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Sorption of Mn(II) Ions From Wastewater Using Dried and Blended Water Hyacinth (*Eichhornia crassipes*) Roots: Adsorption-Desorption Studies and Kinetics

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Abstract: This study examined the sorption of Mn(II) ions from waste using dried and blended water hyacinth root (WHR). It focused on how the major process parameters influenced the sorption process. Mathematical models were proposed to explain both the equilibrium and kinetics of biosorption. A desorption study was conducted using different HNO_3 and NaOH concentrations. An application study using actual industrial effluent was evaluated to analyze the fitness of the biosorbent at optimal batch conditions. The results demonstrated that the increasing initial Mn(II) ion concentration decreased Mn(II) ion removal, while an increase in the sorbent dosage increased its removal. For the rate of biosorption, the contact time was rapid between 15 and 45 min, and the maximum Mn(II) ion was removed within the initial 60 min. Equilibrium sorption was attained at pH 7, where maximum Mn(II) ion uptake was 94 %. The results also showed that Mn(II) ion biosorption at 30 °C and pH 7 for water hyacinth roots could be modeled by Langmuir and Freundlich isotherms and the pseudo-2nd order model. Furthermore, an effective desorption of Mn(II) was obtained with solutions of both NaOH and HNO_3. The results also showed that the percentage biosorption and desorption of Mn(II) from the industrial wastewater were 64.68 and 27.95 %, respectively.

Keywords: Biosorption; Biosorbent; Wastewater; Water Hyacinth; Desorption.

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1. INTRODUCTION

Water, a vital natural resource, is progressively being contaminated by urbanization and industrialization (1). Contamination of water is occurring due to the continuous discharge of both industrial effluents from chemical and textile industries, compromising the biotic and abiotic components of the ecosystem (2). Consequently, problems like alteration of the chemical oxygen demand (COD) by aquatic organisms, and reduced availability of potable water for human consumption have been on the increase due to the increased toxicity of the discharge of colored substances into water bodies from the textile, pulp, dye, and pigment industries (3,4). These colored substances contain a significant amount of heavy metal ions that pose several toxicity risks to the adjoining water bodies (5).

Over the years, conventional wastewater treatment processes, which include membrane filtration, oxidation, precipitation, and coagulation, have been employed to remove toxic heavy metals from wastewater (6–8). However, adsorption has been recognized and reported to be a cost-effective and economical choice for the exclusion of metals from industrial effluents via the use of low-cost biosorbent materials with high adsorption capability and minimizing waste dispersal problems (9).

There has been increased attention on an aquatic plant, the water hyacinth *(Eichhornia crassipes)*, for use in the adsorption of contaminants, especially toxic metal ions, from aquatic environments due to its rapid proliferation resulting from its absorption efficiency, higher than normal growth rate, low operation cost, and renewability (10, 11). The plant has been well studied, and literature has it that *E. crassipes* is capable of improving the quality of effluent from oxidation ponds. *E. crassipes* is also reported to play a key role in the treatment of industrial, agricultural, municipal, and agricultural wastewaters of any sort (12, 13). *E. crassipes* have been reported to thrive in profoundly polluted water and thus possess a high capability for the accumulation of metal ions (14). The dried root of the plant is reported to possess a strong affinity to adsorb metal ions like lead (Pb), copper (Cu), cadmium (Cd), chromium (Cr), and zinc (Zn) from aqueous media, with varying sorption performance for each metal, its solution chemistry and concentration (15-17). With many studies reporting on heavy metal adsorption onto water hyacinth biomass (18), there is currently no literature relating to the biosorption of Mn(II) ions exploitation of powdered roots of water hyacinth. Herein, the kinetics and adsorption-desorption studies on Mn(II) ion sorption from industrial wastewater with the roots of water hyacinth as a biosorbent were investigated and reported.

2. EXPERIMENTAL DETAILS

2.1. Preparation of Water Hyacinth Roots (WHR) Adsorbent

E. crassipes employed in this experiment were derived from wild specimens growing in Oba Dam, Ibadan. The collected plant root was washed several times with tap water, air-dried, and ground into powder using an electric blender, and thereafter sieved using a 2 mm sieve before being used directly as a biosorbent without further pretreatment. The morphology of the dried WHR was captured on an FEI-110730002486 SEM instrument.

2.2. Preparation of Adsorbate

A stock solution of Mn (II) ions was made in a 1L flask by dissolving 3.0766 g of $MnSO_4.H_2O$ in distilled water. The prepared Mn(II) stock solution was standardized with a standard EDTA solution using suitable indicators and buffer solution at different pH. Working solutions of Mn(II) ion were made from the stock by serial dilution. Each sample was analyzed in triplicate to reduce experimental error and obtain reproducible results.

2.3. Batch Adsorption Experiments

The adsorption of Mn (II) ion on WHR, desorption studies and application to real industrial wastewater, isotherms modeling, and kinetics of the sorption process were done using procedures and model equations previously described (13, 18-20).

2.3.1. Effect of pH

The effect of the sorbent pH was determined by providing 25 mL of Mn(II) ions working solution in a flask and adding 1.0 g of the powdered WHR to it. The mixture was gently stirred using a rotary shaker at 30 °C, for 1 h and then filtered using Whatman No.1 filter paper. The Atomic Absorption Spectrophotometer (AAS), Perkin Elmer (Analyst 200) was used to determine the concentration of filtrate of the Mn(II) ions. The pH of these suspensions was adjusted to a range between 4.0, and 9.0 with a 2M HCl or 2M NaOH solution.

2.3.2. Effect of contact time

The effect of the contact time was studied and determined at different time intervals, 15, 30, 45, 60, 75, and 90 min. 1.0 g of powdered WHR was weighed into different flasks for the different contact time intervals. 25 mL of the prepared Mn(II), at pH 7 and containing equal concentrations (5 mg/L), was added and mixed at 30 °C. At 15 min, the bottle was withdrawn, and the mixture was filtered by gravity using Whatman No.1 filter paper. This was repeated for the different time intervals, and the metal ion concentrations of the filtrate were determined using AAS.

2.3.3. Effect of initial metal concentration

Several standard solutions containing equal concentrations of Mn(II) ions were prepared, and 1.0 g of the biosorbents was weighed into several flasks. Thereafter, 25 mL of the prepared Mn(II) (pH 7) containing an equal concentration of 5 mg/L was added to the flask and mixed thoroughly for 1 h using a rotary shaker at 30 °C. At 15 mins, the flask was withdrawn, and the mixture was filtered by gravity using Whatman No.1 filter paper. This was repeated for the different time intervals, and the metal ion concentrations of the filtrate were determined using AAS.

2.3.4. Effect of dosage

The effect of the sorbent dosage was determined at different concentrations (0.2, 0.4, 0.6, 0.8, 1.0, and 1.2 g) of powdered WHR with 25 mL of Mn(II) (pH 7) in each volumetric flask. The mixture was stirred for 1 h at 30 °C with a rotary shaker to ensure equilibrium. At each time interval, the flask was withdrawn, and the mixture was filtered by gravity using Whatman No.1 filter paper. The concentrations of the metal ions in the filtrate were determined using AAS.

2.4. Desorption Studies

The desorption study was conducted at 2, 4, and 6 M concentrations of NaOH and HNO_3 solutions. A 25 mL mixture of the adsorbent and adsorbate was shaken for 1 h and left undisturbed for 24 h; thereafter, the solution was filtered. AAS was used to measure the concentration of Mn(II) ions. The percentage desorption was computed as follows:

$$Desorption\ Efficiency\ (\%) = \frac{\frac{Released\ metal}{concentration}}{\frac{Initially\ sorbed}{metal\ concentration}} \times 100 \quad (1)$$

2.5. Application to Real Industrial Wastewater

Industrial wastewater was obtained from along the Omi-Asoro river, Ilesa, Nigeria, 25 mL of which was introduced to a tightly closed flask wherein 1.0 g of the sorbents was added. The pH of these suspensions was regulated to 7 and the mixture was gently shaken with a rotary shaker at 30 °C for 1 h. Thereafter, the mixture was filtered using Whatman No.1 filter paper, and AAS was used to measure the concentration of Mn(II) ions. The desorption study for the water sample was also carried out as discussed in Section 3.3.5 above using 4M HNO_3.

2.6. Calculation of Mn(II) Ions Concentration Removal by the Sorbent

A series of batch examinations was done, and the amount of Mn(II) sorbed was determined using the mass balance equation expressed below:

$$q_e = \frac{V}{M}(C_0 - C_e) \qquad (2)$$

Where,
q_e = Metal concentration on biosorbent (mg/g) at equilibrium
C_e = Metal concentration in solution (mg/L) at equilibrium
V = Volume of initial metal solution used (mL)
M = Mass of adsorbent used (g)

More so, the percentage removal of Mn(II) ions was calculated using the following equations,

$$CR = C_0 - C_e \qquad (3)$$

$$\%\, R = \frac{C_r}{C_e} \times 100 \qquad (4)$$

Where,
R = Removal
C_o = Initial metal ion concentration in solution (mg/L)
C_e = Metal ion concentration removed or adsorbed by adsorbent (mg/L)

2.7. Equilibrium Sorption, Adsorption Isotherms, and Kinetics of Mn(II) Removal

The sorption equilibrium offers vital physicochemical information for estimating the applicability of the physisorption processes as a unit operation typically defined by isotherm models, whose parameters express the affinity and surface properties of the sorbent at a fixed pH and temperature. The sorption data were tested against two adsorption isotherm models: the Freundlich model and the Langmuir model.

The Langmuir equation was elected for use in the estimation of maximum adsorption capacity equivalent to the biosorbent surface saturation. The linearized equation after reshuffling is given in the equation below:

$$\frac{C_e}{q_e} = \frac{1}{Q_m K_a} + \frac{C_r}{Q_m} \qquad (5)$$

Where,
Q_m is the maximum sorption upon complete saturation of the biosorbent surface (mg/g).
K is a constant related to the adsorption /desorption energy (L/mg).

The experimental data was fitted into equation (4) by plotting C_e/q_e against C_e.

The slope and intercepts were used to obtain the constants.

For the Freundlich model, it was selected for use to estimate the intensity of adsorption by the biosorbent powder. The linear form of the equation is represented below:

$$\log q_e = \log K_f + \frac{1}{n}\,(log C_e) \qquad (6)$$

Where,
q_e = Metal ion uptake/ adsorption density per unit weight of WHR powder (mg/g).
C_e= concentration of a metal ion in solution at equilibrium (mg/l).
The Freundlich constants are n and K_f

K_f measures the degree of adsorption, while 'n' signifies the affinity of the sorbent for the biosorbent powder.

A plot of log q_e against log C_e in equation (5) yielding a straight line is an indication of a positive confirmation of the Freundlich adsorption isotherm. The constant 'n' can be obtained from the slope and K_f from the intercept.

Furthermore, prediction of the rate of sorption is a requisite in the design of batch sorption systems. The kinetics of the solute is also an essential requisite when choosing the optimal operational environment for the batch process. This data is often collected from pseudo-first- and second-order kinetic models. These models are correlative solute uptake, a vital component when forecasting the reactor volume. These models are clarified below.

The pseudo-first-order equation is expressed as follows:

$$\frac{dq_t}{d_t} = k_1(q_e - q_t) \qquad (7)$$

Where,
q_e = sorption capacity (mass of metal ion adsorbed) at equilibrium (mg/g).
q_t = sorption capacity (mass of metal ion sorbed) at time t (mg/g).
k_1 = pseudo-first order sorption rate constant (L/min)

Following the application of boundary conditions and integration, q_t =0 to q_t =q_t at t =0 to t =t; the modified equation (6) is:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \qquad (8)$$

This equation applies only to experimental outcomes but differs from a true first-order equation in the following ways.

- k_1 (q_e - q_t) does not epitomize the summation of the existing sites.

- log (q_e) is modifiable. It is most often different from the intercept of a plot of log (q_e - q_t) against 't', even though log (q_e) should be equal to the intercept of a plot of log (q_e - q_t) against 't' in a true first-order sorption reaction.

To fit the equilibrium sorption capacity (q_e) in equation (7) with the experimental data, q_e must be known. Where 'q_e' is unknown (as in most cases), and as chemisorption tends to be unhurried, the expanse sorbed is significantly lower than the equilibrium volume. Moreover, there needs to be a method for inferring the experimental data to t= infinity, with the assumption that q_e can be resolved by trial and error. It is thus vital to use trial and error to attain the equilibrium sorption capacity (q_e) and to analyze the pseudo-first-order model kinetics. The slope of the plot between log (q_e - q_t) versus time, t, and a linear plot confirms this model and hence can be used to obtain the pseudo-first-order rate constant k_1.

Moreover, if the rate of sorption follows the second-order mechanism, the pseudo-second-order chemisorption kinetic rate equation is expressed as,

$$\frac{dq_t}{d_t} = k\,(q_e - q_t)^2 \qquad (9)$$

Where; q_e = sorption capacity at equilibrium (mg/g).
q_t = sorption capacity at time t (mg/g)
k = rate constant of pseudo-second order sorption (g/mg/min)

For the boundary conditions q_t = 0 to q_t = q_t at t = 0 to t = t; the modified equation (8) becomes:

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + kt \qquad (10)$$

The linear form below is used:

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}\,t \qquad (11)$$

Where t is contact time (min)
q_e is solute adsorbed at equilibrium (mg/g)
q_t is solute adsorbed at time, t (mg/g).

Equation (10) has no problem assigning an effective q_e. If pseudo-second-order kinetics is appropriate, the plot of t / q_t against t in equation (10) would display a linear relationship, from which q_e and K can be deduced from the intercept and slope even when there are no other earlier known parameters.

3. RESULTS AND DISCUSSION

3.1. Morphological Studies

Figure 1 gives the result of the morphology of the sorbent material. Typically, plant roots are critical as they facilitate plant growth, and are responsible for the absorption of water, inorganic salts, and other nutrients. However, WHRs are characterized by tap- and fibrous root systems with potential voids that are capable of adsorbing toxic pollutants (21). This is because these voids could develop into air passages, with numerous support mechanisms for Mn (II) sorption. The water hyacinth powder obtained in this study demonstrated a pore-like spherical morphology that could serve as channels for trapping Mn(II) ions in the wastewater owing to the large space between the root and water (22).

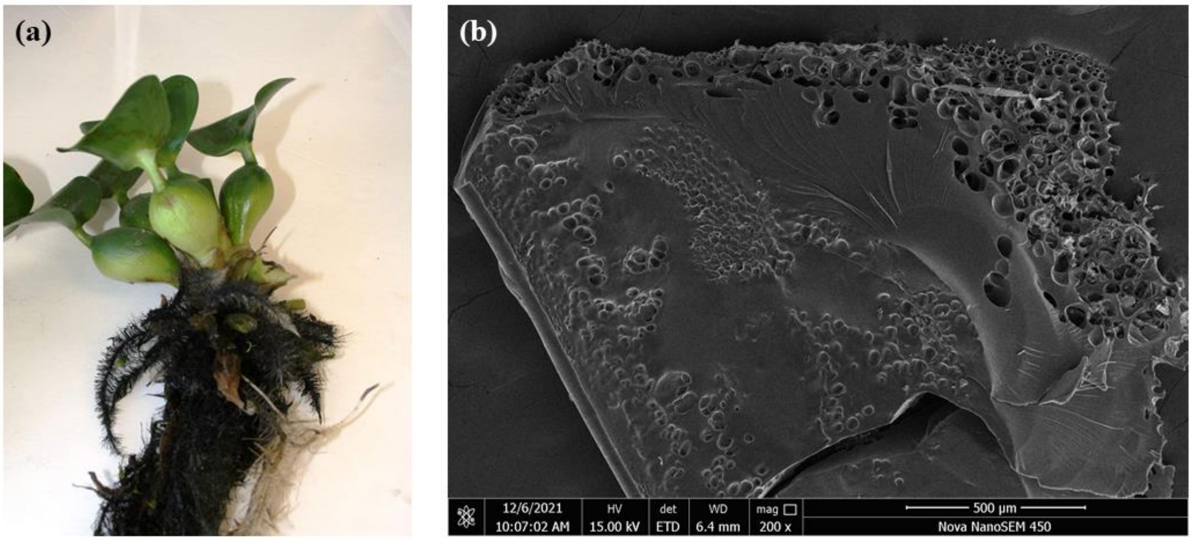

Figure 1: (a) WHR (b) SEM micrograph of dried WHR.

3.2. Effect of pH

It has been established that pH influences the solubility and concentrations of metal ions and counters, respectively on functional groups in the cell walls of the sorbent. It also impacts metal speciation in the solution, calcium carbonate solubility, and the protonation of metal binding sites (23). Therefore, pH is a key factor to be considered when dealing with sorption. Figure 2 shows the impact pH has on the sorption of Mn(II) ions.

From observation, a rise in pH from 4 – 7, influenced the uptake of Mn(II) by WHR, but beyond a pH of 7, the uptake efficiency is almost insignificant (24). The ion-exchange mechanism of sorption explains the effect of pH, and it uses ligands with cation exchange characteristics. At low pH, Mn (II) removal was repressed. This could be contingent upon the interaction of hydrogen and Mn (II) at the sorption spots, with an ostensible superiority of H^+ restricting the action of Mn(II) ion as a result of the action of the abhorrent force. With increasing pH, the oxygen-containing compounds would be exposed, thereby resulting in a total negative charge density on the sorbent and also enhancing the magnetism of positively charged metal ions, permitting sorption (25, 26).

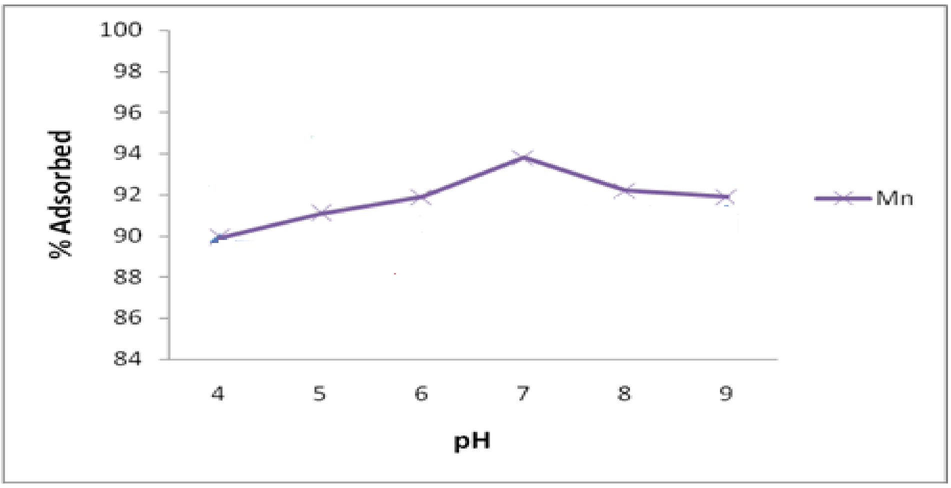

Figure 2: Effect of pH on sorption of Mn(II) by WHR.

3.3. Influence of Contact Time

Contact time influence on the biosorption capacity of Mn(II) with the biosorbent is displayed in Figure 3. By increasing the time of contact from 15 to 90 min, there was a resultant increase in Mn(II) removal from 89.5 % to 91.3 %. The result indicates a higher adsorption rate at the beginning, which resulted from the WHR's many active sites. As the experiment progresses, these sites are used up, and the uptake rate becomes a function of the Mn (II) transportation rate from the outward to the innermost sites of the WHR (27). The highest removal was reached within the first 60 min with no significant change in sorption thereafter. Therefore, the following sorption experiments were limited to a 60 min contact time. This study uses a lower equilibrium time for the adsorbents compared to some reported in the literature. This equilibrium time is significant as it is of vital importance when considering economical water, and wastewater applications. In process applications, swift biosorption is advantageous since the shorter contact time directly results in fewer contact pieces of equipment and ultimately lower processing and operational costs (9).

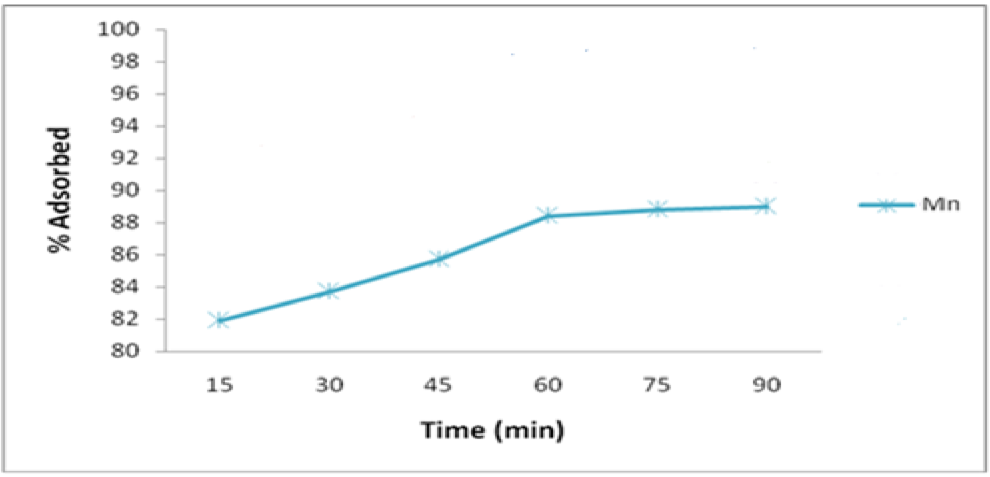

Figure 3: Contact time as a function of Mn(II) removal by WHR.

3.4. Impact of Mn(II) Ion Concentrations

The practicability and effectiveness of biosorption depend both on the absorbate concentration in the solution and the biosorbent properties. The initial absorbate concentration is a vital factor needed to conquer the resisting mass transfer between the aqueous phase and the solid (28). Figure 4 displays the effect of initial Mn (II) ion concentration on the sorption process. As the strength of the Mn(II) ion increased, there was a decrease in Mn(II) removal from 95.9 % to 90.1 %. Certain factors are thought to play a contributing role, including ensuring that adsorption sites stay unsaturated throughout the reaction. However, an accumulation of adsorbent particles at higher concentrations could be a vital contributing factor. Such accumulation causes a reduction of the available surface area needed for adsorption, thereby resulting in length extension of the diffusional path (29, 30). At lower concentrations, however, Mn (II) ions in solution could lead to interactions between the Mn(II) and the binding sites, resulting in a greater sorption percentage compared to a higher metal ion concentration. A lower adsorption yield at higher concentrations is a result of active site saturation.

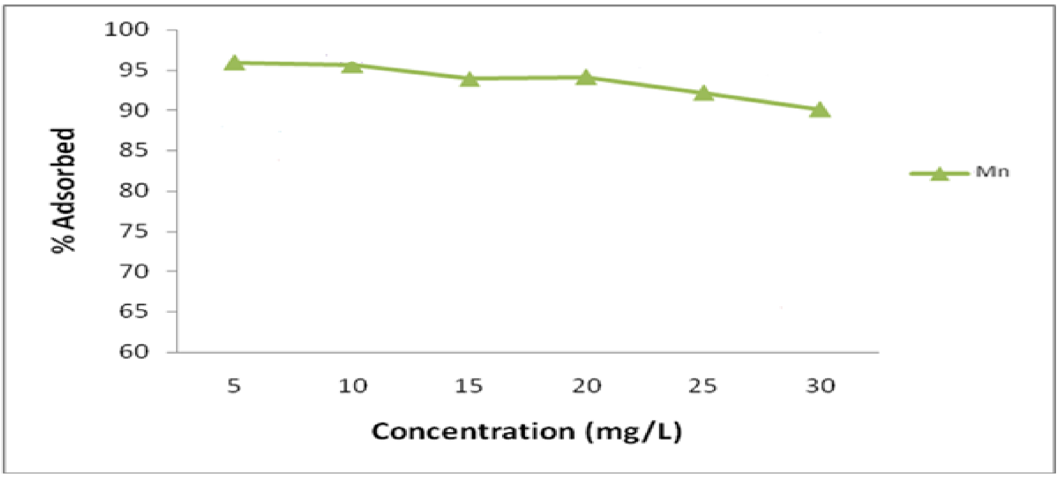

Figure 4: Initial metal ion concentration as a function of Mn(II) removal by WHR.

3.5. Effect of Dosage

The adsorbent dosage has a significant influence on the biosorption process. This was obtained by varying the adsorbent dosage. Figure 5 below clearly displays the removal efficiency. The Mn(II) removal increased with an increase in the WHR mass. The increase in sorption is probably a consequence of the binding site number and the increasing sorbent surface area (16). The removal efficiency increased from 99.5 % to 99.9 % with an increase in dosage from 0.2 g to 1.2 g.

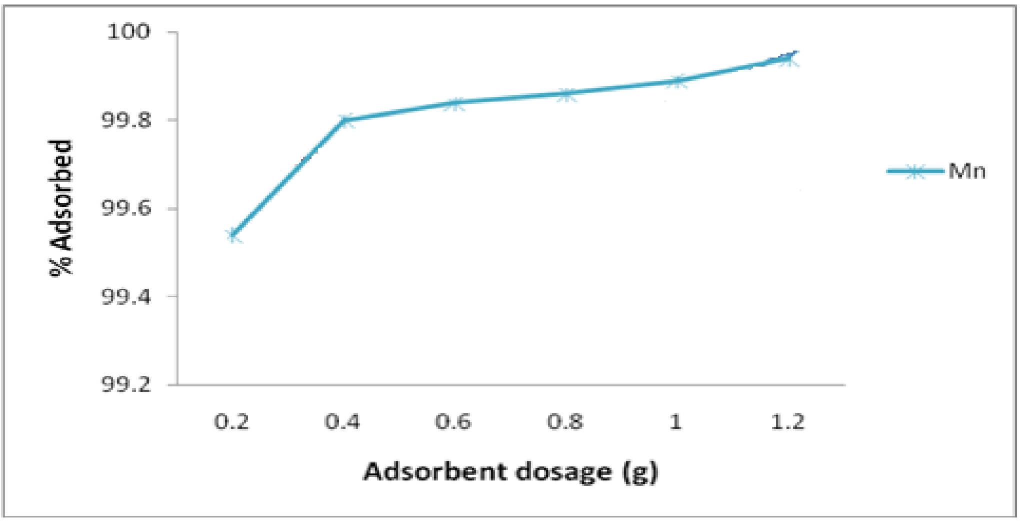

Figure 5: Effect of WHR dosage on Mn(II) removal.

3.6. Adsorption Equilibrium

A plot of C_e/q_e against C_e for Mn(II) for the adsorbent (Figure 6a) with the regression coefficient (R^2) and Langmuir isotherm parameters Q_m, K_a, and K_R, is given in Table 1. The adsorption of Mn (II) ions by WHR fits well into the Langmuir model, as evidenced by the obtained R^2 value of 0.986. The sorption process follows the Langmuir adsorption equation, suggesting that the uptake of Mn(II) ions occurs independently of the type of binding site (31). The adsorption of Mn(II) by WHR ensues principally because of the ion exchange at the surface level. It is therefore expected that the tendency of adsorption will be greatly influenced by the electronegativity of Mn(II) (1.55) (20). However, the adsorption affinity of the metal observed arising from the sorption capacity (Q_m) values opposes the order of electronegativity. It is also known that the removal of metals of greater ionic radius is superior to that of lesser ionic radius. Besides, other factors such as the ionic radius and the initial metal ion may be responsible for the sorption of Mn(II) onto WHR. This infers that initial metal ions and several factors play a role in the sorption process.

The adsorption coefficient K_a (l/mg) about the energy of sorption is given in Table 1. The results show that the energy of sorption is significantly favorable for the adsorbents, with a K_a value of 0.65. The separation factor (K_R) is a crucial property of the

Langmuir isotherm. For this study, the values of K_R obtained revealed that the adsorption of Mn(II) ions by WHR is favorable, as the K_R value obtained was greater than 0 but less than 1 (32).

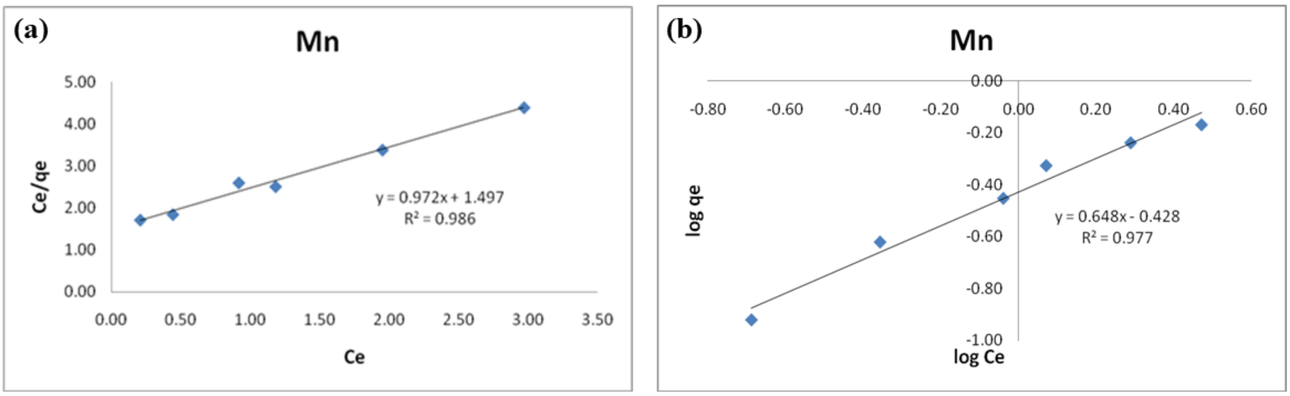

Figure 6: Langmuir (a) and Freundlich (b) isotherms for Mn(II) adsorption by WHR.

Furthermore, a plot of log q_e against log C_o is shown in Figure 6b. The linear plot of the log q_e against log C_o shows that Mn(II) ion adsorption by WHR fits well with the Freundlich model. The Freundlich isotherm parameters R^2, n, 1/n, and K_f of the Mn(II) ion and WHR are shown in Table 1. The R^2 (0.977) value obtained for WHR adsorption of Mn(II) ions showed that the Freundlich model is an ideal model for Mn(II) ion adsorption by WHR. In this study, the 1/n value obtained was < 1 suggesting that there is significant adsorption at lower concentrations (33). The adsorption capacity K_f (0.36) obtained for the Mn(II) ion and adsorbent was appreciable.

Table 1: Freundlich isotherms and Langmuir parameters for the adsorption of Mn(II) ions by WHR.

	Langmuir isotherm parameters				Freundlich isotherm parameters			
	K_a	Q_m	K_R	R^2	1/n	n	K_f	R^2
Mn	0.65	1.03	0.24	0.986	0.65	1.54	0.37	0.977

3.7. Kinetics of Sorption

The plots of log (q_e - q_t) versus t and of t/q_t against t for the adsorption of Mn(II) are shown in Figure 7, while the kinetic data are presented in Table 2. The regression value (0.088) obtained for the 1st order model indicates that the 1st order model is not fitting when describing the process of adsorption of Mn(II) by WHR. The q_e value (calculated) for the adsorbent differs greatly from that of the experimental. This is predictable, as the pseudo-1st order is not an ideal model to describe the process (34).

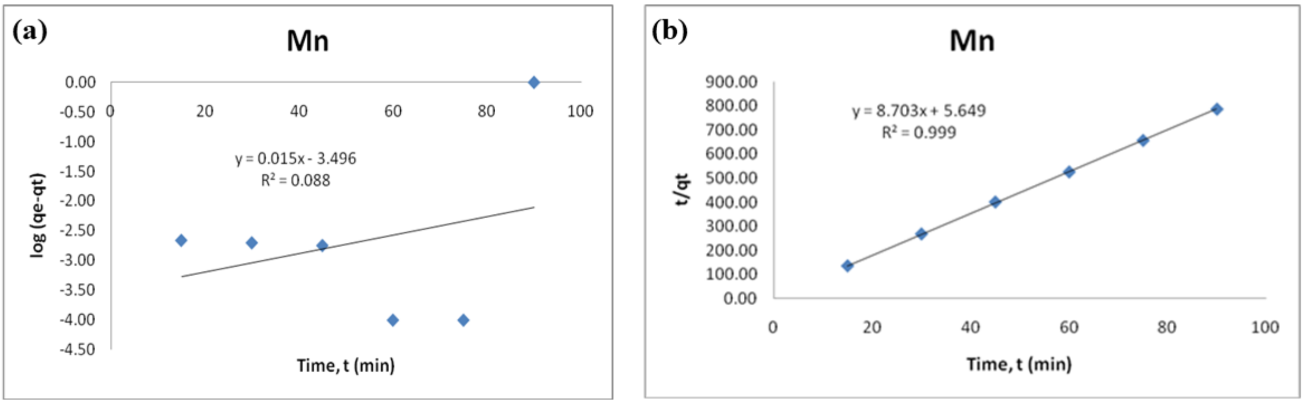

Figure 7: Sorption Kinetics of Mn (II) removal by WHR (a) Pseudo 1st order (b) Pseudo 2nd order.

Table 2: Obtained kinetic data for the biosorption process.

	Experimental q_e (mg/g)	Calculated q_e (mg/g)	k_1 (1/min)	k_2 (g/mg.min)	h (mg/g.min)	R^2
Pseudo-1st order	0.1141	3.19×10^{-4}	-0.03	-	-	0.088
Pseudo-2nd order	0.1141	0.11490	-	13.41	0.18	0.999

However, Figure 7b shows an exceptional fit between the data points. It shows ideal linearity in the experimental data. The R^2 value (0.999) obtained for the 2^{nd} order model was very high, and the q_e (calculated) and q_e (experimental) were very close. These signify that the 2^{nd} order model is fitting when describing the process of adsorption of Mn(II) by WHR and supporting the proposal that chemisorption is the rate-determining stage (35).

3.8. Desorption Studies

Land disposal and incineration are possibilities for discarding used adsorbent. These methods, however, result in pollution of the environment, whether directly or indirectly. A less used possibility would be the regeneration of Mn from the used adsorbent. With the regeneration of Mn, there would be the option of recycling Mn and WHR, hence contributing to the possible protection of the environment. Desorption studies aid in clarifying the adsorption pathway and recovering metals from adsorbent and wastewater (36). Table 3 gives the desorption efficiencies (DE) of the Mn (II) ion from the WHR.

The desorption efficiency increased from 4.58 % to 4.84 % as NaOH concentration increased from 2M to 4M. However, as NaOH concentration increased from 4M to 6M, the DE decreased to 2.05 %. Concerning HNO_3, the DE increased from 32.26 % to 33.22 % as HNO_3 concentration increased from 2 M to 6 M. Overall, effective desorption of Mn(II) was obtained with both NaOH and HNO_3 solutions. The resulting desorption phenomenon seen in both HNO_3 and NaOH is due to ion exchange interaction rather than chemisorption (37). The results indicated that HNO_3 and NaOH are satisfactorily beneficial in desorbing Mn (II) from WHR.

Table 3: Desorption of Mn(II) loaded water hyacinth roots adsorbent.

	Percentage desorption (%)
Strength of NaOH (M)	
2	4.58
4	4.84
6	2.05
Strength of HNO_3 (M)	
2	32.26
4	33.01
6	33.22

3.9. Application Studies Using Real Industrial Wastewater

Further investigation on real industrial wastewater was conducted to examine the fittingness of the adsorbent at optimal batch conditions. The obtained result demonstrates a 64.68 % adsorption of Mn(II) by the adsorbent and a substantial percentage desorption of Mn(II) from WHR of 27.95 %. The result of the desorption study has a similar trend to that obtained for the aqueous solution of synthetic wastewater.

4. CONCLUSION

WHR has been effectively applied as an adsorbent for removing Mn(II) ions from wastewater. The results showed that with an increasing initial Mn(II) ion concentration, there was a decrease in Mn(II) ion removal while increasing sorbent dosage increased Mn (II) ion removal. Equilibrium sorption was attained at pH 7 within the first 60 min. The mechanism of sorption suited Freundlich and Langmuir isotherms, while the kinetics suited the pseudo-2^{nd} order model. Effective desorption of Mn(II) was obtained with both NaOH and HNO_3 solutions, however, desorption was more suited for HNO_3. Appreciable results were also obtained for real industrial wastewater applications.

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