Adsorptive Removal of Heavy Metals in Wastewater using Soy-Pulp Derived Activated Carbon Synthesized via Various Pyrolysis Techniques

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Adsorptive Removal of Heavy Metals in Wastewater using Soy-Pulp Derived Activated Carbon Synthesized via Various Pyrolysis Techniques

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Abstract: *Effective management of wastewater from process industries and laboratories is increasingly critical due to the environmental and health risks associated with heavy metal contamination. This study explores the use of soy pulp, an agricultural by-product of the soy processing industry, as a sustainable precursor for activated carbon production. Soy-pulp derived activated carbon was synthesized via slow, fast, and conventional pyrolysis techniques and assessed for its efficiency in adsorbing cadmium (Cd), copper (Cu), iron (Fe), and lead (Pb) from synthetic wastewater. Adsorption efficiencies across all treatments ranged from 98.00% to 99.81%, with the highest removal recorded for Cu, followed by Cd, Fe, and Pb. Activated carbon synthesized through slow pyrolysis at 350 °C for 4 hours achieved the highest removal rates for Cd (99.65%), Cu (99.81%), and Fe (99.38%), and showed strong performance for Pb (98.83%). The consistent trend in metal removal (Cu > Cd > Fe > Pb) across all pyrolysis methods indicates a dominant affinity for Cu (II) and comparatively lower affinity for Pb (II). These findings demonstrate the potential of soy pulp-derived activated carbon as an effective and sustainable adsorbent for heavy metal removal from wastewater.*

Keywords: Soy pulp; Heavy metal removal; Wastewater; Pyrolysis; Adsorption efficiency.

1. INTRODUCTION

The effective management and treatment of process industries' wastewater have become crucial in society, driven by the escalating concerns over environmental pollution and the potential health hazards caused by heavy metal contaminants. Laboratories engaged in scientific research and industrial processes routinely generate wastewater containing heavy metals such as lead, cadmium, chromium, iron, manganese, nickel, and copper, which are known for their toxic effects on both aquatic ecosystems and human health when discharged into the ecosystem.

Heavy metals refer to any metal and metalloid element that has a relatively high density ranging from 3.5 to 7 g/ml, and is toxic or poisonous at low concentrations. These metals are found widely in the Earth's crust and are non-biodegradable in nature. They enter the human body through air, water, and food [1].

Specific heavy metals that are particularly toxic, such as Arsenic (As), Cadmium (Cd), Lead (Pb), Mercury (Hg), Chromium (Cr), and Nickel (Ni). These metals were categorized based on their carcinogenic properties, highlighting that they are classified as category 1 heavy metals by the International Agency for Research on Cancer (IARC). The accumulation of metals like Arsenic (As), Cadmium (Cd), Lead (Pb), and Mercury (Hg) can lead to chronic diseases, highlighting the urgent need for awareness and preventive measures against exposure to these toxic elements [2]. The persistence of heavy metals in the environment indicates that they do not easily break down or metabolize into less harmful substances.

Natural materials are considered low-cost adsorbents. They are environment-friendly and available in large quantities. The natural polymers like polysaccharides are biodegradable, renewable, and abundantly available and have the natural ability to associate physically or chemically with a large variety of molecules. Hence, polysaccharides are considered excellent low-cost adsorbents for water decontamination [3].

Numerous agricultural wastes like walnut shells, Turkish coffee, waste tea, black gram, neem bark, rice husk, etc., are potent adsorbents of heavy metals. The effectiveness of adsorbents derived from durian husk, banana peel, and mangosteen shell in reducing Nickel concentration in wastewater collected from a mining industry revealed a decrease in nickel concentration, exhibiting the highest reduction efficiency using durian shells [4].

Soy pulp, a low-cost and novel cellulosic biomass which can be used effectively for textile effluent treatment. It shows effective removal of many dyes, besides, it has also been found to have an excellent adsorptive removal capacity even in extreme acidic conditions. It can be used as a low-cost natural adsorbent for the adsorptive removal of dyes from textile effluent. Soy pulp can be a prominent entity in wastewater treatment if used wisely as a natural adsorbent or activated carbon precursor [5].

The microscopic structure of soy pulp is hollow, rod-shaped fibers with a high surface area, which is a necessary feature for an adsorbent. Due to variety of components present in soy pulp, it also contains variety of functional groups on its surface, such as CHO, CO, COOH, which are further responsible for the attachment of different contaminants on the bases of their surface charge. It shows very good results in removing azo dyes such as methylene blue, methyl orange, eriochrome black-T, reactive brilliant blue, and rhodamine-B dyes from textile industry-based effluent [6].

Activated carbon is one of the most proficient and key adsorbents, which has been examined with excellent accomplishment for the treatment of wastewaters for both small laboratory scales as well as industrial applications. The activated carbon, as it is known over the years, has significant properties, which include high internal surface area with a greatly advanced internal pore structure and the availability of broad surface

functional groups [7].
Pyrolysis is a thermochemical process that takes place in the absence of oxygen and at relatively high temperatures. It is commonly used to convert organic materials into bio-oil, bio-char, and syngas. Biomass pyrolysis is usually conducted at or above 500 °C, providing enough heat to deconstruct the strong biopolymers mentioned above. Because no oxygen is present, combustion does not occur rather, the biomass thermally decomposes into combustible gases and bio-char. Most of these combustible gases can be condensed into a combustible liquid, called pyrolysis oil (bio-oil), though there are some permanent gases (CO_2, CO, H_2, light hydrocarbons), some of which can be combusted to provide the heat for the process. Thus, pyrolysis of biomass produces three products: one liquid, bio-oil, one solid, bio-char, and one gaseous, syngas [8].
Depending on the temperature, three different types of pyrolysis can be distinguished: Slow pyrolysis, which is characterized by low heating rates, relatively low temperatures (300–400 ºC), and long residence times. This pyrolysis technique is mostly used to produce high quantities of biochar (yields of about 30%). Fast pyrolysis is characterized by high thermal gradients (100 ºC/s), high temperatures (400–700 ºC), and very short residence times (seconds). This pyrolysis technique is used to obtain high bio-oil yields (over 50%). Conventional pyrolysis, characterized by moderate heating rates (20 ºC/s) and equally moderate reaction temperatures (less than 600 ºC), with residence times ranging from 10 s to 10 min. This type of pyrolysis gives rise to comparable quantities of char, gas, and liquid. Biochar's main use is as a soil amendment; however, it has also been used as an alternative adsorbent to activated carbon since it better complies with the aims of the circular economy [8].
Heavy metals such as cadmium, copper, iron, and lead pose significant health risks and environmental hazards due to their toxicity and non-biodegradable nature. Heavy metals in wastewater come from a variety of sources, including industrial processes such as textile, paint, pesticide, battery manufacturing, metal, and mining. Industrial and Chemical laboratories discharge high concentrations of heavy metals from processes, analysis, and experiments. Addressing these diverse sources is essential for effective wastewater treatment and environmental protection. Traditional removal methods have limitations, making adsorption a preferred alternative due to its efficiency and simplicity. Soy pulp, a nutrient-rich by-product of soy processing, has shown high potential for adsorption due to its porous structure and functional groups. Utilizing soy pulp for this purpose not only mitigates heavy metal contamination but also valorizes agricultural waste, promoting environmental sustainability and economic efficiency.
This research endeavors to bridge this knowledge gap by investigating the adsorptive removal of heavy metals in wastewater using activated carbon produced from soy pulp. Through a systematic exploration of the adsorption efficiency and mechanisms of soy pulp-derived activated carbon, this study aims to provide valuable insights into the feasibility and effectiveness of this sustainable and cost-efficient adsorbent for heavy metal removal.

2. EXPERIMENTAL METHODS

The flowchart in Figure 1 illustrates the flow of necessary steps conducted throughout the duration of experiment.

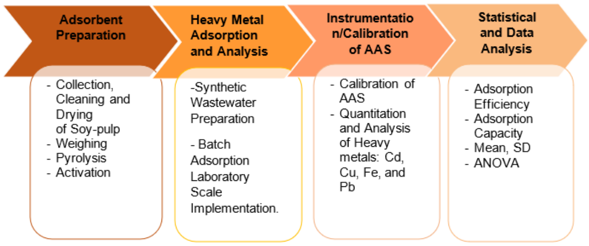


Fig. 1. A schematic diagram of the flow of processes of the experiment.

2.1 Soy-pulp Preparation

The soy pulp was collected from the squeezed-up soy milk and tofu from a local soy products manufacturer. It was then washed with distilled water and squeezed to remove excess water. The pulp was sun-dried and grinded to form into powder with particles of equal size. It was then oven-dried at 105^{O}C for 3 hours. The dried soy pulp powder was removed and cooled in a desiccator containing $CaCl_2$-drying agent for 30 minutes. The dried soy pulp powder was weighed and divided into three (3) equal parts, weighing 100 g. Each soy pulp powder sample was subjected to different pyrolysis treatments for carbonization.

2.2 Pyrolysis

The soy pulp powder was put in a crucible and pyrolyzed using a furnace via various pyrolysis techniques to form biochar. In Slow Pyrolysis Treatment, 100 g of dried soy pulp powder was carbonized by implementing the slow pyrolysis technique at 350 ºC for 4 hours. Another 100 g of dried soy pulp powder was carbonized by the fast pyrolysis technique at 700 ºC for 5 minutes for Fast Pyrolysis Treatment, and another 100 g of dried soy pulp powder was carbonized by the conventional pyrolysis technique at 400 ºC for 10 minutes for Conventional Pyrolysis Treatment.

2.3 Activation

The biochar obtained from each treatment was ground to form fine powder. Each sample was placed in a beaker and subjected to chemical activation using a 30% HNO_3 solution. The content of the beakers was thoroughly mixed until a paste of each was formed. The pastes from each sample were then transferred to crucibles and placed in a muffle furnace. It was heated at 800˚C for 2 hours, producing the soy-pulp derived activated carbon. The activated carbon samples were then cooled at room temperature, washed with distilled water to a pH of 6-7, and dried in an oven at 105◦C for 3 hours until a constant weight of activated carbon samples was obtained. Activated carbon treated with 35% HCl for 24 hours showed four times the elementary mercury adsorption. Acid treatment increased carboxyl, carbonyl, and various other acidic groups on the activated carbon surface, enhancing higher Hg adsorption [9].
The characteristics of the biochar are dependent on the type of raw material, the pyrolysis procedure, and the post-treatment of the biochar. The sorption performance of biochar with modification, such as introducing acids, can eliminate other impurities and introduce more acidic functional groups on the biochar surface. Some common acids include H_3PO_4, H_3BO_3, HNO_3, and HCl. Acidic functional groups may with positive charges and exchange ions with molecules that are negatively charged. Such an interaction is efficient in removing contaminants by adsorbing them onto the biochar surface [10].

2.4 Synthetic Wastewater Preparation

The synthetic wastewater was prepared by making two two-liter stock solution with metals containing Cd (II), Cu (II), Fe (III), and Pb (II), each having a 5 mg/L concentration. The solution contains 0.02754g of $CdN_2O_6 \cdot 4H_2O$, 0.38g of $CuN_2O_6 \cdot 3H_2O$, 0.0724g of $FeN_3O_9 \cdot 9H_2O$, and 0.0212g of $PbN_2O_6 \cdot 6H_2O$ dissolved in two (2) liters of distilled water.

The prepared two liters of synthetic wastewater sample was put in a polyethylene bottle prewashed with 5% v/v HNO_3 and rinsed several times with distilled water to prevent contamination and determination of the exact concentration of the target analyte. 100 mL of the synthetic wastewater sample will be subjected to heavy metal analysis for Cd, Cu, Fe, and Pb using an Atomic Absorption Spectrometer for pre-treatment results. After the analysis, the results will then be recorded.

2.5 Batch Adsorption Laboratory Scale Implementation

A laboratory-scale batch adsorption system setup was conducted using a separatory funnel with a plastic stopper. The separatory funnel is filled with prepared activated carbon from each treatment with a corresponding weight of two (2) grams. The dosage used for every treatment is 0.2 (2 g of carbon/ 100 mL of synthetic wastewater).

In Slow Pyrolysis Treatment, two (2) grams of soy-pulp derived activated carbon prepared from the slow pyrolysis technique will be used for the adsorption of heavy metals in 100 mL of synthetic wastewater. In Fast Pyrolysis Treatment, two (2) grams of soy-pulp derived activated carbon prepared from fast pyrolysis technique will used for the adsorption of heavy metals in 100 mL of synthetic wastewater, and in Conventional Pyrolysis Treatment, two (2) grams of soy-pulp derived activated carbon prepared from conventional pyrolysis technique will used for the adsorption of heavy metals in 100 mL of synthetic wastewater. Another treatment was added, which is two (2) grams of activated carbon derived from coconut shell that will serve as the Positive Control and the Negative Control, where no activated carbon was applied in the treatment.

The effect of adsorbent dose from 2.0 to 12.0 g/L with constant parameters for contact duration of 120 minutes and temperature of 315 K for adsorption of heavy metals: Pb, Cu, Cd, Zn, Ni, and Cr onto activated carbon derived from neem leaf powder shows that percentage removals were increased with increasing dosage of adsorbent up to 10 g/L and then, removals were constant thereafter. This revealed that equilibrium can be attained at 10 g/L for heavy metals. This behavior is because of the enhancement in accessibility of surface' active sites with increasing dose up to 10 g/Land then, all active sites were exhausted after 10 g/L [11].

Whatman no. 1 Filter paper will be used at the base to prevent the escape of the activated carbon when filtering or collecting treated wastewaters. A synthetic wastewater sample will be used as feed for the adsorption setup. From the sample prepared, 100 mL of each was transferred to a 250 mL beaker to be subjected to each treatment. Then, each sample is poured into the filtration system.

For efficient adsorption of heavy metals, 60 minutes of contact time was applied. The time started after pouring the sample from the filtration system. Further shaking after the equilibrium time only results in desorption [12]. After 60 minutes of contact time, 100 mL of the treated water sample will then be filtered and collected from each treatment. Treated wastewater samples will be placed in a 350 mL plastic bottle prewashed with 16% v/v HCl and rinsed with distilled water.

A study by Abdulrazak et al. [13] on the Evaluation of the removal efficiency of heavy metals by low-cost activated carbon prepared from African palm fruit discusses the effect of contact time on the removal of heavy metals: Copper, Nickel, Lead, and Cadmium. Results showed that there are higher treatment efficiencies at 60 min, except for Lead, which shows higher treatment efficiency at 90 min. The result obtained is in agreement with similar work carried out by Srinivasan et al [14] on the elimination of persistent odorous compounds from drinking water using chemical-based adsorbent.

The treated water samples were subjected to heavy metal analysis for Cd, Cu, Fe, and Pb using an Atomic Absorption Spectrometer with a detection limit of 0.001 ppm for Cd, and 0.01 ppm for Cu, Fe, and Pb. The detection limit of the AAS shall be able to quantify the lowest concentration of heavy metals present in water at the post-treatment. The results were then recorded. The experiment will be conducted thrice to obtain samples for three (3) replicates.

2.6 Instrumentation/Calibration of AAS

Calibration of the atomic absorption spectrometer was carried out by using an external calibration curve. The external calibration curve was prepared from a solution of known concentrations of the sample element, which was also known as a stock solution. High-purity metal salts of Cd, Cu, Fe, and Pb dissolved in high-purity acids were used to make the stock solution. Working standards were diluted from the stock standard. Detection limit were set to 0.001 ppm for Cd, and 0.01 ppm for Cu, Fe, and Pb.

2.7 Statistical and Data Analysis

Results obtained from pre-treatment and post-treatment will be used to compute the adsorption efficiency of the activated carbon on each treatment.

$$\%R = \frac{(Co-Ce)}{Ce} x100$$

Legend:

%R-adsorption efficiency
Co-initial concentration of metal in solution, mg/L
Ce-final concentration of metal in solution after adsorption, mg/L

Further, the mass of heavy metal ions adsorbed per unit of adsorbent was calculated by equation:

$$q = \frac{(Co-Ce)*V}{M*1000}$$

Legend:

q- adsorption capacity, mg metal/g adsorbent
V- volume of the heavy metal solution, mL
M- mass of the adsorbent, g

Data will be analyzed using a Randomized Complete Block Design (RCBD) with 3 treatments, positive control, and negative control at 3 replications. The data will be subjected to descriptive statistics using mean and standard deviation. To test the significant difference between treatments, analysis of variance (ANOVA) will be used at $p \leq 0.05$. The LSD and Levene's homogeneity of variance will also be investigated using the IBM SPSS Windows version 22 package (IBM Corp., USA).

2.7 Research Design

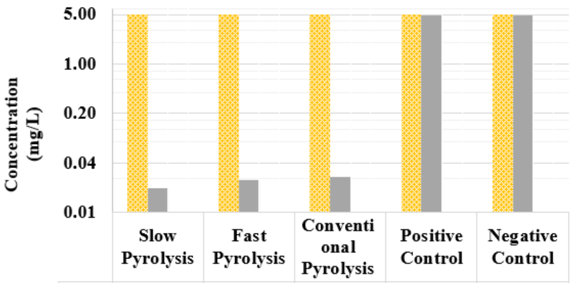
This study utilized a Pretest–Posttest Controlled Group Design. The wastewater for pre-treatment was analyzed for quantitation of the presence of Cu, Cd, Fe, and Pb. The design had five (5) treatments, each had three (3) replicates. The Slow Pyrolysis Treatment, where 100 mL of synthetic wastewater is treated with two (2) grams of adsorbent prepared by the Slow Pyrolysis technique. The Fast Pyrolysis Treatment, where 100 mL of synthetic wastewater is treated with two (2) grams of adsorbent prepared by the fast pyrolysis technique. The Conventional Pyrolysis Treatment where 100 mL of synthetic wastewater with two (2) grams of adsorbent prepared by the conventional pyrolysis technique. The Positive Control, where two (2) grams of commercialized adsorbent derived from coconut shell was used to treat 100 mL of synthetic wastewater. The Negative Control, where no adsorbent was used. All treatments were subjected to the removal of heavy metals Cd, Cu, Fe, and Pb in synthetic wastewater.

After the collection of treated wastewaters in each treatment, the wastewater from post-treatment were analyzed for quantitation of Cd, Cu, Fe, and Pb in the solution to be compared to the pre-treatment results for the computation of adsorption efficiency. Positive and negative control groups were utilized in this study to ensure that the results were due to the independent variable. Replication was utilized to obtain valid and more reliable results.

3. RESULTS AND DISCUSSION

The adsorption efficiency of soy-pulp derived activated carbon in the removal of heavy metals Cd, Cu, Fe, and Pb in wastewater was quantified by computing the %R. Removal of heavy metals is performed via adsorption using various treatments with a contact time of 60 minutes. After 60 minutes, the final concentration (*Ce*) of each treatment was obtained.

3.1 Adsorption Efficiency in the Removal of Cadmium



	Slow Pyrolysis	Fast Pyrolysis	Conventional Pyrolysis	Positive Control	Negative Control
Co	5.0912	5.0912	5.0912	5.0912	5.0912
Ce	0.0177	0.0228	0.0253	4.8125	4.9012
%R	99.65	99.55	99.50	5.47	3.73

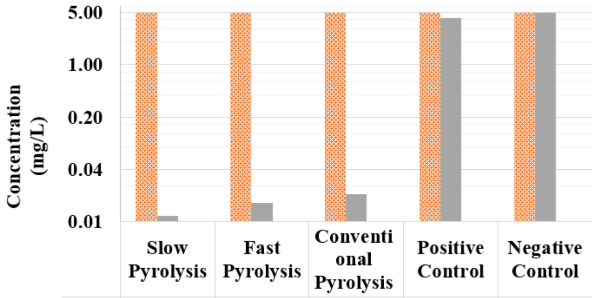
Fig. 2. Mean adsorption efficiency of soy-pulp derived activated carbon in the removal of Cd in wastewater

Figure 2 shows the mean adsorption efficiency in the removal of Cd in wastewater. The initial concentration (*Co*) of Cd in the prepared synthetic wastewater is 5.0912 mg/L.

Slow Pyrolysis Treatment obtained a *Ce* of 0.0177 mg/L and has an adsorption efficiency (%R) of 99.65%. It is then followed by Fast Pyrolysis Treatment with a *Ce* of 0.0228 mg/L and has a %R of 99.55%, and Conventional Pyrolysis Treatment with a *Ce* of 0.0253 mg/L and a %R of 99.50%. All pyrolysis treatments showed high adsorption efficiency for Cd, hovering around 99.55% to 99.65%. However, among the three (3) treatments, Slow Pyrolysis Treatment obtained the highest adsorption efficiency in the removal of Cd.

In the control treatments, the Positive Control had an adsorption efficiency result of 5.47% and the Negative Control, which showed the least adsorption efficiency result of 3.73%. The positive and negative controls are significantly lower, indicating the effectiveness of pyrolysis treatments.

3.2 Adsorption Efficiency in the Removal of Copper



	Slow Pyrolysis	Fast Pyrolysis	Conventional Pyrolysis	Positive Control	Negative Control
Co	4.98209	4.98209	4.98209	4.98209	4.98209
Ce	0.00964	0.01423	0.01882	4.18320	4.94536
%R	99.81	99.71	99.62	16.04	0.74

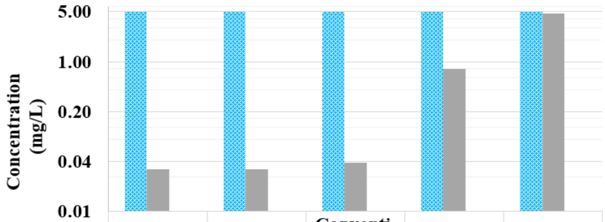
Fig. 3. Mean adsorption efficiency of soy-pulp derived activated carbon in the removal of Cu in wastewater

Figure 3 shows the mean adsorption efficiency in the removal of Cu in wastewater. The initial concentration of Cu in the synthetic wastewater is 4.98209 mg/L.

Based on the data, a *Ce* of 0.00964 mg/L in the Slow Pyrolysis Treatment was obtained, indicating the highest adsorption efficiency recorded of 99.81% in the removal of Cu. Fast Pyrolysis Treatment with a *Ce* of 0.01423 mg/L and Conventional Pyrolysis Treatment with a *Ce* of 0.01882 mg/L, each having a %R of 99.71% and 99.62%, respectively. The mean adsorption efficiency of the experimental treatments in the removal of Cu mirrored the result in the removal of Cd, where each obtained high %R results with a range of 99.62% to 99.81%.

Positive and Negative Control obtained *Ce* of 4.18320 mg/L and 4.94536 mg/L, respectively. The adsorption efficiency of Positive Control is 16.04%. Negative Control, which had the least adsorption efficiency of 0.74% in the removal of Cu. It can be observed that a steep drop in %R values was obtained for both Positive and Negative Controls. The *Ce* values for control groups are still high, indicating that there is minimal removal of Cu during adsorption.

3.3 Adsorption Efficiency in the Removal of Iron



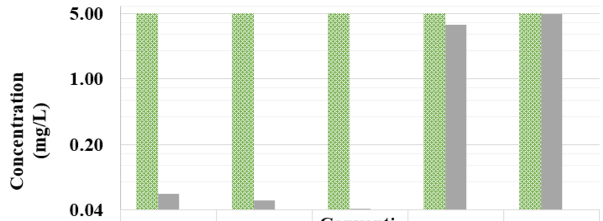
	Slow Pyrolysis	Fast Pyrolysis	Conventional Pyrolysis	Positive Control	Negative Control
Co	5.0080	5.0080	5.0080	5.0080	5.0080
Ce	0.0313	0.0313	0.0379	0.7831	4.7818
%R	99.38	99.38	99.24	84.36	4.52

Fig. 4. Mean adsorption efficiency of soy-pulp derived activated carbon in the removal of Fe in wastewater

Figure 4 shows the mean adsorption efficiency in the removal of Fe in wastewater. The initial concentration of Fe in the synthetic wastewater is 5.0080 mg/L.

Findings showed that in the removal of Fe in wastewater, Slow Pyrolysis Treatment and Fast Pyrolysis Treatment obtained similar results for their final concentration of 0.0313 mg/L, having the same value of %R of 99.38%. Conventional Pyrolysis Treatment with a *Ce* of 0.0379 has a slightly lower adsorption efficiency of 99.24%. The pyrolysis treatments are effective with high adsorption efficiency, ranging from 99.24% to 99.38% in the removal of Fe. For the control treatments, the Positive Control obtained a *Ce* of 0.7831 mg/L, which shows a lesser adsorption efficiency of 84.36%. A notable efficiency drop of 4.52% was noted at the Negative Control, which indicates less removal of Fe in wastewater.

3.4 Adsorption Efficiency in the Removal of Lead



	Slow Pyrolysis	Fast Pyrolysis	Conventional Pyrolysis	Positive Control	Negative Control
Co	5.0822	5.0822	5.0822	5.0822	5.0822
Ce	0.0594	0.0502	0.0411	3.7580	4.9817
%R	98.83	99.01	99.19	26.06	1.98

Fig. 5. Mean adsorption efficiency of soy-pulp derived activated carbon in the removal of Pb in wastewater

Figure 5 shows the mean adsorption efficiency in the removal of Pb in wastewater. The prepared synthetic wastewater has an initial concentration of 5.0822 mg/L of Pb.

Based on the data, a reverse order was observed on the %R for the removal of Pb, where Conventional Pyrolysis Treatment had the highest adsorption efficiency result of 99.19% and a *Ce* value of 0.0411 mg/L. This highlights the high performance of Slow Pyrolysis Treatment in the removal of Pb. It is followed by Fast Pyrolysis Treatment with a *Ce* value of 0.0502 mg/L with a %R of 99.01%. Then, Conventional Pyrolysis Treatment, which has a slightly lower %R of 98.83% and *Ce* value of 0.0594 mg/L. Pb removal also sees high efficiency in pyrolysis treatments from 98.83% to 99.19%, with a sharp decline in the Positive control and even further in the Negative Control. Positive Control with *Ce* of 3.7580 mg/L had an adsorption efficiency of 26.06%, which shows less removal of Pb, and still the Negative Control showed the least adsorption efficiency with a result of 1.98%, having *Ce* of 4.9817 mg/L.

Overall, Slow Pyrolysis Treatment, where soy pulp-derived activated carbon, which was prepared by the slow pyrolysis technique, was pyrolyzed at 350°C for 4 hours, consistently showed high adsorption efficiency in the removal of heavy metals, having the highest %R in removing Cd, Cu, and Fe in wastewater. It can be observed during the experiment that after pouring the wastewater into the filtration setup in Slow Pyrolysis Treatment, the wastewater turned clear, and the yellowish color was reduced immediately. Further reduction of yellowish color was observed after filtration with a contact time of 60 minutes.

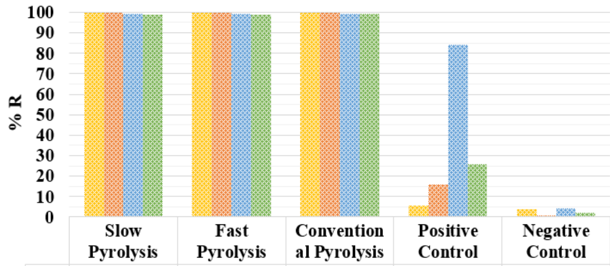
The Fast Pyrolysis Treatment and Conventional Pyrolysis Treatment also show high adsorption efficiencies, which are above 98% for all four (4) metals Cd, Cu, Fe, and Pb. Conventional Pyrolysis Treatment, where soy pulp-derived activated carbon was pyrolyzed at 400 °C for 10 minutes, showed the highest adsorption efficiency in removing Pb in wastewater.

Further, Positive Control shows varying degrees of adsorption efficiency, significantly lower than pyrolysis treatments. It was observed that some of the adsorbent used in the Positive Control had floated on the surface after pouring the wastewater in the separatory funnel. It can be seen that the wastewater had less contact with the surface area of the powdered adsorbent, which leads to low adsorption efficiency results. Since there is no adsorbent applied in the Negative Control, therefore, most of the heavy metals were carried as filtrate. Some were part of the residue attached to the filter paper after the filtration process, which results in the least adsorption efficiency among treatments. This shows the notable impact of applying adsorbents in the removal of heavy metals in wastewater.

The results were aligned to the study of Sizirici, F. et al [15], where activated carbon pyrolyzed at higher temperatures did not produce significant adsorption efficiencies for the removal of heavy metals in water as compared to activated carbon pyrolyzed at lower temperatures. Slow pyrolysis produces biochar with more developed surface areas and functional groups, which enhances their adsorption capacities for heavy metals.

Moreover, in the study of Abdallah, N. et al [16] stated that low temperatures and long residence times enhance biochar yields. Higher pyrolysis temperature decreases the amount of biochar produced due to the thermal degradation of heavy molecular weight hydrocarbons. High pyrolysis temperature and reduced particle size enhance the conversion of hydrocarbons into bio-oil, which leads to low biochar yields. The highest biochar yields were reported at a temperature of 200 °C. The effect of the residence time on the amounts of bio-char produced can is explained that high residence times can be beneficial to the re-polymerization of the elements present in the biomass, which will increase bio-char yields.

3.5 Comparative Mean Adsorption Efficiency (%R)



	Slow Pyrolysis	Fast Pyrolysis	Conventional Pyrolysis	Positive Control	Negative Control
Cd	99.65	99.55	99.55	5.47	3.73
Cu	99.81	99.71	99.62	16.04	0.74
Fe	99.38	99.38	99.24	84.36	4.52
Pb	98.83	99.01	99.19	26.06	1.98

Fig. 6. Comparative Mean Adsorption Efficiency results in removal of heavy metals Cd, Cu, Fe, and Pb

Figure 6 shows the comparative mean adsorption efficiency results of each treatment in the removal of heavy metals. The Slow Pyrolysis Treatment consistently showed high removal efficiencies for all four (4) metals, with the highest result obtained in the removal of Cu at 99.81%. Fast Pyrolysis Treatment also showed high adsorption efficiency, with the highest in removing Cu and lowest in Pb, with results of 99.71% and 99.01% respectively. Similarly, Conventional Pyrolysis Treatment had the highest adsorption efficiency in removing Cu, with a result of 99.62%, and the lowest result at Pb with 99.19%.

For Positive Control, adsorption efficiency showed the highest in removing Fe with the result of 84.36% and lowest in removing Cd at 5.47%. Positive Control shows varying degrees of adsorption efficiency, significantly lower than pyrolysis treatments. This indicates that coconut shell-derived activated carbon as an adsorbent is less effective and has a minimal removal of heavy metals in wastewater compared to soy-pulp derived activated carbon.

Negative Control had the lowest adsorption efficiency in all treatments, where the highest adsorption efficiency was obtained in removing Fe, with a result of 4.52%, and the lowest adsorption efficiency was obtained in removing Cu, with a result of 0.74%. Negative Control exhibits very low efficiencies, highlighting the importance of adsorption using soy-pulp derived activated carbon in removing heavy metals in wastewater and varying pyrolysis techniques in the treatment process.

The order of removal of heavy metals based on decreasing adsorption efficiency of the given Treatments are as follows: Slow Pyrolysis: Cu>Cd>Fe>Pb; Fast Pyrolysis: Cu>Cd>Fe>Pb; Conventional Pyrolysis: Cu>Cd>Fe>Pb; Positive Control: Fe>Pb>Cu>Cd; and Negative Control: Fe>Cd>Pb>Cu. This study highlights the high affinity of copper ions to the synthesized soy-pulp derived activated carbon across different pyrolysis techniques performed.

3.6 Statistical Analysis

In this study, one-way ANOVA was used to determine the statistical difference among treatments. The results revealed in Table 1 that there is a significant difference (p=0.000) on the adsorption efficiency of soy-pulp derived activated carbon in the removal of Cd, Cu, Fe, and Pb in wastewater among treatments.

Table 1. One way ANOVA results for the significant difference on the adsorption efficiency of Soy-pulp derived Activated Carbon in the removal of heavy metals Cd, Cu, Fe, and Pb in wastewater

	C	df	Mean Square	F	p
Cd	Between Groups	4	8117.783	53595.84	0.000
	Within Groups	10	0.151		
Cu	Between Groups	4	7594.477	82797.028	0.000
	Within Groups	10	0.092		
Fe	Between Groups	4	5102.486	14170.926	0.000
	Within Groups	10	0.360		
Pb	Between Groups	4	6719.265	34681.850	0.000
	Within Groups	10	0.194		

Since there is a significant difference in each treatment, a Tukey post-hoc test was used to determine which of the treatments are significantly different from each other.

Results showed that there are no significant differences between Slow, Fast, and Conventional Pyrolysis Treatments in the removal of Cd, Cu, Fe, and Pb. This suggests that all three pyrolysis methods are equally effective, underscoring their potential for environmental remediation in the removal of contaminants, especially the heavy metals, and to be used as treatment of wastewater in process industries.

Moreover, the experimental treatments showed significant differences compared to the control treatments, highlighting that the experimental treatments have substantially higher adsorption efficiency and effective in the removal of heavy metals in wastewater than the control treatments.

3.7 Adsorption Capacity

The data presented in Table 2a illustrate the adsorption capacity of soy-pulp derived activated carbon in binding metal ions at its particle surface, measured in mg of metal/g of adsorbent. Each treatment was measured for its adsorption capacity for the adsorption of Cd, Cu, Fe, and Pb.

The soy-pulp derived activated carbon synthesized via slow pyrolysis showed high adsorption capacities for all four metals- Cd, Cu, Fe, and Pb, with q values ranging from 0.24862 to 0.25368 mg/g. This indicates that this method supports maximum interaction and affinity of metal ions to the particle surface. Further, the soy-pulp derived activated carbon synthesized via fast pyrolysis also demonstrated strong performance for its adsorption capacity, although slightly less effective in the case of Fe, which had a lower q value of 0.23475 mg/g. For the other

Table 2. Adsorption Capacity of Soy-pulp derived Activated Carbon in the removal of heavy metals Cd, Cu, Fe, and Pb in wastewater

(a) Experimental Treatments

Metals	N	V (mL)	M (g)	Slow Pyrolysis Co (mg/L)	Slow Pyrolysis Ce (mg/L)	Slow Pyrolysis q (mg/g)	Fast Pyrolysis Co (mg/L)	Fast Pyrolysis Ce (mg/L)	Fast Pyrolysis q (mg/g)	Conventional Pyrolysis Co (mg/L)	Conventional Pyrolysis Ce (mg/L)	Conventional Pyrolysis q (mg/g)
Cd	3	100	2	5.0912	0.0177	0.25368	5.0912	0.0228	0.25342	5.0912	0.0253	0.25330
Cu	3	100	2	4.98209	0.00964	0.24862	4.98209	0.01423	0.24839	4.98209	0.0188	0.24816
Fe	3	100	2	5.0080	0.0313	0.24884	5.0080	0.313	0.23475	5.0080	0.0379	0.24851
Pb	3	100	2	5.0822	0.0594	0.25114	5.0822	0.0502	0.2516	5.0822	0.0411	0.25206

(b) Control Treatments

Metals	N	V (mL)	M (g)	Positive Control Co (mg/L)	Positive Control Ce (mg/L)	Positive Control q (mg/g)	Negative Control Co (mg/L)	Negative Control Ce (mg/L)	Negative Control q (mg/g)
Cd	3	100	2	5.0912	4.8125	0.01394	5.0912	4.9012	0.0095
Cu	3	100	2	4.98209	4.18320	0.03995	4.98209	4.9454	0.00184
Fe	3	100	2	5.0080	0.7831	0.21125	5.0080	4.7818	0.01131
Pb	3	100	2	5.0822	3.7580	0.06621	5.0822	4.9817	0.00503

metals, the q values were comparable to those from the Slow Pyrolysis Treatment.

The soy-pulp derived activated carbon synthesized via conventional pyrolysis produced results similar to both Slow and Fast Pyrolysis Treatments, with q values closely clustered around 0.248 to 0.253 mg/g across all metals. This indicates a consistent and balanced adsorption process that may serve as a reliable baseline for metal removal. The method appears effective regardless of the type of metal, suggesting broad applicability and stable performance under standard conditions.

In contrast, the Positive Control showed significantly lower adsorption capacities, with q values much smaller than those observed in the three experimental treatments presented in Table 2b. For instance, Cd and Cu had q values of 0.01394 mg/g and 0.03995 mg/g, respectively. This suggests that the positive control, an activated carbon derived from coconut shell, is a less effective adsorbent and not favorable for the adsorption of metal ions. Interestingly, Fe had a relatively higher q (0.21125 mg/g) in this setup, indicating some degree of affinity between the control material and Fe ions. However, the Negative Control confirmed the absence of meaningful adsorption, as evidenced by near-zero q values for all metals.

4. CONCLUSION

Activated carbon is known for its high surface area and adsorption capacity, has emerged as a promising material for heavy metal removal. The development of soy-pulp derived activated carbon, a leveraging of agricultural waste, is transformed into an efficient, eco-friendly, and economically viable solution for heavy metal remediation in wastewater treatment.

The study revealed that soy-pulp derived activated carbon synthesized via slow, fast, and conventional pyrolysis techniques showed high adsorption efficiencies in the removal of heavy metals in wastewater, ranging from 98.00% to 99.81%. Moreover, the soy-pulp derived activated carbon synthesized via slow pyrolysis technique at 350 ºC for 4 hours obtained the highest adsorption efficiency in removal of Cd, Cu, and Fe in wastewater with results 99.65%, 99.81%, and 99.38% respectively and third in removal of Pb with result of 98.83%. It can be concluded that pyrolysis parameters like temperature and residence time can significantly impact biochar properties. Soy-pulp derived activated carbon pyrolyzed by long residence time and low heating temperature can result in high removal efficiency of heavy metals in wastewater. The order of removal of heavy metals based on decreasing adsorption efficiency of the given Treatments is as follows: Slow Pyrolysis: Cu>Cd>Fe>Pb, Fast Pyrolysis: Cu>Cd>Fe>Pb, and Conventional Pyrolysis: Cu>Cd>Fe>Pb. This study underscores the dominant affinity of all pyrolysis treatments for Cu (II) and comparatively lower for Pb (II), indicating a consistent trend across different pyrolysis methods. The findings suggest that pyrolysis-derived adsorbents are particularly effective for Cu removal and may guide future optimization strategies for targeted heavy metal remediation in wastewater treatment.

There are no significant differences between the adsorption efficiencies of soy-pulp derived activated carbon prepared from the three (3) pyrolysis techniques: slow, fast, and conventional, suggesting that all are viable for treatment and removal of heavy metals Cd, Cu, Fe, and Pb in wastewater. These findings highlight the potential of using agricultural waste, specifically the soy pulp, in producing an effective adsorbent that embodies a sustainable approach to waste reduction, waste management, and mitigating the environmental impact of process industries and chemical laboratories. The study supports the feasibility of integrating soy-pulp derived activated carbon into wastewater treatment processes to address heavy metal removal effectively. Bio-based adsorbent aligns with global efforts toward sustainable environmental management and provides a scalable option for chemical and industrial wastewater treatment applications.

5. RECOMMENDATION

Future studies could focus on adsorption isotherms to explore the optimization of pyrolysis conditions, enhancing the adsorptive properties of the activated carbon. Investigating temperatures, heating rates, and durations could yield insights into producing more efficient adsorbents.

Characterization of adsorbent using scanning electron microscope could provide information on the surface morphologies of unactivated biochar, activated carbon, and activated metals-loaded adsorbent. Scanning electron microscope analysis can reveal the structure, texture, pore size, and exposed cavities of these materials, which are critical factors influencing adsorption efficiency. By examining these surface characteristics, researchers can better understand how the activation process and metal loading alter the adsorbent's properties, enabling the optimization of conditions to enhance its performance in removing heavy metals from wastewater.

Researchers must expand their focus on the removal of heavy metals not mentioned in the current research, which pose serious threats to the environment and health. Studies should explore the effectiveness of soy pulp-derived activated carbon in adsorbing a broader range of heavy metals. By expanding the scope of research to include these additional contaminants, a more comprehensive understanding of the adsorbent's capabilities can be achieved, potentially leading to more robust and versatile solutions for heavy metal remediation in wastewater treatment.

Design and development of technologies is recommended to apply soy pulp-derived activated carbon in industrial settings to treat wastewater containing heavy metals. The study's findings indicate that this approach can serve as an effective and sustainable solution for managing heavy metal pollution. Implementing this adsorbent in real-world industrial applications could help industries comply with environmental regulations, reduce the toxicity of their effluents, and promote the use of renewable agricultural by-products, thereby contributing to environmental sustainability and cost-effective wastewater management.

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