove ferricyanide have not been reported in the literature.

In this study, the activated red mud, which contains different metal oxides as adsorbent were used for the removal of ferricyanide complexes (Theis et al., 1988; Cheng et al., 1999; Rennert, 2002; Bushey and Dzombak, 2004; Barakat, 2005). The chemical methods including washing with seawater (Bauxsol), acid treatment Bauxsol (ATB), activated Bauxsol with ammonia (ABA) and activated Bauxsol with cetyltrimethylammonium bromide (ABC) were applied to improve the adsorption property of RM. The morphologies, structures and properties of the activated red mud were evaluated by XRD, XRF, FTIR, SEM, EDX, ICP and BET, respectively.

## 2. Materials and methods

Cetyl trimethylammonium bromide (CTAB) as surfactant, potassium ferricyanide ( $K_4Fe(CN)_6 \cdot 3H_2O$ ) and ammoniac were purchased from Merck. KCl for adjusting the solution ionic strength and HCl and NaOH for adjusting the solution pH was obtained from Merck. The percentage of ferricyanide in the solution was determined by a UV–visible spectrophotometer (model HITACHI U-2000) (Hong et al., 2008). The adsorption capacity ( $q_e$ ) of ferrocyanide ions per unit mass of adsorbent was evaluated from Eq. (1).

$$q_e = (C_i - C_e)v/M \quad (1)$$

where,  $C_i$  is the initial concentration of adsorbate (ppm),  $C_e$  is the final concentration of adsorbate (ppm),  $v$  is the solution volume (lit) and  $M$  is the adsorbent amount (gr).

The RM was obtained from Jajarm mine, 5 km northeast of Jajarm city, North Khorasan province, Iran. The RM chemical component is presented in Table 1. The results show that it mainly consists of oxides of aluminum, calcium, iron, titanium and silicon. It has about 22.17%  $Fe_2O_3$ .

### 2.1. Preparation of activated red mud

The effect of some activation methods (Fig. 1) including washing with seawater, acid treatment, washing with HCl and subsequently precipitating with ammoniac and activated red mud using CTAB on the red mud adsorption capacity were evaluated as the following.

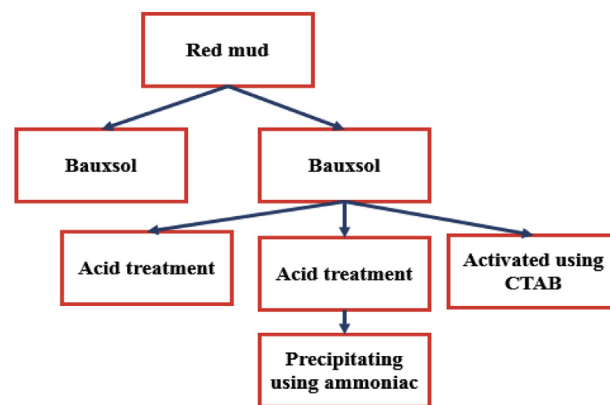


Fig. 1. Activation process of red mud.

#### 2.1.1. Washing with seawater (bauxsol)

The RM was crushed to obtain a grain size of less than 0.149 mm. It was initially suspended in seawater solution (the weight ratio of liquid to solid was 3/1) and stirred for 1 h to obtain the equilibrium pH of 8–8.5. Then, the mixture was allowed to sediment for about 1 h. After filtration (using Whatman 42 filter paper) and the drying of Bauxsol (at 100 °C), it was sieved through a 0.2 mm screen to obtain a uniform sized powder (Genç-Fuhrman et al., 2004a,b).

#### 2.1.2. Acid treatment Bauxsol

The Bauxsol was boiled in 0.5 M HCl solution (at liquid/solid ratio of 25 ml/g) for 1 h. Subsequently, the residue of acidified Bauxsol samples were washed with distilled water and dried overnight at 100 °C. The resulting solid samples were powdered and screened through the sieve of 0.149 mm (Genç-Fuhrman et al., 2004a,b).

#### 2.1.3. Activated Bauxsol using ammonia

The Bauxsol was activated by washing with HCl and then precipitating with ammoniac. 100 ml of HCl solution (%20 wt) refluxes 5gr of bauxsol during 30 min. Subsequently, ammonia was added to complete precipitation until obtaining a pH of 8–8.5. It is optimal to neutralize red mud until reaching a pH of around 8 for a number of reasons including the release of chemically adsorbed Na, neutralizing alkaline buffer minerals, and insolubility of toxic metals in this pH value (Genç-Fuhrman et al., 2004a,b; Hu et al., 2016; Zeng et al., 2017).

#### 2.1.4. Activated Bauxsol using CTAB

CTAB as surfactant was used to modify the surface of Bauxsol. The Bauxsol surfaces have negative charge that reduces the anion adsorption. In order to solve the negative charge repulsion force between the Bauxsol surface and cyanide anions, the cationic surfactant of CTAB with a concentration above the critical micelle concentration (CMC) was used (Ali and Aboul-Enein, 2006). It changes the negative charge of the Bauxsol surface to a positive charge. The appropriate amount of CTAB is dissolved in 100 ml of distilled water. Then, 1% (w/v) CTAB solution was mixed with 20 g Bauxsol and stirred for 2 h. After filtration of the solution, the solid phase was washed three times with distilled water to remove the

Table 1  
Chemical composition of RM.

Red mud	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	MgO	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	CaO	P <sub>2</sub> O <sub>5</sub>	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	LOI
Elements (mass %)	13	13.98	4.2	2.01	0.42	7.17	0.06	24.25	0.16	22.17	1.63	9.55

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