

ADSORPTION STUDIES ON THE REMOVAL OF HEXAVALENT CHROMIUM AND LEAD FROM CONTAMINATED WASTEWATER USING LEAVES OF WATER HYACINTH AS AN ADSORBENT

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ABSTRACT

Present of heavy metals in the aquatic system has become a serious problem. As a result, there has 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 wastewater containing hexavalent chromium and lead was treated with biomass prepared from water hyacinth leaves. It was found that a time of 2 hrs was sufficient for sorption to attain equilibrium. The equilibrium sorption capacity after 2 hrs was 18 mg/L and 15 mg/L for lead and chromium, respectively. The optimum pH was 5 for lead and 2 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 increasing in temperature up to 40⁰C and then started decreasing. Ion exchange was the major removal mechanism along with physical sorption and precipitation. Equilibrium data were analyzed by Langmuir and Freundlich isotherm models and the biosorption data were well fitted to Freundlich isotherm model. It was concluded that adsorbent prepared from water hyacinth leaves can be utilized for the treatment of lead and chromium in wastewater. The water hyacinth leaves can be possible recycled by washes with 0.5 N NaOH solution then with tap water several times to get rid of metals. These results introduce applicable technique for removal heavy metal ions from contaminated waters resulted from industrial activities and

Keywords: Biosorption, water hyacinth leaves, hexavalent chromium, lead , wastewater, removal.

INTRODUCTION

The release of toxic substances in the wastewater from industrial activities such as mining, metal processing, pharmaceuticals, pesticides, organic chemicals, rubber and plastics, etc. may cause serious effects on the environment and human health. It is because the toxic substances have a tendency to accumulate in the soil, sea water, fresh water and sediments due to their high dispersion from where they enter into the food chain *Rathnakumar et al., (2009)*. In order to have a pollution-free environment, the toxic heavy metals should be removed from wastewater before its disposal. The discharge of heavy metals into surface waters has become a matter of concern in Egypt over the last two decades. These contaminants are introduced into surface waters through various industrial operations. The pollutants of concern include 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 and Volesky, 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 the standards set by the Egyptian Ministry of Health for permits to discharge treated industrial liquid effluents into fresh water are 0.05 mg/L. Maximum limit in drinking water is 0.05 mg/L 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 hemoglobin production necessary for oxygen transport and it interferes with normal cellular metabolism. Lead has damaging effects on body nervous system. It reduces Intelligence Quotient (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 diarrhea, and haemorrhage (*Mohanty et al. 2005*).

The conventionally 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 unfavorable or technically complicated, and are used only in special cases of wastewater treatment (*kratochvil and Volesky, 1998 and Sharma, 2003*). Conventional methods for the removal of heavy metals from wastewater, however, are often cost prohibitive having inadequate efficiencies at low metal concentrations, particularly in the range of 1 – 100 mg/L (*Volesky, 2000*). For this reason, low cost adsorbents have been evaluated for the removal of heavy metals from aqueous solutions. Biosorption is a promising technique for the removal of heavy metals from aqueous environments especially when adsorbents are derived from lignocelluloses' materials (*Coelho et al., 2007*). The search for new technologies to remove toxic metals from wastewater has directed attention to biosorption, which is based on metal binding to various biological materials. Biosorption is a fast and reversible reaction of the heavy metals with biomass. *Laszlo and Dintzis (1994)* have shown that lignocelluloses have ion-exchange capacity and general sorptive characteristics, which are derived from their constituent polymers and structure. The polymers include extractives, cellulose, hemicelluloses, pectin, lignin and protein. Agricultural by-products vary greatly in their ability to remove metals from solution. The ability of biological materials to adsorb metal ions has received considerable attention for the development of an efficient, clean and cheap technology for wastewater treatment at metal concentrations as low as 1 mg/L (*Chong and Volesky, 1995*). In recent years, special attention has been focused on the use of natural sorbents as an alternative to replace the conventional adsorbents, based on both the environmental and the economical points of view *Bailey et al., (1999) and Babel and Kurniawan, (2003)*. Natural materials that are available in large quantities, or certain waste products from industrial

or agricultural wastes and operations, may have potential as inexpensive sorbents. Due to their low cost, when these materials the end of their lifetime, they can be disposed of without expensive regeneration. The abundance and availability of agricultural by-products make them good sources of raw materials for natural sorbents. In this research adsorbent prepared from water hyacinth leaves was used for treatment of lead and chromium wastes. Eggshell powder is a waste material and conveniently used for the treatment of industrial wastewater containing Cu and Pb metals. The most important finding of the study showed that in the mixture of metal ions the adsorption percentage is decreased. It clearly indicate that heavy metals need to be removed from the industrial waste before the discharge into the rivers as well as the present study showed that one more metal will decrease the adsorption efficiency of adsorbent (*Agarwal A., 2013*).

Chromium (VI) is present in solution as CrO_4^{-2} and $\text{Cr}_2\text{O}_7^{-2}$ at normal pH values but when pH values are reduced below 3 then chromium exists in the form of H CrO_4^- (*Cimino et al., 2000; Demirbas et al., 2004 ; Park et al., 2006b*). When adsorbent developed from water hyacinth leaves is 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+}). Water hyacinth also has Ca, Mg, Na ions. These are present in the structure of complex organic compounds in water hyacinth and exchange with Pb^{2+} cations during sorption process (*Kratochvil and Volesky, 1998*). Water hyacinth has 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*).

In general, an adsorbent can be assumed as "low-cost" if it requires a little bit processing, is abundant in nature, or is a by-product or a waste from an industry. Natural material or certain waste from industrial or agricultural operation is one of the resources for low cost adsorbents. Generally, these materials are locally and easily available in large quantities. Therefore, they are inexpensive and have little economic value (*Mohana and Pittman, 2007*). The objectives of the present study was to utilize the locally available agricultural waste materials for heavy metal removal from industrial wastewater and the present study are: (1) to study the effect of contact time of Cr(VI) and Pb(II) metal ions adsorption on water hyacinth leaves; (2) to investigate the influence of pH and temperature on the adsorption of Cr(VI) and Pb(II); (3) to find the effect of adsorbent dose and initial concentration on the adsorption of Cr(VI) and Pb(II) and (4) to describe the experimental data of adsorption isotherms through Langmuir and Freundlich models.

MATERIALS AND METHODS

Composition of water hyacinth leaves

Water hyacinth leaves is very versatile natured chemical species as these contain a variety of organic and inorganic compounds. Cellulose,

hemicelluloses, pectin and lignin percent in the cell wall are the most important sorption sites (Volesky, 2003). Water hyacinth has 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).

Adsorption process

Adsorption experiments were carried out in batch mode at ambient temperature. Adsorption is a process that occurs when gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (the adsorbate). Adsorption is operative in most physical, biological, and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins and water purification. Similar to surface tension, adsorption is a consequence of surface energy. Adsorption is usually described through isotherms, that is functions which connect the amount of adsorbate on the adsorbent, with its pressure (if gas) or concentration (if liquid).

Adsorbate Solutions

Synthetic wastewater used in this study was prepared to match the real wastewater samples used in the adsorption studies. Hexavalent chromium solution of 1000 mg/L concentration was prepared by dissolving 2.827 g potassium dichromate in one liter distilled water. Lead solution of 1000 mg/L concentration was prepared by dissolving 1.6 g lead nitrate in one liter distilled water. These solutions were further dilute to get solutions of various known concentrations of lead and chromium (100, 200, 300, 400 and 500 mg/L). The required pH (5 and 2 for Pb(II) and Cr (VI)) was adjusted by drop wise addition of 0.1 N H₂SO₄, depending on the acidity of the sample.

Batch Sorption Experiments

Batch experiments were carried out to find the equilibrium time for sorption of chromium and lead on water hyacinth leaves. All experiments were performed three times and average values were used in all calculation. All experiments were carried out at 25 °C by adding different amount of water hyacinth leaves powder of 50 mesh sizes (0.5, 1.5, 2.0, 3.0 and 5.0 g/L) to different concentrations of 250 ml of chromium and lead ions solution. The agitation rate for all experiments was 300 rpm and the residence time was (15, 30, 45, 60, 75, 90 and 120 minutes). The unknown quantities of lead and chromium after filtrate were determined by Inductively Coupled Plasma (ICP-OES) Perkin – Elmer Optima 7300, to determine the amount of metal left after sorption.

Calculation of cations uptake by water hyacinth leaves

The amount of metal sorbed was calculated by material balance. The amount of adsorption at equilibrium q_e (mg/g) was calculated by:

$$q_e = (C_0 - C_e) V / W \quad \dots\dots\dots(1)$$

Where C_0 and C_e (mg/L) are the liquid –phase concentration of chromium and lead at initial and equilibrium, respectively, $V(L)$ is the volume of the solution and W (g) is the mass of dry adsorbent used. The adsorption efficiency of Cr (VI) and Pb (II) can be calculated as:

$$\text{Adsorption percentage} = (C_0 - C_e) / C_e \times 100 \quad \dots\dots\dots(2)$$

Biomass Preparation

Water hyacinth leaves was soaked in 0.1 M HNO₃ for 24 hrs (50 g water hyacinth leaves were soaked per liter). It was filtered and washed with distilled water to remove acid contents. The washing was continued till the pH of the filtrate become near neutral. It was first dried at room temperature and then in an oven at 105 °C to remove moisture. This biomass was stored in air tight glass bottles to protect it from humidity, so the samples become ready to use (Kaiser et al., 2007). The water hyacinth leaves can be possible recycled by washes with 0.5 N NaOH solution then with tap water. Excess alkali was neutralized with 0.1 N HCL solution and again washed with tap water several times, to get rid of metals; this process will be useful for the removal of residual of metals from adsorbent after treatment to be used for several times (Nagamaik et al., 2002).

RESULTS AND DISUCTIONS

Effect of contact time

The effect of contact time on Cr (VI) and Pb (II) adsorption on water hyacinth leaves powder was investigated to study the rate of Cr (VI) and Pb (II) ions removal (figure 1). It is easily seen from figure (1) that the removal percentage of Cr (VI) and Pb (II) metal ions increased with increasing the contact time. The removal percentage 70% for Cr (VI) and 82% for Pb (II) after 120 min. It is clear that at the beginning % removal increased rapidly in few minutes by increasing contact time, then % removal increased lightly and slowly till reach maximum value and this can be explained on the basis that as initially a large number of vacant surface sites are available for adsorption of metal ions but with passage of time the surface sites become exhausted (Zhan et al., 2000). These results indicate that water hyacinth leaves powder has a very high capacity for adsorption of Cr(VI) and Pb(II) ions in solutions.

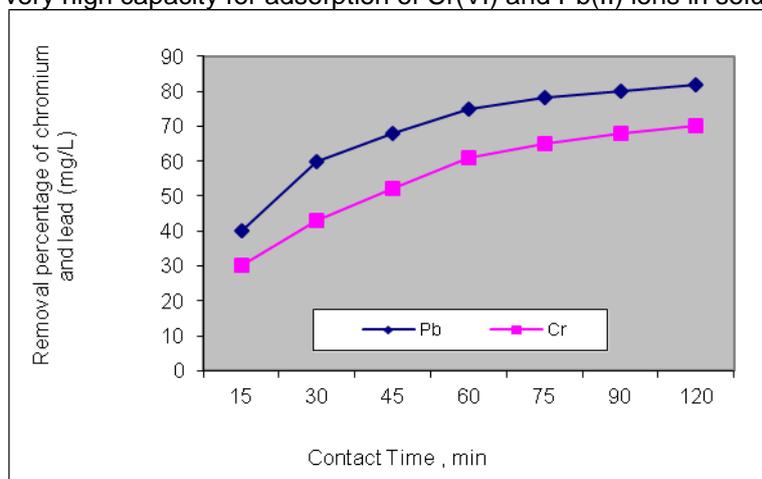
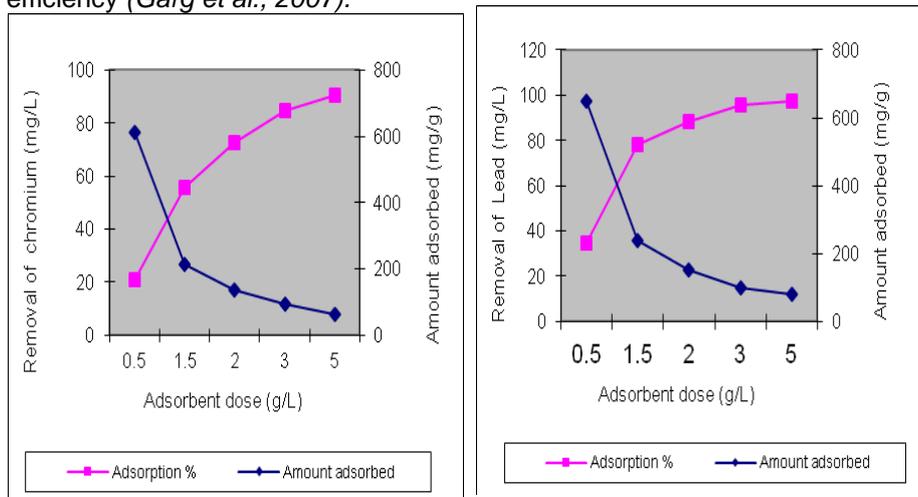


Figure (1) : Effect of contact time on chromium and lead ions removal

Effect of Adsorbent Dose

The percentage adsorption of Cr(VI) and Pb(II) ions onto water hyacinth leaves powder was studied at different adsorbent doses [0.5, 1.5, 2.0, 3.0 and 5 g/L], keeping pH 2 for Cr and 5 for Pb, respectively, temperature (25°C), contact time (120 minutes) and initial concentration for Cr and Pb are constant (500 mg/L). The adsorption capacity (mg/L) and percentage adsorption of chromium and lead at different doses of water hyacinth is shown in fig. (2 and 3). The degree percentage of adsorption increases as the adsorbent dose is increased. It may be concluded that by increasing the adsorbent dose, the removal efficiency of water hyacinth increases, while adsorption efficiency decreases with increase in adsorbent dose fig. (2 and 3). The decrease in adsorption efficiency may be due to the fact that some adsorption sites may remain unsaturated during the adsorption process whereas the number of sites available for adsorption site increase by increasing the adsorbent doses and that results in the increase of removal efficiency (Garg et al., 2007).

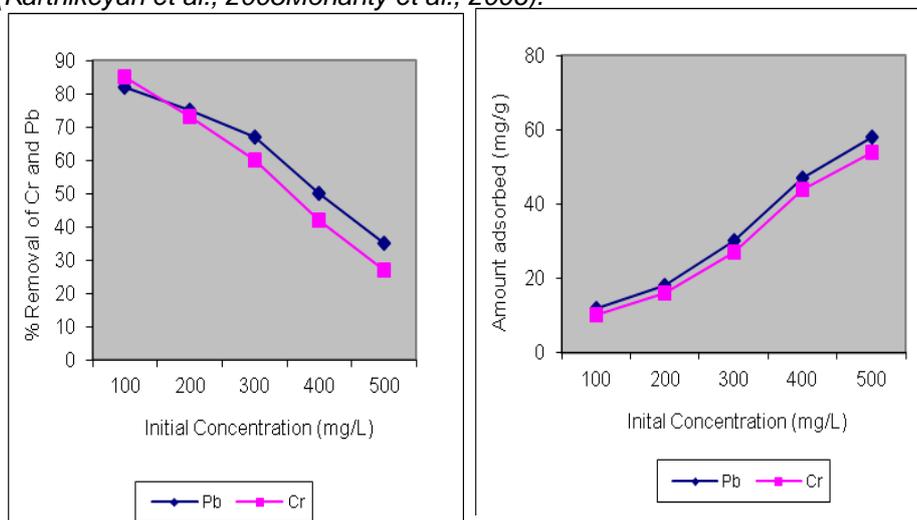


Figures (2 and 3) : The effect of variant water hyacinth adsorbent dosage on the removal and quantity adsorbed of and /or for 500 mg/L chromium and lead ions from wastewater at constant time

Effect of Initial Chromium (VI) and Lead (II) Ions Concentration

The initial heavy metal ions concentration is an important parameter in adsorption since a certain amount of adsorbent can adsorb a certain amount of heavy metal ions. The results represented in figure (4), show that the percentage of Cr(VI) and Pb(II) ions adsorption were decreased with increasing initial concentration. But the actual amount of Cr and Pb ions adsorbed per unit mass of the adsorbent was increased with increasing Cr and Pb ions concentration in the test solution as illustrated in figure (5). At

low concentration the ratio of surface active sites to the total metal ions in the solution is high and hence all metal ions interact with the adsorbent and are removed quickly from the solution. However, the amount of metal ions adsorbed per unit weight of adsorbent, q_e , is higher at high concentration. According to these results, the initial Cr and Pb ions concentration plays an important role in the adsorption capacities. Higher concentrations of metal ions were used to study the maximum adsorption capacity of adsorbent (Karthikeyan et al., 2005; Mohanty et al., 2006).

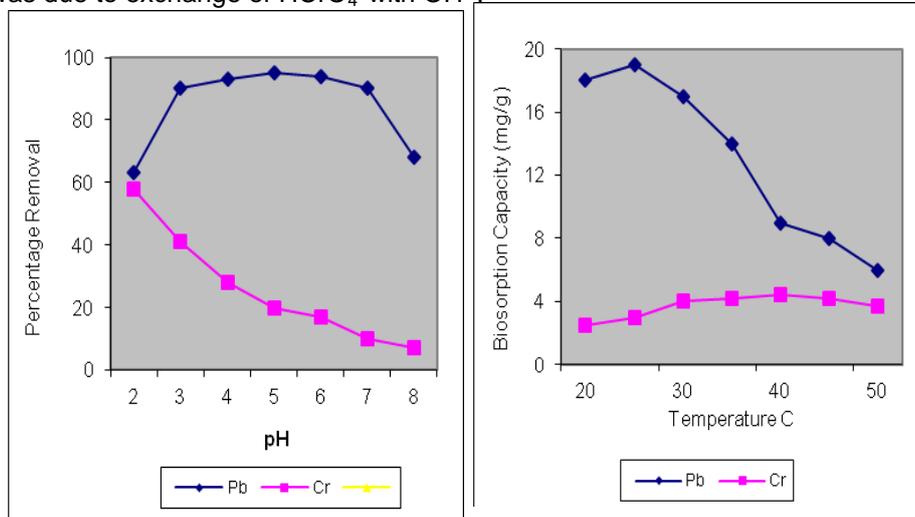


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Effect of pH on the uptake of chromium (VI) and lead (II).

Keeping the same operating condition as mentioned previously, pH of solution was varied from 2 to 8 by the 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, 6). The optimal pH for lead and chromium was 5 and 2 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 lead 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^{-}



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Effect of Temperature

Keeping all other parameters constant temperature was varied from 25^oC to 40^oC. The sorption of chromium increased slightly with the increase in temperature up to 40^oC and then started decreasing, whereas uptake from 25^oC to 40^oC (figure, 7). The temperature higher than 40^oC caused a change in the tissue 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 chemisorptions 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 behavior of chromium and lead uptake with temperature.

Analysis of Adsorption Isotherms

The equilibrium study is important for an adsorption process as it shows the capacity of the adsorbent and the adsorption isotherm is normally applied to describe the adsorption mechanism for the interaction of cations on the adsorbent surface. In the present study, experimental data were analyzed to examine the adsorption isotherm using the Langmuir and Freundlich

models. The Langmuir isotherm is applicable to monolayer adsorbate coverage on the adsorbent surface *Abdulrasaq and Basiru (2010)*, whereas the Freundlich isotherm is an empirical model that considers heterogeneous adsorption on the adsorbent surface *Arunlertaree et al., (2007)*. The linearized equations for the Langmuir and Freundlich isotherms are expressed as (3) and (4), respectively.

$$C_e/q_e = 1 / bq_m + C_e/q_m \dots\dots(3) \quad \log q_e = \log k_F + 1/n \log C_e \dots\dots(4)$$

Where C_e = the adsorbate concentration at equilibrium (mg/L)

q_e = the adsorption capacity at equilibrium (mg/g)

b = the adsorption equilibrium constant (L/mg) related to the apparent energy of adsorption

q_m = the maximum adsorption capacity of adsorbent (mg/g)

n = indicates the bond energies between metal ion and the adsorbent

k_F = (mg/L) related to bond strength *El-Ashtoukhy et al., (2008)*

The linear plots of Langmuir and Freundlich equations representing Cr (VI) and Pb (II) adsorption by water hyacinth leaves are illustrated in Figs. (8, 9, 10 and 11). The adsorption constants of Langmuir and Freundlich equations and their correlation coefficients (R^2) are presented in Table (I). The value of R^2 is a measure of the goodness-of-fit of experimental data on the isotherm models. Results showed that the adsorption process by water hyacinth was better represented by the Freundlich equation compared to Langmuir isotherm, where the R^2 values were found to be 0.9926 and 0.9573 for Cr(VI) and to be 0.9911 and 0.9439, respectively. This indicated that the uptake of Cr(VI) and Pb (II) on water hyacinth were happened through heterogeneous adsorption.

From Table (I), the k_F constant of Freundlich isotherm, which expresses the selective uptake of Cr(VI) and Pb(II) and affinity of adsorbent were 18.446 and 15.829 (mg/L), whereas the $1/n$ value was found to be 0.423 and 0.531 for Cr(VI) and Pb (II), respectively. Generally, the value of $1/n$ that is related to the distribution of site bonding energies is ranging between (0.2 – 0.7) (*Kaewsomboon, 2006*). If $1/n$ is higher than 1, it indicates that the surface areas of adsorbent covered by the metal ions increase, and the energies of adsorption will decrease (*Kaewsomboon, 2006*).

Table(I) : Langmuir and Freundlich isotherm constants for adsorption of Cr(VI) and Pb(II) ions onto water hyacinth leaves at constant temperature 25°C

Metal	Langmuir Isotherm Constant			Freundlich Isotherm Constant		
	q_{max} (mg/g)	b (L/mg)	R^2	k_F (mg/L)	$1/n$	R^2
Chromium	26.25	0.012	0.9573	18.446	0.423	0.9926
Lead	37.45	0.022	0.9439	15.826	0.531	0.9911

Fig8,9,10,11

CONCLUSION

Water hyacinth leaves powder was found to be a very good adsorbent for lead and hexavalent chromium. It has good sorption capacity for both metals. The sorption capacity for hexavalent chromium was 15.0 mg/L and for lead was 18.0 mg/L. The sorption process was dependent on pH and the optimal pH value was 5 and 2 for lead and chromium, respectively. Biosorption of metals was dependent on temperature. Optimal temperature was 25 °C for chromium and 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 of pH at the end of biosorption process. Physical sorption and precipitation also contributed to removal of metals. The biosorption process followed Langmuir and Freundlich models for both metals which indicated that ion exchange took place in a monolayer at the surface of adsorbent.

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دراسات أدمصاصية لإزالة الكروم سداسي التكافؤ والرصاص من المياه الصناعية الملوثة باستخدام أوراق نبات ورد النيل كمادة إدمصاصية
طارق محمد يحيى عمر واحمد محمد الشناوى
المركز الإقليمي للإغذية والاعلاف- مركز البحوث الزراعية – الجيزة ، مصر

إن تواجد العناصر الثقيلة في البيئة المائية أصبح يشكل مشكلة خطيرة . لذا فان الاهتمام الأكبر للتقنيات الحديثة التي تستخدم لإزالة العناصر الثقيلة من مياه الصرف الملوثة. أهم هذه التقنيات هو الإدمصاص الحيوى الذى فيه يتم استخدام مواد طبيعية لإزالة العناصر الثقيلة من مياه الصرف الصناعية. حيث تم معالجة مياه الصرف المحتوية على الكروم سداسي التكافؤ و الرصاص بكتلة حيوية مجهزة من أوراق نبات ورد النيل. وقد وجد ان زمن التلامس (ساعتين) هى مدة كافية لعملية الإدمصاص وان سعة الإدمصاص بعد ساعتين ١٨ مجم /لتر ، ١٥ مجم / لتر من الرصاص والكروم على التوالي. درجة الرقم الهيدروجينى الفعالة هى ٥ للرصاص و ٢ للكروم. وتلعب درجة الحرارة دورا قويا فى عملية الإدمصاص الحيوى، فإزالة عنصر الرصاص تقل بزيادة الحرارة . على الوجه الأخر فإزالة عنصر الكروم تزداد بزيادة درجة الحرارة حتى ٤٠ م ثم تبدأ فى الإنخفاض وأن التبادل الأيونى كان هو العامل الرئيسى لميكانيكية الإزالة. تم تحليل بيانات الإمتزاز باستخدام النماذج الإدمصاصية الحرارية لكل من لانج موير وفرونيدلبيش فقد تم التوصل إلى أن استخدام المواد الإدمصاصية المحضرة من أوراق نبات ورد النيل التى تساعد فى إزالة التلوث الناتج من عنصرى الرصاص والكروم. و أوراق ورد النيل يمكن غسلها بواسطة محلول عيارى ٥ . هيدروكسيد الصوديوم ثم غسلها بالمياه عدة مرات للتخلص من المعادن الثقيلة الموجودة بها حتى يمكن إعادة استخدامها مرات أخرى. وتقدم تلك النتائج تقنية يمكن تطبيقها عمليا فى إزالة ايونات المعادن الثقيلة الموجودة فى المياه الملوثة الناتجة عن الأنشطة الصناعية وتقليل مشاكل البيئة.

قام بتحكيم البحث

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