



Adsorption Of Cr(VI) Ion Using Sugar Palm Fruit Peel (*Arenga Pinnata*) Immobilized Ca-Alginate Bead

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Article History

Received: 28 September 2021; Received in Revision: 14 January 2022; Accepted: 17 January 2022

Abstract

Heavy metal toxicity was an important issue for the environment and human health. Global ecological and public health concerns related to environmental pollution by these metals have been increasing. A new low-cost adsorbent-based of sugar palm (*Arenga pinnata*) fruit peel immobilized Ca-alginate bead was used for the adsorption of Cr(VI) ions in an aqueous solution. Sugar palm fruit peel is used as an adsorbent because it contains of hydroxyl, carbonyl, aldehyde and carboxyl groups, where they can play a role in the adsorption of metal ions. In this study, sugar palm fruit peel waste was immobilized using a Ca-alginate bead to increase adsorption capacity and improve mechanical strength in an aqueous solution. The immobilization improves its mechanical strength, size, rigidity, porosity characteristics, and resistance to environmental restraints. The adsorbent-based sugar palm fruit peel immobilized Ca-alginate bead was characterized by FTIR and SEM-EDX. The effect of pH, contact time, and metal ion concentration on adsorption capacity were studied by batch method. The result showed that optimum pH was pH 2 with an adsorption capacity of 0.3539 mg/g, the contact time was 75 minutes with an adsorption capacity of 0.4131 mg/g and concentration at 10 to 150 mg/L with adsorption capacity was 13.1519 mg/g.

Keywords: Adsorption, sugar palm (*Arenga pinnata*) fruit peel, immobilization, Ca-alginate, Cr(VI) ions.

1. Introduction

Heavy metals are classified as hazardous substances in the aquatic environment (Gupta et al., 2013). One of the dangerous heavy metals is chromium Cr(VI). Both, chromium and its compounds are toxic metals introduced into natural water from various industrial wastes (Ablouh et al., 2019). The Cr(VI) ion is very active and its highly toxic power will work as a barrier to enzyme work in physiological or metabolic processes of the body (Jang et al., 2020). In the metabolic process, Cr(VI) ions will inhibit the work of the benzopyrene hydroxylase enzyme that causes a change in cell growth. The cells will grow wild which, is known as cancer (Castro-Castro et al., 2020). Moreover, Cr(VI) ion could cause lung cancer, skin cancer, anemia, mutagenic and carcinogenic effects. (Alothman et al., 2013). Generally, the majority of sources are textile dyeing, electroplating, leather tanning, and metal finishing industries, which cause severe environmental and public health problems. The presence of Cr(VI) in waters is conceivable removed through various methods like as electrolysis (Wan et al.,

2019), chemical precipitation, membrane processes, filtration, co-precipitation, reverse osmosis, ion exchange, and adsorption processes (Peng and Guo, 2020). Adsorption is one of the methods that are quite effective for removing Cr(VI) ions, due to the adsorption process is much more effective and versatile for removing chromium which solves the problem of sludge disposal and made the system more economically viable, and low-cost adsorbents are used (Cherdchoo et al., 2019). The adsorption method based on the interactions between metal ions and functional groups present on the surface of the adsorbent for removal of metal ions (Lyu et al., 2017).

In this study, the sugar palm (*Arenga pinnata*) fruit peel (SPFP) was used as an adsorbent for Cr(VI) metal ion. *Arenga pinnata* fruit consumed was core of the seed (*endosperm*). While peel of fruit will become waste if not used and pollute the air, water, and soil environment. This sugar palm fruit peel waste is usually only used as raw material for producing compost, and as a substitute for charcoal briquettes. Sugar palm fruit peel

waste is used as an adsorbent because it contains active compounds such as carbohydrates, proteins, fats that can play a role in the adsorption of metal ions (Chaidir et al., 2015). The sugar palm fruit peel contains functional groups -OH, C-H, and COO⁻ serves as a place for adsorption of Cr(VI) metal ions. The utilization of SPFP waste is an effective way to reduce the level of pollution of the aquatic environment. In this study, the SPFP waste is immobilized in Ca-alginate to increase the mechanical strength, adsorption capacity, and cell stability in the adsorption process, which can be used sustainably. Immobilization is a method that is applied to place organelles and cells into a buffer in the form of a membrane or matrix chemically and physically. Alginate acid is a copolymer of two monomer units, 1,4-linked-β-D-mannuronic and α-L-guluronic acids containing carboxylic groups acting as an active site for the binding of metal (Gokila et al., 2017). The binding of inorganic ions to the alginate acid makes it able to show a number of properties and makes it a perfect choice for the use of various applications. Alginate is a natural polysaccharide that is abundantly found in the cell walls of all species of brown algae and plays a role in maintaining cell structure. This research aims to immobilization of sugar palm fruit peel with Ca-alginate as Cr(VI) ion adsorption in an aqueous solution. SPFP immobilized Ca-alginate has been characterized by FTIR and SEM-EDX.

2. Methodology

2.1. Materials

The materials used in this study were sugar palm fruit peel, distilled water, NaOH pro analysis (Merck), HNO₃ pro analysis (Merck), CaCl₂ pro analysis (Merck), K₂Cr₂O₇ pro analysis (Merck), Na-alginate pro analysis. The equipment used in this study were a set of peeler, 150 mesh sieve, hot plate, magnetic stirrer, pH meter, oven type TR 30/R7, analytical balance, pH meter, thermometer, funnel, filter paper, glass ware, dropper, aluminum foil, Fourier Transform Infra-Red (FT-IR), Spectrophotometer type Perkin Elmer Frontier, Atomic Absorption Spectrophotometer (AAS) Type Shimadzu AA 7000, and Scanning Electron Microscopy Energy Dispersive X-ray (SEM-EDX) Type Hitachi.

2.2. Preparation of Stock Solution

The Cr(VI) stock solution was prepared by dissolving 2.707 g K₂Cr₂O₇ was dried at 105 °C for two hours in a 1000 mL volumetric flask

with deionized water to form a stock solution of Cr(VI) with concentration 1000 mg/L. Our experimental solution is prepared at 50, 100, and 125 mg/L with serial dilutions of stock solution of 250 mL.

2.3. Sample Preparation

The raw material used in this study was sugar palm (*Arenga pinnata*) fruit peel (SPFP). The SPFP was taken from Setiris village, Muaro Jambi District, Province of Jambi. The SPFP was cut into small pieces then washed with water to remove impurities that are still attached and aerated in room temperature, and then dried in an oven at a temperature of 100°C and 1atm for 2 hours until the dried SPFP mass being constant. Then, dried SPFP was grounded to powder, mashed using a peeler and filtered using a 150 mesh sieve. The SPFP powder was soaked in 4% NaOH solution in a ratio of 1:10 (m/v). The mixture was stirred for 2 hours at a speed of 100 rpm and then allowed to leave for 24 hours. The SPFP powder was washed with distilled water until the pH neutral and then filtered. Then dried in an oven at 100°C until dry, then pulverized again with a mortar and sieved using a 150 mesh sieve.

2.4. The SPFP immobilized Ca-alginate.

The SPFP immobilization process is accomplished by dissolving 2.0 g of Na-alginate in 100 ml of distilled water, heated at 60°C with constant stirring until homogeneous for 4 hour, cooled at room temperature. Then, 2.0 g of SPFP was added in sodium alginate solution and homogenized for 2 hours. The mixed solution of Na-alginate and SPFP was dropped into 250 ml of CaCl₂ solution using syringe. The granules completed formed in 24 hour, it was called bead. Bead washed using distilled water and dried at room temperature. Bead SPFP immobilized Ca-alginate kept at temperature 4 °C.

2.5. Characterization of Adsorbent SPFP immobilized Ca-alginate.

The adsorbent characterization were characterized using FT-IR instrument to see functional groups, SEM-EDX to determine the surface morphology of adsorbent before and after adsorption of Cr(VI) ions. The FTIR spectra of adsorbent were recorded in PerkinElmer FT-IR Spectrometer in the wavenumber range of 400-4000 cm⁻¹. The morphology, structure and element of adsorbent were examined with SEM-EDX (Hitachi). Cr(VI) ion concentration in solution

analysis using Atomic Adsorption Spectroscopy (AAS-Shimadzu AA 700).

2.6. Adsorption Experimental

The solution stock of Cr(VI) ions 1000 mg/L was prepared by dissolving 5.56 g of $\text{Cr}_2\text{O}_7 \cdot 5\text{H}_2\text{O}$ in distilled water. The batch adsorption experiment conducted in 250 mL conical flask with 100 mL of standard solution using shaker. The Adsorbent 0.1 g was added into these solution and shaker for 30 minute. After reaching adsorption equilibrium, the solution was filtered and filtrate was analysis using AAS. The Cr(VI) adsorption capacity at equilibrium is calculated by Equation 1.

$$Q_e = \frac{C_0 - C_e}{m} \cdot V \quad (1)$$

where, Q_e is adsorption capacity, C_0 (mg/L) and C_e (mg/L) are the initial and equilibrium concentration of Cr(VI), respectively, V is volume of solution, m is mass of adsorbent.

Variable in adsorption are pH, contact time and concentration Cr(VI) ion. The effect of pH on the adsorption of Cr(VI) by bead SPFP immobilized Ca-alginate was studied in a pH range of 1.0–7. The pH of 100 mL solution Cr(VI) 10 mg/L was adjusted to the required pH value using appropriate concentrations of 0,1 M HNO_3 or 0,1 M NaOH solution.

The effect of contact time on Cr(VI) adsorption determined in 30; 45; 60; 75; 105 and 120 minute were chosen with 10 mg/L Cr(VI) ion concentration, 0.1g adsorbent doses and pH value optimum. The mixture was homogenized using a shaker which was then filtered through filter paper and filtrate analysis using AAS. The adsorption isotherm experiments were carried out by adding 0.1 g adsorbent to 100 mL solution with initial Cr(VI) various concentrations of 10; 25; 50; 75; 100; 125; and 150 mg/L. pH values were controlled at 2.0 and the suspensions were shaken for 1 hour at 25°C.

3. Results and Discussion

3.3. Activation of Adsorbent

The activation biomaterial process aims to remove impurities, open pores and activate functional groups on the surface to activate the surface. In this study activation of SPFP has been carried out with 4% NaOH as an activator is because NaOH can remove the lignin contained in cellulose and decompose mineral salts contained in the adsorbent so that it will form -COOH and -OH functional groups on the adsorbent. The OH^- ion from NaOH will break the bond from the basic

structure of lignin, while the Na^+ ion will bind to the lignin to form sodium phenolate. This is due to NaOH being optimal for pulping in plants other than wood such as palm skin fiber. Lignin is removed because it can interfere with the ion transfer process to the active site of the adsorbent.

After the activation process, the adsorbent was allowed to leave for 24 hours to maximize the activation process. The longer the activation process, the more mineral salts are lost so that the functional groups formed on the adsorbent will be better. The longer the activation time, the more maximal the pores will form. The adsorbent was then filtered and washed using distilled water till reach neutral pH, to dissolve the lignin compounds that could interfere with the adsorption process. Furthermore, the adsorbent was dried at a temperature of 70 °C to increase the porosity of the cell wall on the surface of the adsorbent. The dried adsorbent was crushed with a mortar pestle which was then sieved with a size of 150 mesh.

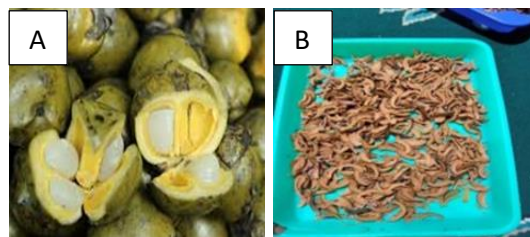


Figure 1. Sugar palm fruit (A) and sugar palm fruit peel dry (B).

3.4. SPFP Immobilized in Ca-Alginate

In this research, SPFP immobilized in Ca-alginate was obtained by providing Calcium ion in the form of CaCl_2 solution. The initial stage was carried out, 2 g of SPFP mixed into 100 ml of Na-alginate, then dripped into a CaCl_2 solution to form beads. The CaCl_2 solution diffuses into the Na-alginate solution so that cross-linking occurs between calcium and alginate ions quickly. Ca^{2+} ions were cross-linked with carboxylate anions ($-\text{COO}^-$) of alginate and ion exchange occurs between Na^+ and Ca^{2+} . After the beads were formed in the CaCl_2 solution, they were left for 24 h to maximize bead formation. This adsorbent SPFP immobilized Ca-alginate has advantages in increasing of adsorption capacity, stability mechanical strength, strong aggregates, and facilitating the separation of the adsorbent from the solution.

3.5. Characterization Of Adsorbent

The FTIR spectra of SPFP Ca-alginate bead are shown in Figure 2. Adsorbent that could affect the adsorption of metal ions. Functional groups like carboxyl and hydroxyl generally are used as a place for metal ions to interact so that absorption occurs (Jafari and Senobari, 2012). The FTIR spectra showed that the presence in Figure 2a.

Table 1. Functional groups contained in the adsorbent.

Functional Groups	Sugar palm fruit peel Adsorbent	
	Before immobilized (cm ⁻¹)	Immobilized Ca-alginate (cm ⁻¹)
O-H	3321.36	3456.37
C-H	2908.11	2917.91
C=O	1618.48	1593.45
CH ₂	1408.17	1425.51
C-O	1029.27	1069.52
C-H	774.69	

The peak at 3321.36 cm⁻¹ and 3855.58 cm⁻¹ assigned -OH stretching. The peak at wave number 2908.11 cm⁻¹ assigned -C-H (aldehyde) stretching. The peak at 1618.48 cm⁻¹ is the strain vibration of the -C=O bond. Figure 2(b) showed peak at wave number 1593.45 cm⁻¹ showed the presence of a -C=O stretching. The peak at 1029.27 cm⁻¹ is

indicated of -C-O stretching, but the resulting peak has the same -OH functional group at wave number 3456.37 cm⁻¹ and wave number 2917.91 cm⁻¹ also indicates occurrence of -C-H bond (Table 1). The peak at 1593 cm⁻¹ is indicated -C=O stretching. This is in accordance to previous research (Chaidir et al., 2015) that at 3423.57 cm⁻¹ there is -OH functional group and carboxylate group.

The morphology of SPFP immobilized Ca-alginate bead image showed in Figure 3. The SEM analysis measurement of SPFP immobilized Ca-alginate bead with magnifications of 1000x; 5000x; 10,000x; and 20,000x.

The morphology of SPFP immobilized Ca-alginate showed in Figure 3. In the SEM results with a magnification of 200x, Figure 3(a) showed an irregular surface of the Ca-alginate immobilized adsorbent, this is following the previous research (Chaidir et al., 2015) accomplished SEM analysis using the same adsorbent as the results of the surface shape of the adsorbent irregularly by forming lumps at 200x magnification. Based on the SEM, it can be seen in Figure 3(b) with 1000x magnification which shows the surface of the Ca-alginate immobilized adsorbent with an irregular structure.

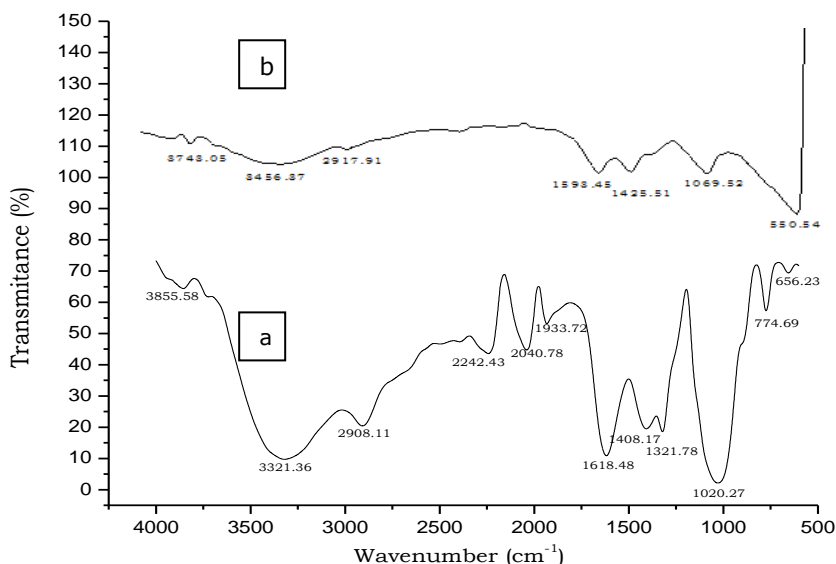


Figure 1. Spectra FTIR of (a) SPFP and (b) SPFP immobilized Ca-alginate.

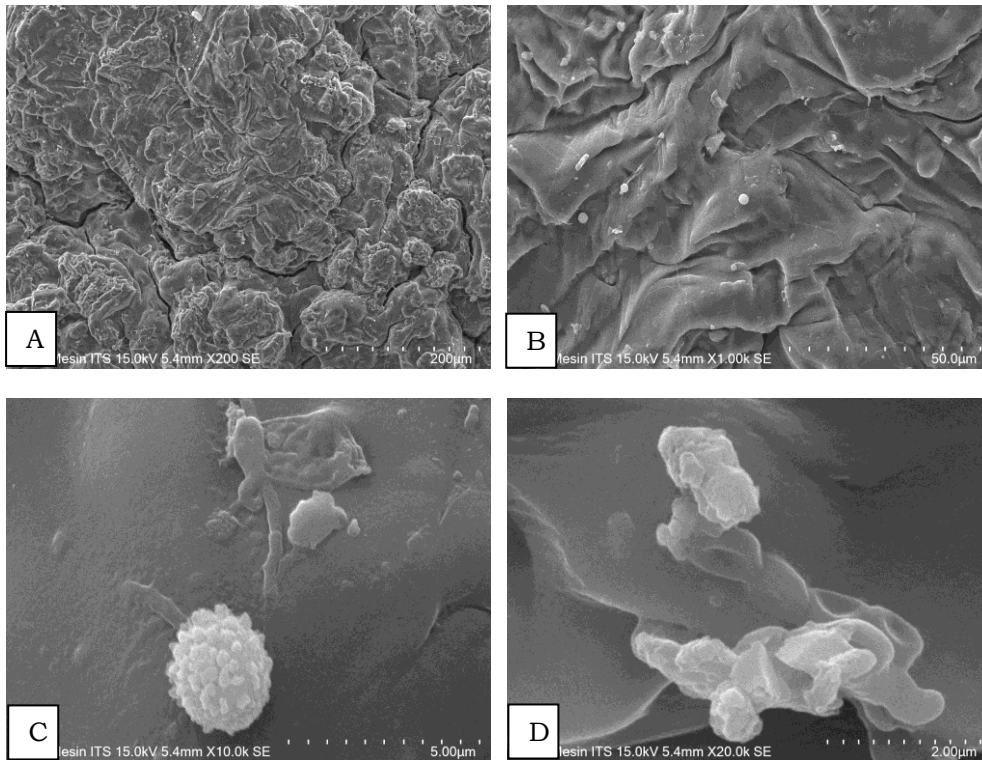


Figure 2. FE-SEM image of the SPFP immobilized adsorbent (a) 200x, (b) 1000x (c) 10,000x and (d) 20,000x magnification.

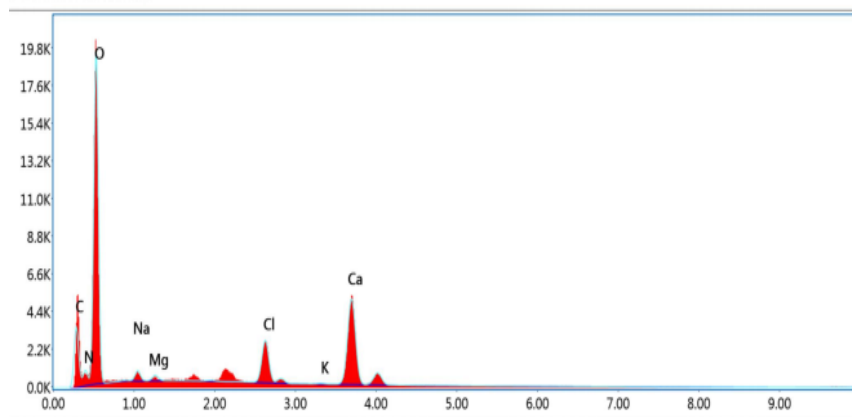


Figure 3. EDX Ca-alginate immobilized adsorbent.

In another hand of the adsorbent, it seems that the adsorbent had bound to Ca-alginate polymer with a shape resembling small white balls and the presence of long-chain Ca-alginate polymer on the surface of the adsorbent. In Figures 3(c) and 3(d) with a magnification of 10,000x and 20,000x, white clumps indicated that bonding formed between the adsorbent and Ca-alginate after the immobilization process due to the carbonyl group (COO^-) of the alginate interacting with the groups contained in the adsorbent. The success of the immobilized Ca-alginate adsorbent confirmed from the EDX spectrums, whichever provides

information about the elements present in the adsorbent.

The main principle of EDX is that X-rays from the SEM results are fired at the analyzed sample, which then appears at a peak that will represent the sample elements. From the results obtained, Ca-alginate immobilized adsorbent can apply as an adsorbent which is indicated by the composition of the general constituent elements, namely C, N, O, Na, and Ca. The elemental composition of Ca-alginate immobilized adsorbent which has been analyzed by EDX.

Table 2. EDX Ca-alginate immobilized adsorbent.

Element	Mass %	Atom %
C	7.32	11.04
N	4.37	5.66
O	62.23	70.49
Na	1.65	1.30
Cl	5.23	2.67
Ca	18.57	8.40
Mg	0.51	0.38
K	0.12	0.06

The highest elemental composition in the adsorbent is element O = 62.23%, atomic mass 70.49%; C = 7.32% atomic mass 11.04% and Ca = 18.57% atomic mass 8.40%. Elements C and O are the main components in cellulose materials. Cellulose can be utilized for the adsorption process. The element Ca indicates the presence of the constituent elements of Calcium Chloride. The high percentage of Ca element indicates that the immobilization of Ca-alginate with SPFP has been successfully synthesized.

3.6. Standard Calibration Curve of Cr(VI) Ion Solution

The standard calibration curve for Cr(VI) ion solution is to determine the linear relationship between absorbance and concentration. This standard calibration curve was obtained from a standard solution of Cr(VI) ions with four different concentrations, 4 mg/L; 6 mg/L; 8 mg/L, and 10 mg/L. Based on the calibration curve, a linear regression was obtained, $y = 1.127x + 1.102$ with a value of $r^2 = 0.993$. It seems expressed as a good coefficient of determination because it was close to 1.

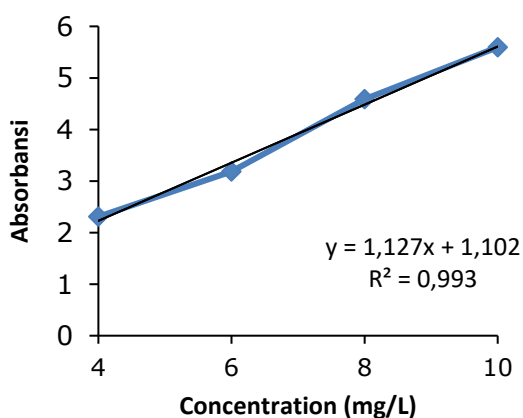


Figure 4. Standard calibration curve of Cr(VI) ion solution.

From the calibration curve results obtained, it seems that the higher the concentration of solution, the greater absorbance produced. Unfortunately, the higher concentration in line

with the more Cr(VI) ions will be in the solution following more Cr(VI) ions are adsorbed. From the calibration curve result, it can be seen that the absorbance is directly proportional to the concentration of the solution.

3.7. Effect of pH on Cr(VI) Ions Adsorption

The acidity of the medium affects the competition of the hydrogen ions and metal ions for the active sites on adsorbent. Variations in pH were conducted to determine the optimum pH conditions in the adsorption process using SPFP immobilized Ca-alginate. Variations in pH will result in changes in the distribution of charge on the adsorbent and adsorbate, this occurred because of the protonation and de-protonation reactions of functional groups. Determination of the pH effect was carried out with several variations pH of 1, 2, 3, 4, 5, 6, and 7 stirred for 30 minutes with a mass of 0.1 g of SPFP immobilized Ca-alginate with 10 mg/L Cr(VI) solution. The large concentrations used in the initial test process can cause agglomeration or lumps in the solution so that the adsorption process will not occur optimally. Based on the graph, the optimum adsorption capacity obtained was at pH 2 of 0.354 mg/g. At low pH the functional group is protonated and increased the electrostatic attraction between the negative Cr(VI) ion (anion) and group on the adsorbent (cation) H^+ ion (Zhao et al., 2020).

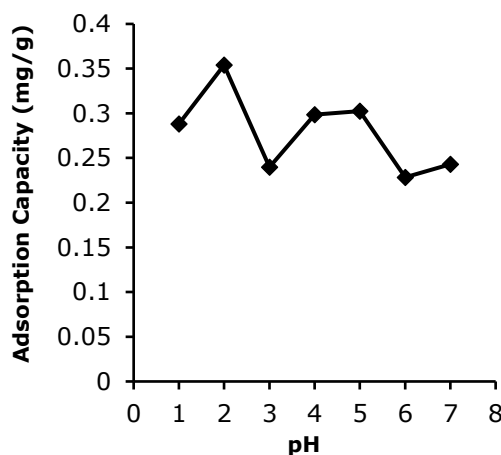


Figure 5. Effect of pH on adsorption capacity Cr(VI) ion.

Decreasing pH will open the carboxyl functional group and carries a negative charge which causes metal ions to be adsorbed. At a higher pH, the number of metal ions will decrease due to competition between

negatively charged Cr(VI) ions and OH⁻ ions so that the adsorption process will decrease. The lower the pH (acid) used, the more adsorbent will absorb the adsorbent, while at high pH (alkaline), less adsorbent will be absorbed.

The results obtained in this research are in line with previous studies (Utama et al., 2016) that the optimum pH in the absorption of Cr(VI) ions is at pH 2. Cr(VI) ions have different shapes depending on the pH of the solution. At pH 1-6 Cr ions have the form Cr₂O₇²⁻, HCrO₄⁻, Cr₃O₁₀²⁻, Cr₄O₁₃²⁻ with HCrO₄⁻ being the most dominating form, but with increasing pH of the solution, the dominant ion forms are Cr₂O₇²⁻ and CrO₄²⁻ (Castro-Castro et al., 2020). With a decrease in pH, the amino, phosphate, and carboxyl functional groups will open and carry a negative charge so that metal ions will be adsorbed (Kanamarlupudi et al., 2018).

3.8. Effect of Contact Time on Cr(VI) Ion Adsorption

The contact time was carried out to determine the optimum time required for the immobilized adsorbent maximum adsorption metal ion. The optimum time is the time required to reach equilibrium when the active site of the adsorbent has metal ions that are maximally bound and (Gorzin and Bahri Rasht Abadi, 2018) when after equilibrium occurs, the bond between the surface-active side of the adsorbent and metal ions will weaken so that it occurs desorption process. In this study, the time contact was varied from 30; 45; 60; 75; 105; and 120 minutes with pH 2 as the optimum pH and Figure showed at Figure 7. It is known that the maximum adsorption capacity at a contact time of 75 minutes is 0.413 mg/g. The optimum time contact for the adsorption of Cr(VI) ions is 20 minutes with an adsorption efficiency of 70%. The adsorption capacity of Cr(VI) ion continued to increase from the initial time of 30 minutes to optimum time of 75 minutes, then continued decreased. This is due to the active site group on the adsorbent are released again during the shaker process which causes unstable bonds so that adsorption was not optimal (Jang et al., 2020).

In addition, it can happen due to the saturation effect of the solution which will reduce the adsorption of adsorbent molecules that have bound all metal ions so that there is no empty active site to bind metal ions remaining in the solution. The longer the time contact, the longer the collision time between

the adsorbent and metal ions so that the adsorption will be maximum. While the decrease in adsorption after equilibrium can occur due to the desorption process so that a reversible adsorption process occurs (Gokila et al., 2017).

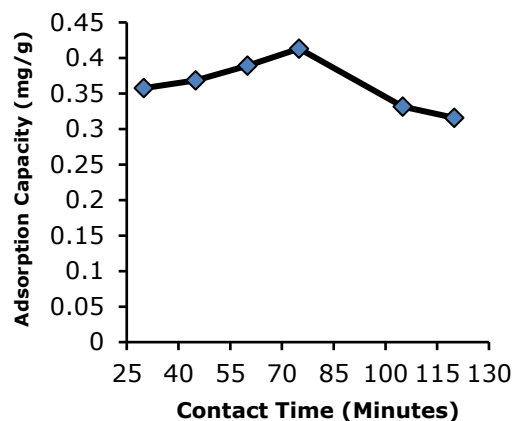


Figure 6. Effect of contact time on adsorption capacity Cr(VI).

3.9. Effect of Concentration on Cr(VI) Ion Adsorption

The higher the metal ions concentration, the adsorption will increase (Isniani et al., 2013). This is due to more and more metal ions will bind to the functional groups on the adsorbent. In this research, various concentrations of 10 mg/L; 25 mg/L; 50 mg/L; 75 mg/L; 100 mg/L; 125 mg/L, and 150 mg/L were carried out using pH 2 with a time contact of 75 minutes. As shown in Figure 8, the optimum concentration of Cr(VI) metal ion was 125 mg/L with adsorption capacity was 11.991 mg/g. The initial concentration solution was carried out in the range of 10-150 mg/L. As the concentration of the solution increases, the adsorption capacity increases. This is due to the driving force of the ions to the surface adsorbent until it reaches an equilibrium state. After reaching an equilibrium state, increasing the concentration does not increase the adsorption capacity, this is because the surface of the adsorbent is saturated with the adsorbed ions (Mondal and Chakraborty, 2020).

The effect of Cr(VI) ion concentration on adsorption capacity showed the maximum concentration at 150 mg/L with a adsorption capacity was 13.152 mg/g. This is according to research (Zein et al., 2014) that the greater

ions concentration, the greater driving force ions towards the surface of adsorbent so that the adsorption capacity increases. The higher of concentration adsorption capacity will increase directly proportional to the concentration of Cr(VI) metal ions because the greater concentration, electrostatic force interaction between Cr(VI) ions and active site group of adsorbent will increase (Gorzin and Bahri Rasht Abadi, 2018)

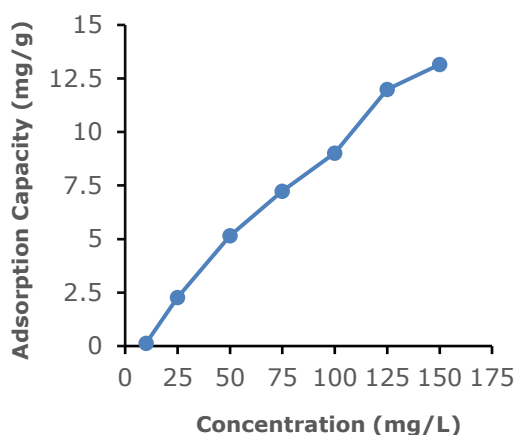


Figure 7. The Effect of Cr(VI) ion concentration on adsorption capacity.

4. Conclusion

The sugar palm (*Arenga pinnata*) fruit peel immobilized Ca-alginate has been effectively adsorption of Cr(VI) ion in aqueous solution. The SPFP biomaterial contains of hydroxyl, carbonyl, aldehyde and carboxyl groups in the FTIR result showed that the adsorbent can be used for the adsorption process of Cr(VI) metal ions. SEM EDX characterization showed that adsorbent has wide pores as a place of adsorption and there are elements of C, N, O and Ca which are common elemental compositions found in adsorbents for the adsorption process. Some variable adsorption metal ions such as pH optimum at pH 2 with a contact time of 75 minutes and an optimum concentration of 150 mg/L with an optimum capacity of Cr(VI) ion was 13.152 mg/g. The present adsorbent could be used for adsorption of metal ions from aqueous solution.

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