

ADSORPTION POTENTIAL OF UNMODIFIED RICE HUSK FOR BORON REMOVAL

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A batch study of boron removal from aqueous solutions by adsorption using rice husk was carried out. The effect of selected parameters such as particle size, pH, adsorbent dosage, and initial concentration of adsorbate on boron removal was investigated in the study. Results showed that the maximum boron removal was obtained with the rice husk particle size between 0.425 mm and 1.0 mm at pH 5. Boron removal was increased with an increasing amount of adsorbent dosage but decreased as the initial concentration of adsorbate increased. A Freundlich isotherm was used to describe the potential of boron adsorption using rice husk. The maximum adsorption capacity of rice husk was found to be 4.23 mg/g for boron removal.

Keywords: Rice husk; Boron removal; Langmuir and Freundlich isotherms; Batch study

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INTRODUCTION

The industrial application of boron results in the accumulation of boron in industrial wastes, from which the boron then may permeate into the natural environment (Weinthal *et al.* 2005). The main industrial wastes that cause boron pollution come from the detergent industry (Weinthal *et al.* 2005; Raymond and Butterwick 1992; Vengosh *et al.* 1994), due to the excellent bleaching properties of sodium perborate. The addition of sodium perborate into detergents will consequently result in the accumulation of boron in domestic wastewater, particularly in industrial countries. Furthermore, high levels of boron concentrations have been found mostly in rivers that are associated with urban and industrial drainage relative to those from rural areas (Neal and Robson 2000), thus indicating a direct link between boron contamination in rivers and sewage pollution (Weinthal *et al.* 2005).

The essentiality of boron in humans has not been established; however, the lowest lethal dose for humans exposed to boron is reported to be around 640 mg kg⁻¹ of body weight by oral exposure, 8600 mg kg⁻¹ body weight by dermal exposure, and 29 mg kg⁻¹ body weight by intravenous injection (Ozturk *et al.* 2010; Stokinger 1981). On the other hand, excessive boron intake causes acute neurological effects, diarrhea, anorexia, weight loss, and testicular atrophy in mice, rats, and dogs. It also causes a decrease in fetal body weight and an increase in skeletal malformation and cardiovascular defects in pregnant female animals (Ozturk *et al.* 2010; Yazbeck *et al.* 2005; Pawa and Ali 2006).

There are no easy methods for the removal of boron from water and wastewaters (Öztürk and Kavak 2004). This is a critical problem in view of boron's potentially environmental damaging effects, especially damages affecting agricultural areas in which high levels of boron will cause toxicity to plants. Unfortunately, efficient treatments are lacking for boron-polluted water (Del-Campo Marín and Oron 2007).

One or more methods may be applied for boron removal, depending on the boron concentration in the medium (Öztürk and Kavak 2004). For boron removal, the main processes that have been studied are precipitation-coagulation, adsorption on oxides, adsorption on active carbon, cellulose, or clay minerals, ion exchange with basic exchanger, solvent extraction, membrane filtration, or use of boron-selective resins (Amberlite XE 243, Amberlite IRA 743) (Öztürk and Kavak 2003, 2004; Okay *et al.* 1985; Receptoglu and Beker 1991; Simonnot *et al.* 1999; Balki 1982; Goldberg *et al.* 1996).

The application of low cost and easily available materials in wastewater treatment has been widely investigated during recent years. Adsorption is a comparatively more useful and economical technique at low pollutant concentrations (Kavak 2009). There is a demand for adsorbents that are made up of inexpensive material and do not require any additional pre-treatment, such that the adsorption process will become economically viable (Sharma *et al.* 2010).

Various agricultural waste materials such as rice bran, rice husk, wheat bran, wheat husk, and kenaf have been evaluated as potential alternative adsorbents for the removal and/or recovery of metal ions from aqueous solution (Dhiraj Sud *et al.* 2007; Hasfalina *et al.* 2010).

The use of rice husk as low-cost sorbent for removing heavy metals such as As(V), Au, Cr(IV), Cu and Pb, Fe, Mn, Zn, Cu, and Cd(II) has been investigated. Apart from that, rice husk is also being used to treat textile dyes such as malachite green and acid yellow (Chuah *et al.* 2005). However, the use of rice husk for boron removal has not been studied elsewhere.

Rice husk has good chemical stability, high mechanical strength, and a granular structure, making it a good adsorbent material for treating heavy metals from wastewater (Wan Ngah and Hanafiah 2008). Apart from that, a great excess of rice husk is abundantly available from local users in developing countries, and this material currently poses disposal problems (Chuah *et al.* 2005). Statistical data of the Food and Agricultural Organization in the year 1995 stated that there is approximately 100 million tons of rice husk available annually for utilization in the developing countries that were considered (Chuah *et al.* 2005).

The general objective of this study was to investigate the potential use of rice husk as a low-cost adsorbent for boron removal from aqueous solution. The specific objectives for the study were to determine parameters such as particle size, pH, adsorbent dosage, and initial concentration of adsorbate affecting adsorption of boron using rice husk. Langmuir and Freundlich isotherms were used to describe the potential of rice husk for boron adsorption.

EXPERIMENTAL

Preparation of Adsorbent

The rice husk was washed with deionized water to remove dirt and dried. Then the rice husk was ground and sieved to classify the particle size of the ground rice husk. The ground rice husk was categorized into three particle class sizes: between 0.150 mm and 0.212 mm, between 0.212 mm and 0.425 mm, and between 0.425 mm and 1 mm.

Preparation of Adsorbate

A stock solution of boron (1000 mg/L) was prepared by dissolving an appropriate quantity of boric acid, H_3BO_3 (Merck, USA) in distilled water. The concentration of boric acid solution required for batch study experiment was prepared from stock solution by dilution using distilled water. The pH of the solution was measured using a pH meter (IQ Scientific Instruments, UK).

Batch Study Experiment

A batch experiment was performed by taking a specified weight of adsorbent and combining it with 500 mL boron solution of specified concentration and pH. After adsorption, the sample was filtered using filter paper (Whatman, USA). Boron concentration was determined using a spectrophotometer (Hach Odyssey DR2500, USA) using the Carmine Method 8015 (Hach, USA).

In order to investigate the effect of particle size on boron adsorption, three different sizes of rice husk at constant initial concentration of adsorbate (100 mg/L) and constant adsorbent dosage (1 gram) were used. To observe the effect of adsorbent dosage on boron removal, different amounts of rice husk were used (1.0, 2.0, 3.0, 4.0, 5.0, and 6.0 grams). The pH of the solution was adjusted to the desired level with 1M NaOH and 1M HCl solutions for pH 5 up to 10. In order to observe the effect of initial concentration of adsorbate on boron adsorption, the initial concentrations of adsorbate was varied accordingly (50, 100, 150, 200, 250, 300 mg/L). All experiments were conducted at ambient temperature.

RESULTS AND DISCUSSION

Effect of Particle Size

The effect of particle size on boron adsorption is shown in Figs. 1 and 2. The highest percentage of removal (84.00%) was achieved by the adsorbent of particle size between 0.425 mm and 1 mm. As shown in Fig. 2, the boron adsorption capacity increased as the particle size was increased. The highest boron adsorption capacity (q_e) was 42.00 mg/g. Therefore, all subsequent studies were carried out using the adsorbent of particle size between 0.425 mm and 1 mm.

Surprisingly, the result was in contradiction to the expected outcome that adsorption capacity will decrease with increasing particle size. This finding suggested that total surface area available for adsorption sites was influenced by the pore structure and not by the particle size of the adsorbent (Punjonharn *et al.* 2008). The pore

diameters that were prevalent in the adsorbent particles in the size range between 0.425 mm and 1 mm might be the most favorable among the three particle sizes of classes in terms of offering access to adsorption sites, and hence this class of particles exhibited the highest adsorption capacity.

Statistical ANOVA analysis was done to determine whether the particle sizes would affect boron adsorption. The p-value obtained ($P < 0.05$) indicated that there was significant difference in the data as a whole. This means that particle size did affect boron adsorption.

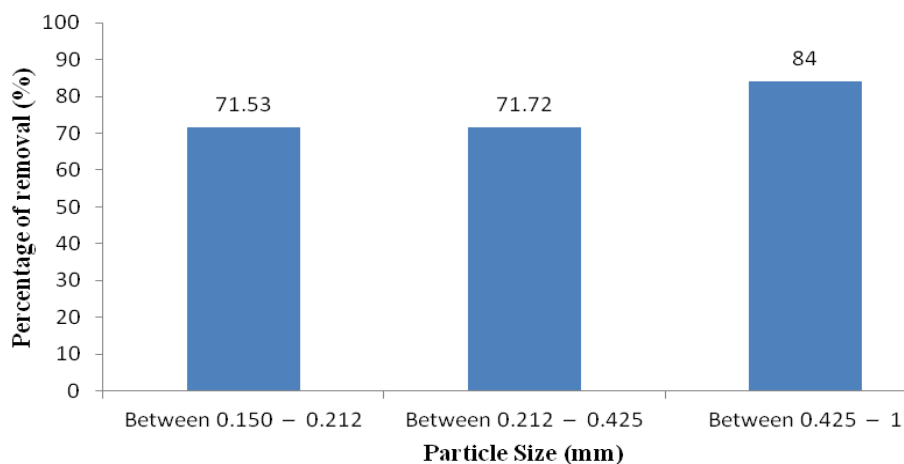


Fig. 1. Percentage of boron removal for three different particle sizes

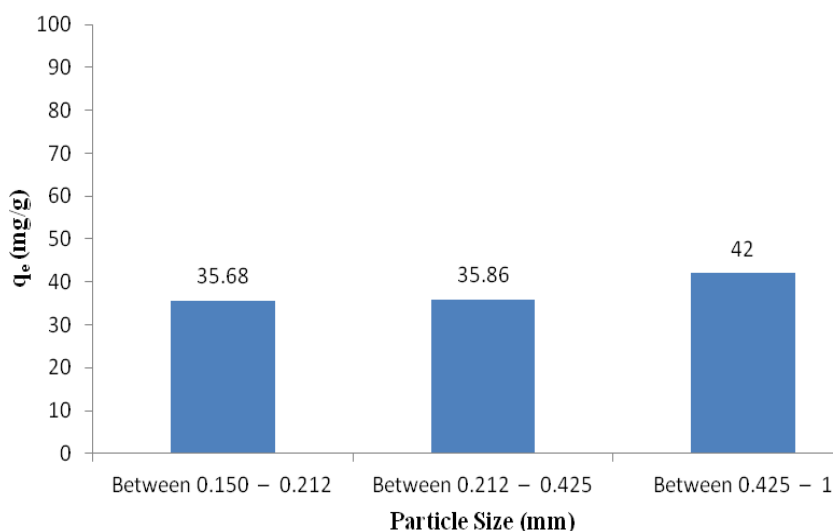


Fig. 2. Boron adsorption capacity for three different particle sizes

Effect of pH

Figure 3 shows the effect of pH variation between pH 5 to 10 on boron adsorption. The uptake of metals from aqueous solution is strongly affected by the pH of the solution (Sari and Tuzen 2009; Anayurt *et al.* 2009). From Fig. 3, the highest boron adsorption capacity ($q_e = 43.20$ mg/g) was achieved at pH 5, and then the boron adsorption capacity dropped gradually until the lowest point ($q_e = 39.30$ mg/g) at pH 8. Above pH 8, the boron adsorption capacity appeared to rise slightly. The boron adsorption capacity at pH 9 and pH 10 was 39.9 mg/g and 40.25 mg/g, respectively. Therefore, the adsorbate pH for all subsequent studies was maintained at pH 5.

For pH values above 5, the adsorption capacity decreased with increment in pH. The similar observation has been reported by Öztürk and Kavak (2005), Onundi *et al.* (2010), Sari and Tuzen (2008, 2009), Uluozlu *et al.* (2008), and Anayurt *et al.* (2009). Decrease in adsorption at the higher pH (>pH 5) can be explained by the formation of soluble hydroxylated complexes of metal ions, and the increasingly negative charge of the cell wall surface of adsorbate (Sari *et al.* 2008). A study by Liu *et al.* (2007) on the effect of pH on biosorption of boron onto cotton had reported that the maximum capacity ($q_e = 11.3$ mg/g) occurred at pH 7. On the other hand, the maximum uptake of boron takes place at pH 2 by using fly ash (Öztürk and Kavak 2005). In this study, the highest capacity was observed at pH 5.

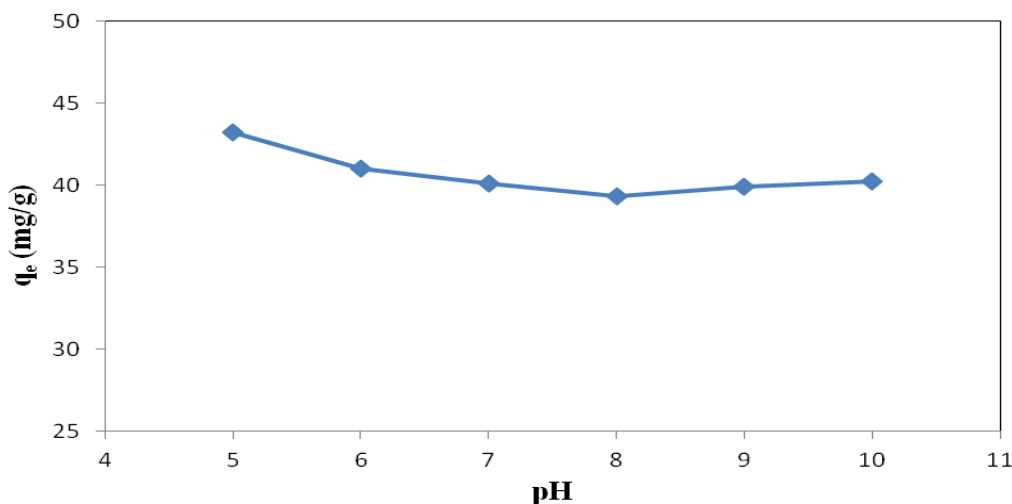


Fig. 3. Boron adsorption capacity for different pH

According to Öztürk and Kavak (2005) in the study of adsorption of boron from aqueous solution using fly ash, at acidic pH values the oxides that are the chief constituents of fly ash tend to form an aqua complex to yield a positively charged surface. Hence, a possible explanation for the highest adsorption capacity at pH 5 in this study is that the impure silicon dioxide (silica) of rice husk (Zakharov *et al.* 1993) may tend to form an aqua complex that has positively charged surface sites. Such positively charged hydroxylated oxide surface sites would be suitable for adsorption of borate anions (Öztürk and Kavak 2005). In addition, the use of hydrochloric acid for acidification might enable the positively charged surface to be associated with chloride ions, which

would subsequently be exchanged for borate anions (Öztürk and Kavak 2005). Therefore, the highest sorption of borate anion occurred at the most acidic pH levels that were tested (Öztürk and Kavak 2005).

On the other hand, the reduction of adsorption above pH 7 can be explained by the amount of tetrahedral boron $[B(OH)_4^-]$ present in the adsorbate (noting that the pKa of boric acid to borate is 9.1) (Demirçivi and Gülhayat 2008). The tetrahedral boron $[B(OH)_4^-]$ becomes the dominant species at pH between 9 and 10 for a total concentration less than 3000 mg L^{-1} (Cengeloglu et al. 2007; Goldberg and Su 2007). An increase in the amount of $B(OH)_4^-$ might not necessarily be the dominant adsorbed species. This could be as a consequence of the negative charge of the oxide surface of the adsorbent, which would have different affinity for the $B(OH)_3$ and $B(OH)_4^-$ species. The neutral $B(OH)_3$ species could be preferred to form boron complexation at basic pH due to its higher affinity for the negatively charged surface of adsorbent, whereas the $B(OH)_4^-$ ion would encounter charge repulsion (Goldberg and Su 2007). Another possibility is that the observed effects may be due, at least in part, to adsorption of other anionic species of boron such as tetraborate ($B_4O_7^{2-}$); such species would have a higher tendency to adsorb on any positive sites on the substrate, especially at very low pH. Moreover, adsorbing ions may not be able to access the surface of adsorbent due to shielding effect of increasing amounts of salt ions.

Statistical ANOVA analysis was done to determine whether the pH of adsorbate would be affected by boron adsorption. The p-value obtained ($P < 0.05$) indicated that there was a difference in the data as a whole. This means that pH of adsorbate to some extent did affect the boron adsorption.

Effect of Adsorbent Dosage

The effect of adsorbent dosage on boron adsorption is shown in Fig. 4. It was observed that the adsorption capacity of boron decreases with an increase in adsorbent dosage. The highest boron adsorption capacity ($q_e = 38.63 \text{ mg/g}$) was achieved with an adsorbent dosage of 1 g. The similar trend was reported by Onundi *et al.* (2000). Further increment of adsorbent above 1 g/L resulted in a decline in adsorption capacity.

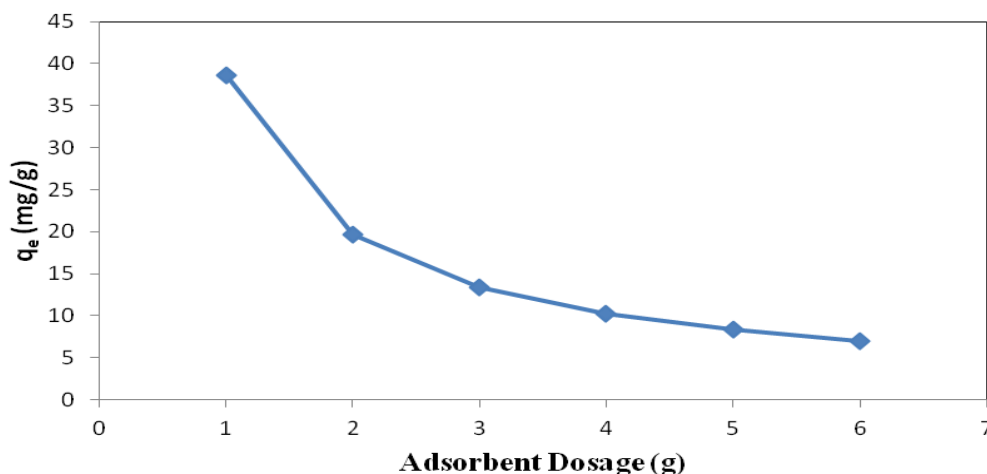


Fig. 4. Boron adsorption capacity for different adsorbent dosage (pH = 5, $C_0 = 100 \text{ mg/L}$)

Ponnusami *et al.* (2007), Cengeloglu *et al.* (2007), and Bouguerra *et al.* (2008) had stated in their study that an increase in adsorbent dosage will enhance the area available for adsorption. Hence, the percentage of boron removal in this study increases as the adsorbent dosage increases because of the adsorbent surface area available for adsorption was increased. In contrast, boron adsorption capacity decreases as adsorbent dosage is increased. A similar trend was also observed in a study of the removal of boron from ceramic industry wastewater by an adsorption-flocculation mechanism using palm oil mill boiler (POMB) bottom ash and polymer conducted by Chong *et al.* (2009). According to the cited authors, the adsorptive capacity of adsorbent available was not fully utilized at a higher adsorbent dosage in comparison to lower adsorbent dosage. Therefore, the trend where boron adsorption capacity decreases as adsorbent dosage increases in this study might be due to the adsorptive capacity of rice husk that was available and was not fully utilized at higher adsorbent dosage in comparison to lower adsorbent dosage. This is possibly because of the equilibrium concentration difference, where there was a lower driving force for adsorption at higher adsorbent dosage occurred (Chong *et al.* 2009; Öztürk and Kavak 2005).

Effect of Initial Concentration of Adsorbate

The effect of initial concentration of adsorbate on boron adsorption is shown in Fig. 5. The results showed that boron adsorption capacity increased as the initial concentration increased. The highest boron adsorption capacity ($q_e = 119.125$ mg/g) was achieved at an initial concentration of 300 mg/L.

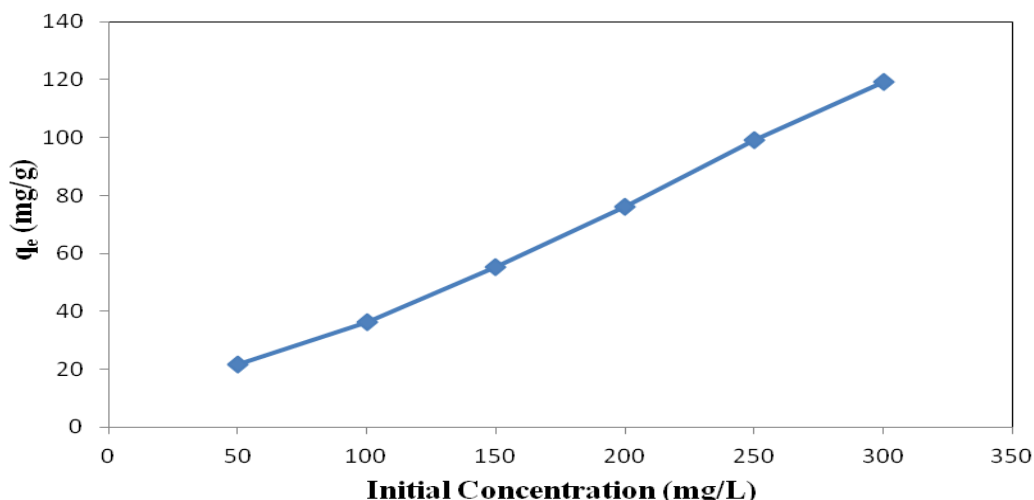


Fig. 5. Boron adsorption capacity for different initial concentration of adsorbate

According to Kavak (2009), the effect of initial concentration of adsorbate on the percentage of boron removal can be explained in terms of the number of exchangeable sites in adsorbent structure and boron-to-adsorbent ratios. Therefore, the reason for a decrease in percentage of removal with increasing initial concentration of adsorbate in this study is that as the ratio of boron to rice husk increases, the exchangeable sites in rice husk structure are saturated, resulting in a decrease of the removal percentage. In

contrast, the boron adsorption capacity in this study increased with increasing initial concentration, which might be due to an increase in the driving force of the concentration gradient, as an increase in the initial concentration of adsorbate. Gulnaz *et al.* (2004) in the study of sorption of basic dyes from aqueous solution by activated sludge stated that an increase in the initial concentration of adsorbate will increase the driving force of the concentration gradient, causing an increase in adsorption capacity.

Adsorption Isotherm Model

The Freundlich and Langmuir adsorption models were applied to experimental data. The R^2 value for Langmuir was found to be 0.086, indicating that the Langmuir model was not able to describe the relationship between the adsorbed amount of metal and biomass. The Freundlich model fit well with the data, and the R^2 value was 0.871. The Freundlich isotherm is given as follows (Freundlich 1906; Chong *et al.* 2009; Öztürk and Kavak 2005),

$$q_e = K_f C_e^{\frac{1}{n}} \quad (1)$$

where K_f (mg/g) and n are the constant for adsorption capacity and intensity, respectively. The constants can be determined from a linearized form of equation as below,

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (2)$$

Figure 6 shows the Freundlich plot for boron removal by adsorption, describing the potential of rice husk as an adsorbent in boron removal. The Freundlich constants K_f (mg/g), indicating the adsorption capacity, and n (dimensionless), indicative of the intensity of adsorption, were calculated from the slope and intercept of the Freundlich plot shown in Fig. 6, respectively. The calculated values were shown in Table 1. R^2 value of 0.871 (as shown in Fig. 6) indicated that the mechanism of boron adsorption using rice husk obeyed the Freundlich isotherm. Therefore, it can be concluded that Freundlich isotherm model best fit the equilibrium data (as in Fig. 6). The good fit of the Freundlich isotherm to an adsorption system assumes there is a multilayer adsorption and reversible adsorption which considers the interaction between adsorbate molecules (Freundlich 1906).

Table 1. Freundlich Constants

Freundlich Constants		
K_f (mg/g)	n	R^2
4.23	1.326	0.871

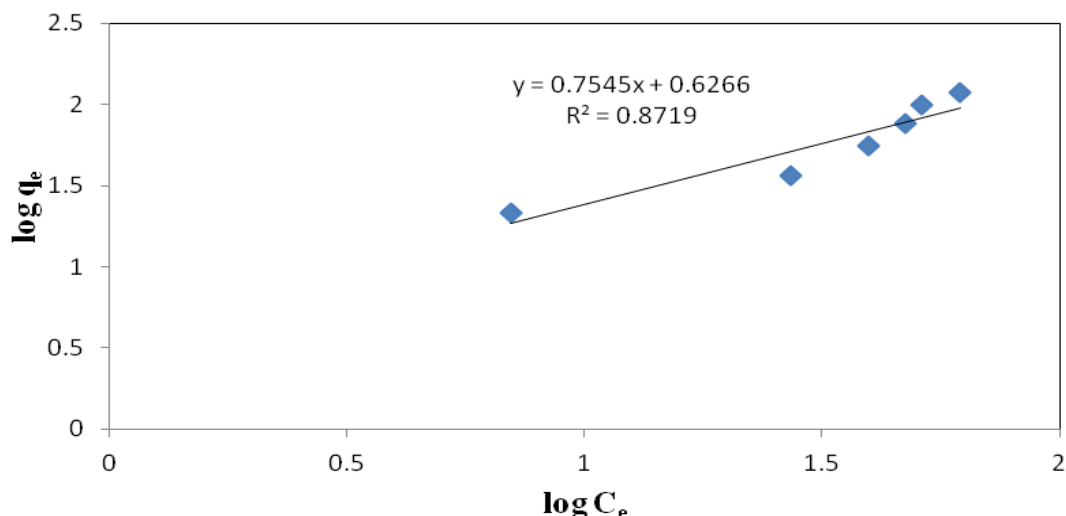


Fig. 6. Freundlich plot for boron removal by adsorption. (Condition: pH = 5; particle size > 425 μm ; volume of solution = 500 mL; adsorbent dosage = 1g; contact time = 8 hours; shaking rate = 90 rpm)

Generally, as the K_f value increases, the adsorption capacity of adsorbent increases (Öztürk and Kavak 2008). On the other hand, the value of n determines whether beneficial adsorption was being achieved. The value of n between 1 and 10 represents beneficial adsorption (Sivaraj *et al.* 2001). From Table 1, the n value obtained was 1.326, which indicates the beneficial adsorption of boron using rice husk occurred.

Although the beneficial adsorption was achieved using rice husk in boron adsorption, the R^2 value obtained for Freundlich isotherms plot was only 0.871. Apart from that, in comparison to the boron adsorption capacity found in other studies, as shown in Table 2, the value of adsorption capacity in the current study is relatively lower, yet shows a promising result.

Table 2. Boron Adsorption Capacity for Different Types of Adsorbent

Adsorbent	Adsorption Capacity (mg/g)	References
Rice Husk	4.23	Current study
Calcine Alunite	3.39	Kavak (2009)
Neutralized Red Mud	5.99	Cengeloglu <i>et al.</i> (2007)
Fly Ash	20.9	Öztürk and Kavak (2003)
Cotton	11.3	Liu <i>et al.</i> (2007)

CONCLUSIONS

- 1) In general it can be concluded that the parameters such as particle size, pH, adsorbent dosage, and initial concentration of adsorbate did affect the adsorption of boron using rice husk. An observed slight increase, rather than a decrease, in capacity with increasing particle size is consistent with a process dominated by absorption on the surfaces of pores within the material.
- 2) Equilibrium data by the Freundlich isotherm ($R^2 = 0.871$) was found to be well fitted in describing adsorption isotherm of rice husk as an adsorbent for boron removal.
- 3) Rice husk fibers could be a potential adsorbent for the boron removal from aqueous solutions

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