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Potential of Corn Stalk Biomass as Biosorbent in Adsorption of Mercury (Hg) Metal Contaminants

Muhammad Fathurrahman*, Sutanto, Yobi Sukresna
Program Studi Kimia, FMIPA, Universitas Pakuan, Bogor
*E-mail: fathur110590@unpak.ac.id

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Abstract

Corn stalks are an agricultural waste that is commonly found in Indonesia. Corn stalks have a high enough cellulose content which has functional groups that can be used as biosorbents. This study aims to optimize the adsorption of corn stalk waste to mercury metal ions and then determine the adsorption capacity and adsorption constant of the Langmuir and Freundlich isotherm model. This research begins with corn stalk preparation, delignification, characterization using FTIR and SEM, determination of optimization of biosorbent weight, pH of Hg solution, contact time, and concentration of Hg solution, then applied to mercury metal contamination waste. Optimization of the weight of biosorbent in the range 0.4-1.2 grams, pH of the Hg solution is 2-4, contact time is 60-120 minutes, and concentration of the Hg solution of 30-100 g/L with a solution volume of 50 mL. Furthermore, determination of mercury levels, manufacture of standard series, and testing using ICP-OES. Followed by the determination of Langmuir and Freundlich's adsorption isothermal model. The results of this study indicate that the optimum conditions for the adsorption process occur at the weight of the biosorbent 1.2 grams, pH of the Hg 3 solution, contact time of 90 minutes, and concentration of Hg solution of 40 ppb with a solution volume of 50 mL. The application of biosorbent to metal contamination waste with adsorbed Hg concentration obtained 27.5772 ppb in 50 mL of waste solution with an adsorption efficiency of 88.44%. In the determination of Langmuir and Freundlich isotherms, a match was found for Langmuir adsorption isothermals with an adsorption capacity of 0.00333 mg Hg/g biosorbent and β = 176.8256.

Keywords: *Corn stalk waste, Biosorbent, Adsorption, Optimization, Langmuir and Freundlich Isotherm*

1 Introduction

In addition to the positive impact of progress in the industrial sector, which is increasingly rapidly nowadays, it has also had a negative impact, including the discharge of liquid, solid, and gaseous waste that can cause pollution to the environment. The industrial waste disposal causes an increase in the number of pollutants and heavy metal toxicity in the environment. Heavy metal pollution that can cause environmental damage include lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As), nickel (Ni), and chromium (Cr). These heavy metals can cause poisoning if absorbed by the body in large quantities [1].

Various methods aimed at eliminating levels of heavy metal contamination in wastewater have been developed, such as electrophoresis methods, membrane separation and ion exchange, but these methods are less effective and require large costs. Various types of pollutants which are organic compounds and inorganic compounds can be absorbed by the adsorbent through the mechanisms of adsorption, ion exchange, filtration, and precipitation. In minimizing the cost of waste treatment, the use of agricultural by-products such as rice husk waste, coconut dregs, coconut fibers, and other agricultural wastes can be done [2]. One of the wastes that has potential as a biosorbent is corn stalks.

Corn stalks are a by-product of the corn plant which are abundant in nature but are generally only used as animal feed and are often left or not used as other products that have more benefits. Corn stalks have the potential to be used as an

adsorbent. This is because corn stalks contain about 45% cellulose, 25% hemicellulose, 15% lignin, and 15% other components [3]. This high content of carbon compounds allows corn stalks to be used as biosorbents. In addition, the reason corn stalks are used as an adsorbent is that it is easy to obtain, cheap, environmentally friendly, and can be decomposed in nature [4].

Corn stalks have been used as an adsorbent to reduce chlorine levels in treated water with a chlorine absorption result of 96.08% with a contact time of 90 minutes and an adsorbent weight of 300 grams [5]. Then in reducing mercury heavy metal contamination, research has been carried out using activated carbon from coconut shells in gold processing waste located in Buru Regency, Maluku Province with a mercury adsorption capacity value of 0.1235 mg per gram of adsorbent and succeeded in reducing mercury levels in waste by the efficiency of 99.4% [6].

Based on these references, it is necessary to conduct research to determine the potential of sweet corn stalk waste as a biosorbent that can be used to overcome environmental damage caused by mercury-heavy metal contamination in waste.

2 Method

2.1 Tools and materials

The material used for the manufacture of corn stalk biosorbent is corn stalk waste (without roots) from corn plants in Sukaraja District, Bogor Regency, West Java. The materials used to experiment with the potential of sweet corn stalk biomass as a biosorbent in the adsorption of metallic mercury (Hg) contaminants were HNO_3, HCl, aquabides, mercury standard solvent (HNO_3 5%) $NaBH_4$, NaOH, Hg standard 1000 mg/L, As standard 1000 mg /L, liquid mercury waste.

Then the tools used are a blender, analytical balance, measuring flask, volumetric pipette, glass cup, micropipette, hot plate magnetic stirrer, pH meter, FTIR, Inductively Coupled Plasma (ICP) 700 series ICP-OES with charge-coupled device (CCD) detector. and the torch used is a one-piece torch, axial 2.4 mm id injector, Anton Paar Multiwave GO Digester, 70 mesh sieve, gray filter paper, and glassware.

2.2 Corn Stalk Biosorbent Preparation

After drying, the corn stalks are cut into small pieces and then mashed with a blender. These results were then filtered using a 70-mesh sieve. 150 grams of fine corn stalk powder was taken, then soaked in 3% NaOH solution until completely submerged and stirred at 150 rpm with a contact time of 60 minutes while heated at 80 C. After that, it was allowed to stand for 60 minutes then filtered and washed with distilled water until neutral. Furthermore, the residue obtained was dried in an oven at 70 C until dry so that the corn stalk powder biosorbent was ready for use [7].

2.3 Optimization of Hg Adsorption Process by Corn Stalk Biosorbent

2.3.1 Preparation of Mercury Standard Solution

Mercury standard with a concentration of 1000 mg/L. 0.50 mL of a 1000 mg/L mercury solution was pipetted into a 100 mL volumetric flask and then dissolved with mercury standard solvent to produce a standard stock solution of mercury with a concentration of 5 mg/L. A standard stock solution of mercury 5 mg/L pipette 0; 0.10; 0.20; 0.30; 0.40; 0.60; 0.80; 1.20; 1.60 and 2.00 mL, then each was put into a 100 mL volumetric flask, added 5 mL of 1:1 HCl and 1 mL of 1:1 HNO_3 then adjusted with mercury standard solvent to produce a mercury standard solution with a concentration of 0; 0.005; 0.010; 0.015; 0.020; 0.030; 0.040 0.060; 0.080 and 0.100 mg/L. The intensity of the standard solution was measured using ICP-OES at a wavelength of 253,650. The absorbance data obtained were used to create a standard curve.

2.3.2 Determination of Optimum Corn Stalk Biomass Weight

Mercury solution with a concentration of 15 ppb was added as much as 50 mL into four different 250 mL beakers. Then the corn stalk biomass was put into a glass cup with a weight of 0.4; 0.8; 1.2 and 1.6 grams. Stirred using a stirrer at 150 rpm for 60 minutes. Then the solution was filtered, and the intensity was measured using ICP-OES together with a standard solution at a wavelength of 253.650 nm.

2.3.3 Determination of Optimum pH

Mercury solution with a concentration of 15 ppb as much as 50 mL was put into 3 250 mL beakers each. In each beaker the pH of the solution was adjusted at pH 2, 3, and 4 by adding 0.1 N HNO_3 solution or 0.1 N NaOH into a glass cup containing a mercury solution, each weight of the optimum corn stalk biomass was added. Stirred with a stirrer at 150 rpm for 15 minutes. Then the solution was filtered, and the intensity was measured using ICP-OES together with a standard solution at a wavelength of 253.650 nm.

2.3.4 Determination of Optimum Contact Time

Mercury solution with a concentration of 15 ppb as much as 50 mL was put into three 250 mL beakers each. Adjusted the pH according to the results of determining the optimum pH. Then added each corn stalk biomass weight according to the results of determining the optimum corn stalk biomass weight. Stirred at a speed of 150 rpm for 60; 90; and 120 minutes using a stirrer. Then the solution was filtered, and the intensity was measured using ICP-OES together with a standard solution at a wavelength of 253.650 nm.

2.3.5 Determination of Optimum Mercury Concentration

50 mL of mercury solution was put into 5 pieces of 250 mL glass beakers with a concentration of 0.030; 0.040 0.060; 0.080 and 0.100 mg/L. Then put in corn stalk biomass with optimum weight and adjust the pH of the solution according to the results of determining the optimum pH. Stirred using a stirrer at a speed of 150 rpm during the optimum contact time. Then the solution was filtered, and the intensity was measured using ICP-OES together with a standard solution at a wavelength of 253.650 nm. The absorbance data obtained were used to create a curve in determining the optimum concentration of biomass absorption.

2.4 Determination of Sorption Characteristics

FTIR characterization was carried out by taking 10 mg of corn stalk biosorbent powder then mixed with 100 mg KBr until homogeneous and molded into pellets and then analyzed using FTIR. Samples that will be characterized by FTIR in this study include corn stalk biosorbent powder before being used for waste adsorption.

2.5 Analysis of the Initial Concentration of Mercury in Metal-Polluted Waste

The metal contamination waste solution that has been homogenized is pipetted 50 mL into a 100 mL glass beaker. Added 2.5 mL of concentrated HNO_3. Heated on an electric heater until the solution was almost dry. Added 25 mL of distilled water, put into a 250 mL volumetric flask, and matched with distilled water and then homogenized. The intensity was measured using ICP-OES together with a standard solution at a wavelength of 253.650 nm.

2.6 Analysis of Mercury Adsorption by Corn Stalk Biosorbent on Metal Contamination Waste

A 100 mL waste sample was put into a 250 mL beaker. Adjusted the pH at the optimum pH by adding 0.1 N HNO_3 or 0.1 N NaOH. The optimum weight of corn stalk biomass was entered. Then stirred with a stirrer at a speed of 150 rpm for the optimum contact time obtained from the determination of the optimum contact time. Then the solution was filtered, and 50 ml of the filtrate was pipetted into a 100 ml glass beaker. Add 2.5 mL of concentrated HNO_3. Heated on an electric heater until the solution was almost dry. Added 25 mL of distilled water, put into a 250 mL volumetric flask, matched with distilled water, and then homogenized. The intensity was measured using ICP-OES together with a standard solution at a wavelength of 253.650 nm.

2.7 Data processing

Determination of the optimization of mercury adsorption by sweet corn stalk biomass in this study was obtained by making a graph of the relationship between the concentration of mercury adsorbed by corn stalk biomass with each optimization treatment carried out.

The maximum adsorption capacity of sweet corn stalk biomass on metallic mercury in a waste can be obtained by making a graph of the determination of the adsorption isotherm of the Langmuir and Freundlich model.

3 Result and Discussion

3.1 Sweet Corn Stalk Biosorbent

Sweet corn stalk biosorbent produced from the preparation of local corn stalk waste has a brownish-white color, and a fine texture with a size of 70 mesh so that it is easy to apply to polluted waste. The yield of corn stalk biosorbent produced from 300 grams of corn stalk waste was 140.20 grams or 46.73%. Figure 1 shows sweet corn stalk biosorbent.

Figure 1. Sweet Corn Stalk Biosorbent

The main components of sweet corn stalks are cellulose, hemicellulose, and lignin, where the functional groups contained therein are hydroxyl

and carboxyl groups. Hydroxyl and carboxyl groups can play a role in adsorption of metal ions [8]. Delignification is a separation process between cellulose and other compounds such as lignin. The purpose of lignin separation is to increase the surface area of the biosorbent so that the adsorption capacity of the biosorbent increases [9]. Lignin binds to cellulose esterically to form lignocellulose complexes. Lignocellulosic complexes have weak ester bonds when subjected to alkaline treatment. Alkaline solutions such as NaOH, KOH or NH_4OH are often used in the delignification process. The alkaline solution used in the delignification process in this study is NaOH where the -OH ion from the solution will attack the ester bond and break it [10].

3.2 Corn Stalk Characterization Using Fourier Transform Infrared Spectroscopy (FTIR)

Characterization by FTIR test aims to identify the functional groups contained in sweet corn stalk biosorbent which is a qualitative test in this study. The results obtained from this analysis are in the form of an IR spectrum that looks like in Figure 2.

Based on the spectrum in Figure 2, in the sweet corn stalk biosorbent, there is a wave number of 3367.71 cm^{-1} which indicates the presence of O-H groups. The absorption peak at wave number 2899.01 cm^{-1} indicates the presence of a C-H group. Then there is a wave number of 1722.43 cm^{-1} which is a C=O vibration. Absorption also appears at wave number 1161.15 cm^{-1} indicating the presence of C-O groups [4].

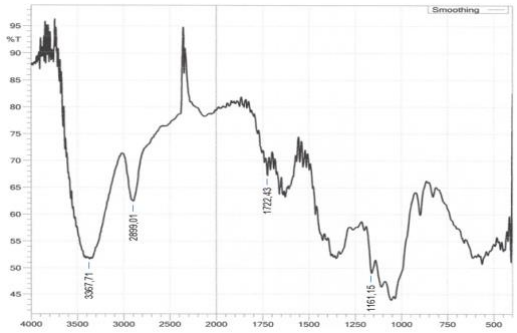

Figure 2. FTIR Spectrum of Sweet Corn Stalk Biosorbent

In the metal adsorption process by sweet corn stalk biosorbent, the functional group that can play an important role is the hydroxyl group (-OH) which is thought to come from alcohol and carboxyl compounds (C=O) derived from carboxylic acid compounds. Several mechanisms that can occur in the process of adsorption of Hg^{2+} ions on sweet corn stalk biosorbents include entrapment and ion exchange. The ion exchange mechanism occurs because Hg^{2+} ions replace H^+ ions present in the biosorbent. Ion exchange occurs due to the electrostatic force between the cation and the negatively charged functional group. In addition, the hydroxyl and carboxyl functional groups in the biosorbent become negatively charged due to deprotonization, so that the functional groups will be reactive in absorbing Hg^{2+} ions. The interaction between Hg^{2+} ions and biosorbent occurs due to the electrostatic force between the negative charge of the biosorbent which acts as an active site and the positive charge of metal ions [8].

3.3 Corn Stalk Characterization Using Scanning Electron Microscopy (SEM)

Testing of biosorbent characterization using Scanning Electron Microscopy (SEM) at a magnification of 3000 times can be seen in Figure 3. The results of morphological characterization of sweet corn stalks using SEM showed that the surface of the biosorbent had pores with pore sizes of 5.321 nm and 7.642 nm. Biosorbents generally have a pore diameter of at least 2 nm, so the results obtained prove that sweet corn stalk biosorbents have the appropriate criteria [11]. Based on these results, it can be stated that the larger the pore size in the biosorbent, the faster or more absorbing pollutants will be [12].

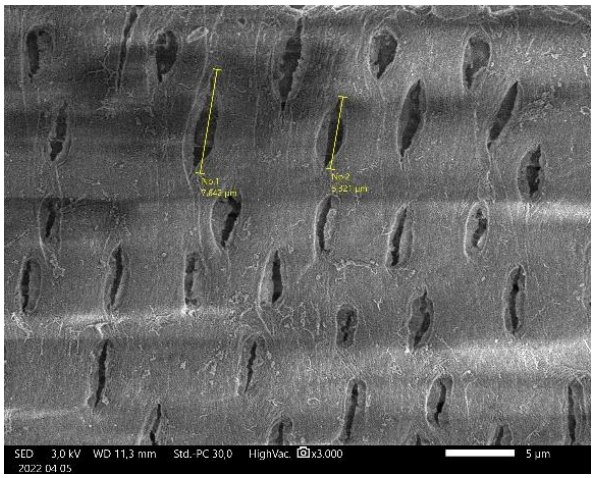

Figure 3. SEM Image of Sweet Corn Stalk Biosorbent

3.4 Optimum Weight of Mercury Adsorption Biosorbent by Corn Stalk Biosorbent

The results of further research, the optimum weight of corn stalk biomass was determined to determine specifically the weight of corn stalks needed to absorb mercury metal. The weight of the

biosorbent used is 0.4; 0.8; 1,2; and 1.6 grams with a fixed concentration of metallic mercury, which is 15 ppb. The experimental results can be seen in Figure 4.

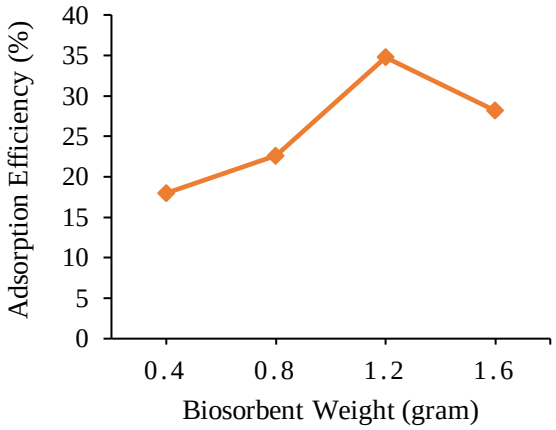

Figure 4. The curve of Determination of Optimum Weight of Mercury Adsorption Biosorbent by Sweet Corn Stalk Biosorbent

The optimum weight was found at 1.2 grams with an adsorption efficiency for mercury metal of 34.75%, the adsorbed concentration was 5.4085 ppb because at that concentration there was a large increase in metal adsorption efficiency compared to the previous efficiency and after that, there was a decrease in efficiency. the adsorbed metal.

The increase in the mass of the biosorbent will increase the surface area. The adsorption process depends on the number of interactions that occur between the adsorbent and the adsorbate. The effective interaction between adsorbent and adsorbate will increase with increasing surface area. However, at a mass of 1.6 grams, the biosorbent no longer experienced an increase in adsorption efficiency, possibly this was because the biosorbent had been in equilibrium at a mass of 1.2 grams. In addition, the stirring speed carried out in this study (150 rpm) may not be able to interact optimally with the adsorbent and adsorbate [13].

3.5 Optimum pH of Mercury Adsorption by Corn Stalk Biosorbent

The volume of the solution used in determining the optimum contact time was 50 mL at a concentration of 15 ppb with a weight of 1.2 grams of corn stalk biomass. The results obtained from determining the optimum adsorption pH can be seen in Figure 5.

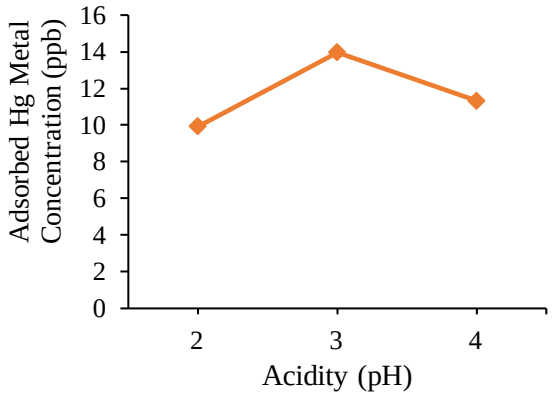

Figure 5. The Curve of Optimum pH Determination of Mercury Adsorption by Sweet Corn Stalk Biosorbent

Based on Figure 5, mercury metal can be adsorbed optimally at pH 3 where the adsorbed mercury is 13.9592 ppb with an adsorption efficiency of 79.22%. At pH 2 the amount of mercury adsorbed is quite low, this is because at pH 2 the surface of the adsorbent is surrounded by H^+ ions, resulting in competition between metal ions Hg^{2+} and H^+ ions in binding to the active groups of the biosorbent. Meanwhile, at pH above 3, there was a slight decrease in the concentration of adsorbed metal ions. This is because, at higher pH conditions, most of the Hg^{2+} is in the form of $HgOH^+$ and $Hg(OH)_2$. The presence of OH^- ligands can cause a decrease in the interaction between Hg^{2+} and the active site of the biosorbent [14].

The pH value has an important role in the adsorption of metal ions that can affect the performance of functional groups owned by biosorbents that play an active role in the absorption of heavy metals [15]. Therefore, the pH of the solution is a factor that is quite influential on the adsorption of mercury metal by corn stalk biosorbent.

3.6 Optimum Contact Time of Mercury Adsorption by Corn Stalk Biosorbent

The optimum contact time is the time corn stalk biomass requires to produce the highest concentration of adsorbed mercury. It is necessary to determine the adsorption equilibrium time to obtain the optimum adsorption of Hg^{2+} ions on the surface of the biosorbent. The longer the contact time in the adsorption process, the higher the value of the adsorbate that will be adsorbed. Therefore, the contact time between adsorption has an important role in an adsorption process.

The volume of the solution used to determine the optimum contact time was 50 mL at a concentration of 15 ppb with a pH of 3 and a corn stalk biomass weight of 1.2 grams. The results of determining the optimum contact time for mercury adsorption by corn stalk biomass can be seen in the curve of the relationship between the concentration of adsorbed mercury and the contact time in Figure 6.

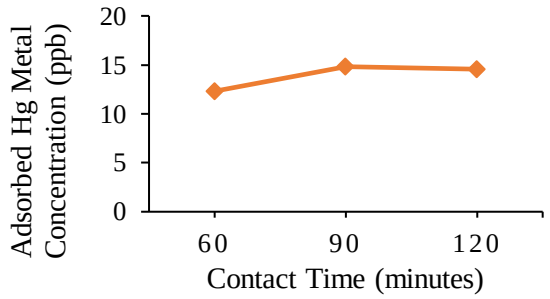

Figure 6. Results Curve of Optimum Contact Time for Mercury Adsorption by Sweet Corn Stalk Biosorbent

Figure 6 shows that the longer the contact time, the more mercury concentration is adsorbed. The optimum contact time of mercury metal sorption with corn stalk biosorbent occurred at 90 minutes, the adsorbed mercury concentration was 14.8141 ppb with an adsorption efficiency of 89.45%. At the contact time of 120 minutes the adsorbed mercury concentration no longer increased, this was because the adsorption process had reached its saturation condition. In addition, the release of adsorbate can also occur because the contact time and stirring that are too long can cause mercury to be desorbed [13].

3.7 Optimum Concentration of Mercury Adsorption by Corn Stalk Biosorbent

The number of active sites on the surface of the biosorbent will affect the amount of absorption of the absorbed Hg^{2+} ion concentration, if the number of active sites on the biosorbent is greater than the number of metal ions to be absorbed, the higher the absorption efficiency will be. However, the absorption efficiency may run constant because the biosorbent has been saturated under certain conditions.

The test was carried out as many as 5 points of mercury concentration were used with the optimum weight of corn stalk biosorbent was 1.2 grams. Concentration starts from the smallest to the largest concentration with the aim of clearly showing the increase and decrease in absorption.

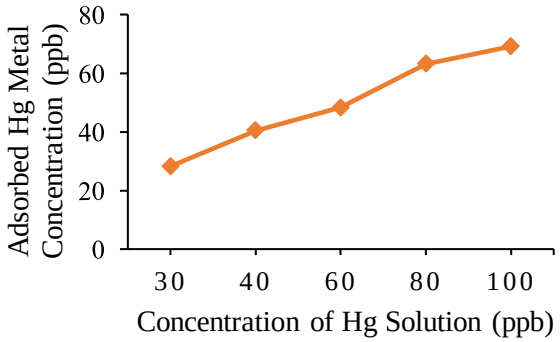

Figure 7. Curve of Determination of Optimum Concentration of Mercury Adsorption by Sweet Corn Stalk Biosorbent

Figure 7 shows the effect of concentration variations on the number of Hg^{2+} ions adsorbed by sweet corn stalk biosorbent, whereas the concentration of Hg^{2+} metal ions increase, the absorption of mercury concentration increases or in other words, the increase in Hg^{2+} ion concentration causes more Hg to interact with the surface. adsorbent and adsorbed where the adsorption value of the largest Hg concentration was 69.1404 ppb. This is because the adsorption ability of sweet corn stalks to mercury ions has not yet reached its saturation point [8].

3.8 Application of Corn Stalk Biosorbent on Metal Contaminant Waste

3.8.1 Initial Concentration of Mercury in Metal Polluting Waste

The liquid waste used comes from PT. X with yellowish orange color. The waste comes from the liquid analysis results. Analysis of the initial concentration of mercury in the waste needs to be carried out to determine the level of mercury adsorbed in the corn stalk biosorbent because the ICP measured is the concentration of mercury remaining in the waste after the adsorption process. The results of the analysis of the initial concentration of mercury in the waste can be seen in Table 1.

Table 1. Results of Analysis of Preliminary Metal Mercury Concentrations in Waste

No.	Parameter	Observation
1	Mercury Level	31.3312 ppb
2	pH	3
3	Appearance	liquid
4	Color	Yellowish orange

3.8.2 Adsorption of Metal Polluted Waste by Corn Stalk Biosorbent

In the results of this study, it is known that the initial mercury concentration in the waste is 31.3312 ppb. The waste used is 50 ml. Then adsorption with corn stalk biosorbent as much as 1.2 grams was carried out with the optimum treatment that was obtained during the initial optimization. The results after adsorption can be seen in Figure 8.

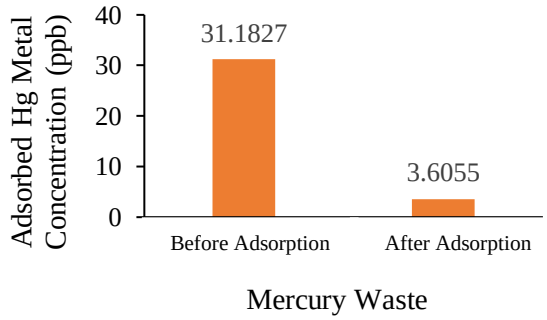

Figure 8. Results of Adsorption of Mercury Metal Waste by Sweet Corn Stalk Biosorbent

Based on Figure 8, it was found that there was a decrease in the concentration of mercury in the waste after adsorption by corn stalk biosorbent. The adsorbed concentration for mercury metal was 27.5772 ppb with an adsorption efficiency of 88.44%.

There was a slight decrease in adsorption efficiency when compared to the optimum adsorption conditions, from 89.45% to 88.44%. This is probably because the waste samples contained metals other than mercury with different concentrations. These other metals can hinder the absorption of mercury metal biosorbent because it can interact with the biosorbent which can cause the absorption of mercury metal to be less than optimal.

3.8.3 Adsorption Isotherm

Adsorption isotherm testing was conducted to determine the appropriate equilibrium model used in a study. Determination of the equilibrium model that is generally used is the Langmuir isotherm and the Freundlich isotherm. Langmuir isotherm assumes that adsorption that occurs on the surface of the adsorbent forms a monolayer, where each adsorption position is equivalent and the presence of particles in adjacent positions does not affect the ability of the adsorbent to bind particles and the interaction between the adsorbate molecules and the adsorbent surface occurs by chemisorption. However, the Freundlich isotherm assumes the opposite where the adsorption that occurs on the surface of the adsorbent forms a multilayer and each position has a different type of adsorption, and the maximum adsorption capacity cannot be determined. In addition, the interaction between the adsorbate molecule and the surface of the adsorbent that occurs is by physisorption [16].

The adsorption isotherm equation of sweet corn stalk biosorbent was made based on the calculation of the adsorption ability at various concentrations. Furthermore, the suitability of two linear regressions is determined, namely the linear regression of the Langmuir isotherm model and the Freundlich isotherm. The match is determined based on the higher linearity, or the regression value (R^2) is close to 1.

Determination of the equilibrium model depends on the value of R^2 with a high value. The results obtained indicate that the Langmuir isotherm is more suitable for the sweet corn stalk biosorbent adsorption isotherm pattern seen from the higher R^2 value of 0.9902 [17]. Based on the equation of the Langmuir isotherm model, namely y = 300.02x + 1.6967, the value of which represents the adsorption capacity of 0.00333 mg Hg/g biosorbent is obtained and the value of which represents the equilibrium constant is 176.8256.

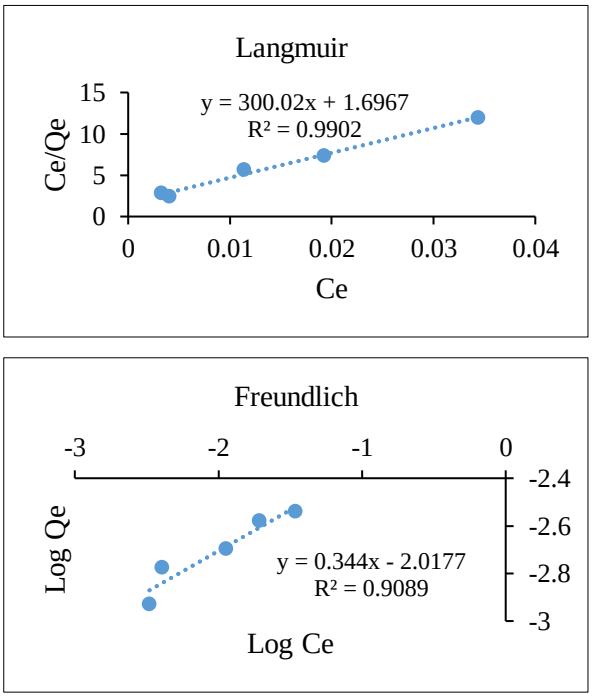

Figure 12. Langmuir and Freundlich Isotherms

Table 2. Langmuir Isotherm of Sweet Corn Stalk Biosorbent

Metal	α	β	R^2
Mercury	0,00333	176,8256	0,9902

4 Conclusion

Corn stalks can be used as biosorbents to adsorb mercury heavy metal contamination. The results of characterization using FTIR showed -OH and C-O functional groups that could play a role in the adsorption process. SEM characterization showed the presence of pores on the surface of the biosorbent with pore sizes between 5.321 m and 7.642 m.

The higher the concentration of metal ions, the greater the absorption of mercury concentration. The optimum conditions for the adsorption process occurred at the biosorbent weight of 1.2 grams, the pH of the Hg 3 solution, the contact time of 90 minutes, and the concentration of the Hg solution of 40 ppb with a solution volume of 50 mL.

Sweet corn stalk biosorbent can absorb mercury metal contamination waste with an adsorbed mercury concentration of 27.5772 ppb with an adsorption efficiency of 88.44%.

It was found that there was a match with the Langmuir isotherm with a value of or adsorption capacity of 0.00333 mg Hg/g biosorbent and 176.8256.

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