

Raffia Palm Seed Shell-Derived Carbon for the Removal of Microplastics, Cadmium (Cd), and Copper (Cu) from Polluted Surface Water

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The presence of microplastics and metals in drinking water constitute human health risk. The objective of this study is to remove plastics through filter bed made from raffia palm seed shell and carry out adsorption study to remove Cd²⁺ and Cu²⁺ from simulated waste water. The water was subjected to Gas Chromatographic mass spectrometry (GC/MS) method of analysis before and after filtration while activated carbon derived from raffia palm seed shell was used to adsorb the metals. Fourier transformed infrared spectrometric (FTIR) analysis and Scan electron spectroscopy (SEM) were used to characterize the adsorbent before and after the experiment. The results showed that traces of degraded microplastics was recorded with a marginal decrease of 0.03% after filtration when the retention time recorded was compared with values in literature. The results of adsorption experiment showed that the maximum adsorption (qm) for Cd²⁺ and Cu²⁺ are 45.1mgL⁻¹ and 202 mgL⁻¹ respectively for Langmuir isotherm while r² of 0.266 and 0.908 were recorded respectively for Freundlich isotherm. The FTIR showed broad O-H peak recorded at 3675.2 cm⁻¹, and carbonyl (-C=O-) at 1617.7 cm⁻¹ before adsorption while after adsorption, small none- intense peaks were recorded. Variation of pore sizes were recorded with SEM analysis. The raffia palm seed shell carbon is effective for the adsorption of Cd and Cu but not very good for the removal of plastics.

Keywords: Adsorption, filtration, metals, microplastics

Introduction

The use of plastics in packaging, construction, automotive, electronics, healthcare, and consumer goods is due to its durability, versatility, lightweight, chemical resistance, and electrical insulation properties (Hossain *et al.*, 2021). Consequently, production of plastics will continue due to high demand. Microplastics are plastic particles with less than 5 mm size, which originated from the degradation of larger plastic debris (Thompson *et al.*, 2004). They infiltrate various ecosystems such as air, water and soil causing threats to terrestrial and aquatic life (Cole *et al.*, 2011). Microplastics consists of unpolymerized monomers, chemical additives, and some miscellaneous substances (Alijagic *et al.*, 2024). Plastics are durable and resistant to degradation, leading to their accumulation in the environment. Improper waste management and plastic pollution have raised concerns about their impact on human life, ecosystems, and health (Sacco *et al.*, 2023). The ingestion of microplastics can accumulate in tissues of organisms, transfer through food chain, cause harm which can affects the physiology and behaviour of higher organisms. It can also act as a carrier of pathogens and harmful chemicals (Zhang *et al.*, 2022). Microplastics can settle in sediments, affecting benthic organisms and change the sediment composition (Li *et al.*, 2023). The source of microplastics in water is through atmospheric deposition, fragmentation of large plastics, microbeads in personal care products, synthetic fibre from textiles, and those carried by erosion from improper wastes disposal.

The contamination of water bodies by heavy metals has emerged as a global concern due to their toxicity, persistence in the environment, and tendency to accumulate in the food

chain (Zhou *et al.*, 2021). Metals such as Cadmium (Cd), Copper (Cu), manganese (Mn) and Chromium (Cr) are among the contaminants reported in waste water which occur as a result of electroplating, mining, tanning, fertilizer production, and steel manufacturing process (Al-Ghouti *et al.*, 2020). Some metals are essential micronutrient while others are carcinogenic. In many developing countries, including Nigeria, water treatment infrastructure is often inadequate, leaving communities vulnerable to unsafe concentrations of heavy metals in their drinking water sources (Isukuru *et al.*, 2024).

Several water treatment methods such as ion exchange, chemical precipitation, membrane filtration, and electro-chemical treatment are effective, but they are expensive (Sanjay *et al.*, 2025). Activated carbon (AC), has been widely used due to its large surface area, high porosity, and chemical stability. However, the high cost of commercial activated carbon has prompted researchers to explore low-cost, renewable, and locally sourced adsorbent (Ghogomu *et al.*, 2016). The present study explored the use of raffia palm seed shell as a filter and as adsorbent for the removal of microplastics and metals from water.

Materials and Methods

Sampling area: The coverage of the area where water samples were collected were Ore and Igbokoda, Ondo South, Ondo State. Igbokoda is located in Ilaje local government between Latitude 6° 21' 9"N Longitude 4° 48' 14"E and Latitude 6° 22' 10"N Longitude 4° 32' 25"E while Ore is located in Odigbo Local government between Latitude 6° 44' 49"N Longitude 4° 52' 33"E and Latitude 6° 45' 10"N Longitude 4° 55' 61"E. All reagents are from Sigma Aldrich

representative in Lagos, Nigeria. The study consists of two parts, namely filtration of water to remove microplastics from surface water collected from the locality named above and adsorption experiments to remove Cd and Cu from simulated waste water.

Collection of samples: Twenty (20) water samples meant for physicochemical parameter determination were collected into plastics and kept in the ice chest and analysed in the laboratory. However, water meant for plastic extraction were kept in glass bottle. All labile parameters (pH, temperature, dissolved oxygen (DO), electrical conductivity (EC)) were determined on site while others were analysed immediately after arrival at the laboratory. Raffia palm seed was collected from Okitipupa and was identified by an expert in the department of Crop, soil and pest (CSP) of Olusegun Agagu University of Science and Technology, Okitipupa, Ondo State, Nigeria.

Adsorbent preparation or carbonization of the raffia palm seed shell: The raffia palm seed shell was washed in distilled water, air dried before it was activated by soaking in 0.1M HNO₃ solution for 1hr. The activated raffia seed shell was transferred into an oven at the temperature of 300°C for 1hr. It was cooled and ground to a size of 0.4mm.

Adsorption Experiment: Batch adsorption experiments were carried out to evaluate adsorption of Cu²⁺ and Cd²⁺ metal ions in solution by carbon produced from raffia palm seed shell. Parameters varied are, mass of adsorbent, concentration of metals, time and pH as described in previous studies (Ayedun *et al.*, 2021).

Percentage adsorbed was calculated with the equation
$$PA = (C_i - C_f) / C_i \times 100 \text{ ----- (1)}$$

Where C_i is the initial Cd concentration before adsorption, C_f is the final Cd concentration after adsorption and PA is the % adsorbed.

The sorption capacity was determined by
$$Q_e = C_i - C_f / m \times V \text{ -----2}$$

Where q_e is sorption capacity (mg g⁻¹); V is the volume of solution (L); m is the mass of material (g), and C_i and C_f are the initial and final (equilibrium) concentrations (mg L⁻¹). Langmuir and Freundlich Isotherms was used to evaluates the extent of adsorption.

Physicochemical analysis: The pH, temperature, dissolved oxygen (DO), and electrical conductivity (EC) were determined using portable meter on site. The alkalinity was determined by titration, Chloride concentration were determined by Mohr's method. Salicylate method was used for nitrate determination, phosphomolybdate method was used for phosphate determination while colorimetric method was used for both sulphates and ammonium determination.

Microplastics analysis: Extraction of microplastics was carried out using the modified method of Ziajahromi *et al.* (2017) and used by Iskandarani *et al.* (2025). Hydrogen peroxide (20mL) was added to 200mL of water samples and

were digested at 60 °C on a heated block. It was stirred with magnetic stirrer at 60 rpm, as digestion progresses, H₂O₂ was added after 10 minutes until the volume reduced to 50mL. The temperature was maintained to avoid degradation of polymer. A cellulose acetate filter (2500 nm, 90 mm diameter) which retain microplastics but excluded smaller size particles was used to filter the water.

Filtration setup: Three compartment were set up as a filter with the stones (2.5mm) in the first compartment. The second compartment contained sand (1.5mm) while the third compartment contained granulated raffia seed powder (1.5 mm thick). The final water sample was collected in the 4th compartment. A steady flowrate of 50 ml per minutes were set up.

Characterization (GC–MS, SEM, FTIR)

Gas Chromatographic (GC-MS Shimadzu model) was used to analyse the water for plastics traces before and after filtration using raffia palm seed shell carbon. The injection temperature was 240°C while the column temperature range between 50°C to 175°C. The column size was 30m length, internal diameter of 0.35mm, thickness of 0.250 μm while total elution time was 45minutes. MS scanning was carried out between 85 to 380m/z.

Fourier Transformed Infrared (FTIR Agilent model) analysis was used to identify the functional groups present on the surface of the raffia palm residue and its activated carbon. The analysis helps to determine changes in chemical bonding that may occur during the activation process, which are essential for metal ion adsorption. The samples were finely ground and mixed with potassium bromide (KBr) to form pellets. The pellets were then scanned over a range of wavelengths (4000–400 cm⁻¹). Scanning Electron Microscopy (SEM Thermo Fisher model) was used to examine the surface morphology and porosity of the raffia palm residue before and after adsorption studies. This technique provides high-resolution images that reveal the texture, pore structure, and surface irregularities of the material. The samples were examined under the SEM operating at an accelerating voltage of 2–10 kV. Atomic Absorption Spectrophotometer (AAS Bulk Scientific model) was used to determine metal concentration before and after adsorption experiments with holo-cathode lamp of Cd and Cu.

Data analysis: Mean and standard deviation were used to evaluate the data generated before and after filtration.

Results and Discussion

The temperature of the water before filtration ranged between 29.1 °C to 30.1 °C with the mean of 29.3±0.3 °C while the ranged between 28.20 °C to 31.1 °C with the mean of 28.4± 1.2 °C (Table1). Temperature between 10 -15 °C is the most suitable for drinking (Omer, 2019). Temperature affects other parameters such as viscosity, solubility, odour, and dissolved oxygen in water (Syed *et al.*, 2023). The pH of the water before filtration ranged between 6.51 to 7.26

with the mean of 6.91 ± 0.03 while the range after filtration was between 6.51 to 7.08 with the mean of 6.85 ± 0.62 . The dissolve oxygen (DO) before filtration ranged between 6.80 mgL⁻¹ to 9.30 mgL⁻¹ with the mean of 7.15 ± 0.25 mgL⁻¹ while the range after filtration was between 7.10 mgL⁻¹ to 9.10 mgL⁻¹ with the mean of 7.12 ± 0.35 mgL⁻¹. The DO varied as a result of handling of water and it depends on temperature and pressure which has no negative consequence on human health.

The Total Dissolved Solid (TDS) of the water before filtration ranged between 70.0 mg L⁻¹ to 1020 mg L⁻¹ with

the mean of 270 ± 10 mgL⁻¹ while the range after filtration was between 20.0 mg L⁻¹ to 210 mg L⁻¹ with the mean of 60.0 ± 7.1 mg L⁻¹. Similarly, Conductivity of water samples before filtration ranged between 140.2 μScm⁻¹ to 2030 μScm⁻¹ with the mean of 510 ± 15 μScm⁻¹ while the range after filtration was between 40.6 μScm⁻¹ to 320 μScm⁻¹ with the mean of 120.0 ± 06 μScm⁻¹. The % TDS and Conductivity removed are 77% and 76% respectively. Industrial effluents are the major source of EC and TDS in surface water (Rahman *et al.*, 2021).

Table 1: Range of physicochemical parameters in water before and after filtration (N = 20)

Parameters	Before Filtration			After Filtration				
	Minimum	Maximum	Mean ± SD	Minimum	Maximum	Mean ± SD	% removed	WHO 2017 limit
Temperature (°C)	29.1	30.1	29.3±0.3	28.20	31.1	28.4±1.2	3	25
pH	6.51	7.26	6.91±0.03	6.51	7.08	6.85±0.62	1.6	6.5-8.5
DO (mg L ⁻¹)	6.80	9.30	7.15±0.25	7.10	9.10	7.12±0.35	3	5
TDS (mg L ⁻¹)	70.0	1020	270±10	20.0	210	60±7.1	77	500
Conductivity(μScm ⁻¹)	140.2	2030	510±15	40.6	320	120±06	76	250
Alkalinity (mg L ⁻¹)	40.0	420	100±5	30.0	300.0	70±20	30	120
Chloride (mg L ⁻¹)	8.29	607.74	132±21	6.4	522	127±21	3.8	250
NO ₃ ⁻ (mg L ⁻¹)	0.07	1.2	0.284±0.383	0.03	0.92	0.199±2.0	28.9	10
PO ₃ ²⁻ (mg L ⁻¹)	0.119	2.59	0.5±0.14	0.071	1.50	0.38±0.55	31.6	NA
SO ₄ ²⁻ (mg L ⁻¹)	0.038	5.29	0.98±0.19	0.003	4.62	0.94±0.16	4	100
NH ₄ ⁺ (mg L ⁻¹)	0.01	0.298	0.067±0.09	0.001	0.13	0.057±0.04	14.9	0.5

Key: NA-Not Available

The Chloride concentration of water before filtration ranged between 8.29 mg L⁻¹ to 607.74 mg L⁻¹ with the mean of 132 ± 21 mgL⁻¹ while the range after filtration was between 6.4 mg L⁻¹ to 522 mg L⁻¹ with the mean of 127 ± 21 mg L⁻¹. Fifty percent (50%) of the water samples has chloride concentration greater than 250 mgL⁻¹ recommended limit while 3.8% of chloride was removed by the filter made up of raffia palm seed shell. The sources of chloride in surface water indicate possible water interaction with chloride containing rock, waste water contamination and agricultural run-off (Chatterjee, 1996). The nitrates concentration of water before filtration ranged between 0.07 mg L⁻¹ to 1.2 mg L⁻¹ with the mean of 0.28 ± 0.383 mgL⁻¹ while the range after filtration was between 0.03 mg L⁻¹ to 0.92 mg L⁻¹ with the mean of 0.199 ± 2.0 mg L⁻¹. The nitrate nitrogen of the raw water does not pose any risk to human health due to its concentration of less than 10 mg L⁻¹. However, the filter under consideration removed 28.9% of nitrates. The phosphates concentration of water before filtration ranged between 0.119 mg L⁻¹ to 2.59 mg L⁻¹ with the mean of 0.5 ± 0.14 mgL⁻¹ while the range after filtration was between

0.071 mg L⁻¹ to 1.50 mg L⁻¹ with the mean of 0.38 ± 0.55 mg L⁻¹. The sulphates concentration of water before filtration ranged between 0.038 mg L⁻¹ to 5.29 mg L⁻¹ with the mean of 0.98 ± 0.19 mgL⁻¹ while the range after filtration was between 0.003 mg L⁻¹ to 4.62 mg L⁻¹ with the mean of 0.94 ± 0.16 mg L⁻¹. The concentration of sulphates below WHO limit obtained in this study is in agreement with published results obtained from Angereb reservoirs, Gondar, Ethiopia (Zewdu *et al.*, 2024). Four percent (4%) of the sulphates was removed by the filter. Agricultural wastes discharge as a results of fertilizer application on soils is the possible source of sulphates in the water. The ammonium concentration of water before filtration ranged between 0.01 mg L⁻¹ to 0.298 mg L⁻¹ with the mean of 0.067 ± 0.09 mgL⁻¹ while the range after filtration was between 0.001 mg L⁻¹ to 0.13 mg L⁻¹ with the mean of 0.057 ± 0.04 mg L⁻¹. The raffia palm seed shell filter removed 14.9% of ammonium ions.

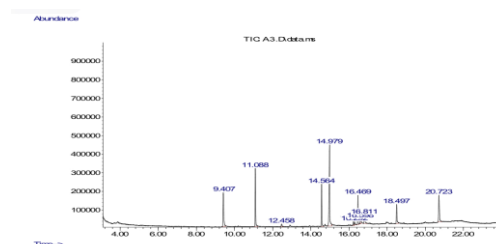


Figure1: GC MS chromatogram of water sample before filtration

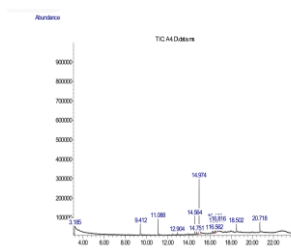


Figure 2: GC MS chromatogram of water sample after filtration

Table 2: GC-MS results of water samples before and after filtration

Present Study		Literature (Zuri <i>et al.</i> , 2025)		
Rt (before filtration)	Rt (after filtration)	Rt (Zuri <i>et al.</i> , 2025)	compound	Microplastic (concentration range μg)
14.979	14.974	13.45	2,4,6- Triphenyl-1-hexene	PS (5-50)
12.458	12.904	12.28	Bisphenol A	PC(5-30)
11.088	11.088	11.97	1,20 Heneicosadiene	HDPE (5-40)
9.407	9.412	9.45	2-Diphenylmethanone	PVC (5-40)

The retention time (Rt) recorded on GC-MS analysis of the water sample before and after passing it over a bed of raffia palm seed shell was compared with results obtained in Literature (Zuri *et al.*, 2025). The retention time very close to those exhibited by 2,4,6-Triphenyl-1- hexene was recorded with a marginal decrease (0.03%) after filtration (Table 2). The Rt corresponding to Bisphenol A (Rt = 12.904) after filtration was higher than values recorded before filtration (Rt = 12.458). Similarly, Rt corresponding to 2-Diphenylmethanone (Rt = 9.412) after filtration was higher than values recorded before filtration (Rt = 9.407). All these pointed at possibility of contamination of the filter bed or outright non-suitability of the filter bed of raffia palm seed shell to remove microplastics from water.

Adsorption studies

Effect of initial concentration

The concentrations of Cu^{2+} and Cd^{2+} used are 20, 50, 75, 100, and 250 ppm with 1g of the adsorbent. The higher the initial concentration of metals, the higher the % metal adsorbed (Figure 3). Cd^{2+} is more adsorbed than Cu^{2+} , this occur due to differences in ionic radius. This indicates that the adsorbent made from raffia palm seed shell carbon possesses abundant active sites capable of binding Cd^{2+} and Cu^{2+} ions. The least % adsorbed for Cd^{2+} and Cu^{2+} are 96.3% and 92.05% respectively while the highest adsorbed are 99.2% and 98.9% respectively.

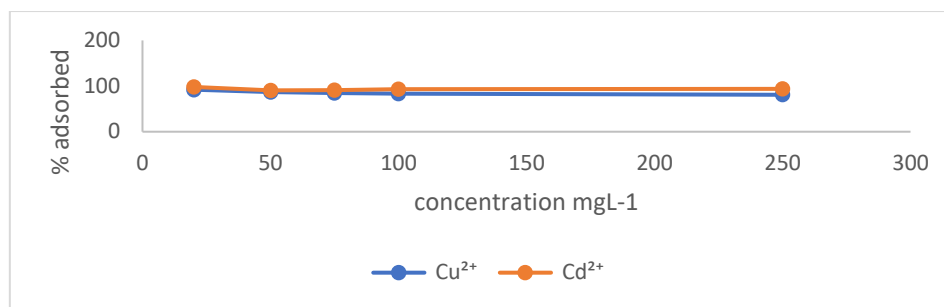


Figure3: percentage of metal adsorbed at various concentration

Effect of pH

The % Cd adsorbed over a pH range of 2 to 10 was investigated and was found to be high at pH 2 (90% adsorbed) while it decreases rapidly after pH 7 (Figure 4). This may be attributed to the fact that surface properties can vary significantly among plastics, and metals present in the water as a result of structural and compositional differences (Tenea *et al.*, 2024).

However, the % Cu²⁺ adsorbed increases toward neutral pH. This suggests the potential formation of copper hydroxide compounds by the adsorbent at pH 7. The decrease in Cu²⁺ concentration on the surfaces of raffia seed carbon adsorbent occur as a result of deprotonation of the Cu hydroxides at the surface of the adsorbent (Yao *et al.*, 2024).

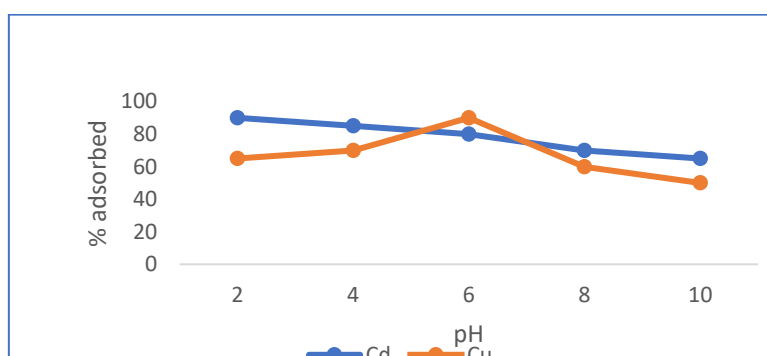


Figure 4: percentage of metal adsorbed at various pH

Effect of mass of adsorbent

The highest % adsorbed (89%) was recorded for Cd²⁺ when 1g mass of raffia palm seed shell carbon was used while 65% of Cu²⁺ was adsorbed under the same

condition (Figure 5). Adsorption decreases as mass of adsorbent exceeds 1g as metal concentration of 20ppm was used at pH 7.

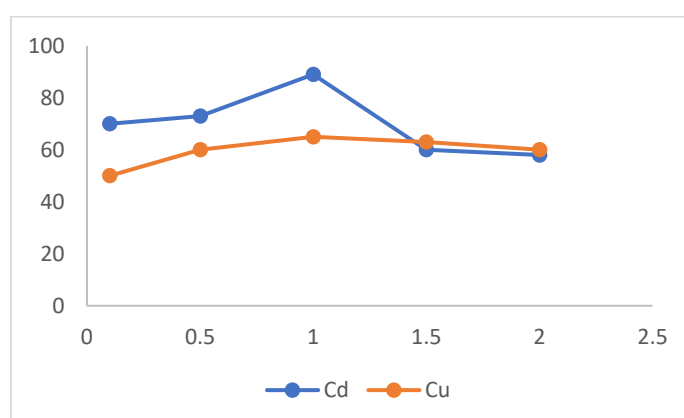


Figure 5: percentage of metals adsorbed at varied mass of adsorbent

Mass of adsorbent variation: More metal concentration was adsorbed as time increases up to 50 minutes (Figure 6). No appreciable increase in % was

recorded for Cd²⁺ while marginal increase in % adsorbed by Cu²⁺.

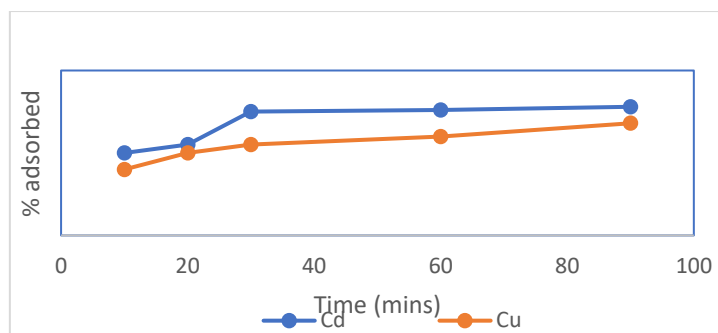


Figure 6: percentage of metal adsorbed at different time range

Variation in adsorption of metals occur as a results of difference in electrostatic interaction between the metals and the adsorbent and electronegativity of the metals (Tang *et al.*, 2021). The electronegativity of Cu^{2+} (1.90) is greater than that of Cd^{2+} (1.69) (Shi *et al.*, 2009). It is expected that Cu^{2+} should be more adsorbed than the Cd but due to the presence of

microplastics and other ions, the reverse was the case. The difference in sorption of the two metals used in the present study is affected by competition for the binding sites as a result of difference in mobility (Du *et al.*, 2018). The presence of organics affects the selectivity sequence of metal adsorption (Jalali & Moradi, 2013).

Table 3: Results of Langmuir and Freundlich Isotherms of adsorption of Cd and Cu to raffia palm seed shell carbon

Langmuir		Freundlich	
Cd	Cu	Cd	Cu
$q_e = 19.6$	$q_e = 18.4$	$1/n = 0.333$	$1/n = 3.236$
$R^2 = 0.073$	$R^2 = 0.725$	$R^2 = 0.266$	$R^2 = 0.908$
$K_L = 0.37 \text{ Lmg}^{-1}$	$K_L = 0.039 \text{ Lmg}^{-1}$	$K_F = 0.277 \text{ mgg}^{-1}$	$K_F = 0 \text{ mgg}^{-1}$
$y = 0.327x + 53.0$	$y = 0.304x + 41.8$	$y = 0.5144x - 1.8908$	$y = 0.5144x - 1.8908$
$q_m = 45.1 \text{ mgg}^{-1}$	$q_m = 202 \text{ mgg}^{-1}$		

The dimensionless constant term K_L in Langmuir isotherm determine extent of adsorption. If $K_L = 0$, the adsorption is favourable, $K_L =$ between 0 and 1, linear, $K_L > 1$ unfavourable (Ahmad *et al.*, 2012). For both Cd^{2+} and Cu^{2+} the adsorption is linear. However, for Freundlich isotherm which was based on multilayer adsorption on heterogeneous surface, if $1/n$ is between 0 and < 1 , it is favourable, if $1/n$ is > 1 it is unfavourable. It is favourable for Cd^{2+} and unfavourable for Cu^{2+} . The maximum adsorption capacity q_m for Cd^{2+} and Cu^{2+} using Langmuir are 45.1, and 202 respectively while with Freundlich the q_m are 3 and 2.5 respectively. The results showed that Freundlich adsorption is the best fit isotherm for Cu^{2+} adsorption ($R^2 = 0.908$) by activated carbon made from raffia palm seed shell.

FTIR

The FTIR of the residue of raffia palm seed shell was carried out before and after adsorption experiment to establish whether there is adsorption of metals to the surfaces of the adsorbent. The broad O-H peak was recorded at 3675.2 cm^{-1} (Figure 7). Nandiyato *et al.* (2023), reported that similar values belong to alcohol and phenols. The presence of various absorption bands before filtration, at 1617.7 cm^{-1} and 1375.3 cm^{-1} , were all characteristic of the carbonyl (-C=O-) group. However, after adsorption, small none- intense peaks were recorded which showed that Cd^{2+} and Cu^{2+} has bound to the surfaces (Figure 8). The functional group on activated carbon surfaces consist of hydrophilic and hydrophobic layers which serve as adsorption centres for water vapour and non-polar species (Fletcher *et al.*, 2007).

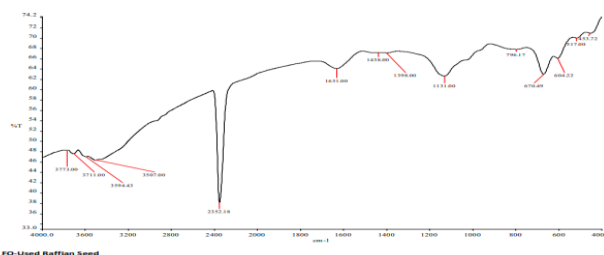


Figure 7: FT-IR Spectrum for carbonised raffia palm seed shell before adsorption.

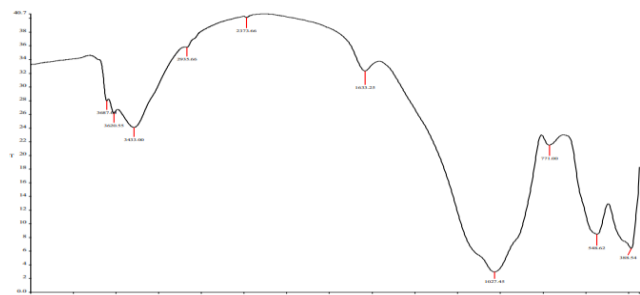


Figure 8: FT-IR Spectrum for carbonised raffia palm seed shell after adsorption.

SEM: The SEM micrographs of the raffia palm seed shell adsorbent at 120 μm and 124 μm revealed an irregular, pore structure with a heterogeneous distribution (Figure 9). These occur as a result of carbonization process which open up channels for

adsorption. After adsorption of Cd^{2+} and Cu^{2+} , the pores were partially or completely filled with adsorbate which change the surface morphology observed (Figure 10). This is in agreement with results reported in literature (Ghogomu *et al.*, 2016).

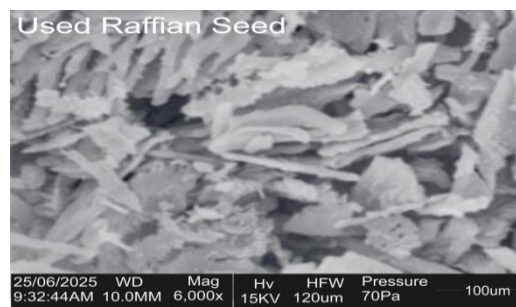
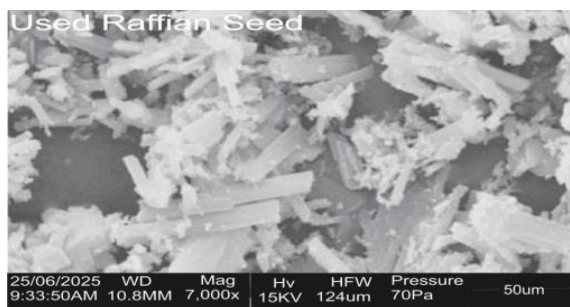


Figure 9: SEM of raffia seed shell used before adsorption

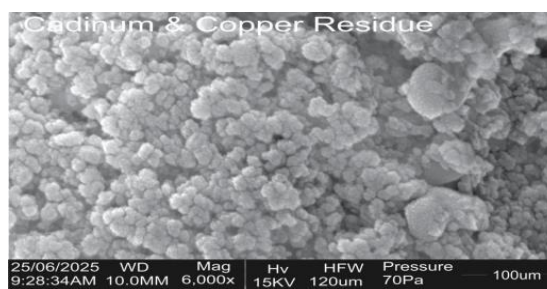
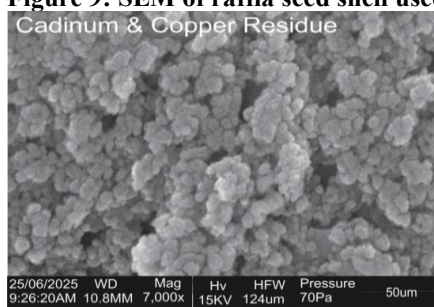


Figure 10: SEM of raffia seed shell residue after adsorption experiment

Conclusion

The study investigated the use of raffia palm seed shell as a water filter and possible use as adsorbent for the removal of plastics, Cd^{2+} and Cu^{2+} . The results showed that physicochemical parameters such as TDS, and EC can be reduced to some extent while others such as chlorides, sulphates, phosphates and nitrates can be removed marginally. GC-MS analysis of water before and after filtration showed removal of 2,4,6-Triphenyl-1-hexene while other plastics related

materials does not show any form of removal. Adsorption experiment showed effective removal of Cd^{2+} and Cu^{2+} while SEM results showed a presence of plastics and metals in the pores. Raffia palm seed shell carbon is not a good material for the removal of microplastics in water but effective for the removal of metals.

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