



Kinetics of Adsorption of Ferrous Ion onto Acid Activated Carbon from *Zea Mays* Dust

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Abstract: The batch removal of ferrous ion from aqueous solution using low cost adsorbents such as *Zea mays* dust carbon(ZDC) under different experimental conditions were investigated in this study. The process parameters studied include agitation time, initial metal ion concentration, carbon dose, pH and temperature. The adsorption followed first order reaction equation and the rate is mainly controlled by intraparticle diffusion. Freundlich and Langmuir isotherm models were applied to the equilibrium data. The adsorption capacity (Q_m) obtained from the Langmuir isotherm plot were found to 37.17, 38.31, 39.37 and 40.48 mg/g. The temperature variation study showed that the ferrous ions adsorption is endothermic and spontaneous with increased randomness at the solid solution interface. Significant effect on adsorption was observed on varying the pH of the ferrous ion solutions. The Langmuir and Freundlich adsorption isotherms obtained positive ΔH^0 value, pH dependent results and desorption of metal ions in mineral acid suggest that the adsorption of ferrous ion on ZDC involves physisorption mechanism.

Keywords: Activated carbon, *Zea mays* dust carbon (ZDC), Ferrous ion, Adsorption isotherm, Adsorption kinetics, Intraparticle diffusion, Regeneration pattern.

Introduction

Toxic metal compounds coming to the earth's surface not only reach the earth's water (seas, lakes, ponds and reservoirs) but can also contaminate ground water in trace amounts by leaching from the soil. Therefore, the earth's water may contain various toxic metals. Metal ion such as ferrous ion produces undesirable effects on human and animal life even in

low concentrations. In recent years many researchers have studied the removal of different heavy metal ions including iron, chromium, nickel and copper from the water. Wastewaters from industries released in to nearby land or rivers without any treatment because the conventional treatment methods are not cost effective in the Indian context. 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 classes of 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 metal ion from water and wastewater¹⁻⁴. The present is study undertaken to evaluate the efficiency of a carbon adsorbent prepared form acid activated *Zea mays* dust for the removal of ferrous ion aqueous solution. In order to design adsorption treatment systems, knowledge of kinetic and mass transfer process is essential. In this paper, the applicability of kinetic and mass-transfer models for the adsorption of ferrous ion onto activated carbon was reported.

Experimental

Carbon was prepared by treating air dried *Zea mays* dust with con sulphuric acid in a weight ratio of 1:1. The resulting black product was kept in a furnace maintained at 500 °C for 12 h followed by washing with water until free from excess acid and dried at 150 ± 5 °C. The carbon product obtained was ground and the portion retained between 10 and 50 µm sieves was used in all the experiments. All chemicals supplied by S.d. fine chemicals with high purity.

Batch equilibration method

All experiments were carried out at 30, 40, 50 and 60 °C temperature in batch mode. Batch mode was selected because of its simplicity and reliability. The experiments were done in different Erlenmeyer glass flasks of 100 mL capacity. Prior to each experiment, a predetermined amount of adsorbent was added to each flask. The stirring was kept constant (120 rpm) for each run throughout the experiment to ensure equal mixing. Each flask was filled with a known volume of sample before commencing stirring such as metal solutions with an initial concentration of 5 mg/L to 25 mg/L. The flask containing the sample was withdrawn from the shaker at the predetermined time interval, filtered and the residual concentration of the ferrous ion was measured by UV-visible double beam Spectrophotometer.

Effect of variable parameters

Dosage of adsorbents

Different doses consisting of 10 to 250 mg/50 mL of the adsorbent is mixed with the ferrous ion and the mixture was agitated in a mechanical shaker. The percentage of adsorption for different doses was determined by keeping all other factors constant.

Initial concentration

In order to determine the rate of adsorption, experiments were conducted with different initial concentrations of ferrous ions ranging from 5 to 25 mg/L. All other factors were kept constant.

Contact time

The effect of period of contact between the adsorbent and adsorbate on the removal of the ferrous ions in a single cycle was determined by keeping particle size, initial concentration, dosage, pH and temperature constant.

Initial pH

Adsorption experiments were carried out at a range of pH of the solution *i.e.* 3-10. The acidic and alkaline pH of the medium was maintained by adding the required amounts of hydrochloric acid and sodium hydroxide solutions. The parameters like particle size of the adsorbent and temperature were kept constant while carrying out the experiments.

Other ions

Adsorption studies of a specific metal ion in the presence of chloride ions were experimentally verified using the adsorbents. This involved the determination of the percentage of ferrous ion adsorbed from 15 mg/L of initial concentration of the ferrous ion solution with varying concentration of the added ion keeping all other factors constant.

Temperature

The adsorption experiments were performed at four different temperatures *viz.*, 30, 40, 50 and 60 °C in a thermostated shaker machine (Remi, India). The constancy of the temperature was maintained with an accuracy of ± 0.5 °C.

Regeneration studies

The regeneration of the adsorbed carbon is done by using 0.2 M mineral acids and sodium chloride solutions.

Results and Discussion

Characterization of the adsorbent

Activated carbons are a widely used adsorbent due to its high adsorption capacity, high surface area, micro porous structure and high degree of surface respectively. The wide usefulness of carbon is a result of their specific surface area, high chemical and mechanical stability. The chemical nature and pore structure usually determines the sorption activity. The physicochemical properties of the chosen adsorbent are listed below.

Table 1. Characteristics of the adsorbent

Properties	ZDC
Particle size, mm	0.042
Density, g/cc	0.3253
Moisture content, %	1.75
Loss on ignition, %	92
Acid insoluble matter, %	2.10
Water soluble matter, %	0.48
pH of aqueous solution	6.85
pH _{zpc}	6.25

Effect of contact time and initial ferrous ion concentration

The experimental results of adsorptions of ferrous ion on the activated carbon at various concentrations (5, 10, 15, 20 and 25 mg/L) with contact time are shown in Figure 1. The equilibrium data were collected Table 2 reveals that, percent adsorption decreased with

increase in initial ferrous ion and concentration, but the actual amount of ferrous ion adsorbed per unit mass of carbon increased with increase in metal ion concentration. It means that the adsorption is highly dependent on initial concentration of ferrous ion.

Table 2. Equilibrium Parameters for the adsorption metal ion onto ZDC

Ferrous ion, mg/L	C_e , mg/L				Q_e , mg/g				Metal ions 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	0.1715	0.1228	0.0812	0.0554	9.6570	9.7544	9.8376	9.8892	96.57	97.54	98.37	98.89
10	1.2587	1.0525	0.7747	0.5756	17.4826	17.8950	18.4506	18.8488	87.41	89.47	92.25	94.24
15	2.8849	2.4254	1.9841	1.5944	24.2302	25.1492	26.0318	26.8112	80.76	83.83	86.77	89.37
20	5.5546	4.9721	4.3599	3.7529	28.8908	30.0558	31.2802	32.4942	72.22	75.13	78.20	81.23
25	7.7945	7.0572	6.3160	5.6435	34.4110	35.8856	37.3680	38.7130	68.82	71.77	74.73	77.42

It is because of that at lower concentration, the ratio of the initial number of ferrous ion 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 ferrous 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 ferrous ion on the carbon surface^{5,6}.

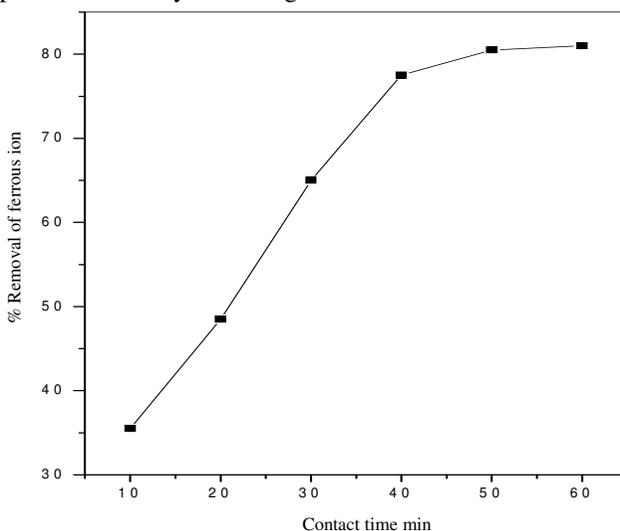


Figure 1. Effect contact time on the adsorption ferrous ion onto ZDC [M] = 15 mg/L; Adsorbent dose = 25 mg/50 mL; temp = 30 °C

Effect of carbon mass

The amount of ferrous ion adsorption increased with the increase in carbon dose and reached a maximum value after a particular dose (Figure 2). Taken an initial metal ion concentration of 15 mg/L, optimum metal ion removal was obtained at a maximum carbon dose of 25 mg. The increase in the adsorption of metal ion with carbon dose was due to the introduction of more binding sites for adsorption and the availability more surface area². Hence the entire studies are carried out with the adsorbent dose of 25 mg of adsorbent /50 mL of the adsorbate solution.

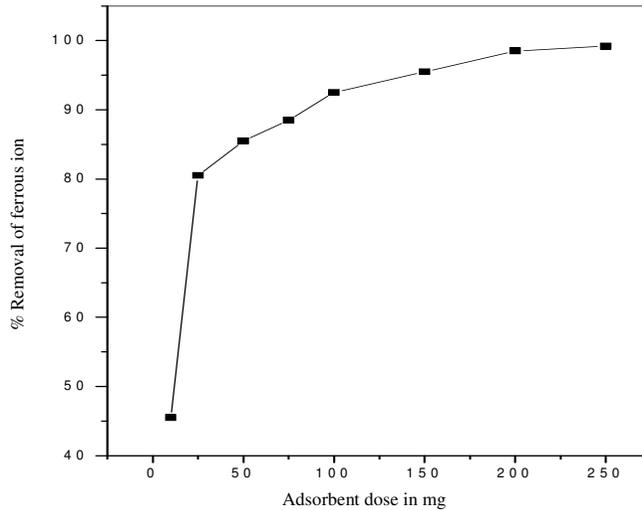


Figure 2. Effect of adsorbent dose on the adsorption of ferrous ion onto ZDC [M] = 15 mg/L, Contact time = 60 min; temp = 30 °C

Adsorption isotherm

The experimental data analyzed according to the linear form of the Langmuir⁷ and Freundlich⁸ isotherms.

The Langmuir isotherm represented by the following equation

$$C_e/Q_e = 1/Q_m b + C_e/Q_m \quad (1)$$

Where 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).

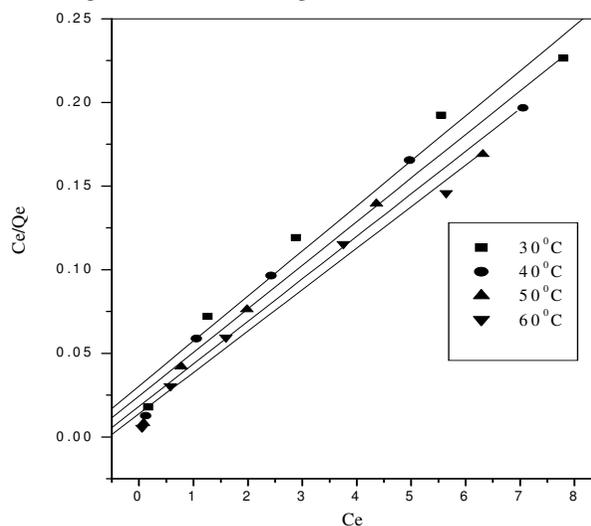


Figure 3. Linear Langmuir isotherm for the adsorption of ferrous ion onto ZDC

Values of Q_m and 'b' were determined from slope and intercepts of the plots and are presented in Table 3. From the results, it is clear that the value of adsorption efficiency Q_m and adsorption energy b of the carbon increases on increasing the temperature. From the values 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^{9,10}.

Table 3. Langmuir isotherm results

Metal ion	Temp °C	Statistical parameters/constants		
		r^2	Q_m	b
Ferrous ion adsorption	30	0.9972	37.174	0.8848
	40	0.9934	38.314	1.0653
	50	0.9905	39.370	1.3956
	60	0.9979	40.485	1.7770

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 adorability of the adsorption process, the separation factor (R_L) has calculated and presented in Table 4. The values were found to be between 0 and 1 and confirm that the ongoing adsorption process is favourable¹¹.

Table 4. Dimensionless separation factor (R_L)

Ferrous ion, mg/L	Temperature, °C			
	30°	40°	50°	60°
5	0.184	0.158	0.125	0.101
10	0.102	0.085	0.066	0.053
15	0.070	0.058	0.045	0.036
20	0.053	0.045	0.034	0.027
25	0.043	0.036	0.027	0.022

The Freundlich equation have also employed for the adsorption of ferrous ion on the adsorbent. The Freundlich isotherm have represented as

$$\log Q_e = \log K_f + 1/n \log C_e \quad (2)$$

Where Q_e is the amount of ferrous 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 ferrous ion follows the Freundlich isotherm (Figure 4). Values of K_f and 'n' were found and given in the Table 5, shows the increase of negative charge on the surface that enhances the electrostatic force like Vanderwaal's between the carbon surface and metal ion, which increases the adsorption of ferrous ion. The values clearly show that dominance in adsorption capacity. The intensity of adsorption is an indicative of the bond energies between ferrous ion and adsorbent and the possibility of slight chemisorptions rather than physisorption^{10,11}. 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^{12,13}.

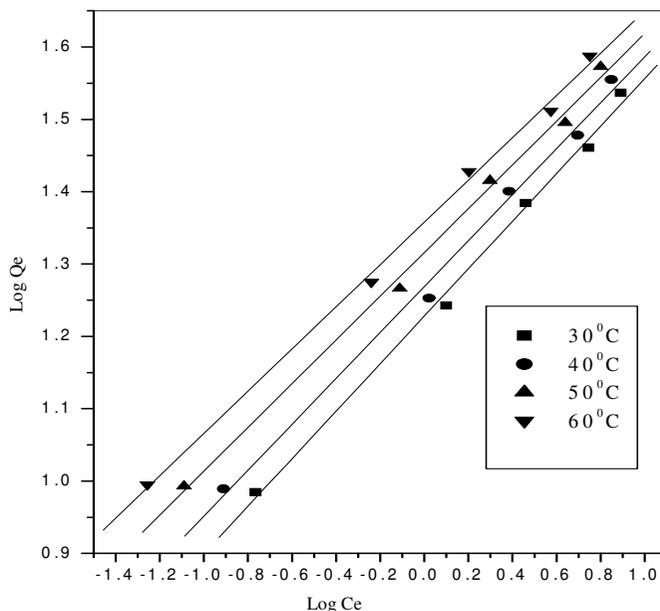


Figure 4. Linear Freundlich isotherm for the adsorption of ferrous ion onto ZDC

Table 5. Freundlich isotherm results

Metal ion	Temp	Statistical parameters/Constants		
	°C	r^2	k_f	n
Ferrous ion adsorption	30	0.9923	3.4140	3.0459
	40	0.9916	3.5561	3.1496
	50	0.9935	3.7284	3.3068
	60	0.9938	3.8876	3.4246

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°), enthalpy (ΔH°) and entropy (ΔS°) were determined using the following equations^{11,12,15,16}.

$$K_0 = C_{\text{solid}}/C_{\text{liquid}} \quad (4)$$

$$\Delta G^\circ = -RT \ln K_0 \quad (5)$$

$$\log K_0 = \Delta S^\circ / (2.303RT) - \Delta H^\circ / (2.303RT) \quad (6)$$

Where 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 6. The values are with in the range of 1 to 93 kJ/mol indicates the favourability of physisorption. From the order we could make out physisorption is much more favourable for ferrous ion. The positive values of ΔH° show the endothermic nature of adsorption and it governs the possibility of physical adsorption. Because 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 ferrous ion is physisorbed onto adsorbent^{12,13,15}

The negative values of ΔG° (Table 6) shows the adsorption is highly favourable for ferrous ion. However, it indicates that the metal ion adsorption was spontaneous. The positive values of ΔS° (Table 6) shows the increased disorder and randomness at the solid solution interface of with adsorbent. While the adsorption there are some structural changes in the ferrous ion and the adsorbent occur. The adsorbed water molecules, which have displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate molecules, thus allowing the prevalence of randomness in the system^{5,12,18}. From the results, we could make out that more efficient physisorption. Enhancement of adsorption capacity of the activated carbon at higher temperatures has attributed to the enlargement of pore size and activation of the adsorbent surface¹¹.

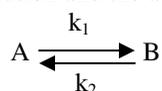
Table 6. Equilibrium constant and thermodynamic parameters for the adsorption of metal ions onto acid activated carbon Temperature, °C

Ferrous ion, mg/L	K_0		$-\Delta G^\circ$							ΔH°	ΔS°
	30 °C	40° C	50° C	60° C	30° C	40° C	50° C	60° C			
5	28.15	39.71	60.57	89.25	8408	9581	11020	12434	32.53	134.89	
10	6.94	8.50	11.90	16.37	4882	5569	6652	7739	24.33	96.05	
15	4.19	5.18	6.56	8.40	3614	4282	5051	5894	19.41	75.86	
20	2.60	3.02	3.58	4.32	2407	2878	3430	4057	14.23	54.80	
25	2.20	2.54	2.95	3.42	1994	2428	2912	3412	12.35	47.28	

ΔH° (kJ/mol), ΔS° (J/K/mol), ΔG° (J/mol)

Kinetics of adsorption

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 ferrous ion removal has carried out by Natrajan-Khalaf plot is used to understand the behaviour of this low cost carbon adsorbent. The adsorption of ferrous ion from an aqueous solution follows reversible first order kinetics, when a single species considered on a heterogeneous surface. The heterogeneous equilibrium between the ferrous ion solution and the activated carbon have expressed as



Where k_1 is the forward rate constant and k_2 is the backward rate constant. A represents ferrous ion remaining in the aqueous solution and B represents ferrous ion adsorbed on the surface of activated carbon. The rate constants calculated as earlier¹¹⁻¹³. The data furnished in Table 6 and 7, is evident 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 given by K_0 . The calculated values presented in the Table 6. The results indicates that K_0 values decreases with increase in the concentration of the ferrous ion and increases with increase in temperature

A clear examination of the effect of ferrous ion concentrations on the rate constant K_{ad} (Table 7), the values help to describe the mechanism of metal ion removal taking place .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 ferrous ion concentration and rate of reaction will not be linear. It shows that pore diffusion limits the overall rate of ferrous ion adsorption¹².

Table 7. Rate constants for the adsorption of ferrous ions ($10^3 k_{ad}$, min^{-1}) and the constants for forward ($10^3 k_1$, min^{-1}) and reverse ($10^3 k_2$, min^{-1}) process

Ferrous ion mg/L	k_{ad}				k_1		k_2		k_1		k_2	
	30 °C	40 °C	50 °C	60 °C	30 °C	40 °C	30 °C	40 °C	50 °C	60 °C	50 °C	60 °C
5	69.57	77.72	88.16	99.79	67.36	2.39	75.81	1.90	86.73	1.43	98.69	1.10
10	35.31	39.16	45.77	53.94	30.86	4.44	35.04	4.12	42.23	3.54	50.84	3.10
15	26.93	29.80	34.02	38.05	21.75	5.18	24.98	4.82	29.52	4.50	34.01	4.04
20	20.82	22.40	24.57	26.84	15.04	5.78	16.83	5.56	19.21	5.35	21.80	5.03
25	18.77	10.48	22.96	24.31	12.92	5.85	14.69	5.78	17.16	5.80	18.82	5.49

The contact-time experimental results can be used to study the rate-limiting step in the adsorption process, as shown by Weber and Morris^{13,14}. Since the particles are vigorously agitated during the adsorption period, it is probably reasonable to assume that the rate is not limited by mass transfer from the bulk liquid to the particle external surface, one might then postulate that the rate-limiting step may be either film or intraparticle diffusion. As they act in series, the slower of the two will be the rate-determining step¹³.

The rate constant for intraparticle diffusion is obtained using the equation

$$Q_t = K_p t^{1/2} + C \tag{3}$$

Here, K_p (mg/g/min) is the intraparticle diffusion rate constant. The K_p values obtained from the slope of the linear portions of the curves. The calculated intraparticle diffusion coefficient K_p value was given by 0.253, 0.278, 0.295, 0.319 and 0.346 $\text{mg}/\text{min}^{0.5}$ for initial dye concentration of 5, 10, 15, 20 and 25 mg/L at 30 °C. The values are increases with increase in the ferrous ion concentration, which reveals that the rate of adsorption is governed by the diffusion of adsorbed ferrous ion within the pores of the adsorbent.

Effect of pH

The experience carried out at different pH show that there was a change in the percent removal of ferrous ion over the entire pH range (Figure 5). This indicates the strong force of interaction between the metal ion and ZDC that either H^+ or OH^- ions could influence the adsorption capacity. In other words, the adsorption of metal ion on ZDC does involve ion exchange mechanism that have been an influence on the metal ion adsorption while varying the pH^{15,16}. This observation is in line with Langmuir and Freundlich isotherms and positive ΔH^0 value obtained, which indicates irreversible adsorption probably due to polar interactions.

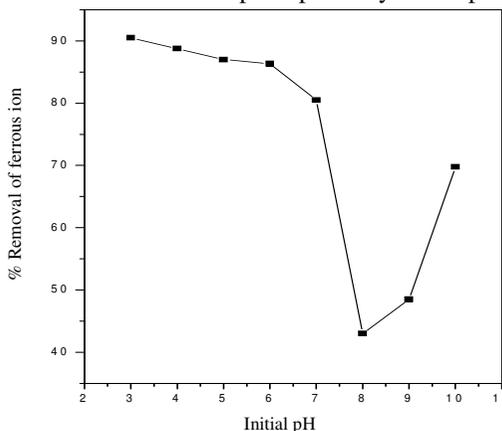


Figure 5. Effect of pH on the adsorption of ferrous ion onto ZDC [M] = 15 mg/L; contact time = 60 min; adsorbent dose = 25 mg/ 50 mL

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 15 mg/L of metal ion solutions and the contents were agitated for 60 min at 30 °C. The results had shown in the Figure 6 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 ions¹⁶⁻¹⁸.

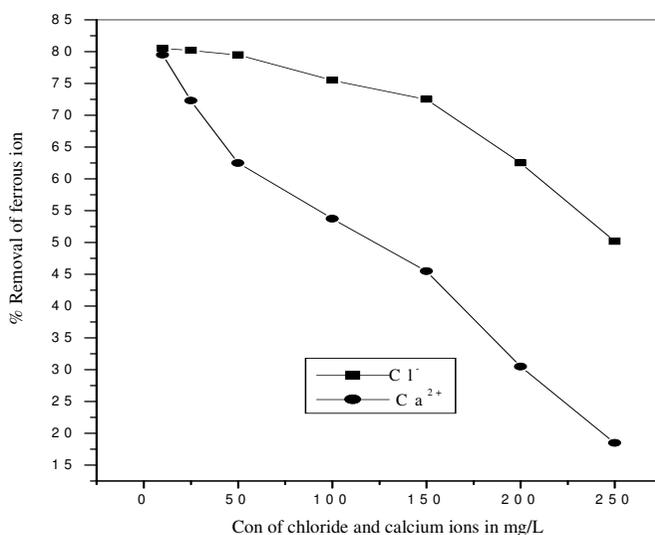


Figure 6. Effect of other ions on the adsorption of ferrous ion onto ZDC [M] = 15 mg/L; contact time = 60 min; adsorbent dose = 25 mg/50 mL

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. The effect of various reagents used for desorption studies indicate that hydrochloric acid is a better reagent for desorption, because we could get more than 94% 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 physisorption as well as by chemisorption mechanisms¹⁶⁻¹⁸.

Conclusion

The objective of this paper was utilization of *Zea mays* dust carbon as an adsorbent materials for ferrous ion removal. The following conclusions have been drawn from the above investigations:

- The removal efficiencies of ferrous ion through adsorption onto *Zea mays* dust carbon were found to be between 37.174 and 40.485 at 30 to 60 °C temperatures.
- The initial pH of aqueous solutions affects the ferrous ion removal. On the other hand percent removal of ferrous ion decreased with increasing initial concentration but increased with increasing adsorbent concentration.
- The best adsorptions were obtained under the condition of 25 mg/50 mL ferrous ion concentration and original pH solution. The suitable contact time was 40, 50 and 60 minutes.
- Adsorption isotherm can be described by Langmuir and Freundlich isotherm equations.
- The values of ΔH° , ΔS° and ΔG° results shows that the carbon employed has a considerable potential as an adsorbent for the removal of ferrous ion.
- *Zea mays* dust carbon has the potential for use as an adsorbent for the removal ferrous ion from wastewater.

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