



Adsorption of Iron Ions from Palm Oil Mill Effluent Using Novel Adsorbent of Alginate–Mangrove Composite Beads Coated by Chitosan

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ABSTRACT

This study was about the investigation of the removal of iron ions from Palm Oil Mill Effluent (POME) by using novel adsorbent which is Alginate–Mangrove Composite Beads Coated by Chitosan (AMCBCC). The adsorbent was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM) with Energy Dispersive X-ray Spectroscopy (EDX) to prove the successful coating by Chitosan and also to provide an evidence of iron ions were adsorbed on the surface of the beads. Batch studies were conducted by using different parameters, such as pH, dosage, contact time, and initial concentration. It was found that at pH value of 3, 300 g/L of AMCBCC concentration, and a contact time of 72 hours the maximum removal of iron ions was 92.7%. The isotherm equilibrium data were followed Freundlich isotherm model and the adsorption kinetic data were well fitted by the pseudo second order.

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INTRODUCTION

In the last century after the industrial revolution, industrial wastewater contain heavy metals is considered as one of the main sources of water pollutions. Through that time, an enormous amount of industrial wastewater was drainage into the water sources, which led to severe damage to the environment and increased water pollution. Heavy metals are non-biodegradable, toxic, and bio-accumulate in aquatic living organisms and can transfer to human through the food chain and cause many diseases such as cancer [1, 2].

Nowadays, Malaysia is the second biggest producer and exporter of the palm oil in the world, after Indonesia [3, 4]. According to United States Department of Agriculture (USDA), production of palm oil in Malaysia has raised from 10.8 million tons in 1999/2000 to 21 million tons in 2015/2016. A huge amount of Palm Oil Mill Effluent (POME) is discharged during Palm Oil production process. POME is a dark brown liquid, effluent temperature is in the range of 80–90°C, and it is acidic where the pH is about 4.5–5 [5]. POME contains

many heavy metals such as lead, zinc, cadmium, manganese, iron, copper, and chromium [3, 6, 7]. Among all these heavy metals, iron ions have the highest concentration and in a dangerous level in POME from 75–164 mg/L [8] which exceeds the permissible limits issued by the Department of Environmental (DOE) Malaysia which is 5 mg/L. In spite of the various studies were used to treat effluent contained heavy metals, adsorption process is considered as one of the suitable methods compared to the other traditional treatments because adsorption is economical especially less energy involved when using low cost material as adsorbents, sufficient in removal of the pollutants, even at low concentrations, simple in operation and handling, and safe [9]. Limited studies had been conducted to treat POME by using natural materials as adsorbent in adsorption process. In this study, a novel type of adsorbent was prepared: alginate-mangrove composite beads coated by chitosan (AMCBCC) in beads shape, because the beads are the most suitable form of biopolymer adsorbent for heavy metals removal in wastewater [10], and also the beads are easy to separate from the wastewater after treatment and

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easy to regenerate. Mangrove bark is obtained from charcoal factory, it contains effective functional groups in their structures, such as carboxylic, phenolic and hydroxyl, which can act very well in heavy metals removal [11], mangrove bark is abundant in nature, so using it will be very helpful in solving disposal problems. Alginate is a biopolymer which can be obtained from the cell walls of brown algae, it contains effective functional groups such as carboxylate and hydroxyl which are well-known in their ability of heavy metals adsorption [12]. Alginate is considered one of the eco-friendly, cost-effective and biodegradable materials [13]. Chitosan is a copolymer which can be obtained from chitin, chitin is produced from arthropods' exoskeleton, yeast and fungi cell wall, clams, krill, shellfish, squid and shrimps [14, 15]. It contains effective functional groups such as amino and hydroxyl, which are perfect in heavy metals adsorption [16-19]. Chitosan is considered biodegradable, non-toxic and has the ability to flocculate and regenerate.

The main purpose of this present research is to remove iron ions from real POME using new type of adsorbent (AMCBCC). Different parameters were performed such as pH, dosage of adsorbent, contact time, and initial concentration during the batch studies to discover the optimum parameters conditions. Moreover, isotherm studies and kinetic studies were also conducted in this work.

MATERIAL AND METHODS

Materials

POME sample was collected from Palm Oil Mill in Seri Ulu Langat, Malaysia, the sample was left to cool and kept in a cooling room at about 4°C. pH of POME was 4.9 and iron ions concentration was 94.6 mg/L.

Mangrove bark was collected from Charcoal factory in Kuala Sepetang Perak, Malaysia.

Chitosan with medium molecular weight was obtained from local factory, Malaysia. All the other chemicals (Na-alginate 99% purity, acetic acid, calcium chloride, hydrochloric acid, nitric acid, and sodium hydroxide) were purchased from R&M Chemicals (Selangor, Malaysia).

Preparation of adsorbent (AMCBCC)

Mangrove bark was washed with distilled water, dried in room temperature, ground, sieved using 250 mesh sieve, and treated by 0.1 M NaOH for 2 hours, due to the fact that NaOH is the best agent to improve the bark by increasing the active sites to get the maximum adsorption capacity [20]; then the bark was washed many times with distilled water till the pH of solution became neutral, after that the bark filtered and dried in oven at 60-63 °C for 24 hours. The treated bark was kept in glass container until used.

The mixture of Alginate and Mangrove bark (AM) was prepared by adding 5 g of Na-alginate into 200 mL of distilled water, then was continuously stirring till the alginate completely dissolved, after that 2.5 g of the treated Mangrove bark was added to the Na-alginate mixture and left to mix till it became homogenous. Using syringe pump device to form the alginate-mangrove composite beads (AMCB) by dropping (AM) mixture into 0.2 M CaCl₂ solution and kept in the solution for 24 hours. The beads were washed with distilled water to remove the excess of CaCl₂, left to dry in room temperature then kept in glass container until used.

In order to coat the beads, Chitosan solution was prepared by adding 7.5 g of chitosan powder to 250 mL of 0.2% acetic acid solution, kept stirring for 3 hours at 45-50°C, to get the viscous gel [21]; then the solution of chitosan was insert in Ultra Sonic device for 10 min, after that the solution left in room temperature for 24 hours. The 40g of AMCB were immersed in 250 mL of chitosan solution with slow stirring at 45-50°C for one hour, after that the beads were removed from chitosan solution and were immersed in 250mL of 0.1M NaOH for one hour to become neutralized with the excess acetic acid [21]. Finally, AMCBCC were washed with distilled water till the pH of the solution became neutral, left to dry in the room temperature and kept in glass container for further experiments.

Batch adsorption studies

The batch adsorption studies were achieved in series of 250 mL glass bottles, each one contain 100 mL of POME, a fixed amount of AMCBCC were added and agitated at 150 rpm at room temperature. The effects of different parameters were studied such as pH, dosage, contact time, and initial concentration. The pH of POME solution was adjusted by using 0.1 M NaOH and 0.1 M HCl in the range of 2-9. The effect of dosage was conducted in the range of 2.5-40 g. The contact time was varied from 3-120 hours, and the initial concentration was between 9.46-85.14 mg/L. The iron ions concentrations in POME before and after the treatment were determined by Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES). To do the analysis the samples were filtered and acidified by adding concentrated HNO₃.

The removal percentage and the amount of iron ions adsorbed by AMCBCC was calculated by the equations (1) and (2), respectively.

$$\text{Removal \%} = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

$$\begin{aligned} \text{Adsorption Capacity (q}_e\text{)} \\ = \frac{(C_i - C_e) \times V}{m} \end{aligned} \quad (2)$$

where C_i and C_e (mg/L) are the initial and equilibrium concentration of the adsorbate respectively, q_e (mg/g) is

the amount of iron ions adsorbed by AMCBCC at equilibrium, $v(L)$ is the volume of solution, $m(g)$ is the mass of adsorbent.

Characterization studies

Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the surface functional groups on AMCBCC, as the spectra were measured from 4000 to 600 cm^{-1} . The surface morphology of AMCBCC before and after adsorption was determined by using Scanning Electron Microscope (SEM), the chemical composition of AMCBCC before and after adsorption process was determined by Energy Dispersive X-ray Spectroscopy (EDX).

Regeneration studies

First, for adsorption process, 30 g of AMCBCC were agitated at 150 rpm in 100 mL of POME at pH 3, for 72 hours at room temperature. After that AMCBCC were filtered, washed with distilled water and left to dry for 30 minute and then were immersed in 100 mL of the regeneration agents which were 0.1 M NaOH and 0.1 M HCl) and agitated at 150 rpm for 1 hour. The treated POME and supernatant from each regeneration solution was taken and analyzed by ICP-OES to determine the adsorbed and desorbed iron ions respectively. The AMCBCC were washed with distilled water and left to dry to be reused. This cycle of Adsorption – desorption was repeated three times with the same parameters mentioned above.

RESULTS AND DISCUSSION

Characterization of AMCBCC

From Figure 1, it is found that the main peak was at the wave number 3395.96 cm^{-1} , which referred to hydroxyl group, this group was presented in mangrove bark [22], alginate [23] and chitosan [17]. The wave number 1642.59 cm^{-1} referred to carboxyl group, which was presented in alginate [24]. Moreover, the wave numbers 1034.87 and 779.23 cm^{-1} indicated to the functional group amine, which can be considered evident of coating by chitosan [25]. The SEM images of AMCBCC before and after adsorption are shown in Figure 2 (a and b); it demonstrated that the AMCBCC has a great change after adsorption; it became smooth in comparison with AMCBCC before the adsorption; that was due to the structure of beads was influenced by the adsorption on iron ions. From Figures 3(a and b), it was obvious that the main elements existed in the composite of AMCBCC before the adsorption were; oxygen O and carbon C, while the elements existed in AMCBCC after adsorption were; oxygen O, carbon C, calcium Ca, manganese Mn, and iron Fe. This meant that, after the adsorption, Fe ions as well as Mn ions were adsorbed by AMCBCC. On the

other hand (Au) appeared in both figures due of coating the beads with gold before the analysis.

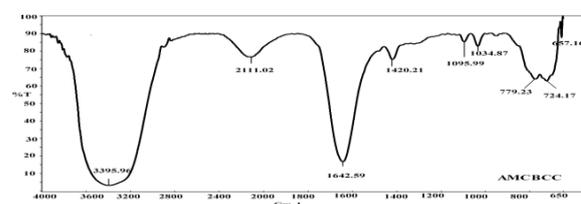


Figure 1. FTIR spectra of AMCBCC

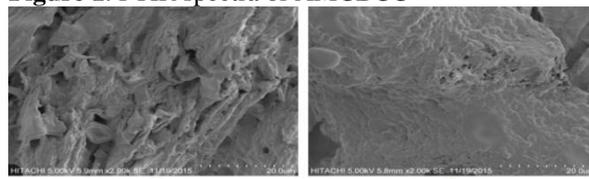


Figure 2. SEM images of: (a) AMCBCC before adsorption and (b) AMCBCC after adsorption

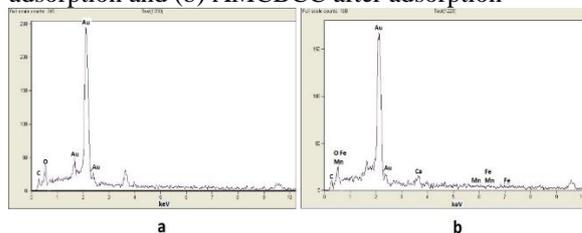


Figure 3. EDX spectra of: (a) AMCBCC before adsorption and (b) AMCBCC after adsorption

Batch adsorption studies

The effect of pH

The adsorption process of Iron ions by AMCBCC is remarkably influenced by the pH of solution [7]. To compute the optimum pH, the pH values of POME solution was ranged from 2-9 (Fig 4). At pH 2 strong competition between hydrogen ions (H^+) and Fe^{3+} ions to be united with the active sites on the surface of AMCBCC, and H^+ occupied most of the active sites, leading to low adsorption of Fe^{3+} ions, as a result of electrostatic repulsion [17]. On the other hand, increasing the pH of POME led to minimize the competition between H^+ and Fe^{3+} ions to be united with the active sites [26], and thus higher removal percentage of Fe^{3+} occurred at pH 3, which is the optimum pH for AMCBCC with maximum removal percentage of 42.56%. Meanwhile, the removal percentage decreased and reached to the lowest level at pH 5, which was 17.89%. This trend was identical to the adsorption of reactive dye in a previous study [27]. Moreover, when the pH increased above 6 the removal percentage increased and reached to 36.25 and 37.11% at pH 7 and 9, respectively. That is due to the fact that iron ions are tending to form their hydroxide $Fe(OH)_2$, $Fe(OH)_3$ and precipitate; therefore, this increase in removal is related to precipitation rather than adsorption [11, 28].

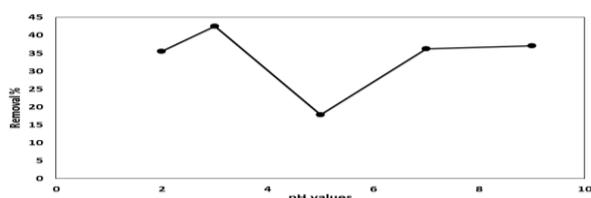


Figure 4. The effect of pH on the adsorption of iron ions

The effect of adsorbent dosage

By increasing AMCBCC dosage from 2.5 to 30 g, the removal percentage gradually increased from 32 to 66.3%, as shown in Fig 5. That is due to increment of available surface area and adsorption sites [29]. A further increase in dosage above 30 g, led to decreasing the removal percentage to 63.95%. This phenomena happened because after the equilibrium between the adsorbent and the adsorbate, the adsorbed iron ions may block the entrance to the initial adsorbent pores [30]. Hence, 30 g of AMCBCC were chosen as the optimum dosage for further experiments.

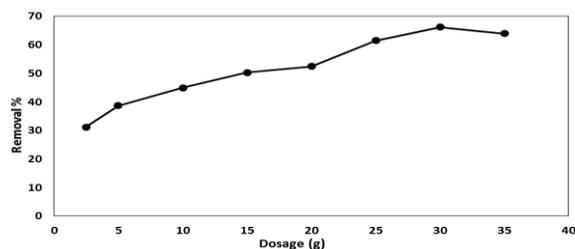


Figure 5. The effect of adsorbent dosage on the adsorption of iron ions

The effect of contact time

The effect of contact time on the removal of iron ions from POME is illustrated in Figure 6. The percentage removal of iron ions rapidly increased through the first 12 hours and reached to 65.4%. Following the same trend, the percentage removal slightly increased in the next 12 hours and reached to 67.3%, and then onward. The percentage removal gradually increased till it reached the equilibrium value of 92.7% at 72 hours. After the equilibrium, the percentage removal decreased to 90.8%; that was due to the fact that the iron ions are tending to desorb from AMCBCC after the equilibrium. The surface of AMCBCC is saturated, and as a result the process of adsorption-desorption may occurred [31].

The effect of initial concentration

The effect of initial concentration on the removal of iron ions is illustrated in Figure 7. It can be noticed that by increasing the initial concentration from 9.5 to 85 mg/L, the removal has decreased. Such phenomena can be explained that at low initial concentration of iron ions, adequate active sites were available, as a result most of the iron ions were adsorbed by AMCBCC; while at high

concentration of iron ions led to fast saturation of AMCBCC [32]. Hence, the initial concentration of iron ions has a great influence on the adsorption of ions on adsorbent.

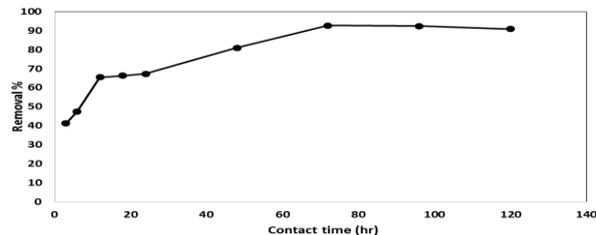


Figure 6. The effect of contact time on the adsorption of iron ions

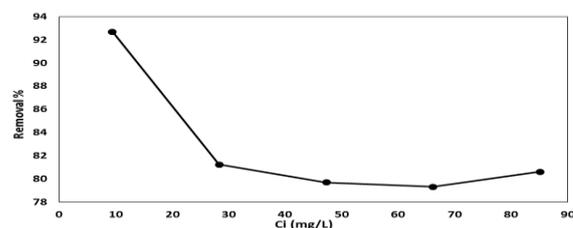


Figure 7. The effect of initial concentration on the adsorption of iron ions

Regeneration of adsorbent

Regeneration and reusability of the adsorbent is a significant feature for the industrial application. It helps to reduce the energy consumption costs and the cost of the adsorption process. The results of the reusability studies were carried by 0.1 M NaOH, 0.1N HCl solutions; the obtained data are illustrated in Figure 8.

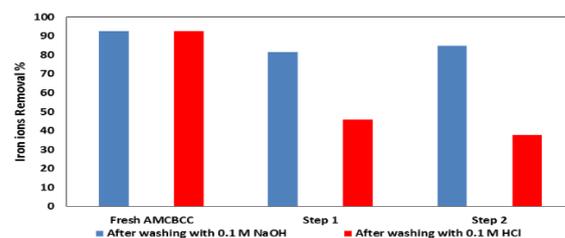


Figure 8. Reusability of AMCBCC for iron ions removal by using NaOH and HCl as a regeneration agents

From Figures 8, it was clear that 0.1 M NaOH was the suitable solution for the regeneration process of AMCBCC; it is obvious that the adsorption capacity of AMCBCC was maintained above 80% after the third cycle, while treatment with HCl solution dropped the adsorption capacity to 37.84%.

This mean, the regenerated AMCBCC by 0.1 M NaOH could be reused for the removal of iron ions without any significant changes of the adsorbent properties; in contrary, acid solution affected and modified the

properties of AMCBCC that caused major reduction in the adsorption capacity.

From Figure 9, it can be noticed that there are weight losses in AMCBCC when using acid solutions as a regenerating agents. The weight losses of adsorbents were 35.8%. That was due to solubility of minerals exist in the chitosan in acid solution [17]. At acidic pH, the H⁺ increased and protonated the amine group of chitosan, which further let the chains of polymer to fall apart [33].



Figure 9. (a) AMCBCC treated with NaOH, (b) AMCBCC treated with HCl

Adsorption isotherm

Adsorption isotherm characterizes the interaction between the adsorbent and adsorbate. Langmuir and Freundlich isotherms are the famous models to explain the relation between the adsorption capacity at equilibrium (q_e) and concentration of adsorbates at equilibrium (C_e). The experiments were conducted by preparing different concentration of iron ions at the optimum operation conditions of contact time, pH, and adsorbent dosage.

The Langmuir model assumed that the adsorption process happens in a monolayer sorption, and the molecular is homogeneously distributed on the surface of adsorbent [7]. The linear form of Langmuir equation stated as follows:

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max} K_L C_e} \quad (3)$$

where q_e (mg/g) is the amount of metal ions adsorbed at equilibrium, C_e (mg/L) is the concentration of metal ions at equilibrium, K_L (L/mg) is Langmuir constant. The q_{max} (mg/g) is the maximum capacity of the monolayer adsorption. The essential characteristics of Langmuir isotherm can be expressed in term of dimensionless parameter (R_L):

$$R_L = \frac{1}{1 + K_L C_i} \quad (4)$$

where C_i (mg/L) is the highest initial concentration of adsorbate. Value of R_L indicate the type of isotherm, if $R_L=1$ the isotherm is unfavorable, $R_L=0$ the isotherm is irreversible, $0 < R_L < 1$ the isotherm is favorable.

A plot of $\frac{1}{q_e}$ versus $\frac{1}{C_e}$ is illustrated in Figure 10, the slope and the intercept of the linear equation were used to calculate K_L and q_{max} , which is presented in Table 1. The

values for K_L and q_{max} were 0.277 L/mg and 0.18 mg/g, respectively. The lower value of K_L was less than 1 which showed the higher affinity of AMCBCC to iron ions [34]. The R_L values was found to be (0.27) which was a favorable adsorption [35].

The Freundlich model assumed that the adsorption process happens in a multilayer sorption, and the molecular is heterogeneously distributed on the surface of adsorbent [7]. The linear form of Freundlich equation shown below:

$$\log q_e = \log k_f + \frac{1}{n} \log C_e \quad (5)$$

where q_e (mg/g) and C_e (mg/L) are defined in Eq. (4), K_f (mg/g) is Freundlich constant, $\frac{1}{n}$ is the constant that indicate the type of isotherm, if $\frac{1}{n} = 0$ the isotherm is irreversible, $\frac{1}{n} > 1$ the isotherm is unfavorable, $0 < \frac{1}{n} < 1$ the isotherm is favorable.

A plot of $\log q_e$ versus $\log C_e$ is illustrated in Figure 11, where the intercept and the slope of the linear equation were used to calculate K_f and $\frac{1}{n}$ which are presented in Table 1. The values for K_f and $\frac{1}{n}$ were 0.033 mg/g and 0.6215, respectively. The value of $\frac{1}{n}$ is between 0 and 1 and the value of n is 1.61 > 1 which indicated a favorable adsorption [36].

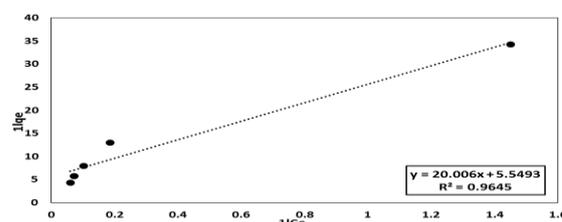


Figure 10. Langmuir Adsorption Isotherm of iron ions onto AMCBCC

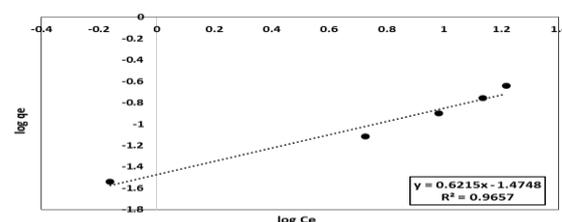


Figure 11. Freundlich adsorption isotherm of iron ions onto AMCBCC

From Table 1 the values of the correlation coefficient of Langmuir and Freundlich isotherms are 0.9645 and 0.9657, respectively. These values suggested that the adsorption of iron ions by AMCBCC showed that both models were quite similar and the R^2 values were very close to each others.

TABLE 1. Parameters of Langmuir and Freundlich isotherm for iron ions

Isotherm Models	Parameters	Values	R ²
Langmuir Isotherm	q _{max} (mg/g)	0.18	0.9645
	K _L (L/mg)	0.277	
Freundlich Isotherm	K _f (mg/g)	0.0335	0.9657
	$\frac{1}{n}$	0.6215	

Adsorption kinetics

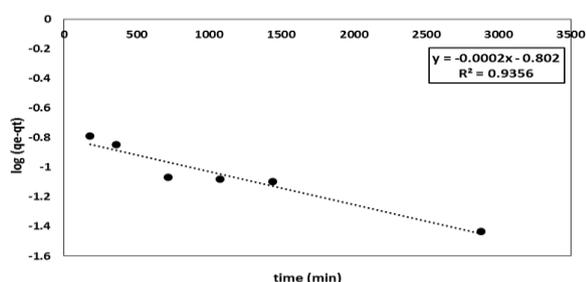
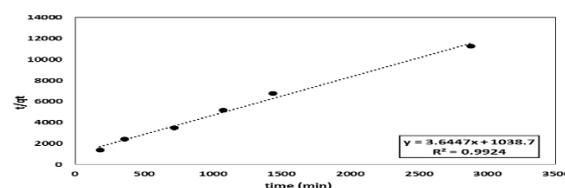
The rate of adsorption can be calculated by two well-known kinetics adsorption models Lagergen's first order equation and Ho's second order equation. The experiments were conducted under different contact time. The linear form of pseudo first order equation stated as follows:

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (6)$$

where q_e and q_t (mg/g) are the amount of metal ions adsorbed at equilibrium and at any time respectively, k_1 (L/min) is the constant of pseudo first order. A plot of $\log (q_e - q_t)$ versus time is illustrated in Figure 12; where the intercept and the slope of the linear equation were used to calculate the $q_{e,cal}$ and K_1 which are presented in Table 2. The values for $q_{e,cal}$ and K_1 were 0.1577 mg/g and $4.606 \times 10^{-4} \text{ min}^{-1}$, respectively. The linear form of pseudo second order equation shows:

$$\frac{t}{qt} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (7)$$

where q_e and q_t (mg/g) are defined in Eq. (6), k_2 (g/mg.min) is the constant of pseudo second order. A plot of $\frac{t}{qt}$ versus time is illustrated in Figure 13, where the intercept and the slope of the linear equation were used to calculate the $q_{e,cal}$ and K_2 which are presented in Table 2. The values for $q_{e,cal}$ and K_2 were 0.2743 mg/g and $0.0127 \text{ g mg}^{-1} \text{ min}^{-1}$, respectively.

**Figure 12.** Pseudo first order kinetic model of iron ions**Figure 13.** Pseudo second order kinetic model of iron ions**TABLE 2.** Parameters of pseudo first and second order kinetics of iron ions

Kinetics Models	Parameters	Values	R ²
Pseudo first order	q _{e,cal} (mg/g)	0.1577	0.9356
	q _{e,exp} (mg/g)	0.2923	
	K ₁ (1/min)	4.606×10^{-4}	
Pseudo second order	q _{e,cal} (mg/g)	0.2743	0.9924
	q _{e,exp} (mg/g)	0.2923	
	K ₂ g/mg.min	0.0127	

From Table 2 the values of the correlation coefficient of the pseudo first order and pseudo second order were 0.9356 and 0.9924, respectively. These values indicated that the adsorption of iron ions by AMCBCC followed the pseudo second order model, because the R² for the pseudo second order (0.9924) was higher than R² for the Pseudo first order (0.9356), as well as the $q_{e,cal}$ for the Pseudo second order (0.2743 mg/g) was much agreeable with the experimental one (0.2923 mg/g) compared to the pseudo first order [31, 35].

CONCLUSION

In this research, AMCBCC were prepared and characterized by FTIR and SEM/EDX, and well performed to adsorb iron ions from POME, by the sufficient functional groups in the beads (i.e. hydroxyl, carboxylate and amine). The maximum removal percentage obtained was above 90 % at pH 3, 30 g of AMCBCC dosage at 72 hours and when the initial concentration increased, the removal percentage decreased, the isotherm data for iron ions was the best fitted with Freundlich isotherm which indicated that the multilayer sorption was dominated. In terms of kinetic studies, iron ions followed the pseudo second order, indicating that the chemical adsorption was the rate limiting step. In addition, the regeneration process was accomplished by using 0.1 M NaOH, the results illustrated that the adsorption capacity of iron ions by AMCBCC maintained above 80% after the third adsorption-desorption cycle.

In the end, this research suggested that Alginate-Mangrove Composite Beads Coated by Chitosan (AMCBCC) is a promising adsorbent for the removal of heavy metals from industrial wastewater.

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Persian Abstract

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چکیده

این مطالعه درباره بررسی حذف آهن از فاضلاب روغن پالم با استفاده از جاذب جدید بر پایه آلژینات پوشش داده شده با کیتوزان انجام گرفته است. بررسی جاذب با استفاده از آنالیزهای FT-IR، SEM، EDX جهت اثبات پوشش کیتوزان و همچنین بررسی شواهد جذب یون آهن بر روی جاذب انجام شد. شرایط مختلف آزمایش در حالت ناپیوسته مورد ارزیابی قرار گرفت. در زمان تماس ۷۲ ساعت ۹۲/۷٪ جذب در بهترین حالت صورت پذیرفت. ایزوترم های فرندلیش و اطلاعات کینتیک جذب با استفاده از مدل شبه درجه دو مورد مطالعه قرار گرفت.
