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A STUDY ON REMOVING HEAVY METALS FROM WASTEWATER BY USING LOW-COST ADSORBENTS

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ABSTRACT

The purpose of this study was to investigate the possibility of the utilization of rice straw, bagasse, maize cob, garlic skin, and onion skin as a low cost adsorbents. Batch experiments were carried out on a laboratory scale to evaluate the adsorption efficiency and removal of heavy metals from aqueous solutions by different natural waste materials under various conditions of pH, contact time, and initial metal ion concentration. The equilibrium time was found to be 120 min. and optimum pH value was found to be 6.0. To apply the finding of the study, real wastewater sample was obtained and different adsorbents were used for its treatment as a case study. The investigated adsorbents showed a good efficiency in removing toxic heavy metals such as lead, cadmium, arsenic, chromium and copper from their aqueous solutions.

INTRODUCTION

The present of heavy metals in the environment is of great concern to scientists and engineers because of their increased discharge toxic nature and other adverse effects on receiving waters. The important toxic metals, which include Cr, Cd, Zn, Ni, Cu, Pb, etc, find there was due to water bodies through industrial wastewater discharges. Industries such as metal cleaning and plating baths pulp, paper and board mils, fertilizer production, tannery operations, chemical manufacturing, petroleum refining, mine drainage, wood preserving, battery manufacturing, and leachates from hazardous waste disposal sites containing metal ions cause heavy metal pollution (Gupta, 1998) & (Sreenivasulu and Komal, 1998).

Lead poisoning causes diseases such as anemia, damage to the central nervous system, mental deterioration, etc. (Moha *et al*., 1998). Excessive copper intakes lead to accumulate in the liver, copper poisoning and gastro-intestinal catarrh. Copper is toxic to aquatic organisms even at very small concentrations. Wastewater from plating shops may contain Pb (II) in the range of 100-250 ppm (Muthukumaram *et al*., 1995).

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, 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 the increase in temperature (Mohan and Singh, 2002).

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 removed 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 in 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 straw can be made into sorbent materials which are used in the environmental remediation. In this study, two types of sorbents made from rice straw 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%.

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*., 2003).

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 roles 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 was 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 .

The optimum contact time for the adsorbents under investigation is comparable to those previously obtained by many authors for example, the equilibrium time needed for the removal of Pb (II), Cd (II), Cu (II), Ni (II) and Zn (II) onto black gram husk was found to be 30 min (Saeed *et al*., 2005), the time needed to reach equilibrium for Cr (VI) removal on wool was 1.5 h (Dakiky *et al*., 2002), equilibrium time for the adsorption of Cd (II) on rice polish at various adsorbate concentrations was found to be 90 min (Singh *et al*., 2005) and the maximum percent metal removal was attained after about one hour of stirring time for the removal of Ni(II) on sawdust (Shukla *et al*., 2005).

In the present study, the adsorption of heavy metal ions on low-cost adsorbents and waste materials (rice straw, bagasse, maize cob, garlic skin, and onion skin) were evaluated under various conditions such as pH value, contact time, and initial metal ion concentration through kinetic studies. The optimum removal condition was also identified for each metal ion. In addition, a series of tests were conducted to investigate how the presence of other metal ions affected the removal of one metal ion. Finally, the effectiveness of natural waste materials in the removal of heavy metals was evaluated using real wastewater.

MATERIALS AND METHODS

The plant tissues (rice husk, bagasse, maize cob, garlic skin, and onion skin) were collected from agricultural area cutted into small pieces by using a clean cutter and oven dried at 80 °C for 72 hrs. 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, these steps are repeated when used their materials again for several times **(**Nagarnaik *et al*., 2002).

Mixed metal ion solutions of (Pb^{2+}, Cd^{2+}, As^{2+}, $Cr^{3+,}$ and Cu^{2+}) were prepared from Merck-analytical grade stock standards of concentration 1000 ppm. The synthetic wastewater was then prepared by diluting the stock standard of each. The pH of the wastewater was adjusted using 1 M HCl and /or NaOH. The final concentrations of metal ions in the wastewater were analyzed by inductively coupled plasma (ICP-OES) Perkin Elmer Optima 2000. Treated sewage water produced from Abu-Rawash treatment station was selected in this study, characterization of the treated sewage water as standard method (APHA, 1992), was obtained in Table (1)

Table (1): Characterization of the treated sewage water

Parameter	Concentration (mg/l) except for pH value
pH	6.25
Pb	0.647
Cd	0.081
As	0.081
Cr	0.241
Cu	0.862

The amount of metal ion removal was determined using the following equation expressed as: Removal (%) = $[(C_0 - C_1) / C_0] \times 100$

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

RESULTS AND DISCUSSION

(1) Sorption studies

Successful application of the adsorption technique demands innovation of cheap, non-toxic, easily and locally available adsorbents of known kinetic parameters and sorption characteristic. Knowledge of the optimal conditions would herald a better design. Thus the effect of some major factors such as pH, contact time, and initial metal ions concentration (Pb^{2+}, Cd^{2+}, As^{2+}, $Cr^{3+,}$ and Cu^{2+}) on the adsorption on the adsorbents materials (rice straw, bagasse, maize cob, garlic skin, and onion skin) were investigated from the kinetic viewpoint. Adsorption studies were performed by batch technique to obtain the rate and equilibrium data. Experiments were carried out by shaking 2 gm of adsorbent with 200 ml of aqueous solution containing a known concentration of (Pb^{2+}, Cd^{2+}, As^{2+}, $Cr^{3+,}$ and Cu^{2+}) ions and by agitating the samples on the shaking machine at a speed of 300 rpm.. Samples containing mixed metal ions were maintained at a desired pH adding 0.5 M HNO_3 or 0.1 M NaOH. All the experiments were conducted at room temperature.

(2) Effect of pH

The initial pH value of the aqueous solution is a very important factor for adsorption of metal ions from aqueous solution because it affects the solubility of the metal ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the adsorbate during reaction. To examine the effect of pH on the removal efficiency of Pb, Cd, As, Cr and Cu on the different adsorbents, the pH was varied from 3.0 to 8.0, as previously mentioned in the experimental part, in separate experiments. Other parameters such as dose of adsorbent, contact time and metal ions concentration were kept constant. In order to avoid hydroxide precipitation of metal ions no further increase in pH was applied (Amer , 1998).

It is clear that, the adsorption of the studied metal cations increased as pH increases and recorded its minimum values at pH 3.0. Thus can be justified on the bases that at lower pH values, the H^+ ions compete with the metal cations for the adsorption sites in the system, which in turn leads to partial releasing the later. The heavy metal cations are completely released under extreme acidic conditions (Forstner and Wittman, 1981). The influence of pH on sorption efficiency of the different natural adsorbents towards Pb (II), Cd (II), As (II), Cr (III) and Cu (II) are illustrated in Table (2), which showed that their removals percentages depend on type of metal ion, nature of adsorbent and the different operating conditions.

The removal percentages increased as the pH values increased from 3.0 to 8.0. The removal of Pb (II) attained its maximum at pH 6.0 on rice husk, maize con and onion skin. The removal of cadmium was less in acidic range and reached a maximum adsorption around pH 8.0. In addition to this effect, the adsorbent surface is highly protonated in acidic medium, which is not favorable for cadmium uptake because in this medium, Cd^{++} is the dominant ion (Huang and Ostovic , 1978). As a result the adsorption of cadmium is hindered due to electrical repulsion. As pH increases the degree of protonation of surface reduces gradually and approaches zero at pH 7.0 where Cd^{++} and $CdOH^{+}$ species are present in solution , the adsorbent surface starts acquiring a net negative charge making the situation electrostatically favorable for a higher uptake of cadmium. Above pH 8.0 removal of cadmium started to decrease due to soluble hydroxyl complexes of cadmium hydroxides (Leckie and James, 1974).

The uptake percentage of Cr (III) and As (II) increased with the pH change from 3.0 – 8.0 then decreased sharply at pH 8.0. These results are in accordance with those found in the literature (Nomanbhay and Palanisamy, 2004) stating that the uptake of free ionic Cr (III) depends on pH, where optimal metal removal efficiency occurs at pH 5.0 and declining at higher pH. Chromium, arsenic, and some other metals, depending on pH, are known to exist as anions. At higher pH, the presence of Oxygen containing functional groups makes the adsorbent surface negatively charged and hence there is repulsive electrostatic interaction between the adsorbent and the anions (Faria *et al*., 2004). At pH greater than 8.0, insoluble chromium hydroxide starts to precipitate from the solution, making true sorption studies impossible.

Table (2) Optimum pH for maximum removal (%) of the studied metal ions onto natural waste materials

Metals \ Adsorbents	Bagasse	Rice husk	Maize cob	Onion Skin	Garlic Skin
Pb	98.88%pH 4.0	90.82%pH 6.0	99.95 %pH 6.0	97.24%pH 6.0	67.85 %pH 4.0
Cd	65.55 %pH 6.0	98.17% pH 8.0	65.73 %pH 6.0	75.88 %pH 6.0	87.45 %pH 6.0
As	96.68 %pH 6.0	97.00% pH 6.0	98.62% pH 6.0	85.17 %pH 6.0	78.24 %pH 6.0
Cr	91.85 %pH 6.0	98.12% pH 6.0	97.95% pH 6.0	88.92 %pH 6.0	85.17% pH 6.0
Cu	67.40 %pH 6.0	80.66% pH 6.0	95.84%pH 6.0	85.40 %pH 6.0	65.80%pH 6.0

(3) Effect of contact time

Tables (3-7) depict the effect of contact time on the rate of uptake of Pb^{2+}, Cd^{2+}, As^{2+}, Cr^{3+} and Cu^{2+} ions. It was found that the removal of metal ions increased with increase in contact time to some extent. Further increase in contact time did not increase the uptake due to deposition of metal ions on the available adsorption sites on the adsorbent materials. Preliminary investigations into the uptake of Pb^{2+}, Cd^{2+}, As^{2+}, $Cr^{3+,}$ and Cu^{2+} ions on the adsorbent materials at their optimum pH values indicated that the processes are quite rapid.

Optimum contact time for all adsorbents was found to be range between 30 – 120 min and hence all further experiments were conducted at 120 min of contact time. This result is important, as equilibrium time is one of the important parameters for an economical wastewater treatment system. Many authors studied the effect of contact time on adsorption process of metal ions from wastewater. Their results indicate that the time of equilibrium was dependent on the nature of the adsorbent and on the metal ions.

Table (3): Effect of contact time on lead removal by different materials

Materials	Time (min)	30	60	90	120	180	240
Bagasse	**Uptake %**	35.55	61.27	87.61	95.77	95.28	95.25
Rice husk	**Uptake %**	25.44	56.32	78.32	90.85	92.54	91.99
Maize cob	**Uptake %**	33.54	65.32	81.24	99.42	98.21	92.1
Onion skin	**Uptake %**	35.21	71.25	85.05	97.14	98.14	98
Garlic skin	**Uptake %**	28.67	54.61	65.09	78.11	80.21	80.11

Table (4): Effect of contact time on cadmium removal by different materials

Materials	Time (min)	30	60	90	120	180	240
Bagasse	**Uptake %**	24.23	45.25	56.27	65.67	70.25	69.25
Rice husk	**Uptake %**	48.35	54.68	79.32	88.36	87.21	86.98
Maize cob	**Uptake %**	22.32	45.32	62.31	65.89	64.21	64.01
Onion skin	**Uptake %**	45.36	65.32	80.21	95.67	90.25	90.11
Garlic skin	**Uptake %**	36.87	44.28	70.54	86.25	87.25	82.32

Table (5): Effect of contact time on arsenic removal by different materials

Materials	Time (min)	30	60	90	120	180	240
Bagasse	Uptake %	48.36	75.21	80.58	96.9	95.21	94.87
Rice husk	Uptake %	54.98	78.25	88.21	97.08	97.08	96.55
Maize cob	Uptake %	39.25	76.21	89.32	98.67	97.25	97
Onion skin	Uptake %	29.39	58.32	69.25	85.24	87.21	88.09
Garlic skin	Uptake %	25.22	48.22	66.26	78.62	77.25	77.19

Table (6): Effect of contact time on chromium removal by different materials

Materials	Time (min)	30	60	90	120	180	240
Bagasse	Uptake %	44.28	65.25	84.25	91.85	91.98	90.11
Rice husk	Uptake %	54.74	69.25	80.24	98.18	98.54	97.21
Maize cob	Uptake %	43.24	70.25	87.21	98.78	97.25	97.11
Onion skin	Uptake %	35.87	55.41	71.26	88.95	89.26	88.25
Garlic skin	Uptake %	33.26	65.36	75.09	85.14	87.26	87.22

Table (7): Effect of contact time on copper removal by different materials

Materials	Time (min)	30	60	90	120	180	240
Bagasse	Uptake %	19.25	35.74	54.01	67.67	70.21	69.25
Rice husk	Uptake %	26.34	56.11	70.26	80.05	82.54	81
Maize cob	Uptake %	35.65	52.32	85.21	95.67	95.22	95.08
Onion skin	Uptake %	38.54	58.94	71.26	85.26	88.25	89.21
Garlic skin	Uptake %	28.25	45.28	58.65	65.33	65.01	64.26

(4) Effect of initial metal ions concentration

The influence of initial heavy metal ions concentration on the removal efficiency of Pb (II), Cd (II), As (II), Cr (III), and Cu (II) on the different adsorbents was studied by varying the initial concentration from 5 – 20 mg/l of mixed metal ions solution, while keeping dose of adsorbents, the pH and the contact time constant. The data are illustrated in Tables (8-12) for the removal of metal ions.

The results indicted that; the increase in initial concentration of Cd (II) decreased their percentage removal on the different adsorbents under investigation. This is because at higher initial concentrations the ratio of initial number of moles of metal ions to the available adsorbent surface area is high; hence fractional adsorption becomes dependent on initial concentration. For fixed adsorbent dose, the total available adsorption sites are limited thereby adsorbing almost the same amount of sorbate thus resulting in a decrease in percentage removal of the adsorbate corresponding to an increase in initial sorbate concentration (Krishnan and Anirudhan, 2003).

The removal efficiency of lead on the different natural waste materials increase with increasing of the initial metal ions concentration and was constant for maize cob, onion skin, and garlic skin. For all adsorbents, the efficiency As (II) and Cu (II) decreased with increase of the initial metal ions concentration, except for rice husk and onion skin in case of Cu (II). The removal efficiency of the different natural waste materials for Cr (III) removal varied for each adsorbent as follows: - In case of bagasse and maize cob the removal efficiency was constant at a concentration 15 & 20 mg/l of metal ion concentration. While for rice husk, onion skin and garlic skin, removal efficiency decreased by increasing initial metal ion concentration.

From all the above results it is clear that the general trend was decrease of the removal percentage with increase in initial concentration from 5 – 20 mg/l. These results are in good agreement with many previous studies found in literature. (Vijayaraghavan *et al*., 2004). Found that the percentage removal of Cu (II) by Ulva retculata decreased as the copper concentration increased. This is because at lower concentration, the ratio of the initial number of moles of Cu (II) to the available surface area is low and subsequently the fractional sorption becomes independent of initial concentration. However, at high concentration the available sites of sorption becomes fewer compared to the moles of Cu (II) present and hence the percentage removal of Cu (II) is dependent upon the initial Cu (II) concentration.

Table (8): Effect of initial metal ions concentration on uptake of lead adsorbed by different natural waste materials from mixed solution

Concentration (ppm)	**5.00**	**10.00**	**15.00**	**20.00**
Materials	**Uptake %**			
Bagasse	92.11	95.78	97.66	98.85
Rice husk	89.21	90.85	95.25	97.31
Maize cob	90.95	99.44	95.28	95.14
Onion skin	95.24	97.24	95.24	95.00
Garlic skin	77.24	78.00	66.56	66.27

Table (9): Effect of initial metal ions concentration on uptake of cadmium adsorbed by different natural waste materials from mixed solution

Concentration (ppm)	**5.00**	**10.00**	**15.00**	**20.00**
Materials	**Uptake %**			
Bagasse	60.88	65.68	50.77	48.42
Rice husk	82.66	88.36	60.69	57.68
Maize cob	60.38	65.89	45.28	33.00
Onion skin	93.55	95.67	65.88	51.54
Garlic skin	80.24	87.24	55.67	43.12

Table (10): Effect of initial metal ions concentration on uptake of arsenic adsorbed by different natural waste materials from mixed solution

Concentration (ppm)	**5.00**	**10.00**	**15.00**	**20.00**
Materials	**Uptake %**			
Bagasse	95.84	96.91	83.47	69.31
Rice husk	93.26	97.08	80.36	65.00
Maize cob	95.29	98.67	82.24	62.15
Onion skin	80.24	85.24	65.17	54.87
Garlic skin	70.67	78.64	53.54	33.00

Table (11): Effect of initial metal ions concentration on uptake of chromium adsorbed by different natural waste materials from mixed solution

Concentration (ppm)	5.00	10.00	15.00	20.00
Materials	**Uptake %**			
Bagasse	**85.64**	**91.85**	**88.47**	**88.31**
Rice husk	**95.26**	**98.18**	**70.36**	**65.13**
Maize cob	**93.09**	**97.77**	**88.19**	**88.02**
Onion skin	**90.24**	**88.94**	**65.97**	**42.87**
Garlic skin	**88.67**	**85.14**	**58.54**	**27.00**

Table (12): Effect of initial metal ions concentration on uptake of copper adsorbed by different natural waste materials from mixed solution

Concentration (ppm)	5.00	10.00	15.00	20.00
Materials	**Uptake %**			
Bagasse	65.24	67.67	55.45	35.87
Rice husk	74.01	80.05	85.68	88.00
Maize cob	90.25	95.67	50.24	30.06
Onion skin	70.25	85.26	86.67	87.17
Garlic skin	62.64	65.34	40.25	27.11

Application of different materials on the treated sewage water by using batch technique: -

Based on the promising results of heavy metal removal from aqueous solution, tests were conducted to evalute these results using real wastewater. The wastewater used treated sewage water, which used for irrigation from Abu-Rawash region. To study the effectiveness of the adsorbents under investigation on the removal of Pb^{2+}, Cd^{2+}, As^{2+}, Cr^{3+} and Cu^{2+} from the wastewater the optimum conditions obtained from the laboratory scale study were applied, 2 g of each of the materials with mixed with 200 ml of wastewater (sewage water) without shaking as optimum. The samples were then analyzed for the remaining metal ions concentration. The results of removal of Pb^{2+}, Cd^{2+}, As^{2+}, Cr^{3+} and Cu^{2+} are given in Table (13). The results revealed the following facts: -

Table (13): Heavy metals remaining concentration and adsorbents removal efficiencies

Absorbent	Remaining concentration (ppm)					Removal efficiency (%)				
	Pb	Cd	As	Cr	Cu	Pb	Cd	As	Cr	Cu
Rice straw	0.031	0.017	0.020	0.009	0.113	95.20	79.01	75.30	96.26	86.89
Bagasse	0.037	0.019	0.022	0.012	0.135	94.28	76.54	72.83	95.02	84.33
Maize cob	0.044	0.021	0.021	0.014	0.193	93.19	74.07	74.07	94.19	77.16
Onion skin	0.038	0.022	0.025	0.015	0.252	91.03	72.83	69.13	93.77	70.76
Garlic skin	0.062	0.024	0.029	0.018	0.269	90.41	70.37	64.19	92.53	68.79

The adsorbents under investigation were effective in the removal of $Pb2^{+}$, Cd^{2+}, As^{2+}, Cr^{3+} and Cu^{2+} ions from treated sewage water to different extents according to their nature and the removal metal ion. While $Cr3^{+}$ and Pb^{2+} ions were the most adsorbed ions on all of the different natural waste materials, rice husk showed higher efficiency for the removal of all metal ions. The removal efficiencies order on rice husk, bagasse, maize cob, onion skin and garlic skin was:

Cr > Pb > Cd > Cu > As

Conclusion

In this study the feasibility of using some locally available materials for wastewater treatment by adsorption was investigated. Significant data were obtained through batch experiment for the removal of Pb2+, Cd^{2+}, As^{2+}, Cr^{3+} and Cu^{2+} ions by adsorption using rice husk, bagasse, maize cob, onion skin and garlic skin. Those locally available materials were appeared to be promising adsorbents for metals removal. The adsorbent metal ions concentration, contact time, and pH of the metals solution. Optimum removal was achieved at 120 min of contact and within pH value ranges between 4-6. For the treated sewage water from Abu-Rawash region that contained lead, cadmium, arsenic, chromium and copper ions, a treatment process using the studied adsorbents was found to be efficient. Based on the results noted a process using those materials for the removal of heavy metals is potentially more economical than current process technology.

More studies are needed to optimize the system from the regeneration point of view and to investigate the economic aspects. More studies are also needed to investigate the possible uses of the treated water either in industrial or agricultural application.

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دراسة على إزالة العناصر الثقيلة من مياه الصرف باستخدام المخلفات الزراعية ذات التكلفة المنخفضة

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الهدف من تلك الدراسة توضيح إمكانية الاستفادة من قش الأرز , مخلفات عصير القصب,فوالج الذرة, قش الثوم وقش البصل كمواد أد مصاص منخفضة التكلفة. أجريت التجربة في إطار معملي لبحث القدرة الأدمصاصية للمخلفات سابقة الذكر وذلك مع تثبيت العوامل التالية : الأيون الهيدروجيني, فترة التلامس و التركيز الأولى لأيون العنصر.

وقد نتج عن ذلك التوصل إلى إن نقطة الاتزان التبادلي التي تحققت بعد120 دقيقة , بينما درجة الإيوان الهيدروجيني المثلى 6, ولتحقيق تلك النتائج تطبيقا تم استخدام مياه صرف صحي معالجة في عدة مصادر باستخدام وسائل أد مصاص مختلفة. وقد أوضحت نتيجة البحث كفاءة مواد الأدمصاص في عمل إزالة العناصر الثقيلة السامة مثل الرصاص,الكادميوم, الزرنيخ, الكروم, والنحاس في الوسط المائي.