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SYNTHESIS OF Na-CMC MODIFIED CELLULOSE MEMBRANE FROM WATER HYACINTH (*Eichhornia Crassipes*) RODS AGAINST Cr (VI) METAL ADSORPTION

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ABSTRACT

Metal ion Cr (VI) is a type of heavy metal waste that is dangerous, so we need a method to reduce the level of pollution. The separation method using a cellulose membrane is an effective method to reduce the level of Cr (VI) metal pollution, by utilizing natural materials such as water hyacinth plants which are known to contain high enough cellulose. The purpose of this study was to determine the effect of concentration variations with time variations on the adsorption capacity of Cr (VI) metal ions from Na-CMC modified cellulose membranes from water hyacinth (Eichhornia crassipes) stems. This research uses experimental research methods, the steps that have been carried out are sample treatment, extraction, bleaching, hydrolysis, Na-CMC modified cellulose membranes. Data analysis was performed by sample characterization test using the FTIR Spectroscopy method and for metal content analysis using the Atomic Absorption Spectrophotometer (AAS) method. The results showed that there was an effect of concentration variations and time variations on the adsorption capacity of Cr (VI) metal ions. At the concentration variation with the largest adsorption capacity of 1.24 mg/gram occurred at a concentration of 30 mg/L and at the time variation the largest adsorption capacity of 0.917 mg/gram occurred at 30 minutes. The results respectively of FTIR showed functional groups O-H, C-H, and C-O in non-modified cellulose and there was no significant effect after the addition of Na-CMC, as evidenced by the similar FTIR spectrum without any shift or new peaks appearing.

Keyword: *Chromium (VI), Water Hyacinth (Eichhornia crassipes), Cellulose Membrane, FTIR, AAS*

INTRODUCTION

The rapid development of industry and the rapid growth of the world's population have led to an increase in the yield of toxic waste materials discharged into the environment. This waste material is difficult to control properly, so it will become waste that pollutes the environment. This waste will cause serious pollution to the environment if the heavy metals contained in it exceed the threshold and have very dangerous toxic properties, which will later cause serious illness for humans if they accumulate in the body. The metal ion Cr (VI) is classified as hazardous and toxic (B3), because chromium is a type of heavy metal waste that is difficult to decompose and can accumulate in the body and the environment [1]. Based on the

Regulation of the State Minister of the Environment Number 3 of 2010, concerning the quality standard of industrial estate wastewater, it is stated that the maximum concentration of Cr (VI) is 0.5 mg/L. Considering the danger of Cr metal ion, necessary to do a method to reduce the level of pollution of Cr (VI) metal ion in the environment [2].

The most common methods to remove metals from wastewater include chemical precipitation, coagulation, flocculation, reverse osmotic, membrane filtration, evaporation, and ion exchange [3]. The filtration method is a method that has been developed to handle heavy metal levels that exceed the threshold. This filtering process can be done with membrane technology. The advantages of membrane separation technology include that it does not require additional chemicals, can be combined with other processes, and the process takes place continuously. One of the membranes currently being developed is the cellulose membrane [4].

Cellulose can be obtained from materials containing high purity cellulose. In general, commercial production of cellulose comes from wood, cotton, and high-quality non-timber plant fibers, such as water hyacinth stem fibers [5]. Water hyacinth has the potential for good quality fiber and cellulose because it has a high cellulose content, which is around 60%, making it an alternative potential raw material for the manufacture of cellulose membranes.

Research [6] has succeeded in isolating cellulose from water hyacinth. Water hyacinth has a high absorption rate for various dyes and heavy metals such as iron (Fe), zinc (Zn), copper (Cu), chromium (Cr), cadmium (Cd), manganese (Mn), mercury (Hg) and arsenic. (As) [7]. Water hyacinth as an adsorbent is able to adsorb chromium (VI). However, until now there has been no research that has found about chemical modification of water hyacinth fiber to increase the adsorption capacity of metal ions [8].

Based on the above background, it is necessary to conduct research to synthesize Na-CMC modified cellulose membranes and to determine the effect of the optimum concentration and time to obtain the best membrane performance in increasing the adsorption capacity of Cr (VI).

METHODS

The tools used in this research are socket set, knife, blender, analytical balance, 44 mesh sieve, filter paper, cloth, beaker, measuring flask, measuring cup, funnel, scissors, hot plate, universal pH, thermometer, magnetic stirrer, stir bar, oven, petri dish, stainless rod, Erlenmeyer, centrifuge, FTIR and AAS. Research methods depend on the research design used.

The main ingredients used in this study were stems of water hyacinth (*Eichhornia crassipes*) obtained from the area of Karangrowo Undaan Village, Kudus Regency, Central Java with criteria for stems in good condition, fresh old condition, green color, and not rotting, distilled water, potassium dichromate ($K_2Cr_2O_7$), ethanol 96%, toluene, 1% NaOH $^b/_v$, and 3% sodium hypochlorite, 5% hydrochloric acid $^b/_v$, 2 M

sulfuric acid, 99% Na-CMC, 99.5% potassium hydrogen phthalate.

1. Research procedure

a. Membrane preparation

Water hyacinth stems were sorted, selected water hyacinth stems were in good condition, old, fresh, green, and not rotten. Then cleaned and washed with running water. Dry in the sun to dry. The dried water hyacinth stems were reduced in particle size by cutting and mashing with a simplicia blender until they became fine fibers then sieved using a 44 mesh sieve [9].

b. Determination of Water Content

Water hyacinth stem powder was weighed as much as 2 g, put in a porcelain cup, then in an oven at 105 °C for 2 hours. Then it was put in a desiccator and weighed until a constant weight was obtained.

c. Determination of Ash Content

Water hyacinth stem powder was weighed as much as 2 g and put in a porcelain dish, then in an oven at 600 °C for 1 hour. Then it was put in a desiccator and weighed until a constant weight was obtained.

d. Isolation of cellulose using the soxhletation method

Water hyacinth stem powder was weighed as much as 50 g and then carried out soxhletation using 96% ethanol: toluene in a ratio of 1:2 for 3.5 hours at 100 °C [10]. The result of this method is a brown solid and produces a brown filtrate.

e. Bleaching

The brown fiber which still contains impurities is bleached using a 500 mL 3% w/v NaClO solution and heated over a water bath at 80 °C for 2 hours while stirring to produce white fiber [11].

f. Hydrolysis

In this process, 500 mL of 1% w/v NaOH solution was used and heated at 60 °C for 2 hours, filtered, washed with distilled water until the residue was neutral, and then dried at room temperature for 2-3 days. The final bleaching process was carried out with 500 mL 1% w/v NaClO solution at 75 °C for 3 hours, stirring occasionally, filtered. The solids were filtered and washed with distilled water to neutral pH using filter paper. Pure cellulose powder is dried in a drying cabinet. After dry cellulose powder was tested using FTIR.

g. Preparation of non-modified and modified Na-CMC cellulose membranes

The cellulose membrane synthesis process consists of two stages, namely the synthesis of non-modified and modified Na-CMC cellulose membranes. Synthesis of non-modified cellulose membranes was carried out by weighing 2 grams of cellulose, added with 20 mL of aqua de mineral, and stirred homogeneously using a magnetic stirrer for 1 hour. While the manufacture of Na-CMC modified cellulose membrane by weighing 2 g of water hyacinth stem cellulose, then adding 20 mL of concentrated sulfuric acid, then stirring for 30 minutes while heated on a hot plate at 45 °C. Then 55 mg Na-CMC was added and stirred for 20 minutes at the same temperature using a magnetic stirrer. The solution formed was poured into a petri dish that had been given a plastic mold with a diameter of 7.5 cm and

waited for it to dry. This stage is a modification of the cellulose membrane to obtain a high level of adsorption of heavy metal Cr by forming complex compounds with Na-CMC. After the dry membrane was tested using the FTIR.

h. Preparation of Cr Solution

1) Preparation of standard solution of Cr 1000 ppm

A total of 3.7348 g of K_2CrO_4 was put into a 1000 ml volumetric flask, then diluted with distilled water, then added with Potassium Hydrogenphthalate (pH 3) to obtain a standard solution of 1000 ppm Cr as a stock solution. Next, a series of standard solutions and calibration curves are made. The absorbance of Cr (VI) metal standard solution was measured using AAS analyst-100 at a maximum wavelength of 545 nm.

i. Treatment and Analysis of Cr Metal Absorption on Cellulose Membranes

1) Determination of Optimum Contact Time

As much as 0.5 g of cellulose was put into an erlenmeyer containing 25 mL of Cr solution with a concentration of 20 mg/L. The solution was stirred for a contact time of 30, 45 and 60 minutes, then centrifuged at 2800 rpm for 5 minutes. The precipitate is filtered and the supernatant is measured. The metal content of Cr in the solution after adsorption was analyzed by AAS analyst-100.

2) Determination of Cr metal ion adsorption capacity

A total of 0.5 g of cellulose into an Erlenmeyer containing 25 mL of Cr metal solution was made with various concentrations of 10, 20, and 30 mg/L. The mixture was shaken and then filtered and measured. The content of Cr metal in the solution after adsorption was analyzed by AAS analyst-100.

2. Data analysis

Determination of the optimum concentration and contact time of Cr adsorption can be obtained from AAS can be calculated by [12]:

$q_e=((C1-C2))/W \times V$

Information:

q_e : Adsorption capacity (mg/g)

C_1: Initial concentration of metal (mg/L)

C_2: Final metal concentration (mg/L)

W: Mass of adsorbent (grams)

V: Volume of metal solution (L)

RESULTS AND DISCUSSION

1. Results of drying and making water hyacinth stem powder

This research begins with the preparation of water hyacinth stems by removing the outer skin of water hyacinth stems which contain a lot of lignin and other impurities. After getting clean water hyacinth stems, a size reduction process is carried out through a cutting or chopping process. This chopping aims to reduce the size, expand the surface and reduce the volume of water hyacinth. Furthermore, drying in the sun for 5-7 days. The drying process is carried out to obtain simplicia that is not easily damaged so that the simplicia can be stored for a long time, the quality of the simplicia is maintained and reduces the water content in the simplicia [13]. The water hyacinth fiber preparation process resulted in mass loss until the final mass became 87.5% of the initial mass. The dried simplicia obtained was then powdered

using a simplicia blender and sieved with a 44 mesh sieve to obtain a fine powder. The purpose of making fine powder and sieved with a 44 mesh sieve is to increase the surface area to speed up the extraction process because increasing the surface area will increase the contact between the powder and the solvent [14].

2. Water hyacinth stem powder analysis

Determination of water content is a parameter to determine the residual water after the drying process. Excessive water content can cause mold and mildew growth so that it can reduce biological activity during storage. In addition, the low water content can increase the reactivity of cellulose because the hydroxyl groups in water are more reactive than the groups in cellulose. Therefore, obtaining a high level of reactivity requires a low water content so that the substitution process can take place properly. Determination of water content was carried out 3 times replication, the results obtained an average water content of 7.461%. This indicates that the percentage of water content in water hyacinth stem powder has met the quality requirements is ≤ 10% [15].

3. Analysis of ash content of water hyacinth stem powder

In the study, the determination of ash content was carried out by direct method, namely burning the powder at a temperature of around 600 °C for 3 hours. The ash content produced from water hyacinth stem fiber is 9.205%. This indicates that the water hyacinth stem powder does not meet the requirements for ash content of SNI No. 0258-79,1989 for the adsorbent standard, which is a maximum of 2.5%. The resulting water hyacinth stem powder has an ash content value that is far from the standard ash content. This is because water hyacinth stem powder contains relatively large amounts of minerals and impurities. The more mineral content, the lower the site or active group that functions to bind Cr (VI) metal ions. Therefore, the mineral content needs to be removed so that the binding process will take place optimally. This high ash content is related to the function of water hyacinth stems as a phytoremediator [7].

4. Cellulose insulation process

This process is carried out using the soxhlet method, which functions to extract fat or wax content (dewaxing process) and remove extractive compounds contained in water hyacinth stem powder (secondary metabolite compounds other than lignin, hemicellulose, and cellulose compounds) [6]. This process produces brown fibers and produces a brown filtrate. Next is bleaching using NaClO solution which functions to remove hemicellulose and lignin compounds more optimally. NaOCl in water will produce hydroxyl ions and hypochlorous acid (HOCl) which is a strong oxidizing agent so that the degree of whiteness in a sample will increase. The use of NaClO causes an oxidation process that will later produce cellulose [5].

The hydrolysis process using NaOH solution aims to remove hemicellulose compounds. In this process, there was a change in color from the initially colorless solution to dark brown and concentrated due to the lignin and hemicellulose contained in

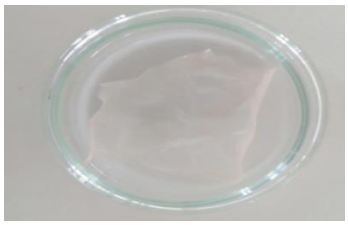
the water hyacinth stem powder which had been successfully degraded into monomers and dissolved in NaOH. Base hydrolysis is carried out to cut hemicellulose, so that it will be cut from the main chain, namely cellulose. In addition, the reaction with NaOH solution will cause lignin to be degraded due to breaking bonds [16]. The result of this delignification process was washed with distilled water until the pH was neutral. The final bleaching process with NaClO aims to remove the remaining lignin that is still contained in the water hyacinth stem powder.

5. Results of making cellulose membranes

In the process of making cellulose membranes, it is done by making non-modified cellulose membranes and modified Na-CMC cellulose membranes. The modified Na-CMC membrane produced a better membrane than the non-modified membrane. This can be seen in Figure 1 which shows that the resulting membrane is not easily brittle and quite elastic and the resulting membrane is a transparent white membrane like plastic. Meanwhile, non-modified cellulose membranes without plasticizers produce very brittle membranes and are difficult to peel off from the mold. This is because the added Na-CMC functions as a plasticizer which aims to increase the elasticity of the membrane [17].

Figure 1. Modified Water Hyacinth Stem Cellulose Membrane Na-CMC Wet

A



B

Figure 2. Non-modified Dry Water Hyacinth Stem Cellulose Membrane (A) and Na-CMC Modified Dry Water Hyacinth Stem Cellulose Membrane (B)

6. FTIR Analysis

Table 1. Test results of non-modified and modified Na-CMC cellulose membranes FTIR instruments

Wave Number (cm^{-1}) Non-modified	Wave Number (cm^{-1}) Modified	Functional Groups	Frequency (cm^{-1}) Library (Sastrohamidjojo, 2001)	Intensity
3343	3372	O-H	3500 – 3200	medium
2901		C-H	2900 – 2800	weak
1740	1737	C=O	1750 – 1730	sharp
1368		($-CH_3$)	1370	sharp
1216		C-O	1350 – 1000	sharp
	1166	C-H	1350 – 1000	Medium-sharp
1057	1042	C-O	1300 – 1000	sharp
	885	C-H alkena	1000 – 650	sharp

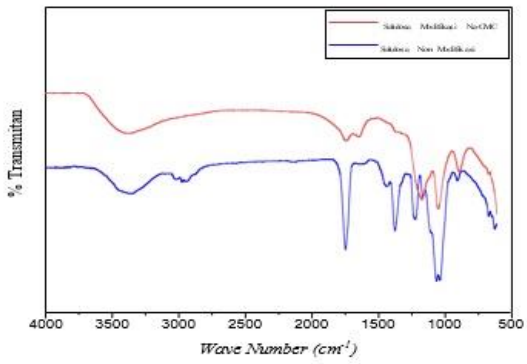


Figure 3. FTIR spectrum of modified cellulose powder (blue) and modified Na-CMC cellulose powder (red)

In Figure 3 and Table 1, it can be seen that the FTIR spectra produced from non-modified cellulose can be analyzed based on the absorption of the stretching O-H group that appears at around 3372 cm^{-1} and has the C-H stretching functional group that appears at the absorption of 2901 cm^{-1}. And the C-O functional group is seen in the 1350–1000 cm^{-1} area which appears at a wavelength of 1216 cm^{-1}. the presence of functional groups O-H, C-H, and C-O are the main groups of cellulose. From the results of the spectrum above, it can be said that in the water hyacinth stem cellulose powder there is a cellulose compound.

As for the FTIR spectrum of Na-CMC modified cellulose, it was observed that the wave number 3343 cm^{-1} was the OH group which is a characteristic of cellulose where both spectra showed the same wave absorption range. The presence of a carboxyl group at a wavelength of 1740 – 1720 cm^{-1}, namely at a wave number of 1737 cm^{-1}. C-H functional group In the absorption area of 1350-1000 cm^{-1}, namely at a wave number of 1166 cm^{-1}. At a wave number of 1042 cm^{-1} there is an absorption area of 1300-1000 cm^{-1} indicating the presence of an ether formed, namely the C-O group. The results of the functional groups measured from the FTIR spectrum with each absorption in a certain wavelength region showed that no new functional groups were formed after the addition of Na-CMC.

7. Results of Cr (VI) Metal Ion Adsorption with Variations in Concentration and Time

The adsorption capacity of Cr (VI) metal ions with variations in concentration and time were carried out to determine which absorption capacity was the best by using modified Na-CMC cellulose. A total of 0.5 g of cellulose was put in an Erlenmeyer 25 mL solution of Cr with a concentration of 20 mg/L with a pH of 3. The degree of acidity (pH) is one of the factors that affect the adsorption process. Acidity affects the ability of the charge on the active site or functional group in which H^+ ions will compete with cations to bind to the active site of the adsorbent. The reason it was carried out at pH 3 was that the acidic nature was needed so that the Cr (VI) sample solution formed ions in the solution and nothing precipitated so that it could be measured perfectly with AAS. Metals contained in water if the pH is more acidic then the solubility is greater, on the contrary if the solution is more alkaline then the solubility is getting smaller which is indicated by the presence of a precipitate. The more acidic the solution, the higher the ionization, on the contrary the more alkaline it will precipitate. It is clear that adsorption is better at high acidity levels because at pH 3 there is greater ionization and adsorption can occur if the metal forms ions and will be bound by active groups on the adsorbent. The adsorption capacity at various times can be seen in Figure 4.

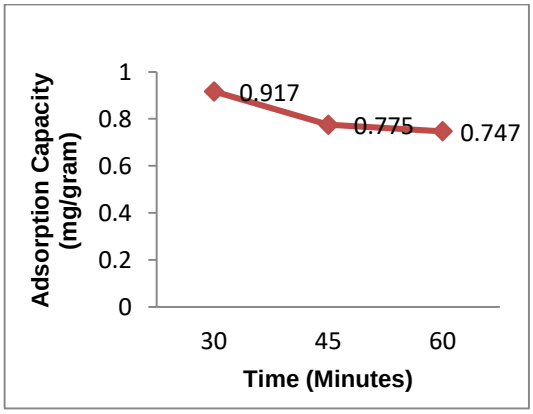


Figure 4. Graph of Relationship between Adsorption Time and Capacity (mg/gram) on Na-CMC modified cellulose membrane

Figure 4 shows that the adsorption capacity decreased from 30 minutes to 60 minutes. This shows that 30 minutes is the highest time for Cr (VI) metal ion adsorption with an adsorption capacity of 0.917 mg/gram. Then within 45 and 60 minutes of experiencing saturation. The process of releasing ions that have been bound to the active group or have experienced saturation. In research based on the determination of the adsorption capacity value, the longer the contact time, the lower the adsorption capacity level. It is advisable to do a second adsorption process with a sampling time of 10 minutes to ensure that the 30th minute is the optimum minute.

As for the adsorption capacity, the optimum concentration was also carried out with the same process with variations in concentrations of 10, 20, and 30 mg/L. Determination of the adsorption capacity of metal ions Cr (VI) was calculated at the optimum time. In this study, the optimum time obtained was 30 minutes. The largest absorption of Na-CMC modified cellulose occurred at a concentration of 30 mg/L, with an adsorption capacity of 1.24 gram/gram. Based on the results of determining the value of adsorption capacity with variations in the concentration of Cr (VI) metal, it shows that the higher the concentration of Cr (VI) metal, the more Cr (VI) metal that is able to bind to the OH group of cellulose.

Figure 5 shows that the adsorption capacity increases with increasing concentration. This is related to the number of active sites on the surface of the adsorbent capable of binding the metal ions. If the number of active sites is quite large compared to the number of metal ions, the adsorption capacity or absorption will be high [18]. The mechanism of binding of Cr metal to cellulose can be seen in Figure 5.

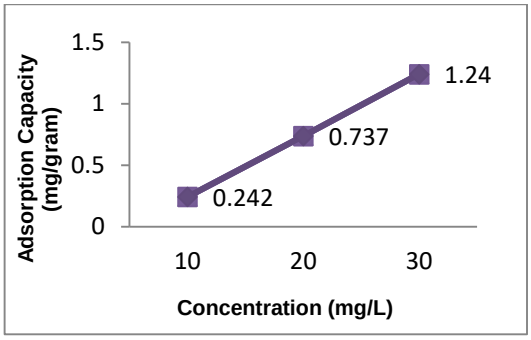


Figure 5. Graph of the relationship between concentration variations and adsorption capacity (mg/gram) on Na-CMC modified cellulose membrane

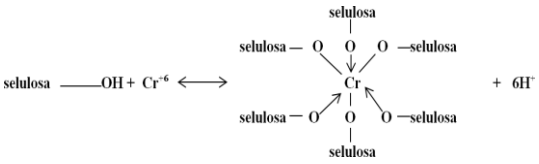


Figure 6. Possible Interaction Between Cellulose and Metal Ions Cr (VI)

In Figure 6 the sign → is a coordinating covalent bond and the sign is a covalent bond. The interaction between cellulose and Cr (VI) is possible due to a covalent bond where the bond occurs due to the sharing of electron pairs by the two bonding atoms and coordination covalent bonds where bonds are formed by sharing electron pairs from one atom. bonded (O).

CONCLUSION

Based on the results of the tests that have been carried out, it can be concluded that the concentration and time affect the adsorption capacity of Cr (VI). At the concentration variation with the largest

adsorption capacity of 1.24 mg/gram occurred at a concentration of 30 mg/L and at the time variation the largest adsorption capacity of 0.917 mg/gram occurred at 30 minutes. The characteristics of the functional groups of Na-CMC modified cellulose using FTIR did not give a significant effect, indicated by the absence of shifts or the appearance of new peaks.

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