Preparation and Characterization of Chitosan with Activated Carbon as Adsorbent to Reduce Level Metal Cadmium (Cd) and Nickel (Ni)

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Abstract: Preparation and characterization of chitosan with activated carbon have been made with the aim of reducing the metal content of Cadmium (Cd) and Nickel (Ni) in standard solutions. Characterization chitosan with activated carbon by FT-IR, SEM, and test adsorption by using AAS. Characterization of chitosan and chitosan - activated carbon by FT-IR shows that there is no difference in wavelength: as for the emerging groups, NH groups (3448.72 cm^{-1}), CH groups (2924.09 cm^{-1}), C = C groups (1635.64 cm^{-1}), C-N group (1381 cm^{-1}), and NH group (3441.01 cm^{-1}). SEM characterization on chitosan - activated carbon shows a rude surface. Absorptions of Cd and Ni in chitosan that best with addition carbon of 0.6 g that is 74.54% and 73.43%.

1 INTRODUCTION

Industrial activities which are rapidly developing have made the contamination of heavy metal ion in water increase. This condition has caused serious environmental problem throughout the world (Li et al., 2016). Heavy metal from industrial waste such as lead, copper, and cadmium can pollute water, sea level, and soil. The International institution has confirmed that cadmium is latent metabolic poison since it is very dangerous for the life of organism and can affect human health.

Electroplating industry has significant risk for environment and human beings because wastewater contains heavy metal ion which is not biodegradable and tends to be accumulated in living organism which causes toxic effect or carcinogenic. One of the contents found in electroplating waste is nickel metal; this metal is usually used in electroplating industry due to its anti-corrosion (Raja Sulaiman et al., 2018). Nickel is a silver white, hard and ductile metal. Ni normally forms cubic crystal lattice (Coman et al., 2013). Nickel is used for production of stainless steel, nonferrous alloys and anticorrosion and temperatures resistance properties.(Reck *et al.*, 2008)

According to the International Board for cancer study, cadmium is known as carcinogenic (Jeon, 2017) . It is accumulated in human kidneys and liver, and it can cause various diseases such as kidney dysfunction, hypertension, diarrhea, stomach-ache, and bone disorder (Pal and Pal, 2017). Waste which contains cadmium metal comes from electroplating industry, the making of batteries, pesticides, and mining which bring about water, air, and soil pollution (Al-Malack and Dauda, 2017).

There are several methods used to remove heavy metals from wastewater is precipitation, membrane filtration, ion exchange (Hegazi, 2013). The adsorption method is the most commonly used because it is more efficient, more economical and uses cheap natural adsorbents (Li et al., 2016).

Chitosan is poly-(2-amino-2-deoxy-*β*-(1-4)-*D*-glucopyranose) with the molecule formula of $(C_6H_{11}NO_4)n$ which can be obtained from chitin deacetylation (Rahate, 2013).

Its molecular weight between 300 – 1000 kDa. Chitosan produced from crustacean shell such as crab and shrimp. These shells contain 30 – 40 proteins, 30 – 50% calcium carbonate and 20-30% chitin. Chitosan is natural chelating make chitosan useful in wastewater treatment by allowing for the binding and removal of metal ions such as copper,

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lead, mercury, and uranium from wastewater. Chitosan (Figure 1) has advantages such as biodegradability, natural origin, abundance, reactivity. It has many application include medical, agricultural, food processing, nutritional enhancement, cosmetics and waste and water treatment. (Paridah et al., 2016).

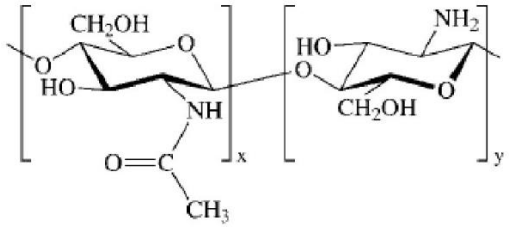

Figure 1: Chemical structure chitosan.

Chitosan is absorbent which is very effective to remove heavy metal ion because chitosan structure has abundant hydrophilic hydroxyl group and polycationic amine group which can bind metal (Ahmad et al., 2017). Among various natural polymers, chitosan is the second largest biopolymer in nature after cellulose (Choi et al., 2016). It is a very effective natural polymer which can be used as absorbent because it is biodegradable (Ahmad et al., 2017) and not poisonous (Choi et al., 2016); it also has bio-compatibility (Liu and Bai, 2014) and bioactivity (Vakili et al., 2014).

Chitosan has several weakness is mechanical properties and low thermal stability, porosity and small surface area. Vakili *et al.*, 2014 modified the structure of chitosan into chitosan beads, membranes and films, to improve adsorption ability, physical and mechanical properties. Liu and Bai, 2014 explained that modifying chitosan into semi-IPN (Interpenetrating Network) hydrogel, nano-magnetic particles, chitosan grafting polymers, and chitosan composites can improve chitosan adsorption ability.

Coffee is the most common drink which is consumed throughout the world. It reaches 400 billion glasses per year and produces around 8,000 tons of coffee grounds per year. Coffee ground is still considered as waste since it takes a very long time to be decomposed, compared with the other types of waste (Zein et al., 2017).

One of the advantages of coffee grounds is that they can be used as absorbent, tannin compounds which contain polyhydroxy and polyphenol groups which can be bound with metal cation which forms chelate (Utomo and Hunter, 2006). Besides that, coffee grounds also contain carbon, nitrogen, lipophilic compound, ethanol, lignin, alkaloid, polysaccharide, and chlorogenic acid (Pujol et al., 2013).

The ingredients which can be used for carbon are tea, coffee grounds, and rice husk. Carbon from active charcoal can absorb inorganic contaminants (Djati Utomo, 2015). Carbon from active charcoal is usually used for absorbent since it is flexible and more effective; besides that, it has wider surface and good capacity to decrease metal and the other poisonous compounds (Salehi et al., 2016) (Zhang et al., 2016).

(Hernández Rodiguez et al., 2018) reported that adsorption of Nickel from aqueous solutions of activated carbon from spent coffee. Activated carbon is effective adsorbent for decrease concentrations of metal ions in aqueous solutions. These adsorbents have high amount of micropores and mesopores, large surface area.

2 MATERIALS AND METHODS

2.1 Material

All materials used in this research were of chitosan, acetate, aquadest, NaOH, activated carbon, standard solution of Cd^{2+} 1000 mg/L, and standard solution of Ni^{2+} 1000 mg/L.

2.2 Methods

2.2.1 The Making of Chitosan

Chitosan 1.2 grams of, dissolved with 3% of acetate, about 60 mL, and stirred until it became homogenous. It was then poured into acrylic glass and dried up in a oven at the temperature of 60^0C within 24 hours. The result was immersed with NaOH 1M within 24 hours. It was then removed from the acrylic glass and washed with aquadest until it was neutral. Finally, it was dried up in room temperature and stored in desiccators. yielded absorbent was analyzed by FT IR, SEM, and tensile strength testing.

2.2.2 The Making of Chitosan – Activated Carbon

Chitosan 1,2 g, dissolved with 3% of acetate about 60 mL, added by 0.3 gram of activated carbon, stirred until it was homogenous, poured into acrylic glass, dried up in an oven at the temperature of 60^0C within 24 hours. The result was immersed with NaOH 1M within 24 hours. It was then removed from acrylic glass and washed with aquadest until it

was neutral. Finally, it was dried up in room temperature and stored in desiccators. The yielded absorbent was analyzed by FT IR, SEM and tensile strength testing. The same treatment was done with the variation of weight of additional carbon of 0.4, 0.5, and 0.6 g.

2.2.3 The Use of Chitosan and Chitosan – Activated Carbon as Adsorbent to Decrease the Concentration of Cadmium (Cd) and Nickel (Ni)

Chitosan adsorbent and chitosan-activated carbon were used to decrease the content of Cd and Ni metal in standard solution. Chitosan and chitosan-activated carbon were put into column. About 50 mL of Cd and Ni standard solution was sucked by a straw, and it was then skipped from the column with vacuum pump, and the solution was collected to be analyzed by using AAS (Atom Absorption Spectrophotometer). The same treatment was done for chitosan-activated carbon of coffee grounds with the variation of weight 0.4, 0.5, and 0.6 g.

3 RESULTS AND DISCUSSIONS

3.1 Characterization of Chitosan and Chitosan – Activated Carbon

The peaks which appeared in the FT-IR spectrum was showed in the Figure 2 and Table 1.

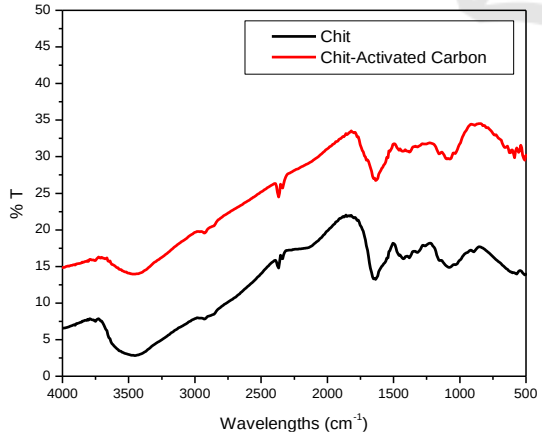

Figure 2: FT-IR Spectrum of Chitosan and Chitosan-Activated Carbon.

Based on FT-IR spectrum in chitosan, there was OH functional group in the wavelength of 3749.62 cm^{-1} and strain vibration of N-H primary amine in wavelength of 3448.72 cm^{-1} (Boggione et al., 2017). In the wavelength of 1381.03 cm^{-1} there was C-N functional group (Omidi and Kakanejadifard, 2019) . There was C-H bound in $–CH_2$ in the wavelength of 2924.09 cm^{-1}. In the wavelength of 1635.64 cm^{-1} there was vibration peaks of C=O of secondary amide group. In the wavelength of 1084.14, 1033.85 cm^{-1} there was asymmetrical vibration from C-O functional group (Paluszkiewicz et al., 2011).

The result of the analysis on FI-IR spectrum in chitosan and chitosan-active carbon of coffee grounds showed that there was the peaks of absorption in the wavelength of 3749.62 cm^{-1} which indicated the existence of OH strain vibration, in the wavelength of 3448.72 cm^{-1} which indicated the existence of N-H strain vibration, in the wavelength of 2924.09 cm^{-1} which indicated that there was C-H group of aliphatic chain, in the wavelength of 1635.64 cm^{-1} which indicated that there was C=O group from secondary amide, and in the wavelength of 1381.03, 1084.14, 1033.85 cm^{-1} which indicated that there were C-N and C-O groups.

Table 1: Functional group of chitosan and chitosan – activated carbon.

Sample	Wavenumber (cm^{-1})	Functional Groups
Chitosan	3749.62	OH
	3448.72	N-H
	2924.09	C-H
	1635.64	C=O
	1381.03	C-N
	1084.14 1033.85	C-O
Chit-Activated Carbon	386.35	OH
	3448.72	N-H
	2924.09	C-H
	1635.64	C=O
	1381.03	C-N
	10722.42 1033.85	C-O

In the spectrum of chitosan and chitosan added by active carbon adsorbent of coffee grounds, there was no difference in wavelength which indicated that physical interaction occurred between carbon and chitosan.

3.2 Characterization of Chitosan and Chitosan – Activated Carbon with SEM

Chitosan adsorbent (Figure 3) characterized by SEM was aimed to find out its morphology.

3.2.1 Morphological Analysis of Chitosan with SEM

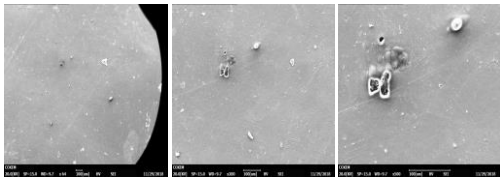

Figure 3: Image of SEM on Chitosan adsorbent surface with enlargement (A) 64x, (B) 200x and (C) 500x.

Chitosan morphological adsorbent was not fine enough and homogenous because chitosan was not distributed equally with acetate solvent. This condition caused the establishment of clump, but it could also be caused by air bubble which was trapped during the mould of adsorbent.

3.2.2 Morphological Analysis of Chitosan with SEM

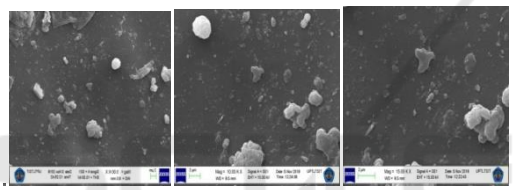

Figure 4: Image of SEM on Chitosan – activated carbon Surface with Enlargement (A) 5.000 times, (B) 10.000 times and (C) 15.000 times.

The result of chitosan morphology with the addition of active carbon could be seen in Figure 4. Rough surface and some undistributed carbon particles were caused by the process of making adsorbent was not homogenous. The addition of active carbon to chitosan adsorbent influenced the smoothness of the surface and the compatibility of the arranging materials (Lessa et al., 2018).

3.3 Characterization of Chitosan and Chitosan – Activated Carbon

Tensile strength testing in this research aims to determine the tensile strength of chitosan and chitosan - activated carbon. The result of the tensile test from the processing of chitosan and chitosan - activated carbon samples with a variation of the weight of carbon addition of 0.3, 0.4, 0.5 and 0.6 g.

Table 2: Tensile strength of chitosan and chitosan – activated carbon.

Sample	Tensile Strength (MPa)
Chitosan	2.266
Chit – C 0.3 g	3.392
Chit – C 0.4 g	3.924
Chit – C 0.5 g	2.123
Chit – C 0.6 g	1.927

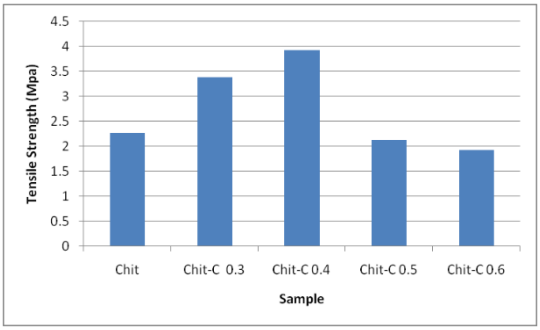

Figure 5: Tensile Strength.

As in Table 2 and Figure 5, chitosan without the addition of carbon have tensile test strength of 2.266 MPa, and chitosan with the addition of variations in the weight of activated carbon have different tensile strength tests. Chitosan with 0.3 g of carbon addition has a tensile strength of 3.392 MPa, addition 0.4 carbon is 3.924 MPa, addition 0.5 g carbon is 2.123 MPa, addition carbon 0.6 is 1.927 MPa.

Optimum tensile strength results in chitosan with the addition 0.4 g carbon. Tensile strength test results have increased with the addition of activated carbon. activated carbon from coffee ground acts as an reinforcing agent due to the non-electrostatic interactions that are bound between activated carbon and chitosan (Lessa et al., 2018). Addition activated carbon more than 0.4 g, the tensile strength has decreases. that with increasing carbon concentration in chitosan composites it will reduce the value of tensile strength.

3.4 Analysis Samples with Atomic Absorption Spectrophotometer (AAS)

In this research, before the sample analysis was carried out, we examined the sensitivity and linearity of atomic absorption spectrophotometer (AAS) instruments with equipment operating conditions such as Table 3 and Table 4 below:

Table 3: Conditions for AAS instruments Shimadzu type AA-7000 on measurement of concentration Cadmium (Cd).

Parameter	Cadmium (Cd)
Wavelength (nm)	228,8
Flame	Air – C_2H_2
Burning gas flow rate (L / min)	1,8
Air flow rate (L / min)	15,0
Gap Width (nm)	0,7
Furnace Heigth (nm)	7

Table 4: Conditions for AAS instruments Shimadzu type AA-7000 on measurement of concentration Nickel (Ni).

Parameter	Nickel (Ni)
Wavelength (nm)	232
Flame	Air – C_2H_2
Burning gas flow rate (L / min)	1,8
Air flow rate (L / min)	15,0
Gap Width (nm)	0,7
Furnace Heigth (nm)	7

Based on Tables 3 and 4 above the wavelength for the measurement of nickel and cadmium is different, the use of cathode lamps that are suitable for the metal to be analyzed. The cathode lamp will emit radiation energy that corresponds to the energy needed for the transition of atomic electrons. With giving a voltage to a certain current the metal begins to glow and the cathode metal atom will be evaporated by sprinkling. The atom will be excited then emit radiation at a certain wavelength.

3.4.1 Linearity Test

Linearity test aims to determine the correlation between the concentration of standard solutions with the response/signal from the absorbance instrument. In this research evaluation is done by making a calibration curve (concentration of standard solution versus absorbance solution) can be seen in the Table 5:

Table 5: Absorbance Linearity Test of Cadmium (Cd).

Concentration (ppm)	Absorbance
0	0,0100
0,2	0,1203
0,4	0,2366
0,6	0,3509
0,8	0,4587
1	0,6773

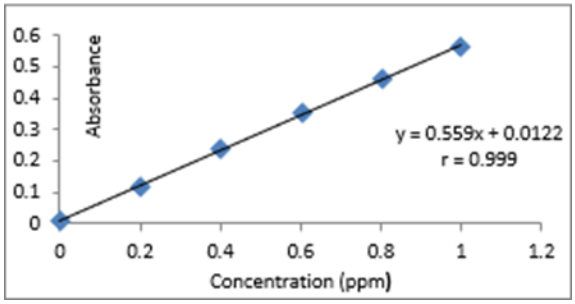

Figure 6: Calibration Curve of Standard solution Cadmium.

Based on the calibration curve above (Figure 6), the correlation coefficient (r) is 0.999, indicating that the instrument used has a good response.

Table 6: Absorbance Linearity Test of Nickel (Ni).

Concentration (ppm)	Absorbance
0	0,056
0,2	0,0234
0,4	0,0441
0,6	0,0672
0,8	0,0857
1	0.1044

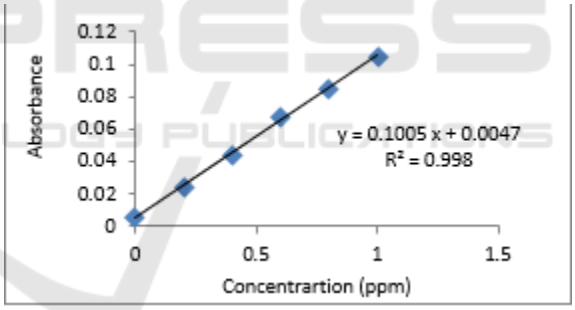

Figure 7: Calibration Curve of Standard solution Nickel.

Based on the calibration curve above (Figure 7), the correlation coefficient (r) is 0.999, indicating that the instrument used has a good response.

3.5 Analysis on Measuring Cadmium Metal

The result of measuring Cd concentration in the samples with AAS could be seen in the following Table.

Table 7: Result of Measuring Cd Concentration in the Samples with AAS.

Sample	Abs	Concentration (ppm)	% Adsorption
Chit	0,5571	0,9764	51,17 %
Chit – C 0,3 g	0,4414	0,7769	61,52 %
Chit – C 0,4 g	0,3734	0,6480	67,59 %
Chit – C 0,5 g	0,3089	0,5327	73,36 %
Chit – C 0,6 g	0,2957	0,5091	74,54 %

Based on the data in Table 7, it was found that the largest amount of Cd metal absorption was in the chitosan with the addition of 0.6 gram carbon (74.54%), while the least amount of Cd metal absorption was in the chitosan without the addition of carbon (51.1%).

The result of measuring Ni concentration in the samples with AAS could be seen in the following Table.

Table 8: Result of Measuring Ni Concentration in the Samples with AAS.

Sample	Abs	Concentration (ppm)	% Adsorption
Chit	0,0897	0,8450	57,71 %
Chit – C 0,3 g	0,0721	0,6706	66,46 %
Chit – C 0,4 g	0,0682	0,6318	68,40 %
Chit – C 0,5 g	0,0609	0,5590	72,03 %
Chit – C 0,6 g	0,0581	0,5313	73,43 %

Based on the data in Table 8, it was found that the largest amount of absorption of Ni metal was in the chitosan adsorbent with the addition of 0.6 gram of active carbon (57.71%). The amount of active carbon added to chitosan adsorbent (73.43%) while the least amount of chitosan adsorbent without the addition of active carbon was 57.71%. The amount of active carbon added to chitosan adsorbent influenced the increase in percentage (%) of metal absorption.

Chitosan has active amine and hydroxyl group and the capacity to stick on some types of metal. It can be used as adsorbent of heavy metal such as Zn, Cd, Cu, Pb, Mg, and Fe. Chitosan active site, either in the form of NH_2 or in the protonated NH_3condition, is able to adsorb heavy metal through the mechanism of establishing chelate or ion exchanging. Chitosan have good complexing ability, -NH_2 groups on chitosan interactions with metals (Obregón-Valencia and Sun-Kou, 2014).

Amine and hydroxyl group of chitosan was able to bind metal through some mechanism, including chemical interaction (like the establishment of chelate) and electrostatic interaction (like ion exchanging or the establishment of ion pair). In the result of their research, it was reported that chitosan which has been coated with active carbon increased its percentage (%) of absorptive power on cadmium metal (Hydari et al., 2012).

Activated carbon is commonly used as adsorbent to absorb metal because it has high capacity to absorb and has good endurance against abrasion. Active carbon has porous structure and wide surface. (Obregón-Valencia and Sun-Kou, 2014) reported that active carbon was able to absorb cadmium metal because it had high chemical reactivity. The use of commercial active carbon is limited since its price is relatively high. Active carbon is very effective adsorbent in absorbing metal in waste water because of its large number of micropores and mesopores, its wide surface, and its big pores, and the functional group on its surface interacts with heavy metal ion (Hernández Rodiguez et al., 2018). Carbon has the capacity to absorb metal because it has large pores. The more the in its pores so that metal content in chitosan decrease by adding more carbon.

4 CONCLUSIONS

Chitosan and chitosan-active carbon can be used as adsorbent to decrease Cd and Ni metal content. The best decrease in Cd metal content is found in chitosan by adding 0.6 g carbon (75.45%) and the best decrease in Ni metal content is found in chitosan by adding 0.6 g carbon (73.43%).

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