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Adsorption of copper, lead and cadmium from aqueous solutions by activated carbon prepared from saffron leaves

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Abstract

Background: Industrial development has caused the release of various pollutants including heavy metals into the environment. These toxic compounds are extremely dangerous to living beings and the environment due to their non-biodegradability, severe toxicity, carcinogenicity, the ability to be accumulated in nature and the ability to contaminate groundwater and surface water. The aim of the present research was to provide an appropriate and cost-effective adsorbent to remove heavy metals from aqueous solutions.

Methods: The activated carbon was produced from the dried. Batch experiments were performed on real and synthetic samples at room temperature. The effect of pH, adsorbent dose, initial concentration, and contact time were studied, and the adsorption isotherms of heavy metals were determined. The removal efficiency was evaluated on real wastewater.

Results: The maximum removal efficiency of heavy metals (copper, cadmium and lead) by activated carbon adsorbent prepared from saffron leaves was obtained in pH 7. The optimum amount of adsorbent was 0.6 g, and the optimum contact times were 45 min for copper and cadmium ions and 90 min for lead ion, respectively. In these optimum conditions the removal efficiencies were 76.36%, 91.25% and 97.5%, respectively. The removal efficiencies of heavy metals from actual samples (copper industry and the battery industry) in the optimum conditions were 82.25%, 69.95% and 91.23%, respectively. The results obtained showed the highest correlation with Langmuir isotherm model.

Conclusion: Based on the results obtained, the activated carbon produced from saffron leaves has a good capability in removal of the metal ions from the aqueous solutions. Considering the availability of saffron leaves in Khorasan, its cost-effectiveness, and high uptake capacity, it can be applied as a proper absorbent to remove the heavy metals from industrial wastewater.

Keywords: Adsorption, Heavy metal, Activated carbon, Saffron leaves

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Introduction

Increasing pollution of municipal and industrial sewages by toxic compounds due to industry development and growth is a concerning issue. Heavy metals because of the nature of their non-degradability, sever toxicity, ability to be accumulated, and carcinogenicity not only endanger aquatic life, but also cause the acceptor waters to be inappropriate for various uses such as drinking (1). Discharging sewage of many industrial processes like plating, photography, nuclear power plants, and petrochemical industries into water resources causes water pollution. Heavy metals like lead and cadmium, even at low concentrations are serious threats to living organisms through their being stimulating, property aggregation ability, carcinogenicity, and mutagenesis (2). These metals are non-degradable and very resistant against biodegradation. The adverse effects of their presence in body can be diarrhea, abdominal pains, severe vomiting, fracture, sterilization, and they can damage the central nervous system, body immune systems, and also cause psychological disorders, possible damage to DNA, and cancer (3-8).

There are a variety of methods such as sedimentation, filtration, oxidation and reduction, ion exchange, and membrane separation for heavy metals removal. These methods are often ineffective or expensive in very low concentrations (3,4).

Adsorption is a process during which a soluble material accumulates on the surface of another substance (9). In fact, adsorption is the function of transferring from liquid phase to solid phase. Adsorption is an important phenomenon in many physical, chemical, and biological processes in the nature. Adsorption is also widely used in water and wastewater treatment (7). One of the most widely used adsorbents for this purpose is activated carbon. Its high cost of activation stages and its low regenerative capacity has limited the use of this material on a large scale. In recent years, using inexpensive adsorbents has attracted the attention of many researchers. These adsorbents are frequently found and are available, and their preparation costs are low (6). The use of bioadsorbents to remove metal ions from aqueous solutions is a cost-effective and environment-friendly method. Therefore in recent decades, it has attracted the attention of many researchers. Successful use of natural materials such as activated carbon produced from olive kernel in the removal of heavy metals like copper, lead and cadmium was experienced by Bohli *et al* (8) in 2013, in Tunisia.

Activated carbon produced from tassel to remove lead was one of the applied adsorbents used by Moyo *et al* (5). Also Hadoun *et al* (10) investigated the removal of cadmium from the aqueous solutions using activated carbon produced from palm stem in Algeria. *Yahaya* et al (11) investigated the removal of copper using activated carbon produced from rice husk. Amuda *et al* studied the heavy metals removal by charcoal made from coconut shell that resulted in a very effective absorbent from cheap row material (11). The study by Song *et al* (12) on the removal of lead by activated carbon made of coconut skin activated by potash had good results. Nale *et al* (13) performed studies of kinetics and copper absorption equilibrium from aqueous solutions on activated carbon produced from corn cob. Studies related to kinetics and cadmium removal equilibrium from aqueous solutions by activated carbon produced of apple skin were carried out by Sartape *et al* (14). Another study was conducted by Arivoli *et al* (15) titled "The dynamic of copper adsorption by low-cost activated carbon". Saffron with the scientific name of Crocus sativus, is a plant from the family of gladiola. There was 57,000 hectares of cultivated area in Razavi Khorasan which was equivalent to 77% total production of country in 2012.

The aim of this study was to investigate the removal efficiency of heavy metals (copper, cadmium and lead) with activated carbon prepared from leaves of saffron in the synthetic and real solutions as an available and inexpensive absorbent in the region. The effective parameters on absorption process such as contact time, pH, and initial concentration of metal ions were studied. Also the adsorption equilibriums were studied.

Methods

In this practical study, the efficacy of activated carbon prepared from saffron leaves for the removal of metal ions (copper, cadmium and lead) from the aqueous solutions was studied.

The chemical materials made by Merch company with high purity were used. The stock solutions of metal ions with concentration of 1000 mg/l were prepared. In all stages of the experiment, the 0.1 N normal solutions of NaOH and H_2SO_4 were used to adjust pH. pH meter (HANNA instruments HI 110 Model) was used to measure the pH of the solution

Adsorbent preparation

Sorbent preparation was performed as follows. Saffron leaves were harvested from the cultivated fields around Torbat Heydarie city located in Razavi Khorasan and collected in the suitable containers and moved to laboratory. The leaves were washed by distilled water, then dried at 60 °C, and were powered after crushing. In order to improve the adsorption capacity, 100 g of obtained powder ere mixed with a special volume of phosphoric acid with a mass ratio of 1:5, and this mixing alternatively continued for one hour to obtain a better contact of acid and saffron leaves. Then the modified powder was washed by distilled water for the removal of outputs. After removing the additional water, the obtained materials in a metal reactor in diameter of 50 mm and length of 100 mm were placed into an electric furnace for one hour in a temperature of 750 °C (16). This carbon was washed by distilled water until pH reached about 6.5 and then was dried in the oven for one hour at 100 °C, and then the two sieves with mesh sizes of 100 and 200 were used to separate the absorbent particles between 0.85 and 0.15 mm. Memert oven, which was made in Germany, and 1200 °C Electric Furnace were used to provide required temperatures.

Determining the characteristics of the adsorbent

In order to investigate the properties of the initial carbon and activated carbon prepared from saffron leaves, several experiments were conducted to determine the percentages of cellulose, lignin, volatile material, humidity, density, and iodine number (17). Morphological properties of activated carbon prepared from saffron leaves were studied by Scanning Electronic Microscope (SEM).

Adsorption tests

The effect of various parameters on the absorption process including the initial concentration of pollutants, the amount of adsorbent, contact time, and the optimum pH was studied. To determine the optimum pH, the value of 0.1 g activated carbon prepared from saffron leaves was poured in 100 ml of the synthetic solution with concentration of 30 ppm. Then experiment was conducted at contact times of 30, 45, 60, and 90 min at various pHs (3, 5, 7, and 9) separately, and then adsorbent was contacted with the metal ions of pollutant in the ambient temperature. Then the samples were filtered using filter paper in size of 0.45 μ and the remaining metal ions were measured by atomic absorption apparatus (YOUNGLTN ASS 8020 Model). The process was separately repeated for all the metal ions.

In the next step considering optimum pH determined, the amounts of adsorbent of activated carbon (0.3, 0.6, 1, and 1.3 g), initial concentration of pollutants (20, 30, 50, and 40 mg/L), and the contact time (30, 45, 60, and 90 min) were separately tested for individual pollutants. The removal efficiency was calculated using the following equation:

$$\text{Removal}(\%) = (C_0 - C_t)/C_0 \times 100 \quad (1)$$

Where C_0 is the initial concentration (mg/l), and C_t is metal ion concentration after each adsorption step.

Langmuir adsorption isotherm

The assumptions of Langmuir equation include the followings: a) maximum absorption occurs when the adsorbent surface is covered by a single molecular layer of soluble material. b) The absorption energy is fixed and identical at all the points. c) The molecules of adsorbed material cannot move in the adsorbent surface.

$$Q_0 = \frac{Q_{max} BC}{1 + BC} \quad (2)$$

Where C is the concentration of soluble material in the steady state mg/L.

Q_{max} is the maximum absorption capacity (mg/g) and B is Langmuir equation constant (L/mg).

Freundlich adsorption isotherm

The Freundlich equation expresses adsorption on a heterogeneous surface in terms of adsorption, and it can be expressed as follows:

$$Q = KC_f^{1/n} \quad (3)$$

Where n is Freundlich intensity parameter, and K is Freundlich capacity factor.

The Freundlich equation is an empirical one, and it is applicable for interpreting the experimental data (7).

With respect to determining optimal conditions in synthetic samples, the efficiency of metals removal from the sewage of Copper Complex Industries was studied to provide copper ion and the battery-making for electronics power (for cadmium and lead ions) was sampled. After determining the characteristics of sewage [pH, concentrations of copper, lead, cadmium ions, Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), and Total Dissolved Solids (TDS)], adsorption experiments were conducted by produced active carbon, and the removal efficiency was calculated (18).

Results

The results of studying the characteristics of activated carbon produced from saffron leaves are shown in the Table 1. The results showed that the density of activated carbon produced is lower than the other initial materials, and the amount of lignin is almost similar to the almond skin and walnut shell. The amount of cellulose is lower than pistachio bark and almond skin and is equal to walnut shell, but the value of the iodine number is less than these materials.

Figure 1 shows the results of the photos provided by the electron microscopy of coal and activated carbon prepared from saffron leaves.

In studying different pHs in the adsorption of metal ions, the optimum pH was 7. In the other pHs, the amount of adsorption was decreased.

According to efficiency results the absorption of copper and cadmium during contact time of 45 min was the highest with 83.34 % and 86.64 %, respectively. The lead adsorption was balanced at 90 min. At this time, the highest observed removal amount was 90.22%. Table 2 shows the results.

To investigate the amount of optimal absorbent, the adsorption experiments at optimum pH 7, the initial concentration of pollutants with 50 ppm, and the optimum contact times from the previous stage in the different values of adsorbents were tested and the results are shown in Table 3.

The results of adsorption experiments of heavy metals by activated carbon prepared from saffron leaves showed that in the optimum amount of adsorbent of 0.6 g and the equilibrium contact time of 45 min, the removal efficiency of cadmium ion is more than that of copper ion (82.52% against 75.56%). Removal efficiency of lead ion in the amount of adsorbent of 0.6 and 90 min was 95.55%. Increasing amount of adsorbent caused no significant change in absorption rate.

In the following research, to investigate the effect of adsorbent prepared on the real sample, samples of the effluents of two copper and battery-making industries were studied. The results related to study the raw wastewater quality of copper and battery-making industries are shown in the Table 4.

Testing optimized results on the actual samples related to the copper and cadmium ions showed that in optimum conditions (absorbent 0.6 g and the equilibrium contact time of 45 min) the removal efficiencies were equal to 68.12% and 78.95%, respectively. The experimental results related to the real lead sample showed that in the optimum contact time of 90 min and adsorbent amount

Table 1. The characteristics of activated carbon prepared from saffron leaves

Ingredients	Iodine number, mg/g	Percent of lignin	Percent cellulose	Density kg/m³	Percentage of total ash	Percentage of volatile material	Moisture (%)	References
Saffron leaves	501	30	34	160	7.84	1.05	5.4	This study
Almond Skin	830	27	39	470	1.5	-	-	Kaghzchi (17)
Walnut shell	806	33	34	457	6	-	-	Kaghzchi (17)
Pistachio bark	953	13	42	448	1.2	-	-	Kaghzchi (17)

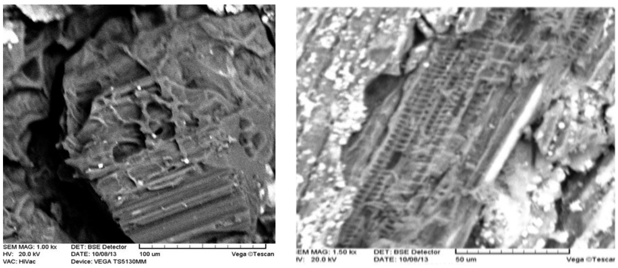

Figure 1. SEM image of charcoal (Left) prepared from saffron leaves without activating and activated carbon (Right).

of 0.6 g, the removal efficiency of this ion was 88.96%. To study the behavior of the adsorbent, Langmuir and Freundlich adsorption isotherms were plotted, and the results are shown in Figures 2 and 3. On the basis of the obtained correlation coefficients, it can be concluded that the adsorption of ions studied on prepared adsorbent given to the correlation coefficient higher than 0.99 follows Langmuir isotherm. Isothermal parameters have been shown in Table 5.

Table 2. The removal efficiency of the heavy metals (%) at different pHs and contact times (initial concentration 30 mg/L of pollutant and adsorbent dose 0.1 g).

Contact time(min)	Metal ion											
	Cd^{2+}				Cu^{2+}				Pb^{2+}			
	30	45	60	90	30	45	60	90	30	45	60	90
pH= 3	33.30	36.65	43.36	46.67	40.25	40.36	53.36	50.58	60.31	63.32	66.96	66.68
pH= 5	43.32	46.60	50.25	60.36	56.63	63.69	56.68	53.32	66.63	70.10	73.30	76.65
pH= 7	82.25	83.34	81.95	70.65	73.35	86.64	73.30	70.11	83.32	86.65	86.66	90.22
pH= 9	59.25	63.56	63.26	66.63	50.22	63.37	56.69	60.11	76.65	80.35	80.35	83.36

Table 3. The removal efficiency of heavy metals at different amounts of adsorbent (optimum contact time, optimum pH 7, initial concentration of 50 ppm)

Heavy metals	Optimum contact time (min)	The amount of adsorbent (g)	The final concentrations of heavy metals (ppm)	Absorption efficiency in synthetic solution (%)
Pb^{+2}	90	0.3	4.9	90.20
	90	0.6	2.22	95.55
	90	1	2.33	95.34
	90	1.3	2.27	95.45
Cu^{+2}	45	0.3	14.21	71.58
	45	0.6	12.22	75.56
	45	1	12.72	74.56
	45	1.3	12.37	75.25
Cd^{+2}	45	0.3	15.61	68.78
	45	0.6	8.75	82.50
	45	1	9.27	81.45
	45	1.3	9.21	81.58

Table 4. Characteristics of the sample of raw wastewater in the copper and battery-making industries

Type of industry	pH	Lead Concentration (ppm)	Cadmium concentration (ppm)	Copper concentration (ppm)	COD mg/L	BOD mg/L	TDS mg/L
Copper	4.21	1.4	0.4	5.88	185	60	260
Battery-making	5.9	53.56	20.32	2.8	350	180	1100

COD: Chemical Oxygen Demand; BOD: Biochemical Oxygen Demand; TDS: Total Dissolved Solids

Table 5. Adsorption isotherm parameters for Cu, Cd and Pb by activated carbon prepared from saffron leaves

Metal ion	Freundlich isotherms			Langmuir isotherm		
	R^2	1/n	K (mg/g)	R^2	B (L/g)	Q_{max} (mg/g)
Copper	0.96	0.476	1.74	0.99	1.90	62.96
Lead	0.96	0.476	1.50	0.99	1.44	79.6
Cadmium	0.98	0.44	1.77	0.99	16.66	68.75

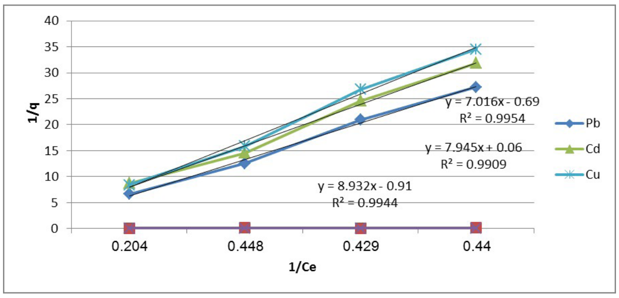

Figure 2. Langmuir adsorption isotherm of copper, cadmium, and lead

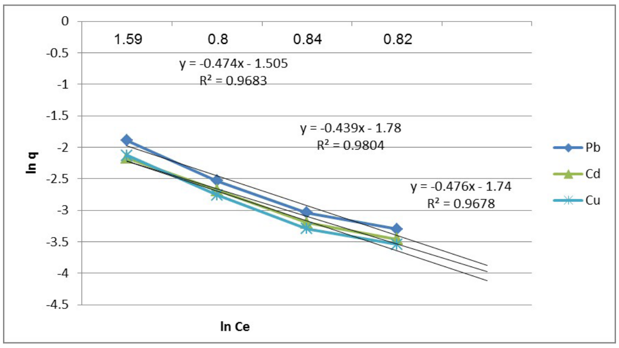

Figure 3. Freundlich adsorption isotherm of copper, cadmium, and lead

Discussion

The experimental results related to the prepared activated carbon shown that the way of activation stages have a significant impact on the quality of activated carbon prepared.

The effect of pH on adsorption efficiency of lead, cadmium, and copper by the activated carbon prepared from saffron leaves

pH is an important parameter in the adsorption process. In this study the effect of pH was tested by changing pH of solution in the range of 3-9 in concentration of 30 ppm from metal ions. Metal ions adsorption indicated more dependence from pH of solution (Table 2). Increasing solution pH increases the rate of adsorption because of electrostatic forces. This process continued until pH reached to 7 after that, increasing pH did not show positive effect on increasing absorption. This is because of reduced mobility of ions resulting from changing the properties and its load .The highest absorption was obtained in pH 7. In most of the studies, best pH for removal heavy metals was between 5-7. pH in terminal experiment was decreased by 1 value.

The results of this study were consistent with those of Amouei *et al*'s (19) research titled "Cadmium removal from canola plant", Madhava Rao *et al* (20) research in India about copper and cadmium removal from the aqueous solutions by activated carbon produced from Ceiba pentandra, and also and also with Venkatesan *et al* (21) about cadmium removal by activated carbon produced from Derris Indica wood. Nwabanne and Igbokwe (22) obtained some similar results related to the copper removal from aqueous solutions by activated carbon produced from various agricultural products in 2012.

The properties of the metal ion and also selectivity of the adsorbent are important factors in the adsorption process. In this study, the orders of ions absorption were observed Pb>Cd>Cu, respectively. This trend has also been the same in other studies (23). Pb ion occupies the adsorption places sooner than Cu and Cd due to its larger ionic radius. Some various trends have been also in the other studies (23).

The effect of adsorbent dose on the adsorption efficiency of copper, cadmium, and lead by activated carbon prepared from leaves of saffron

The dose of adsorbent is another important parameter in the adsorption process. The number of sites available for adsorption of metal ions depends on the amount of adsorbent used. In Table 3, it is observed that the adsorption of metal ions increases by increasing the amount of adsorbent to 0.6 g through holding the other parameters constant in the initial concentration of 50 mg/L. This increase is due to an increase in the surface area of adsorbent. At this value, the adsorbent surface is saturated with metal ions and after that the amount of adsorption reduces (24).

The results of this study regarding the effect of the adsorbent on the adsorption efficiency were consistent with Venkatesan *et al* research (21) related to the removal of cadmium by activated carbon produced from Derris Indica wood ,and also with the results of Madhava Rao *et al* research (20) on copper and cadmium removal from aqueous solutions by activated carbon prepared from Ceiba pentandra and were consistent with Amouei *et al* (19) studies about cadmium removal from water through activated carbon prepared from canola plant. In the study of Awwad and Salem about adsorption of copper and lead from aqueous solutions by modified adsorbent, the optimum amount of adsorbent was obtained 0.5 g (25).

The effect of contact time on adsorption efficiency of lead, cadmium, and copper by activated carbon produced from saffron leaves

The results related to ions adsorption of Cu (II) and Cd (II) on prepared adsorbent indicated that 45 min contact time was enough to reach equilibrium (Table 2). Adsorption does not change with an increase in contact time due to the saturation of adsorbent surface. Thus, this obtained contact time was used in other adsorption studies. The results indicated that this time is less than the contact time of these metal ions on some other adsorbents (25,26).

The results of this research on the effect of contact time were similar to those of Patnukao *et al* research (27) titled "Removal of metal ions (copper and lead) from synthetic wastewater by activated carbon produced from Eucalyptus Camaldulensis Dehnh shell related to copper metal". Also, Madhava Rao *et al* research (20) related to removal of copper and cadmium from aqueous solutions by activated carbon prepared from Povak tree skin was consistent with studies of Nale *et al* (13) on kinetics and equilibrium of lead adsorption from aqueous solutions on activated carbon produced from maize cobs (2012) with equilibrium contact time of 90 min. Additionally, these were associated with the other study by Arivoli *et al* (15) titled "Dynamic of copper adsorption by inexpensive activated carbon".

The isotherm study in the adsorption process of copper, lead, and cadmium from aqueous solutions by activated carbon prepared from saffron leaves

In the present study, the Freundlich and Langmuir isotherm models were studied to describe the relationship between the level of adsorption and the equilibrium concentration in liquid phase. This relationship and adsorption parameters for each model are shown in Figures 1, 2 and Table 5.

The amount of the correlation coefficient (R^2) shows that Langmuir model states a better relationship in this adsorption process. The maximum adsorption capacity for copper, lead, and cadmium ions is 62.92, 79.6, and 68.75 mg/g, respectively. These values represent high potential of the produced absorbent in the adsorption of metal ions studied from aqueous solutions because of its high adsorption capacity.

The results of this study on correspondence of adsorption isotherms are consistent with those of Saeedi *et al* (26) research related to cadmium removal by activated carbon produced from walnut and almond shell and Moreno-Piraján *et al* research (28) about the study of kinetic and removal of Mn, Ni, Cu, Fe ions from wastewater by activated carbon produced from coconut shell. Also, Nikazar and Noorbakhsh's (29) study on the removal of heavy metals Cd, Cr, and pb from aqueous solutions by activated carbon produced from agricultural wastewater (rice bran, wheat bran and straw) was consistent with these studies. Amouei *et al* (19) studied the Freundlich isotherm model related to cadmium adsorption using canola plant.

Comparing the absorption efficiency of heavy metals (copper, cadmium, and lead) from synthetic and real wastewater by activated carbon prepared from saffron leaves

Also in a research done by Elamin *et al* (30) about activated carbon produced from almond shell for the absorption of heavy metals of contaminated water, removal efficiencies of copper, and lead and cadmium in the synthetic model were 98%, 94%, and 49%, respectively and in the real model were 73%, 75%, and 20%, respectively.

The effect of initial concentration had an important effect on the absorption studies because removal efficiency decreased with rising initial concentration. Increasing the initial concentration of metal from 20 to 50 mg/L reduced the absorption efficiency of copper from 76.36% to 75.56% mg/L and those of lead absorption from 97.5% to 95.5% and cadmium absorption from 91.25% to 82.5%.

Conclusion

The results of experiments related to heavy metals absorption including copper, lead, and cadmium on activated carbon prepared from saffron leaves indicated a significant efficiency. The absorption level of Pb>Cd>Cu in the amount of adsorbent for all metals were 0.6 g, and the optimum contact times were 45, 45, and 90 min, respectively.

In these conditions the highest absorption efficiencies were observed 76.36%, 91.25%, and 97.5%, respectively. Also comparing the results of the study on synthetic and real models showed that the level of absorption in real wastewater is less than synthetic wastewater which is justifiable given analyzing the real wastewater of target industries and existence of other competing compounds, such as heavy metals and the other effective materials in absorption.

Due to the abundances of leaves and undesired use, saffron can be used as appropriate absorbent in the heavy metals removal from aqueous solutions.

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Ethical issues

We certify that all data collected during the study is presented in this manuscript and no data from the study has been or will be published separately.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

All authors participated equally in data acquisition, analysis and interpretation. All authors reviewed and approved the manuscript.

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