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REMOVAL OF TOXIC HEAVY METALS FROM SEWAGE REUSE BY USING LOW-COST ADSORBENTS AND WASTE MATERIALS

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ABSTRACT

Treatment of high volumes of sewage reuse containing low concentrations of pollutants is becoming increasingly important as the discharge regulations become more stringent. Most of the past work has focused on the removal of higher concentrations of pollutants and on the more traditional and more expensive adsorbents/ion-exchange materials. This work has focused on the ability of low-cost adsorbents, as compared to the more traditional adsorbents, to remove from a high-volume waste stream, low concentrations of pollutants. The low –cost adsorbents investigated in this research include Eichhornia crassipes (water hyacinth); tree leaves (Eucalyptus camaldulensis and Casuarina glauca); maize cob and rice straw waste. The natural waste materials can be possible recycled by using dilute HCl and NaOH solutions, this process will be useful for the removal of residual of metals from absorbents after treatment to be used for several times. Also, investigations were carried out using a batch sorption process, the efficiency of sorption of lead, cadmium, arsenic, aluminum, chromium, mercury, nickel, cobalt, copper and zinc ions are affected by aqueous solution, pH, contact time, type of adsorbents, initial metal ion concentration all of which factors were investigated. The pollutant removal efficiencies and capacities of these materials were compared with removal efficiencies and abilities of the more traditional adsorbents: Amberlite IR-120 (plus) and CM- Cellulose.

The results experiment for lead ion has shown that the maximum removal capacity was for Amberlite IR with a capacity of approximately 99 %, followed by the CM-cellulose. While the low-cost adsorbents, water hyacinth plant and rice straw had shown the greatest removal capacities, approximately 43 – 90 %, compared to Eucalyptus camaldulensis; Casuarina glauca and Maize cob.

INTRODUCTION

The removal of metal ions from effluents is of importance to many countries of the world for both environmental considerations and water reuse. The application of low-cost sorbents including carbonaceous materials, agricultural products, and waste by-products has been investigated (Nguyen and Do, 2001).

The adsorption process with activated carbon is attracted by many scientists because of the effectiveness for the removal of heavy metal ion at trace quantities. But the process has not been used extensively for its high cost. For that reason, the use of low cost materials as sorbents for metal removal from wastewater has been highlighted. More recently, great effort has been contributed to develop new adsorbents and improve existing adsorbents (Babel and Kurniawan, 2003).

Natural materials that are available in large quantities or certain waste from agriculture operations may have potential to be used as low cost adsorbents, as they represent unused resources, are widely available and environmental friendly materials (Dens and Dixon, 1992).

In recent years, agricultural by-products have been widely studied for metal removal from water. These include peat (Ho and Mckay, 2000), pine bark (Al-Asheh and Duvnjak, 1997), peanut shells (Wafwoyo and Marshall, 1999), hazelnut shell (Cimino *et al*., 2000), rice straw (Daifullah *et al*., 2003), sawdust (Zhang *et al*., 2001), wool (Balkose and Baltacioglu, 1992) and leaves (Zaggout, 2001). Most of this work has shown that natural products can be good sorbents for heavy metals.

An investigation on the use of spheroid cellulose to remove chromium was also conducted on China. Cellulose is the most abundant among renewable and natural polymers and it has three reactive hydroxyl groups. The adsorption capacity of speroidal cellulose was found to be 73.46 mg Cr^{6+}/g at pH of 6.0 (Liu et al., 2001).

Daifullah *et al*; (2003) reported that the surplus, low value agriculture by-product rice husk can be made into sorbent materials which are used in the environmental remediation. In this study, two types of sorbents made from rice husk were characterized and evaluated. The efficiency of the two sorbents in the complex matrix containing six heavy metals which are Fe, Mn, Zn, Cu, Cd and Pb was about 100%.

Ansari *et al*;(1999) reported that Steamed Hoof Powder (SHP), was observed to have high adsorption capacity for Hg (II) with > 95 % removal from a solution containing 100 mg/l of Hg (II) with only 0.1% (W/V) concentration of SHP.

Ajmal *et al*., (2000) carried out an adsorption study on citrus reticulate, an agricultural waste originated from the fruit peel of Ni^{2+} from electroplating wastewater. The use of low-cost activated carbon derived from bagasse, an agricultural waste materials waste material, has been investigated as an alternative for the current expensive methods of removing heavy metals from wasterwater. The uptake of cadmium was found to be slightly greater than that of zinc and the sorption capacity increases with increase in temperature (Mohan and Singh, 2002).

The technical feasibility of using palm tree leaves as a low cost local biosorbent for the removal of nitrite from dairy wastewaters was investigated (Al-Muhtaseb *et al*., 2004). The study proved that the complete removal of nitrite from dairy wastewaters was achieved by using only one gram per liter. A novel biosorbent rice polish has been successfully utilized for the removal of cadmium (II) from wastewater. The maximum removal of cadmium (II) was found to be 9.72 mg/g at pH 8.6, initial Cd (II) concentration of 125 mg/l and temperature of 20 ^{0}C (Singh *et al*., 2005).

During the post two decades, there has interest in the use of aquatic plants in treating polluted effluents. Much of the attention has been addressed to Eichhornia crassipes (water hyacinth), a free-floating weed of worldwide distribution. The rapid, often excessive, growth of this plant allows biomass production that far exceeds the yield of most productive agricultural crops.

Early studies concerning the use of E. crassipes and other aquatic plants in pond systems to remove metal ions and other pollutants from wastewater were conducted by the National space. Technology Laboratories (NASA/NSTL) in United States in the 1970s. It was demonstrated at laboratory scale and a wastewater treatment station that this weed efficiently removes dilute concentrations of heavy metals, including Co, Ni, Sr, Ag, Cd, Hg and Pb.

Conversely, the metal sorption capacity of the dried biomass of aquatic plants has been recently recognized. The main advantages in using the dead biomass instead of living systems appears to be the following:

(a) Problems of metal toxicity on plant metabolism, plant deterioration, odor liberation, and insect's proliferation are avoided.
(b) The dried biomass presents advantages for conservation, transport, and handing and as such becomes ready for usage in wastewater units as a simple sorbent material.
(c) It is possible to recover the sorbet heavy metals by elution techniques using sorption/desorption cycles.

The adsorption of heavy metals into composts derived from rice straw, maize cobs or sawdust and at different states of maturity was examined in relation to their possible role in removing metals from wastewater. The desorption kinetics were determined through electro ultra filtration. The adsorption capacities of composts varied with raw materials used: rice straw > maize cobs > sawdust. The capacity to adsorb the metals increased with the maturity of the compost. Lead was the most readily adsorbed metal. The greatest difficulties in the subsequent desorption of the metals from the compost were encountered with rice straw and maize cob composts and with the metals lead, chromium and nickel .

Before mixing the adsorbate with the adsorbents (coal fly ash), the initial pH of each solution (pH=3 &4) was adjusted to the required value by adding 0.1 – 10M HNO_3 and 0.1 - 10 M NaOH solution (Nagarnaik *et al*., 2002). It should be noted that nitrate anions are not forming precipitates or complexes with the corresponding metals at the test conditions and are considered to be inert. In addition, at pH =3 and pH = 4, the effect of complexing of the metal ions with hydroxide ion are not significant (Mier, 2001). It is assumed that the impact of adjusting the initial solution pH with HNO_3 and NaOH solutions in terms of changing the chemistry of the solution is not significant.

The aim for this research is to develop inexpensive and effective metal ion adsorbents from plentiful sources of natural wastes, such as tree leaves (Eucalyptus camaldulensis and Casuarina glauca); aquatic plants (water hyacinth); maize cob and rice straw waste to offer these adsorbents as replacements for existing commercial materials. Tree leaves and aquatic plants have been proven for its effectiveness for the cleaning of air pollution and balance environment. However, to our knowledge, few such studies have been performed previously to use the tree leaves; aquatic plants; maize cob and rice straw waste to clean the wastewater. As we mentioned earlier natural wastes have been proven for its ability to remove the metal ions from sewage water.

MATERIALS AND METHODS

The synthetic samples which similar qualifications to treated sewage water; were prepared. The qualifications of synthetic samples are shown in Tables (1&2).

The plant tissues (water hyacinth, Eucalyptus Camaldulensis and Casuarina glauca) were gathered from twigs into clean plastic bags, washed with deionized water and laid flat on a clean table to dry (80 ºC for 72 hrs). Dry leaves were grounded with electrical grinder and were stored to be ready for application. Also, rice husk and maize cobs were collected from agricultural area cutted into small pieces by using a clean cutter and oven dried at 80 ºC for 72 hours. The dried materials were grinded by using a clean electric mixer and then stored in clean plastic bags. All natural waste materials were washed with 0.5 N NaOH solutions then with distilled water. Excess alkali was neutralized with 0.1 N HCl solutions and again washed with distilled water several times, to get ride of metals, Table (3) show that chemical analysis of the waste materials adsorbents after treatment, these steps are repeated when used their materials again for several times (Nagarnaik *et al*., 2002).

Table (1): Binding of metal ions with different natural waste materials by equilibrium experiments

Metal Solution	Pb		Cd		As		Al		Cr	
Initial conc.	10.0 mg / l		10.0 mg / l		10.0 mg / l		10.0 mg / l		10.0 mg / l	
Initial pH	6.00		6.00		6.00		6.00		6.00	
Materials	Final conc.	Removed %	Final conc.	Removed %	Final conc.	Removed %	Final conc.	Removed %	Final conc.	Removed %
Amberlite IR 120(plus)	0.113	98.33	0.905	90.95	1.975	80.25	2.2	78.00	1.179	88.21
CM - Cellulose	0.167	89.21	1.513	84.87	2.734	72.66	3.574	64.26	2.05	79.50
Water hyacinth plant	1.189	88.11	3.107	68.93	3.775	62.25	4.491	55.09	3.185	68.25
Eucalyptus camaldulensis	3.199	68.01	5.413	45.87	6.442	35.58	7.579	24.21	5.389	46.11
Casuarinas glauca	4.746	52.54	4.901	50.99	6.953	30.47	6.454	35.46	6.412	35.88
Maize cob	3.653	63.47	6.783	32.17	7.416	25.84	5.865	41.35	4.676	53.24
Rice straw	2.929	70.71	4.373	56.27	5.105	48.95	5.379	46.21	3.975	60.25

Experimental conditions:

1- **2 g adsorbent from materials /200 ml synthetic wastewater**

2- **Shake 120min @ 300 rpm at room temperature**

3- **Plants size 0.5-4.0mm**

The uptake percent of metal ions adsorbed from nitrate solution by water hyacinth, Eucalyptus Camaldulensis, Casuarina glauca, Rice straw, Maize Cobs, Amberlite IR-120 (plus) and CM- Cellulose was determined by batch experimental technique, where a constant V/m ratio was used. The analysis of the liquid phase was carried out using inductively coupled plasma (ICP-OES) Perkin Elmer Optima 2000 and atomic absorptions spectrophotometer (AAS) Perkin Elmer.

The amount of metal ion removal was determined using the following equation expressed as:

$$\text{Removal (\%)} = [(C_0 - C_1) / C_0] \times 100$$

Where: C_0 and C_1 are the initial and equilibrium concentration (mg/l) of metal ion solution, respectively

Table (2): Binding of metal ions with different natural waste materials by equilibrium experiments

Metal Solution	**Hg**		**Ni**		**Co**		**Cu**		**Zn**	
Initial conc.	**10.0 mg / l**		**10.0 mg / l**		**10.0 mg / l**		**10.0 mg / l**		**10.0 mg / l**	
Initial pH	**6.00**		**6.00**		**6.00**		**6.00**		**6.00**	
Materials	**Final conc.**	**Removed %**	**Final conc.**	**Removed %**	**Final conc.**	**Removed %**	**Final conc.**	**Removed %**	**Final conc.**	**Removed %**
Amberlite IR 120(plus)	4.488	55.12	2.479	89.33	3.405	65.95	0.927	90.73	1.579	84.21
CM - Cellulose	5.179	48.21	3.689	75.21	4.413	55.87	1.78	82.20	3.073	69.27
Water hyacinth plant	6.453	35.47	2.489	63.11	4.573	54.27	2.978	70.22	3.175	68.25
Eucalyptus camaldulensis	8.175	18.25	5.729	42.71	6.153	38.47	6.391	36.09	5.586	44.14
Casuarinas glauca	7.791	22.09	6.153	38.47	5.775	42.25	4.875	51.25	6.889	31.11
Maize cob	7.595	24.05	5.946	40.54	6.913	30.87	5.165	48.35	7.492	25.08
Rice straw	7.246	27.54	4.199	58.01	5.601	43.99	3.279	67.21	4.976	50.24

Experimental conditions:

1- 2 g adsorbent from materials /200 ml synthetic wastewater

2- Shake 120min @ 300 rpm at room temperature

4- Plants size 0.5-4.0mm

Batch Sorption Studies

The metal ions uptake from aqueous solutions and/or treated sewage water by the biosorbent was measured by placing the homogenously powdered 2 g (particle size 0.5-4.0 mm) was added to 200 ml of solution containing 10 mg/l metal solutions into contact with biosorbent under stirring. The metal solutions (Pb, Cd, As, Al, Cr, Hg, Ni, Co, Cu, and Zn) were prepared by dilution of 1000 mg/l standard solutions.

3

Preliminary experiments of adsorption kinetics indicated that a period of 120 min was sufficient to attain equilibrium. Separation of the suspended biosorbents was performed by filtration in polypropylene disks to avoid metal uptake by the filters. The concentration of metal ions in solution and the medium pH were determined both before and after biosorption by (ICP-OES) and with a pH - meter, respectively. Each experiment was repeated in duplicate runs. Sorption isotherms were measured at room temperature by the initial metal ion concentration and keeping the biosorbent mass constant. Isotherm adsorption models have been used in waste stream treatment to predict the ability of a certain adsorbent to remove a pollutant down to a specific discharge value. The two commonly used isotherm models are the Langmuir and the Freundlich models.

RESULTS AND DISCUSSION

Adsorption on natural waste materials: Tables (1, 2) listed the adsorption of ten kinds of metal ions on five kinds of natural waste materials. As shown in Tables (1,2) different metal ion on the same absorbent had different removal rate. At the same experimental condition, lead (Pb) had highest removal rate and mercury (Hg) had lowest removal rate. For lead, the highest removal rate in water hyacinth plant reached 88.11%. However, the highest removal rate for copper (Cu), cadmium (Cd), chromium (Cr) and arsenic (As) were 70.22%, 68.93 %, 68.25 %, and 62.25 %, respectively.

For the same metal ion, different waste materials had different removal rates. In the tested waste materials groups, water hyacinth plant, rice straw, and Eucalyptus camaldulensis showed highest removal rate for lead (Pb). Water hyacinth plant and rice straw also showed higher removal rate for cadmium (Cd), arsenic (As), aluminum (Al), chromium (Cr), mercury (Hg), nickel (Ni), cobalt (Co), copper (Cu), and zinc (Zn) ions. The highest removal rate for lead (Pb), arsenic (As), nickel (Ni) and zinc (Zn) were Eucalyptus camaldulensis. They were 68.01 % for lead ion (Pb), 35.58 % for arsenic ion (As), 42.71 % for nickel ion (Ni) and 44.14 % for zinc ion (Zn).

Influence of contact time on adsorption:

The time effect on adsorption of metal ions (Pb, Cd, As, Al, Cr, Hg, Ni, Co, Cu, and Zn) on the different materials (Amberlite IR 120(plus), CM – Cellulose, Water hyacinth plant, Eucalyptus camaldulensis, Casuarinas glauca, Maize cob, and Rice straw) were studied using synthetic solution at pH 6.00. Synthetic solution of 10.0 mg/l was used with V/m ration 200/2. The relation between the shaking time and uptake percent of metal ions on the different materials are given in Figs. (1-5). The results show that the percentage of metal ion adsorption by both sorbents increased with increasing time of equilibration and it reached the plateau value at 120 min. for both metal ions.

Adsorption on synthetic materials: Tables (1, 2) also showed the results of adsorption of metal ions on the Amberlite and CM-cellulose. The experiments were conducted under the same conditions as in the adsorption by natural waste materials. The experimental condition has been indicated in

Tables (1,2). In comparison with the adsorption of metal ions from wastewater by natural waste materials, Amberlite and CM-cellulose showed supreme results. The best removal rate for all metal ions were by Amberlite and CM- cellulose which are higher than five natural waste materials under the same test conditions. Water hyacinth plant removed 88.11 %, 54.27 % and 68.25 % for Pb, Co and Zn , which are quite close results as CM-cellulose . The best removal rate for Pb and Cu were by water hyacinth plant, 88.11 %, and 70.22 % in the tested group of natural waste materials, which is lower than Amberlite and CM- cellulose.

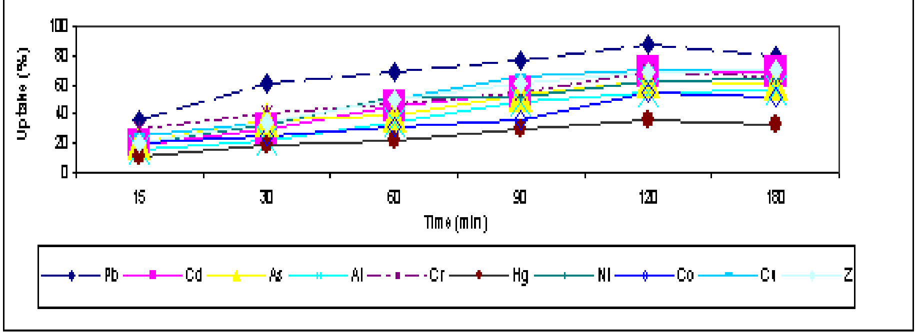

Fig. (1) Effect of contact time on uptake of metal ions adsorbed from synthetic solution by water hyacinth plant

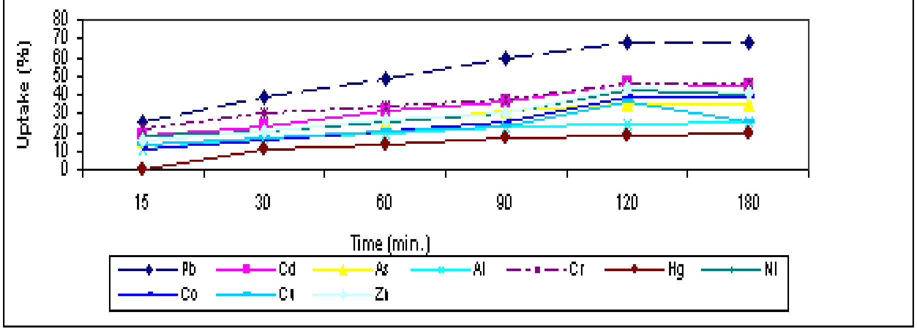

Fig.(2) Effect of contact time on uptake of metal ions adsorbed from synthetic solution by E. camaldulensis

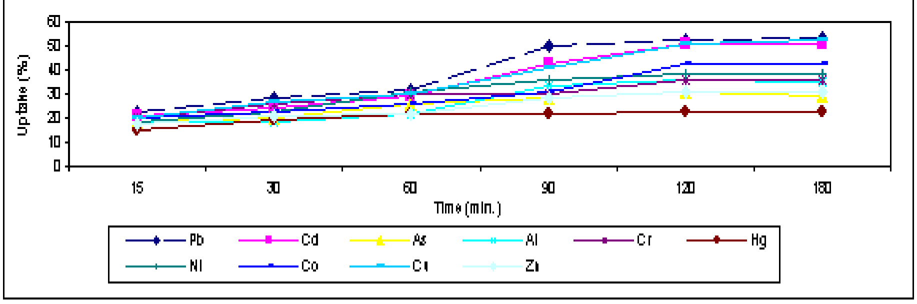

Fig.(3) Effect of contact time on uptake of metal ions adsorbed from synthetic solution by C. glauca

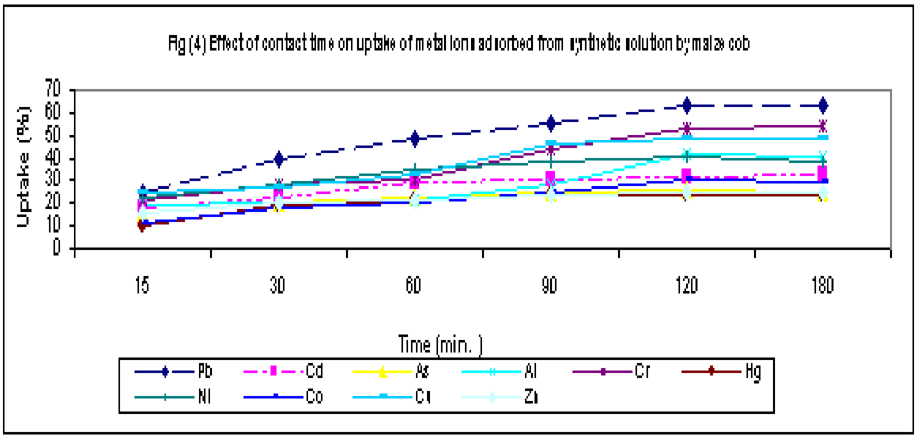

Fig (4) Effect of contact time on uptake of metal ions adsorbed from synthetic solution by maize cob

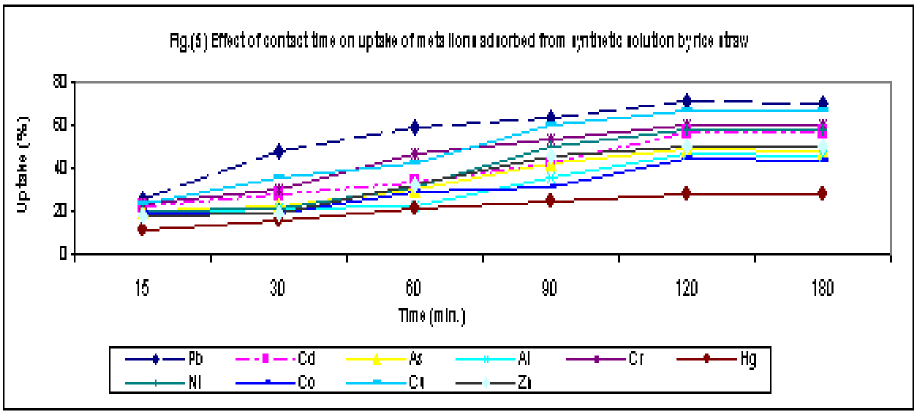

Fig.(5) Effect of contact time on uptake of metal ions adsorbed from synthetic solution by rice straw

Conclusion:

Natural waste materials (Water hyacinth plant, Eucalyptus camaldulensis, Casuarinas glauca, Maize cob and Rice straw) can be used in the treated sewage water treatment process for the removal of metal ions.

The adsorption of the metal ion on natural waste materials reached equilibrium in 120 minutes.

The adsorption of different metal ions on the same natural waste materials had different removal rate, Pb, Cr, Cu, Cd, and Zn had the highest removal rate, the rest of metal ions had the lowest removal rate.

For the same metal ion, different natural waste materials had different removal rate for Water hyacinth plant and Rice straw showed highest removal rate for all metal ions (Pb, Cd, As, Al, Cr, Hg, Ni, Co, Cu and Zn) , while Casuarinas glauca and Mize cob showed the lowest removal rates for (Pb, Cr, Ni, Co, Zn, Cd and As).

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إزالة العناصر الثقيلة من مياه الصرف الصحي المعالج باستخدام مخلفات زراعية منخفضة التكلفة

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أصبح من الضروري الاهتمام بالكميات الكبيرة من مياه الصرف الصحي المعالجة التي تحتوى على تركيزات منخفضة من الملوثات , خاصة بعد إصدار قوانين مشددة بخصوص التلوث البيئي . وقد كان في السابق التركيز ينصب على الملوثات عالية التركيز والطرق التقليدية في عملية التنقية والتي تعتبر عالية التكلفة.

تعتمد الدراسة على كيفية التخلص من الملوثات ذات التركيز المنخفض باستخدام مواد منخفضة التكاليف مقارنة بالطرق التقليدية.

المواد المنخفضة التكاليف المستخدمة في هذا البحث هى نبات ورد النيل , أوراق شجر الكافور والجز والين ومخلفات قش الأرز وقوالح الأذرة. حيث يمكن الاستفادة من هذه المخلفات عدة مرات وذلك بإعادة معالجتها بمحلول مخفف من حامض الهيدروكلوريك وهيدروكسيد الصوديوم , لإزالة العناصر المتراكمة عليها بعد عملية المعالجة.

أجريت المعاملات باستخدام طرق الادمصاص المختلفة, وكذلك فعالية (كفاءة) امتصاص كل من عناصر الرصاص, الكادميوم, الزرنيخ, الألمونيوم,الكروم,الزئبق,النيكل,الكوبالت,النحاس والزنك والتي تتأثر بكل من الوسط المائي , الرقم الهدروجيني, زمن التلامس, نوعية مادة الادمصاص و تركيز البداية لكل العناصر موضع البحث. كذلك تم مقارنة كفاءة و قدرة الامتصاص بين الطرق موضع الاختبار وتلك الخاصة بالطرق التقليدية وهى (Amberlite IR 120 (Plus أو CM-Cellulose.

تبين نتائج التجربة آن عنصر الرصاص أعلى سعة للإزالة باستخدام Amberlite IR 120(Plus) وهى تقريبا99% ثم يليه مادة CM-Cellulose , بينما بقية أيونات العناصر الأخرى فهي اقل من أيون عنصر الرصاص . أما المواد قليلة الثمن مثل نبات ورد النيل و مخلفات قش الأرز لهما قدرة عالية لأزاله العناصر الثقيلة و هي تقريبا من 43 – 90 % , مقارنة بالنباتات الأخرى (أوراق شجر الكافور والجز والين وقوالح الأذرة)

Table (3): Chemical analysis of the waste materials adsorbents

Metals (mg/l)	Water hyacinth	E. Camaldulensis	C. glauca	Maize cob	Rice straw	Water hyacinth	E. Camaldulensis	C. glauca	Maize cob	Rice straw
	Analysis before washed with dilution (NaOH/HCl) and after treatment process					Analysis after washed with dilution (NaOH/HCl)				
Pb	8.109	5.833	5.209	6.082	6.079	0.127	0.087	0.025	0.04	0.011
Cd	6.124	4.201	4.354	3.232	5.431	0.005	0.001	0.004	0.008	0.006
As	6.316	3.345	3.053	2.685	4.516	0.241	0.112	0.078	0.102	0.095
Cr	6.524	4.654	3.697	5.029	6.535	0.012	0.007	0.011	0.09	0.017
Hg	3.247	1.945	2.255	2.412	2.754	0.125	0.098	0.108	0.245	0.187
Ni	7.712	5.482	4.145	4.354	6.502	0.151	0.255	0.111	0.231	0.341
Co	5.321	4.102	4.551	3.219	4.488	0.115	0.095	0.114	0.041	0.301
Al	5.645	2.954	3.946	4.651	4.542	0.150	0.188	0.259	0.084	0.052
Cu	12.212	7.976	8.339	5.421	5.932	5.987	4.012	4.255	0.624	0.246
Zn	9.784	7.114	6.141	4.478	6.124	3.15	4.01	3.15	2	1.7

- ***All natural waste materials were washed with 0.5 N NaOH solutions then with distilled water. Excess alkali was neutralized with 0.1 N HCl solutions and again washed with distilled water several times for used for several tines in the process treatment.**