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OPTIMIZATION OF ADSORPTION PROCESS FOR BORON REMOVAL BY *MORINGA OLEIFERA*

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ABSTRACT: Boron contamination in water sources significantly threatens human health and environmental integrity. Adsorption techniques offer promising solutions for boron removal due to their cost-effectiveness and eco-friendliness. In this research, we investigate the potential of Moringa Oleifera seed, as a natural source, economical, and cost-effective adsorbent in removing boron from water. The process of adsorption is optimized by examining a few parameters such as pH, adsorbent dosage, and initial boron concentration. The primary objective of this research was to optimize several adsorption parameters such as pH (which ranged from 7 to 9), adsorbent dosage (which ranged from 4 to 8 g/L), and initial boron concentration (which ranged from 20 to 100 mg/L) utilizing Face-centred Central Composite Design (FCCCD). The highest removal of boron was attained at an initial concentration of 60 mg/L with an adsorbent dose of 6 g/L and pH 8. Furthermore, adsorption studies revealed a notable correlation between the Freundlich isotherm and the Pseudo-second order kinetic model. In conclusion, these findings look forward to underscoring the potential efficacy of Moringa Oleifera-derived adsorbents in effectively addressing boron contamination.

KEY WORDS: Boron contamination, *Moringa Oleifera*, Adsorption, Isotherm.

1. INTRODUCTION

Boron pollution in water has garnered significant attention due to its detrimental effects on human health and the environment. Sources of boron contamination include geological processes, agricultural runoff, industrial activities, and irrigation practices. Although boron is an essential micronutrient for plant growth in small amounts, excessive boron concentrations can cause toxicity problems in humans and agricultural systems [1]. This has prompted the search for economical and efficient boron removal methods.

Among these, adsorption is considered an environmentally friendly treatment approach due to the availability of natural and low-cost adsorbents [2]. In addition, adsorption stands out as a practical solution for water purification, valued for its simplicity, affordability, and effectiveness even at low contaminant levels. Unlike methods like reverse osmosis, adsorption is more cost-effective and accessible. One promising natural adsorbent is Moringa Oleifera, often called the "wonder tree". The seeds of *Moringa oleifera* contain proteins,

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polysaccharides, and functional groups that can effectively bind contaminants through adsorption and coagulation mechanisms [3]. Its seeds, rich in proteins and polysaccharides, contain functional groups that effectively bind and remove contaminants, making it a sustainable and eco-friendly option for water treatment.

This study focuses on optimizing the use of Moringa Oleifera seeds for boron removal from synthetic seawater using activated *Moringa oleifera* seeds as a biosorbent. The effects of important operational parameters, including pH, adsorbent dosage, and initial boron concentration, were investigated using Response Surface Methodology (RSM). In addition, adsorption equilibrium and kinetic studies were conducted using Langmuir and Freundlich isotherm models as well as pseudo-first-order and pseudo-second-order kinetic models to evaluate the adsorption behavior of boron onto activated *Moringa oleifera* seeds.

2. METHODOLOGY

2.1 Materials and Chemicals

Dried Moringa Oleifera seeds were sourced from a reputable supplier in Malaysia. Analytical-grade reagents, including potassium hydroxide (KOH), hydrochloric acid (HCl), sodium hydroxide (NaOH), boric acid (H_3BO_3), and potassium dichromate ($K_2Cr_2O_7$), were purchased from Sigma-Aldrich in Malaysia.

2.2 Experimental Procedures

2.2.1 Preparation of Stock Solution

All reagents used were analytical grade, and all solutions were made using distilled water. All stock solutions were made using polypropylene volumetric flasks. A stock boron solution (1000 µg mL^{-1}) was prepared by dissolving 0.5636 g of boric acid (Merck) in water and diluting it to 100 mL in a volumetric flask [4].

2.2.2 Adsorbent Preparation

Moringa Oleifera seeds were manually peeled, ground, and sieved using a 212 µm mesh. A boron stock solution (1000 µg/mL) was prepared by dissolving boric acid in distilled water, and a working concentration of 5 mg/L was achieved through dilution. For synthetic seawater preparation, boric acid was used following Zarei et al. [4]. Activated baobab fruit shell (BF-AC) was prepared by chemical reagents ($ZnCl_2$, KOH, or H_3PO_4) at a 1:1 impregnation ratio. Approximately 15 g of baobab shell material was mixed with the activating agent, and distilled water equivalent to four times the mass of the mixture was added. The mixture was stirred continuously for 1 hour at 50°C until a homogeneous paste was formed. The sample was pyrolyzed in a nitrogen-purged reactor at 500°C for 1 hour, then cooled and treated with 0.5 M HCl to remove impurities. After thorough washing and pH stabilization, the BF-AC was dried in a vacuum oven at 110°C for 24 hours, as described by Nedjai et al. [5]. Fig 1 shows the steps to prepare the activated *Moringa Oleifera* seed.

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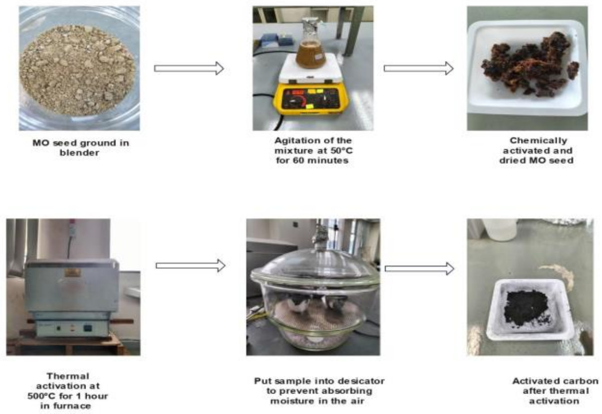

Fig. 1: Adsorbent Preparation.

2.2.3 Preparation of Standard Curve

Boron depletion was analysed using potassium dichromate ($K_2Cr_2O_7$) as a reference material to validate the spectrophotometer's photometric scale in the UV spectral region, following Conceição et al. [6]. Boron solutions (1 mL) at concentrations of 20, 40, 60, 80, and 100 mg/L were added to 1.5 mL cuvettes along with 0.5 mL of potassium dichromate. The dichromate enhanced the colour intensity of the analyte for easier detection. Absorbance was measured at 600 nm, and boron concentrations were calculated using a standard curve. Fig 2 shows the standard curve of boron concentrations.

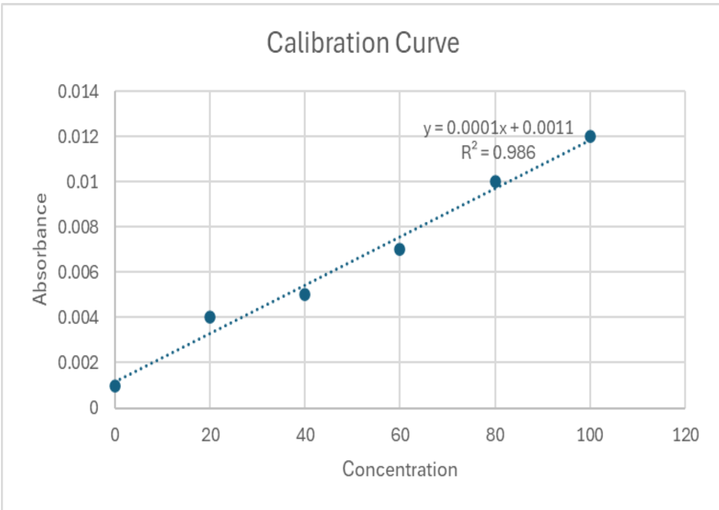

Fig 2: The calibration curve of Boron

2.2.4 Batch Adsorption Experiments

The adsorbent dosage was mixed with 100 mL of boron solution, and the pH was adjusted using 0.1 M NaOH or H_2SO_4 [7]. Batch experiments were conducted in 250 mL Erlenmeyer flasks on a hot plate with a magnetic stirrer, wrapped in aluminium foil to prevent splashes. Agitation was maintained at 200 rpm, with ambient temperature and a 60-minute contact time. Afterward, the flasks were left undisturbed for 10 minutes to allow particle settling [8].

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Samples were filtered through 0.45 µm fiberglass membrane filters before analysing boron removal efficiency, calculated using Eq. 1.

$$Boron\ removal\ (\%) = \frac{C_i - C_f}{C_i} \times 100 \tag{1}$$

C_i and C_f are the initial and final boron concentrations of the solutions (mg/L), respectively.

2.3 Adsorption Isotherm

Equilibrium isotherm experiments were conducted by mixing different initial boron concentrations (20–100 mg/L) with a fixed adsorbent dosage of 6 g/L in a 500 mL volumetric flask. The samples were maintained at ambient temperature, pH 8, with a contact time of 60 minutes and shaking at 200 rpm. Afterward, the samples were filtered, and 1 mL of the supernatant was analysed to determine the remaining boron concentration. The adsorption capacity of Moringa Oleifera was calculated using the Eq. 2

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{2}$$

where C_0 is the initial concentration (mg/L), Ce is the final concentration (mg/L), V is the volume of the solution (L), and m is mass of adsorbent (g). Linearized equation for Langmuir isotherm Eq. 3

$$\frac{C_e}{q_e} = \frac{1}{KQ_m} + \frac{C_e}{Q_m} \tag{3}$$

where, qe is the quantity of adsorbate adsorbed in mg/g, Ce is the equilibrium concentration (mg/L), K is the constant of adsorption equilibrium (L/mg) which is related to the energy of adsorption, and Qm (mg/g) is the maximum adsorption capacity. Linearized equation for Freundlich isotherm Eq. 4

$$logq_e = logK_F + \frac{1}{n}logC_e \tag{4}$$

here KF ($mol^2\ /KJ^2$) is an indicator of adsorption capacity and 1/n (mg/L) is a measure of intensity of adsorption.

2.4 Kinetic Isotherm

Batch adsorption studies were performed by adding fixed amount of adsorbent (6 g/L) to a volumetric flask containing 100 mL of boric acid solution with an initial boron concentration of 60 mg/L at pH 8 and stirring at 200 rpm at ambient temperature for varied contact times. The sample was taken out every 20 minutes until 100 minutes. Following the reaction, the samples were removed and allowed to settle at room temperature for 10 minutes. The supernatant was then removed, filtered with SFM 102 filter paper, and 1 mL of the aliquot was used to determine the quantity of boron left in the sample. The pseudo-first-order kinetic model and pseudo-second order are often used to investigate the boron adsorption kinetics. The linear form of pseudo-first order was stated in Eq. 5

$$ln\ ln\ (q_e - q_t)\ = lnq_e - k_1 t \tag{5}$$

Linearized equation for pseudo-second order Eq. 6

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$$\frac{t}{q_t} = \frac{1}{k_2 {q^2}_e} + \frac{t}{q_e} \tag{6}$$

where, q_e (mg/g), while the adsorption capacity at time t is represented by q_t (mg/g). The rate constant is denoted by k_1 (min–1). The adsorption rate constant is denoted by k_2 (g/mg·min) [9].

3. RESULT & DISCUSSION

3.1 Design of experiment

Table 1 presents the Design of Experiment (DOE) for boron removal using activated *Moringa oleifera* seed under different operating conditions of pH, adsorbent dosage, and initial boron concentration. The results indicate that the adsorption performance was significantly influenced by the interaction between these parameters. The highest boron removal efficiency of 51.67% was achieved at pH 8 with an adsorbent dosage of 6 g/L and initial boron concentration of 60 mg/L. The optimum boron removal observed at pH 8 in this study agrees with previous findings reported by Desta and Bote [10], where *Moringa oleifera* seed powder showed the best treatment performance within the pH range of 7–9. This suggests that alkaline conditions favor boron adsorption due to the increased interaction between borate ions and active functional groups available on the adsorbent surface.

Table 1: Design of experiment (DOE)

Run	Block	Factor 1 A:pH	Factor 2 B:Adsorbent dosage g/L	Factor 3 C:Initial boron concentration mg/L	Response: Percentage of removal %
1	1	9	8	100	21
2	1	7	6	60	35
3	1	7	8	20	40
4	1	8	6	60	51.67
5	1	8	6	20	48
6	1	8	8	60	45
7	1	8	6	60	51.67
8	1	8	6	100	40
9	1	8	4	60	43
10	1	8	6	60	51.67
11	1	7	4	100	28
12	1	8	6	60	51.67
13	1	8	6	60	51.67
14	1	8	6	60	51.67
15	1	7	8	100	30
16	1	9	4	20	5
17	1	7	4	20	35
18	1	9	8	20	10
19	1	9	4	100	15
20	1	9	6	60	18

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Table 2 displays ANOVA results for significant model terms with an F-value of 47.53 and a low probability value "Prob > F" less than 0.0500, indicating model significance. The lack of fit is not significant, which is beneficial to the model. A, A^2, AC, are crucial in this investigation.

Table 2: Analysis of variance for significant model terms.

Source	Sum of squares	Df	Mean squares	F value	Probe >F
Model	4404.87	9	489.43	47.53	< 0.0001
A	980.10	1	980.10	95.18	< 0.0001
B	40.00	1	40.00	3.88	0.0770
C	1.60	1	1.60	0.16	0.7017
A^2	1197.16	1	1197.16	116.26	< 0.0001
B^2	31.13	1	31.13	3.02	0.1127
C^2	31.13	1	31.13	3.02	0.1127
AB	2.00	1	2.00	0.19	0.6688
AC	180.50	1	180.50	17.53	0.0019
BC	0.50	1	0.50	0.049	0.8300
Residual	102.97	10	10.30		
Lack of Fit	102.97	5	20.59		
Pure Error	0.000	5	0.000		

Table 3 shows the ANOVA results for the selected factorial model used in this study. The model obtained a high R^2 value of 0.9772, indicating that the model fits the experimental data well. The adjusted R^2 and predicted R^2 values of 0.9566 and 0.8644 also show good agreement between the experimental and predicted results. In addition, the adequate precision value of 19.653 is higher than 4, indicating that the model has an acceptable signal-to-noise ratio and can be used for optimization purposes. The insignificant lack-of-fit further confirms that the model is suitable for describing the boron adsorption process using activated *Moringa oleifera* seed.

Table 3: Analysis of variance for selected factorial model

Parameters	Value
Standard deviation	3.21
R-squared	0.9772
Adj R-squared	0.9566
Pred R-squared	0.8644
Adeq Precision	19.653

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3.2 Adsorption Isotherm

3.2.1 Langmuir Isotherm

Figure 3 depicts a linear plot of the Langmuir isotherm model, represented as C_e/q_e versus C_e. It was observed from Figure 3 that the R^2 value was 0.879 which means that Langmuir isotherm does not correlate with experimental data. The slope of the graph indicates $1/q_m$ which is 0.8329 with y-intercept $1/Kq_m$ is -16.214. The result may be due to the heterogeneous surface and different active sites available on the adsorbent. Aziz et al. [7] stated that the adsorption data of *Moringa oleifera* and *Musa cavendish* adsorbent cannot be fitted with the Langmuir isotherm model. This finding suggested that the boron was not interacting well with the monolayer of the adsorbent sites for adsorption.

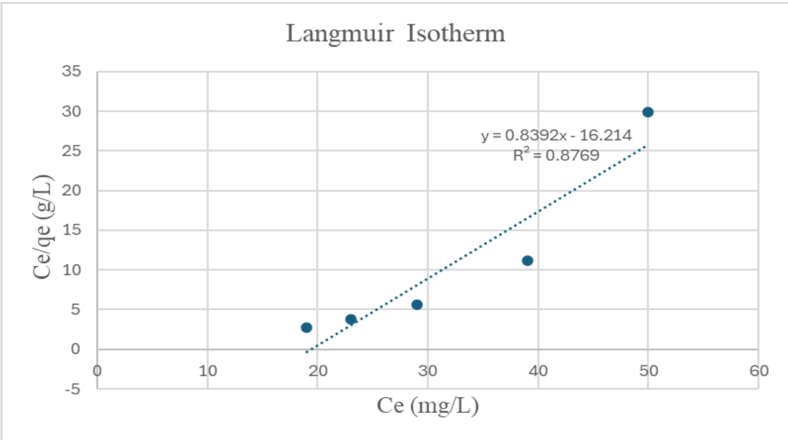

Figure 3: Langmuir isotherm

3.2.2 Freundlich Isotherm

Figure 4 depicts the Freundlich isotherm model. Indeed, the Freundlich isotherm has an R^2 value of 0.9038, showing that boron adsorption on activated *Moringa oleifera* seed is favourable. The slope of the graph indicates $1/n$ which is -1.3923 with y-intercept K_F is 6.1658. Therefore, equilibrium data is fitted better with Freundlich isotherm for boron adsorption on activated *Moringa oleifera* seed. Indeed, Aziz et al. [7] reported that the experimental plots of Pb adsorption by MO, MC and MO+MC, were best fitted with the Freundlich model with R^2 values of 0.97, 0.99 and 0.99, respectively. Therefore, it supports the findings of boron adsorption on activated *Moringa oleifera* seed value of Freundlich isotherm which is 0.9038 was better than Langmuir isotherm which is 0.8769. Hence, the finding suggested that the boron was interacting well with multi-layer adsorbent sites for adsorption.

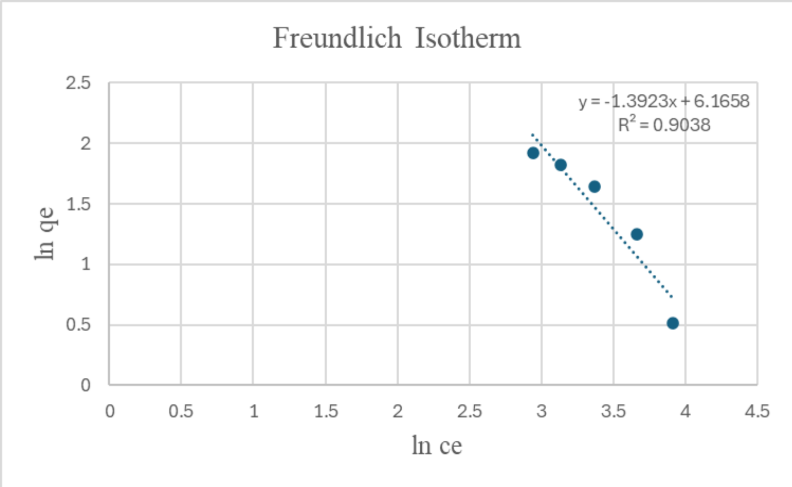

Figure 4: Freundlich Isotherm

3.2 Kinetic Isotherm

3.2.1 Pseudo First Order

Figure 5 depicts a pseudo first order model with an R^2 of 0.75. This kinetic model cannot be fitted to the adsorption process on activated *Moringa oleifera* seed. The slope of the graph indicates $-K_1$ which is -0.0067 with y-intercept ln q_e is 0.7844. This suggests that the adsorption process does not mainly follow pseudo-first-order kinetics. Halim et al. [11] reported that adsorption on *Cur-AC* cannot be fitted with the first pseudo-order with regression (R^2) value of 0.765.

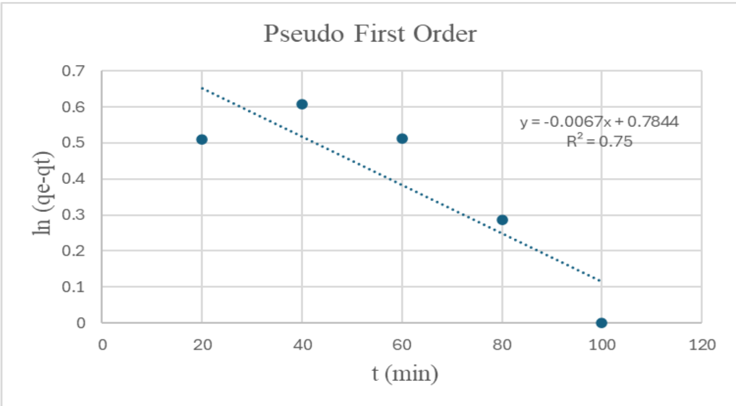

Figure 5: Pseudo-First Order

3.2.2 Pseudo Second Order

Figure 6 depicts a pseudo second order model of adsorption on activated Moringa oleifera seed with coefficient of correlation R^2 value of 0.9106. Thus, the adsorption of boron from aqueous solution on *activated Moringa oleifera* seed is fitted with pseudo second order with coefficient of correlation R^2 of 0.9106. The slope of the graph indicates $1/q_e$ which is 0.5353 with y-intercept $1/K_2q_e^2$ is -9.97. This suggests adsorption kinetics were more likely to adhere to pseudo-second order models. This implies that the physical adsorption and diffusion processes that occur at the surfaces and pores of Cur-AC influenced the boron adsorption kinetics [11]. Therefore, pseudo-second-order kinetics is more suitable for explaining boron adsorption onto activated *Moringa oleifera* seed.

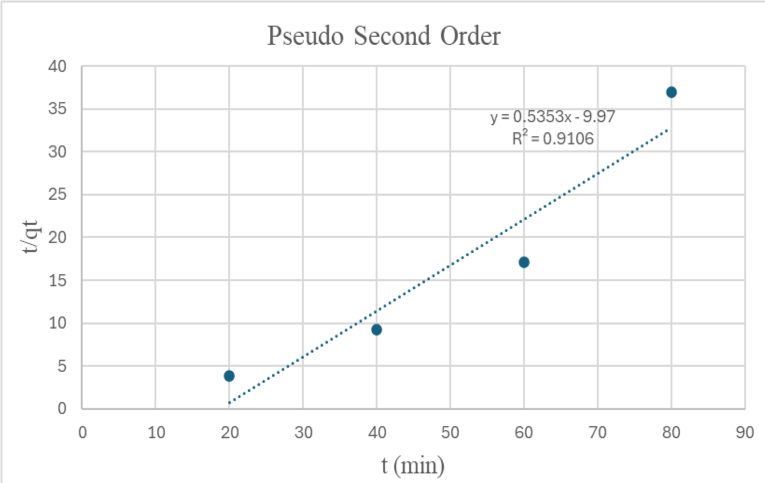

Figure 6: Pseudo second order

4. CONCLUSION

In conclusion, this study demonstrated that Moringa oleifera seeds are an effective adsorbent for boron removal from seawater, achieving a maximum removal efficiency of 51.67% at pH 8, an adsorbent dosage of 6 g/L, and an initial concentration of 60 mg/L. The adsorption process reached equilibrium within 1 hour, with the Freundlich isotherm model and the pseudo-second-order kinetic model providing the best fit for the experimental data, highlighting the potential of Moringa oleifera seeds as a sustainable and efficient solution for boron contamination in wate

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