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TEMPERATURE'S IMPACT ON HEAVY METAL REMOVAL WITH CALCINED CASSAVA PEEL AS AN ADSORBENT

***Tersoo Terrence Akuto, Umar Alfa Abubakar, Donnatus Begianpuye Adie, Samson Igbebe and Terhile Thaddeus Jen**

Department of Water Resources and Environmental Engineering, Ahmadu Bello University Zaria

*Corresponding authors' email: tersooakuto@gmail.com Phone: +2348067939139

ABSTRACT

The use of calcined cassava peel as an adsorbent for the removal of heavy metals from wastewater has gained attention in recent years. This study aims to investigate the effect of temperature on heavy metal removal using calcined cassava peel as an adsorbent. The research problem is to determine the optimal temperature range for heavy metal removal using this adsorbent. The methodology involves preparing the adsorbent, preparing the heavy metal solution, and conducting batch adsorption experiments. The data obtained will be analyzed to determine the effect of temperature on heavy metal removal. The findings indicate that the use of sulphuric acid activation enhanced the charge adsorption of the adsorbent and improved the particle retaining capacity for heavy metals. The Langmuir adsorption capacity was determined to be 0.7796, 0.486, and 0.044 for the Langmuir, Freundlich, and Temkin adsorption isotherm models, respectively. The results show that the modification sample at 300˚C achieved the best lead removal efficiency. The implications of this research include providing valuable information on the optimal temperature range for heavy metal removal using calcined cassava peel as an adsorbent, which can be used to design more efficient and cost-effective wastewater treatment systems. Additionally, this research contributes to the development of sustainable technologies for heavy metal removal and can be utilized by policymakers, researchers, and industries to promote environmental protection and sustainable development.

Keywords: Calcined cassava peel, Heavy metal removal, Adsorption, Temperature, Wastewater

INTRODUCTION

The use of calcined cassava peel as an adsorbent for the removal of heavy metals from wastewater has been a topic of interest in recent years. The adsorption process is influenced by several factors, including temperature, pH, and contact time. In this paper, we will focus on the effect of temperature on heavy metal removal using calcined cassava peel as an adsorbent.

According to a study by Adewoye et al., 2019, the efficacy of cassava peel extracts for the removal of heavy metals from hospital sewage sludge in Nigeria was assessed. Aspergillus niger fermentation and crude fermentation extract were obtained from the cassava peels strain of Aspergillus niger and indigenous microflora, respectively. The experiment was carried out by adding 10 ml of the treatment to 3 g of each sludge sample (extracts and controls) at varied temperatures (room and elevated) and pH (3–5). The mixture was centrifuged after a contact time of 1–12 days at 1000 rpm for 1 hour. The results showed that the efficacy of the cassava peel extracts increased with increasing temperature, with the highest removal efficiency observed at elevated temperatures.

Another study by Schwantes et al., 2015 evaluated residues from the processing of cassava roots, or cassava peels, as chemically modified adsorbents with H_2O_2, H_2SO_4, and NaOH, in the removal of metal ions Cd (II), Pb (II), and Cr (III) from contaminated water. The modified adsorbents were chemically characterized for their chemical composition and point of zero charge, while adsorption tests determined the best conditions of pH, adsorbent mass, and contact time between adsorbent and adsorbate in the process of removal of the metal ions. The experiment was carried out at room temperature. The results showed that modified cassava peels are excellent adsorbent, renewable, high availability, and low-cost materials and a feasible alternative in the removal of metals in industries.

Based on these studies, it can be concluded that the removal efficiency of heavy metals using calcined cassava peel as an adsorbent is influenced by temperature. The efficacy of the adsorbent increases with increasing temperature. However, the optimal temperature range for heavy metal removal using calcined cassava peel as an adsorbent is yet to be determined. Further research is needed to determine the optimal temperature range for heavy metal removal using calcined cassava peel as an adsorbent.

MATERIALS AND METHODS

Materials

Table 1: Materials/Reagents

Materials/ Reagents	Apparatus	Source/Location
Cassava *(manihot esculanta)*	Laboratory mortar and pestle	Gwar Che Tarka Benue State
Deionized and distilled water	Tyler mesh sieve (300µm) Buchner Flask (1 Litre)	Zaria Commercial Market

Table 2: Equipment

S/N	Name	Model	Manufacturer
1	Atomic Absorption Spectrophotometer	Varian AA240FS	Edward, England
2	Weighing Scale (0 – 100kg range)	1455T18	WEDA, Holland
3	Oven (0 - 260˚C)	TMOV – 420	Gallenkamp, England
4	Analytical balance	AB204(0 – 180kg)	Mettler Toledo, Switzerland
5	High Vacuum Pump	ES-100	Edward, England
6	Electric Furnace (0 - 1400°C)	TIKIR 11/12	Heraeys, Holland
7	Band heater with temperature control 600 W (220V) 4cm diameter		
8	Ultra Sonicator	350	HS-VINC, New York
9	Magnetic Stirrer	78HW/AMS 300	

Methods

Sample Collection

Five Litre empty containers were properly rinsed with the wastewater and collected from the influent point of Nigerian Institute of Leather and Science Technology, (NILEST) Zaria, Kaduna State, Nigeria. The sample was labelled and stored in a refrigerator to prevent bacterial multiplication which could alter the result. Two sets of samples were collected, for both characterizations of wastewater to ensure replicative results. Standard procedures (American Public Health Association (APHA, 2017) were followed during sampling.

Digestion of Samples

Heavy metals readily form complexes with organic constituents. Therefore, it is necessary to destroy the organics by digestion with strong acids to avoid interferences and to bring metallic compounds in suspension to the solution. Samples were prepared by digesting 50 ml of wastewater with 1.0 ml of concentrated (conc.) HNO_3 following Carvalho et al., 2020 as thus:

i. The sample was well shaken to form a homogenous mixture before sub-sampling.
ii. 50 mL of the sample was taken and suspended into a digestion vessel, fitted with a reflux cap.
iii. 1.0 mL concentrated HNO_3 was added to the sample.
iv. A laboratory control sample (LCS) was also prepared for every batch sample. 50 mL of LCS solution was added to the digestion vessel.
v. The samples were covered with a reflux cover or watch glass and digested for 2.0 hours at 95°C.
vi. After 2 hours of digestion at 95°C, the samples were removed from the heat source and allowed to cool for at least 30 minutes (to reduce any potential harmful fumes from the sample).
vii. The reflux cover or watch glass was removed and sample (s) reconstituted back to 50 mL with de-ionized water. The samples were properly shaken to mix.

Manihot Esculenta Peel preparation

Cassava peels (***Manihot Esculenta***) that were used for the preparation of the activated carbon were collected from Gwar-che in Tarka LGA of Benue State and were properly washed with distilled water to remove dirt and surface impurities and the optimum moisture content was determined.

Production of Activated Carbon

The collected cassava peels after drying and moisture content was determined underwent a process of washing with distilled water which made them more suitable for use as an adsorbent.

Chemical Pretreatment

Each of the carbonized cassava peels was activated with 1m of sulphuric acid for 3 hours at a ratio of 2:1 (Volume of acid: mass of cassava peel) and later oven dried overnight at 200^0C to ensure proper drying according to (Hung *et al.,* 2012).
The material was removed from the oven, cooled for 2 hours, and then washed with distilled water to bring the pH to 7.0 and again oven-dried overnight at 100^0C (Hung *et al.,* 2012). The samples of thermal and chemical pre-treated cassava were labelled CP300, CP400, and CP500.

Thermal pretreatment

The cassava peel was placed on a ceramic flat surface, charged into a furnace, and heated to a temperature of 300^0C at a heating rate of 20-25^0C/min and reduced time of 1-hour Collivignarelli et al. (2017) method. The charred residue was collected and cooled at room temperature. The procedure above was repeated for 400^0C and 500^0C. The thermal pre-treated cassava peel (Carbonised) was labelled C300, C400, and C500 representing carbonization temperatures of the cassava peel at 300, 400, and 500^0C.

Characterization of Adsorbate (Activated Carbon)

Scanning Electron Microscope (SEM) Analysis

The surface morphology of the produced activated carbon was obtained using a scanning Electron Microscope (SEM), and the results presented and discussed

Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The surface functional groups of the samples were estimated by Fourier Transform Infrared (FTIR) spectroscopy analysis using a spectrometer. The pellet for infrared studies was prepared by mixing a given sample with potassium bromide (KBr) crystals and pressed into a pellet. The pellet which was homogenous in appearance and inserted into the IR sample holder for the analysis. The FTIR spectra of the samples and their resolutions were recorded to know the functional group responsible for the lead (Pb) removal.

RESULTS AND DISCUSSION

Characterization of Wastewater

The results obtained from water samples collected at the Leather Research Institute reveal significant concentrations of three heavy metals: lead, chromium, and cadmium. These samples were taken at a pH of 5.7 and a temperature of 22°C. Delving into the specifics:
The concentration of lead in the water sample is 1.11 mg/L. This value exceeds the World Health Organization (WHO) guideline for drinking water, which stands at 0.01 mg/L. Lead, a toxic metal, poses serious health risks to humans at such levels. It can lead to neurological, cardiovascular, and reproductive problems. The primary sources of lead contamination in water include corroded pipes, solder, brass

fittings, mining, and smelting activities, as well as industrial effluents.

The possible causes of the contamination are natural geology, human activities, or both. The water quality should be improved by using appropriate treatment methods and monitoring systems.

In a report written by Hung et al. (2020), water characterization of metal ion concentration was carried out for heavy metals such as lead, cadmium, and chromium and the results showed 0.48 -20.17 mg/L for lead. This confirms a similar pattern of ion contamination through the same industrial process that produces tannery wastewater. As shown in the Table 3 below.

Table 3: Characterization of wastewater

S/No	Heavy metal	pH	Concentration mg/L	Range of Contamination Isa and Jimoh (2015)
1	Lead	5.7	1.11	0.48 -20.17mg/L

Characterisation of Adsorbent

FTIR Results

The major functional groups of the untreated and treated cassava peel were identified via the FTIR spectra test carried out at the multiuser chemistry laboratory. FTIR curves for both treated and untreated cassava peels are shown in Figures 1 and 2 respectively.

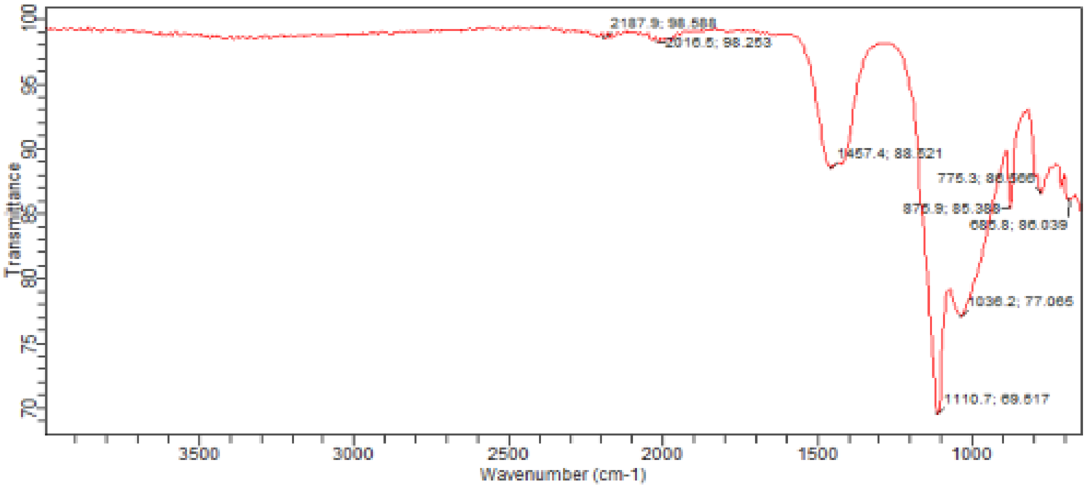

Figure 1: FTIR Curve for untreated cassava peel

The FTIR spectrum of the untreated cassava peel as presented in Figure 1 above shows that there is a fit in a broad band from 2187.9 cm^{-1} which falls between 2260 -2189 cm^{-1}, which indicates a weak stretching alkyne with a molecular formula of CΞC. This group is however not likely to affect the uptake of heavy metals in wastewater, alkyne functional groups are not known to be strong chelating/inhibitor agents (chemical compounds forming stable complexes with metal ions). To enhance the ability of cassava peel or any other biomass material to remove heavy metals from wastewater, various modifications and treatments can be used. These may include, chemical modification as it is so used in the research, to introduce specific functional groups with better metal binding properties, physical treatments like activation or grinding to increase surface area, and pH optimizing and contact time during the adsorption process.

Also noted in the broadband is a wavelength distributed at 2016.5 cm^{-1} which lies within 2140-1990 with a strong stretching isothiocyanate with a molecular formula of N=C=S was observed and could be attributed to organic sulfides. These compounds present in cassava peel as a defense mechanism against pests and diseases need to be processed before they can fit for use as an adsorbent material for heavy metals to prevent the release of cyanogenic glycosides during treatment. The complexes formed from cyanide groups can compete with adsorbent for binding sites on adsorption surfaces potentially reducing the adsorption capacity and decrease the efficiency of metal removal. In some processes like Chemical precipitation cyanide complexes can interfere with the process by forming more stable complexes with metal ions making it more difficult to achieve precipitation and removal. pH changes can alter the speciation and solubility of both heavy metal ions and the cyanide complexes which can disrupt the adsorption process. It is essential to consider these interactions when designing wastewater treatment processes that involve the presence of cyanogenic glucosides from cassava peel. Proper treatment and conditioning may be necessary to mitigate these effects and ensure effective heavy metal uptake. Also, worthy of note is the fit in the band of 1457.4 cm^{-1} assigned to C-H identified to be in the methylene group. This group does not directly affect the uptake of heavy metals in wastewater but is part of the complex structure of cassava peel and the overall composition of the peel contributes to its adsorption capacity. These results were in line with the findings of Nahil and Williams 2012, who reported in their work that the shift in broadband within the range of 2260 – 2189 cm^{-1} could be a weak stretching alkyne.

The S=O bond in the stretching sulfoxide link or bond indicates the presence of a sulfuric-oxy-containing group in the activated cassava peel implying that traces of sulfuric acid could be left in the cassava peel even after repeated washing. The appearance of only the broadband in the regions, 1124-1087 cm^{-1}, 850-550 $cm^{-1,}$ and 690-515 cm^{-1} as captured in Table 4 below and Figure 2 which is assigned to C-O, C-Cl, C-Br could indicate the introduction of haloalkanes or alkyl halides containing functional groups resulting from sulfuric acid and thermal modification.

The presence of a stretching secondary alcohol group indicates an increase in the binding property properties because of the presence of carboxyl (-COOH). These alkyl halide-containing functional groups on the activated cassava peel surface contribute to enhancing charge adsorption.

The result is in line with the findings of Xing et al., 2011 show the bend in the region of 1300-900 cm^{-1} could be caused by sulfuric-Oxy-containing functional groups for the FTIR spectrum of carbon prepared by sulfuric acid activation.

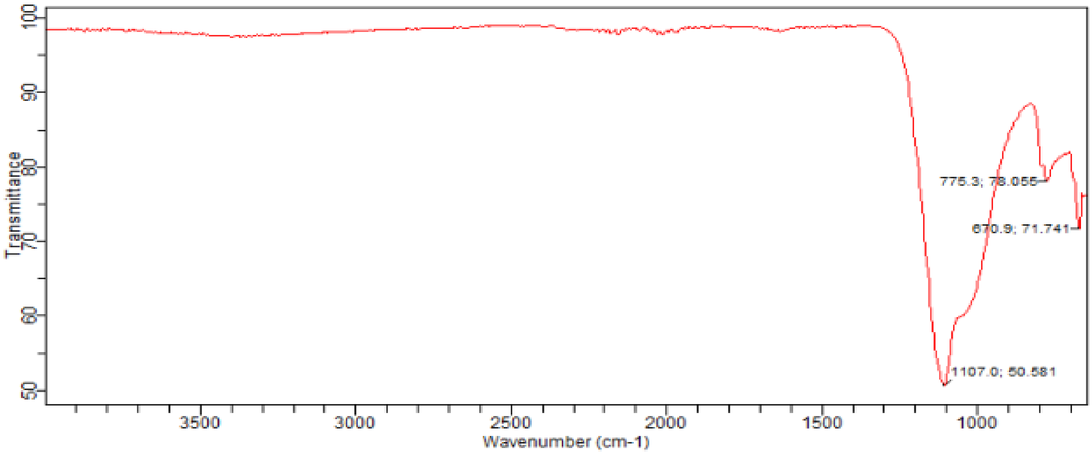

Figure 2: FTIR curve for treated cassava peel

Table 4: FTIR spectra of the studied cassava peel

S/N	Wavelength (cm^{-1})	Molecular formula	Functional Group
1	2187.9	CΞC	Weak alkyne
2	2016.5	N=C=S	Strong isothiocyanate
3	1457.4	C-H	Medium bending alkane
4	1110.7	C-O	Stretching secondary alcohol
5	1036.2	S=O	Stretching Sulfoxide
6	775.3	C-Cl	Strong stretching Halo compound
7	685.8	C-Br	Strong stretching Halo compound

Scanning Electron Microscopy

The SEM image of the cassava sample as shown in Plate I depicts that the SEM structure of the untreated cassava peel is micro-porous with pores diameters of less than 2 µm and makes trapping the pollutants from the effluent extremely difficult, due to low surface area which offers less binding sites with the metal ions while Plate II explains how the activated carbon is highly porous with a major distribution of mesoporous as well as an insignificant number of macropores, which is the characteristic feature of activation using sulfuric acid.

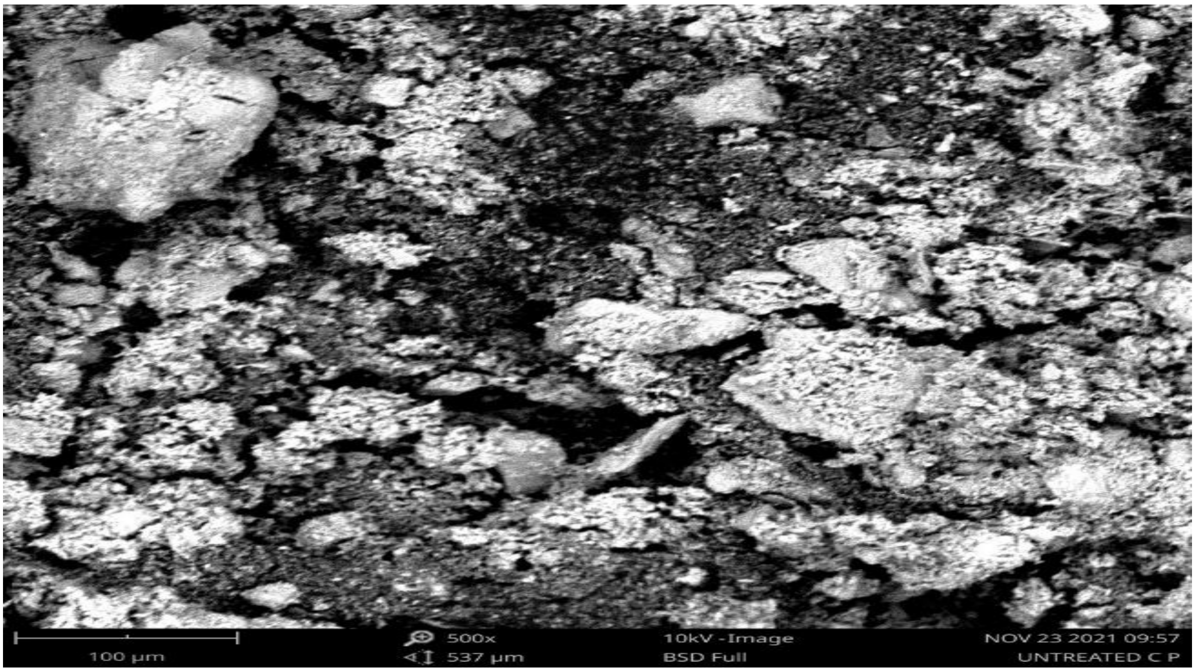

Plate 1: Untreated Cassava Peel SEM

The gases generated from the chemical activation are energized by the thermal energy and erupt with pressure such that to cause cleavage of the bonding in the network structure of the precursor material used in preparing the activated carbon. The honeycomb structure found in the SEM of the treated cassava peel (Plate II) shows how the activation leads to pore widening and thus transforms into mesopores and further into macropores. The geometry and distribution of the pores are controlled by the molarity of the acid used during activation, the bonding characteristics of the precursor material, the activation temperature, the pressure employed during the activation, and the time of activation (Sakhiya *et al*., 2020).

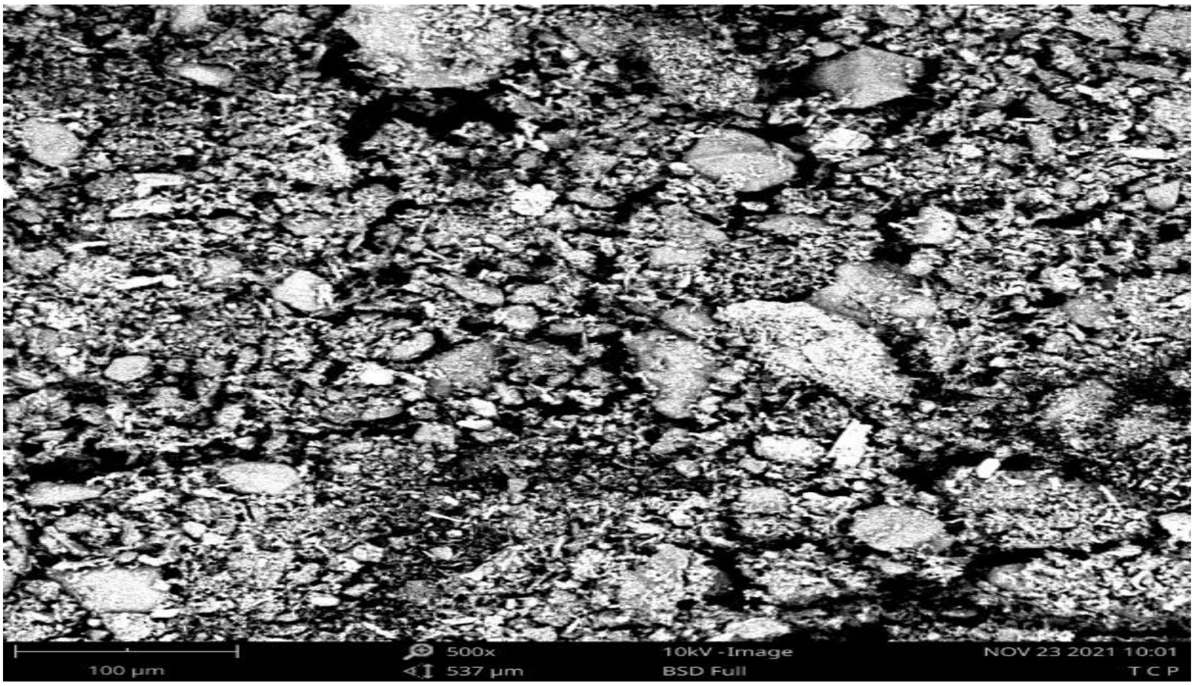

Plate 2: Treated Cassava Peel SEM

Heavy Metal Removal Efficiency Of *Manihot Esculent* Peel

After obtaining the design of the experiment of *Manihot esculent* peel as an adsorbent for removal of heavy metals contaminated tannery water, removal efficiency and adsorption capacity were calculated after the laboratory experiment. The experimental runs were performed. This performance was evaluated with the second-order model to establish the process.

The most commonly employed approach for solid catalyst preparation from plant materials is the calcination process and it is performed at temperatures ranging from 300 - 1200°C as reported by Nath *et al.,* (2023).

Effect of Temperature Variation of Adsorbent on Removal Efficiency of Lead

The elevated temperature had a great impact on removal efficiencies irrespective of the different factors considered (dosage, contact time, and pH) as seen in Figure 3.

While the modification sample at 300˚C gives the best result for lead removal efficiency as it reduces steadily from H3 to H5 as seen in Figure 3, the possible reason for this decline in removal efficiency percentage could be due to the loss of functional groups on the surface of the cassava peel adsorbent as a result of the elevated calcination temperature which caused a breakdown of the chemical bonds that hold the functional groups to the surface of the adsorbent (Shwantes *et al.,* 2015). Gomez *et al.,* 2023 in a report discussed dissociation equilibria as a major influencing factor on adsorption which requires acid/base activation with a trigger of temperature, the result varied temperature above 20°C and found an equilibrium at 95°C which proved that the majority of compounds exhibit an exothermic apparent adsorption enthalpy, which means energy is released and in the case of figure 3 the energy release causes a dissociation of functional groups.

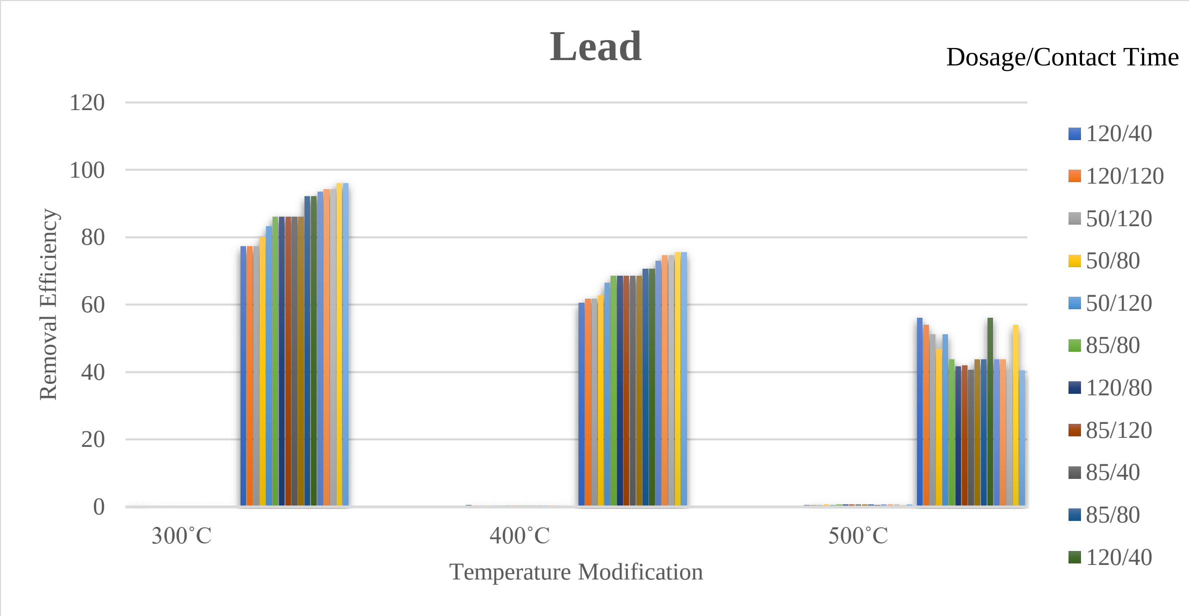

Figure 3: Effect of Elevated Temperature, Contact Time, and Cassava Peel Dosage on Removal Efficiency of Lead

From the experimental results obtained in Table 5, the maximum heavy metal removal efficiency for Lead was 96.036 % at a dosage of 120 mg/L, contact time of 120 min, pH of 6, and its adsorption capacity was 8.883 mg/g. The highest adsorption capacity of Lead removal was 20.9 mg/g, at a pH of 6, contact time of 120 min, and dosage of 50 mg/L which revealed that contact time and pH have the highest influence on adsorption capacity for removal of Lead irrespective of the dosage. It also shows that the removal efficiency is mostly influenced by the dosage of the adsorbent. This agrees with the findings of Chitthaluri et al., (2023) which says that wastewater treatment works best at pH ranges of 6 - 7.

Table 5: Central Composite Design (CCD) Matrix and Output Response for Lead

	Factor 1	**Factor 2**	**Factor 3**	**Response 1**	**Response 2**
Run	**A: Dosage**	**B: Contact Time**	**C: pH**	**Removal Efficiency**	**Adsorption Capacity**
	(mg/L)	(Min)	-	(%)	(mg/g)
1	120	40	8	92.072	8.517
2	**120**	**120**	**6**	**96.036**	**8.883**
3	50	120	8	94.144	20.900
4	50	80	7	93.514	20.760
5	**50**	**120**	**6**	**94.144**	**20.900**
6	85	80	7	86.036	11.235
7	120	80	7	83.243	7.700
8	85	120	7	80.090	10.459
9	85	40	7	77.207	10.082
10	85	80	8	86.036	11.235
11	85	80	7	86.036	11.235
12	120	40	6	92.072	8.517
13	85	80	6	86.036	11.235
14	85	80	7	86.036	11.235
15	50	40	8	77.207	17.140
16	120	120	8	96.036	8.883
17	50	40	6	77.207	17.140

Adsorption Isotherm Study

Two-parameter (Freundlich and Temkin) and three-parameter (Langmuir) adsorption isotherm models were investigated to ascertain the model that best describes the adsorption mechanism of Lead, Cadmium, and Chromium. The linear fitting of Freundlich, Langmuir, and Temkin for lead is shown in Figures 4, 5, and 6.

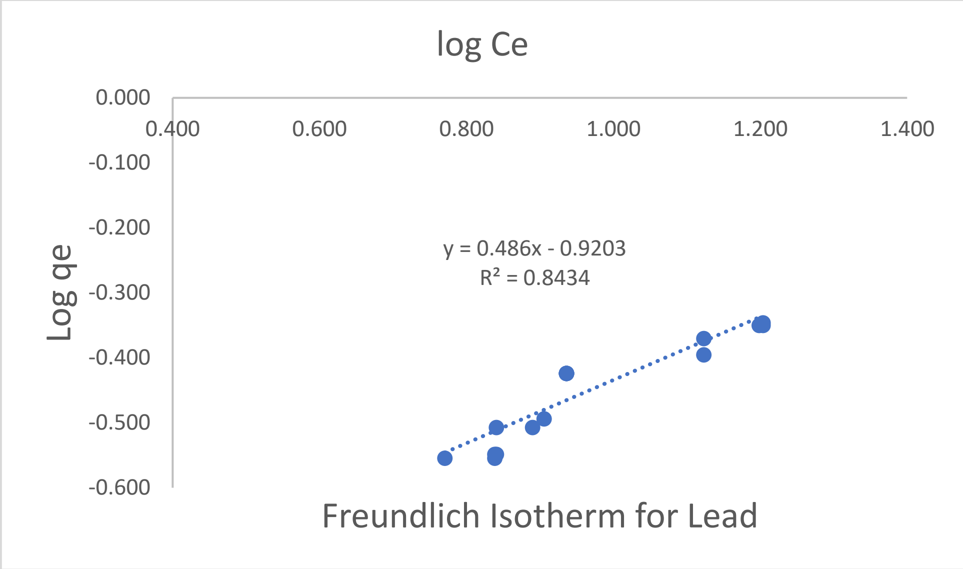

Figure 4: Freundlich Isotherm model for Pb adsorption

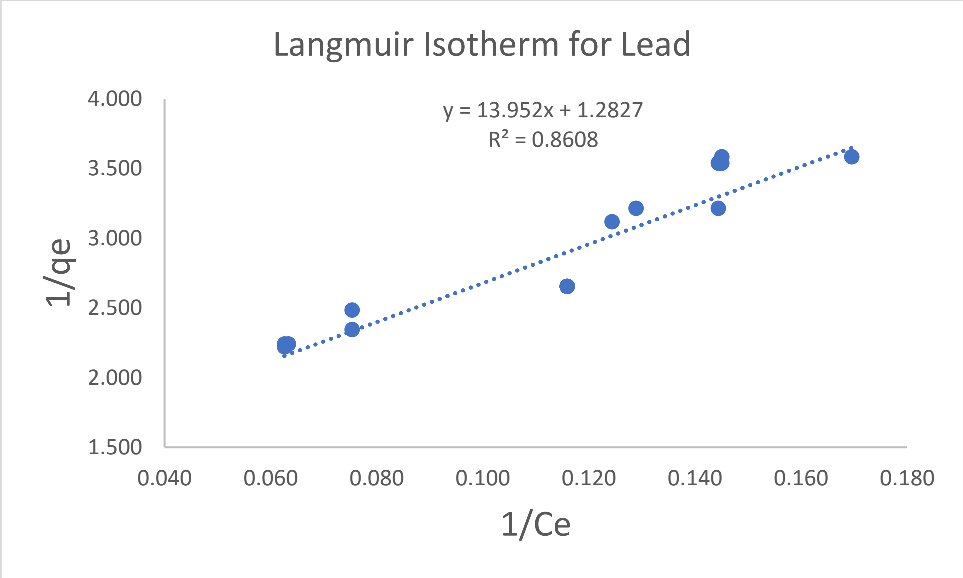

Figure 5: Langmuir Isotherm model for Pb adsorption

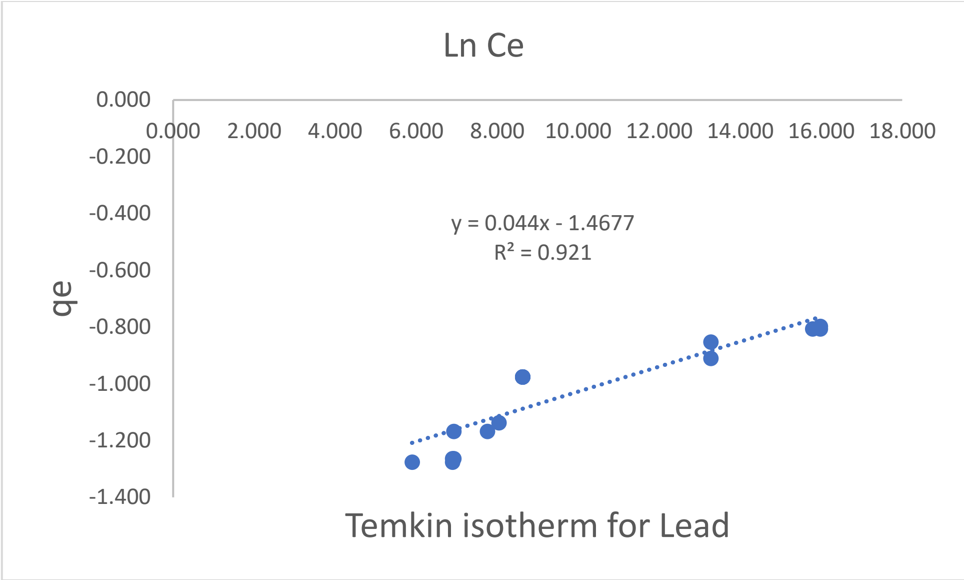

Figure 6: Temkin Isotherm for Pb adsorption

From the isotherm models investigated, it was found that only Temkin isotherm as seen in Table 6 fitted well to the experimental data with a good correlation coefficient (R^2) of 0.921 for lead as compared to Freundlich and Langmuir Models. The fitness of the experimental data to the Temkin model validates that it is a multilayer adsorption which implies that the adsorption sites increase exponentially with adsorption having a high Temkin maximum adsorption capacity of 15.980 mg/g for Lead. Consequently, the fitness of the Temkin model further validates that the adsorbent has a heterogeneous surface concerning the interactions it has with the adsorbate. Shimizu & Matubayasi, (2023) and Chang *et al*., (2020) in their work showed that the Langmuir isotherm strongly agrees that the adsorption mechanism is a mixed type and does not follow the ideal monolayer adsorption. This is because the Langmuir isotherm is a three-parameter model, hence the adsorption of the metal ions could occur either on homogeneous or heterogeneous surfaces of the adsorbent.

Table 6: Isotherm Models and Their Calculated Parameters

Isotherms	Parameters	Lead (Pb)
Freundlich	1/n	0.486
	K_f	0.1201
	R^2	0.8434
Langmuir	q_{max}	0.7796
	K_L	17.896
	R_L	20.865
	R^2	0.8608
Temkin	B_T	0.044
	K_T	3.26E-15
	R^2	0.921

The value of 1/n in Table 6 for Freundlich Isotherm indicates the heterogeneity of the adsorption process. If 1/n is close to 1, it suggests favorable adsorption behavior. A lower value implies stronger adsorption at low concentrations, meaning that a 1/n value of 0.486 indicates that there is higher adsorption at lower concentrations. A high K_f value means a strong adsorption capacity

The Freundlich constant, K_f, represents the adsorption capacity. A higher K_f value means greater adsorption capacity, while The coefficient of determination (R^2) assesses how well the Freundlich model fits the data. An R^2 value close to 1 indicates a good fit.

If the Freundlich model fits well (high R^2), it suggests that the adsorption process is heterogeneous and multi-layered. Therefore, an R^2 value of 0.8434 indicates that the Freundlich model fits the data and the adsorption process is heterogeneous and multi-layered.

Similarly, in the Langmuir Isotherm, the maximum monolayer adsorption capacity q_{max} represents the maximum amount of adsorbate that can be adsorbed on the surface. A higher q_{max} value indicates a higher adsorption capacity, which means that a q_{max} value of 0.7796 depicts a higher adsorption capacity.

The Langmuir constant, K_L relates to the energy of adsorption. If K_L is small, it suggests strong adsorption and in this case, a K_L value of 17.896 suggests strong adsorption. When K_L is relatively high, it indicates favorable adsorption behavior, meaning that the adsorbate molecules are efficiently binding to the surface of the adsorbent material. In practical terms, this implies that the adsorption process is robust and effective for capturing the target substance (in this case, lead, Pb) (Giles et al., 1960).

The separation factor, R_L indicates whether adsorption is favorable or unfavorable. If R_L is between 0 and 1, it's favorable. a R_L value of 20.865 is significantly greater than 1, which implies unfavorable adsorption. This means that the adsorption process for lead (Pb) onto the adsorbent material is less efficient or less ideal according to the Langmuir model (Langmuir, 1918).

CONCLUSION

The following conclusions were drawn from the study:
The wastewater was characterized and temperature, pH, and heavy metal (Lead) initial concentration was determined to be 22°C, 5.7, and 1.11 mg/g respectively. The use of sulphuric acid activation introduced haloalkanes and alkyl halides functional groups enhancing charge adsorption of the adsorbent-adsorbate interaction. The SEM results for both treated and untreated samples showed that the micropores on the untreated sample grow into meso and macro pores with acid activation, improving the (particle retaining capacity of the heavy metals) for each particle mass of the adsorbent material. The adsorption isotherm models such as Langmuir, Freundlich, and Temkin with Langmuir adsorption capacities of 0.7796, 0.486, and 0.044 respectively. The modification sample at 300˚C gives the best result for lead removal efficiency.

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