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Removal of Lead (II) and Iron (II) ions from Aqueous Solutions Using Watermelon (Citrillus Lanatus) Peels as Adsorbent

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ABSTRACT

This present work illustrates the adsorption of iron (II) and lead (II) ions from aqueous solutions onto watermelon peels. The research was carried out to assess the possible use of watermelon peels as a low cost adsorbent. The influence of the following factors; solution pH, temperature, contact time and initial metal ion concentration on the adsorption capacity has been studied. The percentage removal of the metals was observed to increase with increase in contact time; 10-40 minutes. The adsorption of the metals decrease with increase in temperature indicated the exothermic nature of the reaction. The percentage sorption of the metal ions onto the adsorbent increased with increase in pH and optimum sorption efficiency occurred at pH 2 for all the investigated metal ions. The result indicated that the watermelon peels prepared could be used effectively for the adsorption of Fe(II) and Pb(II) ions from aqueous solutions. Langmuir and Freundlich isotherms were also studied and the adsorption conformed to both models but Freundlich model fitted better because of the higher values of the correlation coefficients.

Keywords: *Adsorption, Lead (II), Iron (II), Watermelon, Isotherm*

INTRODUCTION

Water pollution raises a great concern nowadays since water constitutes a basic necessity in life and development of industries. Industries such as metal mining operations, fertilizers and paper industries and pesticides have deliberately discharged various types of pollutants into the environment especially in developing countries [1]. The presence of various pollutants such as industrial wastes is continuously discharged into the water system and further affecting our ecosystem due to their lethal effects. Among other issues, water contaminations by heavy metals are more pronounced than other pollutants especially when heavy metals are exposed to the natural ecosystem. 'Heavy metals' refers to any elements with the atomic weights between 63.5 and 200.6 and a specific gravity greater than 5.0. Cadmium, zinc, copper, nickel, lead, mercury and chromium, are some examples of heavy metals which originate from activities of metal plating, mining, battery manufacture, petroleum refining and paint manufacturing [1].

Owing to the toxic and adverse effects of heavy metals, most industries are advised to treat waste waters systematically so that the metal contents can be minimized in their wastes. Various conventional treatments have been applied for removing heavy metals such as chemical precipitation, ion exchange, filtration and electrochemical treatment, but most of these methods are only suitable for large scale treatments and incur high cost to be practiced [2]. Generally, all these treatments lead to certain disadvantages such as incomplete removal of heavy metals, high-energy requirements and production of toxic sludge [2]. Numerous approaches have been studied for the development of more effective methods in removing metal pollution and the adsorption process is found to be more practicable over other techniques. Adsorption process is one of the easiest, safest and more cost-effective methods for heavy metal removal from industrial effluents and this process is already established as a simple operation and an easy-handling process [2]. This work focused on the adsorption of Lead (II) and Iron (II) ions from aqueous solutions using watermelon peels as adsorbent. Experimental parameters utilized include; solution pH, contact time, temperature and initial metal ion concentration, and Langmuir and Freundlich isotherms were used to establish adsorption equilibria.

MATERIALS AND METHODS

Adsorbent Preparation

Watermelon peels were obtained from local fruit market and washed with tap water followed by doubly-distilled water. After thorough washing, watermelon peels were cut into small pieces and dried under sunlight for seven days. The dried watermelon peels were washed repeatedly with hot water (70 ^{o}C) to remove any soluble matter present and then dried in an oven at 85 ^{o}C for 48 hours. The oven dried watermelon peels were pounded using mortar and pestle and sieved through a 250 μm sieve. The watermelon peels granules were stored in air tight polyethylene bottles and used for sorption experiment [3].

Activation of Adsorbent

A 100 g of the screened adsorbent was soaked in excess of 0.3 M trioxonitrate (V) acid, HNO_3 for 24 hours to remove any debris or soluble biomolecules that might interact with the metal ions during the sorption process. This constitutes the chemical activation of the sample. The adsorbent was then filtered through whatman No. 41 filter paper and rinsed first with filtrate and then with deionised water. The rinsed adsorbent was later air dried for 12 hours and stored in a plastic container for further experiment [3].

CHARACTERIZATION OF THE ADSORBENT

Bulk density determination

The tapped density of the adsorbents was determined by a tapping procedure using a 50 mL graduated plastic measuring cylinder [4]. The cylinder was tapped on the work-bench until the volume of the sample stopped decreasing. The mass and volume were recorded and density calculated using equation 1 [5].

$$\rho = \text{Mass/Vol. occupied} \qquad (1)$$

pH determination

The pH value of the prepared adsorbent was determined by socking 1 g of the sample in 100 mL of deionized water and stirring for 1 hour [6].

Preparation of Iron (Fe) Stock Solution

A stock solution of iron (II) (1000 mg/L) were prepared by dissolving 7.019 g of ammonium ferrous sulphate in 0.1 M KNO_3 solutions, KNO_3 served as an electrolyte to control the ionic strength of the metal ion. The stock solution was then diluted to 1000 cm^3 to give 1000 mg/L. Other concentrations of 60, 80, 100 and 120 mg/L from the stock solution were obtained by serial dilution, which was used for sorption experiment.

Preparation of Lead Stock Solution

A stock solution of Lead (II) was prepared by dissolving 1.830 g of lead acetate in 0.1 molar KNO_3. KNO_3 served as an electrolyte to control the ionic strength of the metal ion, the solution was then diluted to 1000 cm^3 to give 1000 mg/L. Other concentrations of 60, 80, 100 and 120 mg/L from the stock solution were obtained by serial dilution, which were used for sorption experiment.

Adsorption Experiment

Biosorption studies were carried out by optimizing the following parameters: solution pH, contact time, temperature and initial metal ion concentration.

EFFECT OF PH

The biosorption experiment was carried out at different pH of the metal ion Fe(II) and Pb(II) solutions in order to investigate the effect of pH on metal uptake. The pHs studied were pH 2, 4, 6 and 8. A 0.25 g of the adsorbent was agitated with 15 mL of 100 mg/L at 500 rpm for 20 min at 30 °C temperatures. The pH adjustment was done using 0.1 M sodium hydroxide NaOH and 0.1 molar hydrochloric acid. The separation of the adsorbent and solution was carried out by filtration with whatman filter paper No 42 and the filtrate stored in sample bottles in a refrigerator prior to analysis. The residual metallic ion concentrations were determined using an Atomic Absorption Spectrophotometer (AAS).

Effect of Initial Metal Ion Concentration

The effect of initial metal ion concentration on percentage sorption of metal ions (Fe(II) and Pb(II)) were investigated for optimizing the initial metal ion concentration, 60, 80, 100 and 120 mg/L. A 0.25 g of adsorbent was shaken at 500 rpm with 15 mL of each of the investigating concentrations.

The temperature was maintained at 30 °C. the separation of the adsorbent and solution was carried out using whatman filter paper No. 42 and the filtrate stored in sample bottles in a refrigerator prior to analysis. The residual metal ion concentrations were determined using an Atomic Absorption Spectrophotometer (AAS).

Effect of Contact Time

The effect of contact time on percent sorption of metal ion Fe(II) and Pb(II)) was investigated over contact time of 10, 20, 30 and 40 minutes. A 0.25 g of adsorbent was shaken at 500 rpm with 15 mL of 100 mg/L sorbate concentration. The temperature was maintained at 30 ^{o}C. The separation of the adsorbent and solution was carried out by filtration with whatman filter paper No. 42 and the filtrate stored in sample bottles in a refrigerator prior to analysis. The residual metal ion concentrations were determined using an Atomic Absorption Spectrophotometer (AAS).

Effect of Temperature

The biosorption experiment was carried out at different temperature of metal ions; Fe(II) and Pb(II) solutions in order to investigate the effect of temperature on metal uptake. The temperatures studied were 10, 20, 30 and 40 °C. The solutions were agitated with 15 mL of 100 mg/L of the solution at 500 rpm for 30 minutes at each of the investigating temperature. The separation of the adsorbent and solution was carried out using whatman filter paper No. 42 and the filtrate stored in sample bottles in a refrigerator prior to analysis. The residual metal ion concentrations were determined using an Atomic Absorption Spectrophotometer (AAS).

CALCULATION OF UPTAKE CAPACITY FOR EACH SAMPLE

The uptake capacity for each sample was calculated as follows;

$$\text{qe} = \frac{(\text{Co} - \text{Ce})\text{v}}{\text{m}} \qquad (2)$$

Where;

Co= $initial\ metal\ ion\ concentration\ \frac{mg}{L}$

Ce= $the\ equilibrium\ metal\ ion\ concentration \frac{mg}{L}$

V = $volume\ of\ the\ solution\ in\ L$

m = $weight\ of\ the\ adsorbent\ used\ in\ g$

CALCULATION OF PERCENTAGE SORPTION FOR EACH SAMPLE

The extent of sorption in percentage was calculated using the equation;

$$\%\ \text{sorption} = \frac{\text{Co} - \text{Ce}}{\text{C}_\text{o}} \times 100 \qquad (3)$$

Where;

C_o = initial metal ion concentration (mg/L)

C_e = final metal ion concentration (mg\L)

RESULTS AND DISCUSSION

Characteristics of Watermelon Peels

Bulk density is an essential parameter because it determines how many grams of adsorbent that can be contained in a filter of a given solid capacity and how much treated liquid is retained by the filter cake. Adsorbent with adequate density also helps to improve the filtration rate by forming an even cake on the filter surface [7]. The America Water Work Association had set a lower limit on bulk density at 0.25 g/cm^3 for granular activated adsorbent to be of practical use [8]. The bulk density of the activated adsorbent used for this work is within that limit, which is 0.33 g/cm^3.

The activated adsorbent considered in this study was observed to be acidic with pH value of 3.3. The acidic or basic nature of an activated adsorbent depends on its preparation, inorganic matter and chemically active oxygen group on its surface as well as the kind of treatment to which the activated adsorbent was subjected [7].

Table1. *Characteristics of Adsorbent*

Characteristic	**Value**
Bulk density	0.33 g/cm^3
pH	3.32

Effect of Different Parameters on Adsorption

Effect of pH

The pH of the solution is an important parameter affecting adsorption of heavy metal ions. The effect of pH on the adsorption of Fe(II) and Pb(II) ions by watermelon peels is as shown in figure 1. The maximum removal efficiency for Fe(II) is 89.03 and for Pb(II) 95.74 all at pH 8.

At low pH value, the hydrogen ions concentration was high and therefore, protons can compete with the metal ions for surface sites. At low pH values, the metals are present as Fe^{2+} and Pb^{2+} free cations. When pH increased, there was a decrease in positive surface charges due to the deprotonation of the sorbent functional group which resulted to a lower electrostatic repulsion between the positively charged metal ions and the surface of the adsorbent [9, 10, 11].

As pH value was higher, more exchangeable cations contained in the adsorbent can be exchanged with metal ions due to weak competitive adsorption of hydrogen ions.

Therefore, biosorption of meal ions increased with increase in pH [12].

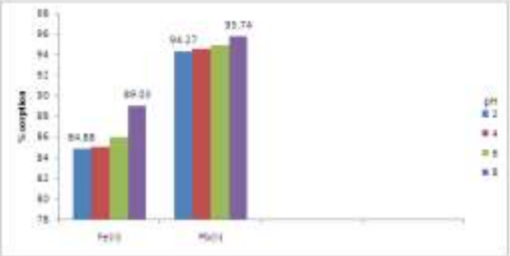

Fig1. *Effect of pH on Percentage Sorption by Watermelon Peels*

Effect of Initial Metal ion Concentration

The rate of adsorption is a function of initial concentration of metal ions. Figure 2 indicated that higher adsorption was found to take place at lower concentrations. This may be due to the interaction of all metal ions present in the solution with binding sites. At higher concentrations, more metal ions are left unadsorbed in solution due to saturation of adsorption sites [9, 13, 14]. The number of ions adsorbed from a solution of higher concentrations is more than that removed from less concentrated solutions.

According to Senkumar *et al*. [15], at lower metal ions concentration, the percentage uptake was higher due to larger surface area of adsorbent being available for adsorption. When the concentration of metal ions became higher, the percentage removal decreased since the available sites for adsorption became less due to saturation of adsorption sites. At a higher concentration of metal ions, the ratio of initial number of moles of metal ions to the adsorption sites available was higher, resulting in lower adsorption percentage.

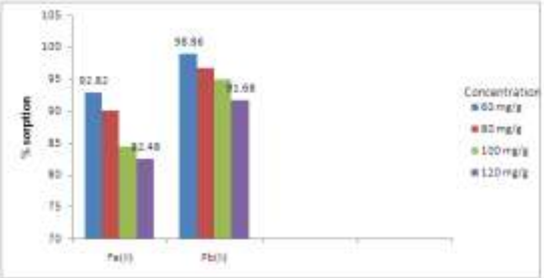

Fig2. *Effect of Initial Metal ion Concentration on Percentage Sorption by Watermelon Peels*

Effect of Temperature

The data presented in figure 3 below showed that the adsorption of metal ions by the watermelon peels increased with increase in temperature which is typical for biosorption of many metal ions from their solution [16]. At high temperature, the thickness of the boundary layer decreases due to the increased tendency of the metal ion to escape from the biomass surface to the solution phase which resulte to a decrease in adsorption as temperature increased. The decrease in adsorption with increasing temperature suggests weak adsorption interaction between adsorbent surface and the metal ion which supports Physisorption [17].

According to Giles classification as reported by Vinod [18] and Anirudhan [19], the adsorption isotherm at 50-60 °C belongs to sub-group II while above 70 °C is sub-group I. In this investigation, temperature of 10, 20, 30, and 40 °C in figure 3 tend to define a plateau, therefore, it seems reasonable to support the proposal that for the experiment condition used, the formation of a complete mono layer covering the adsorbent surface belongs to group III, meaning that saturation of the adsorbent surface seemed to be reached at 10-40 °C and the optimal temperature of adsorption for the investigated metal ion could be within this range.

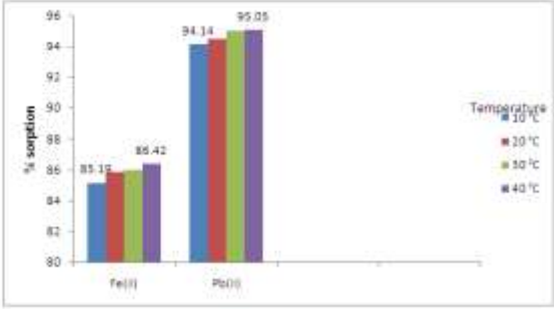

Fig3. *Effect of Temperature on Percentage Sorption by Watermelon Peels*

Effect of Contact Time

The effect of contact time on the adsorption efficiency is as shown on figure 4. Adsorption experiments were carried out at different time intervals, 10, 20, 30 and 40 minutes in the individual metal ions; Fe (II) and Pb(II). It was observed that adsorption of the tested metal ions was optimum within a short period of 30 minutes. Adsorption of Fe (II) and Pb(II) increased with increase in contact time. Generally, the observed trend of metal removed was Pb>Fe. Previous results revealed that removal of all tested metals was rapidly removed within a short period of 30 minutes [20]. The effects of contact time on adsorption process of metal ions from wastewaters were studied by many authors [21, 22, 23].

In addition, Lead with smaller ionic size 1.80 Ǻ was observed to have been adsorbed at faster rate than Iron. This may be explained by considering the ionic radius of Lead being lower, with higher rates of removal at equilibrium than Iron (2.48 Ǻ). This was observed on other biological adsorbents as reported earlier [24]. Thus, the smaller the ionic sizes, the greater the affinity to active sites, and hence the metal ions with smaller ionic radii diffuse faster through the adsorbent pores than the bulkier ones [12].

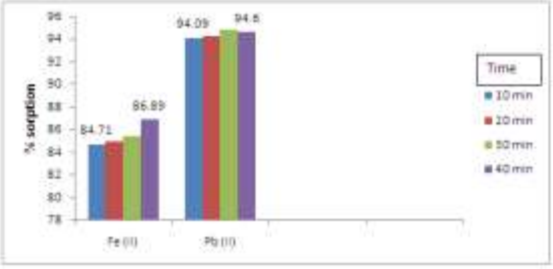

Fig4. *Effect of Contact Time on the Percentage Sorption by Watermelon Peels*

Adsorption Isotherms

The adsorption isotherms indicate how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches equilibrium state [25]. Since the adsorption isotherms are important to describe how adsorbates will interact with adsorbents and so are critical for design purposes; therefore, the correlation of equilibrium data using an equation is essential for practical adsorption [12]. In this study, adsorption data were fitted with both Langmuir and Freundlich isotherms. The general form of Freundlich isotherm model is given by:

$$q_e = KC_e^{1/n} \quad (4)$$

The line arised form of equation 3 is given by:

$$\log q_e = \log k + 1/n \log C_e \quad (5)$$

Where qe = amount adsorbed at equilibrium (mg/g),

Ce = equilibrium concentration of the adsorbate (mg/L),

K and n = Freundlich constants related to adsorption capacity and adsorption intensity respectively.

When log q_e was plotted against log C_e, straight lines were obtained (Fig. 5-6). K and 1/n were also determined from intercept and slope of the isotherm respectively as presented in Table 2. The K values are in the following order: Pb(II) (3.906)> Fe(II) (2.115) confirming the order of adsorptivity Pb(II)>Fe(II).

Since the values of 1/n for almost all the metal ions Fe(II), Pb(II), are less than 1, this indicates favourable adsorption. A smaller value of 1/n indicates favorable adsorption mechanism and formation of relatively stronger bond between the adsorbate and adsorbent.

The correlation coefficient (R^2) as indicated in Table 2 with R^2 values greater than 0.9 for all the metal ions also depict a good fit for Freundlich adsorption isotherm [25].

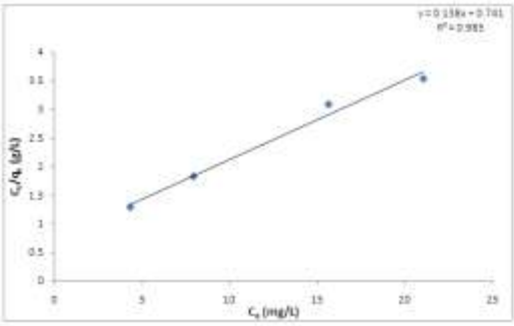

Fig5. *Langmuir Plot for the Adsorption of Fe(II)*

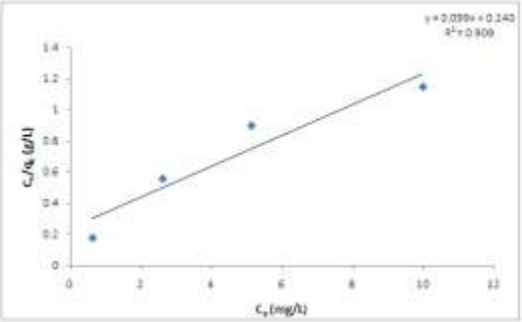

Fig6. *Langmuir Plot for the Adsorption of Pb(II)*

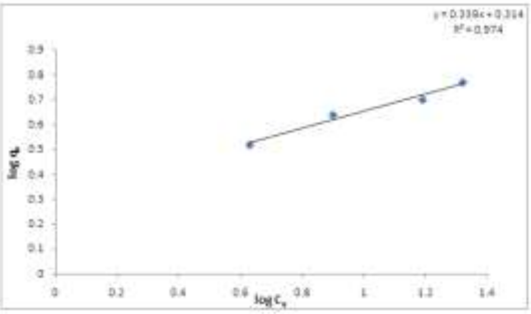

Fig7. *Freundlich Plot for the Adsorption of Fe(II)*

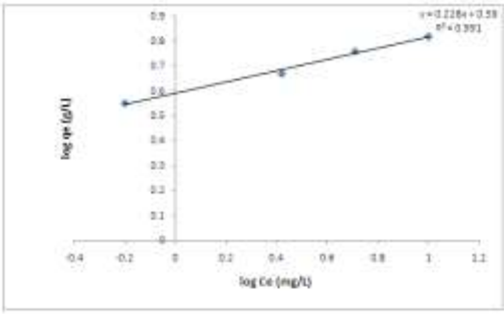

Fig8. *Freundlich Plot for the Adsorption of Pb(II)*

Table2. *Isotherm parameters for the adsorption of Fe (II) and Pb(II)*

Heavy metal	Langmuir model				Freundlich model		
	R^2	Q (mg/g)	b (L/mg)	R_L	R^2	K	1/n
Fe(II)	0.985	7.246	0.186	0.082	0.974	2.115	0.339
Pb(II)	0.909	10.101	0.413	0.039	0.991	3.906	0.228

The Langmuir adsorption isotherm is represented by the following equation:

$$q_e = \frac{Qb\,C_e}{1+b\,C_e} \qquad (6)$$

The linear from of the Langmuir isotherm is given by:

$$C_e/q_e = \frac{1}{Qb} + \frac{1}{Q}C_e \qquad (7)$$

Where C_e = the equilibrium concentration of the adsorbate (mg/L),

q_e = The amount of the adsorbate adsorbed per unit mass of adsorbent (mg/g),

Q and b = Langmuir constants related to adsorption capacity and rate of adsorption respectively.

When C_e/q_e was plotted against C_e, straight lines were obtained (Fig. 7-8), indicating that the adsorption of the metal ions Fe(II) and Pb(II) followed the Langmuir isotherm. The Langmuir constants Q and b were determined from the slopes and intercepts of the plots, and are reported in Table 2. Pb(II) ion had the greater maximum adsorption capacity (Q) of 10.01 mg/g compared to Fe(II) 7.25 mg/g. Pb(II) ion had the higher rate of adsorption b (0.413) than Fe(II) (0.19). The correlation coefficients (R^2) of all the metal ions are high > 0.9, also confirming that the sorption of the metal ions by the watermelon peels followed the Langmuir isotherm as reported in the literature.

The essential characteristics of Langmuir isotherm may be expressed in terms of dimensionless separation parameter, R_L, which is indicative of the isotherm shape that predicts whether an adsorption system is favorable or unfavorable.

R_L is defined as:

$$R_L = \frac{1}{1+bC_o} \qquad (8)$$

Where b = Langmuir constant (L/mg), and C_o = Optimum initial concentration of the adsorbates (mg/L).

The value of R_L indicates the type of the isotherm to be either unfavorable ($R_L > 1$) linear ($R_L = 1$), favorable ($O < R_L < 1$), or irreversible ($R_L = O$). The R_L values for the metal ions Fe (II) and Pb (II) were found to be less than 1 as reported in Table 2 and confirmed that the watermelon peels adsorbent is favorable for the adsorption of metal ions.

CONCLUSION

Watermelon peels could be used as a very effective low-cost adsorbent for the removal of Fe (II) and Pb (II) ions from aqueous solution considering the maximum percentage removal of the metal ions studied through the various adsorption experiments. Batch studies on Iron and lead removal showed significant effects of the variables: contact time, temperature, pH and initial metal ion concentration. The results provide a good indication of the different operating conditions that would be required for efficient removal of each heavy metal from aqueous solutions. Both Langmuir and freundlich isotherm model ($R^2 \approx 1$) were in good agreement with the experimental data but conforms better to Freundlich model with higher value of the correlation coefficient.

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