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Removal of boron from aqueous solution by using neutralized red mud

Yunus Cengeloglu [a], Ali Tor [b,*], Gulsin Arslan [a], Mustafa Ersoz [a], Sait Gezgin [c]

[a] Selcuk University, Department of Chemistry, Campus, 42031 Konya, Turkey

[b] Selcuk University, Department of Environmental Engineering, Campus, 42031 Konya, Turkey

[c] Selcuk University, Agriculture Faculty, Campus, 42031 Konya, Turkey

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Abstract

The adsorptive removal of boron from aqueous solution by using the neutralized red mud was studied in batch equilibration technique. The effects of pH, adsorbent dosage, initial boron concentration and contact time on the adsorption were investigated. The experiments demonstrated that boron removal was of a little fluctuation in pH range of 2–7 and it takes 20 min to attain equilibrium. The adsorption data was analyzed using the Langmuir and the Freundlich isotherm models and it was found that the Freundlich isotherm model represented the measured sorption data well.

Keywords: Adsorption; Equilibrium isotherm; Freundlich isotherm; Langmuir isotherm; Boron; Red mud

1. Introduction

Boron minerals occur in nature in more than 200 [1]. Among them boric acid and boron salts have widely used in the glass manufacture, soaps and detergents, flame retardant, neutron absorber for nuclear installations, and also used in fertilizers for the treatment of boron deficient soils. Boric acid is used as disinfectant and as food preservative due to its bactericidal and fungicidal properties [2,3].

The presence of boron in water has two differentiated origins, one of which is natural origin due to the boron in silts present in the aquifer and the other one is the anthropogenic origin, such as wastewater discharge from boron mines and boric acid plants [4,5]. The most important boron source in the world is located in West Anatolia [6]. It was reported that boron concentrations in the aquifers in West Turkey were in the range of 1–63 mg/L [5]. Moreover, some boron plants in Turkey cause some environmental problems. Wastewaters having high boron content (approx. 1500 mg/L) from plants are discharged to ponds. These ponds occupy a wider area than the boron plant area. In case of over-flow from the ponds, the wastewater might deteriorate the agricultural area [7].

Thermal waters are generally discharged to agricultural areas for irrigation after being used in thermal baths. Their boron contents are accumulated in the soils and this cause the changing of physical, chemical and biological features of soils. Also, these waters could mix with underground waters by passing through the soil and constitute complexes with Pb, Cu, Cd and Ni ions. The toxicities of these complexes are higher than those of the heavy metals [8].

Safe concentration of boron in irrigation water is 0.3 mg/L for sensitive plants, 1–2 mg/L for semi-sensitive plants and 2–4 mg/L for tolerant plants [9]. Boron is also an important micronutrient for animals and humans [10], but the range between deficiency and excess is narrow. The approvable daily intake is 0.3 mg boron/kg [11]. The World Health Organization (WHO) has given a recommendation of below 0.3 mg/L boron for the quality of drinking water [12]. In order to prevent the environmental problems of high concentration of boron in waters, their boron contents should be removed by a suitable method.

Numerous researches, including coagulation–precipitation [13], adsorption on active carbon and fly ash [7], ion exchange [14–18], solvent extraction after complexation [19], reverse osmosis [4], donnan dialysis [20] and electrodialysis [21] have been conducted for boron removal from water. Because of the bactericidal effect of the boron in boric acid and sodium borate form, the biological treatment of boron wastewater is limited [5].

* Corresponding author. Fax: +90 332 241 06 35.
E-mail address: ali.alitor@gmail.com (A. Tor).

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These methods, given such as, ion exchange, coagulation–precipitation, electrodialysis, are expensive. In recent years, considerable attention has been devoted to the study of different types of low-cost materials and other waste materials for adsorption of some toxic substances [22].

Studies using red mud residues from alumina refineries as unconventional adsorbents for water and wastewater treatment purposes are motivated by the fact that red mud is a fine-grained mixture of oxides and hydroxides, capable of removing several contaminants, as well as being widely available. Thus, several studies have reported that red mud or activated red mud can be utilized for adsorbing different pollutants from water, including phosphate [23], fluoride [24], cadmium, lead, copper [25–28], nitrate [29], arsenic [30–32], phenol [33] and dye [34]. However, the study about utilization of red mud for removal of boron from aqueous phase has not been reported in the literature. Therefore, in the present paper, the possibility of utilization of the neutralized red mud as an adsorbent for removal of boron from water was studied.

2. Materials and method

2.1. Materials

About 500,000 m^3 of strongly alkaline ($pH \approx 12$–13) red mud-water pump is dumped annually into specially constructed dams around Seydişehir Aluminium Plant (Konya, Turkey). The grain size of red mud was mostly (>94%) less than 10 μm and had the following average composition (by wt.%)—Al_2O_3: 18.71 ± 0.59, Fe_2O_3: 39.70 ± 0.67, TiO_2: 4.90 ± 0.54, Na_2O: 8.82 ± 0.96, CaO: 4.47 ± 0.56, SiO_2: 14.52 ± 0.37 and loss on ignition: 8.15 ± 0.40.

The alkaline red mud was suspended in distilled water with a liquid to solid ratio of 2/1 on a weight basis, stirring it until the equilibrium pH is 8.0–8.5, and drying. The BET surface area of neutralized red mud was measured with nitrogen as 14.2 m^2/g by Apak et al. [25].

H_3BO_3 was of analytical grade obtained from R.P. Normapur® (Paris, France). NaCl, HCl were of also analytical grade obtained from Merck Co. (Darmstad, Germany).

2.2. Method

The boric acid solutions were prepared in distilled water by diluting the prepared stock solutions (1000 mg/L) to desired concentrations. The adsorption experiments were carried out by a batch method. All experiments were carried out at a constant ionic strength of 0.01 M maintained with NaCl. A known amount of red mud and boric acid solution were taken in a 100 mL stoppered conical flask. Sodium chloride was added to maintain ionic strength, and pH was adjusted to the desired level with 0.1 M NaOH or 0.1 M HCl solutions. The final volume was adjusted to 50 mL with distilled water and agitated at constant speed (500 rpm) with magnetic stirrer in room temperature over a period of time and filtered through Whatman No. 42 filter paper. The experimental parameters studied are adsorbent amount (1–8 g/L), contact time (20–200 min), initial boron concentration (12–200 mg/L) and pH (2–7). The concentration of boron in filtrate was determined by use of an ICP-AES (Varian, vista\AX CCD Simultaneous ICP-AES). The amount of boron removal was calculated from the ratio of boron taken and that remaining in the solution.

3. Results and discussion

3.1. Effect of contact time

Fig. 1 shows the effect of contact time on the equilibrium of boron adsorption onto neutralized red mud. The removal increases with time in the first 20 min. Then the adsorption equilibrium curve leveled off after this time. The removal of boron from aqueous solution by using polymer supported iminodipropylene glycol functions was investigated by Senkal and Bicak [6], who reported that polymer used was efficient in chelation with boric acid at ppm levels in contact time of 12 min. In addition, it was reported by Ozturk and Kavak [35] that the equilibrium time needed for adsorption of boron by fly ash was 24 h. For present study, the short time needed to achieve equilibrium suggests that neutralized red mud have high adsorption efficiency and have a potential for removal of boron from aqueous phase.

3.2. Effect of the pH

The amount of boron adsorbed depends on the distribution of $B(OH)_3$ and $B(OH)_4^-$ which are controlled by pH of the solution. The two species, $B(OH)_3$ and $B(OH)_4^-$, in the solution compete for the adsorption on the neutralized red mud. The tetrahedral $B(OH)_4^-$ becomes the dominant species at pH between 9 and 10 for total boron concentration less than 3000 mg L^{-1} for all temperatures [36].

In addition, the solution pH relative to the point of zero charge (pHpzc) for the red mud also needs to be considered. At pH

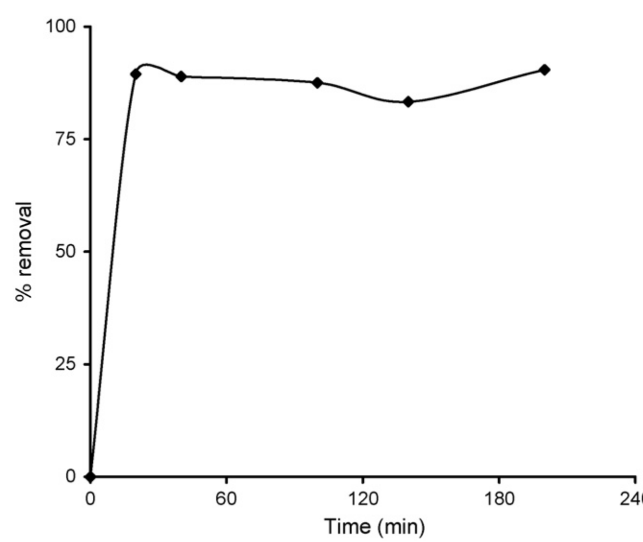


Fig. 1. Removal of boron as a function of equilibrium time (pH 7, initial boron concentration: 43 mg/L, red mud dose: 4 g/L, agitation speed: 500 rpm and temperature: 25 ± 1 °C).

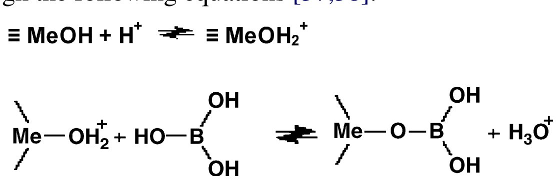
Table 1
Effect of pH on the adsorption of boron by neutralized red mud (initial boron concentration: 43 mg/L, red mud dose: 4 g/L, agitation speed: 500 rpm and temperature: 25 ± 1 °C)

pH of the solution	q (mg/g)
2	9.236
4	9.541
7	9.686

values above the pHpzc of the adsorber, the surface of adsorber particles is negatively charged and as the pH rises above the pHpzc, anion adsorption decreases. The pHpzc for neutralized red mud has been reported as about 8.3 in the literature [23].

Because, at pH above 8, there are repulsive forces between the $B(OH)_4^-$ and negative charged surface of the red mud, the adsorption of boron on the neutralized red mud was studied at three different pH values, ranging from 2 to 7. The results are given in Table 1, which indicated that the amount of boron adsorbed by neutralized red mud had a little fluctuation in the pH range of 2–7. Therefore, in further experiments, pH of the solution was adjusted to 7.

Red mud is a mixed adsorbent with different metal oxides. In a humid environment, hydroxylated surfaces of these oxides develop positive charge on the surface. As a consequence, at pH below 8.3 $B(OH)_3$ molecules are favour in aqueous solution. In this pH, the chemical interaction between boric acid and hyroxylated surface of red mud can be represented schmetically through the following equations [37,38]:



where Me is the metal ion (Si, Fe or Al).

3.3. Adsorption isotherms

The relationship between the amount of boron adsorbed and the boron concentration remaining in solution is described by an isotherm. The two most common isotherm types for describing this type of system are the Langmuir and the Freundlich isotherm. The most important model of monolayer adsorption came from the work of Langmuir [39]. This isotherm is given as follows:

$$q_e = \frac{Q_0 b C_e}{1 + bC_e} \tag{1}$$

The constants Q_0 and b are characteristics of the Langmuir equation and can be determined from a linearized form of Eq. (2):

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \tag{2}$$

where C_e is the concentration of boron (mg/L) at equilibrium, Q_0 the monolayer capacity of the adsorbent (mg/g) and b is the Langmuir adsorption constant (L/mg).

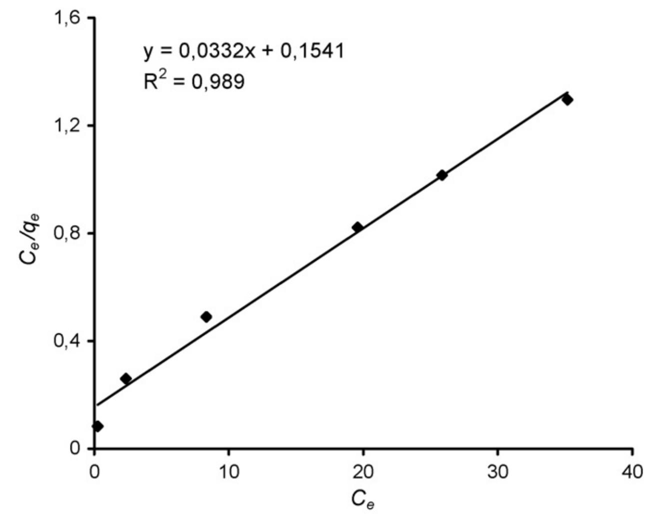


Fig. 2. The Langmuir isotherm plot for boron adsorption on red mud. Red mud dose: 4 g/L, pH 7, agitation speed: 500 rpm and temperature: 25 ± 1 °C.

The Freundlich isotherm [40] is derived to model the multilayer adsorption and for the adsorption on heterogeneous surfaces. The Freundlich model is formulated as follows:

$$q_e = kC\,e^{1/n} \tag{3}$$

The equation may be linearized by taking the logarithm of both sides of Eq. (6) and linear form of Freundlich isotherm can be given as follows:

$$\log q_e = \log k + \frac{1}{n}\log C_e \tag{4}$$

where C_e is equilibrium concentration, k the sorption capacity (mg/g) and n is an empirical parameter.

The values of the constants for isotherms were obtained from the slope and intercept of the plots of each isotherm (Figs. 2 and 3). The monolayer sorption capacity, Q_0, and sorption equilibrium constant, b, are 30.120 mg/g and 0.215 L/mg for

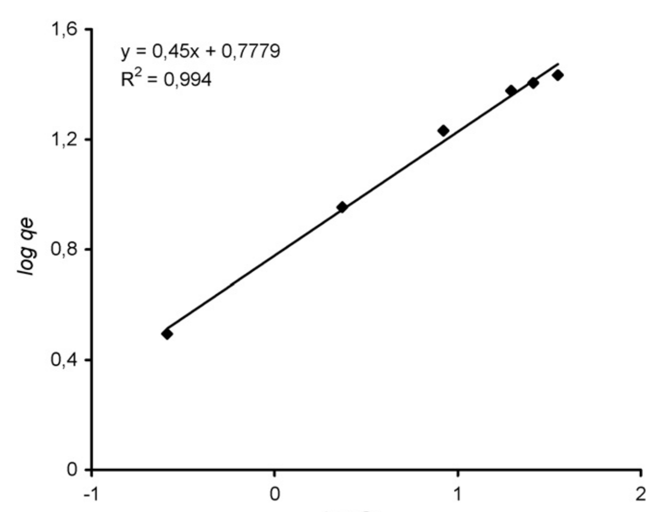


Fig. 3. The Freundlich isotherm plot for boron adsorption on red mud. Red mud dose: 4 g/L, pH 7, agitation speed: 500 rpm and temperature: 25 ± 1 °C.

Langmuir isotherm. The Freundlich sorption isotherm constant k is 5.996 mg/g and n is 2.221.

An error analysis is required in order to evaluate the fit of the adsorption isotherms to experimental data. In present study, the linear coefficient of determination (R^2) was employed for the error analysis. The linear coefficient of determination is calculated by using the equation [41]:

$$R^2 = \frac{S_{xy}^2}{S_{xx}S_{yy}} \tag{5}$$

where S_{xx} is the sum of squares of X:

$$S_{xx} = \sum_{i=1}^{n} x_i^2 - \frac{\sum_{i=1}^{n} x_i}{n} \tag{6}$$

where S_{yy} is the sum of squares of Y:

$$S_{yy} = \sum_{i=1}^{n} y_i^2 - \frac{\sum_{i=1}^{n} y_i}{n} \tag{7}$$

where S_{xy} is the sum of squares of X and Y:

$$S_{xy} = \sum_{i=1}^{n} x_i y_i - \frac{\left(\sum_{i=1}^{n} x_i\right)\left(\sum_{i=1}^{n} y_i\right)}{n} \tag{8}$$

The value of R^2 ranges between zero and one. A R^2 of one shows that 100% of the variation of experimental data is explained by the regression equation. The coefficient of determination, R^2, was applied to determine the relationship between the experimental data and the isotherm in most studies. The R^2 values for linear form of Langmuir and Freundlich isotherms are 0.989 and 0.994, respectively. According to R^2 values, the Freundlich isotherm best represents the equilibrium adsorption of boron on the neutralized red mud. This result is attributed that various active sites or heterogenous mixture of several minerals on neutralized red mud has different affinities to molecule of $B(OH)_3$ [42].

That the adsorption data provide a fit to the Freundlich isotherm compared to the Langmuir model was also supported by the distribution coefficient values (K_D, m^3/kg), which reflects the binding ability of adsorbent surface for a sorbate. The K_D value of a system mainly depends on pH and type of surface. The distribution coefficient K_D values for boron and red mud at pH 7 were calculated [43] with the following equation:

$$K_D = \frac{C_s}{C_w} \tag{9}$$

where C_s is the concentration of boron on the red mud (mg/kg) and C_w is the equilibrium concentration in solution (mg/m^3). As can be seen in Fig. 4, K_D increases with increasing red mud concentration at constant pH that implies the heterogeneous surface of the neutralized red mud. If the surface is homogeneous, the K_D values at a given pH should not change with red mud concentration.

Freundlich capacity constants of activated carbon and activated alumina for boron adsorption were reported as 0.190 and 0.440 mg/g, respectively (Table 2) [44]. It was also reported that k and $1/n$ values for three kinds of mine spoils for boron adsorption: 0.004, 0.005 and 0.002 mg/g for k and 0.694, 0.615 and 0.510 for $1/n$ [45]. These data indicate that the adsorption capacity of the neutralized red mud is higher than that of activated carbon, activated alumina and mine spoils. In addition, it was shown using mathematical calculations that n was between 1 and 10 representing beneficial adsorption [46]. Therefore, it can be concluded that neutralized red mud can be used as an adsorbent for beneficial adsorption of boron from aqueous solution.

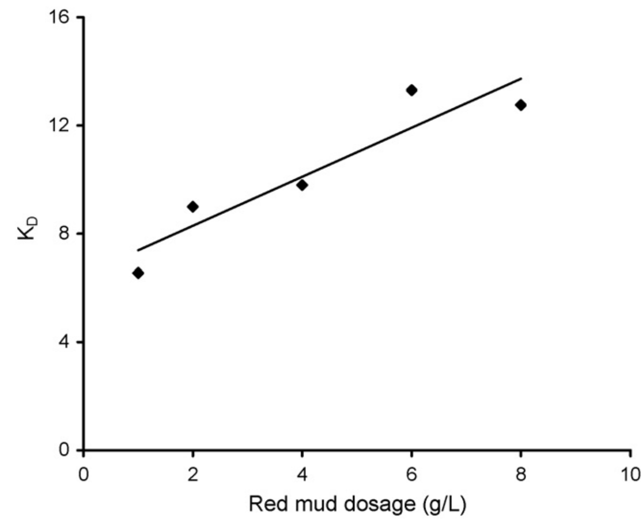


Fig. 4. The variation of K_D values with red mud dose. Initial boron concentration: 43 mg/L, pH 7, agitation speed: 500 rpm and temperature: 25 ± 1 °C.

3.4. Effect of red mud dosage

The effect of the amount of neutralized red mud on the boron removal is presented as % removal and mg boron adsorbed/g red mud versus the amount of red mud in Fig. 5. As expected, the removal of boron was increased with increasing red mud dosage, which is due to the increase in surface area of the red mud. The concentration of surface hydroxyl ions is related to red mud concentration through surface site density. Accordingly the percent of adsorption was increased with red mud dose, whereas loading capacity decreased since the unit of q is mg boron/g red mud (Fig. 5).

Table 2
The Freundlich isotherm constants (k, n) for adsorption of boron by different adsorbents

Adsorbents	k (mg/g)	n	Reference
Activated carbon	0.190	2.5	[42]
Activated alumina	0.440	1.4	[42]
Minespoils (Wooley Edge)	0.004	1.4	[43]
Minespoils (Hoyland Common)	0.005	1.6	[43]
Minespoils (Crowedge)	0.002	1.9	[43]
Neutralized red mud	5.996	2.2	Present study

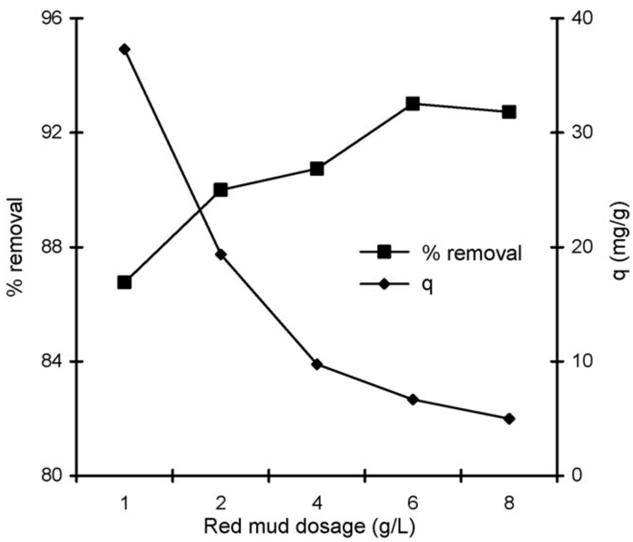


Fig. 5. The variation of boron removal with red mud dose. Initial boron concentration: 43 mg/L, pH 7, agitation speed: 500 rpm and temperature: 25 ± 1 °C.

4. Conclusion

In the present study, the removal of boron from aqueous solution was investigated by using the neutralized red mud with respective different parameters. The other point, the utilization of red mud as adsorbent for removal studies was considered. The following results can be summarized as:

i. The adsorption equilibrium was attained within 20 min contact time.
ii. Compared to the Langmuir isotherm, the Freundlich isotherm model represented the measured sorption data well. This indicates that the heterogeneous adsorption occur, which is also supported by the distribution coefficient (K_D).
iii. The adsorption of boron on the neutralized red mud has a little fluctuation in the pH range of 2–7.

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