

Adsorption of Pb (II) Ions in Water with Natural Kenaf Beads

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ARTICLE INFO	ABSTRACT
Article history: Received 3 May 2023 Received in revised form 22 July 2023 Accepted 28 July 2023 Available online 30 August 2023 Keywords: Kenaf; polluted; heavy metals; adsorption; Sustainable Development	The purpose of this research is to study plant-based kenaf adsorbents on the adsorption of polluted Pb (II) in aqueous solutions. CHT and KNF core powder were added to the sodium alginate solution and stirred homogeneously. The blend solution was dripped into calcium chloride to form smooth magnetic KNF-CHT-ALG beads. The functional group presence in the beads and percentage adsorption of the heavy metals were checked using Fourier Transform Infrared Spectroscopy (FTIR) and Inductively Coupling Method analyses, respectively. It was found that the surface morphology of kenaf core was rougher than that of kenaf fibre, with the presence of a larger amount of micropores as observed in the FESEM analysis. In addition, the FTIR pattern of the KNF- CHT-ALG beads had shown the existence of functional groups such as hydroxyl and carboxyl, which could attract more positively charged heavy metals. The ICP analysis confirmed the successful 95% adsorption of the heavy metals. Kenaf is an abundant crop available in Malaysia that may reduce the production cost of the adsorbents. The significant outcomes of this study would be its contribution to minimising the dependency on the chemical adsorbent and to accelerating the removal process of heavy metals in real water bodies. Thus, this study aims to create a more sustainable
Goals	future, especially by reaching SDG 6: Clean Water and Sanitation.

1. Introduction

Heavy metal is commonly found in many aquatic environments and is a non-biodegradable constituent, unlike all other contaminants that can be degraded in one way or another [1]. In an aquatic life ecosystem, the level of heavy metals is naturally low, but it can get higher due to both natural and anthropogenic sources, which can easily enter the water body and harm aquatic life. Thus, to treat these heavy metals toxicity, several methods can be applied, such as ion exchange, membrane filtration, and chemical precipitation. However, those are still considered expensive and

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ineffective. Recently, much attention has been given to the adsorption method using natural adsorbent materials like chitosan and kenaf for metal ion uptake based on their good adsorption properties [2]. Adsorption can be defined as the consumption of liquid molecules from a mixture onto the surface of a solid [3]. The adsorption method offers low capital and operational costs, is easy to operate, and is not easily affected by toxic or harmful substances [4].

Chitosan has received high consideration for its capability to absorb various heavy metal ions, owing to its functional groups known as amino (NH₂) and hydroxyl (-OH) [5]. The film-forming properties of chitosan are excellent, but its hydrophilic nature limits some of its applications [6]. In the previous study, sodium alginate was used and acted as the cross-linking agent that enhanced the hydrophobicity of chitosan [7]. Kenaf, as an abundant local plant, is also another potential adsorbent that has many functional groups that can enhance the adsorption stability towards heavy metals [8-10].

For the present study, the researchers proposed the synthesis of a new hybrid kenaf-chitosanalginate hydrogel composite. Herein, the kenaf was sampled from the core or the fibre to identify the potential adsorbent. Characterisation of the composites was carried out using Field Emission Scanning Electron Microscopy (FESEM), Thermogravimetric Analysis (TGA), and Fourier Transform Infrared (FTIR) Spectroscopy analyses. The aim was to demonstrate the physical and chemical properties of the proposed composite adsorbent. The FESEM and the TGA analyses were carried out to demonstrate the physical properties by assessing the surface morphology and the thermal properties of the proposed composites [11]. Meanwhile, the FTIR spectroscopy analysis was performed to demonstrate the chemical properties of the proposed composites and identify the functional groups present in the proposed kenaf composites. For the adsorption study, batch mode sorption was conducted using KNF-CHT-ALG beads as the sorbent, and the percentage adsorption of heavy metals was further checked using Inductively Coupling Method (ICP). With regards to the increasing need to achieve SDG 6: Clean Water and Sanitation, an improved and economical approach using green kenaf is, therefore, more desirable.

2. Methodology

2.1 Materials

The Pb (II) ion solution was prepared using Pb (NO₃)₂ purchased from RandM Chemicals. The raw kenaf sample was obtained from the Raw Material Collecting Centre (RMCC) in Cherating Kuantan, Pahang. The centre is owned by Lembaga Kenaf Dan Tembakau Negara (LKTN), Pahang. The raw kenaf was collected from the site and transported to the MyBiorec laboratory at the School of Civil Engineering, UiTM Shah Alam.

2.2 Preparation of Kenaf Core and Fibre

The procedure was the same as our previous work in Lokman *et al.*, [12]. The collected raw kenaf samples were then cut into smaller chunks of about 30 cm in size and kept in the water basin for about 17 days. The water retting process was used to ease the separation process of kenaf fibre from its core. Then, both kenaf core and fibre were sieved and kept well in airtight plastic bags to be further analysed.

2.3 Characterisation of Kenaf Core and Fibre

The characterisation of both kenaf core and kenaf fibre was analysed using (a) Field Emission Scanning Electron Microscopy (FESEM), (b) Fourier Transform-Infrared (FTIR) spectroscopy, and (c) Thermogravimetric Analysis (TGA), respectively. The surface morphologies of kenaf core and kenaf fibre were observed at an accelerating voltage of 3 kV by FESEM Zeiss MERLIN in Research Instrumental Management (RIM), UKM. The FESEM analysis offers physical information at magnifications of 10x to 300000x, with nearly infinite depth of view. Compared to Traditional Scanning Electron Microscopy (SEM), the FESEM produces a simpler and less electrostatically distorted image. The presence of functional groups in kenaf core and kenaf fibre was observed by FTIR spectroscopy using the Spectrum One FT-IR Spectrometer at Instrumental Lab 2, Faculty of Chemical Engineering, UITM. The FTIR spectroscopy analysis is one of the most important analytical techniques designed for analysing chemical properties of various substrates. One of the greatest advantages of this approach is that any samples can be examined such as liquids, solutions, pastes, powders, films, fibres, and gases. Next, the thermal or physical properties of both kenaf core and kenaf fibre were carried out using the TGA analysis. The equipment used was Mettler-Toledo TGA/DSC Model at the Instrumental Lab 2, Faculty of Chemical Engineering, UiTM. The test was conducted in a nitrogen atmosphere with a flow rate of 50 ml / min from 30°C to 900°C at a heating rate of 10°C / min. This technique offers easy operation and needs only a small amount of sample (1-20 mg).

2.4 Preparation of KNF-CHT-ALG Beads

CHT powder and KNF core powder were added to the sodium alginate solution and stirred homogeneously. The blend solution was dripped into 100 mL of calcium chloride to form smooth magnetic KNF-CHT-ALG beads. Finally, the beads were rinsed with ultrapure water three times and dried at 60°C for two hours.

2.5 Batch Adsorption Study

In order to study the removal of heavy metals using KNF-CHT-ALG beads, a batch sorption experiment was conducted in a 250-ml glass flask. A certain amount of adsorbent (0.5 g) was added to a 250-ml flask containing 50 ml of Pb (II) solution at a concentration of 50 mg/l and then shaken in an incubator shaker at 125 rpm for 60 minutes. Next, the beads were separated from the solution through filtration. Then, the residual concentration of lead ions in the solution was analysed by Inductively Coupling Method (ICP). Eq. (1) gives the percentage adsorption equation [13].

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

(1)

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

3. Results

3.1 Surface Morphology of the Kenaf Core and Fibre

The Field Emission Scanning Electron Microscopy (FESEM) micrographs provide further investigation into the morphology of kenaf core and fibre. Figure 1(a) and 1(b) display the FESEM

images of the kenaf core and fibre at 2000x magnification, respectively. As shown in Figure 1(a), kenaf core exhibits physical properties containing a higher number of micropores and being coarser as compared to kenaf fibre in Figure 1(b). This is aligned with the study done by [14,15], whereby it was found that pore development could increase the surface area, and the pore volume would promote the diffusion of heavy metal molecules into the pores. Raw kenaf often has a rough surface, as reported in Ref. [16]. This finding is in accordance with the one discovered by Ref. [17], that the kenaf core demonstrates better adhesion properties for the adsorption process when the surface roughness or surface area is higher.

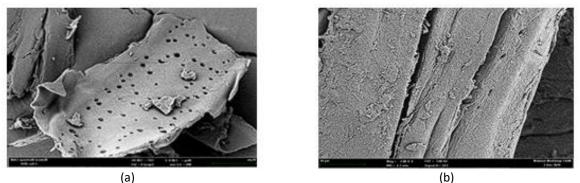


Fig. 1. The FESEM micrographs of raw (a) kenaf core (b) kenaf fibre at 2000X magnification

3.2 Functional Groups Spectra of the Kenaf Core and Fibre

The chemical properties from the FTIR spectra patterns of the kenaf core and kenaf fibre are shown in Figure 2(a) and 2(b), respectively. A wide band appeared at 3300 cm⁻¹, confirming the presence of free OH groups imposing the hydroxyl and carboxyl functions, while the peak IV band position around 1000 cm⁻¹ corresponded to C-OH groups [18]. In addition, the peaks at 1734 cm⁻¹ were attributed to the presence of (–C=O), which confirmed the existence of carbonyl in the carboxyl group (-COOH) [19]. As presented in the FTIR spectra below, both patterns showed the same presence of functional groups. Both kenaf core and kenaf fibre have the potential to promote the binding process of the metal ions, but in relation to higher surface roughness conditions. From the FESEM analysis, the kenaf core demonstrated a better surface that may enhance the binding process of the heavy metals.

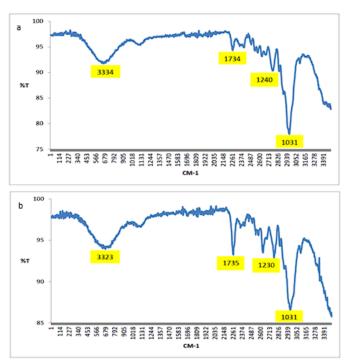


Fig. 2. FTIR spectra for (a) kenaf core (b) kenaf fibre

3.3 Thermal Properties of the Kenaf Core and Fibre

Another important physical property is the determination of thermal properties for evaluating the applicability of the adsorbents at high temperatures. Table 1 shows the parameters of decomposition temperature range, maximum Derivative Thermogravimetric (DTG), residue percentage, and weight loss percentage of lignin, cellulose, and hemicellulose in kenaf core and kenaf fibre obtained from TGA thermograms. The functions of lignin, cellulose, and hemicellulose in kenaf plants are to support the plant mechanically, strengthen the plant, and control biodegradation, thermal degradation, and moisture adsorption. From the table tabulated below, the initial mass decomposition for both kenaf core and kenaf fibre happened approximately at a temperature of 200-600°C with the average maximum loss at around 370°C. This agrees with the study conducted by Ref. [20], which concurred that the initial mass decomposition for kenaf core and kenaf fibre occurred at temperatures of 200-400°C, with the highest loss at 300°C. More raw materials such as lignin, cellulose, and hemicellulose decompose, and their mass continued to reduce as the temperature rose. His study also pointed out that higher degradation promotes a smoother and cleaner surface of kenaf composites since more raw material is degraded.

The DTG max of kenaf core was slightly higher than that of kenaf fibre, which indicates the rate of material weight changes upon heating was slower. Moreover, the residue of kenaf core was higher in percentage compared to kenaf fibre due to lignin content. The weight loss of kenaf core (24.09%) was lower than the weight loss of kenaf fibre (56.91%), which clearly confirms that the surface roughness of the kenaf core is higher and more irregular [8,21] as compared to kenaf fibre. Thus, the kenaf core can potentially help the adsorption process of the metal ions.

TGA results for kenaf core and kenaf fibre						
Decomposition temperature	DTG max (°C)	Maximum decomposition				
range (°C)		Residue (%)	Weight loss (%)			
202-595	372	75.91	24.09			
227-469	366	43.09	56.91			
	Decomposition temperature range (°C) 202-595	Decomposition temperatureDTG max (°C)range (°C)372	Decomposition temperatureDTG max (°C)Maximum derange (°C)Residue (%)202-595372			

3.4 Functional Groups Spectra of KNF-CHT-ALG Beads

From the results of the characterisations, the kenaf core presented a higher potential for binding affinity and was thus being used to synthesise the KNF-CHT-ALG beads. The chemical properties from the FTIR spectra patterns of the beads are shown in Figure 3. A wideband appeared at 3264 cm⁻¹, confirming the presence of free OH groups imposing the hydroxyl and carboxylic functions [19]. A small band perceived at 1417 cm⁻¹ was ascribed to COO- symmetrical stretching vibrations, while the peak V band position around 1024 cm⁻¹ corresponded to C-O stretching vibrations of the chitosan biopolymetric matrix [22]. In addition, the peaks at 1734 cm⁻¹ were attributed to the presence of CO, which confirmed the existence of carbonyl in the carboxyl group (-COOH) [19]. As presented in the FTIR spectra below, the KNF-CHT-ALG beads have the potential to promote a higher binding process for metal ions.



Fig. 3. FTIR spectra of KNF-CHT-ALG beads

3.5 Adsorption of Pb (II) Ions

Figure 4 shows the lead adsorption on the beads. The KNF-CHT-ALG beads revealed good adsorption towards Pb (II) ions, with a maximum adsorption of 95% from an initial 50 mg/l lead concentration to 2.51 mg/l.

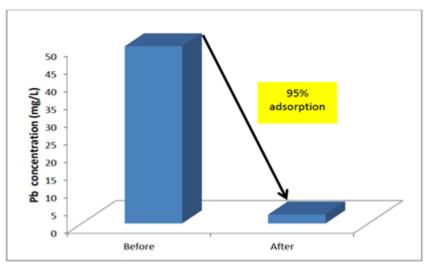


Fig. 4. Percentage adsorption of Pb (II) ions using KNF-CHT-ALG beads

Table 2 shows several studies that have been conducted on green material-based adsorbents for capturing heavy metals in wastewater. It was reported in Ref. [23] that an agro-based adsorbent, Hibiscus Cannabicus kenaf fibre, investigated for Cr (VI) adsorption from water demonstrated a maximum Cr (VI) removal of 73%. In another study, the kenaf functionalized with phosphoric acid removed 88.2% of copper ions from electroplating wastewater [24]. In other studies, the surface modification of chitosan beads using an anionic surfactant, sodium dodecyl sulphate (SDS), effectively removed up to 89.7% of lead ions from wastewater [25].

Moreover, ZnCl₂ was used as a carbonising promoter for kenaf in the treatment of heavy metal contamination and removed up to 91.2% manganese ions [26]. Yang *et al.*, [27] also reported that crosslinking sodium alginate with CaCl₂ demonstrated higher adsorption capacity towards Cu (II) at a 92% adsorption rate. Based on the results, the current study, which proposed the KNF-CHT-ALG beads, exhibited 95% adsorption of Pb (II) ions upon further investigation. Additionally, the kenaf core increased the adsorption binding capacity of the beads towards the Pb (II) ions. Agricultural wastes are potential adsorbents for heavy metal removal owing to the fact that they are relatively cheap and display high adsorption capacities [28].

Table 2

The percentage adsorption of green-material based adsorbent

Adsorbents	Heavy metals	Percentage removal	References
Kenaf fibre	Chromium (VI)	73.0%	[23]
Phosphoric acid modified kenaf fibre (K-PA)	Copper (II)	88.2%	[24]
Surfactant modified chitosan (SMCS)	Lead (II)	89.7%	[25]
ZnCl ₂ carbonized kenaf	Manganese (II)	91.2%	[26]
Calcium alginate	Copper (II)	92.0%	[27]
Kenaf core-chitosan-alginate	Lead (II)	95.0%	This study

4. Conclusions

This study was initially conducted to enhance understanding of the physical characterisation of kenaf core and kenaf fibre using the analyses of Field Emission Scanning Electron Microscopy (FESEM), Fourier Tranform-Infrared (FTIR) spectroscopy, and Thermogravimetric Analysis (TGA). From the FESEM micrographs, it can be concluded that the kenaf core exhibits higher micropores and

surface roughness compared to kenaf fibre. Thus, it is expected that the kenaf core could improve the adhesion process between the metal ions and the surface of the kenaf core. The FTIR spectra depict the presence of free OH groups imposing the hydroxyl and carboxyl functions and (–C=O) imposing the existence of carbonyl in the carboxyl group (-COOH) that can attract positively charged heavy metals to bind with the functional group. Further results from the TGA analysis confirmed that the kenaf core demonstrates higher surface roughness as compared to kenaf fibre. The influence of these factors led to the synthesis of the KNF-CHT-ALG beads, which truly discovered the higher adsorption of 95% Pb (II) ions. Thus, this finding identified an alternative to chemical dependency by using green kenaf as the adsorbent of heavy metals in wastewater treatment.

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