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Enhanced Biosorption of Heavy Metals from Aqueous Solution Using Chemically Modified Banana (*Musa Sapientum*)

***Onuchuku, C.D., Ekpete, O.A., Kpee, F.**
Department of Chemistry, Ignatius Ajuru University of Education, P.M.B 5047, Port Harcourt, Rivers State, Nigeria

***Corresponding author email:** dcob44love@gmail.com

Abstract
Due to the growing concerns about heavy metal contamination and its devastating impact on the economy, human health and the environment, developing efficient approaches for removing such contaminants has become essential. Adsorption is a cost-effective technique for removing these pollutants. In this study, banana peel adsorbent was prepared and chemically activated with 0.3M HNO_3. Carbonization was carried out at 290°C for 15 mins prior to activation. Physicochemical characterization of the adsorbent revealed that the pH, moisture content (%), ash content (%), volatile matter content (%), bulk density (g/mL) and iodine number mg/g) was 6.70 ± 0.15, 9.24 ± 1.15, 5.41 ± 1.22, 12.53 ± 1.71, 0.15 ± 0.02 and 654.10 ± 0.01 respectively. FTIR spectrum of the adsorbent showed peaks at 3205, 2922, 1565, 1371 and 1028 cm^{-1}. The SEM characterization of adsorbent depicted a semi-regular and heterogeneous morphology, characterized by an abundance of pores with diverse forms and sizes. Metal solutions containing Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+} ions were prepared and batch experiments conducted to evaluate the effect of pH, temperature, dosage, contact time and initial concentration of ions. The optimum conditions for the removal of the four metals were at pH of 6, temperature of 50°C, adsorbent dosage of 0.2 g, contact time of about 75 mins and concentration of 100 mg/L for adsorbent and residual metal concentration were determined using AAS. The physicochemical properties of these adsorbents as well as the initial and residual metal concentrations after treatment showed statistically significant differences at 95% confidence limit ($p \leq 0.05$), demonstrating the effectiveness of the adsorbent and the treatment process. The results of the present work highlighted potential usage of agro-waste in removing heavy metal pollutants and as such be implemented in water purification processes as these wastes are not only effective and efficient but sustainable and cost-effective.

Keywords: Heavy Metals, Banana Peels, Carbonization, Activation, Biosorption

Introduction

Heavy metal contaminants are scientifically proven to be present in wastewater from many industries such as metal manufacturing, dye and paint, chemicals and fertilizer (Ekpete et al. 2010a). According to Napitupulu et al. (2019), these pollutants pose serious threat to the ecosystem and their concentrations must be reduced owing to their toxicity, bioaccumulative, persistence, non-biodegradable nature, and have been consistently desired that their levels be reduced in industrial and municipal effluents prior to their ultimate repository in the ecosystem. Conventional techniques employed to remove these metals from industrial wastewaters include reduction followed by chemical precipitation, ion exchange, solvent and electrolytic extraction, electrodialysis, cementation, reverse osmosis, biodegradation, membrane separation, advanced oxidation processes, biosorption and adsorption etc (Egbosiuba et al. 2024). These processes in addition to being economically expensive have disadvantages such as high reagent and energy requirements, incomplete metal removal especially at low concentration, non-environmental friendly and generation of a large quantity of toxic waste sludge, which necessitates careful disposal in further steps (Ekpete et al.,

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2017). Recently, biosorption (a specialized form of adsorption) has emerged as one of the remediation treatment techniques as it employs agricultural by-products as biosorbents for the removal of heavy metals from aqueous solutions (both high and low concentrations) and any organic matter in waste streams. This is because adsorbents provide large surface area, has low cost, possess simplicity, reusability and higher performance (Khattari et al. 2024).

Many agricultural wastes reported in previous studies have been reported to be capable of removing substantial amounts of metal ions from aqueous solutions by biosorption, chelation and ion exchange (Chopra & Pathak, 2020; Harshala & Wagh, 2022). Owing to their low cost, after these adsorbents have been expended, they can be disposed off without resulting in environmental degradation (Ekpete et al., 2010b). Some of the activated carbons from low-cost materials have been exploited to remove heavy metals from the aqueous solutions include coconut shell, rice husk, *Ricinus communis* seed shell, *Canarium schweinfurthii* seed shell, wood apple shell, pomegranate husk, saw dust, tea and coffee waste, orange peel, peanut shells, *Trapa natans* husk, hazelnut shell, dry tree leaves and barks etc. (Jolly et al., 2022). These biosorbents of agricultural origin have polymeric groups like cellulose, hemi-cellulose, pectin, lignin and proteins that can adsorb heavy metal cations in aqueous solution (Anwar et al., 2010). The aim of this research was to determine some physicochemical properties of the banana peel adsorbent, characterize the adsorbent using FTIR and SEM-EDX analyses, study the adsorption of heavy metals (Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}) from aqueous solutions by activated banana peel by evaluating the effect or influence of various parameters such as pH, temperature, adsorbent dosage, contact time and initial metal concentration in order to ascertain the optimum conditions as well as nature of the adsorption process of these heavy metals (Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}) onto the activated banana peel.

Materials and Methods

Sample collection and treatment

Fresh ripe banana fruits were obtained from a local household garden and an adequate amount of the peels gotten after consumption of the edible portion. After the separation of banana peels from fruit, they were washed several times with tap water and then with distilled water to remove impurities and salts, water soluble, extractable organics and acids. The washed material was dried for 2 days, cut into small pieces using a cutter machine and then dried in an oven at 100 °C overnight after which it was then carbonized in a furnace at 290 ºC for 15 mins (Ekpete et al., 2010a).

Preparation of adsorbent

The adsorbent was prepared as described by Ekpete et al. (2010b). About 500 g of the carbonized material above was mixed and soaked in 500 mL 0.3M nitric acid (HNO_3) (purity 85% Merck, Germany) in a glass jar. The mixture was left overnight for proper activation. This was then repeatedly washed with distilled water until the pH of the filtrate fell between 6 and 8. After filtration, the residue was then dried in an oven at 105°C for 1 hour and stored in a desiccator. This was the prepared adsorbent.

Preparation of adsorbates

Analytical Reagent grade lead nitrate ($Pb(NO_3)_2$,), copper sulphate ($CuSO_4.5H_2O$), iron sulphate, ($FeSO_4.7H_2O$) and zinc sulfate ($ZnSO_4.7H_2O$) were used to prepare the adsorbates. Distilled water was used for the preparation of all solutions and adsorption experiments. The stock solution containing 1000 mg/L stock solution of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+} was prepared by dissolving 0.6256, 0.2542, 0.2009 and 0.2274g of $Pb(NO_3)_2$, $CuSO_4.5H_2O$, $FeSO_4.7H_2O$ and $ZnSO_4.7H_2O$ respectively in distilled water and diluted to 1000mL. Several dilutions of the stock solution were made to obtain specific desired concentrations required for the adsorption study. For every biosorption study, a fresh dilution of the stock solution was made. Required initial concentration of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+} standards were prepared by appropriate dilution of the above stock Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+} ion solutions. The concentrations of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+} ions in the solution before and after equilibrium were determined by Atomic Absorption Spectrophotometer (SavantaAA GBC).

Physicochemical characterization of the adsorbent

Determination of pH

This was done using the method of Abdullah et al. (2019) with a slight modification. In a beaker containing 100 mL of distilled water, 1.0 g of adsorbent was added and then boiled for 5 minutes. The resulting solution was diluted to 200 mL, allowed to cool to room temperature and the pH was measured. The pH meter was calibrated with distilled

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water prior to ensure accuracy of the readings and rinsed with distilled water in-between use. Also, the pH of the solution was adjusted to the required value by using 0.1M HCl or 0.1M NaOH solutions where necessary.

Determination of moisture content

Thermal drying method was used in the determination of moisture content of the samples. 1.0 g of adsorbent was weighed in triplicate and placed in clean, dried, and weighed crucible. Following the method by Ekpete et al. (2017), these preweighed crucibles were placed in an oven at 105°C to constant weight for 4 hr. The percentage (%) moisture content was computed using the difference between the initial and final mass as shown in equation 1:

$$\text{Moisture (\%)} = \frac{\text{Loss in weight on drying (g)}}{\text{Initial weight (g)}} \times 100 \qquad (1)$$

Determination of ash content

This involved preheating the crucibles to about 500°C, cooling them in a desiccator, and weighing them. 1.0 g of the adsorbent was transferred into the crucibles and reweighed. The crucibles the adsorbent were then placed in the furnace and the temperature allowed to rise to 300°C for about 1 hr and allowed to cool in a desiccator to room temperature (30°C) and reweighed. The percentage (%) ash content was derived using equation 2:

$$\text{Ash (\%)} = \frac{\text{Ash weight}}{\text{Oven dry weight}} \times 100 \qquad (2)$$

Determination of volatile matter

1.0 g of the adsorbent was heated at a temperature of 500°C for 10 minutes. The % volatile matter was derived using equation 3:

$$\text{Volatile (\%)} = \frac{\text{Weight of volatile component (g)}}{\text{Oven dry weight (g)}} \times 100 \qquad (3)$$

where, volatile weight is the difference in weight prior to heating and after heating of the samples.

Determination of bulk density

Bulk density determination was done using the methods of Sugumaran et al. (2012) although with a slight modification. A glass cylinder (25 mL) was filled to a specified volume with 40-mesh powder carbon sample of the adsorbent and dried in an oven at 105°C overnight. The cylinder was tapped for 1-2 minutes to compact the content of the cylinder and the bulk density calculated in g/mL following the formula in equation 4:

$$\text{Bulk density} = \frac{\text{Mass of sample (dry)}}{\text{Volume of measuring cylinder}} \qquad (4)$$

Determination of iodine number

For iodine number test, 0.7 g of the adsorbent was put into 250 mL conical flask and to this 10 mL of 5% HCl was added and stirred. Then stock iodine solution of 100 mL (2.7 g of iodine and 4.1 g of KI in 1L of distilled water) which was standardized using a standard solution of sodium thiosulphate (0.1 M) was added to this and the mixture was shook for precisely 20 mins. The resulting mixture was filtered using a filter paper. Using starch as an indicator, an aliquot portion of 30 mL was titrated with 0.1 M sodium thiosulphate. The percent iodine adsorbed by the adsorbent was calculated by applying the formula in equation 5:

$$\text{Iodine number (\%)} = \frac{(\text{mL of } Na_2S_2O_3 \text{ in blank - mL } Na_2S_2O_3 \text{ in sample})}{\text{mL of } Na_2S_2O_3 \text{ in blank}} \times 100 \qquad (5)$$

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Characterization of the adsorbents

FTIR characterization of prepared adsorbent

The analysis was conducted using the Buck Scientific M530 USA FTIR. This device was equipped with a detector of deuterated triglycine sulphate with a beam splitter of potassium bromide (KBr). The spectra were obtained and manipulated using the Gram A1 software. Adsorbent weighing approximately 1.0g were combined with 0.5ml of nujol, thoroughly mixed, and then set on a salt pellet. FTIR spectrum was shown as transmittance values and collected during the measurement at wavenumber range of 4,000–600 cm^{-1} and co-added at 32 scans with a resolution of 4 cm^{-1}.

SEM-EDX characterization of prepared adsorbent

Investigations into the morphology of the adsorbents were carried out using SEM Quanta FEG 450 (FEI) (APOLLO X - EDAX). Approximately 0.5g of the adsorbent was coated with Au/Pd (gold/lead) film and the SEM images were obtained using a secondary electron detector. Two independently chosen particles were subjected to point chemical analysis. This interaction generated various signals, including secondary electrons (for topography), backscattered electrons (for atomic number contrast), and characteristic X-rays. The EDX detector measured the energy and intensity of these X-rays, generating a spectrum that showed peaks corresponding to the elements present in the adsorbent.

Batch adsorption studies

Determination of the effect of pH

The effect of pH on the amount of each metal ion was analyzed at pH range of 3 - 7. A 250 mL conical flask was filled with 50 mL of metal ions at a concentration of 100 mg/L, then 0.2 g of biomass was added and shaken at 150 rpm. This was allowed to settle for 1 hour in a water-bath at 30°C, filtered and analysed to evaluate the residual metal ions conducted.

Determination of the effect of temperature

An aliquot of 50 mL of the working solution (metal ion concentration of 100 mg/L) measured into eight 250 mL conical flasks and 0.2 g of the biomass which was earlier sieved was weighed and added. The flasks were heated on a thermostatic water bath at 30, 40, 50, 60, and 70°C while being shaken at 150 rpm. After 5-mins centrifugation, the suspensions were filtered through Whatman No. 40 filter paper and subjected to analysis. This same procedure was carried out for all heavy metals under investigation.

Determination of the effect of adsorbent dosage

To determine the effect of dosage, accurately weighed biomass samples, ranging in mass from 0.2 to 0.7 g, were filled into 250 mL conical flasks. To each conical flask, 50 mL 100 mg/L of metal ion solutions were added. The opened top of the conical flask was covered tightly with cellophane and shaken for 1 hr, after which the suspensions were filtered using Whatman No. 40 filter paper, centrifuged for 5 min at 150 rpm, and examined.

Determination of the effect of contact time

At 15-minute intervals, the impact of contact time on the removal of the metal ions was investigated i.e duration of contact between the adsorbent and the metals ions in the solution for 15, 30, 45, 60 and 75 mins. Here, 50 mL of metal ions at a concentration of 100 mg/L and 0.2 g of biomass was added into a 250 mL conical flask. This was shaken at 150 rpm for 5mins after which it was centrifuged for 5mins, filtered and analysed to evaluate the residual metal ions conducted.

Determination of the effect of initial metal concentration

Seven conical flasks containing 50 mL of varied solutions ranging from 20 to 120 mg/L were prepared and used for each metal ion independently to assess the impact of different metal ion concentrations. One gram of weighed adsorbent was introduced into each flask, followed by an hour of agitation, filtering, centrifuging for five mins at 150 rpm, and AAS analysis was carried out. All solutions were brought to the ideal pH level by gradually adding 0.1 M

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NaOH and 0.1 M HNO_3, except for the pH effect experiment, which was conducted at varying pH levels. Calibration was done for each metal and controls of each of the metal solutions were run to detect any metal precipitation or contamination.

Determination of metal removal efficiency

The efficiency of metal removal was determined to ascertain the effectiveness of the treatment using the relation in equation 6;

$$\% \text{RE} = \frac{C_0 - C_1}{C_0} \times 100 \qquad (6)$$

where, RE is the metal removal efficiency relative to the initial metal concentration in water,

C_0 is the initial concentration of the metal in water, and C_1 is the residual concentration of the metal in the treated water.

Results

The pH, moisture content (%), ash content (%), volatile matter content (%), bulk density (g/mL) and iodine number (%) of the prepared adsorbent is shown in Table 1.

Table 1: Physicochemical parameters of the adsorbent

Parameter	***Adsorbent***
pH	6.70 ± 0.15
Moisture content (%)	9.24 ± 1.15
Ash content (%)	5.41 ± 0.31
Volatile matter (%)	12.53 ± 1.18
Bulk density (g/mL)	0.15 ± 0.02
Iodine number (%)	65.41 ± 0.01

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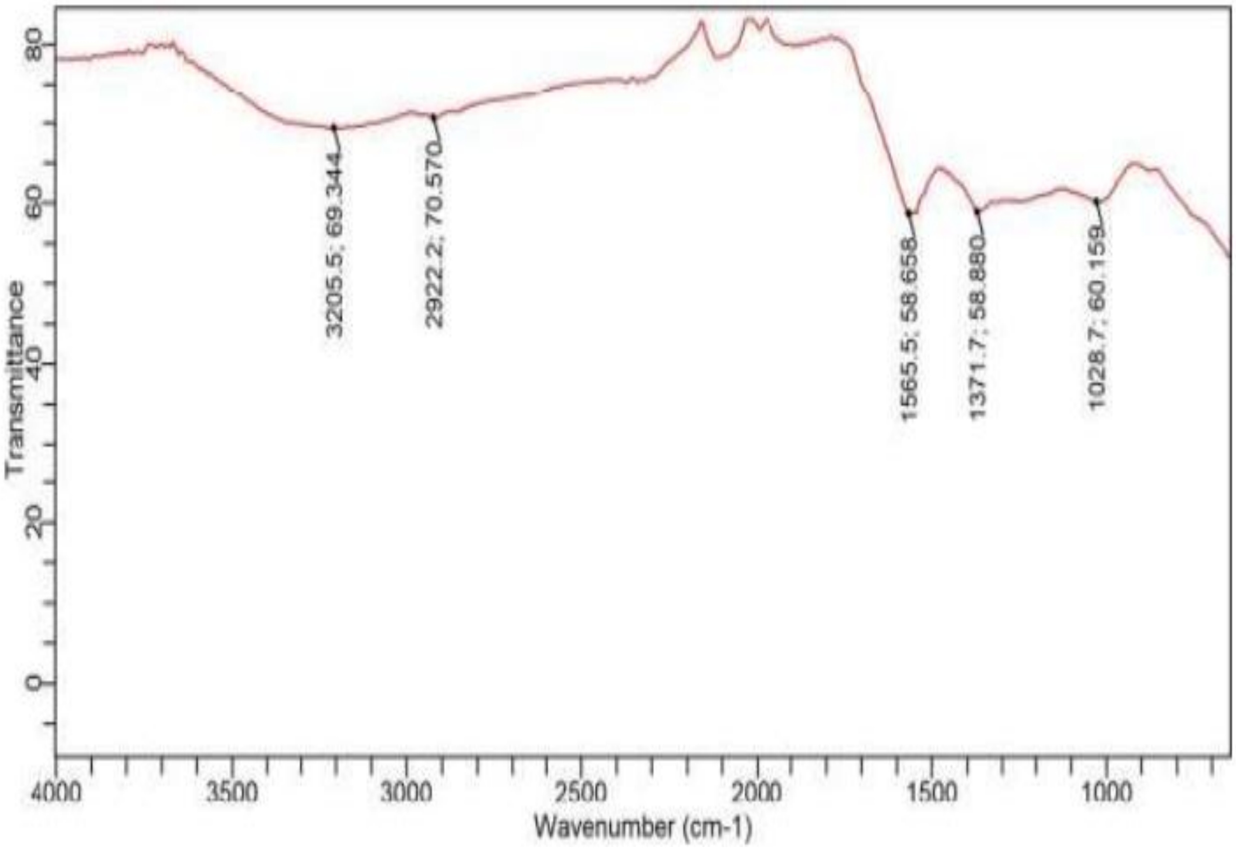

Fig. 1: FTIR spectrum of banana peel adsorbent

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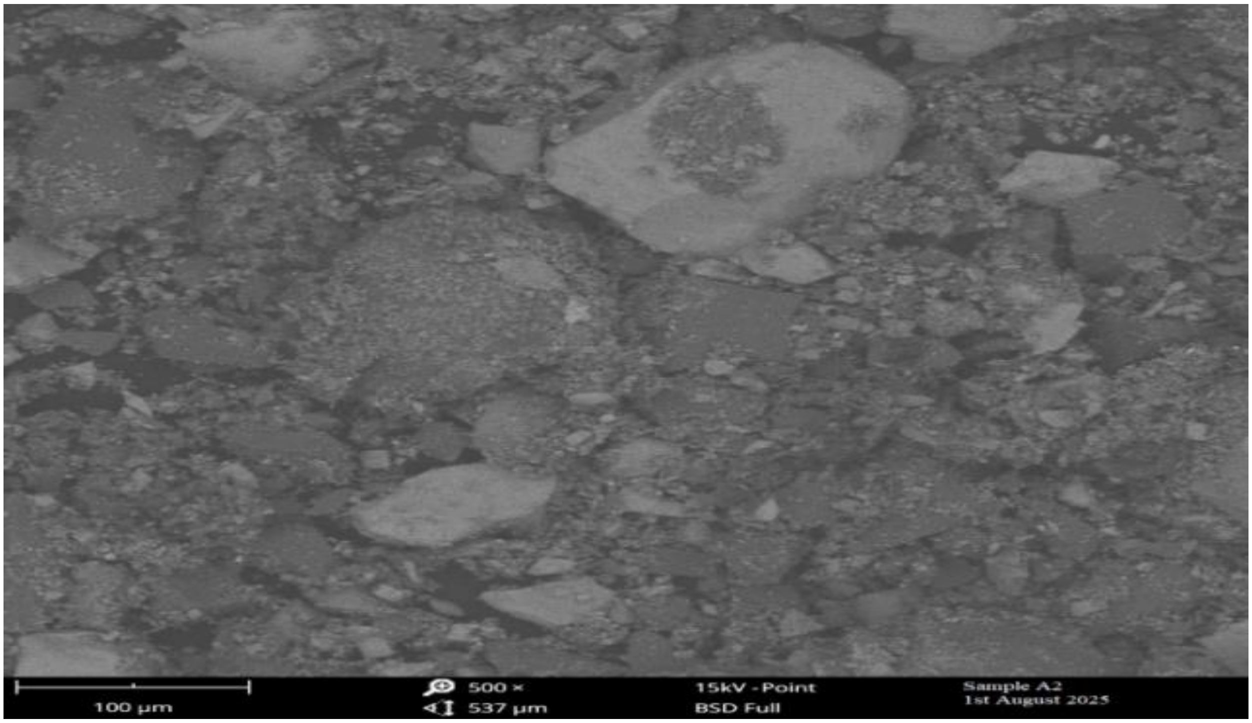

Fig. 2a: SEM micrograph at 500x

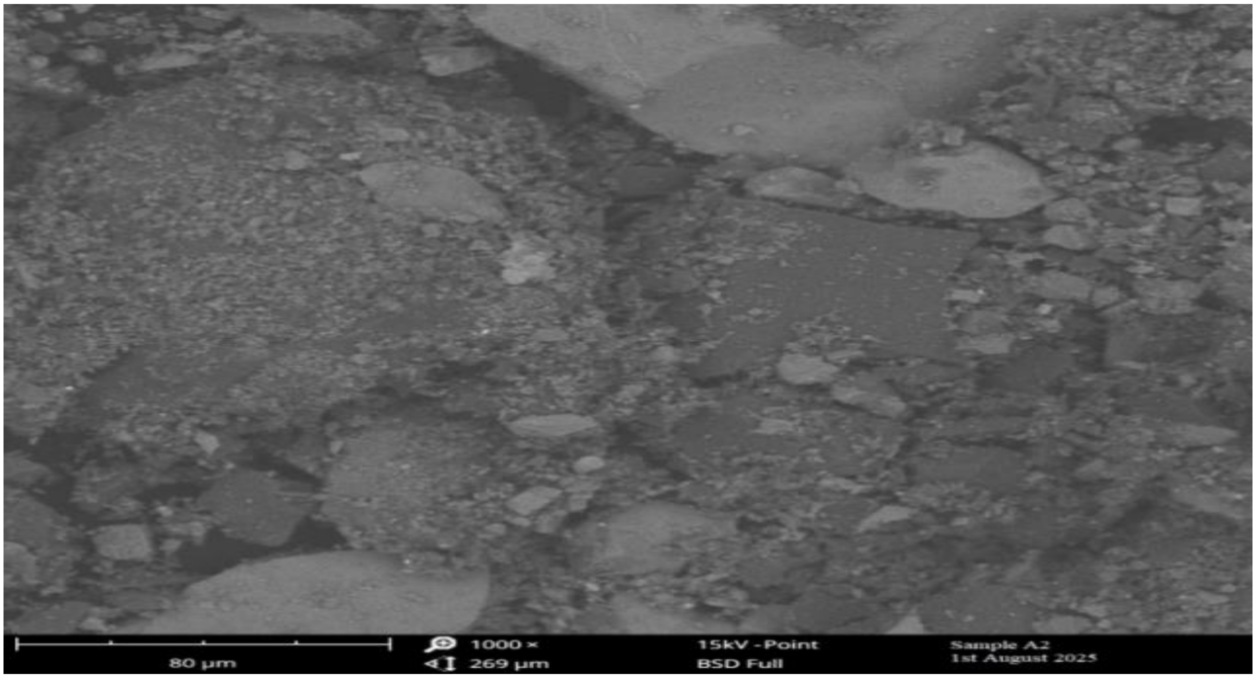

Fig. 2b: SEM micrograph at 1000x

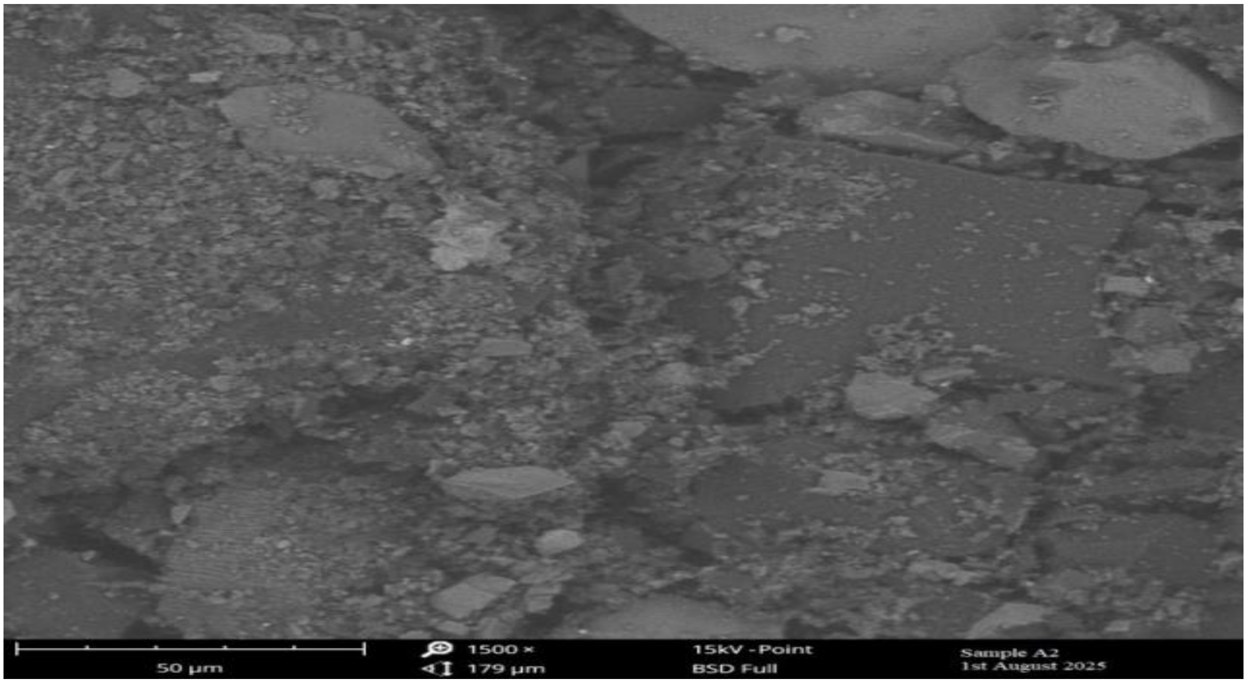

Fig. 2c: SEM micrograph at 1500x

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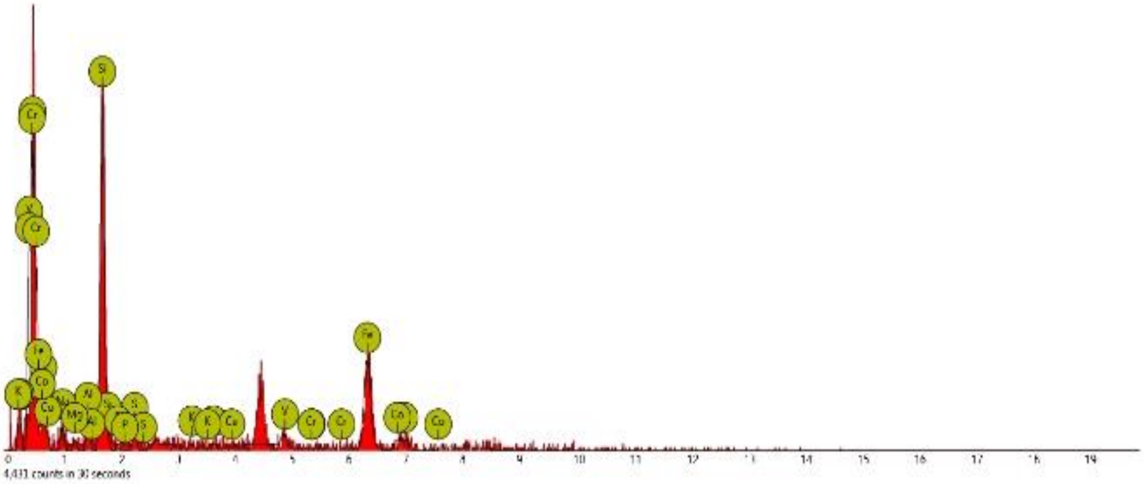

Fig. 2d: EDX spectrum of banana peel adsorbent

Table 2: Effect of pH on the percentage removal of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}

	Pb^{2+}		**Cu^{2+}**		**Fe^{2+}**		**Zn^{2+}**	
pH	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**
3	4.50 ± 0.06	78.24	1.45 ± 0.03	76.68	2.37 ± 0.06	14.59	0.68 ± 0.05	90.19
4	3.98 ± 0.03	80.76	1.37 ± 0.03	77.95	2.28 ± 0.03	17.76	0.57 ± 0.02	91.78
5	3.54 ± 0.05	82.88	1.22 ± 0.04	80.36	2.27 ± 0.04	18.19	0.53 ± 0.04	92.33
6	3.46 ± 0.04	83.27	1.14 ± 0.02	81.68	1.92 ± 0.02	30.73	0.50 ± 0.03	92.82
7	3.57 ± 0.03	82.74	1.28 ± 0.03	79.41	1.99 ± 0.05	28.24	0.73 ± 0.05	89.47

MC is mean concentration, R_E is percentage removal, Results are given as mean ± standard deviation (triplicate values).

Table 3: Effect of temperature on the percentage removal of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}

	Pb^{2+}		**Cu^{2+}**		**Fe^{2+}**		**Zn^{2+}**	
T (°C)	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**
30	0.21 ± 0.03	99.41	1.98 ± 0.05	68.22	0.30 ± 0.04	89.01	0.90 ± 0.04	86.94
40	0.09 ± 0.02	99.58	2.06 ± 0.03	66.95	0.28 ± 0.03	89.81	0.89 ± 0.06	87.12
50	0.01 ± 0.00	99.99	2.07 ± 0.04	66.67	0.17 ± 0.02	93.95	0.67 ± 0.05	90.34
60	0.05 ± 0.03	99.74	2.36 ± 0.04	62.07	0.26 ± 0.03	90.49	0.88 ± 0.04	87.25
70	0.08 ± 0.03	99.63	2.46 ± 0.04	60.51	0.29 ± 0.02	89.52	0.92 ± 0.03	86.64

T is temperature, MC is mean concentration, R_E is percentage removal, Results are given as mean ± standard deviation (triplicate values).

Table 4: Effect of adsorbent dosage on the percentage removal of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}

	Pb^{2+}		**Cu^{2+}**		**Fe^{2+}**		**Zn^{2+}**	
D(g)	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**
0.2	3.98 ± 0.05	80.74	1.37 ± 0.05	77.95	2.27 ± 0.05	18.19	0.57 ± 0.04	91.78
0.3	4.74 ± 0.04	77.08	1.71 ± 0.03	72.49	2.46 ± 0.04	11.28	0.77 ± 0.05	88.85
0.4	5.07 ± 0.03	75.51	1.67 ± 0.05	73.09	2.39 ± 0.05	13.76	0.96 ± 0.03	86.03
0.5	5.49 ± 0.05	73.45	1.84 ± 0.04	70.47	2.32 ± 0.04	16.57	1.03 ± 0.02	85.10
0.6	6.23 ± 0.03	69.85	2.06 ± 0.05	66.91	2.55 ± 0.02	8.03	1.25 ± 0.03	81.81
0.7	6.72 ± 0.04	67.51	2.41 ± 0.06	60.58	2.75 ± 0.04	1.04	1.65 ± 0.04	76.11

D is adsorbent dosage, MC is mean concentration, R_E is percentage removal, Results are given as mean ± standard deviation (triplicate values).

Table 5: Effect of contact time on the percentage removal of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}

	Pb^{2+}		Cu^{2+}		Fe^{2+}		Zn^{2+}	
t (min)	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**
15	5.46 ± 0.07	73.61	1.68 ± 0.05	72.95	2.54 ± 0.05	8.68	0.69 ± 0.04	89.93
30	5.28 ± 0.04	74.46	1.57 ± 0.04	74.73	2.44 ± 0.05	12.03	0.65 ± 0.03	90.56
45	4.32 ± 0.05	79.11	1.46 ± 0.04	76.60	2.47 ± 0.02	10.95	0.61 ± 0.02	91.09
60	4.26 ± 0.04	79.38	1.40 ± 0.05	77.50	2.07 ± 0.04	25.32	0.67 ± 0.04	90.31
75	3.56 ± 0.05	82.80	1.22 ± 0.03	80.36	1.92 ± 0.03	30.73	0.58 ± 0.03	91.64

t is contact time, MC is mean concentration, R_E is percentage removal, Results are given as mean ± standard deviation (triplicate values).

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Table 6: Effect of initial metal concentration on the percentage removal of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+}

	Pb^{2+}		**Cu^{2+}**		**Fe^{2+}**		**Zn^{2+}**	
C (mg/L)	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**	**MC (mg/L)**	**% R_E**
20	2.09 ± 0.03	63.76	0.55 ± 0.03	55.96	1.15 ± 0.04	7.86	0.56 ± 0.03	63.83
40	3.74 ± 0.04	63.92	0.81 ± 0.04	54.22	1.25 ± 0.03	9.91	0.74 ± 0.04	74.04
60	4.03 ± 0.05	70.71	1.30 ± 0.05	67.57	1.53 ± 0.04	14.14	1.06 ± 0.02	77.50
80	5.35 ± 0.04	66.83	1.42 ± 0.03	73.21	1.84 ± 0.03	8.13	0.94 ± 0.03	84.74
100	3.98 ± 0.06	80.74	1.37 ± 0.04	77.95	1.92 ± 0.05	30.73	0.71 ± 0.04	89.76
120	5.67 ± 0.03	81.53	1.86 ± 0.05	80.04	2.70 ± 0.04	21.18	1.21 ± 0.03	86.43

C is initial metal concentration, MC is mean concentration, R_E is percentage removal, Results are given as mean ± standard deviation (triplicate values).

Discussion

The pH value was 6.70 ± 0.15. The near-neutral pH is advantageous for many adsorption applications as it minimizes interference with the target pollutants. It has been reported that for most applications, pH of 6-8 is considered acceptable (Ekpete et al., 2017). This is also in tandem with the results obtained by Akpomie and Conradie (2020) who presented that pH of banana peel was between 5.0 and 7.0. The moisture content was 9.24 ± 1.02%. Carbonization, a high-temperature process, effectively removes moisture, which is a common finding in similar studies (Kabenge et al., 2018). The lower moisture content makes the carbonized material more stable and efficient, as water molecules do not compete for active adsorption sites. The ash content was 5.41 ± 0.31%. These values are in agreement with those reported by Ekpete et al. (2017) in the range of 1 - 20 % and also with that by Romello et al. (2016) who presented that ash content of 12.45 ± 0.38 %. While carbonization often leads to an increase in ash percentage due to the concentration of inorganic material, a decrease can occur if some volatile inorganic compounds are also released during the process.

The volatile matter was 12.53 ± 1.18%. Volatile matter consists of organic compounds that are driven off during pyrolysis and these compounds emitted are hydrocarbons such as aldehydes, alcohols, ketones, esters (Rosenkranz & Schnitzler, 2016). The percentage of volatile matter in banana peel is due to its organic nature comprising lipids, proteins and carbohydrates (Yunus et al., 2020). The volatile matter indicates that the carbonization process was successful in creating a more concentrated carbon structure, which is the primary component of an effective adsorbent (Vieira et al., 2014).

The bulk density is another important parameter especially when the adsorbent is intended for filtration applications. This is because it determines the mass of carbon that can be contained in a filter of given solids capacity and the quantity of treated liquid that can be retained by the filter cake (Ekpete et al., 2017). The bulk density of the adsorbent was 0.15 ± 0.02g/mL which is a critical result of carbonization. The process created a highly porous, lightweight structure by removing mass and creating voids, which is essential for increasing the surface area for adsorption (Chafidz et al., 2018). Pathak et al. (2017) and Yunus et al. (2020) found that the bulk density of banana peel was 0.39 g/mL and 0.56 g/mL respectively and these low values were good for the adsorption process.

The iodine number is a measure of the adsorbent's surface area, particularly its micropore volume which also represents the milligram of iodine adsorbed by one gram of carbon (Akter et al., 2021). From the study, the iodine number obtained was 654.10 ± 0.01 mg/g. In a study reported by Napitupulu et al. (2019) using *Musa paradisiaca formatypica*, iodine number for banana peel carbonized at 400°C for 30 mins, 450°C for 10 mins and 500°C for 10 mins was 994.05 mg/g, 1006.74 mg/g and 998.28 mg/g. This variation is attributed to the fact that iodine number of banana peel varies from species to species due to differences in chemical composition, lignin, cellulose, and surface properties among cultivars (Emaga et al., 2007; Mohapatra et al., 2010).

Research on banana peels by Kabenge et al. (2018) demonstrates a decrease in moisture content and volatile matter, while the ash content and fixed carbon content increased. This confirms that the process effectively converts the raw biomass into a carbon-rich material. Also, the lower bulk density and volatile matter, directly correlate with enhanced adsorption capacity (Khairiah et al., 2021). This is supported by numerous studies on the use of banana peel as well as other adsorbents for pollutant removal as it has shown its effectiveness in removing heavy metals like lead and cadmium from wastewater due to its high surface area and porous structure (Vieira et al., 2014). Romello et al. (2016) reported that the ash content for pineapple and mango was 4.39 ± 0.14 % and 3.24 ± 0.18 % respectively while Yunus et al. (2020) obtained bulk content for pineapple peel and melon peel as 0.33 ± 0.01 g/mL and 0.64 ± 0.02 g/mL respectively. Also, Pathak et al. (2017) reported the bulk density of pineapple peel to be 0.52 g/mL. Yunus et al. (2020) presented moisture content, ash content and volatile matter for pineapple peel and melon peel as 84.86 ± 0.46 % and 68.26 ± 3.06 %, 0.93 ± 0.30 % and 2.06 ± 0.30 %, 14.46 ± 0.57 % and 24.86 ± 1.62 % respectively.

The biosorbent was characterized using the FTIR technique. The spectrum was obtained to recognize the functional groups on its surface as the chemical activation with nitric acid introduced oxygen-containing functional groups, which are crucial for applications like adsorption (Zamri et al., 2019). Fig 1 shows the FTIR spectrum for the prepared biosorbent which was characterized by five (5) distinct peaks covering not only the analytical region (> 1500 cm^{-1}) but also the fingerprint region (< 1500 cm^{-1}) typical of a lignocellulosic material. The peaks are 1028

cm^{-1} at 60, 1371 cm^{-1}at 58, 1565 cm^{-1} at 58, 2922 cm^{-1} at 70 and 3205 cm^{-1} at 69. The peak at 1028 cm^{-1} is assigned to the presence of C-O or C-O-C stretching peak of an ether group. The peak at 1371 cm^{-1} corresponded to C-H bending vibrations. The peak at 1565 cm^{-1} is indicative of the presence of C=C stretching of alkenes, aromatic rings or amino acids, which are present in lignin (Annadurai et al., 2003). This was similarly reported by Bhaumik and Mondal (2014), and Deshmukh et al. (2017). The presence of this group is normal and often enhanced by acid treatment. The peak at 2922 cm^{-1} is attributed to the C-H, $-CH_2$ or $-CH_3$ stretching vibration of aliphatic chains, showing a significant presence of hydrocarbon components. Similar occurrence was observed and reported by Zamri et al. (2019). The last peak at 3205 cm^{-1} was characteristic of the O-H stretching vibration of hydroxyl groups, including those from alcohols, phenols, and carboxylic acids, which are abundant in the cellulose, hemicellulose, and lignin components (Mondal & Kar, 2018). The high intensity of this peak (69) indicates a large presence of these functional groups.

SEM micrographs coupled with EDX were employed to give insights into the morphology and elemental composition of the biosorbent as shown in Fig 2a, 2b, 2c and 2d. Fig. 2a, 2b and 2c show the morphology at magnification of 500x, 1000x and 1500x which revealed a rough, heterogeneous, and disordered surface morphology. The images also showed particles of varying sizes and shapes, with some exhibiting sharp edges and a semi-regular, heterogeneous morphology similar to that reported by Nadew et al. (2023). The carbonization process, which involved high-temperature pyrolysis, created a highly developed, porous carbon structure with well-defined pores and a more organized morphology (Dermawan et al., 2025). The surface appeared as an orderly, honeycomb-like or crater-like surface, indicating the formation of activated carbon (Hussain et al., 2023). The subsequent nitric acid activation provides this porous structure to a much higher specific surface area and a greater number of active sites (Al-Sareji et al., 2024). The surface of the biosorbent is a clear example of the successful conversion of the biomass into a

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functional, highly porous material, which is ideal for applications requiring high adsorption capacity, such as water purification and gas separation (Hussain et al., 2023).

The energy dispersive x-ray (EDX or EDS) spectrum of the adsorbent is shown in Fig 2d. The main elements are carbon (C) and oxygen (O), which are the fundamental components of the lignocellulosic biomass (Al-Sareji et al., 2024). Additionally, the spectrum reveals a significant presence of inorganic elements such as potassium (K), and smaller amounts of silicon (Si), magnesium (Mg), and calcium (Ca) (Hussain et al., 2023). The presence of these elements is characteristic of natural plant-based materials. The nitric acid activation step, while creating a porous surface, does not remove the inherent mineral content, which is why these elements were present in the final product (Shrestha, 2016). The spectrum is dominated by a higher percentage of carbon (C) in comparison to other elements. This agrees with the study presented by Basirun et al. (2023). This is because the carbonization process (pyrolysis) at high temperatures burned most of the volatile organic compounds and other impurities, leaving behind a carbon-rich biochar (Hussain et al., 2023). The high-temperature treatment also reduced or eliminated many of the metallic and non-metallic impurities, resulting in a cleaner product (Hussain et al., 2023). The significant ratio of carbon to oxygen was a key indicator of successful biochar production and a desirable characteristic for materials used in applications like adsorption (Basirun et al., 2023).

Tables 2–6 shows the effect of pH, temperature, dosage, contact time and initial metal concentration on the adsorption of Pb^{2+}, Cu^{2+}, Fe^{2+} and Zn^{2+} by the adsorbent. The pH had a pronounced influence on the removal efficiency and adsorption capacity of nitric-acid-activated carbonized banana peel. For all metals studied (Pb^{2+}, Cu^{2+}, Fe^{2+}, and Zn^{2+}), removal efficiencies generally increased as pH increased from 3 to 6 as shown in Table 2, reaching maximum values at pH 6, after which a slight decline or plateau was observed at pH 7. The improvement with increasing pH is attributed to reduced competition between H^+ ions and metal cations for adsorption sites, as well as increased deprotonation of surface functional groups (–COOH, –OH), which enhances electrostatic attraction and complexation. Similar pH-dependent trends with optimum adsorption near neutral pH have been reported for banana peel and chemically modified banana peel adsorbents in heavy-metal removal studies (Annadurai et al., 2003; Hossain et al., 2012; Nathan et al., 2021).

The effect of temperature on biosorption is shown in Table 3. Temperature variation affected the adsorption performance differently for the metals. Pb^{2+} and Fe^{2+} exhibited maximum removal efficiencies at 50 °C, suggesting enhanced diffusion of metal ions into the pores and increased interaction with active sites at moderate temperatures. In contrast, Cu^{2+} and Zn^{2+} showed a gradual decrease in removal efficiency with increasing temperature, indicating possible exothermic adsorption behavior. These observations suggest that adsorption on nitric-activated banana peel is largely physical with some chemisorption contribution, where excessive thermal energy may weaken adsorbate–adsorbent interactions. Similar mixed temperature responses have been reported for banana peel–derived carbons and biosorbents (Abdullah et al., 2019; Ahsan et al., 2022).

The amount of adsorbent is an important parameter that affects the adsorption process.. The efficiency of metal removal is significantly influenced by the amount of adsorbent used (Hossain et al., 2012). Increasing the adsorbent dosage from 0.2 g to 0.7 g resulted in a consistent decrease in percentage removal efficiency for all metals as shown in Table 4, although the total number of available adsorption sites increased. The reduction in efficiency at higher dosages can be attributed to particle aggregation, overlap of active sites, and reduced effective surface area, as well as a lower adsorbate-to-adsorbent ratio under fixed metal concentration and volume. Similar trends have been observed in banana peel adsorption systems where an optimum low dosage yields higher percentage removal efficiency per unit mass (Nathan et al., 2021).

Contact time is another important factor in the batch adsorption process. Effect of contact time on adsorption was studied and the results are given in Table 5. Removal efficiency increased with increasing contact time for all metals, indicating time-dependent adsorption kinetics. Rapid adsorption occurred at shorter contact times due to the availability of abundant active sites, followed by a slower uptake phase as equilibrium was approached. Maximum or near-equilibrium removal was observed between 60 and 75 minutes, beyond which further increases in contact time produced marginal improvements. This behavior is consistent with banana peel-based adsorbents reported in the

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literature, where equilibrium is typically attained within 60–90 minutes depending on metal type and surface modification (Annadurai et al., 2003; Hossain et al., 2012).

As shown in Table 6, initial metal concentration influenced both removal efficiency and adsorption capacity by altering the mass-transfer driving force. For Pb^{2+}, Cu^{2+} and Zn^{2+}, percentage removal generally increased with increasing concentration up to an optimum range (100–120 mg/L), indicating enhanced interaction between metal ions and available adsorption sites. Beyond this range, fluctuations or slight decreases suggest surface saturation and limited binding sites. Fe^{2+} showed comparatively lower removal across concentrations, reflecting weaker affinity or competitive effects. Similar concentration-dependent behavior has been widely reported for banana peel adsorbents, where higher concentrations enhance adsorption capacity until site saturation occurs (Abdullah et al., 2019; Nathan et al., 2021).

At optimum conditions, the overall trend for the percentage removal of the metal ions with respect to pH, adsorbent dosage, contact time and initial metal concentration was $Zn^{2+} > Pb^{2+} > Cu^{2+} > Fe^{2+}$ while at optimum temperature, it was $Pb^{2+} > Fe^{2+} > Zn^{2+} > Cu^{2+}$.The optimum conditions for each metal analyzed in this study agree with the findings of Ali and Saeed (2015), Ince et al. (2016), and Li et al. (2016) but contrasted with that obtained by Hossain et al. (2012), and Ashfaq et al. (2021). Statistical analysis of the physicochemical properties of the adsorbent and the experimental data obtained from the batch adsorption studies using ANOVA revealed statistical differences at 95% confidence limit ($p \leq 0.05$).

Conclusion

This research showed that activated banana peel is an effective adsorbent for Pb^{2+}, Cu^{2+}, Fe^{2+}, and Zn^{2+} removal from aqueous solutions. It was observed that the uptake of the selected heavy metal ions by the activated banana peel increases with increasing ph, temperature, contact time and initial metal ion concentration with optimum conditions at 0.2g adsorbent, 100 mg/L initial metal concentration, 75 min time, 50°C room temperature at pH 6. It is however concluded that powder of banana peel has successful application as an adsorbent and shows high efficiency for the removal of Pb^{2+}, Cu^{2+}, Fe^{2+}, and Zn^{2+} from aqueous solution.

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