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RESEARCH ARTICLE

Heavy metal uptake by agro based waste materials

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Abbreviations:
FTIR: fourier transform infrared
M: molarity
Pb^{+2}: lead cation
rpm: rotations per minute

Presence of heavy metals in the aquatic systems has become a serious problem. As a result, there has been a great deal of attention given to new technologies for removal of heavy metal ions from contaminated waters. Biosorption is one such emerging technology which utilized naturally occurring waste materials to sequester heavy metals from industrial wastewater. The aim of the present study was to utilize the locally available agricultural waste materials for heavy metal removal from industrial wastewater. The wastewater containing lead and hexavalent chromium was treated with biomass prepared from ficus religiosa leaves. It was fund that a time of one hr was sufficient for sorption to attain equilibrium. The equilibrium sorption capacity after one hr was 16.95 ± 0.75 mg g^{-1} and 5.66 ± 0.43 mg g^{-1} for lead and chromium respectively. The optimum pH was 4 for lead and 1 for chromium. Temperature has strong influence on biosorption process. The removal of lead decreased with increase in temperature. On the other hand chromium removal increased with increase in temperature up to 40ºC and then started decreasing. Ion exchange was the major removal mechanism along with physical sorption and precipitation. The biosorption data was well fitted to Langmuir adsorption model. The kinetics of biosorption process was well described by the pseudo 2nd order kinetics model. It was concluded that adsorbent prepared from ficus religiosa leaves can be utilized for the treatment of heavy metals in wastewater.

The application of biosorption in environmental treatment has become a significant research area in the past ten years. Heavy metal ions are reported as priority pollutants, due to their mobility in natural water ecosystems and due to their toxicity (Volesky and Holan, 1995). The discharge of heavy metals into surface waters has become a matter of concern in Pakistan over the last two decades. These contaminants are introduced into surface waters through various industrial operations. The pollutants of concern include

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Table 1. Langmuir adsorption parameters for lead and chromium biosorption.

Metal	q_{max} (mg g^{-1})	b (l mg^{-1})	R^2
chromium	26.25	0.012	0.996
lead	37.45	0.022	0.972

lead, chromium, zinc, and copper. Heavy metals such as zinc, lead and chromium have number of applications in basic engineering works, paper and pulp industries, leather tanning, petrochemicals fertilizers, etc. The hexavalent and trivalent chromium is often present in electroplating wastewater (Kratochvil et al. 1998). Other sources of chromium pollution are leather tanning, textile, metal processing, paint and pigments, dyeing and steel fabrication. Lead is used as industrial raw material in the manufacture of storage batteries, pigments, leaded glass, fuels, photographic materials, matches and explosives (Raji and Anirudhan, 1997).

Lead and chromium are toxic metal contaminants in water. According to Pakistan standards the maximum discharge limits for lead and chromium in wastewater are respectively 0.5 mg l^{-1} and 1.0 mg l^{-1}. Maximum limit in drinking water is 0.05 mg l^{-1} for both metals. In fact there is no safe level of these metals in drinking water and even a very dilute content can cause adverse health effects. Lead is toxic to living organisms and if released into the environment can bio accumulate and enter the food chain. Lead is known to cause mental retardation, reduces haemoglobin production necessary for oxygen transport and it interferes with normal cellular metabolism. Lead has damaging effects on body nervous system. It reduces I.Q level in children. Strong exposure of hexavalent chromium causes cancer in the digestive tract and lungs and may cause gastric pain, nausea, vomiting, severe diarrhoea, and haemorrhage (Mohanty et al. 2005).

The conventional methods for treatment of lead and chromium wastes include: lime and soda ash precipitation, adsorption with activated carbon, ion exchange, oxidation and reduction, fixation or cementation. These methods are economically unfavourable or technically complicated, and are used only in special cases of wastewater treatment (Kratochvil et al. 1998; Sharma, 2003).

Table 2. Values of constant separation factor for lead and chromium biosorption.

Initial concentration C_0, mg l^{-1}	(chromium) $R_L = 1/(1 + bCo)$	(lead) $R_L = 1/(1 + bCo)$
10	0.895	0.819
25	0.774	0.645
50	0.631	0.476
100	0.461	0.312
250	0.255	0.154
500	0.146	0.083
1000	0.079	0.043

Biosorption of heavy metals from aqueous solutions is a relatively new technology for the treatment of industrial wastewater. Adsorbent materials derived from low cost agricultural wastes can be used for the effective removal and recovery of heavy metal ions from wastewater streams. The major advantages of biosorption technology are its effectiveness in reducing the concentration of heavy metal ions to very low levels and the use of inexpensive biosorbent materials (Holan and Volesky, 1994; Kratochvil and Volesky, 1998).

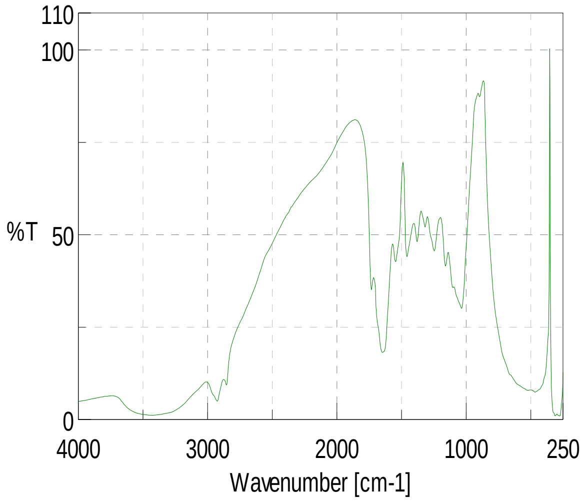

Figure 1. FTIR spectra of acid treated ficus religiosa leaves.

Removing metals from wastewater requires development of new sorbents. A wide range of commercial sorbents including chelating resins and activated carbon are available for metal sorption, but they are relatively expensive. In recent years, numerous low cost natural materials have been proposed as potential biosorbents. These include moss peat, algae, leaf mould, sea weeds, coconut husk, sago waste, peanut hull, hazelnut, bagasse, rice hull, sugar beet pulp, plants biomass and bituminous coal. (Lee and Volesky, 1997; Singh and Rawat, 1997; Gupta et al. 1998; Quek et al. 1998; Brown et al. 2000; Chong and Volesky, 2000; Dakiky et al. 2002; Johnson et al. 2002; Reddad et al. 2002; Babel and Kurniawan, 2003; Pagnanelli et al. 2003; Sekhar et al. 2003). In this research adsorbent prepared from ficus religiosa leaves was used for treatment of lead and chromium wastes. Effect of operating conditions like temperature, pH and initial metal concentration, on lead and chromium biosorption were investigated.

Composition of leaves

Leaves of different trees are very versatile natured chemical species as these contain a variety of organic and inorganic compounds. Cellulose, hemicellulose, pectins and lignin present in the cell wall are the most important sorption sites (Volesky, 2003). Leaves have chlorophyll, carotene, anthocyanin and tannin which contribute to metal biosorption. The important feature of these compounds is that they contain hydroxyl, carboxylic, carbonyl, amino and nitro groups which are important sites for metal sorption (Volesky, 2003). Fourier transform infrared (FTIR) spectra of ficus religiosa leaves also indicated the presence of these functional groups (Figure 2).

Cr(VI) is present in solution as CrO_4^{-2} and $Cr_2O_7^{-2}$ at normal pH values but when pH values are reduced below 3 then Chromium exists in the form of $HCrO_4^-$ (Cimino et al. 2000; Demirbas et al. 2004; Park et al. 2006b). When adsorbent developed from ficus religiosa leaves is intimately mixed with chromium solution at low pH values then OH^- group present in biomass are replaced by chromate ions in the solution. At pH values close to five the adsorbent surfaces are negatively charged due to release of H^+ ions, therefore these attract lead cation (Pb^{+2}). Leaves also have Ca, Mg, Na ions. These are present in the structure of complex organic compounds in leaves and exchange with Pb^{+2} cations during sorption process (Kratochvil et al. 1998). Leaves have considerable amounts of CaO, MgO, Na_2O, K_2O etc. When leaf powder is mixed in water these oxides are converted into hydroxides. These hydroxides precipitate the metal cations (Schneider et al. 2001).

Table 3. Kinetics parameters for lead and chromium biosorption.

Metal	k (g $mg^{-1}min^{-1}$)	q_e (mg g^{-1})	R^2
chromium	0.0436	5.86	0.998
lead	0.0163	17.54	0.997

MATERIALS AND METHODS

Chemicals and instruments

All chemicals used were of analytical reagent grade. Lead solution of 1000 mg l^{-1}concentration was prepared by dissolving 1.6 g lead nitrate in one litre distilled water. Hexavalent chromium solution of 1000 mg l^{-1} concentration was prepared by dissolving 2.827 g potassium dichromate in one litre distilled water. These solutions were further diluted to get solutions of various known concentrations of lead and chromium. A variable speed shaker 20-500 rotations per minute (rpm) was used for batch experimentation. It has the capacity of holding eight Erlenmeyer flasks simultaneously. It was equipped with heating water bath. A vacuum filter assembly having Pyrex filter funnel of porosity grade 4 was used for separating adsorbent from solution. The unknown quantities of lead and chromium were determined by Shimadzu 6800 atomic absorption spectrophotometer using an air-acetylene flame. The adsorbent was analyzed by JASCO FTIR 4000 spectrometer.

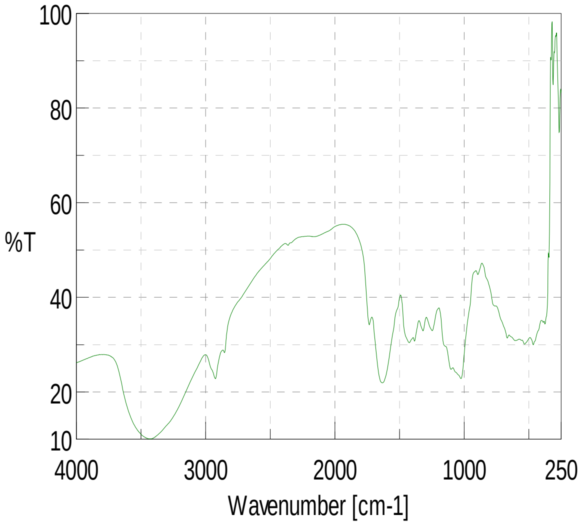

Figure 2. FTIR spectra of un-treated ficus religiosa leaves.

Biomass preparation

Ficus religiosa leaves were collected from local environment of University of Engineering and Technology Lahore, Pakistan. These leaves were washed with tap water and dried in shadow. Dried leaves were ground and sieved to 50 mesh sizes. This powder was soaked in 0.1 molarity (M) HNO_3 for 24 hrs (50 g leaves powder was soaked per

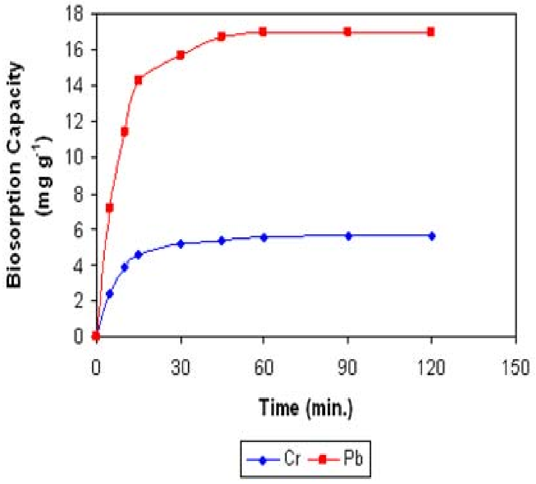

Figure 3. Equilibrium time for lead and chromium biosorption.

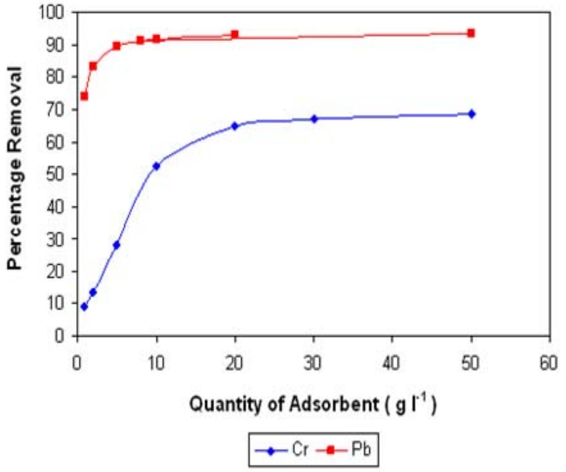

Figure 4. Effect of adsorbent dose on percentage removal of lead and chromium.

litre). It was filtered and washed with distilled water to remove acid contents. The washing was continued till the pH of the filtrate became near neutral. It was first dried at room temperature and then in an oven at 105ºC to remove moisture. This biomass was stored in air tight glass bottles to protect it from humidity.

Fourier transform infrared analysis

FTIR spectroscopy was used to identify the chemical groups present in leaves. The samples were examined using JASCO FTIR 4000 spectrometer within range 400-4000 cm^{-1}. KBr was used as background material in all the analysis. 0.0035 g leaves powder was mixed with 0.5 g KBr and pressed to form a pellet. FTIR spectra of leaves indicated the presence of hydroxyl, carboxyl, carbonyl, amino and nitro groups which are important sorption sites. There were also indications of presence of heavy metals and inorganic compounds below 870 cm^{-1}. These metals were removed by treatment of leaves with nitric acid to improve the sorption capacity. FTIR spectra of acid treated ficus religiosa leaves and untreated ficus religiosa leaves are shown in Figure 1 and Figure 2 respectively.

RESULTS AND DISCUSSION

Determination of equilibrium time

Batch experiments were carried out to find the equilibrium time for sorption of chromium and lead on ficus religiosa leaves. All experiments were performed three times and average values were used in all calculations. 1.0 g ficus religiosa leaves powder of 50 mesh sizes was mixed in 100 ml solutions of lead and chromium. The initial concentration of each solution was 100 mg l^{-1}. It was shaken at 200 rpm and samples were collected at different time intervals. The pH for the experiment was taken as original pH of solutions which was 5.2 for chromium and 5.8 for lead. After completion of each batch of experiments the solution was filtered using vacuum filter assembly. Filtrate was analyzed using atomic absorption spectrophotometer to determine the amount of metal left after sorption. The amount of metal sorbed was calculated by material balance. Sorption capacity q was determined using the formula:

$$q = \frac{V\,(C_0 - C_f)}{m}$$

Where C_0 and C_f are the initial and final concentrations of metal in solution, V is the volume of solution and m is the mass of adsorbent. As shown in Figure 3 about 80% removal was attained in first 15 min and concentration became almost constant after 45 min. The fast initial uptake was due to the accumulation of metal ions on surface of adsorbent which is a rapid step. More time was consumed on diffusion of ions to binding sites. It was concluded that one hr was sufficient for sorption to attain equilibrium. The equilibrium capacity obtained after one hr of sorption was 5.66 ± 0.43 mg g^{-1} and 16.95 ± 0.75 mg g^{-1} for chromium and lead respectively.

Effect of adsorbent dose

Keeping all other parameters constant adsorbent dose was varied from 1.0 to 50 gm l^{-1}. It can be seen from Figure 4 that an adsorbent dose of 10 gm l^{-1} is sufficient for optimal removal of both metals. Increasing the dose further did not affect the percentage removal. The removal capacity was low at high dose rate and vice versa. This was due to metal concentration shortage in solution at high dose rates.

Effect of pH

Keeping the same operating conditions as mentioned previously, pH of solution was varied from 0.5 to 8 by the

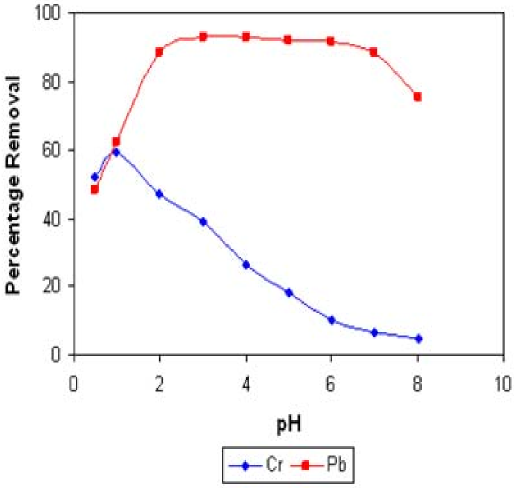

Figure 5. Effect of pH on percentage removal of lead and chromium.

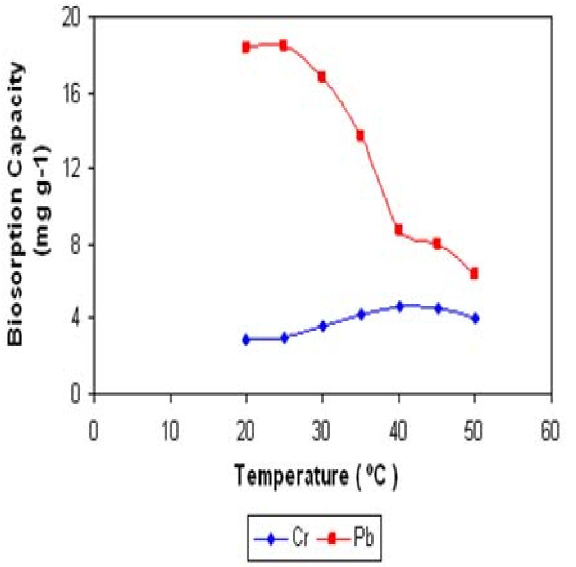

Figure 6. Effect of temperature on biosorption capacity of lead and chromium

addition of 0.1 M nitric acid and 0.1 M ammonia solution. It was found that sorption of chromium was more at low pH where as lead has almost same sorption in the pH range of 3 to 5 (Figure 5). The optimal pH for lead and chromium was 4 and 1 respectively. At pH higher than 6 both metals were precipitated due to formation of hydroxides and removal due to sorption was very low. At low pH the concentration of proton was high and metal binding sites became positively charged repelling the Pb^{+2} cations and attracting the anions like $HCrO^{-}_{4}$ and CrO_4^{-2}. Thus at low pH removal of lead was low and removal of hexavalent chromium was high. Also more chromium removal at low pH was due to the reduction of Cr(VI) to Cr(III) (Park et al. 2006a), which was then precipitated at the biomass surface by forming $Cr(OH)_3$. In this reduction H^+ was consumed and increase in pH was observed at the end of experiments. The reduction of Cr(VI) to Cr(III) was confirmed by analyzing the solution for Cr(VI) using 1,5 diphenylcarbazide in spectrophotometer. At pH close to 5 the binding sites became negatively charged due to presence of hydroxyl, carboxylic and amino groups. So at this pH Pb^{+2} was attracted by the adsorbent. A decrease in pH was observed at the end of experiments in case of lead sorption. This was due to the release of proton as result of ion exchange between Pb^{+2} and H^+ ions. In case of hexavalent chromium there was an increase in pH which was due to exchange of $HCrO_4^-$ with OH^-.

Effect of temperature

Keeping all other parameters constant temperature was varied from 20ºC to 50ºC. The sorption of chromium increased slightly with the increase in temperature up to 40ºC and then started decreasing, where as uptake of lead decreased considerably with increase in temperature from 30ºC to 50ºC (Figure 6). The temperature higher than 40ºC caused a change in the texture of the biomass and thus reduced its sorption capacity.

Biomass contains more than one type of sites for metal binding. The effect of temperature on each site is different and contributes to overall metal uptake. The effect of temperature on biosorption also depends on the heat of sorption. Usually for physical sorption heat of sorption is negative; sorption reaction is exothermic and preferred at lower temperature. For chemisorption the overall heat of sorption is combination of heat of various reactions taking place at sorption sites. It depends on type of metal and adsorbent. That is the reason for having different behaviour of lead and chromium uptake with temperature.

Effect of initial metal concentration

Initial concentrations of both metals were varied from 10 to 1000 mg l^{-1} and quantity of adsorbent was kept constant at 10 gm l^{-1}. It was observed that removal capacity decreased with decrease in metal concentration.

Langmuir adsorption model was applied to data:

$$q_e = q_{max} \frac{b\,C_e}{1 + bC_e}$$

This equation was rearranged to get:

$$\frac{C_e}{q_e} = \frac{1}{q_{max} b} + \frac{C_e}{q_{max}}$$

Where q_e is the amount of metal sorbed per unit weight of biomass at equilibrium, C_e is the residual equilibrium metal

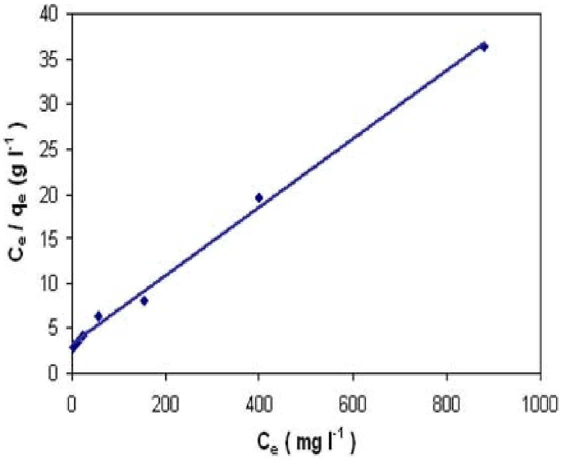

Figure 7. Langmuir plot for hexavalent chromium biosorption.

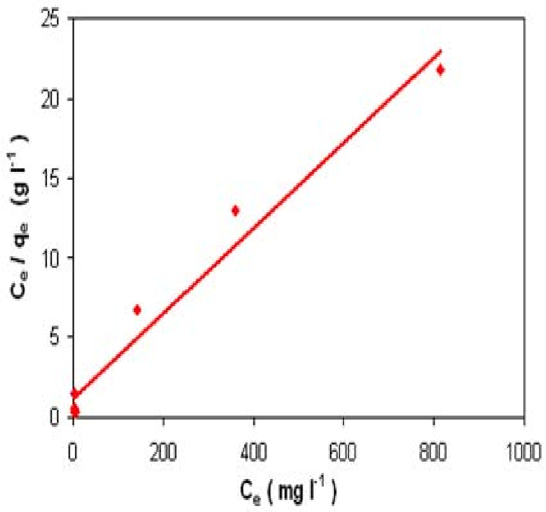

Figure 8. Langmuir plot for lead biosorption.

concentration left in solution after binding, q_{max} is the maximum possible amount of metal ion adsorbed per unit weight of biomass and b is the equilibrium constant related to the affinity of the binding sites for the metals, lower is b more is the affinity of metal to biomass.

Equilibrium concentration C_e and equilibrium capacity q_e were calculated for each initial metal concentration. C_e was plotted against C_e/q_e and a straight line was fitted in the data. Correlation coefficient of 0.996 for chromium and 0.972 for lead indicate that sorption followed Langmuir model (Figure 7 and Figure 8). Values of Langmuir constants q_{max} and b were calculated from slope and intercept of lines in Figure 7 and Figure 8 and are shown in Table 1.

Low values of parameter b indicate that ficus religiosa leaves have high affinity for chromium and lead. The values of equilibrium relation parameter, R_L were calculated for various initial concentrations for both metals. As shown in Table 2, R_L values lie between 0 and 1 which indicate favourable sorption isotherm for both metals.

Biosorption kinetics

In order to explain the kinetics of biosorption pseudo second order kinetics model was applied.

$$\frac{dq_t}{dt} = k(q_e - q_t)^2$$

This equation was integrated and arranged to get the following equation.

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t$$

Where k is the sorption coefficient, q_e is the equilibrium capacity and q_t is the sorption capacity at any time t.

Time, t was plotted against t/q_t and straight lines having correlation coefficient of 0.99 were fitted in the data for both metals. The sorption coefficient k and equilibrium capacity q_e were calculated from the slope and intercept of lines in Figure 9 and are shown in Table 3.

The values obtained for equilibrium capacity for both metals were very close to those obtained experimentally after one hr of sorption. It confirmed that one hr was sufficient for sorption to attain equilibrium and sorption followed pseudo 2nd order kinetics

CONCLUDING REMARKS

Ficus religiosa leaves powder was found to be a very good adsorbent for hexavalent chromium and lead. It has good sorption capacity for both metals. The sorption capacity for hexavalent chromium was 5.66 ± 0.43 mg g^{-1} and for lead was 16.95 ± 0.75 mg g^{-1}. The sorption was pH dependent and optimal pH was 4 and 1 for lead and chromium respectively. Biosorption of metals was temperature dependent. Optimal temperature was 40ºC for chromim and 25ºC for lead. Main removal mechanism was ion exchange between protons and metal cations in case of lead and between metal anions and hydroxyl ions in case of hexavalent chromium. This fact is indicated by the change

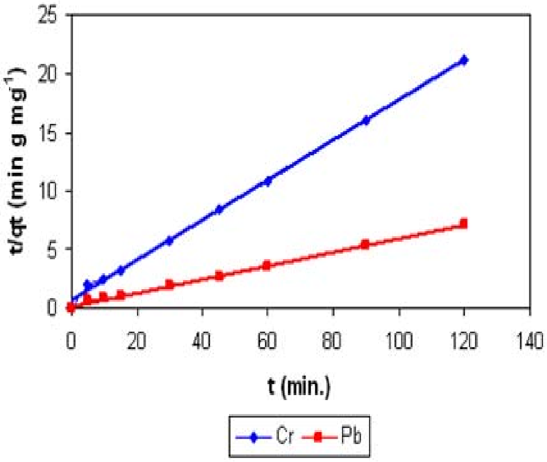

Figure 9. Kinetics of lead and chromium biosorption.

of pH at the end of biosorption process. Physical sorption and precipitation also contributed to removal of metals. The biosorption process followed Langmuir model for both metals which indicated that ion exchange took place in a monolayer at the surface of adsorbent. The kinetics of biosorption process was represented well by pseudo 2nd order kinetics model.

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