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USE OF MORINGA-DERIVED ACTIVATED CARBON TO REMOVE BORON FROM SYNTHETIC SEAWATER

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ABSTRACT: Boron has been identified as a contaminant in drinking water across different countries; this is a threat to sensitive plant species and human health. Therefore, regulatory standards require that boron concentrations be lowered to 0.3 mg/L for potable water and between 0.5 mg/L to 1 mg/L for irrigation water. Traditional methods for boron removal are frequently expensive or inefficient at low concentrations. In this study, the feasibility of using *Moringa oleifera* seed husk to produce activated carbon for the removal of boron from synthetic seawater was investigated. The main objective of this study was to determine the boron removal efficiency of various preparation methods of activated carbon produced from *Moringa oleifera* seed husk, (chemical activation and physical activation) through a batch adsorption process with synthetic seawater. The process parameters (adsorbent dose, temperature, and initial boron concentration) of the best sorbent were optimized. The surface properties of the best sorbent were characterized through FTIR and SEM. Equilibrium isotherm and kinetics for the best sorbent based on the optimized parameters were determined. The result showed that chemically activated carbon was found to possess best adsorption for removal of boron from synthetic seawater with 88.02 % at neutral pH and room temperature (25°C±2°C). SEM showed that chemical treatment on *Moringa oleifera* seed husk improves the porosity and the extent of surface functionality of *Moringa oleifera* seed husk. FTIR analysis showed that, hydroxyl (–OH), carboxyl (–COOH), and carbonyl (C=O) groups were present, which involved in adsorption of boron by chemically activated carbon. The highest removal efficiency of 89.72% was obtained at a temperature of 55°C, 0.9 g/L biosorbent dose, and an initial boron concentration of 1 mg/L, obtained using Response Surface Methodology (RSM). The equilibrium data of this study was fitted with Langmuir isotherm with regression value of R^2= 0.9685, while the adsorption kinetics were adequately described by the sequential fitting of the pseudo-second -order models with regression value of R^2= 0.9942.

KEYWORDS: *Boron, Moringa oleifera, Activated carbon, Chemical treatment, Adsorption.*

1. INTRODUCTION

Boron naturally occurs in many environmental compartments, such as water bodies. Although it is needed only in trace amounts, higher levels of boron can cause phytotoxicity in plants and pose potential health risks to humans [1]. To reduce these adverse effects, organizations such as the World Health Organization (WHO) have established guideline limits for boron concentration in water, recommending a maximum concentration of 2.4 mg/L in drinking water and approximately 1 mg/L in irrigation water [2,3]. However, due to its high solubility and chemical stability in water, removing boron from aqueous environments remains

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a significant challenge. The desalination by reverse osmosis (RO) and ion exchange is expected to remove most of the dissolved solids, however it is not that efficient when it comes to boron. Economic and environmental limitations of current desalination technologies, particularly in boron removal, there is a significant interest in exploring more sustainable and cost-effective alternatives. Using activated carbon to increase boron adsorption rate is another promising direction. This activated carbon obtained from natural and renewable sources such as Moringa Oleifera is a potential method., a plant that has already proven to be effective in eliminating water pollutants [4]. The high surface area and porosity of Moringa Oleifera seed husks make them an excellent source of activated carbon that may be used to effectively absorb a variety of contaminants.

Therefore, the focus of this study is to assess the ability of several activated carbon preparation methods, such as physical, chemical activations, to adsorb boron using a batch adsorption procedure utilizing synthetic seawater polluted with boron. Then the optimal process parameters determined for the most effective sorbent out of the two different methods used to prepare activated carbon. The surface properties of the optimal sorbent are characterized using FTIR and SEM. Under various operating conditions, including adsorbent dose, temperature, and initial boron concentration, the effectiveness of the best activated carbon in removing boron examined.

2. MATERIALS AND METHODS

2.1 Materials

Moringa oleifera seed husks (MOSH) are a biomass precursor used to produce activated carbon, obtained from the MitoMasa company in Selangor, Malaysia. After collecting seed husks, it was washed several times with tap water and then with distilled water to remove dust, oil residue, and other surface impurities. The washed husks were subsequently dried in an oven at a constant temperature of 60 °C for 48 hours until a steady weight was attained. Samples were dried and grind using a mechanical grinder and sieved to get a particle size of less than 106 µm.

The chemical reagents involved potassium hydroxide (KOH) purchased from R&M Chemicals (Malaysia). To prepare MOSH for chemical activation, it was reacted at a weight ratio of 1:1 with KOH. HCl to remove any residual from the AC.

2.2 Activated Carbon Preparation

The activation process involved mixing 10 g of dried seed with KOH ration of 1:1. Then 80 mL distilled water was added and the mixture poured into a rotary shaker and shaken at 50 °C, for an hour. The samples were dried using an oven at 100 °C temperature and overnight to result in a thick paste. The samples were subsequently introduced into a Carbolite horizontal tube furnace and were heated to 500 °C in 1 hr, at 10 °C / min under a constant flow of nitrogen gas. The process of carbonisation was performed in a horizontal tubular quartz reactor (England, UK) provided with the controller, the pressure regulating valve, and the Carbolite horizontal tube electrical furnace (Model CTF, UK). The end products were then taken to a desiccator to cool off finally.

The second activation method is the raw moringa oleifera seed husk subjected to carbonization in a furnace at 500 °C temperature using same condition for 0.5 hr then activation step at 600 °C for 1 hr under a constant flow of nitrogen gas at 10 °C / min. The sample was then cooled to ambient temperature (20 °C) under the same nitrogen flow rate.

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Removal of the remaining activating agents was by washing the produced activated carbon with hot distilled water until the pH remained neutral. Then, the washed samples were dried at 105 °C and then put in closed containers to carry on with further analysis. All the other chemical reagents employed were of analytical purity without further purification. The synthesis of seawater with boron was made by using boric acid (H_3BO_3) that was bought at R&M and diluted in deionized water to resemble marine environments infected with boron.

2.3 Boron Adsorption Studies

To determine the most effective adsorbent for boron removal, different preparation methods of Moringa oleifera seed husk were investigated, including raw Moringa oleifera seed husk, physical activated carbon, and chemically activated carbon. Preliminary batch adsorption experiments were conducted under constant operating conditions to screen the best adsorbent prior to the optimization study using Response Surface Methodology (RSM). In each experiment, 0.9 g/L of adsorbent was added to 25 mL boron solution with an initial concentration of 5 mg/L at neutral pH (7). The mixtures were agitated at 150 rpm at room temperature (25 ± 2 °C) for 2 h. The adsorbent showing the highest boron removal efficiency was selected for further optimization and adsorption studies.

2.4 Characterization of the Carbonized Carbon

2.4.1 Fourier Transform Infrared (FTIR)

FTIR Fourier transform infrared spectroscopy has been used to examine the surface chemistry and functional groups of the activated carbon of the Moringa oleifera seed husk raw, activated chemically, as well as activated by physical. It was done to determine the functional group and analyze the surface chemistry. This was done using a Perkin Elmer Frontier FTIR spectrometer. Samples were dried in air, finely ground in small quantities, and compressed into transparent pellets appropriate to infrared analysis. Each sample was noted for its percentage transmittance (or absorbance) in the spectral range between 400 and 4000 cm^{-1}. This characterization allowed determining some important active groups, like hydroxyl (OH-), carboxyl (COOH-), and carbonyl (CO-), that were critical to boron species binding via surface interactions [5].

2.4.2 Scanning Electron Microscope (SEM)

SEM (scanning electron microscope) used to determine the surface morphology and porosity of raw moringa husk, chemical activated carbon, and physical activated carbon. The samples set on carbon tape, and JEOL-IT100 SEM (JEOL, Japan) used to capture the pictures. The JEOL IT100 SEM's InTouchScopeTM version 1.060 measurement tool used to measure the size of the pores.

2.5 Optimization of Boron Adsorption

The optimization of boron adsorption using chemically activated carbon derived from Moringa oleifera seed husk was performed using Response Surface Methodology (RSM) through Design-Expert® software (Version 11.0). Three independent variables were selected: temperature (35, 45, and 55 °C), initial boron concentration (1, 3, and 5 mg/L), and biosorbent dose (0.3, 0.6, and 0.9 g/L), while the response variable was the percentage removal of boron. The selected parameter ranges were based on previous boron adsorption studies. The experiments were conducted in batch mode at pH 7, agitation speed of 150 rpm, and contact time of 2 h.

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2.6 Adsorption Isotherm

Adsorption was carried out using adsorption isotherm to ascertain the adsorption equilibrium of boron on the moringa derived activated carbon using optimized conditions. The isotherms at equilibrium were performed in batch analysis by diluting various initial concentrations of boron, 1, 3, 5, 7, 9 mg/L, in 25 mL volumetric flasks containing a fixed amount of 0.9 g/L of biosorbent at room temperature (55 °C), pH 7, 2 hours contact period, and shaking rate of 150 rpm. The samples taken out of the shaker after 2 hours were taken to room temperature and allowed to sediment overnight i.e. 24 hours. After that, the samples were filtered, and 1 mL of the supernatant was filtered with the remaining boron concentration in the solution after adsorption being measured [6]. Langmuir and Freundlich are the most common studied adsorption process equilibrium isotherm models encountered in this study.

The linear form of the Langmuir equation can be written as follows (1):

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (1)$$

Meanwhile, for the linearization of the Freundlich model is (2):

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

2.7 Adsorption kinetics Studies

The experiments were performed under the optimized conditions obtained from the RSM study. Batch adsorption experiments were carried out using 25 mL boric acid solution containing 0.9 g/L biosorbent at pH 7 and temperature of 55 °C with agitation speed of 150 rpm. Samples were collected at different contact times of 30, 60, 90, 120, and 180 min to evaluate the adsorption behavior and determine the adsorption rate. The adsorption kinetics were analyzed using pseudo-first order and pseudo-second-order kinetic models.

The models in kinetics were adopted to correlate the mechanism of control, the behavior of the adsorption process, and the prediction of the rate at which the adsorbate was adsorbed out of solution. The experiments were performed under the optimized conditions obtained from the RSM study. Two different kinetic models have been used in this research, which are the pseudo-first order and pseudo-second order. Adsorption kinetics study was performed in a volumetric flask of 25 mL of boric acid mixed with adsorbent dose 0.9 g /L balanced in a shaker at 150 rpm, pH 7, 55 °C temperature level, and the samples were collected at different contact times of 30, 60, 90, 120, and 180 min.

The supernatant was removed, then filtered with (Structure from Motion (SFM) 102 filter paper. 1 mL of the aliquot was taken to measure the remaining boron in the sample.

A pseudo-first-order linear equation is determined using the following equation:

$$ln\ (q_{e-}q_{t)} = ln\ q_e\ + K_1\ t \quad (3)$$

The linear form of pseudo-second order can be formulated as following:

$$\frac{t}{q_t} = \frac{1}{(K_2 {q_e}^2)} + \frac{1}{q_e}\ t \quad (4)$$

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3. RESULTS AND DISCUSSION

3.1 Screening of Adsorption Potentials of Various Preparation Methods of

3.1.1 Boron adsorption Studies

Different methods of preparing the activated carbon give different percentage of boron removal. Chemically activated carbon better than the raw moringa seed husk and the physical activated carbon in terms of boron adsorption. The percentage of removal 88.02%, 70.87%, and 59.56% for chemically activated carbon, physical activated carbon, and raw moringa seed husk, respectively as shown in fig.1.

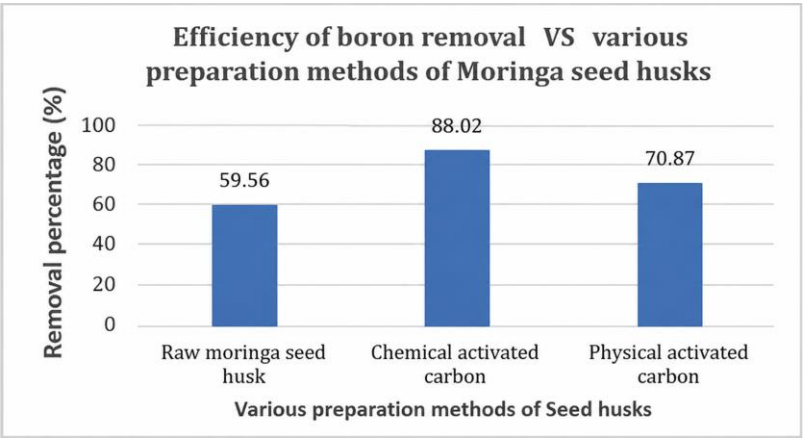

Fig. 1: Removal percentage as function of various preparation methods of AC

3.2 Characterization of Adsorbent

3.2.1 Morphological Structures of the Activated Carbon

The surface morphology of the raw Moringa oleifera seed husk (MOSH) and chemically activated carbons was analyzed with SEM. The structural deformations, porosity, and alterations in the surface texture can be observed in SEM pictures obtained when activating using different methods, Fig. 2 (a). The surface seems to be thick and linked together meaning that a lignocellulosic network exists. This establishes that the raw material possesses small surface area and poor availability to adsorption as it should be without activation.

Once activated, there is an expected change in morphology. The SEM photomicrographs of chemically activated MOSH in Fig. 2 (b) show a very porous structure using the SEM image. The surface appears rugged and irregular, highly microporous, highly mesoporous. This high pore formation shows that the KOH activation successfully pulled out the volatiles and dented the lignocellulosic structure, making it more porous. This improved pore development is important in adsorption applications, and this serves to increase the ability of the materials to trap and hold contaminants.

The SEM microstructure of physically activated MOSH is presented in Figure 2(c). Compared with chemical activation, a less well-defined pore structure with more cracked and fibrous surface morphology is obtained with physical activation. During thermal treatment, organic components are destroyed leaving observable cracking and fissures across the material.

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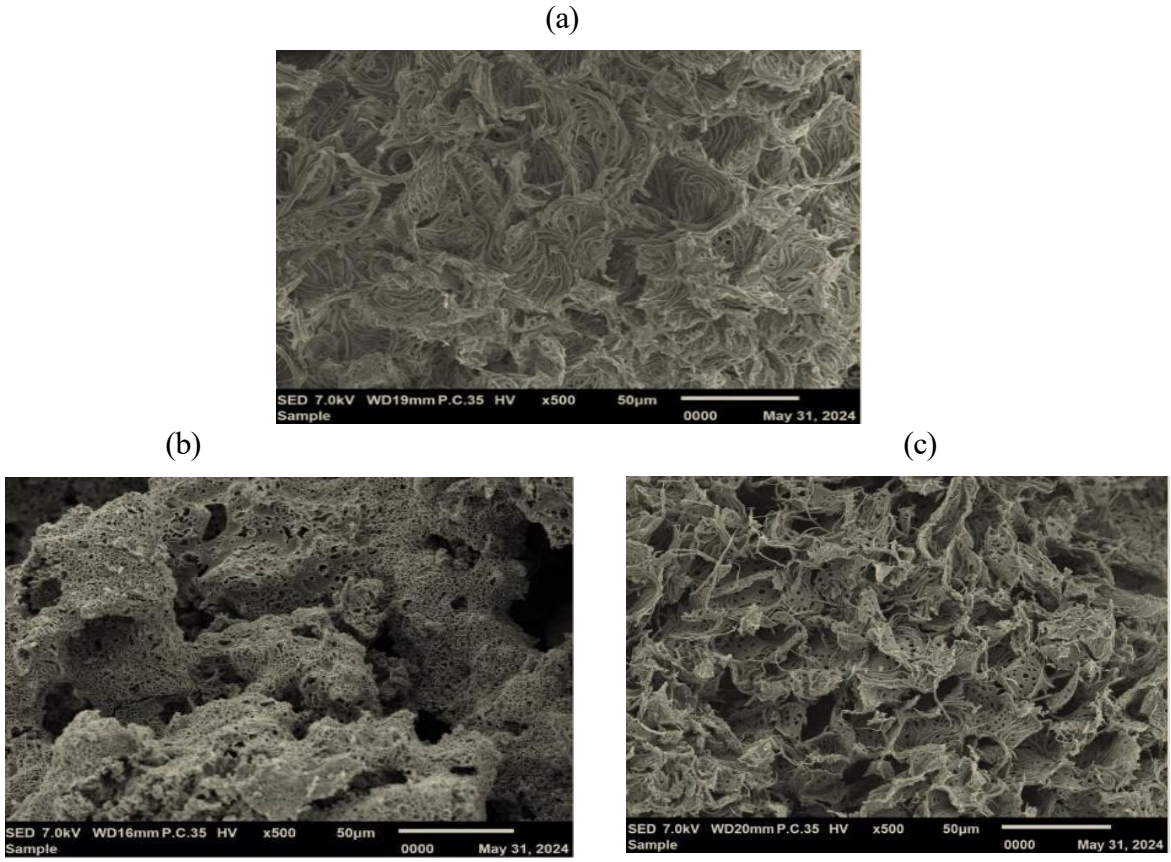

Fig .2: SEM photomicrographs of (a) Raw moringa oleifera seed husk, (b) Chemically activated MOSH and (C) Physical activated MOSH

3.2.2 Surface Chemistry

FTIR was used to analyse the functional groups present on the surface of raw and activated Moringa oleifera seed husk. FTIR spectrum of the raw material showed a great number of functional groups that are related to lignocellulose biomass, such as broad O-H stretching signals (3475-3182 cm^{-1}), aliphatic C-H stretching peaks (2925 and 2858 cm^{-1}), and one C=O stretch peak (1731 cm^{-1}) that relates to carbonyl-containing compounds. The lignin was confirmed in aromatic C=C and C-H vibrations (1600-1450 cm^{-1}), and the presence of esters, ethers, and polysaccharides was determined in C-O and C-O-C (1110 and 1045 cm^{-1}), Fig .3.

Such groups of functions, specifically the hydroxyl (OH), carboxyl (COOH), carbonyl (C=O), and ether (C-O-C) ones, are reported to be of great importance in the mechanism of adsorption. Hydroxyl and carboxyl present the creation of hydrogen and ion exchange of pollutants, and carbonyl and ether groups provide polarity of the surface via the formation of dipoles. π-π interactions are also made possible by aromatic C=C bonds, mainly in the presence of organic molecules.

On activation, substantial alterations in spectral characteristics were realized. The functional groups were O-H stretch (3436 cm^{-1}), aliphatic C-H (alkyl group) was diminished, aromatic, and functional groups (such as C=C 1574 cm^{-1} and C-O 1045 cm^{-1}) remained intact,

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which are crucial in adsorption. However, physical activation of carbon led to a decrease in quantity and intensity of peaks, which suggests the destruction of functional groups through high temperatures during carbonization. This left a mostly carbon-based based chemically inert surface.

Comparing the two approaches, it also appears that chemical activation (e.g., using KOH) tends to preferentially maintain or insert important surface functional groups that are desirable for adsorption, whereas physical activation could be used to preferentially improve porosity, but inhibits access to surface reactivity. Thus, it is of the essence that the functional groups of –OH, -COOH, and C=O are not destroyed to enhance the adsorption capability of the contaminants, dyes, and heavy metals.

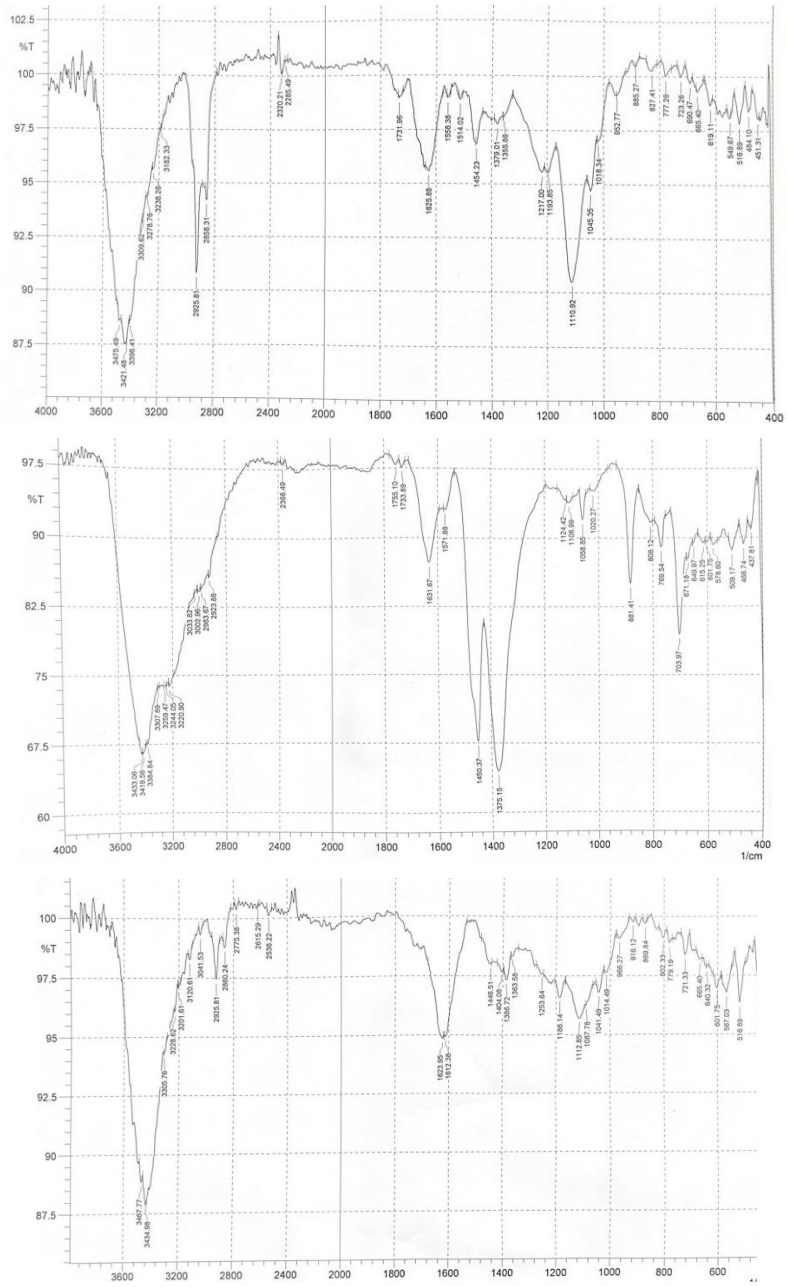

Fig. 3: Fourier transform infrared spectrum for Raw moringa oleifera seed husk, chemically activated MOSH and Physical activated MOSH

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3.3 Optimization

The removal of boron using chemical activated carbon was statistically analysed through Design Expert software to determine the optimum conditions for boron adsorption. In this study, three factors used in the optimization process, temperature (35, 45, and 55 °C), initial boron concentration (1, 3, and 5 mg/L) and biosorbent dose (0.3 ,0.6, and 0.9 g/L), as shown in Table 1.

Table 1: Design of experiment (DOE)

Run	**Factor1** Temperature °C	**Factor2** Initial Concentration mg/L	**Factor 3** adsorbent Dose g/L	Removal percentage
1	45	3	0.9	87.60
2	35	1	0.3	80.59
3	55	1	0.9	89.72
4	55	1	0.3	81.84
5	45	3	0.3	76.21
6	55	5	0.3	71
7	55	5	0.9	87.29
8	35	1	0.9	88.28
9	35	5	0.9	82.89
10	45	1	0.6	87.08
11	35	3	0.6	83.07
12	55	3	0.6	83.90
13	45	5	0.6	82.68
14	45	3	0.6	84.47
15	35	5	0.3	69.02

Table 1 it is obvious that maximum boron removal 89.72% was found at temperature of 55 °C, initial boron concentration 1 mg/L, biosorbent dose 0.9 g/L. m. To determine the optimum adsorption conditions, the regression model was developed to help identify the effect of individual factors on boron removal by chemical activated carbon.

The optimization experiment employed based on the Response Surface Methodology (RSM) demonstrated that temperature, initial concentration of boron, and biosorbents dose were important variables influencing the removal efficiency of boron, with the highest reduction (89.72 %). The removal went higher in elevated temperatures and lesser amounts due to the increased rate of reactions and the adsorption strength that is endothermic, [7] and [8]. Increase the temperature and the dose of biosorbent contributed to the increased efficiency as it stimulated the adsorption sites and improved the mixing processes, as [9] reported it in his work. The correlation between the biosorbent dose and initial concentration was observed to be negative, where a higher dose was more active at lower concentrations since less competition on the active sites [10]. Overall, the dose of adsorbent was the most influential parameter, initial concentration of boron, and temperature.

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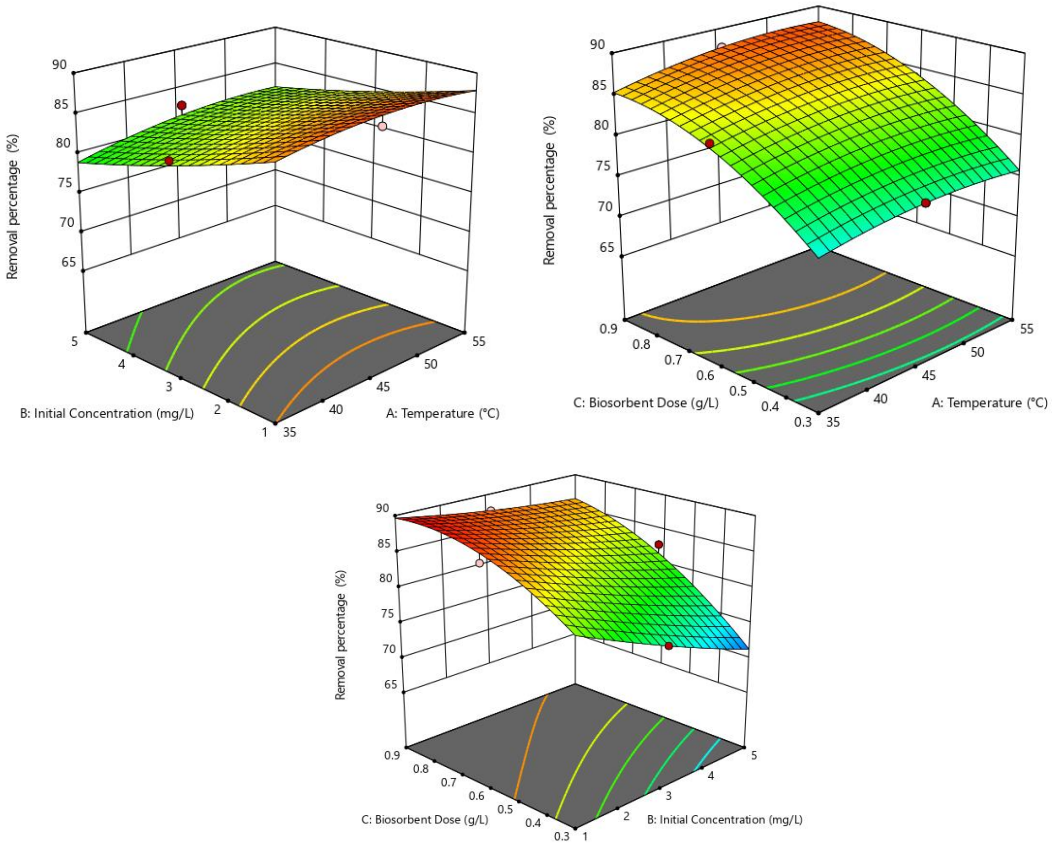

Fig. 4: The 3D Surface Plot of the Interaction Between the Three Factors

3.4 Adsorption Isotherms Models

The distribution of the adsorbate molecules between the liquid and solid phases has been displayed by use of adsorption isotherms. That is usually important to the design of the adsorption system. Adsorbed chemical concentration (mg/g) and remainder of the solution (mg/L) are normally plotted to show the results of adsorption isotherms. In this work, the data of adsorption equilibrium was modelled to Langmuir and Freundlich isotherms.

3.4.1 Langmuir Isotherm

Langmuir isotherm model proposes a single layer with non-transmigration of the sorbate adsorption on the surface without interaction between the sorbed molecules and the constant energy of absorption [11]. Linear form of the Langmuir model shown in Eq. (5), and a plot of C_e/q_mvs. C_e is shown in Fig. 5. The maximum adsorption capacity (q_m) and adsorption rate (K_L) are determined by using the slope and the interception respectively.

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \tag{5}$$

where K_L (L/mg) and q_m (mg/g) are Langmuir constants related to rate of adsorption and adsorption capacity, respectively. C_e is the equilibrium concentration of the adsorbate (mg/L).

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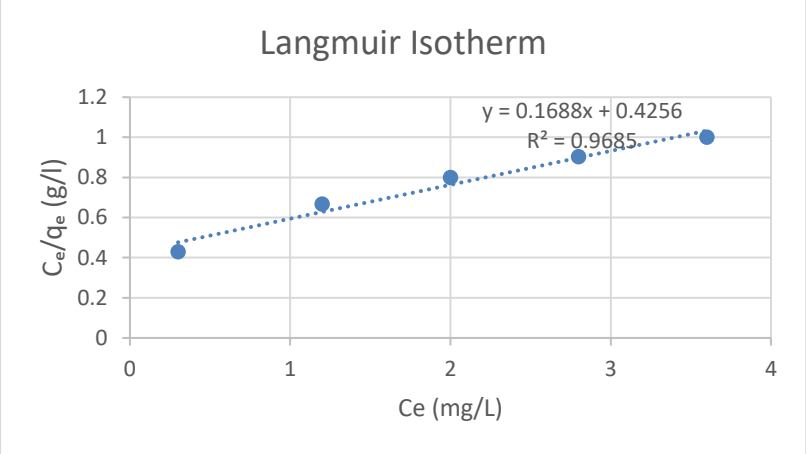

Fig. 5: Langmuir isotherm plot for the removal of boron

3.4.2 Freundlich Isotherm

The Freundlich isotherm is an empirical model based on heterogeneous adsorption along with sites of varying energies [12]. The linear form of the Freundlich isotherm is expressed as:

$$log\ q_e = \log K_f + \frac{1}{n}\ log\ C_e \quad (6)$$

where q_e is the amount of boron adsorbed at equilibrium (mg/g); $C_e$is the equilibrium concentration of the adsorbate (mg/L); K_f ((mg/g) (L/mg)$^{1/n}$) and n is Freundlich constants relating respectively to the adsorption capacity of the adsorbate and the favourability of adsorption process. A plot of $log\ q_e$ vs. $log\ C_e$ is shown in Fig. 6

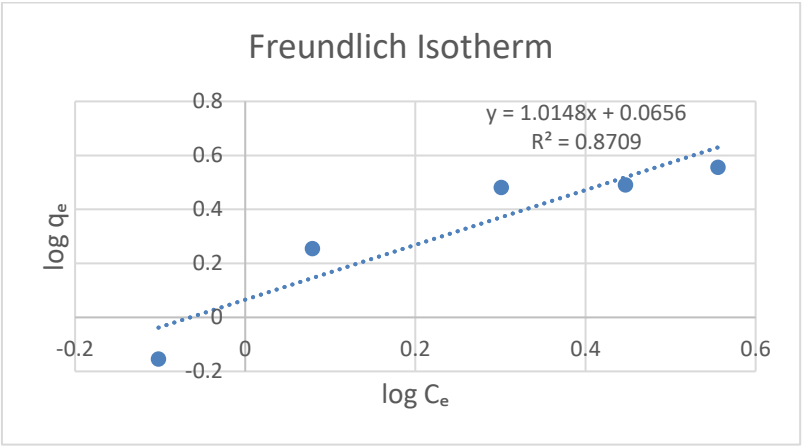

Fig. 6: Freundlich isotherm plot for the removal of boron

The R^2 values indicating the favorability of boron adsorption on chemically activated carbon. The values were 0.8709 and 0.9685 for Freundlich and Langmuir isotherms, respectively. Therefore, the data in equilibrium fits the Langmuir isotherm better. This suggests that adsorption occurred on a flat surface and there was no transmigration of boron.

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3.5 Adsorption Kinetic Studies

The experimental data that depend on time were analyzed to determine the rate-limiting step by fitting them against various kinetic models, namely the pseudo-first-order model [13] and the pseudo-second-order model. The pseudo-first-order and pseudo-second-order equations are linear equations in the form of Eq. (7) and Eq. (8), respectively.

$$ln\,(q_{e-}q_{t)} = ln\,q_e\ + K_1\,t \tag{7}$$

$$\frac{t}{q_t} = \frac{1}{(K_2 q_e{}^2)} + \frac{1}{q_e}\,t \tag{8}$$

The correlation coefficient (R^2) values of pseudo-first-order and the pseudo-second-order, 0.9535 and 0.9942, respectively. Adsorption of boron from aqueous solution by chemical activated carbon is fitted with pseudo second order, as shown in Fig. 8

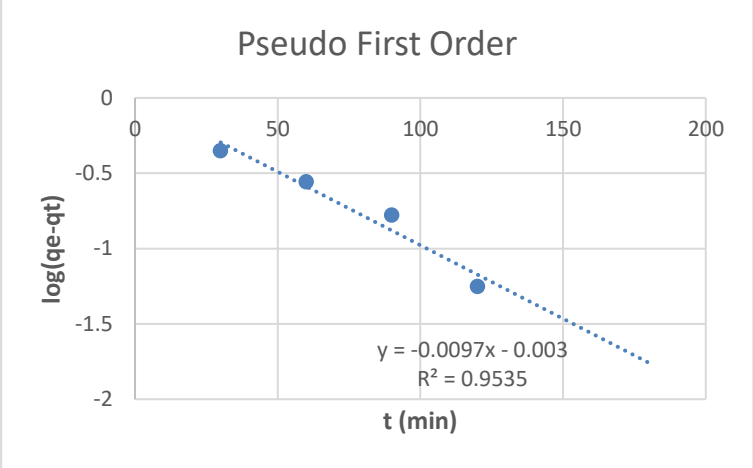

Fig. 7: Pseudo First Order

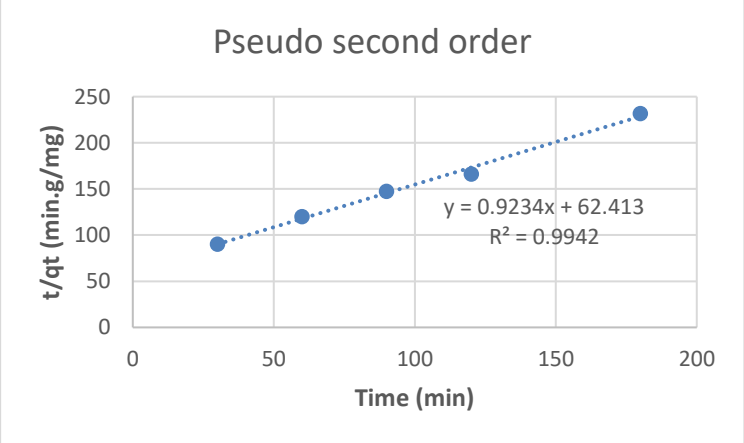

Fig. 8: Pseudo Second Order

4. CONCLUSION

This investigation revealed the fact that activated carbon prepared via the husks of the seeds of Moringa oleifera, especially obtained through chemical activation with KOH, is an excellent biomaterial with adsorption capabilities in relation to the removal of boron from synthetic seawater. The maximum removal efficiency of 89.72 percent was obtained by chemically activated carbon under optimized conditions of 55 °C, 0.9 g/L adsorbent dose, and 1 mg/L initial boron concentration. The presence of functional groups, including hydroxyl, carboxyl, and carbonyl, was confirmed by FTIR, as well as by SEM, which showed an increase in porosity and surface roughness because of chemical activation. The adsorption data were found to conform to the Langmuir isotherm (R^2= 0.9685), which implies the monolayer adsorption, and the kinetic results obeyed the pseudo-second-order model (R^2= 0.9942), which implies chemisorption as the rate-limiting step. Generally, the results indicate that chemically activated Moringa oleifera seed husks demonstrate potential as a cost-effective and sustainable adsorbent in the removal of boron during water treatment processes.

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