

활성탄소를 이용한 수용액으로부터의 Ni(II), Cu(II) 그리고 Fe(III) 이온의 흡착

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Sorption of Ni(II), Cu(II) and Fe(III) ions from Aqueous Solutions Using Activated Carbon

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요약. 활성탄소를 가지고 수용액으로부터 전이금속이온을 제거 능력에 대해 시험했다. 탄소의 물리적, 화학적 그리고 액체-상 흡착 특징은 다음의 기준 절차로 수행하였다. Ni(II), Cu(II) 그리고 Fe(III)이온의 제거 연구는 흡착물질의 양, 금속이온 용액의 pH, 배치 모드에서 시간을 다양하게 하여 시도되었다. 평균 흡착 데이터는 Freundlich와 Langmuir로 맞춰졌고 등은 상수는 계산되었다. 다른 세가지 금속 이온의 평균 시간은 결정되었다. pH는 흡착에 있어서 중요한 역할을 하는 것을 발견했다. 이 과정은 흡열반응이었고, 열역학 인자는 계산되었다. 흡착 연구는 이온교환 메커니즘이 작용되는 것을 나타내고 있다.

주제어: 활성탄소, 흡착, pH, 탈착, 금속 이온 제거

ABSTRACT. An activated carbon was tested for its ability to remove transition metal ions from aqueous solutions. Physical, chemical and liquid-phase adsorption characterizations of the carbon were done following standard procedures. Studies on the removal of Ni(II), Cu(II) and Fe(III) ions were attempted by varying adsorbate dose, pH of the metal ion solution and time in batch mode. The equilibrium adsorption data were fitted with Freundlich and Langmuir and the isotherm constants were evaluated, equilibrium time of the different three metal ions were determined. pH was found to have a significant role to play in the adsorption. The processes were endothermic and the thermodynamic parameters were evaluated. Desorption studies indicate that ion-exchange mechanism is operating.

Keywords: Activated carbon, Adsorption, pH, Desorption, Metal ions removal

INTRODUCTION

The removal of metal contaminants from effluent streams has the advantage of reducing the cost of waste disposal. In most cases, the treatment of waste water gives rise to secondary effluents. Efficiency of such process can be improved by recycling treated water and/or metal recovery. General methods applied to the removal of metals include ion exchange or adsorption, precipitation, floatation, evaporation and membrane processes. By using ion exchange and adsorption most of the water can be recycled without the need for further treatment. In some cases, the metal can also be recovered in a useful form.

A variety of materials have been investigated for the removal of metals from metallurgical effluents. Conventional activated carbons are extensively used in water treatment for removal of colour, odour and organic contaminants. These carbonaceous materials possess the potential for removal of inorganic species from effluent streams.^{1,2}

Activated carbons are of high porosity, high surface area materials and are prepared from readily available carbonaceous precursors. These precursors are normally exposed to a number of different activation methods in an effort to achieve an activated carbon with the most favorable properties for a particular application. The texture of activated carbons may be adopted to suit the situation by adequate choice of

the activation procedure. The nature and concentration of the surface functional groups may be modified by suitable thermal or chemical post-treatments. Introduction of oxygen-containing groups not only improves the cationic uptake capacity of the precursor but it also makes the surface more hydrophilic amenable to wetting.^{2,3}

It has been shown that, for adsorption of inorganic compounds on activated carbons from aqueous solutions, the chemical nature of the adsorbent determined by the amount and the nature of the surface complexes has in general more influence than the surface area and porosity of adsorbent. Removal of metals by conventional activated carbons has been studied by a number of authors. In general, ordinary activated carbons possess large surface areas but have a relatively low capacity for metal ions.³ Modified activated carbons have been examined as alternatives to conventional polymeric ion exchange resins.

Wastewater containing heavy metal pollutants cause direct toxicity, both to human and other living organisms due to their presence beyond specified limits. The main goal today is to adopt appropriate methods and to develop suitable techniques either to prevent metal pollution or to reduce it to very low levels. Nickel is largely present in the wastewaters of electroplating, motor vehicle and aircraft industries.¹ Acute nickel(II) poisoning causes dizziness, head ache, nausea and vomiting, chest pain, dry cough and shortness of breathe, rapid respiration, cyanosis and extreme weakness.² World Health Organization, WHO has suggested that the maximum amount of nickel in drinking water should be 0.1 mg/L.³ But in many electroplating effluent water it is as high⁴ as 50 mg/L. Human body requires about 2 mg/L of copper but prolonged oral administration of excessive quantities of copper may result in liver damage and chronic poisoning and gastro-intestinal catarrh.⁵ Copper is also toxic to aquatic organisms even at very small concentrations.⁶ It is found in sizeable amounts in the liquid effluent streams of printed circuit board plants⁷ and other effluents. Iron(III) is also toxic at higher concentrations. It is desirable, therefore, to undertake investigations on the removal of these metal ions from water. Among the various cleanup methods available for metal ions removal, namely, electrolytic reduction, precipitation, oxidation, ultra filtration, ion exchange, adsorption, etc, adsorption (especially, activated carbon adsorption) appears to have the least adverse effects. It includes a broad range of carbonaceous materials at a high degree of porosity and large surface area⁸ and finds use for the removal of toxic, biodegradable and non-biodegradable substances from wastewaters. It is attractive as it can treat wastewater to acceptable quality suitable for reuse.

The removal efficiency of a commercial activated carbon (M/s. Loba Company, Mumbai) towards nickel(II), copper(II) and iron(III) was attempted in the present study. The effects of adsorbate dose, pH, time and temperature were studied.

EXPERIMENTAL

Adsorbent.

The granular activated carbon was ground and the portion retained between 150 and 250 μm sieves was used for study. The adsorbent was named as CC.

Characterization of CC.

Physicochemical characteristics such as, moisture content, density, volatile matter content, total ash content, water solubles, acid extractable content and pH were determined following standard procedures.⁹ Boehm titrations were performed to find out the amounts of surface functional groups.¹⁰ Liquid phase adsorption characterizations were done following the method of Maria J. Martin *et al.*¹¹ The infrared spectrum was recorded in an Impart - 420, Nicolet spectrometer. BET surface area of the sample was measured using nitrogen adsorption isotherms.

Surface characterization of prepared sorbents.

Iodine number

For the test with iodine, a stock solution is made with 2.7 g of iodine and 4.1 g of KI in 1 L distilled water. The adsorbent sample is dried for a minimum of 3 h in electric drying oven at 110 °C, and then 1.0 g of dried adsorbent is weighed on analytical balance produced by Bosch (Germany), having maximum sensitivity of 10^{-4} g and accuracy of $\pm 0.01\%$.

The sample is transferred into a dry, glass-stoppered, 250-mL Erlenmeyer flask. A 10-mL volume of 5% HCl is added to the flask and stirred until the adsorbent is wetted. After allowing the flask to cool at room temperature, 100 mL of standardized 0.1 N iodine solution is added to the flask. The flask is immediately stoppered and agitated vigorously for 30 sec.

The mixture is filtered by gravity immediately after the 30 sec. shaking period through a Whatmann filter paper. The first 20 to 30 mL is discarded and the rest are collected in a clean beaker. The residue on the filter paper must not be rinsed. The filtrate is stirred in a beaker with a rod, and 50 mL of filtrate is pipetted into 250 mL Erlenmeyer flask. The 50 mL is titrated with standardized 0.01 N sodium thiosulfate solutions until the yellow color has almost disappeared. Approximately 2 mL of starch solution is added and the titration continued until the blue indicator color just dis-

appears, the volume of sodium thiosulfate used is recorded.

Phenol and phenol number

A sample, 12.5 mL, of phenol (100 mg/L) is introduced into 100 mL Erlenmeyer flask. The adsorbent to be tested, 0.03 gm, is added and the flask shaken for 48 h at 25 °C. This procedure is repeated for several other concentrations of phenol. The adsorbent-phenol mixture is filtered through a 15 cm No.5 Whatmann filter paper under gravity, allowing all the solution to filter, and the concentration of phenol is determined by using UV-spectrophotometer at $\lambda_{max} = 271$ nm. The results so obtained provide the distribution of phenol at particular initial ratios of adsorbents masses to phenol concentration and volume. In doing so, one may construct an adsorption isotherm.

Analysis of metal ions

All the metals were estimated following a suitable colorimetric method. Nickel(II) was estimated by the dimethylglyoxime method,¹² copper(II) and iron(III) by the thiocyanate methods.^{13,14}

Isotherm procedure

Prior to isotherm studies, minimum contact times for adsorption equilibria to become established were estimated. Each experiment comprised three replicate 100 mL glasses stopper bottles containing appropriate amount of adsorbent and 50 mL of adsorbate solutions of selected concentrations. Control flasks without the adsorbents are also prepared simultaneously. Mixtures were maintained in a rotary shaker (orbitek) at constant temperature (30, 45 or 60 °C). After the attainment of equilibrium the contents of each flask were filtered through Whatmann (No.41) filter paper, with the first 10 mL discarded. The filtered samples were then analyzed for unadsorbed metal ions. The equilibrium adsorption data were then fitted to Freundlich and Langmuir isotherms equations:

$$\text{Freundlich} \quad q_e = K_F C_e^{(1/n)}$$

$$\text{Langmuir} \quad q_e = K_L C_e / (1 + b C_e) = q_m b C_e / (1 + b C_e)$$

where q_e is the adsorption capacity in mg/g; C_e is the equilibrium concentration of adsorbate (mg/L); K_F and n are Freundlich constants; K_L and b are Langmuir constants; q_m is the Langmuir monolayer adsorption capacity.

pH variation studies

In order to find out the optimum pH for maximum removal

of adsorbate, experiments were carried out with solutions of same metal ion concentration but adjusted to different initial pH values (with HNO₃ or NaOH). Measurements were carried out below which chemical precipitation of metal hydroxides do not occur. These values have been estimated to be 7.8 for Ni(OH)₂; 2.5 for Fe(OH)₃; and 7.5 for Cu(OH)₂.

Desorption Studies

Some desorption experiments were also conducted in order to explore the feasibility of recovering both the adsorbed species and the adsorbent and to elucidate the nature of adsorption processes. They were carried out as follows. After adsorption experiments using the selected adsorbent and adsorbate doses, the adsorbate loaded adsorbents were separated and washed gently with several portions of distilled water to remove any unadsorbed species. The samples were then air-dried and agitated with 0.1M solutions of HCl, AcOH or water for a period of 10 hours and the amounts of desorbed species were determined in the usual way.

RESULTS AND DISCUSSION

Characterization of the adsorbent

Iodine number

The iodine number is the number of mg iodine adsorbed by 1 g sorbent. Silica gels formed as a result of action of alkali leaching of the biomass followed its acid precipitation, show a good capacity of removing iodine which is shown in Table 1, these adsorbents uptake iodine in amounts somewhat lower than the N₂. It should be remarked that the iodine adsorption seems suitable also to estimate approximately the surface area of silica equally as well as activated carbon. Silica gels under the prescribed conditions possess a reasonable surface area mostly contained within micropores and mesopores, with a low contribution micro porosity which recommends them for purification and separation processes involving medium size molecules in addition to cationic species.

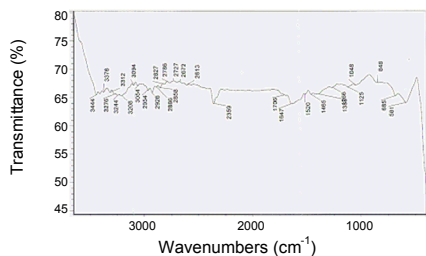
Phenolics removal capacity

Phenol number is the mass of adsorbent required to reduce the concentration of phenol/L water from 0.1 to 0.01 ppm. Phenolic compounds present one of the most frequent pollutants encountered in surface water and water streams. Their presence in minute amounts impart characteristic objectionable (undesirable) taste and odour.

A 100 mL of standard concentration for either phenol or p-nitro phenol compound was selected as 100 ppm (100 mg/L) mixed with constant weights of adsorbents and the observed

Table 1. Characteristics of Activated Carbon

Characteristics	Result	Characteristics	Result
Proximate Analysis, %		Iodine Number, mg/g	482.299
Moisture	1.6454	Phenol Value, g/L	46.610
Ash	16.7338		
Volatile matter	3.9360	Methylen Blue Number, mg/g	9.482
Fixed carbon	77.6848		
Particle size, μm	150 - 250	Tannin Value, mg/L	955.690
Surface area (N2-BET), m^2/g	503.1523	Carboxyl groups, mequiv/g	0.052
Apparent density, g/mL	0.7421	Phenolic groups, mequiv/g	0.205
Water solubles, %	1.8239	Lactonic groups, mequiv/g	0.163
Acid-extractable content, %	10.1276	Acidic groups, mequiv/g	0.420
pH	9.42	Basic groups, mequiv/g	0.452

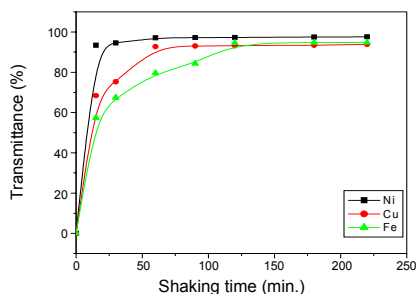
**Fig. 1.** FT-IR spectrum of CC.

percent removals of are shown in *Table 1*.

It is generally observed that:- Removal of phenol appear to be less feasible than p-nitro phenol under same experimental conditions (initial phenolic concentration, adsorbent weight, temperature, contact time, and volume of aqueous solution).

The parameters evaluated are listed in *Table 1*.

Table 1. reveals that the carbon under study is of high surface area and that it has quite good amounts of micro and mesopores as well. Apart from having the desired textural characteristics of a good adsorbent, the carbon was also found to have quite good amounts of surface groups like carboxyl, phenolic, lactonic and basic groups. The presence of these groups is also evident from the FT-IR spectrum of the carbon (*Fig. 1*). The IR spectra showed characteristic peaks of carboxylic acid. The peaks corresponding to carboxyl groups of frequency in the region 2954 cm^{-1} . There are small humps around $1705 - 1647\text{ cm}^{-1}$ characteristic of the carbonyl group of carboxylic acid. In addition to this, other characteristic peaks related to OH group of the carboxyl group are observed at 1760 , and 1125 cm^{-1} and lactones appear at $1160 -$

**Fig. 2.** Effect of Shaking time on sorption of Ni^{+2} , Cu^{+2} and Fe^{+3} ions.

1370 cm^{-1} and $1675 - 1790\text{ cm}^{-1}$ and C-H (stretching) appear at 2613 cm^{-1} .

Effect of shaking time on adsorption of different metal ions

The effect of shaking time on the adsorption of metals ions are shown in *Fig. 2*, the adsorption equilibrium takes place within 15 min. for Ni^{+2} onto CC with sorption medium molarity 0.01 M HCl (i.e. fast kinetic) compared to equilibrium steady state conditions for Cu^{+2} and Fe^{+3} onto sorbent RS-5 (equilibrium time takes place after 60 min. in case of Fe^{+3} with the same sorption medium).

Adsorption Isotherms: Adsorption Models

The assumptions associated with the Langmuir isotherm are well known;¹⁵ Adsorption can not proceed beyond a monolayer coverage and all adsorption sites are equivalent. The Freundlich model, on the other hand, assumes a heterogeneous adsorption surface with sites that have different energies of adsorption and are not equally available. For

Table 2. Isotherm parameters for the adsorption of metal ions on CC

Metal ion	Model	Model Parameters				
		K_f		1/n	n	
		mmol/g	mg/g			
Ni (II)	0.3869	1.4173	0.5065	1.9743	0.9589	
Cu(II)	Freundlich	0.4977	4.2362	0.2285	4.3764	0.9918
Fe(III)	0.2171	6.6024	0.1517	6.5920		

Metal ion	Model	Model Parameters			
		K_L	b	q_m	
		r^2			
Ni (II)	0.5272	0.4110	1.2827	0.9849	
Cu(II)	Langmuir	1.8132	2.4509	0.7398	0.9876
Fe(III)	5.6017	26.2230	0.2136	0.9260	

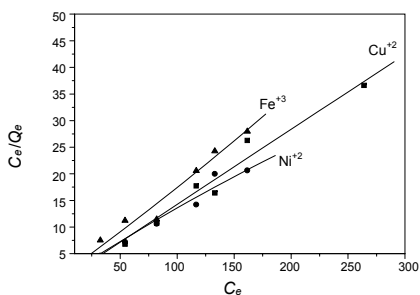


Fig. 3. Langmuir adsorption plot for the adsorption of Cu(II), Ni(II) and Fe(II) ions on activated carbon.

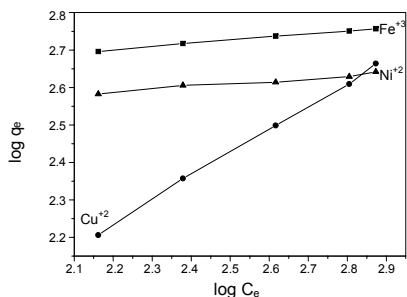


Fig. 4. Freundlich Adsorption isotherms of Ni²⁺, Cu²⁺ and Fe³⁺ ions on activated carbon.

each and every individual adsorption system, the data were fitted to the two isotherm equations and the results are given in Table 2 and Figs. 3 and 4 and [Conditions: adsorbent dose

0.1 g/50 mL of adsorbate solution; initial concentration of metal ion solutions, $C_i = 15 - 100$ mg/L for Ni; $20 - 100$ mg/L for Cu and $15 - 80$ mg/L for Fe(III)]. All the curves were of L type under Giles classification.¹⁶

According to Treybal¹⁷ it has been shown using mathematical equations that n values between 0 and 1 represent beneficial adsorption. Indeed, the n values found for all the adsorption systems fall in this range. The Langmuir constant b is a measure of adsorption intensity and the parameter q_m is a measure of adsorption capacity. Adsorption capacity of the adsorbents toward metal ions decrease in the order Ni(II), Cu(II), Fe(III) [values in Table 2], whereas the adsorption intensity decrease in the opposite order. The b values found indicate stronger interaction forces between carbon surface and Fe(III) ions compared to Ni(II) and Cu(II), in agreement with the higher ionic potential of Fe(III). The b values determined are further used to calculate the dimensionless separation factor, R_L ,^{18,19} defined as $R_L = 1/(1 + bC_i)$ where C_i is the initial solute concentration. The magnitude of R_L value gives an idea about the nature of adsorption equilibrium: the process is non-spontaneous when R_L is greater than one; favorable when R_L lies between 0 and 1; and irreversible when R_L is zero. In all the systems studied, R_L values were comprised between 0 and 1 (values not listed) indicating favorable adsorption of all the metal ions on the activated carbon.

Effect of pH

The effects of pH in external solutions on adsorption extent are presented in Fig. 5 (adsorbent doses: 0.1 g/50 mL, $C_i = 50$ mg/L).

For the three metal cations (Ni²⁺, Cu²⁺ and Fe³⁺) increase in solution pH through out the range studied resulted in increased adsorption. This trend is in agreement with the fact that the metal ions are adsorbed by ion-exchange mecha-

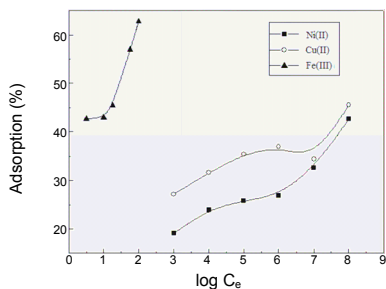


Fig. 5. Adsorption of metals on CC: Effect of pH.

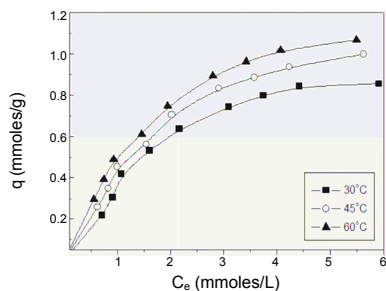


Fig. 6. Effect of temperature: Adsorption of Ni(II) on activated carbon.

nism. As the solution pH is lowered, concentration of H⁺ ions will increase proportionally which will effectively compete with metal cations for active adsorption sites on carbon surface, thereby reducing the available sites for metal cations.

Effect of Temperature

The equilibrium studies for all the systems were conducted at two more temperatures in addition to room temperature (30 °C), namely 45 and 60 °C in Fig. 6. For all the systems increase in temperature resulted in greater adsorption. A representative graph for showing the effect of temperature is shown in Fig. 3 for the Ni (II)-CC system.

The increased adsorption at higher temperatures can be due to one or more of the following reasons. Acceleration of some originally slow step(s)²³ creation of some new activation sites on the adsorbent surface²⁴ and decrease in the size of the adsorbing species.²⁵ This could well occur due to progressive desolvation of the adsorbing ion as the solution temperature increases.

Thermodynamic parameters such as Gibbs's free energy

change (ΔG), enthalpy change (ΔH) and entropy change (ΔS) were calculated using the following expressions:

$$K_C = \frac{C_{ae}}{C_e} \quad (1)$$

$$\Delta G = -RT \ln K_C$$

$$\log K_C = \left[\frac{\Delta S}{2.303R} \right] - \left[\frac{\Delta H}{2.303RT} \right]$$

Where K_C is the equilibrium constant for the distribution of metal ions between the liquid and solid phases; C_{ae} is the solid phase metal ion concentration, mg/L; C_e is the liquid phase metal ion concentration, mg/L; T is absolute temperature, °K and R the gas constant. Equation (1) was used to construct Van't Hoff plots and ΔH and ΔS were calculated from the slope and intercept of the Van't Hoff plot, respectively. Thermodynamic parameters evaluated for varied metal ion concentrations are listed in Table 3.

The ΔG values obtained for most CC-metal ion systems were positive indicating that CC is less effective in removing metal ions from aqueous solutions. The positive values of ΔH obtained for all the processes are further confirmations that they are endothermic, which is an indication of strong interaction between the adsorbate and the adsorbent. The positive values of ΔS suggest increased randomness at the solid-liquid interface during the adsorption of metal ions. The adsorbed solvent (water) molecules, which are displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate ions. Furthermore, before the adsorption process takes place the adsorbate ions are heavily solvated (the system is more ordered) and this order is lost when the ions are adsorbed on the surface, due to the release of solvated water molecules.

Desorption Studies

Attempts were made to regenerate the adsorbed metal ions with water, 0.1 M acetic acid and 0.1 M hydrochloric acid as regenerating agents. The results are presented in Table 4.

These results indicate that the metal ions are adsorbed by ion-exchange mechanism by the surface groups present on the carbon surface.

CONCLUSION

The work described has shown that the activated carbon under study can be successfully used for the adsorptive removal of metal ions from solution. The carbon possessed good textural and chemical properties. The three parameter

Table 3. Thermodynamic parameters for the adsorption of metal ions on CC

Metal Ion	Ci (mg/L)	ΔG (kJ mol ⁻¹)			ΔH (kJ mol ⁻¹)	ΔS (JK ⁻¹ mol ⁻¹)
		30 °C	45 °C	60 °C		
Ni(II)	15	1.2332	0.4290	-0.1344	1.5192	46.0948
	25	0.6259	0.2501	-0.1250	8.3305	25.4083
	35	1.0334	0.8013	0.4532	6.4164	17.6900
	45	1.3395	0.9146	0.6598	7.5700	20.6349
	60	1.8508	1.4022	1.1286	8.0422	20.5066
	70	2.1516	1.7710	1.4510	7.7833	18.5995
	80	2.4279	2.0570	1.7471	7.5631	16.9625
100	3.1272	2.6101	2.3842	8.2926	17.1807	
Cu(II)	20	-1.4933	-1.8412	-2.1525	-	-
	40	0.5437	0.2723	0.0857	5.0964	15.0573
	60	1.7783	1.3624	1.0723	7.8549	20.1026
	80	2.4484	2.0748	1.8570	6.5878	13.7266
	100	3.1185	2.6670	2.4018	7.9800	16.1123
Fe(III)	20	-3.6507	-3.9855	-4.0034	-	-
	30	-1.3317	-1.4240	-1.7235	40.3057	148.6469
	40	0.0063	-0.3659	-0.6050	39.6068	148.8901
	50	0.4643	0.2764	-0.1250	39.1781	149.5028
	60	1.1151	0.8407	0.5514	-	-
	80	2.1857	2.0171	1.6457	-	-

Table 4. Results of desorption

Metal ion	% Desorption with		
	Water	0.1 M AcOH	0.1 M HCl
Ni(II)	24.4	50.1	77.9
Cu(II)	29.6	49.4	85.5
Fe(III)	31.5	43.3	78.4

Redlich-Peterson model can be used to represent the equilibrium adsorption of Ni(II), Cu(II) and Fe(III) and was found to be superior than Freundlich and Langmuir models. Increase in solution pH result in greater retention of metal ions on CC. The adsorption processes were found to be endothermic and the thermodynamic parameters were evaluated. Dilute hydrochloric acid can be used for desorption of these metal ions.

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