₹ 200 ISSN - 2249-555X
Volume : 1 Issue : 7 April 2012

Indian Journal Of Applied Research

Journal for All Subjects

www.ijar.in
Listed in International ISSN Directory, Paris.

ISSN - 2249-555X

Indian Journal of Applied Research

Journal for All Subjects

Editor,
Indian Journal Of Applied Research
8-A, Banans, Opp. SLU Girls College, New Congres Bhavan, Paldi,
Ahmedabad-380006, Gujarat, INDIA
Contact.: +91-9824097643 E-mail : editor@ijar.in

INDEX

Volume : 1 | Issue : 7 | April 2012 ISSN - 2249-555X

Research Paper Chemistry

Heavy Metal Biosorption Using A Biopolymer Chitin

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ABSTRACT

In this study the low cost seafood processing wastes chitin was blended with bentonite with and without the presence of crosslinking agent and characterized using various analytical techniques (FTIR, DSC, TGA and XRD analysis), also binding of metals to polymers was investigated. The adsorption was studied by batch method. The influence of initial concentration, dosage of adsorbent, contact time and pH were experimentally verified. Adsorption studies were done on both cross-linked and non-crosslinked chitin for Cr(VI) and Cu(II) ion. The adsorption behavior could be described using the Langmuir and Freundlich isotherm, the thermodynamic and equilibrium parameters of the reaction were determined to understand the sorption behavior of chitin blend. The results revealed that the adsorption of above metals by chitin blend is spontaneous, endothermic and favorable. Hence, the blended copolymer could open way for waste water treatment in industrial level. The cross linking agent incorporated into the polymer blends to improve the mechanical properties such as surface hardness, stiffness, resistance to temperature and solvent attack.

Keywords : Adsorption, Thermal stability, blend, Effluent

Introduction

Industrial and mining waste waters are important sources of pollution of heavy metals (Quek et al., 1998). It is well known that Cr(VI) is toxic to living systems, several can even cause lung cancer and must be removed from waste water before it can be discharged (Howard, 2002; Zahir et al., 2005). The presence of Cu (II) in waste water is also a problem, when Cu is ingested at high concentration it can become toxic to humans, causing cancer and promoting oxidation.

A wide range of physical and chemical processes is available for the removal of Cr(VI) and Cu(II) from wastewater, such as electrochemical precipitation , ultra filtration, ion exchange, reverse osmosis (Rengaraj et al., 2001; Yurlova et al., 2002; Benito and Ruiz, 2002). A major drawback with precipitation is sludge production. Ion exchange is considered a better alternative technique for such a purpose. However, it is not economically appealing because of high operating cost. Adsorption using commercial activated carbon (CAC) can remove heavy metals from wastewater, such as Cr (Ouki et al., 1997) Cu (Monser and Adhoum, 2002). However, CAC remains an expensive material for heavy metal removal.

Natural biopolymers are industrially attractive because of their capability of lowering transition metal- ion concentration to parts per billion concentrations. Natural materials that are available in large quantities may have potential to be used as low cost adsorbents and are environmentally friendly (Deans and Dixon, 1992).

This study deals with the removal of Cr(VI) and Cu(II) from tannery effluent. Chitin is a polysaccharide and is a major component of arthropod and crustacean shells, like those of lobsters, shrimps or crabs (Deshpande, 1986). Chitin is a nontoxic and biodegradable [polymer of N-acetyl glucosamine and glucosamine residues (Gonzalez-Davila et al., 1990). It is easily filmable, spinnable and the materials processed have very good mechanical properties. It is insoluble in water and alkali but soluble in strong mineral acids in anhydrous formic acid.

Chitin forms complexes with transition metal ions (Dursun et al, 2005; Da silva et al., 2004), allowing recovery or removal of the transition metal ions from solution. Since the lone pair of electrons available on nitrogen in acetamido group (Kaminski et al., 1997) and hydroxyl group can serve as chelating sites.

A systematic study of the adsorption of Cr (VI) and Cu(II) by chitin blend in the presence of cross linking agent (Jayakrishnan et al., 1996) under different experimental conditions has been carried out to understand the nature of the reaction.

2. Experimental

2.1 Materials

Chitin was obtained from India sea foods, Cochin which is 99% pure. All other materials such as formaldehyde and powdered bentonite are of analytical grade.

2.2 Blend Preparation

A known weight of chitin and bentonite were dissolved in trichloro acetic acid separately. The chitin and bentonite solutions were mixed at various ratios with moderate agitation for 30 minutes. The blend films were prepared by casting the mixed solutions onto polystyrene plated and allowing the solvent to evaporate at room temperature. Similar experiments were performed in the presence of formaldehyde.

2.3 FTIR analysis

The films were dried at 60 ˚C overnight before measurement. Chemical structure of pure and the blended films were investigated by FTIR measurement, FTIR measurements were performed using KBr pelleted samples with a Perkins Elmer 200 FTIR spectrophotometer with a resolution of 4cm-1 in the range of 400 – 4000 cm-1.

2.4 X-ray diffraction

X-ray diffractometer was used to characterize the crystallinity of pure and the blend films. X-ray diffraction (XRD) patterns were recorded by reflection method with nickel-filtered

Cu Kα radiation using a Rigaku X-ray diffractometer operated at 40kV and 30mA in the 2θ scanning mode from 5° to 80°.

2.5 Thermal gravimetric analysis
The thermo gravimetric analysis of the bentonite/chitin blends without and with cross linking agents such as formaldehyde was carried out on TGA Q500 V20.10 Build 36 instrument. In this technique the mass of the substance and thermal decomposition of polymer blend are measured as a function of temperature.

2.6 Differential scanning calorimetry
The glass transition temperature (Tg) and melting temperature (Tm) of these blend were carried out with the NETZSCH DSC 200 PC in a pan Al, pierced lid in the nitrogen atmosphere at a heating rate of 10 degree Kelvin per minute.

3. Results and Discussions
3.1 FTIR
As shown in Figure 1a, the spectrum of pure chitin film shows a broad band at 3434 cm-1 which is due to the OH stretching. The band at 1561 cm-1 is assigned for the NH2 bending (amide II) while the small peak at 1654 cm-1 is attributed to the C=O stretching (amide I) O=C-NHCH3. The bands at 2926, 1414, 1317 and 1262 cm-1 are assigned to CH2 bending due to pyranose ring. The band at 1378 cm-1 is due to CH3 wagging. The characteristic features of chitin spectrum in this study are similar to that of previous reports. Figure 1b presents the FTIR spectrum of chitin/bentonite (1:1) blend. The peak at 3434 cm-1 corresponding to OH group of chitin is significantly shifted to lower wave number at 3402 cm-1 in the chitin/bentonite blend, which indicates that both the chitin and bentonite have good interaction through intermolecular hydrogen bonding. Two peaks in the range of 3650-3400 cm-1 were observed due to OH group of bentonite and OH group of chitin. As can be seen, the presence of bentonite in the chitin caused remarkable shift for the C=O stretching peak at 1654 cm-1 of chitin to a higher wave number at 1658 cm-1. In addition, the bands at 2926 and 1317 cm-1 of chitin disappeared in the spectrum of chitin/bentonite blend. These observations indicate the existence of good miscibility between chitin and bentonite. Figure 1c, represents the IR spectrum of chitin blend with cross linking agent formaldehyde.

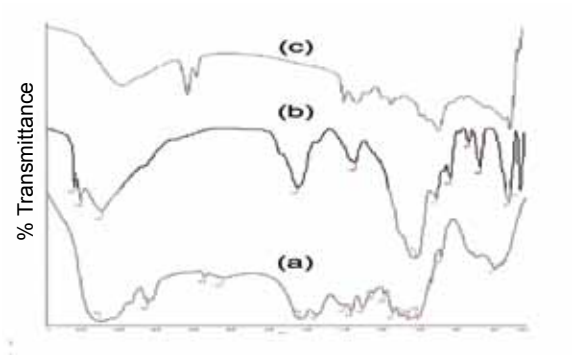

Figure 1: FTIR spectra of (a) pure chitin, (b) CT/BE (1:1) blend, (c) CT/BE (1:1) blend with cross linking agent (Formaldehyde)

3.2. X-ray analysis
The XRD spectrum of chitin shown in Figure 2a, It has high crystallinity and the characteristic peak at 2θ=10° and 20° are assigned to crystal forms I and II. While small amount of bentonite (copolymer) was added into the chitin matrix (Figure 2b), two diffraction peaks shifted to lower and intensity of peaks decreased compared with pure chitin, indicating strong interaction between chitin and bentonite composites. Hence crystallanity of the blended polymer had decreased much. Figure 2c, represents the XRD spectra of chitin/bentonite(1:1) blend with cross linking agent (formaldehyde) .

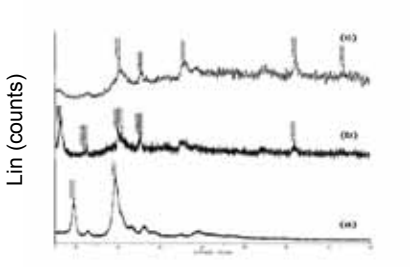

Figure 2: XRD spectra of (a) pure chitin, (b) CT/BE (1:1) blend, (c) CT/BE (1:1) blend with cross linking agent (Formaldehyde)

3.3 Thermo gravimetric analysis
The TGA thermograms of chitin, CT/BE (1:2) and CT/BE (1:1) with formaldehyde polymer blend are presented in Figures 3a – 3c. Chitin has two degradation temperatures which the temperature lower than 100 °C indicating the loss of water. The temperature above 100 ° C indicates the decomposition of pyranose ring structure. The TG of blended polymer shows five decomposition temperatures. Maximum decomposition was observed around from 350 °C, which may be due to the decomposition of side chains.

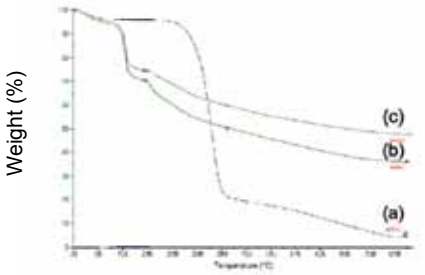

Figure 3: TGA Spectra of (a) pure chitin, (b) CT/BE (1:1) blend, (c) CT/BE (1:1) blend with cross linking agent (Formaldehyde)

3.4 DSC analysis
The Figure 4a shows the DSC curves of pure chitin and Figure 4b is chitin/bentonite (1:1) polymer blend. Broad endothermic peaks are observed at various temperatures indicating the crystallization of the polymer blend as well as evaporation of water and decomposition of side chain. The glass transition temperature of pure chitin is 75.1 °C, whereas the glass transition temperature of CT/BE blend (1:1) is 82.7 ° C. On comparing the DSC curves of chitin, CT/BE (1:1) has higher glass transition temperature. Hence, it was found that the endothermic peaks and the glass transition temperatures of chitin/bentonite blend shifted to higher values. It confirms that the blended polymer has higher thermal stability than pure chitin with the formation of different crystalline forms.

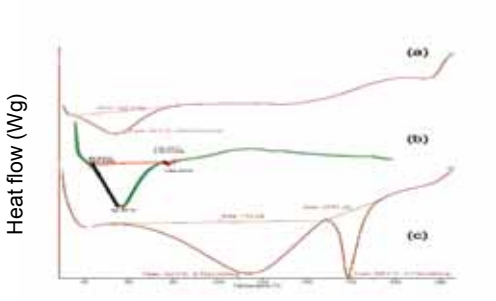

Figure 4: DSC spectra of (a) pure chitin, (b) CT/BE (1:1) blend, (c) CT/BE (1:1) blend with cross linking agent (Formaldehyde)

3.5. Tannery effluent treatment

The physico – chemical parameters and heavy metals content of the tannery industry effluent collected from an industry from Ambur industrial area. All the parameters along with the heavy metal contents were found to be high than the accepted limits.

Table 1: Physico-chemical factors and Heavy metals of tannery effluent collected from AMBUR INDUSTRIAL area

Parameters	Accepted limits	Tannery Effluent
pH	7 – 8.5@	9.4
Electrical Conductivity dSm^{-1}	7.5 – 20.0@	12.49
BOD mg/L	100@	1250
COD mg/L	250@	4210
TDS mg/L	850 – 1500@	8140
Alkalinity mg/L	250@	798
Chloride mg/L	200 – 600@	1200
Total Hardness mg/L	500@	3610
Sodium mg/L	1000 – 1500@	2140
Potassium mg/L	20#	840
Ammonia mg/L	10	140
Nitrite mg/L	0.1	8.41
Nitrate mg/L	0.1	105
Chromium mg/L	2.0#	1200
Copper mg/L	3.0#	141

Values expressed as mean of 6 individual values

Table 2: EFFECT OF ADSORBENT DOSE (CHITIN/BENTONITE 1:1 - FORMALDEHYDE) ON THE PHYSICO-CHEMICAL FACTORS AND HEAVY METALS OF TANNERY EFFLUENT

Various dosages of the prepared chitin–bentonite blend cross linked with formaldehyde have been used to treat tannery effluent. Table 2 explains the parameters such as COD, TS, BOD, and also heavy metals such as copper, chromium, have been decreased drastically with the increase in the adsorbent dosage. The purification of water has been found at minimum amount of sample required (5 g/L). Hence 5 g was found to be the optimum dosage treating the tannery effluent (Singh et al., 2009).

Parameters	Raw effluent	Adsorbent Dose				
		1g	2g	3g	4g	5g
Electrical Conductivity dSm^{-1}	12.49	5.12	3.33	2.11	1.91	1.09
BOD mg/L	1250	665	426	252	135	115
COD mg/L	4210	2450	1280	710	525	271
TDS mg/L	8140	1221	548	212	114	94
Alkalinity mg/L	798	425	124	77	45	41
Chloride mg/L	1200	745	421	288	175	115
Total Hardness mg/L	3610	1245	955	276	177	98
Calcium mg/lit	1010	577	433	275	166	101
Magnesium mg/L	2600	1123	750	424	220	125
Sodium mg/L	2140	997	455	249	162	118
Potassium mg/L	840	442	220	114	99	69
Total Nitrogen mg/L	258	155	85	59	48	45
Chromium mg/L	1200	640	225	135	77	45
Copper mg/L	141	79	31	11	7.7	7.1

Values expressed as mean of 6 individual values

Table 3: EFFECT OF CONTACT TIME OF ADSORBENT (cHITIN/ BENTONITE 1:1 - FORMALDEHYDE) WITH THE EFFLUENT ON THE PHYSICO-CHEMICAL FACTORS AND HEAVY METALS OF TANNERY EFFLUENT

The Table 3 shows the effect of time on the treatment of tannery effluent by the chitin –bentonite (1:1) with formaldehyde. On increasing the time the initial concentration reduces. After the treatment for 6 hrs all parameters reduced to the maximum extent. Hence 6 hrs was found to be an optimum treatment time.

Parameters	Raw Effluent	Contact Time in Hours					
		1 hr	2 hrs	3 hrs	4 hrs	5 hrs	6 hrs
Electrical Conductivity dSm-1	12.49	5.22	3.12	2.11	1.99	1.75	1.5
BOD mg/L	1250	630	444	325	218	118	111
COD mg/L	4210	2450	1650	1140	710	510	310
TDS mg/L	8140	2265	1145	750	364	170	116
Alkalinity mg/L	798	327	175	115	101	86	77
Chloride mg/L	1200	527	444	315	277	211	177
Total Hardness mg/L	3610	1221	720	458	276	115	99
Calcium mg/L	1010	764	423	215	110	94	71
Magnesium mg/L	2600	1450	650	315	218	115	75
Sodium mg/L	2140	1150	750	445	278	177	107
Potassium mg/L	840	485	312	218	117	105	78
Total Nitrogen mg/L	258	155	111	95	75	59	41
Chromium mg/L	1200	412	322	211	108	99	71
Copper mg/L	141	85	65	41	29	21	20

Table 4: EFFECT OF pH (cHITIN / BENTONITE 1:1 - FORMALDEHYDE) ON THE PHYSICO-CHEMICAL FACTORS AND HEAVY METALS OF TANNERY EFFLUENT

The effect of pH on tannery effluent treatment was studied. The reduction of all the parameters in the effluent by the blended polymer was found to be pH dependant. Table 4 showed that the there was maximum adsorption of all the parameters in the effluent by chitin –bentonite with formaldehyde at pH 5. Hence pH 5 was found to be the optimum pH for treating the tannery effluent.

Parameters	Raw Effluent	pH						
		3	4	5	6	7	8	9
Electrical Conductivity dSm-1	12.49	7.4	5.9	**2.6**	2.9	3.9	6.9	8.2
BOD mg/L	1250	729	355	**129**	265	325	475	585
COD mg/L	4210	2101	1100	**379**	740	1180	1320	790
TDS mg/L	8140	3915	1950	**760**	1650	1999	2411	2595
Alkalinity mg/L	798	313	129	**78**	195	315	375	495
Chloride mg/L	1200	575	310	**110**	325	610	640	680
Total Hardness mg/L	3610	1250	670	**312**	578	795	855	1110
Calcium mg/L	1010	595	345	**111**	495	610	775	895
Magnesium mg/L	2600	1220	775	**121**	692	895	974	1210
Sodium mg/lit	2140	950	440	**111**	565	744	985	1211
Potassium mg/L	840	420	275	**71**	150	225	310	375
Total Nitrogen mg/L	258	127	79	**51**	82	127	165	188
Chromium mg/L	1200	511	311	**145**	263	345	445	485
Copper mg/L	141	69	55	**42**	53	79	89	125

Values expressed as mean of 6 individual values

3.6. Adsorption kinetics

The rate constants for adsorption of reactive orange dye onto chitin polymer blend were treated with Lagergren first order model (Lagergren et al., 1998) which is generally expressed as

$dq_t/dt = k1(qe - q)$ -------- (1)

Where, k1 is the first-order-rate constant. Integrating Eq. (1) with respect to integration conditions

$q = 0$ to $q = q_t$ at $t = 0$ to $t = t$, the kinetic rate expression becomes

$\log (q_e - q_t) = \log q_e - k1_t/2.303$ ---------- (2)

The first-order-rate constant k1, can be obtained from the slope of the plot log (qe – qt) vs time.

The coefficients of correlation for the first-order-kinetic model were not high for all adsorbents and concentrations. Also, the estimated values of qe calculated from the equation differed from the experimental values (Table 3), which shows that the model is not appropriate to describe the adsorption process.

Adsorption kinetics were explained by the pseudo-second-order model developed by (Ho and Mckay, 2000)

$t/q_t = 1/k_2q_e2 + t/q_e$ ----------(3)

where k2 is the second-order-rate constant (g mg−1 min−1). The values of k2 at different initial dye concentrations for all adsorbents were calculated from the slopes of the respective linear plots of t/qt vs t (Figure 6). The correlation coefficients were 0.997–0.999, suggest a strong relationship between the parameters and also explain that the process follows pseudo-second-order kinetics.

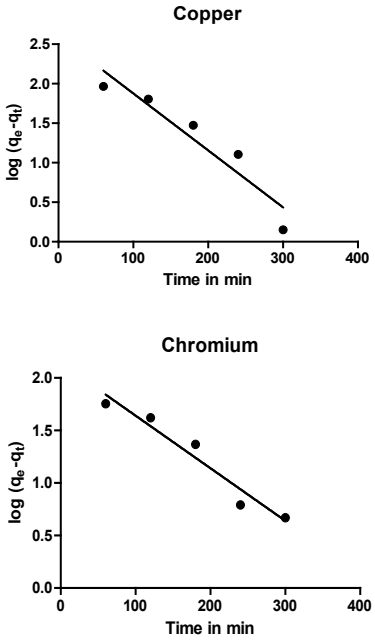

Figure 5: Pseudo-first-order kinetics of chromium and copper

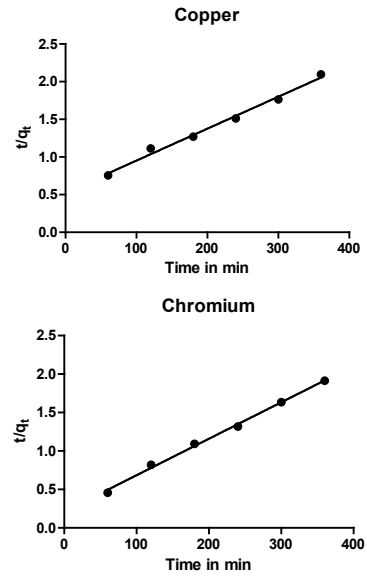

Figure 6: Pseudo-second-order kinetics of chromium and copper

Table: 5 Comparison between Lagergren's pseudo-first-order and Pseudo-second- order- kinetic models for chromium and copper

Metal ion	Pseudo-first-order kinetic model			Experimental value	Pseudo-second-order kinetic model		
	qe (mg/g)	k1 (min-1)	R2	qe (mg/g)	qe (mg/g)	k2 (g mg-1 min-1)	R2
Cr(VI)	427.86	0.005004	0.9438	188.17	44.46	0.004739	0.9966
Cu(II)	360.43	0.007208	0.8979	171.63	124.59	0.004238	0.9899

3.7. Sorption isotherm

3.7.1. Langmuir model

Langmuir theory (Langmuir.1918) was based on the assumption that adsorption was a type of chemical combination or process and the adsorbed layer was unimolecular. The theory can be represented by the following linear form:

$C_e/q_e = 1/Q^0b + Ce/Q^0$ ---------- (4)

where C_e is the equilibrium concentration (mg L−1), qe is the amount adsorbed at equilibrium (mg g-1) and Q^0 (mg g−1) and b (L mg−1) are Langmuir constants (Aksu, 2001) related to adsorption capacity and energy of adsorption respectively. The linear plots of C_e/q_e vs. C_e show that the adsorption obeys Langmuir isotherm model for all adsorbents (Figure 7). The values of Q^0 and b were determined for all adsorbents from intercept and slopes of the linear plots of C_e/q_e vs C_e (Table 6). The good fit of the experimental data and the correlation coefficients (R^2) higher than 0.94 indicated the applicability of the Langmuir isotherm model.

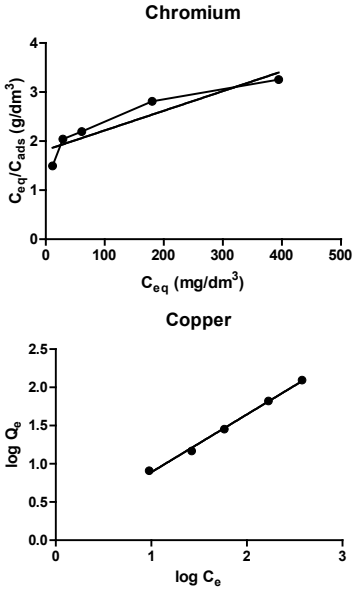

Figure 7: Langmuir adsorption model of chromium and copper

3.7.2. Freundlich model

The Freundlich adsorption model (Freundlich, 1907) stipulates that the ratio of solute adsorbed to the solute concentration is a function of the solution. The empirical model was shown to be consistent with an exponential distribution of active centers, characteristic of heterogeneous surfaces. The amount of solute adsorbed, qe, is related to the equilibrium concentration of solute in solution, Ce, following:

$Qe = K_FC_e^{1/n}$ ---------- (6)

This expression can be linearized to give the following equation:

$\log q_e = \log K_F + 1/n \log C_e$ -------- (7)

where K_F is a constant for the system, related to the bonding

energy. K_F can be defined as the adsorption or distribution coefficient and respects the quantity of dye adsorbed onto carbon adsorbents for a unit equilibrium concentration (a measure of adsorption capacity, mg g^{-1}). The slope 1/n, ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for 1/n below one indicates a normal Freundlich isotherm while 1/n above one is an indicative of cooperative adsorption. A plot of log (q_e) vs log (C_e) is shown in Figure 8, where the values of K_F and 1/n are determined from the intercept and slope of the linear regressions (Table 8).

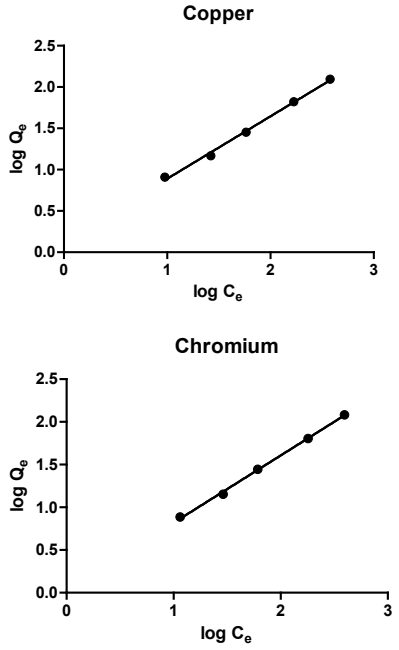

Figure 8: Freundlich isotherm model of chromium and copper

Table 7: Adsorption isotherm constant, Cmax and correlation coefficients

Metal ions	Langmuir constants				Freundlich constants		
	Q0 (dm^3/g)	b (dm^3/ mg)	Cmax (mg/g)	R^2	K_F	n	R^2
Cr(VI)	1.818	0.004007	453.71	0.8616	1.0730	1.2687	0.9985
Cu(II)	1.574	0.004242	371.05	0.8331	1.3611	1.3233	0.9982

Conclusion

In this work, chitin polymer blend have been used successfully as an adsorbing agent for the removal of Cr (VI) and Cu (II) from tannery effluent waste water. Adsorption was influenced by various parameters such as initial pH, initial dye concentration and dose of adsorbent. The maximum adsorption occurred at the optimum pH of 5.0. The Langmuir and Freundlich adsorption isotherm models were used for the description of the adsorption equilibrium. The data were in good agreement with Freundlich isotherms. It was shown that the adsorption of metals by chitin was best fitted by pseudo second order model. Since chitin polymer blend is used in this study, locally available, the adsorption process is expected to be economically viable for wastewater treatment.

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Printed at Unique Offset, Novatsing Rupam Estate, Opp. Abhay Estate, Tavdipura, Shahibaug, Ahmedabad