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Effects of concentration and pH on the potential of a locally sourced activated carbon from *Balanites Egyptiaca* stems on the adsorption of Fe²⁺ ions on the potential

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Abstract

Activated carbon was generated by the carbonization of *balanites egyptiaca* stems at 700°C for 5 hours. The carbonized stems were cooled for 24 hours, after which they were activated at 130°C using ZnCl₂ solution. The activated carbon obtained was purified and dried at 100°C. For the investigation of the effect of concentration, 0.30g, 0.40g, 0.50g, 0.60g and 0.70g each of the activated carbon was used in 20.00cm³ aqueous solution of FeSO₄.7H₂O; for the investigation of the effect of pH, 0.60g of the activated carbon was added to each of the aqueous buffer solutions of FeSO₄.7H₂O of pH values: 5.00, 6.00, 7.00, 8.00 and 9.00 and ; for the analysis of the adsorption isotherm of the process, the equilibrium data obtained at different concentration of the activated carbon used were analysed using Langmuir isotherm model. The work showed that the amounts of Fe²⁺ ions adsorbed, q_e (mg/g) were 1.48, 4.45, 6.67, 8.52 and 6.31 using 0.30g, 0.40g, 0.50g, 0.60g and 0.70g of the activated carbon, respectively; the amount of Fe²⁺ adsorbed, q_p (mg/g) at different pH were 8.58, 8.54, 8.53, 6.92 and 6.19 from aqueous solutions of pH 5.00, 6.00, 7.00, 8.00 and 9.00, respectively and ; the maximum adsorption capacities, q_m (g/g) x 10⁻³ of the activated carbon were 1.60, 4.87, 7.42, 9.65 and 6.70, for 0.30g, 0.40g, 0.50g, 0.60g and 0.70g, respectively. The work also showed that the adsorption isotherm of the process was favourable and monolayer on the surface of the adsorbent (activated carbon).

Keywords: *Balanites egyptiaca*; activated carbon; concentration; pH; adsorption

Introduction

Advances in science and technology have brought tremendous progress in many areas in our day to day activities and, on the other hand, it brought a lot of environmental hazards due to very little attention paid to the treatment of industrial effluents (Ebiekpe *etal*, 2014). In addition to industrial effluents, there are other sources from which environmental hazards (pollutions) occurred, they include agricultural activities, where pesticides, herbicides, fungicides, fertilizers e.t.c are used; automechanic and battery chargers workshops, where there may have been metallic waste products deposits and; metal fabricators workshops, where metals of different kinds are deposited into the environment, all these present variety of health hazards (Bello 1999). Pollution by heavy metals in the environment has become major threat to plants, animals and humans due to their bioaccumulation and toxicity and, therefore, must be removed from municipal and industrial effluents before discharge (Yakasai *etal*, 2015).

The conventional methods for heavy metals removal from water and wastewater include: precipitation, waguulation/flocculation, ion exchange, electrolysis, solvent extraction, electroplating, evaporation, oxidation, reduction membrane separation and reversed osmosis. However, these methods are often prohibitively cost and non renewable with inadequate efficiency at low metal concentration (Ebiekpe *etal*, 2014; Gardea *etal*, 1998). The prohibitive nature of the conventional methods for heavy metals removal in terms of high cost and low efficiency led to the search for alternative methods for heavy metals removal that would be cheap, efficient and environmentally-friendly.

In recent time, the use of adsorption method in which natural adsorbents from the available resources in the environment (locally sourced) are used for adsorbing of heavy metals is now attracting the attention of most researchers. For instance, Yakasai *etal* (2015) used activated

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carbon prepared from ground nut husk for heavy metal removal at varying temperatures; Ebiekpe *et al* (2014) studied the potentials of activated carbon generated from plantain suckers for the removal of Cu^{2+} from aqueous solution; Aman *et al* (2008) used potato peels for the removal of Cu^{2+} from waste water; Annadurai *et al* (2003) investigated the potentials of banana and orange peels as heavy metals adsorbent; e.t.c.

Adsorption is a process in which a component (adsorbate) is taken up (removed) from a solution by a stationary phase (adsorbent) via some physical or chemical interactions. If the adsorbate is held on the surface of the adsorbent by physical intermolecular forces, the process is called physical adsorption and; if the adsorbate formed chemical (covalent) bond with the surface of the adsorbent, the process is called chemisorption (Bayawa, 2000).

This paper reports studies on the effects of concentration of a locally sourced activated carbon from *balanites egyptiaca* and pH of aqueous solution of metallic ions on the adsorption of metallic ions (Fe^{2+}) from its aqueous solution. The paper also reports studies on the analysis of adsorption isotherm of the process at different concentrations of activated carbon using Langmuir isotherm model.

Materials and Methods

Collection and Treatment of the Experimental Sample

Balanites egyptiaca stems were collected from Hura grazing land, along kano-katsina high way in Dawakin – Tofa L.G.A., Kano State Nigeria. The stems were fresh and mature at the time of collection. The stems were chopped into logs of desired sizes and sun-dried for 2 weeks.

Chemicals / Reagents

The chemicals/reagents used were iron(ii)sulphate(vi)heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), concentrated tetraoxosulphate(vi)acid (H_2SO_4), disodium hydrogen phosphate(v) (Na_2HPO_4) and sodium dihydrogen phosphate(v)monohydrate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$). All the chemicals/reagents were analytical reagent (AR) obtained from British drugs house (BDH). The chemicals/reagents were of good purity, hence they were used directly without any purification.

Preparation of 0.05M $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (13.90 g/dm³)

For the preparation of 0.05M $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 13.90g of the salt was first dissolved in a small quantity of distilled water and 15.00cm³ of concentrated H_2SO_4 (as dehydrating agent) was added. The resulting solution obtained was subsequently diluted to 1dm³ using distilled water in a 1dm³ volumetric flask.

Preparation of 0.20M Buffer Solution of Different pH Values

In order to adjust the pH of 0.50M $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution, the Henderson-Hasselbalch equation was used (Ekwenchi and Yaro, 2013). The equation is

$$\text{pH} = \text{pKa} + \log \frac{(\text{base})}{(\text{acid})} \text{-----} (1)$$

For a buffer solution of any molar concentration and pH to be formed, the molar concentration of the acid component must be equal to the molar concentration of the base

component, hence [acid] = [base] as reported by Ekwenchi and Yaro (2013).

The pKa of the buffer salts [$\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ (as an acid) and Na_2HPO_4 (as a base)] was first determined from the pKa value of the buffer salts (i.e 7×10^8) using the relation,

$$\text{pKa} = - \log \text{Ka} \text{-----} (2)$$

The pKa obtained from equation (2) and the required pH value of the buffer solution to be prepared were then used in equation (1), from which the volume of acid (V_A) and that of base (V_B) were determined and subsequently mixed and formed a buffer solution of required pH (Ekwenchi and Yaro, 2013).

Production of Activated Carbon

The activated carbon used for the research was produced by the carbonization of the logs of sun-dried stems of *Balanites egyptiaca* in muffle furnace at 700°C for 5 hours, where char was produced. The char obtained was cooled overnight, after which it was activated by soaking in 1.00M ZnCl_2 solution and heated at 130°C for 3 hours (Yakasai *et al*, 2015). The activated char (i.e activated carbon) was filtered using Whatman filter paper (18.5cm), washed several times with distilled water and dried in an oven at 100°C. The oven-dried activated carbon was finally crushed and sieved with 120µm mesh.

Effect of Concentration of the Activated Carbon on the Adsorption of Fe^{2+} Ions

Different masses of the activated carbon (0.30g, 0.40g, 0.50g, 0.60g and 0.70g) were placed in five (5) different boiling tubes, which were labeled A,B,C,D and E, respectively. To each of A,B,C,D and E, 20.00cm³ of 0.05M aqueous solution of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was added, where solution mixtures of activated carbon in aqueous $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ of different mass concentrations (15.00 g/dm³, 20 g/dm³, 25 g/dm³, 30 g/dm³, and 35 g/dm³), were respectively formed. The adsorption mixtures in the boiling tubes were uniformly and continuously agitated for equilibrium time of 2 hours on a vibrator, after which the content of each boiling tube was filtered using Whatman filter paper (18.5cm). The concentration of the metal ions (Fe^{2+}) in each filtrate was determined using HGA850, USA, atomic adsorption spectrophotometer (AAS) (Ebiekpe *et al*, 2014). Prior to the analysis, the AAS was set at 0.00mg/dm³ and stabilized for 10 minutes.

The amount of Fe^{2+} adsorbed at equilibrium time of 2 hrs, q_e (g/g) for each concentration of activated carbon was evaluated using the following equation as adopted by Ebiekpe *et al* (2014)

$$q_e = \frac{[(C_0 - C_e)V]}{W} \text{-----} (3)$$

where C_0 (g/dm³) = concentration of aqueous $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ used

C_e (g/dm³) = concentration of Fe^{2+} in aqueous solution

V (cm³) = volume of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution used

W (g) = Weight of activated carbon used,

Effect of pH of Aqueous $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ Solution on the adsorption of Fe^{2+} ions

For the investigation of the effect of pH of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ on the adsorption of Fe^{2+} , 0.60g of the activated carbon was dissolved in 20.00cm³ of buffered $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solutions of different pH values: 5.00, 6.00, 7.00, 8.00 and 9.00 in five

(5) boiling tubes, which were labeled A,B,C, D, and E, respectively. The adsorption mixtures were uniformly and continuously agitated for 2 hours on a vibrator, after which the content of each boiling tube was filtered off using Whatman filter paper. Concentration of Fe²⁺ in the filtrate for each boiling tubes was determined using HGA850, USA, atomic adsorption spectrophotometre (AAS) (Ebiekpe *etal*, 2014). Prior the analysis, the AAS was set at 0.00mg/dm³ and stabilized for 10 minutes. The amounts of Fe²⁺ adsorbed at equilibrium time of 2 hours, for each pH q_{ep} (g/g) was evaluated using the modified form of equation (3). The equation is q_{ep} = [(C_o - C_{ep})V]/ W ----- (4)

Where C_{ep} = concentration of Fe²⁺ in the aqueous FeSO₄.7H₂O at equilibrium time of 2 hours for a particular pH

Analysis of Adsorption Isotherm

The equilibrium data collected at different concentration activated Carbon were analysed using Langumur Isothem model (equation 5) where the measure the maximum adsorption capacity of the adsorbent, q_m (g/g) was evaluated while equation (6) was used for the evaluation of the essential characteristics of the Langumuir isotherm, which was expressed in terms of dimensionless constant of separation factor, R_L.

$$\frac{1}{q_e} = \left(\frac{1}{Ka q_m} \right) x \frac{1}{C_e} + \frac{1}{q_e} \text{----- (5)}$$

$$R_L = \left(\frac{1}{1 + KaCAC} \right) \text{----- (6)}$$

The constant Ka in equations (5) and (6) is a constant related to energy of adsorption = l/mg, which indicates that the adsorption of Fe²⁺ is monolayer adsorption on the surface of the adsorbent while R_L is an indicator of the shape / nature of the adsorption isotherm of the process; if R_L > 1, the adsorption isotherm is unfavorable; if R_L = 1, the adsorption isotherm is linear, if R_L = 0, the adsorption isotherm is irreversible and; if 0 < R_L < 1, the adsorption isotherm favourable (Ebiekpe *etal*, 2014).

Results and Discussion

Results

The result of all the experiments and analyses in this work are shown in the Table 1 – 3 and depicted on Figs 1 – 3. Table 1 shows the different concentrations of activated carbon used (CAC), the initial concentration, Co (g/dm³) of FeSO₄.7H₂O used, the equilibrium concentration, Ce (g/dm³) of the metal ions (Fe²⁺) remained in the aqueous solution after adsorption and the amount of metal ions adsorbed, q_e (g/g) by different concentrations of activated carbon (CAC) . Table 2 gives the pH of FeSO₄.7H₂O, the Co (g/dm³) of the activated carbon the equilibrium concentration, C_{ep} (g/dm³) of the metal ions remained in the aqueous solution after adsorption and, the amount, q_{ep}

(g/g) of the metal ions adsorbed by different concentrations of activated carbon (CAC) at different pH of FeSO₄.7H₂O. Table 3 gives the results of the analysis of adsorption isotherm for the equilibrium data obtained at different concentration of activated carbon.

Fig 1 shows the pattern of adsorption of the Fe²⁺ by the activated carbon at different concentrations. Fig 2 depicts the effect of pH of the FeSO₄.7H₂O on the adsorption of Fe²⁺ by the activated carbon at different concentrations. Fig 3 shows the shape / nature of the adsorption isotherm based on the values of dimensionless constant of separation factors (R_L) at different concentrations of activated carbon.

Table 1: Effect of Concentration of Activated Carbon (CAC) on the Adsorption Fe²⁺ at Equilibrium Time of 2 Hours.

Parameter	Concentrations				
CAC (g/dm ³)	15.00	20.00	25.00	30.00	35.00
Co (g/dm ³)	13.90	13.90	13.90	13.90	13.90
Ce (g/dm ³)	12.79	10.56	8.90	7.51	9.17
q _e (g/g) x 10 ⁻³	1.48	4.45	6.67	8.52	6.31

Table 2: Effect of pH of FeSO₄.7H₂O on the Adsorption of Fe²⁺ at Equilibrium Time of 2 Hours

Parameters	pH of FeSO ₄ .7H ₂ O and corresponding concentrations				
pH	5.00	6.00	7.00	8.00	9.00
CAC (g/dm ³)	0.60	0.60	0.60	0.60	0.60
Co (g/dm ³)	13.90	13.90	13.90	13.90	13.90
Ce (mg/dm ³)	1.03	1.09	1.11	2.52	4.62
q _{ep} (g/g) x 10 ⁻³	8.58	8.54	8.53	6.92	6.19

Table 3: Analysis of Adsorption Isotherm at Different Concentrations of Activated Carbon

Parameters	concentrations of activated carbon and the evaluated quantities				
CAC (g/dm ³)	0.30	0.40	0.50	0.60	0.70
q _m (g/g) x 10 ⁻³	1.60	4.87	7.42	9.65	6.70
R _L	0.77	0.71	0.67	0.63	0.59

Discussion

The effect of concentration of the activated carbon on the adsorption of Fe²⁺ from the aqueous solutions of FeSO₄.7H₂O is shown in Table 1 and depicted on Fig 1. From the table , it could be seen that the amount of Fe²⁺ adsorbed, q_e(g/g) increases with increase in the concentration of the activated carbon (CAC) from 15.00 g/dm³ to 30.00 g/dm³, after which it declined at 35.00 g/dm³. The increase in the amount of Fe²⁺ adsorbed within the stated concentration range may be attributed to the sufficient adsorption sites available on the surface of the adsorbent (Meena *etal*, 2005). On the other hand, the low adsorption of Fe²⁺ observed at 35.00 g/dm³ may be attributed to the high solute (activated carbon) content in the solution mixture, which led to low mobile phase (solvent), which will convey the Fe²⁺ from FeSO₄.7H₂O to the surface of the stationary phase (activated carbon) in the solution mixture (Bayawa, 2000).

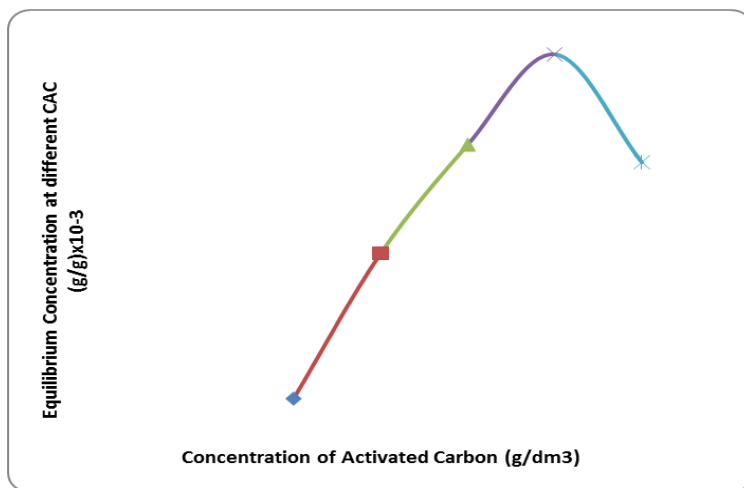


Fig. 1: Effect of Concentration of Activated Carbon on the Adsorption of Fe²⁺ from the Solution

Table 2 shows the effect of pH of FeSO₄.7H₂O on the adsorption of Fe²⁺ by the activated carbon, which was also presented on Fig 2. The result showed that the amount of Fe²⁺ adsorbed q_{ep} (g/g) decreases with the increase in the pH of FeSO₄.7H₂O. The high adsorption observed at relatively low pH may be connected to acidic nature of the medium (low pH of the solution mixture), which affects the surface charge and degree of ionization of the activated carbon (Ebiekpe *etal*, 2014), which resulted in the attraction of Fe²⁺ from FeSO₄.7H₂O by the opposite charges on the surface of the adsorbent. The curve of Fig 2 also evident that the adsorption of Fe²⁺ from the solution mixture decreased with the increase in the pH of FeSO₄.7H₂O. From the figure, it could also be seen that the amounts of Fe²⁺ adsorbed q_{ep} (g/g) were high and, almost the same at pH range of 5.00 – 7.00. This indicated

that the adsorption was high at acidic and neutral media while at alkaline medium (pH range 8.00 – 9.00), the adsorption was comparatively low. The high adsorption observed at pH values: 5.00 and 6.00 could be associated with the increase in the surface charge and degree of ionization of the activated carbon at relative low pH of FeSO₄.7H₂O (Ebiekpe *etal*, 2014); the high adsorption of Fe²⁺ at pH 7.00 may be connected to the neutral nature of the medium within which the activated carbon was added, which maintained the actual nature of the activated carbon due common ions effect and; the low adsorption of Fe²⁺ observed at pH values: 8.00 and 9.00 could be connected to common ion effect between the charges in the solution mixture, which may lead to interfered of the adsorption of Fe²⁺ on the surface of the adsorbent (Meena *etal*, 2003).

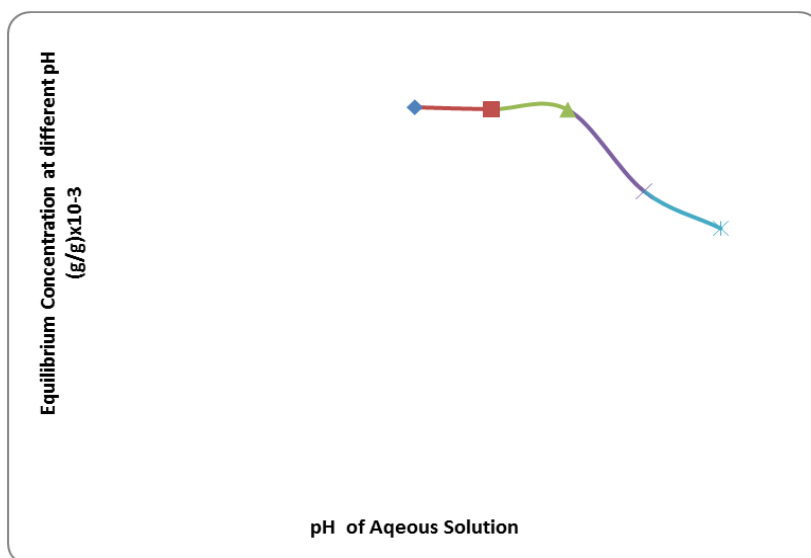


Fig. 2: Effect of pH of Aqueous Solution on the Adsorption of Fe²⁺ by Activated

The results obtained from the analysis of adsorption of isotherm are shown in Table 3 and, depicted on Fig .3. The results showed that the maximum adsorption capacity of the activated carbon, q_m (g/g) increases with the increase in the concentration of the activated carbon from 15.00 g/dm³ to 30.00 g/dm³ and, declined at 35.00 g/dm³. The high q_m within the stated concentration range (15.00 g/dm³ to 30.00 g/dm³) may be attributed to the sufficient adsorption sites available on the surface of the adsorbent (Meena *etal*, 2005). In contrast, the decrease in q_m (g/g) at 35.00 g/dm³ may be associated with the saturation of the solution mixture by the activated carbon, which led to low mobile phase (solvent), that will carry Fe²⁺ in the solution from FeSO₄.7H₂O to the solute (activated carbon) which was used as stationary phase (Bayawa, 2000). The results also showed that the R_L value for each CAC was greater than 0.00, but less than 1.00. this indicated that the adsorption isotherm was favourable (Ebiekpe *etal*, 2014).

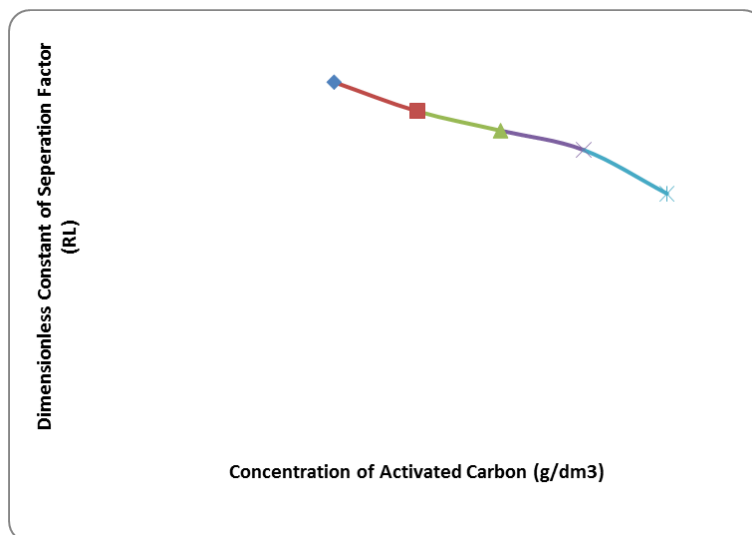


Fig. 3: Analysis of Adsorption of Isotherm at Different Concentrations of Activated Carbon

The curve of Fig. 3 also shows that the value of dimensionless constant of separation factor (R_L) for all the CAC were in accordance with the expression of favourable adsorption isotherm (i.e $0 < R_L < 1$) as reported by Ebickpe *etal* (2014).

Conclusion

The use of a locally sourced activated carbon from *balanites egyptiaca* stem as metallic ions adsorbent was established. From the findings of this work, it could be concluded that the adsorption of metallic ions using activated carbon at characteristic concentrations and pH was favourable and monolayer on the surface of the activated carbon; the adsorption of the metallic ions from the aqueous solution increases with the increase in the concentration of activated carbon to a certain extent and drastically decreases at extremely high concentration; an increase in the pH of aqueous solution brings about the decrease in the amount of metallic ions to be adsorbed and; the maximum adsorption capacity of the activated carbon was found to be high in acidic and neutral media and, low in alkaline medium.

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