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The Effect of Moringa Oleifera Seeds Biosorbent (MSB) Dosage Concentration, Contact Time and Ph on Cadmium Removal from Contaminated Water

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Abstract:-This study investigates the efficacy of Moringa oleifera seeds biomass in removing cadmium (Cd) from contaminated water. The biomass was characterized using scanning electron microscopy, infrared spectroscopy, and point of zero charge techniques. Adsorption experiments were conducted with varying adsorbent dosages (2 mg - 10 mg), pH conditions (4.5, 5.5, 6.5, 7.5, and 8.5), and contact times (ranging from 40 to 200 minutes) to assess the impact of these parameters on Cd removal. Optimal conditions for adsorption were identified as 10 mg of adsorbent at pH 8.5 with a contact time of 200 minutes. The M-Langmuir model was employed to linearize the adsorption isotherms, revealing a superior fit for Cd adsorption. The model suggested a multilayer adsorption process with a chemical nature characterized by an Amino mechanism. The maximum adsorption capacity (Q max) of the Moringa oleifera seeds biomass was determined to be 9.66 mg g−1, indicating a robust interaction with the metal. In conclusion, this study demonstrates the effectiveness of Moringa oleifera seeds biomass as a bio-sorbent for the adsorption of Cadmium from contaminated water. The findings suggest its potential as a viable alternative material for water purification and treatment, offering valuable insights into the adsorption process and mechanisms involved.

Keywords:- *Moringa Oleifera Seeds Biosorbent; Contaminated Water, Dosage Concentration; Contactact Time; Ph; and Cadmium Removal Efficiency***.**

I. INTRODUCTION

➢ *Background of the Study*

Groundwater is often considered a reliable source of safe drinking water due to its inherent characteristics, which generally require minimal or no prior treatment. However, Cadmium (Cd) as a non-essential trace element found in groundwater through geogenic and anthropogenic sources. Its presence in elevated concentrations in drinking water as micro-pollution has been generating various concerns worldwide [1]. [2][3]'' have reported that Cd when consumed with human diet can become toxic in several organs of the body. [4]' posited that elevated concentration of Cd (an average of 25 µg/L) in groundwater in Assam, India were caused by geogenic contamination. World health organization (WHO) has recommend a guideline value for Cd of 3 µg/L for Drinking-Water Quality (GDWQ).Certain bacteria, fungi, and plants have proved to have great potentials for bioremediation of contaminated media through natural process. These organisms in carrying out their normal life functions can alter contaminants by use of negatively charged carboxyl, amino, phosphoryl, and sulfo groups as potential ion exchange sites and metal sinks [5]. Thus, the techniques of bioremediation can potentially destroy or render harmful contaminants in water through biological activity [6]. Bio-sorption on agro-biomass has found useful application in environmental remediation and most especially in water and wastewater treatment. Several agro biomass materials have been tested for their bio-sorption capacities for water purification: Raffia palm epicarp [7], coconut shell [8], coconut husk [9], Almond shells [10]. The use of Moringa oleifera extends beyond its traditional applications, and recent studies have explored its potential as an active low-molecular bioactive peptide for antibacterial activity. In a study referenced as [11], Moringa oleifera was examined as a plant-based coagulant and antimicrobial peptide with a molecular weight of less than 7.5 kDa. The effectiveness of this bioactive peptide was confirmed through the identification of amino acid sequences. The antibacterial activity of Moringa oleifera was assessed against various pathogenic organisms. The results indicated that the plant-based bioactive peptide exhibited significant antibacterial properties, highlighting its potential as a natural antimicrobial agent. Moreover, the combination of extracts from both the seeds and leaves of Moringa oleifera was investigated for its impact on water quality in groundwater samples. The combined use of seed and leaf extracts resulted in a substantial improvement in the removal of various water quality parameters. These parameters included hardness, pH, turbidity, Total Dissolved Solids (TDS), and E. coli count. The combined application demonstrated enhanced efficacy compared to using individual extracts from either the seeds or leaves alone. [12]. However, its efficacy in removal of metal like Cd has not been fully investigated in literatures considering dosage concentrations, contact time and pH.

This study has potential benefit to government, industrialists and individuals involved in the water sector economy. This study has potential for employment generation in the agro and water sector of the economy as many individual may be interested in the cultivation of M*oringa oleifera* plant for its subsequent harvest and sale to individuals and industrialist for application in household and industrial water treatment, which will also go a long way in preserving the human society from the toxicity-related hazards of consuming heavy metal-laden waters. In this study, the effect of MSB dosage concentration, contact time and pH on cadmium removal efficiency has been evaluated using response surface methodology (RSM).

II. MATERIALS AND METHODS

The experimentation necessary to achieve the desired results were all performed in the African Institute of vertinary Research Vom in Jos Plateau state of Nigeria, Agricultural and bioengineering Department A.B.U Zaria, Chemical and Physical Laboratory Nigeria Institute of Geo-science Jos. Chemistry Department Benue State University.

➢ *Preparation of Bio-Sorbent*

Moringa seeds that had been dehusked (peeled) were crushed in a lab using a mortar and pestle to reduce size (Plate 1), and the crushed seeds were then defatted following the process outlined by [13][14]. Following drying, it was standardized to a powder known as moringa seed bio-sorbent (MSB) and utilized for the adsorption of cadmium from solution using a 32 mesh particle size. The bio-sorbents was stored in a dry place, free from light until use.

Plate 1 Preparation of Bio-Sorbent; A (Peeled Moringa Seed); B (Floating Liquid); and C (Crushed Moringa Seed)

In order to observe any obvious changes throughout the bio-sorption process, the MSB materials were evaluated using a Perkin Elmer Nicolet iS10 FT-IR Spectrometer, spectrum 100, and scanning electron microscopy (SEM) both before and after the adsorption. Additionally, the pH of the point of zero charge (pHp), BET-surface area, micro-pore volume, meso-pore volume, and total pore size of the bio-sorbent material were measured (Plate 2).

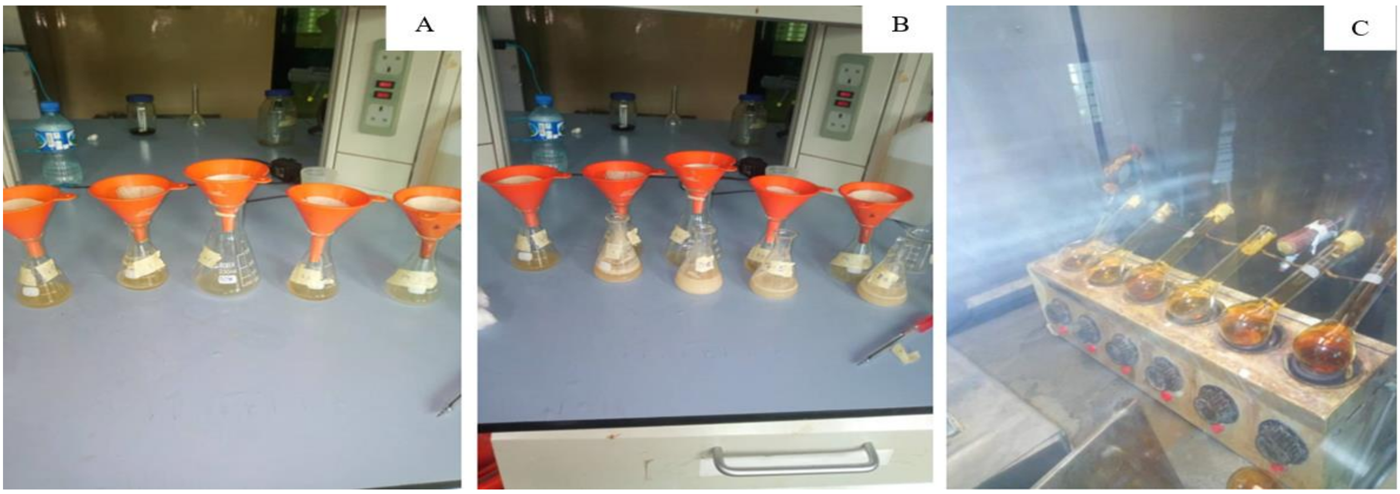

Plate 2 Characterization of MSB. A) Filtrate of PH Variation; B) During Filtration and C) Digestion in Progress

The bio-sorbent was combined with KBr (Sigma-Aldrich) in a weight/weight ratio of 1:10, and an FT-IR analysis was conducted using a PerkinElmer Spectrum 100 spectrophotometer. In absorbance mode, the FT-IR spectra were obtained within the range of 4000–400 cm−1. The Superscan SS550 microscope was used to assess the surface morphology of the bio-sorbents before they were placed on a double-sided carbon tape that had been coated with gold to a thickness of about 30 nm. At 120 °C to 350 °C, water evaporated, and the toggle was turned up to heat the area. Depending on the bio-sorbent conditions, the green light will begin to blink once degassing has begun and has been permitted to continue for more than one to two hours.Degassing of bio-sorbent was done according to standard laboratory process. Specific surface area of bio-sorbent material was evaluated using Kozeny-Carman equation.

$$S_W = \frac{14}{\varepsilon}\sqrt{\frac{\Delta P.A.t.\varepsilon^3}{\eta.L.Q(1-\varepsilon)^2}}\,cm^3/g \quad \text{and} \quad \varepsilon = 1 - \frac{W}{\delta.A.L} \qquad (1)$$

Where;

Sw is the Specific surface area of sample (cm^2/g)
e: Porosity of sample

W = Weight of sample (g)

L = Height of sample bearing (5-15 mm)

A = Cross sectional area of sample bearing (2 cm^2)

Δ = Viscosity of liquid or solid (0,000183poisse) g/cm.s

Q = Amount of gas passing from inside of sample (0-20 cm^3)

The proximate and elemental composition of the bio-sorbent was determined using energy-dispersive X-ray spectroscopy (EDX) where an X-ray spectroscopy module attached to the SEM Perkin Elmer 2400 Analyzer (PerkinElmer) was used.

➢ *Preparation of Cd(II) Solutions and Quantitative Determination*

Standard operating procedures in line with [15] were adhered to when handling and gathering samples as a precaution. The samples were analyzed as soon as they were collected, and five (5) sterile glassware containers were kept in a refrigerator for additional analysis.Digestion of Cd solution was performed by optimizing the procedure of [16]. About 5ml of the Cd solution was measured into a digestion flask which was followed by the addition of 1%ml HNO_3 and 2.5 ml of perchloric acid. This was heated on a digester in a fume cupboard at a temperature of 125°C until a transparent colour was obtained. The suggested setting for Cd (II) with background correction using high purity grade acetylene fuel was followed in order to calibrate and optimize Cd (II) using Atomic Absorption Spectrophotometer (AAS) in accordance with Standard Methods for Examination of Water and Wastewater according to [15].

- *The Absorbance Experiment*

To obtain the calibration plots and determine the concentrations of Cd adsorbed by MSB, the net absorbance was plotted against the concentration of Cd (II) in the standard solutions. The water utilized in the solution preparation process was purified using a MQ-UV water device (Millipore), but no additional purification was required for any of the reagents because of their standard grade. To prepare the 1000 mg/L Cd stock solution, a known mass of Na2HAsO4.7H2O was dissolved in tap water. Cd (II) intermediate solutions (100 mg/L) were generated using diluted stock solutions containing deionized water, and Cd (II) spiked water solutions at different concentrations (300 and 600 µg/L).

- *Quantitative Determination of Cd*

Using a UV-visible spectrophotometer system (PU8700 Philips) at a wavelength of 535 nm, the silver diethyldithiocarbamate method (minimum detectable quantity = 1µg As), also known as SDDC and in line with [17] method, was used to quantitatively measure Cd(II).

- *Adsorption Experiments*

Adsorption experiment was carried out according to the methods adopted by [18]. 4mg/l of Cd (II) was measured into a 250 ml conical flask, followed by the addition of 10 g of the biomass. The pH was adjusted to 8.5. These were heated to a temperature of 90^0c and removed at the interval of 40, 80, 120,160 and 200 minutes. The samples were digested and quantified for the amount of cadmium.For pH, 5 samples as all other parameters are kept constant 500 ml of 4 mg/l of Cd(II) was measured into 250 ml conical flask and 10g of the biomass added. Before the addition of the biomass the pH of the samples were adjusted to 4 5, 5.5, 6.5, 7.5 and 8.5.

For Dosage; All Parameters were kept constant and 50 ml of Cd(II) were measured into a 250 ml conical flash and 2 g of the biomass added. The temperature was maintained at 90 °c for 200 mins. This was repeated for other samples. The samples were digested and analyzed for Cd(II).Two to ten grams of biosorbent were tested in terms of dose. Equations (2) and (3) were used to determine the percentage of Cd(II) removed and the amount adsorbed (Qe, mg/g) by the bio-sorbent, respectively.

$$Removal\ (\%) = \frac{C_o - C_e}{C_o} \times 100 \qquad (2)$$

$$Q_e = \frac{V}{m}(C_o - C_e) \qquad (3)$$

Where:

Co = Initial concentration of metal ion in mg/L,

Ce = Equilibrium metal ion concentration in mg/L,

V and m indicate the volume of the solution treated in mL and the adsorbent mass in mg, respectively.

➢ *Experimental Design for Optimization of Cd Adsorption Model on MSB*

To derive optimal adsorption for Cd(II) of Cd by MSB, Design Expert Software Version 10.0 was used. The experimental design adopted was the Box Benkhen Design (BBD) with three factors (dosage, Contact Time and pH) each set at 3 levels. This gave rise to 15 experimental runs consisting of five (5) centre points and fifteen (15) axial points. The responses considered are the Cd (II) removal efficiency and MSB adsorption capacity, calculated from equations 2 and 3, respectively. The factor levels considered is as shown in Table 1. The desirability criteria in design of experiments were used for the optimization process using M-Langmuir model. Constraints were defined as appropriate and optimal solutions generated, from where the preferred solution was chosen. Confirmation experiments were conducted at the selected optimal conditions for determination of model adequacy and reliability.

Table 1 Factors Considered and Their Levels

Factors	**Low (-1)**	**Medium (0)**	**High (+1)**
Dosage (g)	2	6	10
Contact Time (min)	40	120	200
Ph	4.5	6.5	8.5

III. RESULTS

➢ *Characterization of Moringa Oleifera Seeds*

Table 2 is the result for characterization of M*oringa oleifera* seed for varying temperatures between 150 ^oC to 650 ^oC, the specific area of the bio- sorbent was found in the range of 178 to186. The pore size distribution varied from 2.05 to 99.99 nm while relative pressure varied from 3.0 to 8.0 at varying pore diameter between 6.20 to 8.80 nm

Table 2 Characterization of Moringa Oleifera Seed Bio-Sorbent (MSB)

Temperature ($T/^0C$)	**SpecificSurfaceArea (m^2/g)**	**Sieve Mesh (micron)**	**Pore Size Distribution**	**RelativePressure (P/P_0 X 10^{-2})**	**PoreDiameter (nm)**
150	178	<20um	2.05	3.00	6.20
250	180	20-40um	12.85	4.00	6.40
350	182	40-100um	54.00	6.00	6.50
450	184	100-350um	30.35	6.00	7.50
550	184	350um	74.0	7.00	7.60
650	186	450um	99.99	8.00	8.80

➢ *Results of FT-IR Technique of SEM*

The primary functional groups in Moringa oleifera seeds that are capable of adsorbing metal ions were found using the FT-IR technique of SEM, as demonstrated in Plate 3. The carbonyl groups in both the protein and fatty acid structures, as well as the C-H of the CH2 group found in fatty acids, are the functional groups found in moringa seeds.

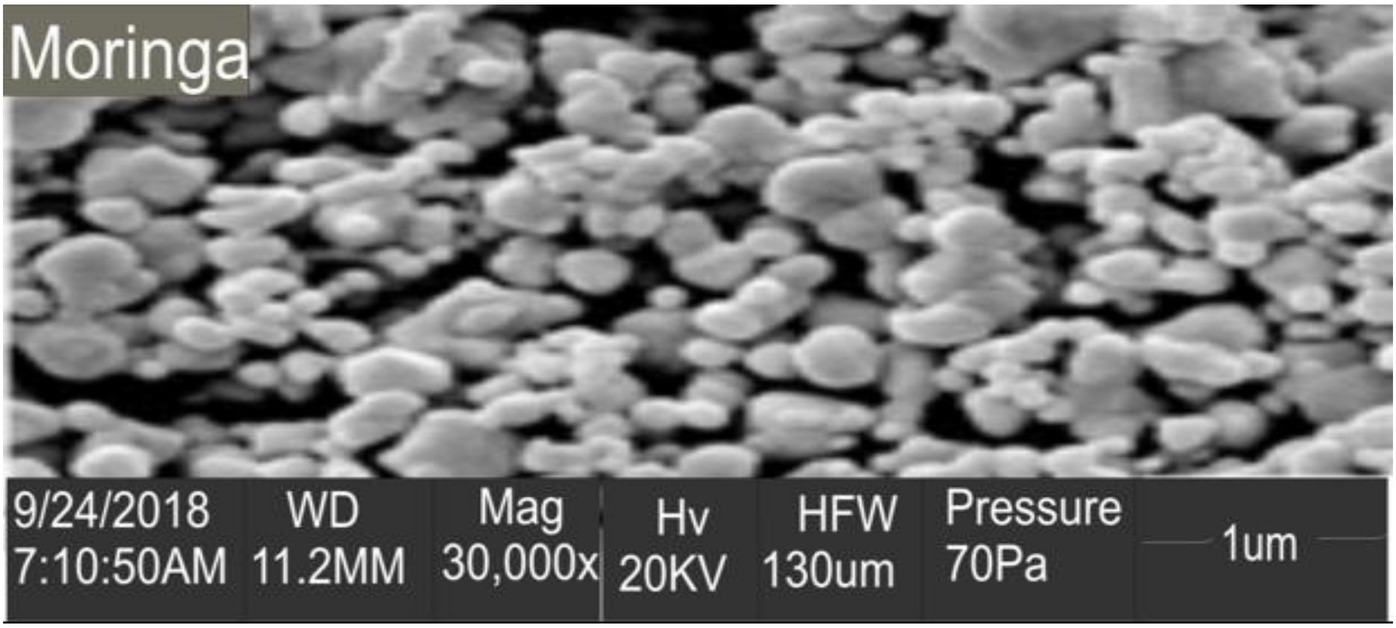

Plate 3 Scanning Electron Micrograph of Moringa Oleifera

➢ *Effects of MSB Dosage of Cd on Removal and Adsorption Efficiencies*

Table 3 presents the results of effects of M*oringa oleifera* biomass dosage on removal of Cd (II) and adsorption (removal) efficiencies. The first concentration of simulated Cd water was 9.600 ppm. The biomass dosage was varied between 2 and 10g. The corresponding concentrations of Cd were between 1.1905ppm to 1.5476ppm. The corresponding/adsorption (removal) efficiencies were between 83.88 % to 87.60 %.

Table 3 Effect of MSB Dosage on Removal Efficiencies of Cd(II)

Heavy Metals	**Biomass Dosage (g)**	**Cd (ppm) beforetreatment**	**Cd (Ppm) after treatment**	**Cd (ppm)n removed**	**Sorption (Removal)Efficiency (%)**
Cd^{2+}	2	9.600	1.5476	8.0524	83.88
Cd^{2+}	4	9.600	1.5476	8.0524	83.88
Cd^{2+}	6	9.600	1.1905	8.4095	87.60
Cd^{2+}	8	9.600	1.3095	8.2905	86.36
Cd^{2+}	10	9.600	1.1905	8.4095	87.60

➢ *Effect of Contact Time on Cd (II) Removal (Adsorption) Efficiencies*

Table 4 presents the results of effects of contact time on removal of Cd (II) and adsorption (removal) efficiencies. The Contact time (min) was varied at between 40 and 200mins. From the results, the concentrations of Cd were 0.5714 ppm for contact times 40, 80 and 200 mins and 0.6857 for contact times 120 and 160 mins. The corresponding removal (absorption) efficiencies were between 94.05% for contact times 40, 80, and 200 and 92.86% for contact times 120 and 160 min.

Table 4 Effect of Contact time (min) on Removal and Absorption Efficiencies of Cd (II) by MSB

Heavy Metals	**Contact time (min)**	**Cd (ppm) before treatment**	**Cd (ppm) after treatment**	**Cd (ppm) removed**	**Sorption (Removal) Efficiency (%)**
Cd^{2+}	40	9.600	0.5714	9.0286	94.05
Cd^{2+}	80	9.600	0.5714	9.0286	94.05
Cd^{2+}	120	9.600	0.6857	8.9143	92.86
Cd^{2+}	160	9.600	0.6857	8.9143	92.86
Cd^{2+}	200	9.600	0.5714	9.0286	94.05

➢ *Effect of pH on Removal and Adsorption Efficiencies*

Table 5 presents the results of effects of pH on removal of Cd (II) and adsorption (removal) efficiencies. pH was varied by 1.0 from 4.5 to 8.5. Cd (II) concentration after treatment varied from 1.4286ppm for pH of 6.5 and 8.5 to 1.6667ppm for pH 5.5 and 7.5. Removal efficiencies varied from 82.15% for pH 5.5 and 7.5 to 85.12% for pH 6.5 and 8.5.

Table 5 Effect of pH on Removal Efficiencies of Cd (II) by MSB

Heavy Metals	**pH**	**Cd (ppm) before treatment**	**Cd(Ppm)after treatment**	**Cd(ppm)removed**	**Sorption(Removal) Efficiency (%)**
Cd^{2+}	4.5	9.6	1.5476	8.0524	83.88
Cd^{2+}	5.5	9.6	1.6667	7.9333	82.64
Cd^{2+}	6.5	9.6	1.4286	8.1714	85.12
Cd^{2+}	7.5	9.6	1.6667	7.9333	82.64
Cd^{2+}	8.5	9.6	1.4286	8.1714.	85.12

➢ *Optimization of Cd Adsorption Model (M-Langmuir model) and Qmax- Adsorption capacity*

Table 6 shows the M-Langmuir model and Qmax- Adsorption capacity of Cd (II) by MSB, The Qmax (mg g-1) for the three factors considered for this experiment ranged between 0. 87 and 0.90 and removal efficiency between 87.0 and 90.0% for an amino mechanism.

Table 6 M-Langmuir Model and Qmax- Adsorption Capacity of Cd (II) by Moringa Oleifera Seed

pH	**Biomass Dosage (g)**	**Contact time (min)**	**Qmax (mg g-1)**	**Types of Metal Solution**	**Model**	**% Removal**	**Mechanism**
4.5	2	40	0.87	Ternary	LM	87.0	Amino
5.5	4	80	0.87	Ternary	LM	87.0	Amino
6.5	6	120	0.90	Ternary	LM	90.3	Amino
7.5	8	160	0.90	Ternary	LM	89.8	Amino
8.5	10	200	0.89	Ternary	LM	89.0	Amino

Qmax = the maximum capacity for Cd removal (mg g-1),

➢ *Comparing Optimal Cd (II) Recommended Guidelines*

Table 7 shows the optimal removal of Cd (II) by *MSB* for the simulated and treated water and were compared with the recommended NAFDAC and WHO guidelines for drinking water quality.

Table 7 Comparing Optimal Cd (II) with Recommended Guidelines

Contaminated Cd Concentration (mg/L)	**Cd Concentration after treatment (mg/L)**	**NAFDAC Drinking Water Standard (mg/L)**	**WHO Drinking Water Standard (mg/L)**
9.6	0.90	0.01	0.01

IV. DISCUSSION

➢ *Characterization of Moringa Oleifera Seeds Bio-Sorbent*

- *FT-IR Analysis:*

The spectra from FT-IR analysis revealed a broad band at 3,420 cm^{-1}, indicative of OZH stretching, highlighting the prevalence of functional groups in the protein and fatty acid structures of Moringa seeds.Peaks at 1632.42 cm^{-1} and 1712.70 cm^{-1} were assigned to symmetrical and asymmetrical stretching of the C-H of CH2 group in fatty acids.Intense bands between 1,275.00 and 1445.75 cm^{-1} were associated with C-O bond stretching.Specific peaks at 1,337.00 cm^{-1} (fatty acids) and 1,445.75 cm^{-1} (amide group in protein) were observed.A peak at 1,275.00 cm^{-1} was attributed to C-N stretching and/or N-H deformation.

- *Transmittance and Metal Removal Efficiency:*

Increasing transmittance with rising frequency of Moringa seeds coagulants was observed, and concentrations were analyzed using AAS.Moringa seeds demonstrated high efficiency in removing Cd(II) from contaminated water.

- *Mechanism of Coagulation:*

Electrostatic Patch Charge Mechanism: The flocculation activities of Moringa seeds were attributed to the electrostatic patch charge mechanism, consistent with findings from previous research ([19]).The seeds exhibited the ability to adsorb metal cations and attract highly toxic compounds.Adsorption and Neutralization: The coagulation process of Moringa Seed Bio-sorbent (MSB) involved adsorption and neutralization of colloidal positive charges.This mechanism attracted negatively charged impurities and metals in water.

- *Water Quality Improvement:*

Toxic Effects and Water Quality:Local Moringa seeds did not demonstrate significant toxic effects. Instead, they functioned as catalysts, notably improving water quality for drinking purposes.Comparable Performance:The coagulation process using MSB demonstrated results comparable to previous studies, particularly in heavy metal removal, aligning with the performance achieved by [19] using Moringa oleifera extracts.

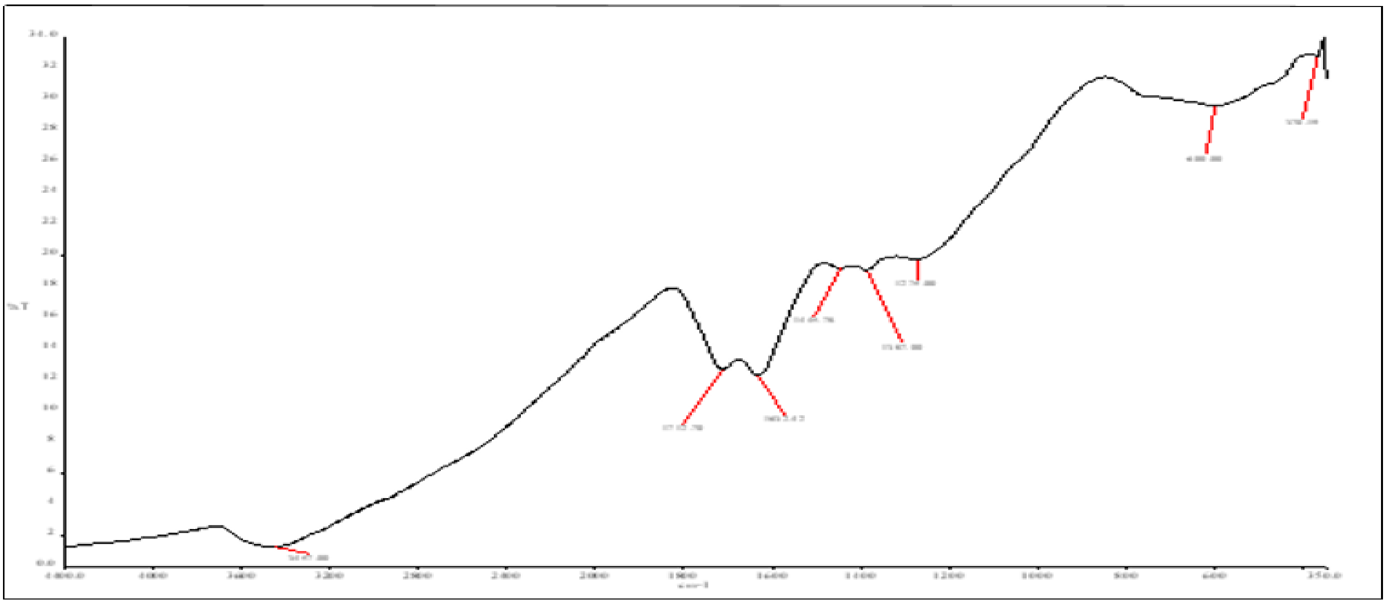

Fig 1 FT-IR Spectrum of Moringa oleifera seeds
Frequency (cm^{-1})

➢ *Sorption (Removal) Studies*

In the laboratory batch tests, the effects of dose concentration, contact time, and pH were investigated with respect to the removal of Cd (II) ion using MSB. For the experimental settings, the % sorption as reported in Tables 3, 4, and 5 has demonstrated a significant degree of removal efficiency.

➤ *Effect of Biomass Dosage on the Sorption Efficiency*

Sorption efficiency seems to increase with the increase of biomass dosage from 2.0 to 10.0 g exponentially (Figure 2). The higher the biomass dosage, the higher the adsorption and consequently Sorption Efficiency. Table 6 shows good performance of the model in relation the experimental data. Maximum Cd (II) percentage Sorption (removal) Efficiency was 87.60% at dosages of 6 g and 10 g as shown in Figure 2. Even the minimum Sorption efficiency of 83.88% for dosage of 2 g and 4 g are a good result compared to the initial concentration of 9.6 ppm. Cd concentrations in unpolluted natural water are below 1µg/l. A maximum value of 100 µg/l has also been documented [20]. In Nigeria, polluted groundwaters have been found to have concentrations between 0.02 to 0.28mg/l in industrial Area in Southwestern parts [21]. It is also very likely that surface waters in industrial areas can be highly polluted from industries that have Cd as one of elements in their wastes into the air, water and soil. This work has shown that *moringa olefiera* seed biomass can remove Cd II from water at different doses with good efficiencies.

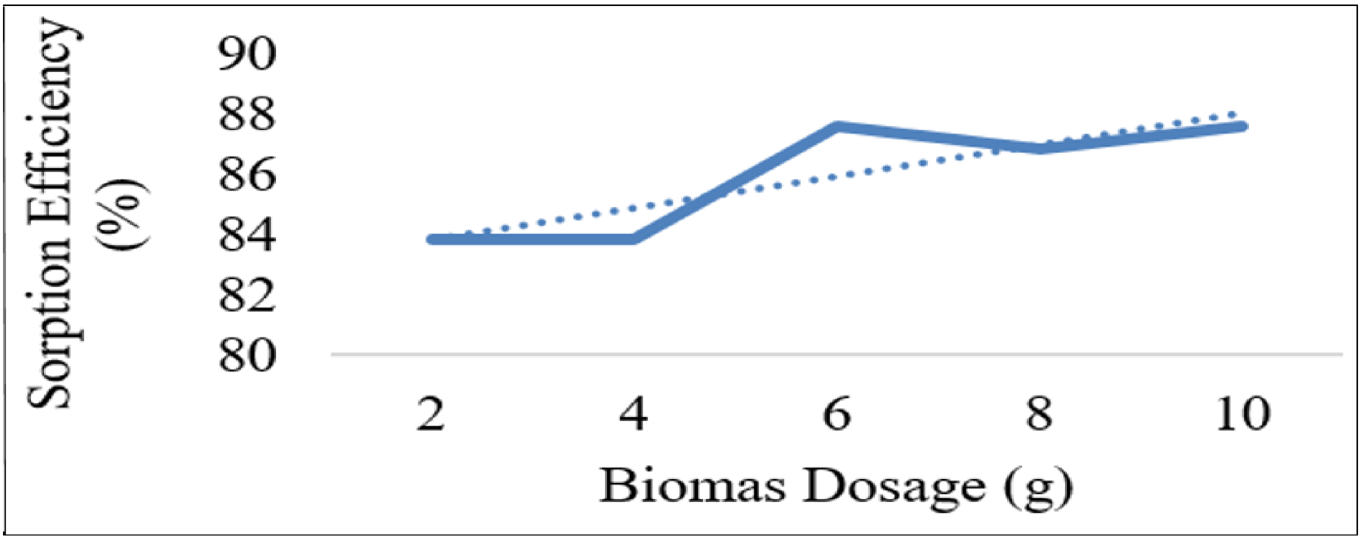

Fig 2 Effect of Biomass Dosage on the Sorption Efficiency

➤ *Effect of Contact Time on the Sorption (removal) Efficiency*

For a period of 40 to 200 minutes, the sorption of Cd (II) by MSB was investigated. With sorption efficiency (%) of 94.05, 94.05, 92.86, 92.86, and 94.05, respectively, the percent sorption of Cd ion varied with contact time from 40, 80, 120, 160, and 200 contact time (mins). As seen in Figure 3, two of the contact periods had a consumption efficiency of 92.86%, while three of them had the same percentage of 94.05%. The two numbers of 94.05% and 92.86% are close, therefore even though the trendline appears to imply a reduction with increasing Contact time, the actual findings did not appear to exhibit any trend. Further increasing the duration after equilibrium had no effect on the percentage sorption of Cd (II) ion.

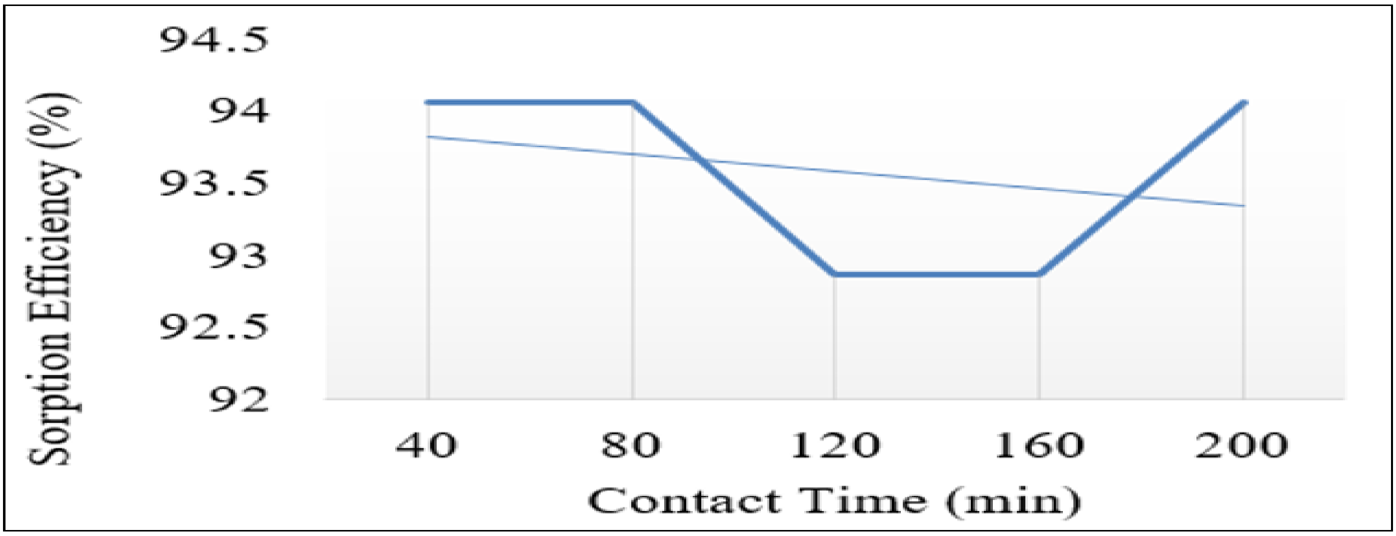

Fig 3 Effect of Contact Time on the Sorption Efficiency

➤ *Effect of pH on Sorption Efficiency*

One significant variable that has been shown to have an impact on the proportion of Cd (II) ion sorption on MSB is pH. This investigation showed that when the polluted water's pH rose from 4.5 to 8.5, sorption efficiency improved (Figure 4). The pH profile of the seed powder demonstrates that Cd(II) sorption is dependent on pH, with maximal sorption occurring at pH 6.5 and pH 8.5 and an 85.12% sorption efficiency. Additionally, the study demonstrates that the pH range of 4.5 to 8.5 allowed MSB to eliminate 82.64% to 85.12% of the Cd II concentration found in the tainted water.

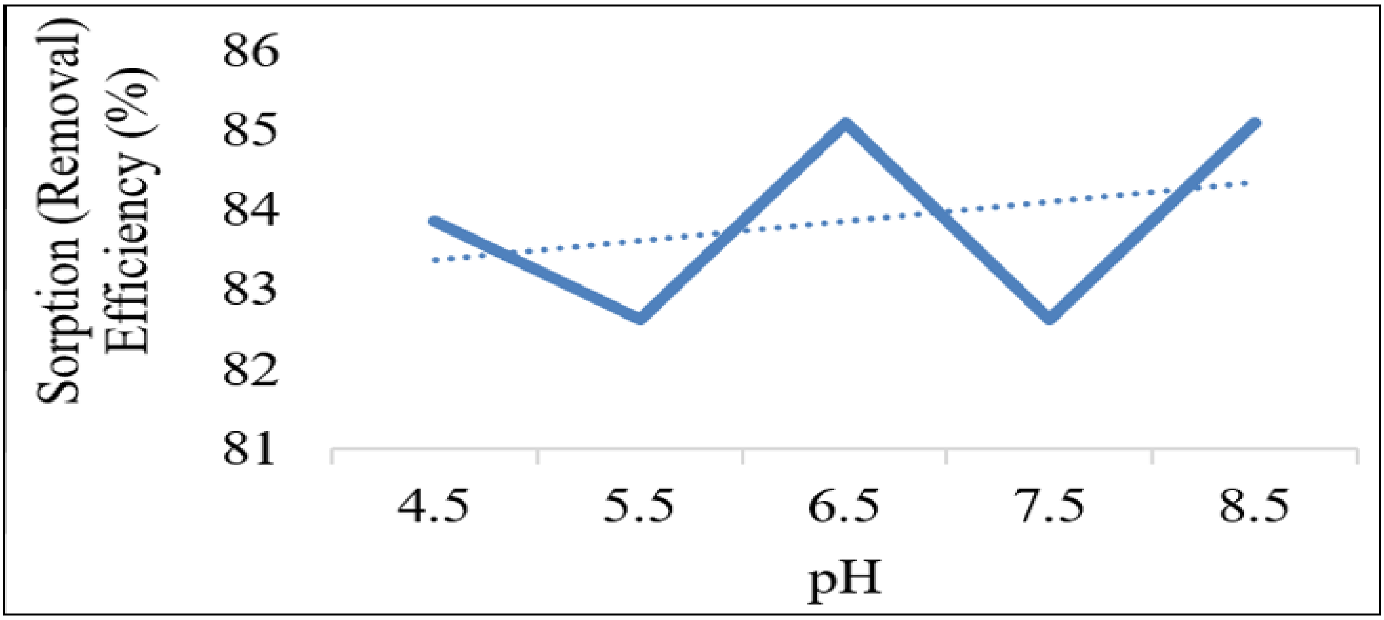

Fig 4 Effect of pH on Sorption Efficiency

V. CONCLUSION AND RECOMMENDATIONS

➢ *Conclusion*

The characterization results indicate that the seeds of Moringa oleifera contain amino acids. One important component of the functional groups that helped remove heavy metal from single solutions is amino acids. After treating the Cd (II) polluted water with varying dosages of MSB at varying contact periods and pH settings, the concentration of 9.60 ppm was significantly reduced through sorption. Solubility efficiencies of various MSB dosages range from 83.64% to 87.50%, whereas efficiencies of contact times and pH conditions range from 92.86% to 94.05% and 82.64% to 85.12%, respectively.

➢ *Recommendations*

The following recommendations are made:

- Further research should be carried out on experimental parameters that can influence metal biosorption in multi-ions solution of water.
- Since M*oringa Oleifera* Seeds are locally available, it should serve as a cheap Bio sorbent for removing other heavy metals from water.

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