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Adsorption of Copper(II) by “Waste” Fe(III)/Cr(III) Hydroxide from Aqueous Solution and Radiator Manufacturing Industry Wastewater

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ABSTRACT

“Waste” Fe(III)/Cr(III) hydroxide has been used as an adsorbent for the effective removal of copper from aqueous solution. The parameters studied include agitation time, Cu(II) concentration, adsorbent dose, temperature, and pH. The percent adsorption of Cu(II) increased with a decrease in the concentration of Cu(II) and an increase in temperature. Quantitative removal of Cu(II) by 50 mg/50 mL adsorbent was observed at pH 5.0 for a Cu(II) concentration of 40 mg/L. The equilibrium data fit well with the Langmuir isotherm. The adsorption capacity (Q_0) calculated from the Langmuir isotherm was 92.59 mg/g at an initial pH of 5.0 at 32°C. Desorption of Cu(II) from a Cu(II)-loaded adsorbent was 55.4% at pH 3.0. Application of the adsorbent for the removal of Cu(II) was successfully demonstrated using radiator manufacturing industry wastewater.

INTRODUCTION

Environmental pollution due to copper arises from industrial and agricultural emissions. It is found in municipal wastes as a by-product from the metal finishing and processing industry and agricultural sources such as fertilizers and fungicides (1). The tolerance limit for copper for discharge into

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inland surface waters is 3.0 mg/L (2) and in drinking water is 0.05 mg/L (3). As a result, there is concern regarding the fate and transport of copper in soils and aquatic systems.

Conventional methods for the removal of Cu(II) from wastewater include precipitation coagulation/flocculation ion-exchange, cementation, complexation/sequestration, electrochemical operation, and biological treatments (4). These methods are not cost-effective in the Indian context. The search for efficient, cost-effective options for treating metal-bearing wastes has intensified in recent years in light of increasingly stringent discharge limits. A number of low-cost, nonconventional adsorbents including nontoxic agricultural and industrial solid wastes for the treatment of wastewater containing heavy metals and dyes have been recently reviewed (5, 6).

Fe(III)/Cr(III) hydroxide is produced in the treatment of wastewaters containing Cr(VI). Hexavalent chromium is reduced to trivalent chromium using Fe(II), which is generated electrolytically (4). The resulting Fe(III)/Cr(III) hydroxide ions are precipitated as hydroxides by the addition of lime, which is discarded as waste material. Recycling solid wastes not only helps the economics of wastewater treatment but also helps solve solid waste disposal problems. In developing countries like India, which is heavily populated, most industries do not treat effluents properly before they are discharged into rivers. Solid waste treatment methods encourage industries to adopt pollution control measures.

Namasivayam and coworkers have employed waste Fe(III)/Cr(III) hydroxide for the treatment of wastewaters from industries such as fertilizer (7), dairy (8), dyeing (9, 10), distillery (11), tannery (12), and the removal of toxic ions and dyes (13). It is also efficient for the removal of chromium(VI) (14), iron(II) (15), nickel(II) (16), lead(II) (17), cadmium(II) (18), mercury(II) (4), a mixture of Pb(II), Cd(II), and Ni(II) (19), and pesticides (20) from wastewaters.

The objective of the present study was to examine the kinetics of adsorption and to evaluate the adsorption capacity of the "waste" Fe(III)/Cr(III) hydroxide for the removal of copper(II) from aqueous solution and radiator manufacturing industry wastewater. Parameters studied include initial Cu(II) concentration, agitation time, temperature, and pH.

EXPERIMENTAL

Materials

Dry Fe(III)/Cr(III) hydroxide, obtained from Southern Petrochemical Industries Corporation Limited (SPIC, Tuticorin, Tamil Nadu, India) was used as the adsorbent. It was ground, washed with distilled water to remove very fine particles, dried at 60°C for 16 hours, and sieved to different particle



TABLE 1
Characteristics of "Waste" Fe(III)/Cr(III) Hydroxide

Parameter	
Apparent density, g/L	0.895
Acid insoluble matter, % (w/w)	9.66
Water-soluble matter (pH range 3.5–10.0):	
Fe(III), mg/L	<0.10
Cr(III), mg/L	<0.05
Mechanical moisture content, % (w/w)	16.78
Loss on ignition, % (w/w)	32.80
Total Fe, % (w/w)	29.30
Total Cr, % (w/w)	5.30
Total Ca, % (w/w)	5.00
pH of 0.2 % (w/w)	8.11
Conductivity of 0.2% solution, $\mu\text{S}/\text{cm}$	68.20
Surface area, m^2/g	424.00
pH_{zpc}	8.30

size ranges (0.075–0.15, 0.15–0.25, 0.25–0.50 mm). The characteristics have been reported (19) (reproduced in Table 1). A stock solution of Cu(II) (1000 mg/L) was prepared from $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in distilled water with a few drops of concentrated nitric acid to prevent hydrolysis. The radiator manufacturing industry wastewater containing copper was collected at Coimbatore, India, and characterized as per standard procedures (21). All chemicals used were of analytical reagent grade and were obtained from SD's and Glaxo/BDH.

Batch Mode Adsorption Studies

Our adsorption study was carried out by agitating 50 mg of Fe(III)/Cr(III) hydroxide with 50 mL of Cu(II) solution of the desired concentration in 100 mL glass conical flasks at 120 rpm for a predetermined time using a temperature-controlled shaking water-bath. The pH was adjusted using dilute nitric acid or sodium hydroxide solutions. The solution was agitated as above for the time required to attain equilibrium. The adsorbent and adsorbate were separated by centrifugation at 10,000 rpm, 8700g for 40 minutes, and Cu(II) was estimated spectrophotometrically (22).

Langmuir isotherm studies were carried out with different initial concentrations of Cu(II) from 40 to 200 mg/L and a fixed concentration of adsorbent, 2 g/L. The adsorbent did not release either Cr(III) or Fe(III) into water in the initial pH range studied (3.0–10.0) (20).

To correct for any adsorption of Cu(II) on containers, control experiments were carried out without adsorbent. There was no adsorption by container walls.



Experiments were generally carried out with a Cu(II) concentration of 40 mg/L and an adsorbent dose of 50 mg per 50 mL and at 32°C, unless otherwise stated. Experiments were carried out in duplicate. The error was within 5%.

Desorption Study

After an adsorption experiment with 40 mg/L of Cu(II) and 1.0 g/L of adsorbent, the adsorbent laden with Cu(II) was separated and gently washed with distilled water to remove any unadsorbed Cu(II). Several such spent adsorbent samples were prepared. Then the spent adsorbent was agitated with 50 mL of acidified water, adjusted to different pH values varying from 3.0 to 7.0, and agitated for 120 minutes at 120 rpm at 32°C. The desorbed Cu(II) was analyzed as before.

Different doses of adsorbent (25–200 mg) were added to 50 mL of the industrial wastewater and agitated for 5 hours at 120 rpm at 32°C. An aliquot (10 mL) of the supernatant was digested with a mixture of 10 mL concentrated hydrochloric acid and 5 mL concentrated nitric acid over hot sand bath for 30–45 minutes. The solution was made up to 50 mL with water. Copper(II) was estimated as before.

RESULTS AND DISCUSSION

Effect of Agitation Time and Initial Concentration

Figure 1 shows that the removal of Cu(II) increased with agitation time and attained equilibrium after 30, 80, 120, and 150 minutes for initial concentrations of 10, 20, 30, and 40 mg/L, respectively. The percent adsorption increased with a decrease in Cu(II) concentration at any time prior to equilibrium. At equilibrium the percent removal was close to 100.

Effect of Agitation Time and Particle Size

Figure 2 shows that the percent removal increased from 25.25 to 99.50 with a decrease in particle size of the adsorbent until equilibrium. This shows that the smaller the adsorbent particle size, the faster will be the adsorption rate at any agitation time prior to equilibrium. The equilibrium times for particle size ranges of 0.075–0.150, 0.150–0.250, and 0.250–0.500 mm were 20, 150, and 180 minutes, respectively. At equilibrium the percent removal reached close to 100. A longer equilibrium time for larger particle sizes is required to make intraparticle diffusion efficient.

Effect of Agitation Time and Temperature

The effect of agitation time at different temperatures on Cu(II) adsorption by “waste” Fe(III)/Cr(III) hydroxide showed an increase in percent removal



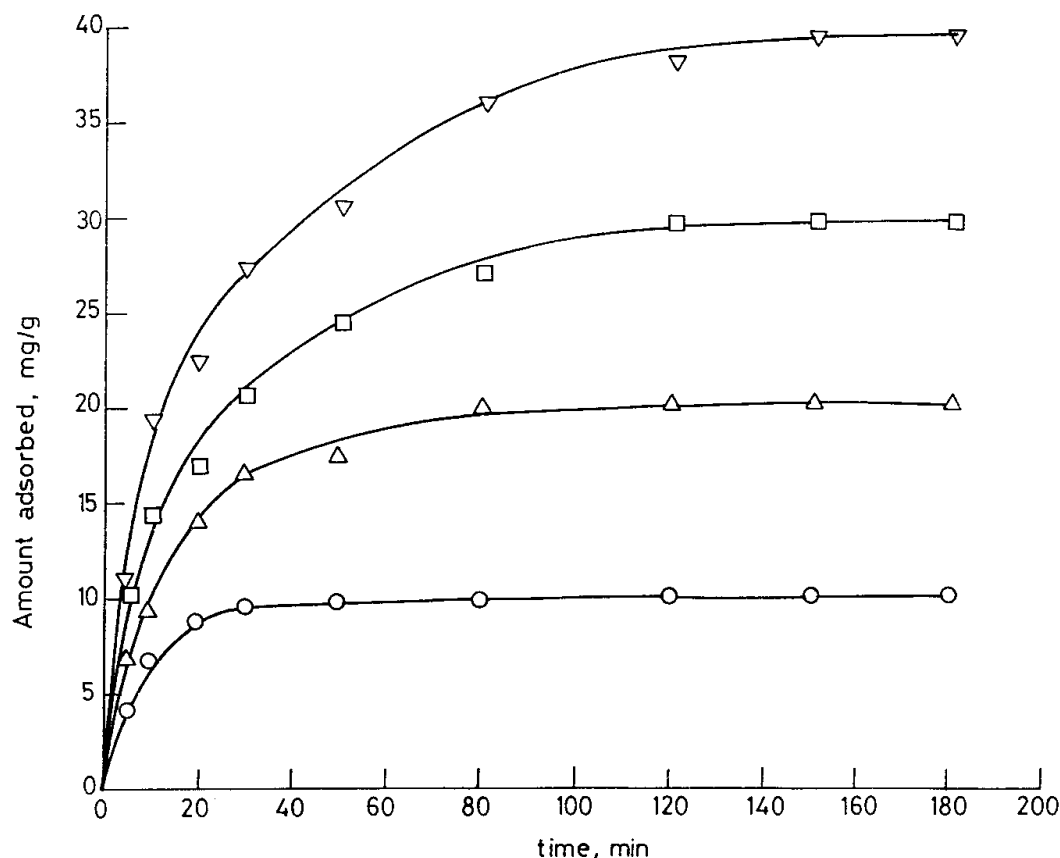


FIG. 1 Effect of agitation time and initial concentration of Cu(II) on the adsorption of Cu(II) on "waste" Fe(III)/Cr(III) hydroxide. Conditions: Cu(II) concentration = 10 mg/L (○); 20 mg/L (△), 30 mg/L (□), 40 mg/L (▽); adsorbent dose = 50 mg/50 mL; pH = 5.0; particle size = 0.15–0.25 mm; temperature = 32°C.

of Cu(II) with an increase in temperature. The equilibrium times were 180, 180, 150, and 150 minutes for 12, 22, 32, and 42°C, respectively. The percent adsorption at equilibrium increased from 90.6 to 100 with an increase in temperature from 12 to 42°C. Figures 1 and 2 show plots that are smooth and single, indicating monolayer adsorption of Cu(II) on the adsorbent (23). The increase in adsorption of Cu(II) with temperature is probably due to "activated diffusion." Such activated adsorption would widen and deepen the very small micropores, i.e., cause "pore burrowing" and so create more surface for adsorption.

Assuming that the activity coefficients are unity at low concentrations (in the Henry's law sense), thermodynamical parameters were calculated using the following relations (14):



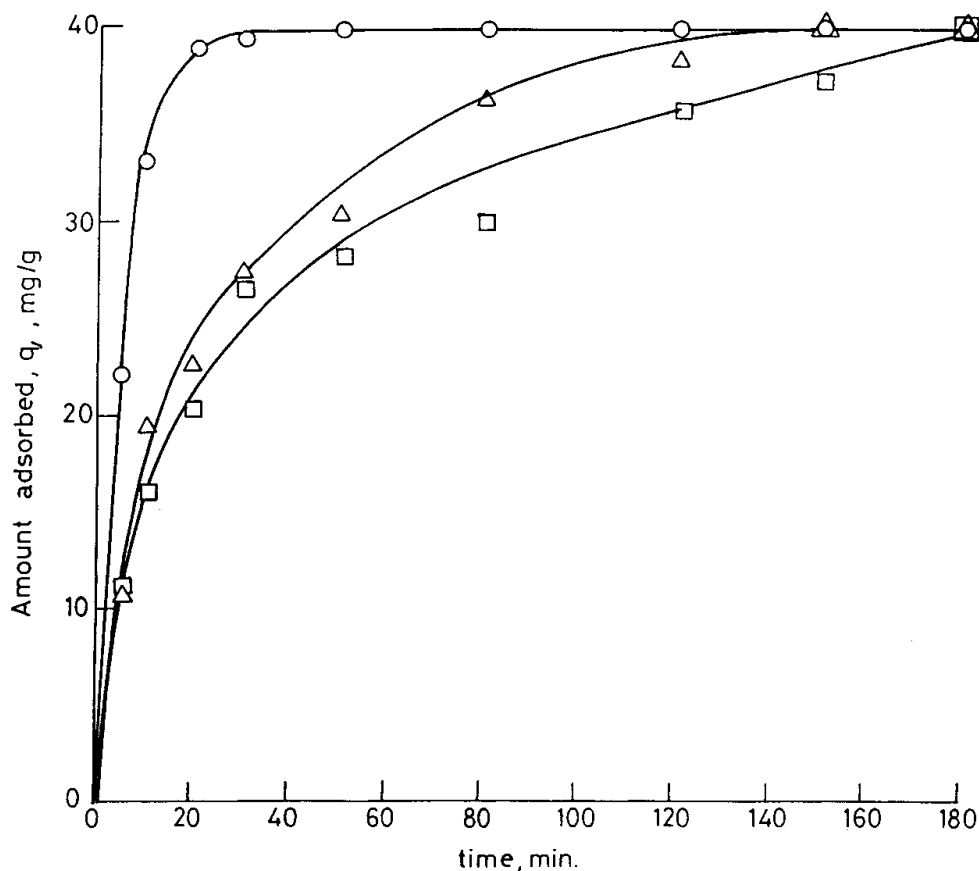


FIG. 2 Effect of agitation time and particle size on Cu(II) removal. Conditions: Cu(II) concentration = 40 mg/L; pH 5.0; adsorbent dose = 50 mg/50 mL; temperature = 32°C; particle size = 0.075–0.15 mm (○), 0.15–0.25 mm (△), 0.25–0.50 mm (□).

$$K_c = C_{Ae}/C_e \quad (1)$$

$$\Delta G = -RT \ln K_c \quad (2)$$

$$\log_{10} K_c = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \quad (3)$$

where K_c is the equilibrium constant, C_e is the equilibrium concentration in solution (mg/L) and C_{Ae} is the solid-phase concentration at equilibrium (mg/L). ΔG , ΔH , and ΔS are changes in free energy, enthalpy, and entropy, respectively. ΔH and ΔS were obtained from the linear van't Hoff plot of $\log_{10} K_c$ vs $1/T$ and are presented in Table 2. The positive value of ΔH confirms the endothermic adsorption of Cu(II) on Fe(III)/Cr(III) hydroxide. The negative values of ΔG (Table 2) indicate that the adsorption is spontaneous. The positive values of ΔS suggest increased randomness at the



solid–solution interface during the adsorption of Cu(II) ions on Fe(III)/Cr(III) hydroxide. In the adsorption of Cu(II), the adsorbed solvent molecules, which are displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate ions, thus allowing for the prevalence of randomness in the system (14).

Effect of Adsorbent Dose

Figure 3 shows that the removal of Cu(II) increased with adsorbent dose (10 to 200 mg per 50 mL) for the different concentrations of Cu(II) (10 to 40 mg/L) studied.

Adsorption Isotherm Studies

The equilibrium data were correlated by the Langmuir equation (24). The Langmuir treatment is based on the assumption that maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on the adsorbent surface, that the energy of adsorption is constant, and that there is no transmigration of adsorbate molecules in the plane of the surface. The isotherm data in Fig. 4 are well described by the linear form of the Langmuir equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0} + \frac{C_e}{Q_0 b} \quad (4)$$

where C_e is the equilibrium concentration (mg/L), q_e is the amount adsorbed at equilibrium (mg/g), b is the “affinity” parameter or Langmuir constant (L/mg), and Q_0 is the “capacity” parameter (mg/g). Q_0 and b were determined from the slope and intercept of the Langmuir plot and are 92.59 mg/g and 0.5714 L/mg, respectively.

Values of Q_0 and b of other metal-oxide-based nonconventional adsorbents are given in Table 3 for comparison. The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless parameter, R_L (16). R_L describes the type of isotherm and is defined as $R_L = 1/(1 + bC_0)$,

TABLE 2
Thermodynamical Data

Temperature (°C)	K_c	$-\Delta G$ (kJ/mol)	ΔH (J/mol)	ΔS (J/mol/k)
12	9.13	5.24	21.5	94.2
22	13.29	6.34		
32	19.00	7.47		
42	21.22	8.00		



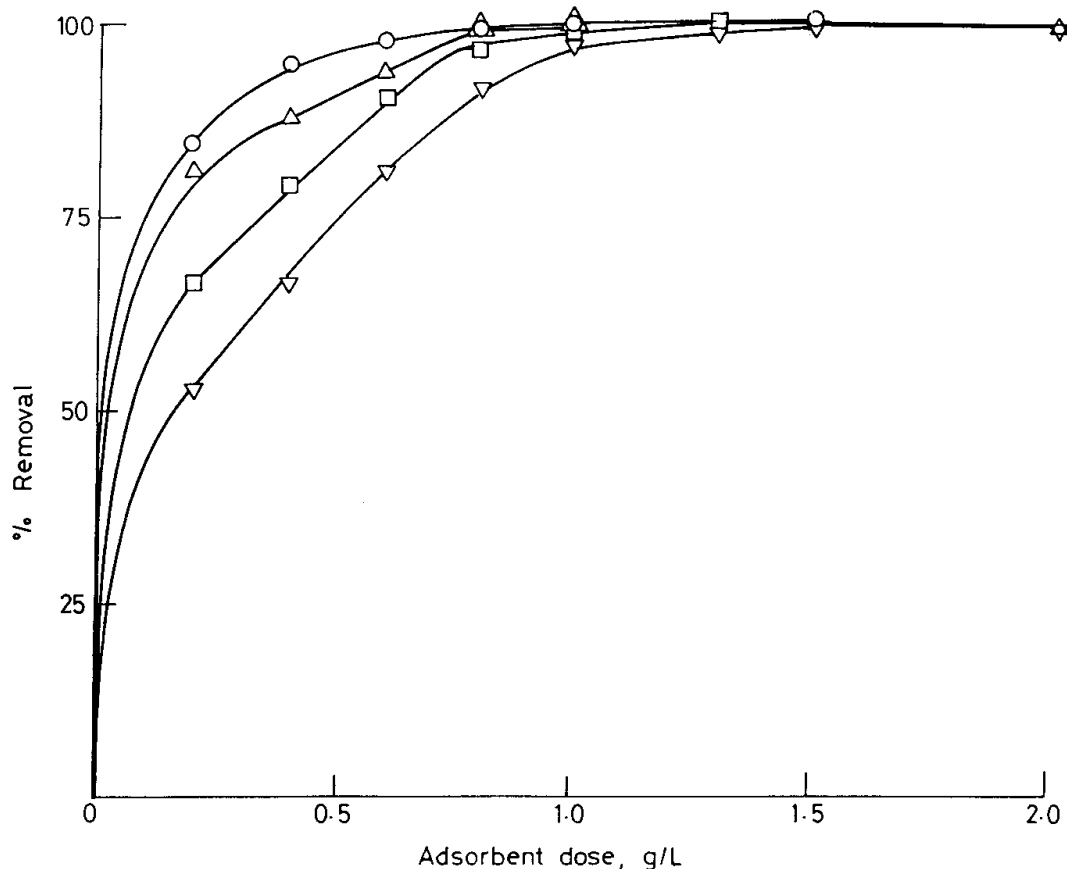


FIG. 3 Effect of adsorbent dose and initial concentration of Cu(II) on the adsorption. Conditions: Cu(II) concentration = 10 mg/L (○), 20 mg/L (△), 30 mg/L (□), 40 mg/L (▽), initial pH 5.0; agitation time = 160 minutes; particle size = 0.15–0.25 mm; temperature = 32°C.

where b is the Langmuir constant and C_0 is the initial concentration of Cu(II). R_L values between 0 and 1 indicate favorable adsorption of Cu(II) on Fe(III)/Cr(III) hydroxide for all concentrations of Cu(II) and temperatures studied.

Adsorption Dynamics

The two important aspects for parameter evaluation of the adsorption study are the kinetics and equilibria of adsorption. The adsorption of heavy metals from the liquid phase to the solid phase can be considered as a reversible reaction with equilibrium being established between two phases (16). A simple first-order kinetic model is represented by the Lagergren equation.

$$\log_{10}(q_e - q) = \log_{10} q_e - k_{ad}t/2.303 \quad (5)$$

where k_{ad} (1/min) is the rate constant of adsorption, and q and q_e are the amounts of Cu(II) adsorbed (mg/g) at time t (min) and at equilibrium time, respectively. Values of k_{ad} were obtained from linear plots of $\log_{10}(q_e - q)$



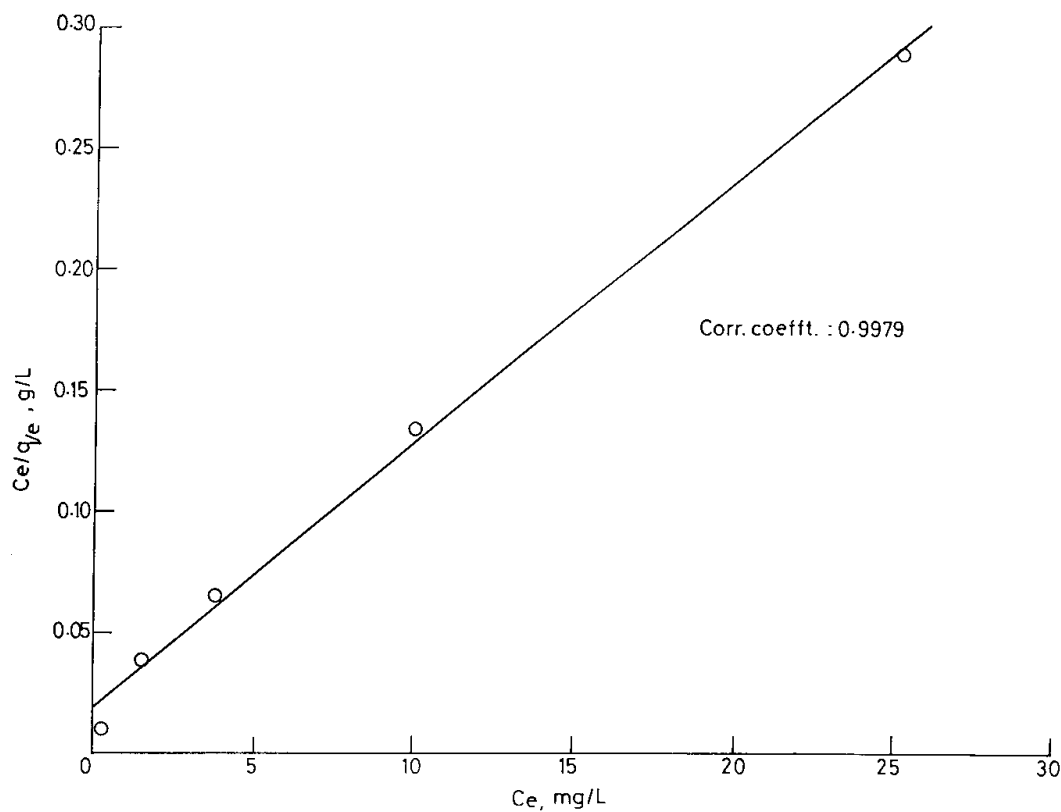


FIG. 4 Langmuir plot for Cu(II) adsorption. Conditions: Initial pH 5.0; adsorbent dose = 100 mg/50 mL; agitation time = 120 minutes; temperature = 32°C; particle size = 0.15–0.25 mm.

TABLE 3
Comparison of Adsorbent Capacity of Other Adsorbents

Adsorbent	Q_0 (mg/g)	b (L/mg)	Reference
Raw fly ash	207.30	0.94	28
“Waste” Fe(III)/Cr(III) hydroxide	92.59	0.57	This work
Red mud washed with water	75.20	0.96	28
Chemically modified silica gel	16.52	0.05	30
Bentonite	2.32	1.18	29



vs t for concentrations of 10, 20, 30, and 40 mg/L and found to be 0.1298, 0.0569, 0.0276 and 0.0260 1/min, respectively.

EFFECT OF pH

One of the conventional methods of removing metals from waters is their precipitation as metal hydroxides using alkali/lime. This method has the limitation that metal cannot be completely removed from solution owing to the solubility product of metal hydroxides. Hence, comparison is made between adsorption of Cu(II) by Fe(III)/Cr(III) hydroxide and precipitation as a metal hydroxide (Fig. 5). Throughout the pH range investigated ($3.0 < \text{pH} < 10.0$),

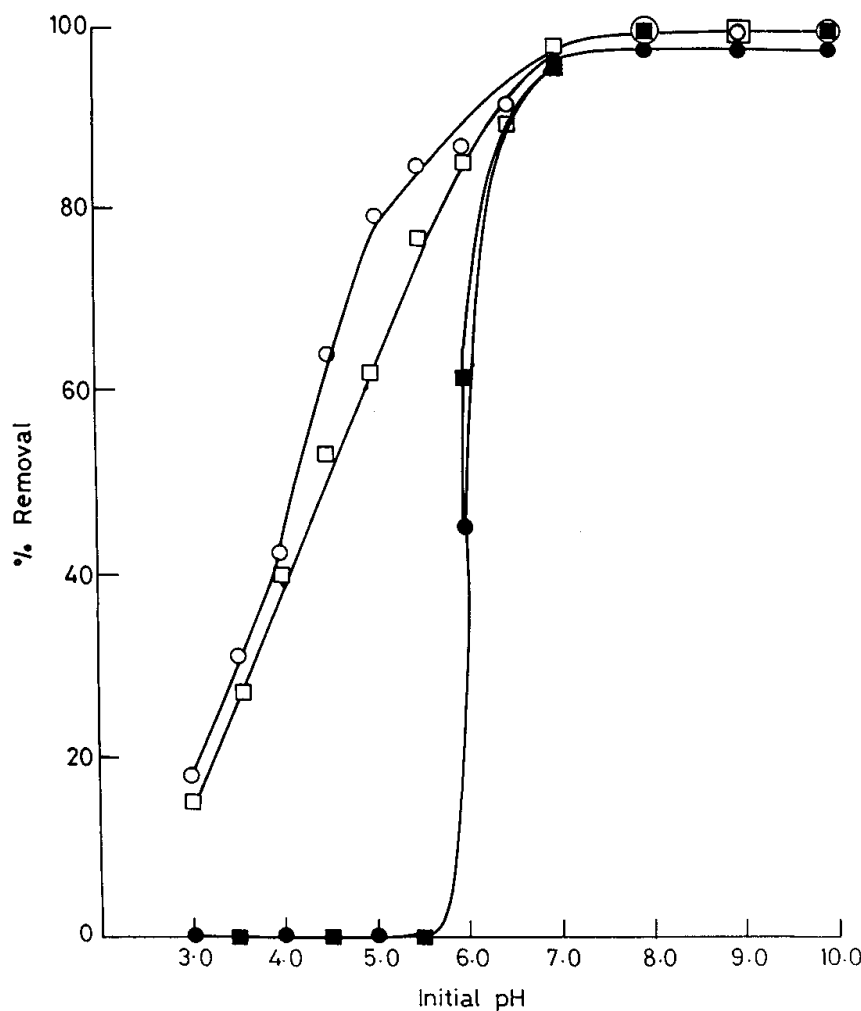
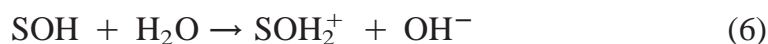


FIG. 5 Effect of pH on Cu(II) removal. Conditions: Cu(II) concentration = 20 mg/L (\circ), 30 mg/L (\square); adsorbent dose = 10 mg/50 mL; particle size = 0.15–0.25 mm; temperature = 32°; agitation time = 3 hours; copper hydroxide precipitation = 20 mg/L (\bullet), 30 mg/L (\blacksquare).



Cu(II) removal in the system with Fe(III)/Cr(III) hydroxide was more efficient than the Fe(III)/Cr(III) hydroxide-free system. Precipitation of copper starts at pH 5.5 whereas adsorption starts at a lower pH, 3.0. Adsorption of a metal cation on an adsorbent depends upon the nature of the adsorbent surface and the species distribution of the metal cation. Species distribution mainly depends on the pH of the system. The primary metal ion species in the pH range studied are Cu^{2+} and $\text{Cu}(\text{OH})^+$. The percent adsorption of copper decreased with the decrease in pH of the system because protons compete with the metal ion for sorption sites on the adsorbent surface as well as the concomitant decrease of the negative charge of the same surfaces. Similar results of the pH effect were reported for the adsorption of copper on ferric hydrite (25). Appreciable adsorption of Cu(II) occurred at pH values below pH_{zpc} (the pH at which the adsorbent surface is neutral) of the adsorbent (8.3) although the adsorbent surfaces were mostly positively charged. Since electrostatic attraction was not possible between the positively charged adsorbent surface and the positively charged metal ion species [Cu^{2+} and or $\text{Cu}(\text{OH})^+$], it seems that some nonelectrostatic force called specific adsorption was involved in the adsorption process.

The surface is considered to be nonpolar at pH_{zpc} , but in actual practice hydroxide complexes (SOH) are present. They render the surface slightly polar depending on the pH. Since there is no satisfactory method for determining the polar character of the surface quantitatively, the above statement is relative (26). The surface hydroxide complex hydrolyze water molecules as shown by (27),



Since lime is added for the treatment of electrolytically-generated Fe(III)/Cr(III) ions for precipitation, SOCa^+ and $(\text{SO})_2\text{Ca}$ are also assumed to be present. Ca^{2+} in the above groups is also exchanged with H^+ in the medium as follows:



Figure 6 shows the effect of initial pH on final pH at different concentrations of Cu(II). The curve referred to as “blank” was obtained under conditions such that $[\text{Cu(II)}] = 0$. Reactions (6) to (8) contribute to an increase in pH in the blank curve. When Cu(II) is present in solution, its adsorption will free some H^+ and the final pH will be lower than that in the blank (Eqs. 9 and 10). This is also observed in the 3–7 pH range. Release of Ca^{2+} ions was also observed according to Eqs. (13) and (14). Tests performed by agitating 50 mg of adsorbent for 3 hours at an initial pH of 5.0 led to a calcium



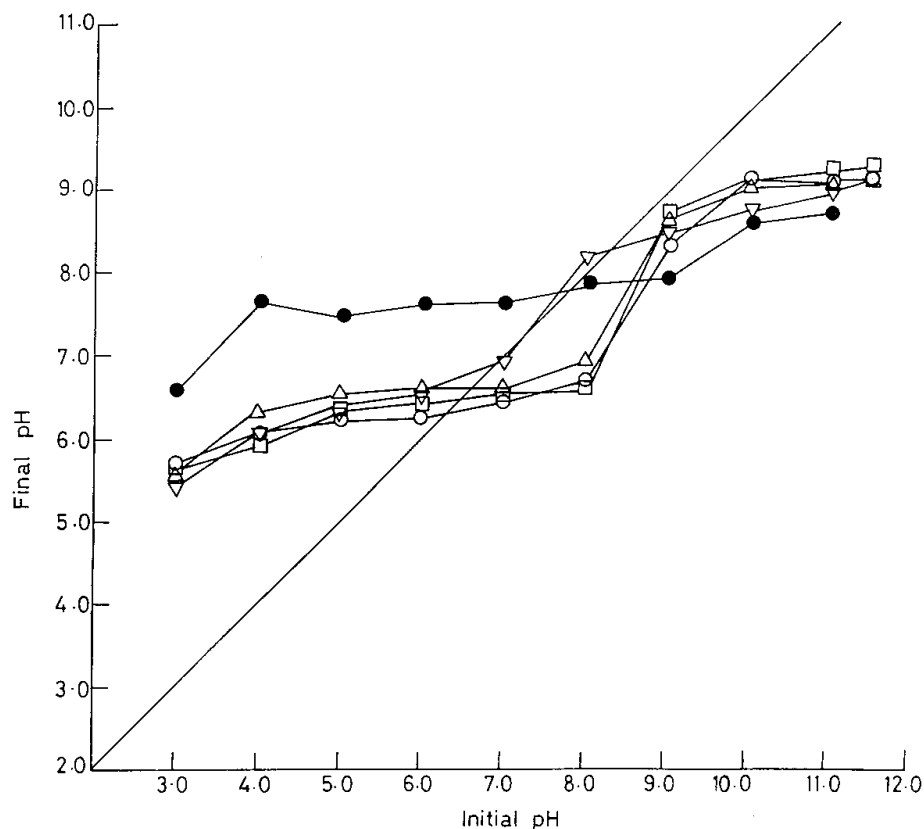
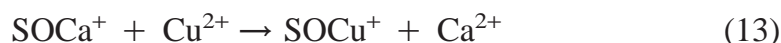
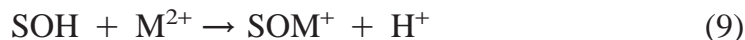


FIG. 6 Effect of pH on final pH on the adsorption of Cu(II). Conditions: (●) blank ($[\text{Cu(II)}] = 0$; adsorbent dose = 50 mg/50 mL; particle size = 0.15–0.25 mm; temperature = 32°C; agitation time = 3 hours; Cu(II) concentration = 10 mg/L (○), 20 mg/L (△), 30 mg/L (□), 40 mg/L (▽).

concentration of 2.0 mg/L. Calcium release was found to increase from 2.0 to 5.0 mg/L when the Cu(II) concentration increased from 0 to 40 mg/L. This shows that apart from the exchange of H^+ ions on the adsorbent with Cu(II) ions, significant Ca^{2+} ions which were present in the adsorbent were also exchanged with Cu(II) ions.



where SO^- denotes a negatively charged surface, SOH denotes a neutral surface, and M denotes a copper metal ion. With an increase in metal ion concentration, the final pH should decrease correspondingly. As the difference in the final pH values is within the error limits (3%), the decreasing trend in the final pH values with an increase in $Cu(II)$ concentration could not be observed.

DESORPTION STUDIES

Desorption studies help recycle the adsorbent and recovery of metal, apart from protecting the environment from solid waste disposal in landfills. The percent desorption increased with a decrease in pH and reached 60% at pH 3.0. H^+ ions protonate the adsorbent surface at acidic conditions, leading to desorption of the positively charged metal ion species. At $pH \geq 3.0$, the adsorbent did not release either $Fe(III)$ or $Cr(III)$ into solution (20).

Test with Copper Containing Radiator Manufacturing Industry Wastewater

The characteristics of radiator manufacturing industry wastewater are shown in Table 4. The effect of pH on copper removal by “waste” $Fe(III)/Cr(III)$ hydroxide is shown in Fig. 7(A). Increasing the pH increased copper removal. The effect of adsorbent dosage on copper removal is shown in Fig. 7(B). There was complete removal of $Cu(II)$ from 50 mL of wastewater containing 125 mg/L $Cu(II)$ at 125 mg of adsorbent dosage.

TABLE 4
Characteristics of Radiator Manufacturing
Industry Wastewater

Parameter	Value
pH	2.91
Turbidity, NTU	Nil
Total solids, mg/L	5264.00
Sulfate, mg/L	251.25
Chloride, mg/L	1489.32
Hardness as $CaCO_3$, mg/L	1868.50
COD, mg/L	140.00
Calcium, mg/L	63.50
Sodium, mg/L	218.75
Potassium, mg/L	11.50
Copper, mg/L	125.00



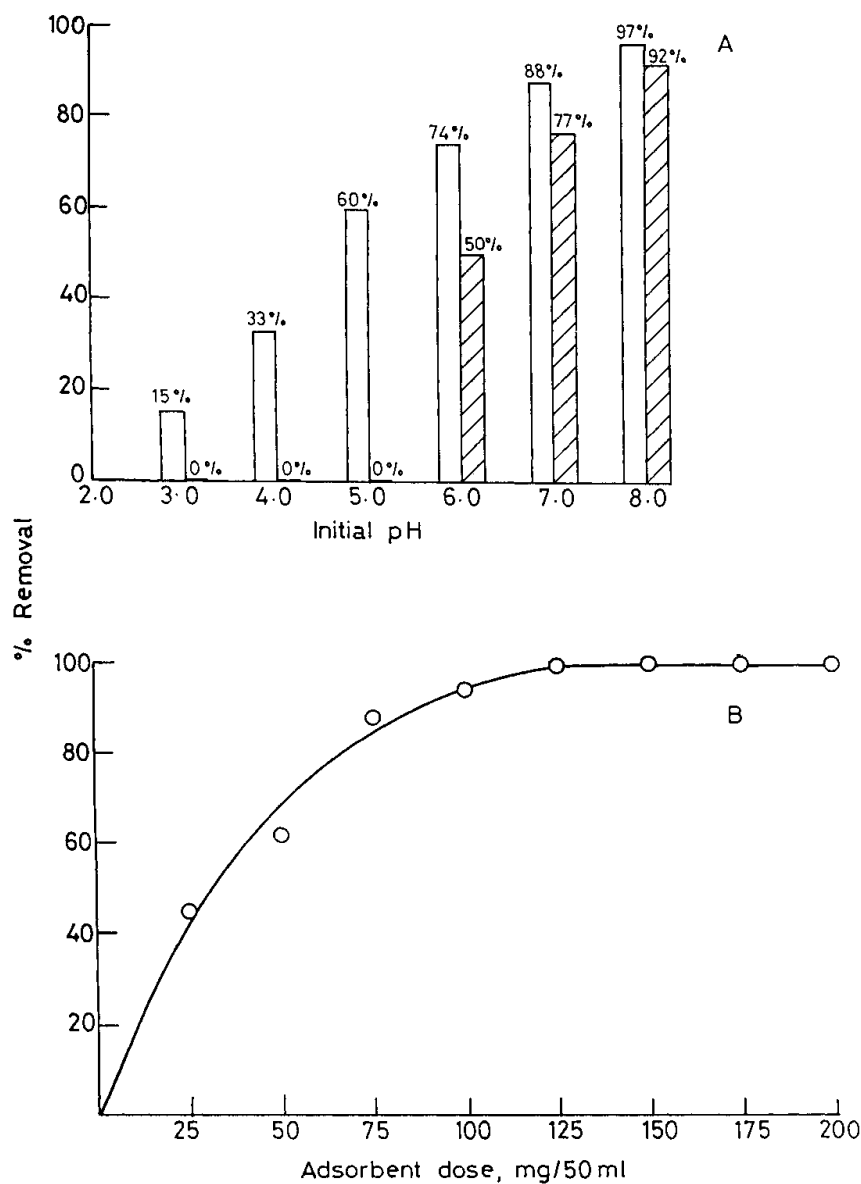


FIG. 7 (A) Effect of pH on the removal of Cu(II) from radiator manufacturing industry wastewater. Conditions: Agitation time = 5 hours, particle size = 0.15–0.25 mm; adsorbent dose = 50 mg/5–0 mL; temperature = 32°C; (□) adsorption; (▨) hydroxide precipitation. (B) Effect of adsorbent dose on the removal of Cu(II) from radiator manufacturing industry wastewater. Conditions: Initial pH 5.0; agitation time = 5 hours; particle size = 0.15–0.25 mm; temperature = 32°C.



The adsorbent, Fe(III)/Cr(III) hydroxide, is a solid waste in fertilizer industries. It requires a great deal of space for land disposal. Hence, it is available free of cost. The conventional adsorbents are not only expensive but also generate large volumes of solid sludge. Only the transportation cost is required to procure "waste" Fe(III)/Cr(III) hydroxide. Moreover, the adsorbent can be recycled after desorbing Cu(II) at pH 3.0. Hence, treatment using the waste Fe(III)/Cr(III) hydroxide seems to be cost-effective.

CONCLUSION

1. The results show that the "waste" Fe(III)/Cr(III) hydroxide can be used as an adsorbent for the effective removal of Cu(II) from aqueous solution.
2. Kinetic data would be useful for the fabrication and designing of wastewater treatment plants. The adsorption capacity was 92.59 mg of Cu(II) per gram of adsorbent.
3. Quantitative removal of copper from radiator manufacturing industry wastewater confirms the validity of results obtained in batch mode studies.
4. Application of "waste" Fe(III)/Cr(III) hydroxide to wastewater treatment is expected to be economical.

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