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Comparative Study on the Adsorption Kinetics and Thermodynamics of Metal Ions onto Acid Activated Low Cost Pandanus Carbon

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Abstract: Batch experiments were carried out for the sorption of Copper and Ferrous ions onto acid activated carbon prepared. The operating variables studied were initial metal ion concentration, pH, and temperature and contact time. The equilibrium data were fitted to the Langmuir and Freundlich isotherm equations. From this adsorption efficiency, adsorption energy, adsorption capacity, intensity of adsorption and dimensionless separation factor were calculated. From the kinetic studies the rate constant values for the adsorption process was calculated. From the effect of temperature thermodynamic parameters like ΔG° , ΔH° , and ΔS° were calculated. The mechanism of adsorption for metal ions onto carbon was investigated by using the experimental results and confirmed by FT- IR, XRD and SEM images.

Keywords: Adsorption Kinetics, Pandanus Carbon, Langmuir and Freundlich isotherm equations.

Introduction

The presence of heavy metals in the environment is one of the major concerns because of their toxicity and threat to human life and the environment. Wastewater from industries released into nearby land or rivers without any treatment because the conventional treatment methods are not cost effective in the Indian context¹. On the other hand, low cost technologies never allow a wishful metal ion removal and it has certain disadvantages. Adsorption is one of the most effective methods and activated carbon is the preferred adsorbent widely employed to treat wastewater containing different metal ions, recognizing the economic drawback of commercial activated carbon. Many investigators have studied

the feasibility of using inexpensive alternative materials like pearl millet husk, date pits, saw dust buffing dust of leather industry, coir pith, crude oil residue tropical grass, olive stone and almond shells, pine bark, wool waste, coconut shell etc., as carbonaceous precursors for the removal of metals from water and wastewater^{2,3}. The present study undertaken to evaluate the efficiency of a carbon adsorbent prepared from acid activated Pandanus carbon for removal of metal ions in aqueous solution. In order to design adsorption treatment systems, knowledge of kinetic and mass transfer processes is essential. In this paper, we report the applicability of kinetic and mass-transfer models for the adsorption of Copper and Ferrous ion onto activated carbon.

Experimental

Dried stem of *Pandanus* plant were carbonized with concentrated sulphuric acid in the weight ratio of 1:1(W/V). The carbonization and activation was completed by heating for twelve hours in a furnace at 400 °C. The resulting carbon washed with distilled water until a constant pH of the slurry reached. Then the carbon has dried for four hours at 100 °C in a hot air oven. The dried material was ground well to a fine powder and sieved

All Chemicals used were of high purity, commercially available AnalaR grade. Stock solutions of 1000 mg / L of metal ions were prepared using doubly distilled water.

Metal ions were made to a known concentration. From that, 50 ml of 5,10,15,20,25 and 30mg/L metal ion solutions have taken and added with the 100mg of activated carbon adsorbent. Then these were agitated at 30, 40, 50, and 60 °C in a defined time intervals. After that the samples were withdrawn from the shaker and filtered. Then they were analyzed in UV-Visible spectrophotometer.

Desorption studies were carried out using the spent carbon. The carbon loaded with metal ion separated and gently washed with distilled water to remove any unadsorbed metal ions. The metal ions-laden carbons were agitated with 50 mL each of water, 0.2 M sulphuric acid, hydrochloric acid, and nitric acid and sodium chloride solutions separately for 30 minutes and analyzed. The XRD patterns of the adsorbents before and after adsorption were recorded at the Regional Research Laboratory, Thiruvananthapuram, S.India. The SEM images were taken for the adsorbent at Madurai Kamaraj University, Madurai, S.India.

Results and Discussions

Effect of contact time and initial metal ion concentration

The experimental results of adsorptions of Copper ion on the activated carbon at various concentrations (5,10,15, 20, 25 and 30 mg/L) with contact time are shown in Figure 1. The equilibrium data are given in Table 1. It reveals that, percent adsorption decreased with increase in initial metal ion concentration, but the actual amount of metal ions adsorbed per unit mass of carbon increased with increase in metal ions concentration. It means that the adsorption is highly dependent on initial concentration of metal ions. It is because of at lower concentration, the ratio of the initial number of metal ions to the available surface area is low subsequently the fractional adsorption becomes independent of initial concentration. However, at high concentration the available sites of adsorption becomes fewer and hence the percentage removal of metal ion is dependent upon initial concentration. Equilibrium have established at 40 minutes for all concentrations. Figure 1 reveals that the curves are single, smooth, and continuous, leading to saturation, suggesting the possible monolayer coverage of the metal ions on the carbon surface^{4,5}.

Table 1 Equilibrium parameters for the adsorption of Metal ions onto PD activated Carbon

[M] ₀		C _e (mg / L)				Q _e (mg/g)				Metal ion removed (%)			
		30° C	40° C	50° C	60° C	30° C	40° C	50° C	60° C	30° C	40° C	50° C	60° C
5	Copper ion adsorption	1.2520	1.0281	0.9256	0.8351	1.874	1.985	2.037	2.082	74.96	79.43	81.48	83.30
10		2.5780	2.2528	2.0281	1.9821	3.711	3.874	3.986	4.009	74.22	77.47	79.71	80.18
15		4.7812	4.2121	4.0121	3.8559	5.109	5.394	5.494	5.572	68.13	71.92	73.25	74.29
20		7.8521	7.2821	7.0421	6.8529	6.074	6.359	6.479	6.574	60.74	63.59	64.79	65.74
25		11.2821	10.9251	10.4281	9.5790	6.859	7.037	7.286	7.711	54.87	56.30	58.29	61.68
30		14.7281	14.2832	14.059	13.1512	7.636	7.858	7.971	8.424	50.81	52.39	53.14	56.16
5	Ferrous ion adsorption	0.5250	0.4578	0.4072	0.3875	2.2375	2.2711	2.2964	2.3062	89.50	90.84	91.85	92.25
10		1.1250	1.0750	0.9852	0.7558	4.4380	4.4610	4.5074	4.6221	88.75	89.22	90.14	92.44
15		1.8325	1.7240	1.5570	1.2541	6.5838	6.6350	6.7215	6.8729	87.78	88.50	89.62	91.63
20		2.7876	2.5494	2.2542	2.0490	8.6062	8.7254	8.8729	8.9755	86.06	87.25	88.72	89.75
25		3.8980	3.7150	3.4715	3.1749	10.5510	10.6425	10.7642	10.9125	84.40	85.14	86.11	87.30
30		5.8520	5.6852	5.2820	4.9957	12.0740	12.1574	12.3575	12.5021	80.40	81.04	82.38	83.34

Comparative Study on the Adsorption Kinetics

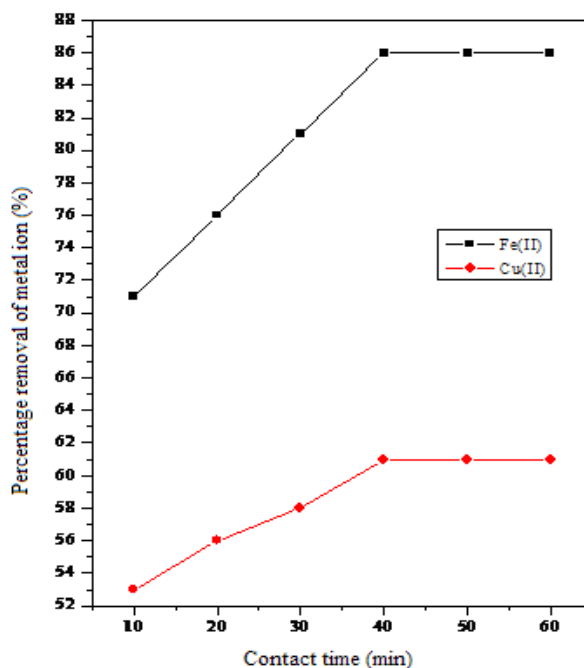


Figure 1. Effect of contact time on the removal of metal ions by PDC

Effect of carbon concentration

The adsorption of the metal ion on carbon was studied by varying the carbon concentration (50-250mg/50mL) for metal ion of 20 mg/L. The percentage of adsorption increased with increase in the carbon concentration (Figure 2). This has attributed to increased carbon surface area and availability of more adsorption sites.

Adsorption isotherm

The experimental data are analyzed according to the linear form of the Langmuir and Freundlich isotherms. The Langmuir isotherm represented by the following equation⁶

$$C_e/Q_e = 1/Q_m b + C_e/Q_m$$

Here C_e is the equilibrium concentration (mg/L), Q_e is the amount adsorbed at equilibrium (mg/g) and Q_m and b is Langmuir constants related to adsorption efficiency and energy of adsorption, respectively. The linear plots of C_e/Q_e versus C_e suggest the applicability of the Langmuir isotherms (Figure 3). The values of Q_m and b were determined from slope and intercepts of the plots and are presented in Table 2. From the results, it is clear that the value of adsorption energy b of the carbon increases on increasing the temperature. From the values of adsorption efficiency Q_m we can conclude that the maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on adsorbent surface with constant energy and no transmission of adsorbate in the plane of the adsorbent surface. The trend shows that the adsorbent prefers to bind acidic ions and that speciation predominates on sorbent characteristics, when ion exchange is the predominant mechanism. Further, it confirms the endothermic nature of the processes involved in the system. To confirm the favorability of the adsorption process, the separation factor (R_L) is calculated and presented in Table 3. The values were found to be between 0 and 1 and confirm that the ongoing adsorption process is favourable⁷

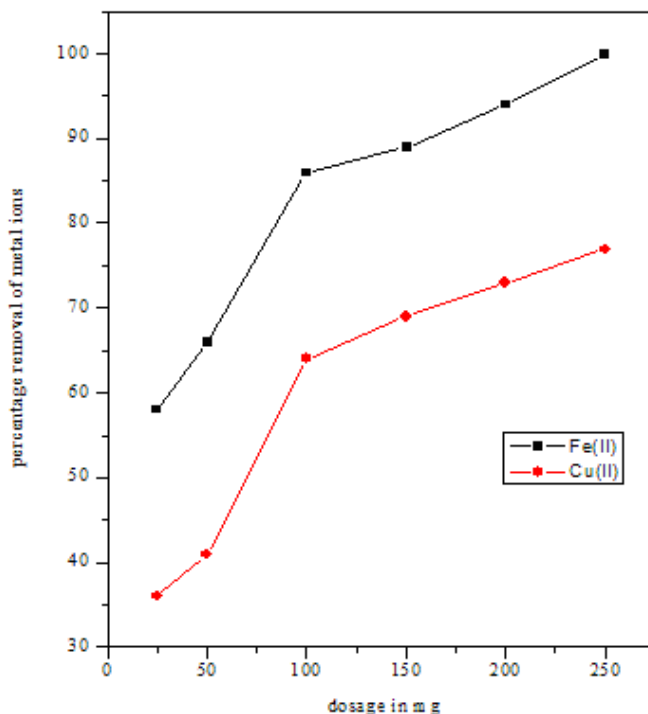


Figure 2. Effect of dosage on the removal of metal ions

Table 2. Langmuir isotherm results

Metal ion	Temp (⁰ C)	Statistical Parameters/Constants		
		r ²	Q _m	b
Copper ion	30	0.9968	10.0979	0.2008
	40	0.9976	9.7181	0.2707
	50	0.9986	9.6993	0.3089
	60	0.9987	10.4515	0.2926
Ferrous Ion	30	0.9926	21.4915	0.2339
	40	0.9946	20.2675	0.2792
	50	0.9936	20.0680	0.3196
	60	0.9917	18.9286	0.4144

The Freundlich equation have also employed for the adsorption of metal ions on the adsorbent. The Freundlich isotherm⁸ have represented as

$$\log Q_e = \log K_f + 1/n \log C_e$$

Where Q_e is the amount of metal ion adsorbed (mg/ g), C_e is the equilibrium concentration of metal ion in solution (mg/L) and K_f and n are constants incorporating all factors affecting the adsorption capacity and intensity of adsorption, respectively. Linear plot of log Q_e versus log C_e shows that the adsorption of metal ion follows the Freundlich

isotherm (Figure 4). The values of K_f and n were found and given in the Table 4, shows the increase of negative charge on the surface that enhances the electrostatic force like Vander waal's between the carbon surface and metal ions, which increases the adsorption of metal ions. The values clearly show that dominance in adsorption capacity. The intensity of adsorption is an indicative of the bond energies between metal ions and adsorbent and the possibility of slight chemisorptions rather than physisorption. The possibility of multilayer adsorption of metal ion through the percolation process cannot be ruled out. However, the values of n is greater than one indicating the adsorption is much more favourable^{5,9}

Table 3 - Dimensionless Separation factor (R_L)

[M] ₀ (mg/L)		Temperature, (°C)			
		30	40	50	60
5	Copper ion adsorption	0.5000	0.4255	0.3923	0.4081
10		0.3333	0.2702	0.2439	0.2565
15		0.2500	0.1980	0.1769	0.1869
20		0.2000	0.1562	0.1388	0.1470
25		0.1666	0.1290	0.1144	0.1212
30		0.1428	0.1098	0.0970	0.1030
5	Ferrous ion adsorption	0.4609	0.4166	0.3846	0.3257
10		0.2994	0.2631	0.2380	0.1945
15		0.2222	0.1923	0.1724	0.1386
20		0.1763	0.1519	0.1351	0.1077
25		0.1461	0.1254	0.1111	0.0881
30		0.1248	0.1067	0.0943	0.0745

Table 4 – Freundlich isotherm results

Metal ion	Temp (°C)	Statistical parameters / constants		
		r^2	K_f	n
Copper ion	30	0.9753	1.3366	1.8587
	40	0.9715	1.4328	2.0169
	50	0.9712	1.4837	2.0955
	60	0.9830	1.5059	2.0325
Ferrous Ion	30	0.9894	1.8047	1.4074
	40	0.9900	1.8648	1.4560
	50	0.9887	1.9340	1.4764
	60	0.9762	2.0301	1.5304

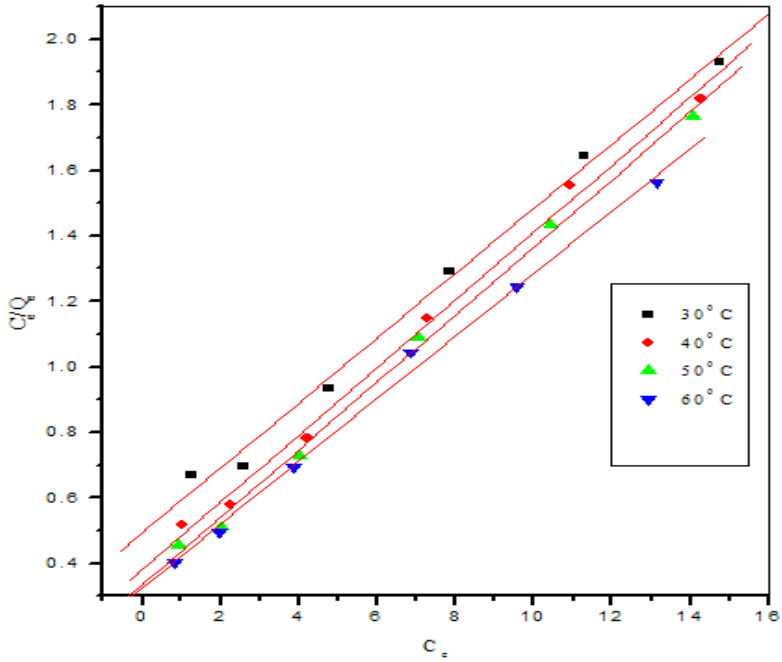


Figure 3. Langmuir isotherm for the adsorption of copper ion onto PDC

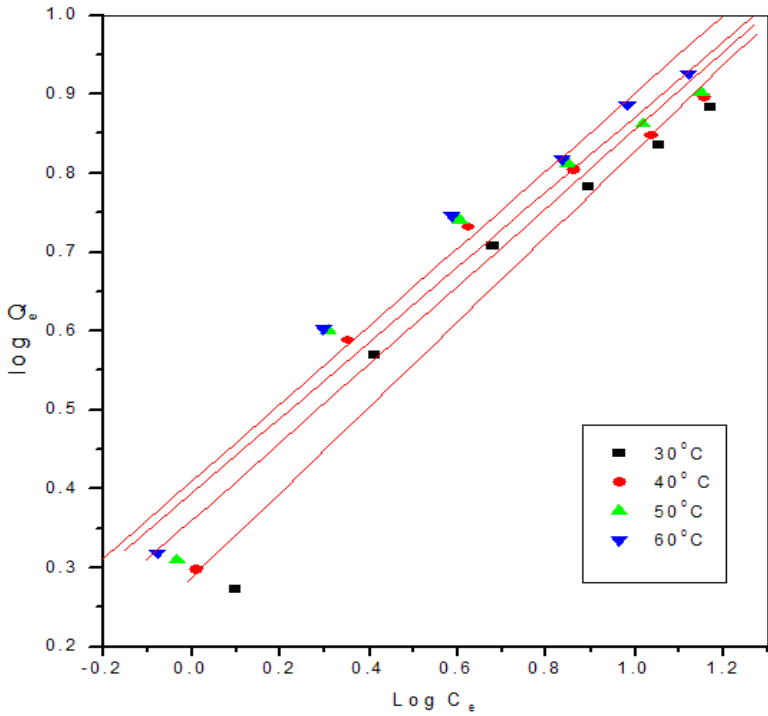
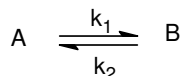


Figure 4. Freundlich isotherm for the adsorption of copper ion onto PDC

Kinetics of adsorption

The kinetics of sorption describes the solute uptake rate, which in turn governs the residence time of sorption reaction. It is one of the important characteristics in defining the efficiency of sorption. In the present study, the kinetics of the metal ion removal has carried out to understand the behaviour of this low cost carbon adsorbent. The adsorption of metal ions from an aqueous solution follows reversible first order kinetics, when a single species considered on a heterogeneous surface. The heterogeneous equilibrium between the metal ion solutions and the activated carbon have expressed as



Here k_1 is the forward rate constant and k_2 is the backward rate constant. A represents metal ion remaining in the aqueous solution and B represents metal ion adsorbed on the surface of activated carbon. The rate constants calculated as earlier^{10,11}. The data furnished in Table 6, shows that the forward rate constant is much higher than the backward rate constant suggesting that the rate of adsorption is clearly dominant. At equilibrium, the rate is the ratio of the concentration of adsorbate in adsorbent and concentration of adsorbate in aqueous solution is given by K_0 . The results shown in Table 5 indicates that K_0 values decreases with increase in the concentration of the metal ions and increases with increase in temperature. Examination of the effect of metal ion concentrations on the rate constant K_{ad} (Table 6), the values help to describe the mechanism of metal ion removal taking place¹¹. In cases of strict surface adsorption a variation of rate should be proportional to the first power of concentration. However, when pore diffusion limits the adsorption process, the relationship between initial metal ion concentration and rate of reaction will not be linear¹⁰

Effect of temperature

The adsorption capacity of the carbon increased with increase in the temperature of the system from 30-60 °C. Thermodynamic parameters such as change in free energy (ΔG°) kJ/mol, enthalpy (ΔH°) kJ/mol and entropy (ΔS°) J/K/mol were determined using the following equations^{12,13}

$$K_0 = C_{solid}/C_{liquid}$$

$$\Delta G^\circ = -RT \ln K_0$$

$$\log K_0 = \Delta S^\circ / (2.303RT) - \Delta H^\circ / (2.303RT)$$

Here K_0 is the equilibrium constant, C_{solid} is the solid phase concentration at equilibrium (mg/L), C_{liquid} is the liquid phase concentration at equilibrium (mg/L), T is the temperature in Kelvin and R is the gas constant. The ΔH° and ΔS° values obtained from the slope and intercept of Van't Hoff plots have presented in Table 5. The values are with in the range of 1 to 93 KJ/mol indicates the favourability of physisorption. From the order we could conclude that physisorption is much more favourable for metal ions. The positive values of ΔH° show the endothermic nature of adsorption and it governs the possibility of physical adsorption¹⁴. In the case of physical adsorption, while increasing the temperature of the system, the extent of metal ion adsorption increases, this rules out the possibility of chemisorptions. However, the very low ΔH° value depicts metal ions are physisorbed onto adsorbent^{15,16}

The negative values of ΔG° (Table 5) shows that the adsorption is highly favourable for metal ions. However, it indicates that the metal ion adsorption is spontaneous. The positive values of ΔS° (Table 5) shows the increased disorder and randomness at the solid solution interface of the adsorbent. During adsorption there are some structural changes in the metal ion and the adsorbent occur¹⁷. From the results, we could make out that more efficient physisorption. Enhancement of adsorption capacity of the activated carbon at higher temperatures is attributed to the enlargement of pore size and activation of the adsorbent surface⁴.

Table 5. Equilibrium constant and thermodynamic parameters for the adsorption of metal ions onto PD carbon

[M] ₀		K ₀				ΔG°				ΔH°	ΔS°
		30° C	40° C	50° C	60° C	30° C	40° C	50° C	60° C		
5	Copper ion adsorption	2.9936	3.8633	4.4019	4.9873	-2.761	-3.517	-3.979	-4.445	13.895	55.21
10		2.8789	3.4389	3.9309	4.0451	-2.663	-3.213	3.675	-3.867	9.709	41.05
15		2.1372	2.5611	2.7386	2.8901	-1.913	-2.446	-2.704	-2.937	8.170	33.52
20		1.5470	1.7464	1.8399	1.9184	-1.098	-1.449	-1.635	-1.803	5.855	23.10
25		1.2158	1.2883	1.3973	1.6098	-0.491	-0.659	-0.896	-1.317	7.591	26.49
30		1.0369	1.1003	1.1338	1.2811	-0.091	-0.247	-0.335	-0.683	5.461	18.22
5	Ferrous ion adsorption	8.823	9.5495	11.2789	11.9032	-5.397	-5.871	-6.503	-6.854	8.215	49.935
10		7.888	8.2764	9.1502	12.2307	-5.201	-5.498	-5.943	-6.938	11.547	54.807
15		7.1855	7.7006	8.6339	10.9616	-4.967	-5.310	-5.787	-6.627	11.319	53.398
20		6.1746	6.8455	7.8723	8.7608	-4.585	-5.003	-5.539	-6.006	9.872	13.182
25		5.4135	5.7294	6.2006	6.8761	-4.252	-4.540	-4.898	-5.336	6.569	35.598
30		4.1264	4.2768	4.6796	5.0051	-3.569	-3.782	-4.142	-4.457	5.528	29.907

Table 6. Rate constants for the adsorption of metal ions ($10^3 k_{ad}, \text{min}^{-1}$) and the constants for forward ($10^3 k_1, \text{min}^{-1}$) and reverse ($10^3 k_2, \text{min}^{-1}$) process.

M_0		k_{ad}				30 ° C		40 ° C		50 ° C		60 ° C	
		30° C	40° C	50° C	60° C	k_1	k_2	k_1	k_2	k_1	k_2	k_1	k_2
5	Copper ion adsorption	13.270	15.770	14.891	17.680	9.9472	3.3228	12.5274	3.2436	12.130	2.7564	14.727	2.9530
10		10.508	11.639	1.881	12.521	7.7931	2.7069	8.9960	2.6340	9.471	2.4097	10.039	2.4816
15		7.3903	7.738	10.913	9.492	5.0346	2.3557	5.5651	2.1729	7.992	2.9186	7.052	2.4403
20		5.9878	6.476	7.024	6.694	3.6369	2.3509	4.1181	2.3579	4.551	2.4733	4.404	2.2943
25		8.1226	8.177	8.705	9.142	4.4569	3.6657	4.6042	3.5737	5.074	3.6311	5.639	3.5032
30		6.4253	6.722	6.766	7.339	3.2695	3.1518	3.5221	3.2006	3.595	3.1709	4.122	3.2175
5	Ferrous ion adsorption	4.5737	3.7568	3.4545	2.8780	4.0935	0.4802	3.3980	0.3558	3.1732	0.2813	2.6550	0.2230
10		27.935	25.7930	20.3664	4.3200	24.7920	3.1430	23.0192	2.7738	18.2424	1.9936	3.9930	0.3265
15		26.046	24.4110	23.2830	22.2460	22.8641	3.1821	21.6053	2.8057	20.8660	2.4170	20.3860	1.8600
20		24.365	25.5860	24.3420	21.5300	20.9130	3.3962	22.3248	3.2614	21.5977	2.7443	19.3300	2.2089
25		22.138	21.5260	16.6960	14.3840	18.8686	3.4521	18.3271	3.1989	14.3772	2.3188	12.5580	1.8263
30		21.222	19.6960	16.1348	13.7300	17.0800	4.1400	18.9581	3.7319	13.2939	2.8409	11.4440	2.2864

Effect of pH

The experiments carried out at different pH shows that there was a change in the percent removal of metal ions over the entire pH range of 3 to 9 shown in the Figure 5. This indicates the strong force of interaction between the metal ion and the activated carbon that, either H^+ or OH^- ions could influence the adsorption capacity. Here the interaction is more at pH 6, due to the competence of acidic H^+ ion with metal cation for the sorption sites. The percentage of sorption increased at the above pH value is due to the presence of ionic COOH groups. The adsorption of metal ions on the activated carbon does involve ion exchange mechanism. The adsorption of metal ions through ion exchange mechanism due to that there should be an influence on the metal ion adsorption while varying the pH¹⁸.

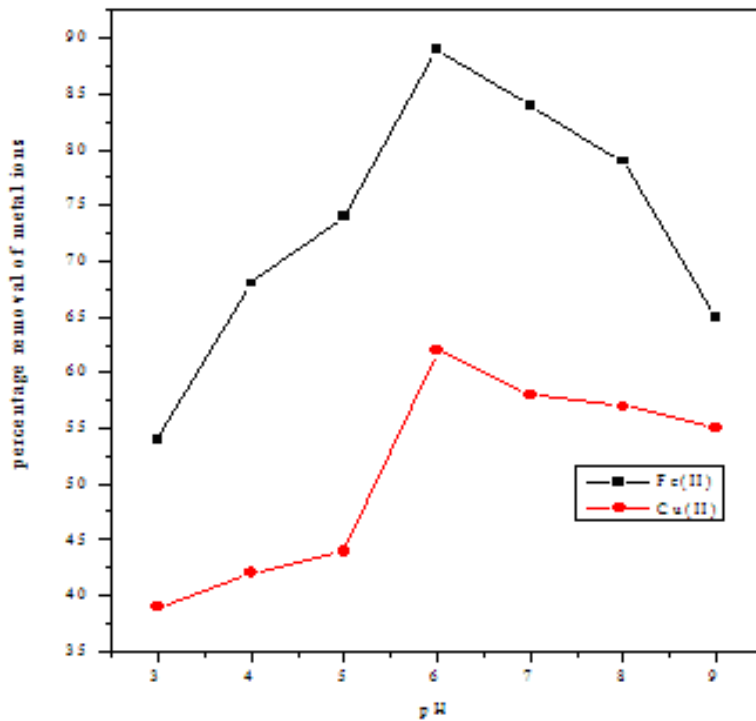


Figure 5. Effect of pH on the removal of metal ion

Effect of other ions

The effect of other ions like Ca^{2+} and Cl^- on the adsorption process studied at different concentrations. The ions added to 20mg/L of metal ion solutions and the contents were agitated for 60 min at 30 C. The results had shown in the Figure 6 and 7 reveals that low concentration of Cl^- does not affect the percentage of adsorption of metal ion on activated carbon, because the interaction of Cl^- at available sites of adsorbent through competitive adsorption is not so effective. While the concentration of other ion Ca^{2+} increases, the interference of these ions at available surface sites of the sorbent through competitive adsorption increases that, decreases the percentage adsorption. The interference was more in the presence of Ca^{2+} compared with Cl^- ion. This is so because ions with smaller hydrated radii decrease the swelling pressure with in the sorbent and increase the affinity of the sorbent for such ions^{5,19}

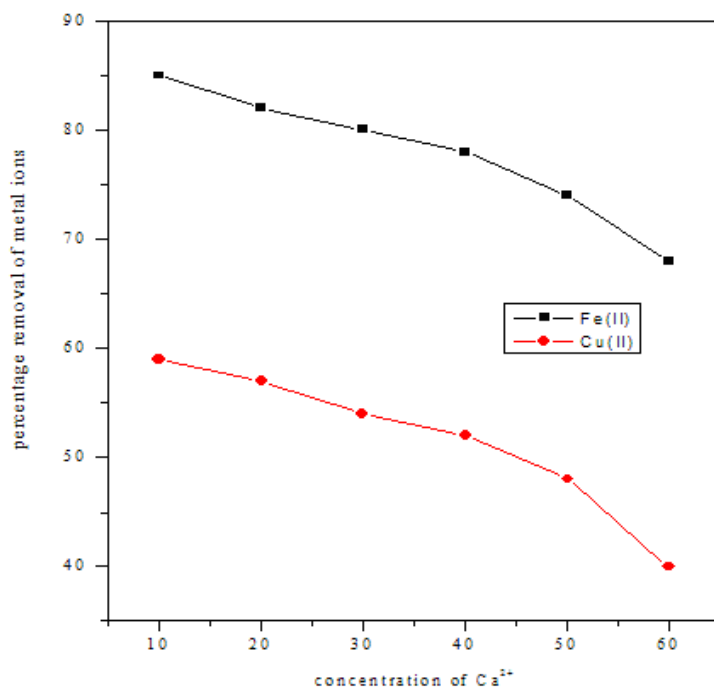


Figure 6. Effect of Ca²⁺ on the removal of metal ion

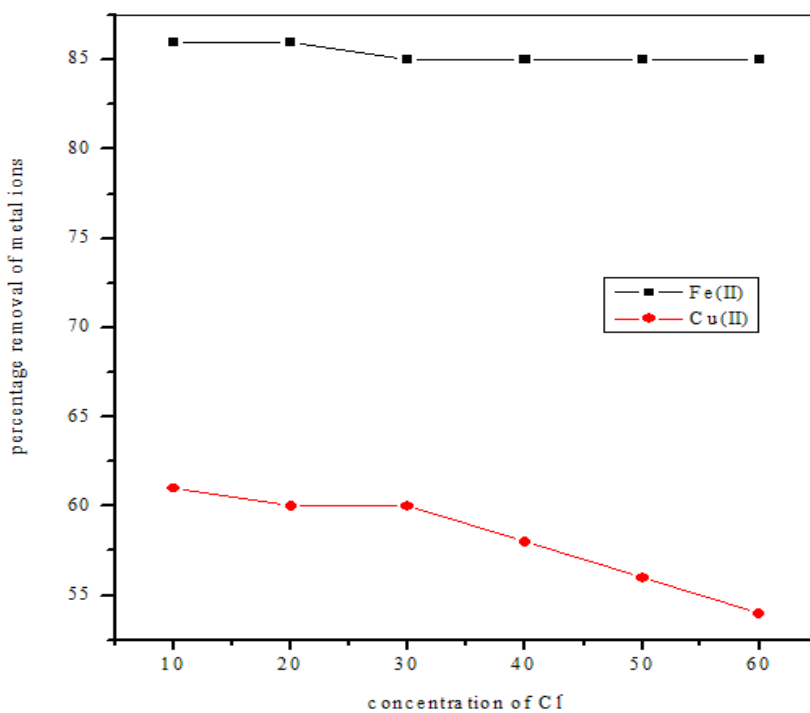


Figure 7. Effect of Cl⁻ on the removal of metal ion

Desorption studies

Desorption studies help to elucidate the nature of adsorption and recycling of the spent adsorbent and the metal ions. If the adsorbed metal ions can be desorbed using neutral pH water, then the attachment of the metal ion of the adsorbent is by weak bonds. Sulphuric acid or alkaline water desorb the metal ions then the adsorption is by ion exchange²⁰. The effect of various reagents used for desorption studies is shown in Figure 8. The results indicate that hydrochloric acid is a better reagent for desorption, because we could get more than 90% removal of adsorbed metal ion. The reversibility of adsorbed metal ion in mineral acid or base is in agreement with the pH dependent results obtained. The desorption of metal ion by mineral acids and alkaline medium indicates that the metal ion was adsorbed onto the activated carbon through by physisorption²¹

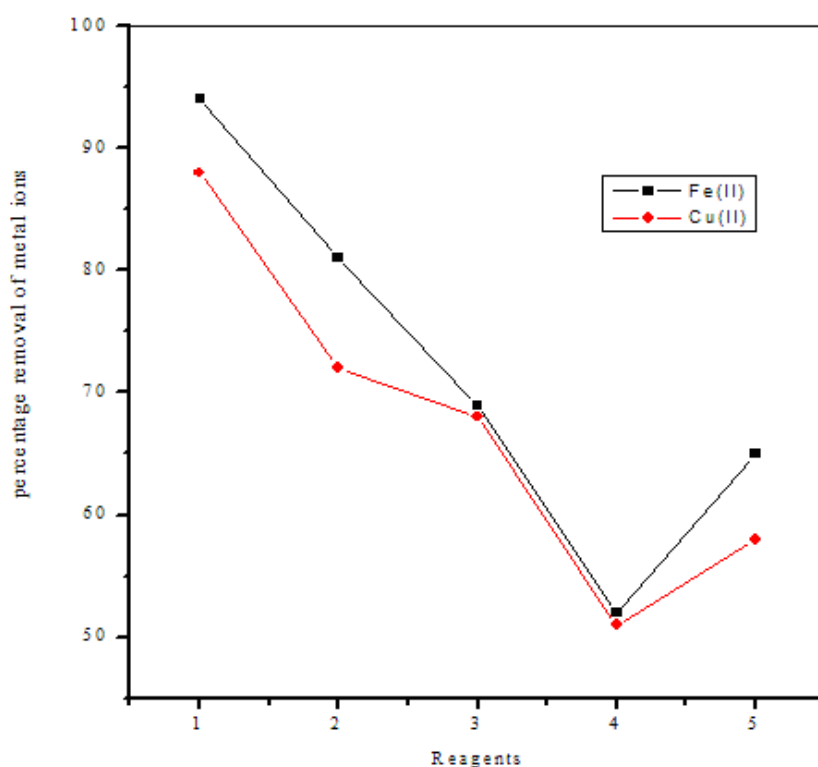


Figure 8. Regeneration pattern (1-HCl, 2-HNO₃, 3-H₂SO₄, 4- NaCl, 5-NaCl+H₂O)

Evidences for adsorption

The IR spectra of the raw activated carbon and after adsorption of metal ions have shown in representative Figs. 9 and 10. It could be seen that the slight reduction of stretching vibration adsorption bands. This clearly indicates the adsorption of metal ion on the adsorbent by physical forces not by chemical combination. The XRD diagrams of activated carbon and metal ions-adsorbed carbon have shown in representative Figs. 11 and 12. The intense main peak shows the presence of highly organized crystalline structure of raw activated carbon²², after the adsorption of metal ion, the intensity of the highly organized peaks are slightly diminished. This has attributed to the adsorption of metal ions on the

upper layer of the crystalline structure of the carbon surface by means of physisorption. The SEM diagrams of raw activated carbon and metal ions-adsorbed activated carbon have shown in representative Figure 13 and 14. The bright spots, shows the presence of tiny holes on the crystalline structure of raw activated carbon, after treatment with metal ions the bright spots became black shows the adsorption of the metal ions on the surface of the carbon.

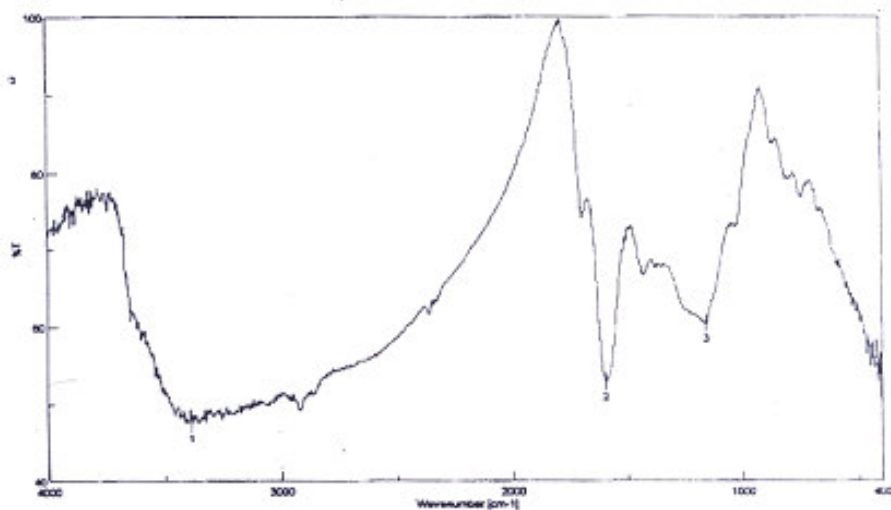


Figure 9. FT-IR Spectrum of absorbent before absorption

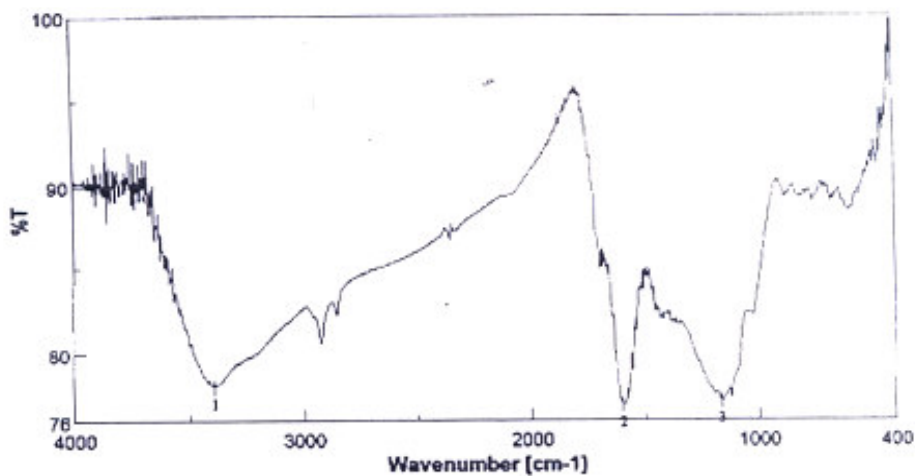


Figure 10. FT-IR Spectrum of absorbent after absorption

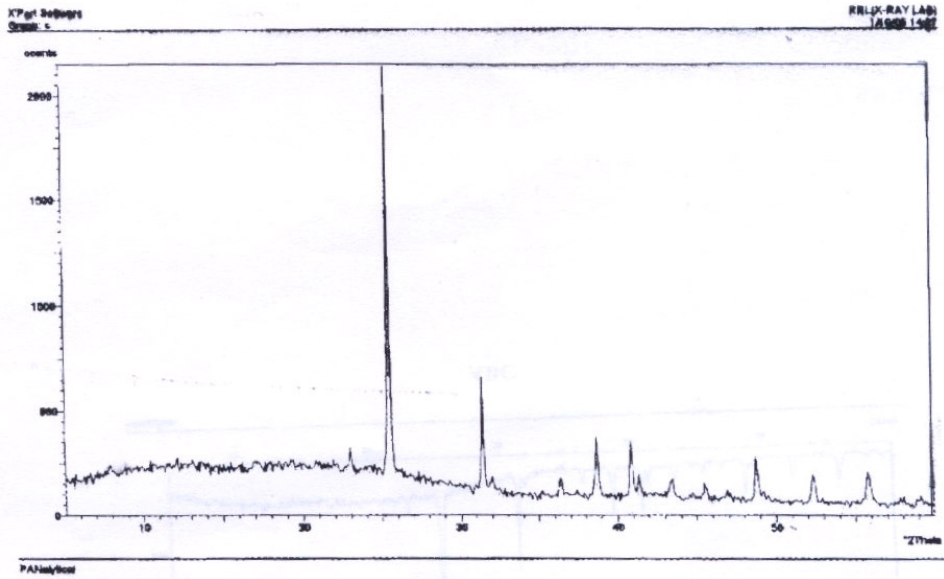


Figure 11. XRD pattern of the adsorbent before adsorption

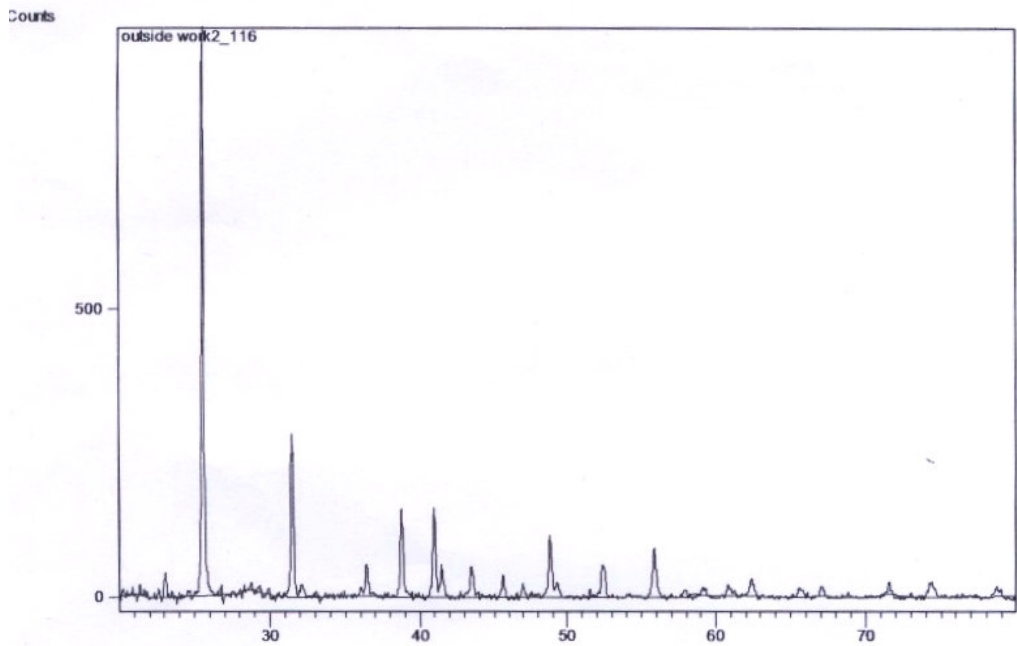


Figure 12. XRD pattern of the adsorbent after adsorption

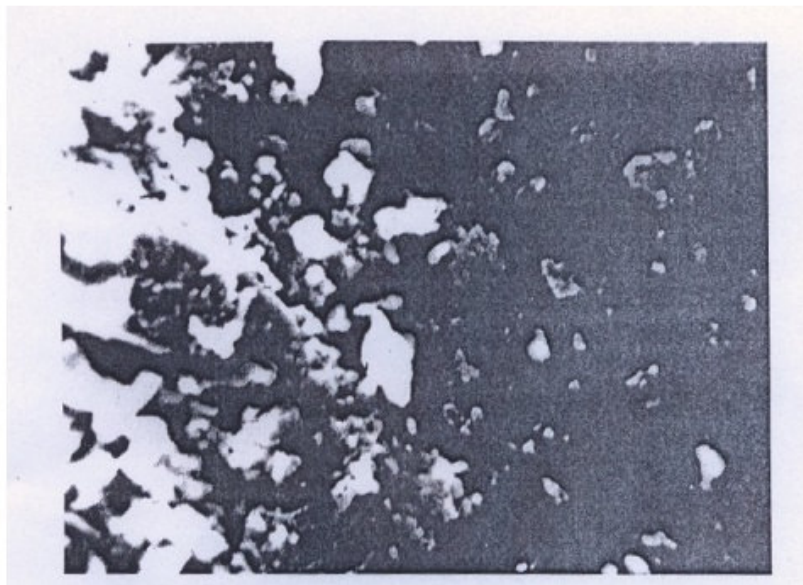


Figure 13. SEM image of absorbent before absorption



Figure 14. SEM image of absorbent before absorption

Conclusions

The experimental data correlated reasonably well by the Langmuir and Freundlich adsorption isotherms and the isotherm parameters were calculated. The amount of metal ions adsorbed increased with increase in pH of the medium. The amount of metal ion adsorbed slightly decreased with increasing ionic strength and increased with increase in temperature.

The dimensionless separation factor (R_L) showed that the activated carbon can be used for the removal of metal ions from aqueous solution. The values of ΔH° , ΔS° and ΔG° results shows that the carbon employed has a considerable potential as an adsorbent for the removal of metal ions.

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