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POTENTIAL OF CELLULOSE FIBERS FROM SUGAR PALM (*Arenga pinnata*) SEED SHELL FOR REMOVAL OF Cr(VI) IONS

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Abstract. *It is well-known that chromium containing wastewater is one of the major pollutants of the environment. The removal of Cr(VI) from wastewater by adsorption approach using bio-sourced materials remain an attractive way since combines reasonable cost and renewability character. In the present study, it was demonstrated that the cellulose fibers could be a good candidate material for removing the Cr(VI) ions from aqueous solution via adsorption mechanism. Herein cellulose fibers were successfully extracted from sugar palm (Arenga pinnata) seed shell using alkaline and bleaching treatments. The characteristics of the as-prepared cellulose fibers were evaluated using several characterization techniques, and then used as bio-absorbent for removal of Cr ions from concentrated aqueous solutions using batch experiments. The effect of pH, contact time, Cr ions concentration on the removal capacity was evaluated. It was found that the performed adsorption studies determined that the adsorption equilibrium was reached within 120 min, and the highest adsorption capacity was for the pH of 3. The isotherm study showed that the maximum adsorption capacity was 20.34 mg/g*

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Keywords: *cellulose; sugar palm (Arenga pinnata); adsorption; Cr(VI).*

1. Introduction

One of the metals that has received much attention as an environmental contaminant and a potential hazard is hexavalent chromium or Cr(VI). Cr(VI) is persistent, bioaccumulative, toxic metal, unable to decompose in the environment, and accumulates in the human body through the food chain [1][2]. Chromium is released into the environment with the most significant releases in the industry. Industries that remove a lot of chromium metal into the environment are metal processing, tanning facilities, chromate production, stainless steel welding, chrome pigment production. The maximum allowed chromium level in industrial waste is 0.5 mg/L. The entry into the human body of large amounts of chromium can cause health problems because chromium harms the liver kidneys and is toxic to the protoplasm of living things. It is also a carcinogen (cancer-causing), teratogen (inhibitor of fetal growth), and mutagen [3].

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One method that can reduce pollution due to metal ions Cr(VI) is the adsorption method. The adsorption process has advantages such as not causing toxic side effects, removing organic materials, and being more economical (Fig. 1). The adsorption process can be carried out using natural materials from agricultural residues [4][5].

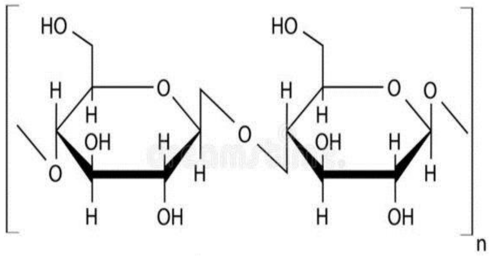


Figure 1. Chemical Structure

One of the natural materials widely used as an adsorbent is cellulose. Cellulose is a polymer with a long chain of D-glucose (pyranose) units joined with single oxygen atoms between C1 of one pyranose ring and C4 of the following ring called -1,4 glucopyranose bonds [6]. The cellulose can absorb metal ions Cr(VI) because a –OH (hydroxyl) group is in the cellulose structure. Positively charged metal ions will be bound to hydroxyl groups rich in electrons [7]. In addition, heavy metal ions will be attached to hydroxyl groups that have pairs of free electrons to act as ligands. The mechanism of complex formation occurs in the adsorption process [8]. This mechanism occurs because the oxygen atom in the hydroxyl groups has a pair of free electrons. In contrast, the heavy metal ion has empty d-orbitals to be filled by free electrons from the oxygen atom in the –OH groups [9].

Some of natural fibers have been used as alternative biomaterials such as natural fiber composites are used instead of environmentally friendly materials synthetic materials are harmful and help control pollution problems [10], sustainable bio-based material as assessment on bio-fibers, biofilm and biopolymer and bio-composite [11], [12], and reinforced hybrid polymer composite [13].

The existence of cellulose in nature is incorporated in lignocellulose bonds (lignin, cellulose, and hemicellulose). The cellulose as an adsorbent of metal ion Cr(VI) was obtained from breaking the bond between cellulose with lignin and hemicellulose. Cellulose will have good physical properties because lignin is rigid and brittle. The presence of lignin bonds in cellulose can act as an inhibitor of the adsorption process due to the rigid structure of lignin. The structure of cellulose, which is rich in pores and the –OH groups, will be covered with a rigid lignin structure because lignin is a wrapper or protector of cellulose. In addition, there is a methyl group (-CH_3) in lignin, which can be a steric factor or steric barrier in the adsorption process using lignin adsorbents. The steric factor is caused by the difference in the bonding of the atoms or groups in their space. The

–CH_3 group is a group that has a significant molecular weight, causing lignin to be very bulky (occupying an ample space). The state of space (bulk) in lignin makes it difficult for lignin to interact with heavy metal ions, which will accept a lone pair of electrons from lignin so that lignin is not stronger to be used as an adsorbent [14].

A promising material which could be turned into adsorbent is cellulose nanofibrils (CNF). One of the plants that can become an adsorbent is sugar palm (*Arenga pinnata*), the sugar palm seed shell has a lot of active chemicals compound like as lignin, hemicellulose and cellulose and it have functional groups such as alcohol and carboxyl [6]. In the processing of CNF is the content of lignin, lignin and hemicellulose form the matrix in natural fibers while cellulose strengthens them. Therefore, to extract CNF, precursors with low lignin content are preferred. Acidified chlorite bleaching can be used to remove lignin and its surface carboxylation although this process was originally used to determine holocellulose content. The cellulose fibers from agro waste material have been used as adsorben. The cellulose micro fibrils from Cocoa bean shell with tendril helical morphology has been used for removal dye. [15] and highly hydrated cellulose microfibrils (CMFs) from coffee pulp waste using specific chemical treatments has been used for water treatmen [16], Cellulose nanofibrils were prepared from kenaf core (KC) has been used for removal methylene blue [17].

In this study, cellulose fiber was separated from lignin through a hydrolysis process using H_2SO_4, the fiber was bleached with H_2O_2. The cellulose used as an adsorbent for Cr(VI) ions by studying some parameters adsorption like as pH, contact time and concentration Cr(VI) ions.

2. Materials and Experimental Details

2.1. Materials.

Sugar palm seed shell (*Arenga pinnata*) used form cellulose extraction was obtained from Setiris Village, Ma. Jambi, Jambi Indonesia. The chemicals analytical grade reagent used in this research are ethanol 70% (Sigma Aldrich), toluene 99,9% pro analysis (Merck), HCl 38% (Merck), NaOH 99% (Merck), H_2O_2 25% (Merck), HNO_3 65% (Merck), H_2SO4 98% (HmbG Chemicals), and $K_2Cr_2O_7.5H_2O$ (Merck), and distilled water.

2.2. Fiber extract and preparation of cellulose

The sugar palm seed shell used was an old sugar palm fruit. The raw product was dried in sunlight for 5 days. The dried seed was crushed using a grinder to obtain a powder. The sugar palm seed shell powder was sieved using a 120 mesh sieve to get powder. 15 g of palm seed shell powder fibers was waxed by soxhletation in 180 mL of a mixture of ethanol : toluene (1:2) as a solvent using extractor for 6 hours. The obtained fibers were washed several times with distilled water to remove the traces of toluene and were soaked in 2% NaOH solution overnight. After that, The fibers were treated were heated with 8% NaOH solution in 1:8 ratio at 60 ± 10 °C for 4 h to remove the excess lignin associated with cellulose fibers. The fibers were then subjected to steam treatment in alkaline medium (8–10% NaOH) in an autoclave at pressure of about 20 bars for 4 h at 180 ± 10 °C, followed by sudden release of pressure causing mechanical the disintegration of fibers [14]. The fibers were washed with distilled water several times untill the pH turned neutral. Then the obtained fibers were subjected to a bleaching treatment using 8% H_2O_2 solution for 24 h followed by washing for complete removal of hemicellulose. Finally, the cellulose fiber were washed with distilled water and the pH was neutralized followed by drying in oven at 60 °C.

2.3. Characterization of Cellulose fibers

Fourier Transform Infrared (FTIR)

The FTIR analysis was performed using a PerkinElmer FTIR spectrometer (Nicolet Nexus 670 instrument). FTIR test was used to identify the functional groups of cellulose before and after chemical treatment. Spectrum scans recorded in the wavenumber range 4000–400 cm^{-1}.

Scanning Electron Microscopy

The surface morphology of cellulosic fibers was observed using Scanning Electron Microscopy (SEM), Model: Hitachi SU8010 Field E with a voltage of 20 kV and a probe current of 8 mA. The test sample is placed on the SEM sample stub. The pre-prepared sample was coated with carbon and then further coated with gold to reduce the electron charge and to avoid overcharging.

2.4. The adsorption kinetic study process of Cr(VI) ions

Stock solution of 1000 mg/L Cr(VI) concentration was prepared by dissolving 5.65 g of $K_2Cr_2O_7.5H_2O$ into 1000 mL of distilled water.

The study process Cr(VI) ions conducted as followed : 0.1 g the cellulose powder was mixed with a Cr(VI) metal solution adjusted to a particular concentration, volume, and pH into a 100 mL erlenmeyer glass. The adsorption process was carried out with variations in pH (2, 3, 4, 5, 6, and 7) for 15 minutes. The pH was adjusted using a 0.1 M HNO_3 solution or 0.1 M NaOH.

The adsorption kinetic study of Cr(VI) ions conducted as followed : 0.1 g the cellulose powder was mixed with a Cr(VI) metal solution adjusted to a particular concentration, volume, and pH into a 100 mL erlenmeyer glass. The adsorption process was carried out with variations in with variations in contact time (15, 30, 45, 60, 75, 90, 120, and 150 minutes).

2.5. Isotherm Adsorption Study of Cr(VI) Ions Concentration.

The studied concentrations of Cr(VI) solution were 10; 25; 50; 75; 100; 125; and 150 ppm with the optimum pH and contact time obtained and has been carried out by batch method. As much as 0.1 g adsorbent was added to a flask containing 100 mL of Cr(VI) ions and shaker at 150 rpm, at optimum time and immediately filtered using whatman filter paper. The concentration of Cr(VI) ions in the filtrate was determined using Atomic Absorption Spectroscopy (AAS- Hitachi). Amount of adsorbed Cr(VI) ions per adsorbent mass unit at equilibrium is calculated using the following equation :

$$Q_e = \frac{C_o - C_e . V}{m} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(1)}$$

where are :

Q_e = Adsorption Capacity (mg/g)
C_o = Initial concentration (mg/L)
C_e = Final concentration (mg/L)
m = Adsorbent mass (g)
V = Solution volume (L)

3. Results

3.1. Analysis of Cellulose fiber AP

Dewaxing. The dewaxing treatment aims to dissolve extractive compounds, which are compounds other than lignin, cellulose and hemicellulose, such as waxes, secondary metabolites, natural pigments that act as coloring agents, and fats [18]. The dewaxing process uses organic solvents are ethanol and toluene. These two solvents will extract extractive compounds according to their properties, according to the rule like dissolves like, which means that polar solvents will dissolve polar compounds. In contrast, non-polar solvents will dissolve non-polar compounds.

This process is carried out using soxhletation with the working principle of the extraction process being carried out repeatedly using a relatively constant organic solvent, and the results obtained are perfect [19]. During the soxhletation process, the colour of the solvent will change to brownish-yellow, which indicates the presence of extractive compounds extracted. The soxhletation process will continue until the color of the solvent changes to clear again. The Palm seed shell powder was reduced after undergoing the dewaxing process from 45 - 41.14 g.

Delignification. Delignification is the process of separating or breaking cellulose's bonds with lignin and hemicellulose. The lignin and hemicellulose bonds need to be separated because they can interfere with cellulose to bond with Cr(VI) metal ions. The delignification process was carried out using 4% NaOH solvent, and NaOH can cause breaking of bonds between lignin and cellulose, lignin and hemicellulose, and bonds between lignin [21].

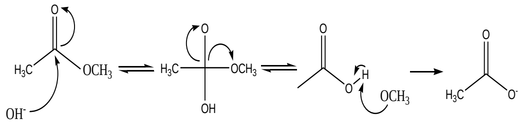


Figure 2. Mechanism of the hydrolysis reaction of OH^- ions to ester bonds in lignocellulose

Figure 2 shows the reaction mechanism of the hydrolysis of OH- ions to the ester bonds in the lignocellulosic structure. Lignin is linked to hemicellulose due to a complex structure called Lignin-Carbohydrate (LCC). This LCC links phenolic lignin units and arabinoxylan (hemicellulose) using ferulic acid, and arabinoxylan are linked by ester bonds which are very susceptible to OH^- ions because they can increase the rate of hydrolysis that occurs than water. In addition, ester bonds exist between the bonds that connect lignin to lignin and lignin to cellulose. The mechanism that occurs is that the OH^- ion will attack the carbon in the ester bond so that the double on the oxygen atom will be released and become a negative oxygen atom. However, the negative oxygen atom quickly removes the alkoxide group ($-OCH_3$) and restores the double bond to the oxygen atom to form a carboxylic acid. The resulting $–OCH_3$ group will act as a base and then deprotonate the carboxylic acid so that the H^+ cation is released (Modenbach dan Nokes, 2014). In addition, Na^+ ions will bind to phenolic groups on lignin and form phenolic salts (Sodium Phenolic), which are readily soluble. The dissolution of lignin into NaOH solution is indicated by the color of the solution changing to a dark black color (black liquor) [23].

Bleaching. Bleaching is a step that is carried out after the delignification stage because this stage aims to increase the purity of the cellulose extract obtained. In addition, the bleaching step serves to increase the whiteness by removing the light-absorbing chromophore in the lignin functional group and the remaining modified lignin. It is indicated by the colour of the cellulose extract powder, which is still dark, such as brown or black [24]. The bleaching process is carried out using a solution of hydrogen peroxide (H_2O_2). Color changes can occur because H_2O will decompose into HOO^-. H_2O_2 is a potent oxidizing agent that can oxidize natural dyes or natural pigments, which are organic compounds and have double bonds into simpler compounds, into a single bond as a stable white color is obtained. The optimum H_2O_2 works in an alkaline or alkaline environment, which causes more HOO^- groups so that the oxidation process of the chromophore groups in the lignin structure will also accelerate [25].

In Figure 3 showed the mechanism of lignin decomposition using H_2O_2. About 90% of the lignin structure is composed of phenolic content, and H_2O_2 can oxidize phenolic units in the lignin structure to form cation radicals. The Cα with Cβ bonds can be broken using H_2O_2 so that the lignin ring opens and can react with other reactions. The oxidation of non-phenolic aromatic compounds is catalyzed by H_2O_2 to form aryl cation radicals. Hydrogen catalyzes the oxidation of non-phenolic lignin compounds in the presence of a change from veratryl alcohol compounds to veratryl aldehyde [26].

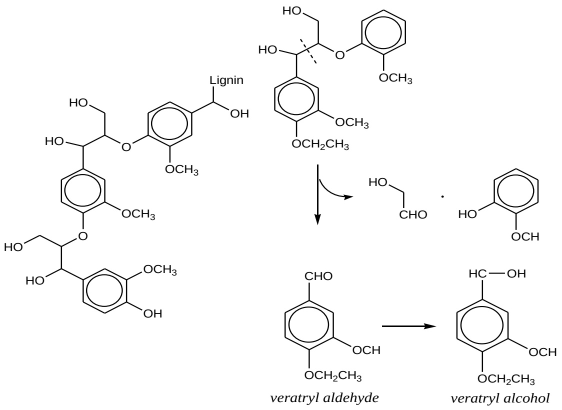


Figure 3. The mechanism of the lignin decomposition reaction using H_2O_2

3.1. Characterization of cellulose

Fourier Transform Infrared (FTIR)

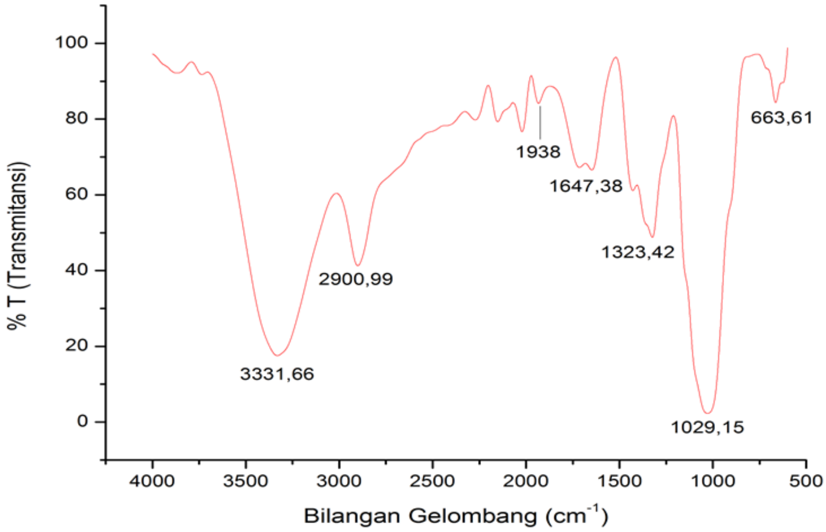
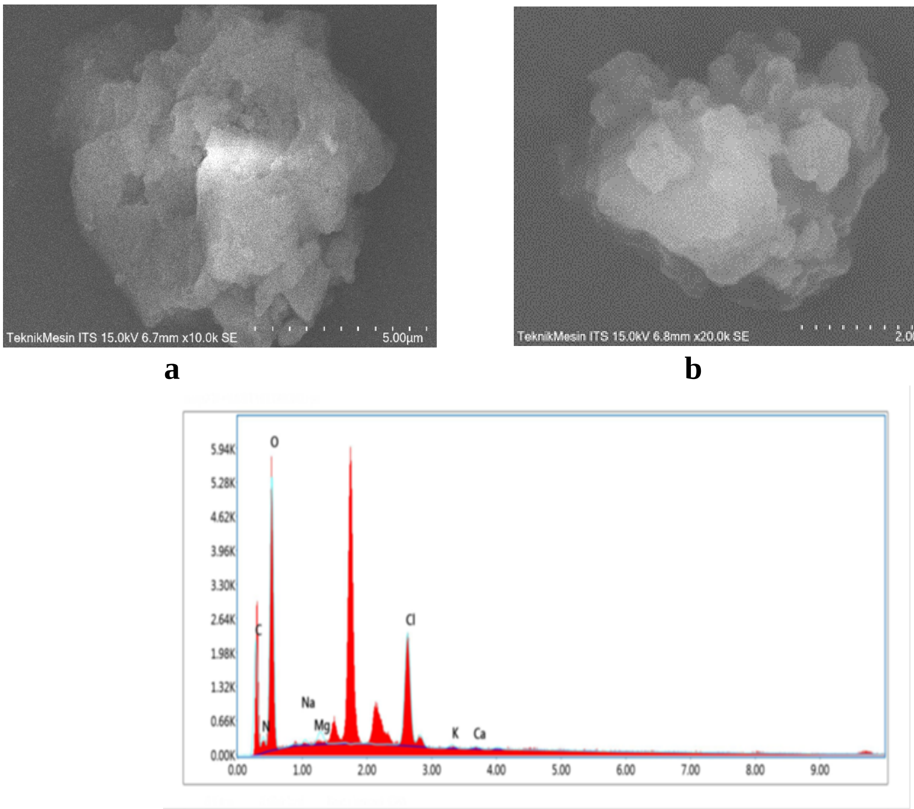


Figure 4. FTIR Spectra of cellulose AP

Figure 4 showed the specific absorption of functional groups in cellulose at wavenumber 3331.66 cm^{-1} is the stretching vibration of the hydroxyl group (-OH). At wavenumber 2900.29 cm^{-1} shows stretching vibration of $-CH_2$ group. The emergence of -OH and $-CH_2$ groups are groups found in the structure of cellulose. At a wavenumber of 1029.15 cm^{-1} is the stretching vibration of the C-O group, which is the connecting group of the carbon chain in the cellulose structure or is called a glycosidic bonding. The peak at 1323.42 cm^{-1} is a stretching vibration of -OH. In addition, the FTIR spectrum shows the absorption of the lignin functional group in the C=O and C=C groups at wavenumbers 1938 cm^{-1} and 1647.38 cm^{-1}, respectively.

SEM-EDX analysis



a **b**

c

Figure 5. SEM image of cellulose AP 10.000x (a) and 20.000x (b) magnification and EDX analysis (c).

The figure 5 shows that the results of the cellulose characterization obtained using the SEM-EDX instrument at magnifications of 10,000x, and 20.000x, the cellulose surface has cavities that are pores in cellulose. It is indicates that the delignification and bleaching processes have been successfully combined to sever and dissolve the lignin content. At 1000x magnification, the cellulose material has agglomeration. It is usually occurs in the natural cellulose extract process due to van der Waals forces [27]. The EDX image of the cellulose obtained contains elements C, N, O, Na, Mg, Cl, K, and Ca. Elements C and O are the main components in cellulose materials. Elemental Na is the residue from the delignification process using NaOH solution, which shows less than optimal neutralization and drying treatment. While the elements K, Cl, Ca, and Mg are inorganic elements or are the remaining nutrient elements, indicating that the dewaxing process is less than optimal. The percentage of cellulose constituent elements, which consists of C 30.10%, N 5.61%, O 49.22%, Na 0.63%, Mg 0.66%, Cl 12.78%, K 0.53%, and Ca 0.47%.

3.2. The Adsorption of Cr(VI) Ions

The effect of pH on adsorption capacity of Cr(VI) ions

The effect of pH on the adsorption capacity of Cr(VI) metal ions can bee seen in Figure 6.

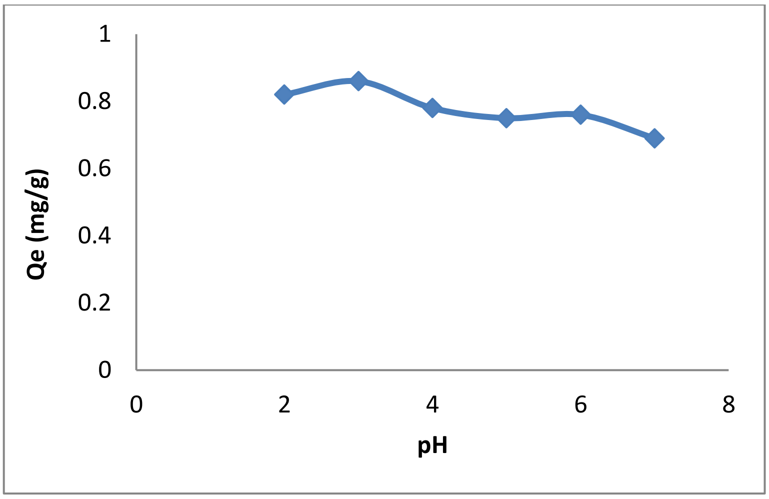


Figure 6. The effect of pH on the adsorption capacity of Cr(VI) ions.

The optimum adsorption capacity occurred at a low pH, especially pH 3 of 0.86 mg/g. The adsorption of Cr(VI) ions was carried out with cellulose adsorbent, pH is obtained under acidic conditions at pH 4 with an adsorption capacity is 0.06 mg/g [28]. At low pH, protonation will occur, in which H^+ ions on the surface of the adsorbent will have positive charges so that they are very reactive to speciation in the form of anions. At high pH, the functional group of -OH on the cellulose surface adsorbent will deprotonate and have negative charges, thereby reducing the adsorbent's ability to bind Cr(VI) metal anions.

The Cr(VI) has been ionic forms different in the solution depending on the pH of the solution. In the range of pH 2 to pH 6, metal ions Cr(VI) are in the form of anions such as $Cr_2O_7^{2-}$, $HCrO_4^-$, $Cr_3O_{10}^{2-}$, and $Cr_4O_{13}^{2-}$ and the dominant anion is $HCrO_4^-$. At low pH conditions, the H^+ present on the surface of the cellulose adsorbent will increase, and the positive charge on the adsorbent surface with dichromate ions will produce a strong electrostatic bond. Meanwhile, at high pH conditions, the OH^- in the solution will increase and cause the surface of the cellulose adsorbent to be negatively charged slowly. As a result, the strength of the adsorbent to bind Cr(VI) metal ions will decrease and reduce the ability in the adsorption process. In addition, Cr(VI) metal ions at high pH will experience precipitation to become $Cr(OH)_3$, which causes a decrease in the solubility of Cr ions in solution so that the cellulose adsorbent absorbs only a few Cr(VI) ions.

The Effect of contact time on adsorption capacity of Cr(VI) Ions

The effect of contact time on adsorption capacity of Cr(VI) ions can bee seen in Figure 7.

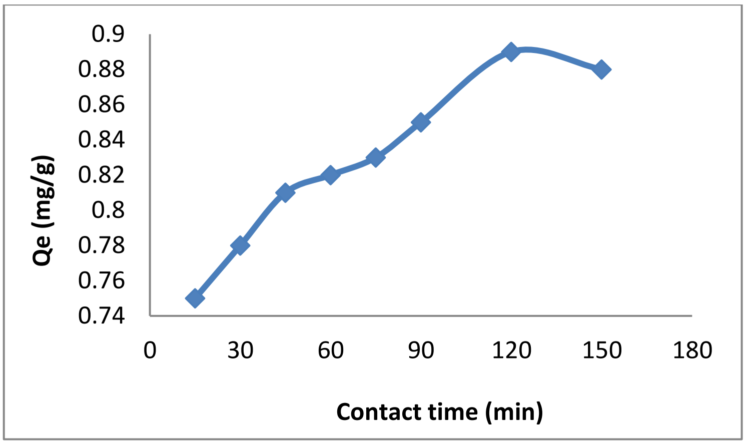


Figure 7. Effect of contact time on adsorption capacity of Cr(VI) ions.

The adsorption capacity of Cr(VI) ions at a contact time of 15-120 minutes increased, and then after 150 minute the adsorption capacity of Cr(VI) ions decreased The optimum adsorption capacity of the contact time was obtained at 120 minutes of 0.89 mg/g. The optimum adsorption capacity was received at the contact time of 60 minutes of 0.51 mg/g. Under these conditions, the adsorbent has reached its maximum limit because the functional group on the adsorbent has bound metal ions Cr(VI). Then, there is a decrease in adsorption capacity because the adsorbent has reached saturation, and the desorption process occurs. Meanwhile, compared with the results obtained in this study, it has a greater yield due to the extraction of the cellulose adsorbent through the delignification and bleaching stages. The steps have the function to decide and dissolve the lignin content, causing more pores on the surface of the adsorbent and can bind more metal ions Cr(VI) [29].

The constant increase in adsorption capacity indicates that the longer the contact time, the more Cr(VI) metal ions are adsorbed on the cellulose adsorbent. The longer the time, the longer the collision time and the interaction between the adsorbent and Cr(VI) metal ions. More active groups on the cellulose adsorbent bind to Cr(VI) metal ions. At a contact time of 120 minutes, the adsorbed Cr(VI) metal ion had reached its maximum limit, causing a decrease in the adsorption efficiency at 150 minutes. This decrease occurred because the active groups on the cellulose adsorbent had been saturated so that the cellulose adsorbent was unable to absorb more Cr(VI) metal ions.

The effect of concentration of Cr(VI) ions.

The effect of concentration Cr(VI) ions on adsorption capacity can bee seen in Figure 8.

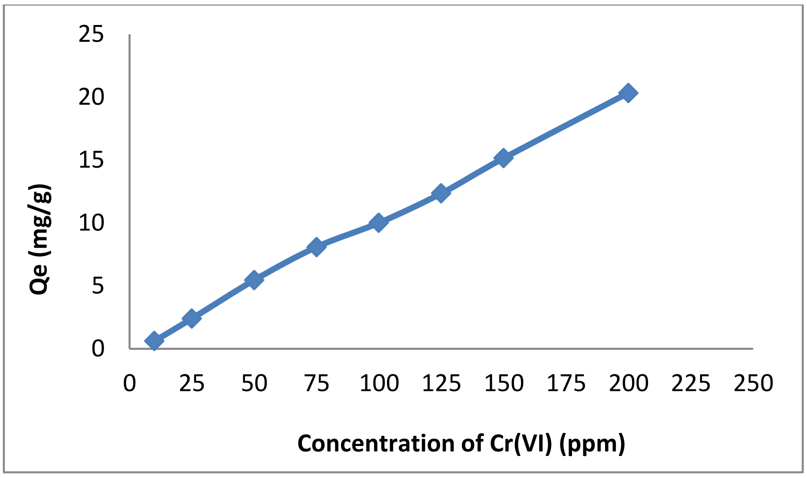


Figure 8. Effect of concentration on the adsorption capacity of Cr(VI) ions.

The initial Cr(VI) ion concentration affected to adsorption process on cellulose AP. Adsorption capacity Cr(VI) ions increased from 10-150 ppm. The maximum adsorption capacity of Cr(VI) metal ion was obtained at a concentration of 150 ppm of 15.15 mg/gr. The increase in adsorption capacity occurs because the metal ion Cr(VI) contained in the solution increases. There is also an increase in collisions between the adsorbent and the adsorbate. Then it causes a lot of Cr(VI) metal ions to be adsorbed on the cellulose adsorbent. There was an adsorption capacity increased at a concentration of 250 ppm with an adsorption capacity of 21.980 mg/g. The surface of the adsorbent is still able to adsorb metal ions Cr(VI) because the increasing concentration of metal ions will encourage metal ion molecules to bind to the active groups on the surface of the adsorbent so that it reaches a state of equilibrium. If a higher concentration is used, saturation will be found on the adsorbent because the active groups on the surface of the adsorbent have been bound and covered by metal ions so that the adsorbent is no longer able to absorb the remaining metal ions [30].

Conclusion

The cellulose fibers has been used potential to adsorber of Cr(VI) ions in aqueous solution. The characterized of cellulose fibers using SEM-EDX instrument showed cavities on the surface of cellulose, which indicated success in the delignification and bleaching process, and the using FTIR instrument which indicate there are -OH, $-CH_2$, and -C=O functional groups which are the building blocks of the structure cellulose. The effects of several adsorption parameters, such as pH, contact time, and Cr(VI) ions concentration on the metal uptake, were investigated. The effect of adsorption study Cr(VI) ions parameter using cellulose resulted in the optimum pH at pH 3 with an adsorption capacity is 0.86 mg/g, the optimum contact time at 120 minutes with an adsorption capacity is 0.89 mg/g, and the concentration of Cr(VI) ions at 150 ppm with adsorption capacity is 20.34 mg/g.

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