SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Applied Sciences and Engineering Technology

Journal homepage:
https://semarakilmu.com.my/journals/index.php/applied_sciences_eng_tech/index
ISSN: 2462-1943

Isotherm, Characterisation and Regeneration Studies for the Adsorption of Pb(II) Ions in Water

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ARTICLE INFO

Article history:
Received 12 December 2022
Received in revised form 3 January 2023
Accepted 25 January 2023
Available online 16 February 2023

Keywords:
Regeneration; KENAF adsorbent; Heavy metals; Isotherm; Characterisation

ABSTRACT

The adsorption isotherms and regeneration study of the removal of Pb(II) ions from water using kenaf-chitosan-alginate (KNF-CHT-ALG) beads were evaluated. The effects of initial Pb(II) ions concentration on the adsorption capacity of KNF-CHT-ALG beads were carried out in a batch study mode and analysed using the Inductively Coupled Plasma (ICP) technique. In the present research, the linear models of Langmuir and Freundlich were used to predict the adsorption isotherms. The adsorption process was excellently well fitted with the Langmuir isotherm model. The maximum adsorption capacity recorded 33.557 mg/g. For the regeneration study, after five times of the recycling process, the KNF-CHT-ALG beads still showed good adsorption towards Pb(II) ions with maximum removal of 95% and regeneration of 98%. Clearly, from the research conducted, the KNF-CHT-ALG beads are found to be a suitable adsorbent for Pb(II) ions removal.

1. Introduction

The discharges of untreated industrial wastewater containing high amounts of heavy metal are highly toxic at low concentrations and can affect human, animals and the environment. Promptly, appropriate removal of heavy metal is crucial, but common traditional methods such as solvent extraction, coagulation, ion exchange and electrochemical technologies are suffering from high instrumentation cost, inadequate removal and energy consumption [1]. Among all the used method to remove heavy metals, adsorption is known to be flexible in the operation and design process [2].

A study by Florence *et al.,* [3] on the novel of chitosan/kenaf fibres as a good adsorbent for metal ions uptake had attained 89% of Nickel ions. In another study, Zinc chloride $Zn(Cl_2)$, used as a carbonising promoter for kenaf core in treating heavy metal contamination, removing up to 91.2%

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https://doi.org/10.37934/araset.29.3.175184

manganese ions [4]. Both studies are good benchmarking for the current study. However, with many benefits offered by kenaf as a natural sorbent, the gap is that no study has been conducted on (KNF-CHT-ALG) hydrogel composites as the adsorbent. It is interesting to note that many previous studies recommended that chitosan/kenaf composite is an effective and low-cost adsorbent for the adsorption of metal ions from water.

In the present work, the researcher proposes an adsorption method to remove Pb(II) ions in water by (KNF-CHT-ALG) beads using batch mode experiments. Recently, kenaf has attracted attention as green and renewable biomass with hydroxyl (-OH) functional groups that help the adsorption process of heavy metal [5,6]. Chitosan also has attracted high consideration due to its various functional groups such as hydroxyl (-OH) and amino groups ($-NH_2$) that are capable to adsorb various heavy metal ions [7]. However, it has a limitation of swelling effect, especially under acidic conditions that limits the metal ion uptake. To overcome the weakness, this study also utilised sodium alginate (ALG), a biological polysaccharide, as the cross-linking agent that enhanced the hydrophobicity of chitosan [8].

The objectives of this study include the evaluation of the maximum adsorption capacity of the synthesized adsorbent and also the regeneration study of the adsorbent in the adsorption of Pb(II) ions in water. For the adsorption capacity, the effects of initial Pb(II) ions concentration are investigated. On the other hand, the linear models of Langmuir and Freundlich will be fitted to the experimental data in order to develop equilibrium isotherms. Adsorption isotherms are of importance to understand the relationship between the Pb(II) ions and the KNF-CHT-ALG beads. The regeneration study of the KNF-CHT-ALG beads after a few adsorption-desorption cycles is also reviewed.

2. Methodology

2.1 Materials

Lead (II) nitrate $Pb(NO_3)_2$ purchased from R&M Chemicals was used for preparing a Pb(II) ions solution in the desired concentrations. For the adsorbent used in this research, the KNF plants were obtained from the Raw Material Collecting Centre (RMCC), which is owned by Lembaga Kenaf Dan Tembakau Negara (LKTN), in Cherating Kuantan, Pahang. In addition, sodium alginate (ALG) and chitosan (CHT) powder purchased from Sigma Aldrich, Germany were also used to produce KNF-CHT-ALG beads.

2.2 KNF-CHT-ALG Beads Synthesis

CHT powder and KNF core powder were added to a sodium alginate (ALG) solution and stirred homogeneously. The blended solution was dripped into 100 ml of calcium chloride ($CaCl_2$) solution to form smooth magnetic KNF-CHT-ALG beads.

2.3 Effect of Initial Pb(II) Ions Concentration

Standard Pb(II) ions solution at a concentration of 1000 mg/L were diluted into desired concentrations prior to use. Then, the KNF-CHT-ALG beads of 0.5 g were mixed with 50 mL of the Pb(II) ions solution in five 250 mL conical flasks having concentrations ranging from 50, 100, 150, 200 and 250 mg/L. At the same time, the pH and contact time were fixed at normal pH at pH 4.43- and 60-minutes agitation time, respectively. After that, the mixtures were centrifuged and filtered. The amount of Pb(II) ions left in the solution after contacting with the beads was analysed using

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The percentage removal was calculated using the following function:

Percentage adsorption (%R) = $(C_0-C_t)/C_0 \times 100$ (1)

where C_0 is the Pb(II) ions concentration before adsorption (mg/L) and C_t is the Pb(II) ions concentration after adsorption (mg/L).

2.4 Adsorption Isotherms

The Langmuir and Freundlich isotherm equations were implemented to the batch equilibrium data. The linear form of both isotherms can be written as follow:

$$C_e/q_e = (1/q_m)(C_e) + 1/q_mK_L \quad (2)$$

$$\log q_e = 1/n \log C_e + \log K_F \quad (3)$$

where C_e (mg/L) is Pb(II) ions concentration after adsorption and q_e is the amount of Pb(II) ions adsorbed by the adsorbent (mg/g). Through C_e/q_e versus C_e plot, $1/q_m$ as slope and $1/q_mK_L$ as intercept were used to compute the values of maximum adsorption capacity, q_m and Langmuir constant, K_L.

From function (3), a line graph was plotted using log C_e as the x axis and log q_e as the y axis, thus the K_F (Freundlich constant) and n (adsorption intensity) values were obtained from intercept and slope [9].

Another important parameter that should be noted in the adsorption study is the separation factor, R_L. This dimensionless constant is derived from the Langmuir isotherm model and is suitable to be used in evaluating the effectiveness of an adsorbent [10]. The separation factor, R_L equation can be expressed as follow:

$$R_L = 1/(1+bC_o) \quad (4)$$

where b is the Langmuir constant (L/mg) and C_o is the initial concentration of the Pb (II) ions (mg/L). The value was correlated with a linear ($R_L = 1$), irreversible ($R_L = 0$) or favourable ($0 < R_L < 1$) process.

2.5 Regeneration Cycles Study

Regeneration cycles study is the environmentally favourable and economically prime important factor for an effective adsorption material. The regeneration of the KNF-CHT-ALG beads was determined up to five times the regeneration process. The desired Pb(II) ion solution was obtained by dissolving 0.8 g of $Pb(NO_3)_2$ salts into 1 L conical flasks. The regeneration study was performed by agitating 0.5 g KNF-CHT-ALG beads in 50 mL of Pb(II) ions solution at 50 mg/L for 60 minutes. After that, the beads were separated from the solution by filtration and washed with ultrapure water before subsequent reused.

3. Results

3.1 Surface Morphology of the KNF-CHT-ALG Beads

The surface morphology of the KNF-CHT-ALG beads is depicted in Figure 1. The KNF-CHT-ALG beads displayed rough and irregular surfaces, indicating the presence of agglutinative flakes of fibrous cellulose materials due to the inclusion of KNF. The coarse structure of the beads indicates it has high porosity that could act as active sites for the adsorption of Pb(II) ions [11].

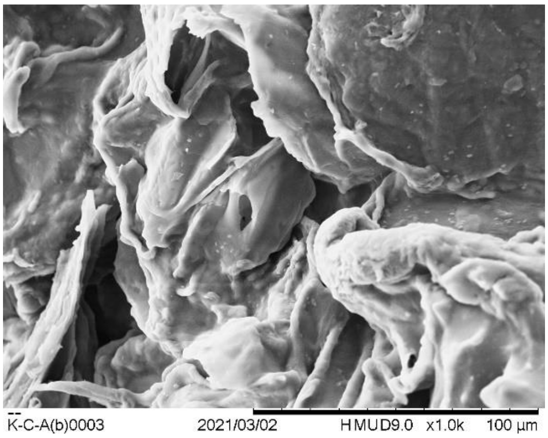


Fig. 1. FESEM images at 1000× magnification and an accelerating voltage of 3.0 kV of the KNF-CHT-ALG

3.2 Functional Groups Spectra of the KNF-CHT-ALG Beads

FTIR analysis was utilised to investigate further the functional groups present in the KNF-CHT-ALG beads to confirm the adsorption of Pb(II) ions. The results are displayed in Figure 2. A wideband was observed within the 3200–3300 cm^{-1} range, confirming the presence of free OH groups imposing the hydroxyl and carboxylic functional groups in the beads [12]. The peak at 1734 cm^{-1} of the KNF-CHT-ALG beads confirmed the existence of the –C=O group in the carboxyl (-COOH) group [13]. The FTIR spectrum at around 1590 cm^{-1} might be due to the immobilisation of C=C groups onto the lignin groups of the KNF powder [14]. The band perceived within the 1416–1418 cm^{-1} region represented COO− and C-O stretching, while the peak at the 1244 cm^{-1} region corresponded to the phenols C-O [15,16]. On the other hand, the band within the 1008–1028 cm^{-1} range demonstrated the C-O stretch of cellulose and lignin structure [14]. Overall, the KNF-CHT-ALG beads exhibited numerous functional groups, as illustrated by the FTIR spectra, confirming Pb(II) ions binding capabilities.

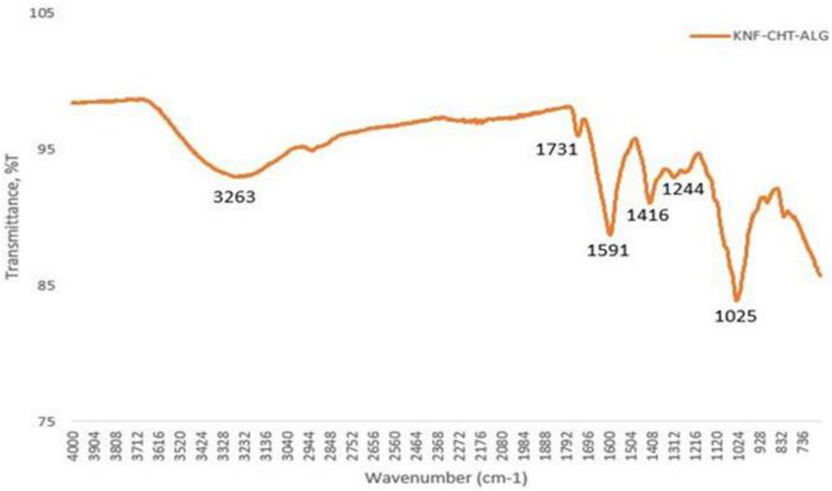


Fig. 2. FTIR spectra of the KNF-CHT-ALG beads within the 700–4000 cm^{-1} scanning range

3.3 Effect of Initial Pb(II) Ions Concentration onto KNF-CHT-ALG Beads

Figure 3 shows trend decreasing of Pb(II) ions removal with increasing of Pb(II) ions concentration. At lower range of Pb(II) ions concentration, the percentage removal exhibits almost 92% of Pb(II) ions. However, at higher concentrations, the performance was reduced with only 84% of Pb(II) ions was removed. This scenario illustrated that at lower Pb(II) ions concentration, the ratio between the number of Pb(II) ions and the number of adsorption sites available is small resulting in high removal percentage of Pb(II) ions. In the case of higher concentrations, excessive Pb(II) ions available thus increasing the probability to contact with the available adsorption sites. Conclusively, the available sites are rapidly occupied and faster saturated as each unit mass of adsorbent is subjected to a larger number of Pb(II) ions. Due to the fixed number of available sites in the adsorbent, the excess ions were remained in the solution leading to lower percentage removal [17].

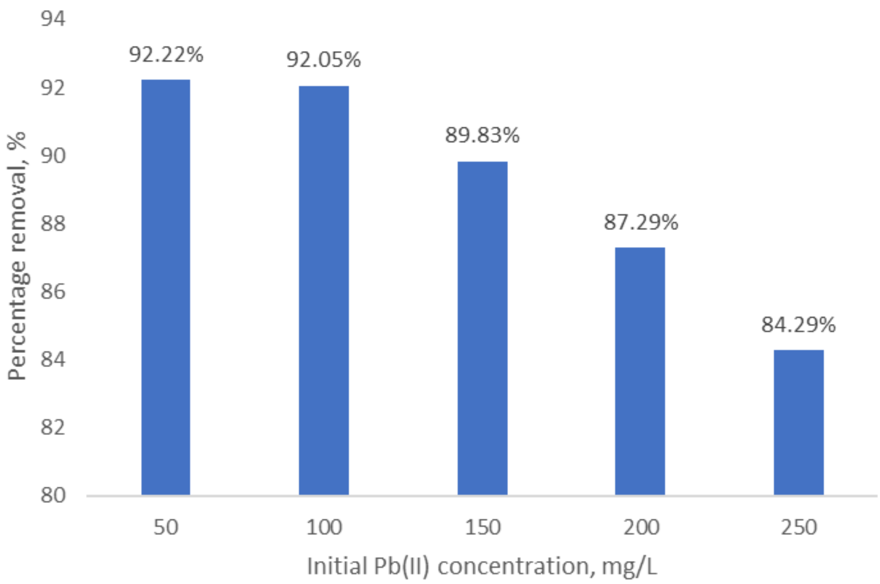
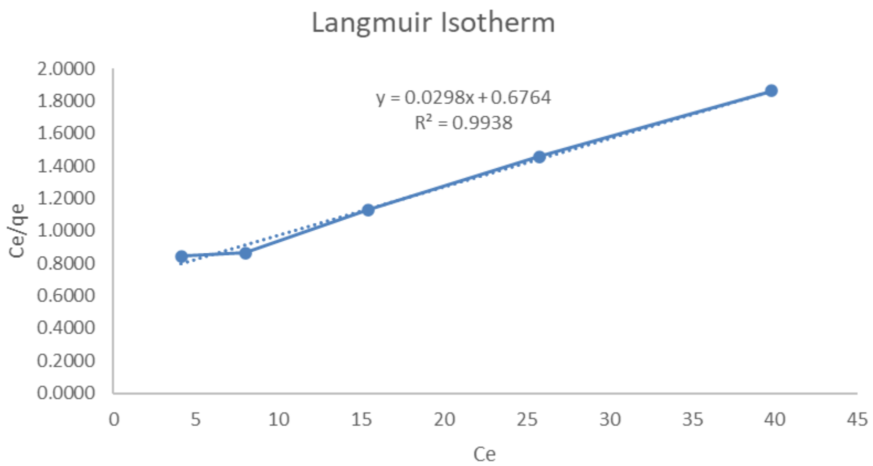


Fig. 3. Effect of initial Pb(II) concentration on adsorption of Pb(II) ions

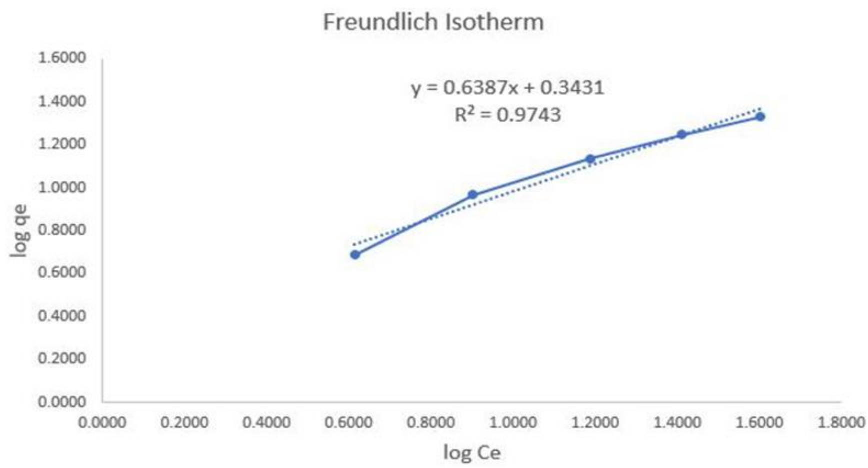
3.4 Adsorption Isotherms

Adsorption isotherms are of importance to understanding the relationship between the Pb(II) ions and the KNF-CHT-ALG beads. Langmuir and Freundlich are the most common isotherm models used to fit the experimental data. The Langmuir isotherm forms monolayer adsorption on the adsorbents. This model predicts maximum adsorption occurs at a fixed homogeneous site that forms saturated monolayer at the exterior surface of the adsorbent. Langmuir isotherm also assumes that all adsorption sites are exact and energetically equivalent. The Freundlich isotherm describes the adsorption of Pb(II) ions at multilayer heterogeneous surfaces. The adsorption process can occur at any site of the adsorbent due to the random distribution of active sites and their energies [10].

The regression coefficient (R^2) value helps in deciding which isotherm model fitted well with the adsorption data. R^2 value near to 1 shows more precision of the sorption process [18]. Figure 4(a) plots the C_e/q_e versus C_e graph which is found to be linear for the Langmuir isotherm. From the figure, the R^2 value is 0.9938. Meanwhile, the graph of log q_e versus log C_e is also found to be linear for Freundlich isotherm as depicted in Figure 4(b). From the figure, the R^2 value is 0.9743, which is slightly lower than the Langmuir isotherm.



(a)



(b)

Fig. 4. Adsorption isotherms model of Pb(II) ions on KNF-CHT-ALG beads (a) Langmuir (b) Freundlich

Table 1 shows the Langmuir and Freundlich isotherm constants and correlation coefficients for Pb(II) ions adsorption on KNF-CHT-ALG beads. Based on the correlation coefficient (R^2) values, the adsorption of Pb(II) ions onto KNF-CHT-ALG beads fitted well to the Langmuir model. The linear Langmuir plot indicates the formation of monolayer coverage of Pb(II) ions on the surface of KNF-CHT-ALG beads. Similarly, a previous study on adsorption of Pb(II) ions using surfactant modified chitosan beads was fitted to the Langmuir model with an R^2 value of 0.984 [9]. The maximum adsorption capacity, qm of Pb(II) ions by KNF-CHT-ALG beads was 33.557 mg/g. This study was in agreement with research done by Thakur *et al.,* [19] whereby the employment of Chinaberry leaves did adsorb maximum capacity of 38.46 mg/g Pb(II) ions. K_L and K_F values describe Langmuir and Freundlich constant, the values recorded were 0.0441 L/mg and 2.2034 mg/g, respectively. n value related to adsorption intensity and the value was found to be 1.5657.

The dimensionless Langmuir separator factor (R_L) was determined using the equation in section 2.4 and the R_L value can be classified as a favourable process. The linear process can occur only if b=0. This means that the adsorption isotherm is a straight line while the irreversible process can occur when the b value is very large, which means that adsorption is too strong. Lastly, a favourable process is the standard case when adsorption occurs normally under our conditions. Not so strong, but noticeably occurs [20]. In this research, the R_L changed from 0 to 1 (0.312, 0.185, 0.131, 0.102 and 0.0832 respectively for Pb(II) ions concentration of 50, 100, 150, 200 and 250 mg/L). The Langmuir isotherm model assumed that the adsorbed layer was one molecule in thickness and all the adsorption sites had equal energies and enthalpies of adsorption [3].

Table 1
Langmuir and Freundlich isotherm constants and correlation coefficients for Pb(II) ions adsorption on KNF-CHT-ALG beads

Isotherm Models	Langmuir					Freundlich				
	q_m (mg/g)	K_L (L/mg)	R^2	Linear equation	Equation	K_F (mg/g)	n	R^2	Linear equation	Equation
Pb(II)	33.557	0.0441	0.9938	$C_e/q_e = (l/q_m)$ (Ce) + l/q_mK_L	y = 0.0298x + 0.6764	2.2034	1.5657	0.9743	$log\ q_e = l/n$ log C_e + log K_F	y = 0.6387x + 0.3431

3.5 Regeneration Cycles of the Adsorbent

Figure 5 and Figure 6 show the percentage removal of Pb(II) ions upon regeneration cycles of KNF-CHT-ALG beads and percentage regeneration of KNF-CHT-ALG beads with Pb(II) ions removal, respectively. After 5 times of regeneration process, the KNF-CHT-ALG beads still showed good adsorption towards Pb(II) ions with maximum removal of 95% and regeneration of 98%. This is because, natural adsorbents usually rich in functional groups, possess higher surface area, easy to synthesize and low in cost which help the adsorption process of the heavy metals [21,22]. A study done by Kulkarni and Kaware [23] also proved that the regeneration cycles using natural adsorbent is good because it can reduce the need of new adsorbent and also reduce the problem of disposal of used adsorbent.

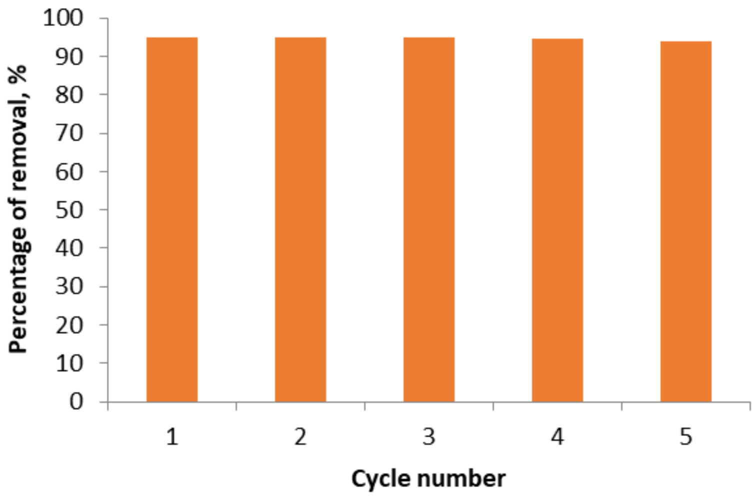


Fig. 5. Effect of regeneration of KNF-CHT-ALG beads on percentage removal of Pb(II) ions

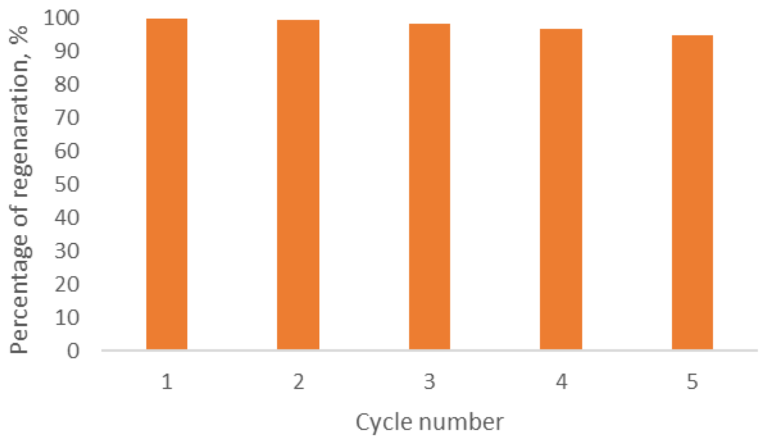


Fig. 6. Effect of regeneration cycles on percentage regeneration of KNF-CHT-ALG beads

Based on the study reported by Yang *et al.,* [24] on the sodium alginate crosslinked with $CaCl_2$ to produce calcium alginate beads, it revealed that after five cycles of the regeneration process, the CA beads are still attracted to adsorb Cu(II) ions and the percentage removal was 92%. Thus, it can be said that CA beads have good reusability during recycling experiments and have economic potential in wastewater treatment. In another studies, the adsorbed concentration of Pb(II) ions by chitosan/alginate/Fe_3O_4@SiO_2 hydrogel composites had a reduction of 18% of the removed Pb(II) ions after three cycles of regeneration study [25]. Razak *et al.,* [26] have found that iminodiacetic acid-modified kenaf fibre for Cu^{2+} ions removal can be used up to 4th cycle and the decrease in the percentage removal can be due to a decrease in the number of active sites.

4. Conclusions

The present study has been carried out with KNF-CHT-ALG beads for the successful removal of Pb(II) ions. The KNF-CHT-ALG beads exhibited numerous suitable functional groups (OH, –C=O, C-O-C, COO− and C-O) and picturized porous microstructure with sufficient active sites for Pb(II) ions to bind. The adsorption data were in good agreement with Langmuir isotherm with a maximum uptake capacity of 33.557 mg/g. Additionally, after 5 times of recycling process, the KNF-CHT-ALG beads still showed good adsorption towards Pb(II) ions with maximum removal of 95% and regeneration of 98%.

The kenaf is a crop found in abundance in Malaysia, which had the potential to reduce the production cost of adsorbents. The significant outcomes of this study are to minimise the dependency on chemical adsorbents and accelerate the removal process of heavy metals in real water bodies.

Acknowledgement
This study was financially supported by the Fundamental Research Grant Scheme (FRGS) (Grant number: FRGS/1/2019/TK10/UITM/02/7). The authors are grateful to the Chemical Engineering Laboratory, Universiti Teknologi MARA (UiTM), Lembaga Kenaf Dan Tembakau Negara (LKTN) Pahang and the Centre for Research and Instrumentation Management (CRIM), UKM, for all the facilities provided. Special thanks also to Penerbitan Yuran Prosiding Berindeks (PYPB) for the financial support of the publication fees.

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