



Influence of Dosage and Type of Red Mangrove Sorbent on the Adsorption of Chromium (VI) Ions in Aqueous Solution

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Abstract

To present the influence of dosage and type of red mangrove sorbent on adsorption of chromium [Cr] (vi) ion in aqueous solution, adsorption data of raw and sulphuric acid (H₂SO₄)-treated red mangrove biomass (leaves, bark and root) sorbents, obtained from a recent dissertation, was re-viewed, as function of dosage for raw and H₂SO₄-treated sorbents. The adsorption was influenced by the dosage and type of red mangrove sorbent. The lower the dosage applied the higher the adsorption capacity obtained. Also, H₂SO₄-treated red mangrove sorbent adsorbed Cr (vi) ion more than raw sorbent. Values of adsorption capacity recorded for raw sorbents were lower than those for H₂SO₄-treated sorbents, meaning raw red mangrove biomass was not as efficient as H₂SO₄-treated red mangrove biomass. Isothermic data indicate stronger Cr (vi) adsorption by H₂SO₄-treated sorbents than raw sorbents; fitted well with the Freundlich model for raw sorbents. Kinetic data indicate a high Cr (vi) adsorption rate by both H₂SO₄-treated and raw sorbents; showing the best correlation with the pseudo-second-order model, suggesting chemisorption for both sorbents. H₂SO₄-treated red mangrove leaves, bark, or root powder can be employed as an adsorbent for the treatment of Cr (vi) ion in aqueous solution.

Keywords: Sulphuric acid, Adsorbent, Biomass, Freundlich, Capacity

Introduction

For reasons of ready availability and low processing cost, agro-based materials are now being researched for use as adsorbents for the treatment of heavy metal contamination in wastewater. Amongst them are the research carried out by Ofudje et al. (2014) and Maduelosi et al. (2023). In a recent dissertation titled 'Evaluation of the adsorptive capacity of mangrove biomass on chromium (vi) in aqueous solution', Nduka (2019) researched the adsorption of chromium [Cr] (vi) ion using raw and sulphuric acid (H₂SO₄)-treated red mangrove (*Rhizophora mangle*) leaves, bark, and root sorbents. The results of the study are still being considered. The focus of the present review is to re-analyze the data as a function of dosage for sulphuric acid (H₂SO₄)-treated and raw red mangrove biomass and re-present the same as influence of dosage and type of red mangrove biomass sorbents on adsorption of Cr (vi) ion in aqueous medium.

Materials and Methods

Materials and Instrumentation: Details of the collection and pre-treatment of red mangrove sample, instrumentation as well as preparation of adsorbate solution have been given by Nduka (2019). Accordingly, parts (leaves, bark, and root) of a red mangrove plant (*Rhizophora mangle*) were collected from a mangrove swamp forest (Plate 1) near Bakana community in the Degema Local Government Area of Rivers State, Nigeria. Samples of the leaves, bark, and root are illustrated in Plate 2. These were washed with tap water to remove dirt, soil particles, and other soluble materials. Leaves were sun-dried for three days while the bark and root were sun-dried for five days. These were also ground to powder with mechanical grinder and sieved through 5 × 10² μm sifter to the particle size depicted in Plate 3. 30 g each of the raw red mangrove leaves, bark and root samples were mixed with 50ml of 0.5 M sulphuric acid (H₂SO₄) solution and placed in an oven for 24 hrs and then soaked in 50 ml de-ionized water until stable pH was obtained according to the method used by Nordiana et al. (2013) and Iboroma et al. (2020). The mixture was then filtered and the residues were oven dried. The H₂SO₄-treated samples were soaked in 50 ml of 0.5 M sodium bicarbonate (NaHCO₃) for 1hr and then washed with de-ionized water until all residual acid was removed. This was confirmed by testing the wash water with blue litmus paper. The samples

were dried for 5 hours at 110 °C, cooled, and stored in a tight container for further analysis. The treated samples were labeled as H₂SO₄-treated red mangrove leaves, H₂SO₄-treated red mangrove bark, and H₂SO₄-treated red mangrove root sorbents. The untreated samples were also labeled as raw red mangrove Leaves, raw red mangrove bark, and raw red mangrove root sorbents. The morphology of the samples (H₂SO₄-treated and raw) was also analyzed using Phenom world SEM-Pro (model X800-07334) Scanning Electron Microscope (SEM), at the Multi-User-Laboratory, Umaru Musa Yaradua University, Katsina, Katsina State, Nigeria. Stock solution (1000 ppm) of chromium [Cr] (vi) ion, was prepared by dissolving 2.828 g of Potassium dichromate [K₂Cr₂O₇] (Plate 4) in 1 liter of deionized water. The working solutions (10 ml, 20 ml, 40 ml, 60 ml, and 100 ml) were prepared from stock solution by dilution of the stock solution of [K₂Cr₂O₇] with deionized water.



Plate 1: Red Mangrove (*rhizophora mangle*) Trees (Source: Nduka, 2019)



019; Iboroma



019;



Plate 4: Potassium Dichromate in Solid Form (Source: Nduka, 2019)

Experimental procedures: Batch adsorption experiments were conducted using 60 ml beakers into which the Cr (vi) solution and red mangrove biomass samples were mixed. 0.2 g of either H₂SO₄-treated or raw red mangrove leaves, bark, and root was weighed into a beaker containing 20 ml of 40 ppm chromium (vi) solution. The beakers were agitated on an orbital shaker at a constant speed of 150 rpm. Samples were withdrawn at predetermined time intervals, filtered, using Whatman No. 1 filter paper, and analyzed for residual Cr (vi) concentration using an atomic absorption spectrophotometer (AAS). The experiment was repeated using 1, 2, 3, and 4 g dosages for the H₂SO₄-treated and raw mangrove sorbents. pH of the solutions was determined with a pocket-sized pH Meter

(HANNA instrument: H196107 pH). The experiments were carried out at room temperature. The amount of Cr (vi) ion adsorbed (q_e) was calculated from the differences between the initial (C_o) and final (C_f) Cr (vi) concentration using equation 1. The percentage of Cr (vi) ion removed by various parts of red mangrove biomass was calculated from the differences between the initial and final Cr (vi) concentrations using Equation 2.

$$q_e = \left(\frac{C_o - C_f}{m} \right) V \quad (1)$$

$$\% \text{ Chromium (vi) Ion Adsorbed} = \left(\frac{C_o - C_f}{C_o} \right) 100 \quad (2)$$

In Equations 1 and 2, q_e is the concentration of Cr (vi) ion adsorbed at equilibrium (mg/g), V is the volume of solution (l) and m is the mass of the adsorbent (g), and C_o and C_f are as defined earlier.

Adsorption Isotherms: Isotherm data for Cr (vi) ion adsorption were generated by using 0.2 g of the 500 μ m particle size and by varying the concentration of Cr (vi) from 10, 20, 40, 60 to 100 ppm at solution pH of 2.0. Other experimental procedures were followed and the residual Cr (vi) concentration was determined using AAS. Isotherm data for both acid-treated and raw sorbents were generated.

Langmuir and Freundlich isotherm models given as Equations 3 and 4 were used to interpret the adsorption process.

$$\frac{C_e}{q_e} = \frac{K_L}{q_{max}} + \frac{1}{q_{max}} C_e \quad (3)$$

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

where q_{max} is the maximum adsorption at monolayer (mg/g), C_e is the equilibrium concentration of the metal ion in solution, q_e is the amount of ion adsorbed per unit weight of the adsorbent, K_L is the Langmuir constant related to the affinity of binding sites, K_f and n are called Freundlich constants. K_f defines the relative adsorption capacity while n indicates the intensity of the adsorption.

Adsorption Kinetics: For kinetic properties, 0.2 g of either sorbent was added to 20 ml of 60 ppm Cr (vi) solution and agitated for time intervals of 30, 60, 90, 120, 150, 180, 210, and 240 mins. Other experimental procedures were followed and the residual Cr (vi) concentration was determined using AAS. Kinetic data for both modified and raw sorbents were generated.

The kinetics process was also analyzed using the Pseudo-first order (equation 5) and pseudo-second-order (equation 6) adsorption models by Lagergren (Ngugi, 2013).

$$\log(q_e - q_t) = \log q_e - \left(\frac{K_1}{2.303} \right) t \quad (5)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

In equations 5 and 6, q_e and q_t represent the amount of Cr (vi) ion adsorbed at equilibrium and at a given time, t . K_1 and K_2 are the rate constants of the pseudo-first order and pseudo-second order adsorption processes, respectively.

RESULTS

Adsorbents Surface Morphology

SEM micrographs of the acid-treated and raw red mangrove biomass sorbents taken at 200 μ m are presented in Figures 1A, B, and C. They indicate that the acid-treated red mangrove sorbents (ATML, ATMB, and ATMR) have wider surface structures and porosity than the raw ones (RML, RMB, and RMR). In Figure 1A, the surface of raw mangrove leaves (RML) was smooth and had a small opening while the acid-treated red mangrove leaves (ATML) had a dented surface. In Figure 1B, ATMB showed white patches (Poly crystallites), that look like a second-phase material. These patches were less on the root and the leaves. In Figure 1C, ATMR was rough while RMR appears more compact.

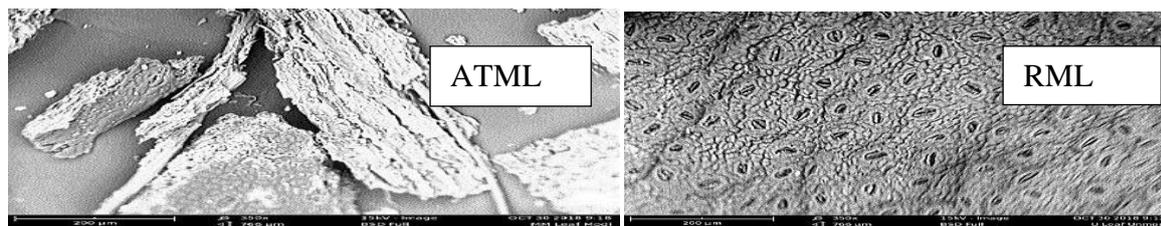


Figure 1A: SEM Images Showing Acid-treated Red Mangrove Leaves (ATML) and Raw Red Mangrove Leaves (RML) (Source: Nduka, 2019; Nduka et al., 2019; Coockey et al., 2019)

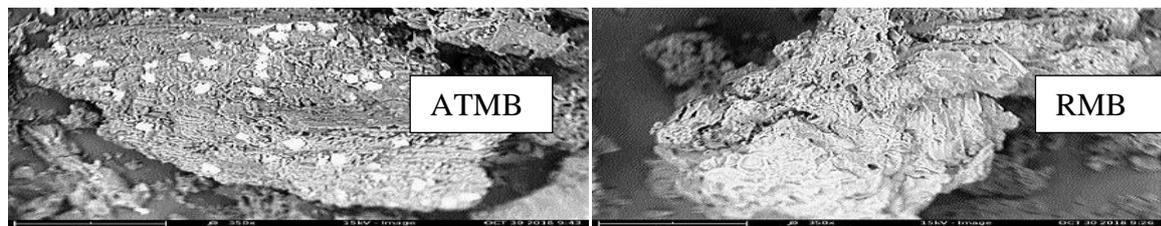


Figure 1B: SEM Images Showing Acid-treated Red Mangrove Bark (ATMB) and Raw Red Mangrove Bark (RMB) (Source: Source: Nduka, 2019; Nduka et al., 2019; Coockey et al., 2019)

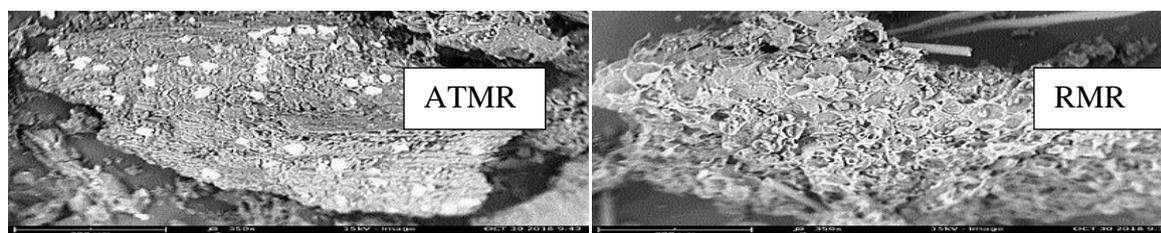


Figure 1C: SEM Images Showing Acid-treated Red Mangrove Root (ATMR) and Raw Red Mangrove Root (RMR) (Source: Source: Nduka, 2019; Nduka et al., 2019; Coockey et al., 2019)

Adsorption Properties of H₂SO₄-Treated and Raw Red Mangrove Sorbents

Table 1 shows the adsorption capacities of H₂SO₄-treated leaves, bark, and root sorbents at different dosages. It shows that acid-treated leaf had the highest adsorption capacity, followed by acid-treated root and acid-treated bark as the least. Similarly, Table 2 displays the adsorption capacities of raw red mangrove leaf, bark, and root sorbents at different sorbent dosages. It shows that at the various dosages, except the lowest, raw red mangrove leaf had the highest followed by raw red mangrove root and raw red mangrove bark as the least.

Table 1: Absorption Capacity and % Adsorption as Function of Dosage for H₂SO₄-Treated Red Mangrove Biomass Sorbents

Adsorbent Dosage	Adsorption Capacity, (mg/g)			% Adsorption		
	Leaves	Bark	Root	Leaves	Bark	Root
0.2	2,996.00	2,172.50	2,819.50	76.90	55.76	72.37
1	563.90	458.02	575.68	72.37	58.78	73.88
2	329.01	205.48	317.25	84.45	52.74	81.43
3	223.87	158.40	225.60	86.19	60.99	86.86
4	175.05	132.40	175.50	89.86	67.97	90.09

Table 2: Absorption Capacity and % Absorption as Function of Dosage for Raw Red Mangrove Biomass Sorbents

Adsorbent Dosage	Adsorption Capacity, (mg/g)			% Adsorption		
	Leaves	Bark	Root	Leaves	Bark	Root
0.2	1,707.90	701.90	1,819.50	43.68	18.02	46.70
1	493.30	234.50	340.38	63.31	30.09	43.68
2	234.89	134.89	140.78	60.29	34.62	36.13
3	167.40	104.67	83.07	64.45	40.30	33.71
4	143.39	90.05	41.05	73.61	46.23	21.07

Isotherm Parameters for Modified and Raw Red Mangrove Sorbents

Isotherm parameters of modified red mangrove sorbents are presented in Table 3. Maximum adsorption, Q_{max} , obtained for leaves, bark, and root were 1111.1, -3.3333, and 1111.1 respectively. Freundlich constant, n , obtained for modified red mangrove leaves, bark, and root were 1.1164, 0.619, and 1.1767, respectively. Table 3 also indicates high correlation coefficient values ($0.806 \leq R^2 \leq 0.963$) for the Freundlich model. Langmuir model had poor correlation coefficient values ($0.11 \leq R^2 \leq 0.38$). Isotherm parameters of raw red mangrove sorbents evaluated from Langmuir and Freundlich plots are given in Table 4. For raw red mangrove leaves, bark, and root, maximum adsorption, Q_{max} , obtained were -1250, -3.3333, and 2500. Freundlich constant, n , obtained were 0.6301, 0.7567, and 0.9960, respectively. Table 4 also indicates high correlation coefficient values ($0.93 \leq R^2 \leq 0.99$) for the Freundlich model. Langmuir model had poor correlation coefficient values ($0.31 \leq R^2 \leq 0.76$).

Table 3: Langmuir and Freundlich Isotherm Parameters of Chromium (VI) ion Adsorption by H₂SO₄-Treated Red Mangrove Biomass Sorbents (Source: Source: Nduka, 2019; Nduka et al., 2019; Cooney et al., 2019)

Sorbent	LANGMUIR			FREUNDLICH		
	Q_{max} (mg/g)	K_L (l/mg)	R^2	K_F (mg/g)	n	R^2
Leaves	1111.1	178.89	0.383	74.67	1.1164	0.963
Bark	-3.3333	-41.33	0.807	32.92	0.6191	0.887
Root	1111.1	145.56	0.110	122.15	1.1767	0.806

Table 4: Langmuir and Freundlich Isotherm Parameters of Chromium (VI) ion Adsorption by Raw Red Mangrove Biomass Sorbents (Source: Source: Nduka, 2019; Nduka et al., 2019; Cookey et al., 2019)

Sorbent	LANGMUIR			FREUNDLICH		
	q_{max} (mg/g)	K_L (l/mg)	R^2	K_F (mg/g)	n	R^2
Leaves	-1250	-70.00	0.761	5.1310	0.6301	0.931
Bark	-3.3333	-89.67	0.740	9.5017	0.7567	0.978
Root	2500	76.50	0.307	18.4884	0.9960	0.993

Kinetic Parameters for Modified and Raw Red Mangrove Biomass Sorbents

The evaluated kinetic parameters of chromium (VI) ion adsorption by modified leaf, bark, and root are presented in Table 5. Results indicated perfect conformity with second-order kinetics ($R^2 \sim 1$) than that of first-order ($R^2 \geq 0.84$). The evaluated kinetic parameters of chromium (VI) ion adsorption by raw red mangrove leaf, bark, and root are presented in Table 6. Results indicate perfect conformity with second-order kinetics ($R^2 \geq 0.99$) than that of first-order ($R^2 \geq 0.77$).

Table 5: Kinetic Parameters of Cr (vi) ion Adsorption by H₂SO₄-Treated Red Mangrove Biomass Sorbents (Source: Source: Nduka, 2019; Nduka et al., 2019; Cookey et al., 2019)

Sorbent	Pseudo-First Order			Pseudo –Second Order		
	q_e (mg/g)	K_1 (min ⁻¹)	R^2	q_e (mg/g)	K_2 (g/mg/ min)	R^2
Leaves	1.6×10^3	3.73×10^{-2}	0.821	5.0×10^3	5.405×10^{-6}	0.988
Bark	9.2×10^3	3.45×10^{-2}	0.788	3.3×10^3	1.525×10^{-5}	0.996
Root	1.5×10^3	3.89×10^{-2}	0.910	5.0×10^3	5.333×10^{-6}	0.994

Table 6: Kinetic Parameters of Cr (vi) ion Adsorption by Raw Red Mangrove Biomass Sorbents (Source: Source: Nduka, 2019; Nduka et al., 2019; Cookey et al., 2019)

Sorbent	Pseudo-first Order			Pseudo-second Order		
	q_e (mg/g)	K_1 (min ⁻¹)	R^2	q_e (mg/g)	K_2 (g/mg/min)	R^2
Leaves	9.6×10^3	3.52×10^{-2}	0.812	5.0×10^3	8.889×10^{-6}	0.996
Bark	1.3×10^3	3.57×10^{-2}	0.782	3.3×10^3	6.250×10^{-6}	0.994
Root	1.5×10^3	3.55×10^{-2}	0.733	5.0×10^3	4.819×10^{-6}	0.993

Discussion

Surface Morphology of the Adsorbents

SEM images of modified and raw red mangrove leaves, bark, and roots indicate that the modified materials have more porous and irregular structures than the raw sorbents. The images of the raw mangrove sorbents presented

in Figures 1A, B, and C show fewer pores on the surface and are more compact in shape than those found in the modified sorbents. The bark had a second-phase material as a result of acid modification which may have led to its low adsorption. The acid treatment increased the surface area of the adsorbent by opening the pores thereby exposing the binding sites which enhanced the adsorption process.

Adsorption of Cr (vi) on H₂SO₄-Treated and Raw Red Mangrove Biomass Sorbents

Tables 1 and 2 show the adsorption capacities of H₂SO₄-treated and raw red mangrove leaf, bark, and root sorbents at different dosages. As may be seen, the adsorption was influenced by dosage and the type of red mangrove biomass sorbent used. The lower the dosage applied the higher the adsorption capacity obtained. Also, H₂SO₄-treated sorbents generally produced higher adsorption capacities than the raw (untreated) sorbents. This can be a result of greater pores and irregular surfaces observed on the H₂SO₄-treated sorbents as given by the SEM results (Figure 1A, B, C). Furthermore, Table 1 indicates that H₂SO₄-treated red mangrove leaf produced the highest adsorption capacity followed by modified red mangrove root and then modified red mangrove bark as the lowest. In the case of raw red mangrove leaves, bark, and root, Table 2 indicated that the root produced the highest adsorption capacity followed by the leaf and the bark as the least. It can be seen from this comparison that the H₂SO₄-treated sorbents adsorbed Cr (vi) ion more than the raw sorbents and that the leaves and root adsorb Cr (vi) ion adsorbed more than the bark compared to the leaves and root. Raw mangrove bark produced low adsorption capacity which implies that it is not a very good adsorbent for Cr (vi) ion. The comparison also showed that H₂SO₄-treated mangrove adsorbents especially the leaf and root are best for effective and efficient adsorption of dissolved hexavalent Cr ion.

Isotherm Properties of H₂SO₄-Treated and Raw Red Mangrove Sorbents

Langmuir (Equation 3) and Freundlich models (Equation 4) are widely employed to describe the interaction between adsorbent and adsorbate in the adsorption process. These models, establish sorption mechanisms through correlation plots (Isotherm) and are characterized by certain constants whose values express the surface properties of adsorbents (Eren et al., 2009; Cooney et al., 2018). The evaluated adsorption parameters presented in Tables 3 and 4 were deduced from Langmuir and Freundlich isotherm plots. Correlation coefficient (R^2) values of 0.963, 0.887, and 0.806 recorded for H₂SO₄-treated mangrove leaf, bark, and root indicate good linearity with Freundlich. The H₂SO₄-treated mangrove leaf and root show low correlation coefficient values (0.383, and 0.110) indicating non-conformity with Langmuir while H₂SO₄-treated mangrove bark with a correlation coefficient of 0.807 shows good linearity. The results therefore suggest monolayer as well as multilayer adsorption for H₂SO₄-treated mangrove bark and multilayer adsorption or heterogeneous adsorbent surface for the H₂SO₄-treated mangrove leaf and root. Other parameters such as the Freundlich constant, n , indicate the feasibility of an adsorption process. When $n > 1$ it shows that adsorption intensity is favorable (Eren et al., 2009). The n values for H₂SO₄-treated mangrove leaf, bark, and root which were 1.20, 0.70, and 1.20 indicate favorable adsorption. It was observed that H₂SO₄-treated mangrove leaf and root were in the range of $n > 1$, while the bark was between 0 and 1. Therefore, all were in the range of 1 to 10 indicating favorable adsorption. A similar report was made by Prasad and Abdulah (2010) using the fruit shell of *Gulmohar* as an adsorbent.

The evaluated parameters of Langmuir and Freundlich isotherm models for the adsorption of Cr (vi) ion by raw mangrove as presented in Table 4.3. The adsorption data also follows the multilayer adsorption model according to the correlation plots (Figures 4.17 and 4.18) and coefficient (R^2) values which fitted Freundlich better than Langmuir. Furthermore, n also provided information concerning the favourability of adsorption. The values of n for raw mangrove leaf, bark, and root in this study are 0.630, 0.757, and 0.996 respectively. This satisfies the condition of $0 < n < 1$ for favorable adsorption. Tawde and Bhatarao (2010) made the same observation using Neem adsorbent. The isotherm data in Tables 3 and 4 also highlight the influence of the type of sorbent on adsorption. For example, with large values of Langmuir and Freundlich isotherm constants, (K_L and K_F), recorded for the H₂SO₄-treated red mangrove leaves (178.89; 74.67), H₂SO₄-treated red mangrove bark (-41.33; 32.92) and H₂SO₄-treated red mangrove root (145.56; 122.15) and low values recorded for raw red mangrove leaves (-70.00; 5.1310), raw red mangrove bark (-89.67; 9.5017) and raw red mangrove root (76.50; 18.4884), results in Tables 3 and 4 suggest stronger adsorption of Cr (vi) ion by H₂SO₄-treated red mangrove than raw red mangrove.

Kinetic Properties of H₂SO₄-Treated and Raw Red Mangrove Sorbents

The evaluated adsorption kinetic properties of H₂SO₄-treated mangrove sorbents presented in Table 5 are based on the pseudo-first (Equation 5) and pseudo-second (equation 6) order kinetics models. It can be deduced from the results that chemisorption is the rate-determining step in the adsorption of the Cr (vi) ion. However, for H₂SO₄-treated mangrove root, both physisorption and chemisorption could be suggested considering the linearity ($R^2 > 0.91$) recorded in both pseudo-first and -second order kinetics. Similar observations were made by Ofudje et al. (2014) using adsorbent prepared from acid-modified sugarcane. Table 6 shows the correlation coefficient (R^2) of

Cr (vi) ion adsorption onto raw mangrove leaf, bark, and root as 0.812, 0.782, and 0.733 for the pseudo-first-order model and 0.996, 0.994, and 0.993 for a pseudo-second-order model. Again, these results suggest that chemisorption is the rate-determining step in the adsorption of Cr (vi) ion from an aqueous solution. These observations are in agreement with those made by Nameni et al. (2008). In contrast to high and low isothermic constants, K_L and K_F , values recorded for H_2SO_4 -treated and raw sorbents, kinetic pseudo-first and pseudo-second order rate constants, (K_1 and K_2), values recorded for the sorbents are all similarly low. For example, (K_1 and K_2) values (Tables 5 and 6) recorded for H_2SO_4 -treated red mangrove leaves, bark, and root are (3.73×10^{-2} and 5.405×10^{-6}); (3.45×10^{-2} and 1.525×10^{-5}); and (3.89×10^{-2} and 5.333×10^{-6}). Again, (K_1 and K_2) values recorded for raw red mangrove leaves, bark, and root are (3.52×10^{-2} and 8.889×10^{-6}); (3.57×10^{-2} and 6.250×10^{-6}); and (3.55×10^{-2} and 4.819×10^{-6}). This suggests a high Cr (vi) ion adsorption rate by both H_2SO_4 -treated and raw red mangrove sorbents.

Conclusion

This review revealed that the effectiveness of red mangrove biomass sorbent in adsorbing Cr (vi) ion from aqueous solution decreases with the dosage of the biomass sorbent applied. The review has also shown from the surface morphology that H_2SO_4 -treated red mangrove biomass sorbents have more porous and more binding sites than raw sorbents which enhance their adsorption capacity. Isothermic data indicate stronger Cr (vi) adsorption by H_2SO_4 -treated red mangrove than raw red mangrove sorbents fitted well with the Freundlich model for both H_2SO_4 -treated and raw sorbents. Kinetic data indicate a high Cr (vi) adsorption rate by both H_2SO_4 -treated and raw sorbents, fitting the pseudo-second-order model, and suggesting chemisorption for both sorbents. The investigations indicate that red mangrove biomass, especially the H_2SO_4 -treated leaves, and roots, can be used as adsorbents for the treatment of water and wastewater containing Cr (vi) ion.

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