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DETOXIFICATION OF LEAD AND CADMIUM IN PHARMACEUTICAL EFFLUENT BY HOME-MADE FOOD WASTES

Parisa Ziarati[1,2*], Sepideh Tajik[3], Barbara Sawicka[4], Luis Cruz-Rodriguez[1,5,6], Viola Vambol[7,8], Sergij Vambol[9]

[1]ELIDAN genome SAS, France
[2]Department of Medicinal Chemistry, Tehran Medical Sciences, Azad University, Tehran, Iran
[3]Pars Arya LTD, Food Sciences Research Center, Tehran, Iran
[4]Department of Plant Production Technology and Commodities Science, University of Life Sciences in Lublin, Lublin, Poland
[5]CEO, ELIDAN America LLC, Fl, USA
[6]CEO, Elidan Dynamic Corp, Florida, USA
[7]Department of Environmental Engineering and Geodesy, University of Life Sciences in Lublin, Lublin, Poland
[8]Department of Applied Ecology and Nature Management, National University Yuri Kondratyuk Poltava Polytechnic, Poltava, Ukraine
[9]Department of Occupational and Environmental Safety, National Technical University Kharkiv Polytechnic Institute, Kharkiv, Ukraine

Abstract. The environmentally novel method of wastewater and effluent purification from heavy metal ions, utilizing home-made food waste bioadsorbent was studied. The initial concentration of heavy metals/metalloid such as Arsenic, Cadmium, Cobalt and Lead in the untreated effluents and treated by citrus peel (Grapefruit/orange and sour lemon peels) which treated by 2% coffee waste beans were analyzed. After especial times : 48, 72 hours and 1 and 2 weeks (by/ without stirring), final concentration of heavy metals in effluent samples were analyzed using Inductively Coupled Plasma Optical Emission Spectroscopy. Batch adsorption experiments were carried out to evaluate the effects of adsorbent-dosage, contact time and ionic strength of solution on the adsorption of heavy metals onto bio-adsorbent that was simply gathered from household-food waste at room temperature and then dried at 80 C^0 and grind to specify sizes. The results revealed that the adsorbent content and percentage in the studied effluent solution played a major role in adsorption. The potential of adsorptive amount of Cd^{2+} in presence of bio-mass after 48 hours removed 98.9% and 100% removed the initial contents of Lead after 72 hours by stirring the waste water which shows efficiently, and environmentally method of purification of aqueous. Current study indicates that home-made wastes which are commercially available can be considered as an efficient adsorbent for removing toxic heavy metals in this novel method.

Keywords: *Bio-adsorbent, environmentally method, waste water treatment, Arsenic, Cadmium.*

[*]***Corresponding Author:*** *Parisa Ziarati, ELIDAN genome SAS, France, 1 avenue du Lycée, 77130 Montereau Fault Yonne, France; Department of Medicinal Chemistry, Tehran Medical Sciences, Azad University, Tehran, Iran, e-mail: Ziarati.p@iaups.ac.ir parziarati@gmail.com*

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1. Introduction

Shortage of water is a main concern of many countries in the world. The presence of chemical and microbial pollutants in natural waters is a severe threat to the biosphere and for the health of the public in particular (Ziarati *et al*., 2022; Zahirnejad *et al*., 2017; Mahmoud *et al*., 2020). Pharmaceuticals (PhAs) are the most common pollutants in the water because 30–90% of them remain in our bodies without decomposition as well as enter the environment after excretion from humans or animals (Mahmoud *et al*., 2020; Rivera-Utrilla *et al*., 2013). The study of PhAs sources is always essential as a first step for pharmaceutical wastewater management because various substances from these sources becomecomponents of the chemical composition of the environment. PhAs sources mean the origin of the micropollutant which exists in greater concentrations than the natural ones due to human activities. In this case, their continuous appearance in the environment affects both the health of organisms and the environment (Ziarati *et al*., 2020a; Sobhani & Ziarati, 2017). Both non-prescription and prescription drugs, consisting of assorted compositions, are used in the evasion or cure of diseases (Yuan et al., 2009; Benotti *et al*., 2009). The physicochemical and biological properties of pharmaceuticals micropollutants make their biological power different when they are dispersed in the surroundings (Arabian *et al*., 2020; Fatta-Kassinos *et al.*, 2011). According to World Health Organization, PhAs concentrations are typically, 0.1 μg L–1 in groundwater, surface waters, and partially treated water while they are ,0.05 μg L–1 in treated water (Ziarati *et al*., 2019a; Ziarati & Mirmohammad Makki, 2017). From these investigations, numerous water sources receiving effluents of wastewater contain pharmaceuticals at nominal concentrations.

There are approximately 6832 documents about pharmaceutical wastewater in the Scopus database. We focus on the published documents from 2000 to 2020 for the graphical representation of these documents. The continuous increment of the published documents in the last 20 years displays the top countries that have a contribution to more than 100 publications in pharmaceutical wastewater up to 2020. The top country is the USA with 1140 documents, and after that is China with 1031 documents. Spain and Germany are the leading European countries regarding pharmaceutical issues.

The positive effect observed in the proposed method can be explained by the following features of the selected adsorbent.

It was found that the main factors that affect the degree of adsorption are the concentration of metal ions in contaminated water, the concentration of waste of coffee and its ratio of citrus peels (Ziarati *et al*., 2019b; Tajik *et al.*, 2020).

Advantages of Using Agricultural/ Food Waste

- Agricultural wastes can be a valuable resource for improving food security. If left untreated, maintained or disposed of properly, agricultural wastes are likely to contaminate the environment and harm human health (Vambol *et al*., 2021).
- This calls for increased public awareness of the benefits and potential dangers of agricultural waste, especially in developing countries. Also, the use of new methods in solving the agricultural waste problem can greatly help reduce pollution and improve the production cycle (Ziarati *et al*., 2022).
- Food and agricultural losses and waste have been approached by two different angles: either from a waste perspective, with the associated environmental concerns, or from a food perspective, with the associated food security standards (Ziarati *et al.*, 2020b; Khan *et al.*, 2020).

- Food waste is produced all along food life cycle: agricultural production, industrial manufacturing, processing and distribution. They have high-value components such as phytochemicals, proteins, flavor compounds, polysaccharides and fibers that can be re-used as nutraceuticals and functional ingredients (Sawick *et al.* 2019).

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The proposed utility model relates to technologies for water treatment, water treatment and sewage treatment, namely adsorption removal of metal ions from polluted natural and industrial wastewater and can be used for the preparation of drinking water, water for agricultural purposes, chemical, petroleum, food processing for enhancing biological and physical-chemical parameters of drinking water, and for organizing a closed cycle.

Known methods of treating wastewater from metal ions, which include the treatment of water with sorbents, based on inorganic and organic materials. Among various wastewater treatment techniques, ***adsorption*** is the most common technique to remove heavy metal in wastewater (Ziarati *et al*., 2019).

The main disadvantages of inorganic sorbents are the need to extract adsorbed metal ions from them. This requires additional technological equipment and reagents. The use of specially synthesized adsorbents (eg, zeolites, silica) is more efficient but more expensive. At the same time, the use of natural adsorbents (bentonites, saponites) is related to the variability of their adsorption capacity ((Ziarati *et al*., 2019; Ziarati & Mirmohammad Makki, 2017).

The basis of the proposed utility model is the task of creating a method of extracting heavy metal ions from contaminated waters which:

- provides a high degree of purification from heavy metal ions;

The extraordinary impact of Food & Agro-Industrial wastes to a new environment methodology:

- Increasing crops production with reduced costs
- has high performance;
- does not introduce additional contaminants;
- Easily removed from purified water;
- Easily recyclable.

The food waste such as fruit-processing industry generates enormous solid waste in the form of pomace, peels, kernels, and pulp following the industrial processing of fruit juices. The fruit residues are not only by products of industry, but are also found in the household kitchens, restaurant trash bins, hotel, juice, local shops and also contribute significantly to the municipal solid waste. One of these products is apple pomace that is alternative by-product obtained from apple juice industries in huge amounts and by fruit juice shops in other scale annually. Chemical composition of final pomace is linked to the morphology of original feed stock and the extraction technique used. According to NRC 2001, apple pomace (AP) in some regions is very low in protein (contains only 6.4% protein on DM basis) and it also serves as a useful energy source for ruminants. Another important abundant nutritive fruit waste is grapes that are consumed both as fresh and as processed products such as wine, jam, juice, jelly, grape seed extract, dried grapes, vinegar and grape seed oil. The cultivation of grapes is widely spread around the world. Ziarati et al. (2017) worked upon protein, mineral contents of pulps, seeds and peels of 5 different grape pomaces (Red Rishbaba, Shahroudi, Shoush Aroos, Yaghouti and Askari) in Iran based on dry weight (Sawick *et al.,* 2019; Gholizadeh & Ziarati, 2016). Comparing with Dietary Reference Intakes (DRI) , the iron content in grape pomace found in current study (46.02 mg/100 g) supplies the adult daily requirements for iron (8 mg/day for men and 8 to 18 mg/day for women). On the whole, supplement ingredients may contain traces, minerals, vitamins, herbs or other herbal medicine or herbal plants, amino acids, enzymes, organ tissues, gland extracts, or other dietary substances, which are mainly derived from food and agricultural waste materials. In the case of fruit and vegetables, there is a large amount of antioxidant compounds, usually in skins, grains or seeds, i.e. parts that are removed during processing and become waste (Sawick *et al.,* 2019). It could be considered that currently recovering bioactive compounds from fruit waste that is beneficial for health is a research trend not only to reduce the burden of waste, but also to meet the society's intense demand for phenolic compounds that are thought to have a protective effect against chronic diseases (Sawick *et al.*, 2019).

The use of organic sorbents or biosorbents can more effectively purify natural or waste water from heavy metal ions, but they require additional technological solutions for the manufacture or modification of such sorbents, methods for their introduction and removal from water, regeneration or disposal of such sorbents.

The use of organic wastes from food or processing industries as sorbents makes it possible to combine the solution of the problem of disposal of such wastes with the problem of purification of wastewater contaminated with heavy metals (Gholizadeh & Ziarati, 2016; Razafsha & Ziarati, 2016; Ziarati *et al.*, 2016; Motaghi & Ziarati, 2016; Ziarati *et al*., 2019a,b).

2. Material and Methods

Effluents from 4 in pharmaceutical educational Laboratories in Pharmacy Faculty , in Tehran medical Sciences , Azad University as well as Nutrition and Food Sciences Research Center (Effluent 1&2), Toxicology (Effluent 3&4), Analytical chemistry (Effluent 5) were collected in 2020 and used in this investigation. Effluent 1 and 2 were from the same laboratory but were collected on separate occasions with a 3 week time interval. Although Effluent 1 and Effluent 2 come from the same effluents' treatment plant, they were studied and considered as 2 different waste water samples due to the variability of their characteristics, heavy metal contamination and pHs. The characteristic is attributed to the significant experiments which occurred following the first sampling event. After collection, the wastewater effluent was instantly transported to the main research laboratory for analysis. Physico-chemical parameters such as Total Solids, Total hardness, pH, Electrical Conductivity, Total Dissolved Solids, Chloride, Sulphate, Dissolved oxygen, Calcium, Sodium, Cadmium, Lead, Zinc, Copper, Chrome, Manganese, Iron and Potassium were analyzed as per the standard methods (Shahsavan-Davoudi & Ziarati, 2020; Yazdanparast *et al*., 2014; Manshadi *et al*., 2013).

The initial concentration of heavy metals/metalloid such as Lead, Cadmium , Cobalt and Lead in the untreated effluents and treated by citrus peel (80% sour lemon peel + 10% orange + 10% grapefruit peels) which treated by 2% coffee waste beans were analyzed. After especial times: 48, 72 hours and 1 and 2 weeks (by/ without stirring) final concentration of heavy metals in effluent samples were analyzed using ICP-OES system and standard solutions. All required precautions were taken to avoid any possible contamination of the sample as per the AOAC guidelines (Ziarati *et al*., 2013; AOAC, 2000; Lahiji *et a*l., 2016). The efficiency of *citrus peels treated by coffee waste bean* in accumulating heavy metals was investigated.

Contaminated water is contacted with the bio-adsorbent in a flow reactor or in a stirred reactor, which is prepared as a mixture of spent coffee waste and citrus peels in a certain proportion. The contact time is determined based on the initial concentration of metal ions and the desired final concentration. At the end of the adsorption process, the adsorbent is separated on the filter and sent for disposal.

Citrus Peel Sampling Method

45 kilograms Tangerine, Orange and Grapefruit were purchased from different recognized Tehran market. The fruits were washed extensively under tap water to remove adhering dirt, rinsed with de-ionized water, cut into small spices by small clean cutter and naturally dried in sunlight. Dried peel was grounded using a clean electric mixer, sieved through (Retsch GmbH & CoKG, Germany) mesh size (250 µm) to retain fine particles. To reduce enzymatic browning, the peels were then dipped in a 1% (w/v) citric acid solution for 10 min, drained and dried in an oven at 150°C for 8 hours and homogenized in a blender to utilize in adsorption experiments.

Coffee waste Sampling Method

Coffee bean waste is collected from remarkable local coffee shops in Tehran in June of 2020, and dried up in an oven at 50°C for 48 hours. The studied Coffee waste samples during the Bio-adsorption investigation for removing heavy metals were at natural state with no chemical or thermal treatment.

Adsorbent particle size

The crusher sieve was used for the reduction of particle size of the dried Tangerine ,Orange and Grapefruit peel samples and for particle size distribution, respectively. The sieves were mechanically vibrated for 15 minutes which was sufficient for separation to take place. The screens were subjected to weighing balance before and after the vibration to get the mass and size of the banana peel particles retained on each sieve. The particle size range used in this study was 0.15 mm to 1.5 mm.

Biochemical oxygen demand (BOD)

For Biological Oxygen Demand, 15 samples were processed after collection immediately for the determination of initial oxygen and incubated at 20°C for 5 days for the determination of BOD5 (Ziarati *et al*., 2022; Lahiji *et a*l., 2016; FDA, 2013).

Preparing the device

The settings of the device for measuring lead and cadmium elements were made based on the instructions provided by the device manufacturer, and then the digested samples were injected into the ICP device, and finally the concentration of heavy metals was read in mg/kg (FDA, 2013; Mokhtarzadeh *et al*., 2021; Keyseruxskaya, 2023; Alizade, 2022; Ziarati *et al*., 2020c).

Statistical methods

All measurements were carried out in triplicate. Data were expressed as the mean ±SE (standard error). Data were analyzed using one-way analysis of variance (ANOVA) and SPSS Software v.12 (SPSS INC., Chicago, IL, USA). Duncan's multiple range tests was used to assess differences in Lead and Cadmium contents in Pharmaceutical effluents and waste water samples, different dose of bio-adsorbent, different time and pH of the samples. The values reported here are means of five values. Each sample data was the mean of 5 subsamples. A *p* value of 0.05 or less was considered as statistically significant.

3. Results and Discussion

As compared to BOD, COD was very high which is normal for effluent of such pharmaceutical laboratories. The minimum and maximum values ranged between 1983–3219 and mean of 2603 mg/L and the average values ranged between 1250 – 7300 and mean of 2417 mg/L for the studied effluent.

Table 1. Characteristics of studied Pharmaceutical Effluent and waste waters

Parameters	Concentration Range	Average
pH	0.18 – 6.8	2.5
BOD5 at 208C (mg/L)	1983–3219	2603
COD (mg/L): chemical oxygen demand	1250 – 7300	2417
TSS (mg/L): total suspended solids	31 – 63	47
Total alkalinity as $CaCO_3$(mg/L)	70 – 1200	610
TVA (mg/L)	85 – 2100	784
Lead (mg/L)	4.75– 26.54	20.00
Tin (mg/L)	0.9 – 11.4	6.5
Cadmium (mg/L)	2.5 – 13.33	10.00
Mercury (mg/L)	0.09 – 0.28	0.16
Zinc (mg/L)	10.42 -54.82	17.04
Cobalt (mg/L)	1.99– 16.23	7.18
Chromium (mg/L)	4.92 – 19.65	7.22
Chloride (mg/L)	500 – 1360	894
Sulfide (mg/L)	2-18	8
Nitrate (mg/L)	380-4200	1400

The results of determination of Cadmium concentrations in the samples of Pharmaceutical effluents and waste water treated by bio-adsorbents were accomplished by ICP-OES in and were reported as mean concentration in mg / L in Table 2, after 48 and 72 hours respectively. All concentrations were expressed as (mg/L ± SE). The amount obtained for each sample is the average of 3 repetitions of the measurement.

Table 2. Treating Cadmium in wastewater and Pharmaceutical effluents by Bio-adsorbent

Cadmium content(mg/L ±SE) in waste water	**48h**	**72h**
0.2% Bio-adsorbent	b4.125±0.102	b3.714±0.006
0.3% bio-absorbent	c3.006±0.211	b3.487±0.024
0.4% bio-absorbent	c3.002±0.124	c2.218±0.32
0.5% bio-absorbent	c2.984±0.112	c2.906±0.021
0.6% bio-absorbent	d2.663±0.092	d2.843±0.042
1% bio-absorbent	e1.009±0.083	e1.608±0.031
2% bio-absorbent	e1.583±0.088	e1.428±0.052
untreaad sample	a10.002±0.011	a10.002±0.11

*SE= Standard Error
**Different letters in the columns indicate statistically significant difference (P<0.05)

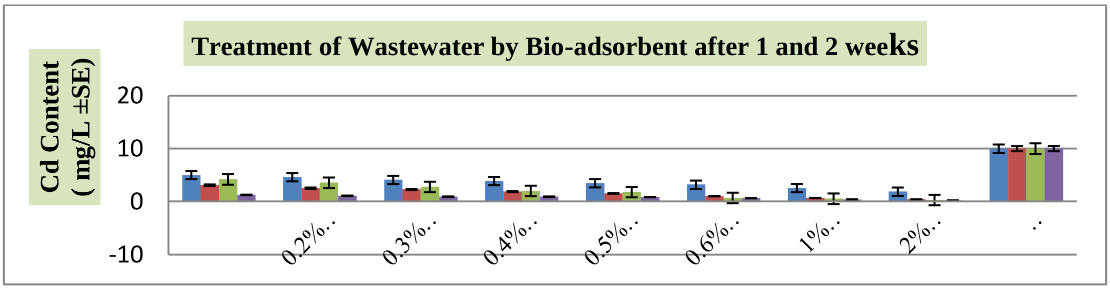

Figure 1. Effect of contact time on the removal of cadmium (initial Cd concentration=10.002 mg/l, bio-adsorbent doses=0.2,0.3,0.4,0.5,0.6,1 and 2 mg Grapefruit/orange and sour lemon peels treated by 2% coffee waste beans /100 ml of *waste water* respectively, temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.2

Results in figure 1 in presence of 2% coffee waste + 10% and 8% Grapefruit-Orange Peel+ 90% sour lemon peels showed significant difference in Cadmium up -taking by bio-adsorbent after 2 weeks with and without stirring. The data showed that accomplishing of agitation speed= 200 rpm during 14 days (2 weeks) as well as without stirred studies have a potential of taking up Cadmium by increased significantly in statistic state (p <0.001) and Cadmium content in untreated sate 10.01 mg/Lit after 2 weeks decreased to 0.0.008 and 0.001 mg/Lit respectively which prove that biomass of adsorbent can reduce *98.9%* of heavy metal by utilizing of 1% and 2% of biomass.

The results of determination of mean level of Lead (Pb) in the samples of Pharmaceutical effluents and waste water treated by bio-adsorbents were accomplished by ICP-OES in and were reported as mean concentration in mg / L in Table 3, after 48 and 72 hours respectively. All concentrations were expressed as (mg/L ± SE). The amount obtained for each sample is the average of 3 repetitions of the measurement.

Table 3. Treating Lead in wastewater and Pharmaceutical effluents by Bio-adsorbent

Lead content(mg/L ±SE) in waste water	**48h**	**72h**
0.1% bio-adsorbent	[b]15.562±0.024	[b]14.889±0.003
0.2% bio-adsorbent	[b]15.249±0.015	[b]13.729±0.003
0.3% bio-adsorbent	[b]14.021±0.014	[c]12.501±0.008
0.4% bio-adsorbent	[c]12.984±0.009	[c]11.333±0.004
0.5% bio-adsorbent	[c]11886±0.008	[c]10.192±0.006
0.6% bio-adsorbent	[c]10.723±0.006	[d]8.004±0.004
1% bio-adsorbent	[c]10.009±0.006	[e]6.886±0.005
2% bio-adsorbent	[c]9.556±0.006	[e]4.281±0.003
untreaad sample	[a]20.004±0.003	[a]20.004±0.003

[*]SE= Standard Error
[**]Different letters in the columns indicate statistically significant difference (P<0.05)

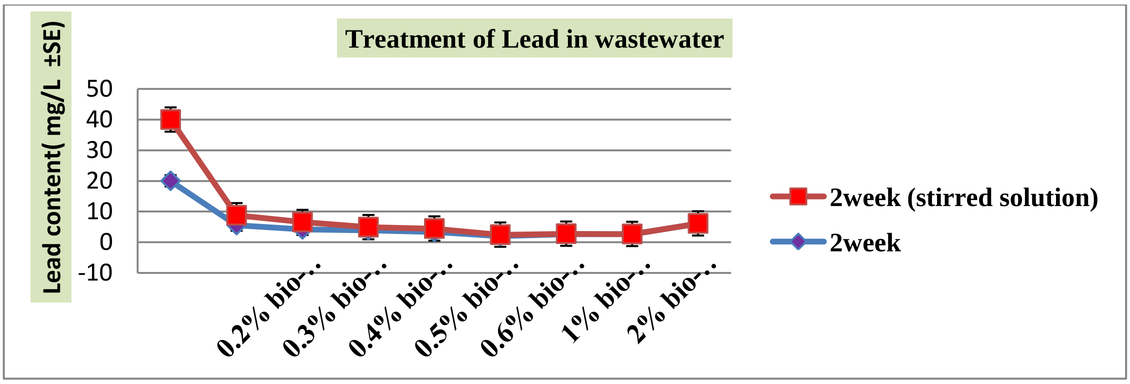

Figure 2. Effect of contact time on the removal of Pb (initial lead concentration=20.004 mg/l, bio-adsorbent doses=0.2,0.3,0.4,0.5,0.6,1 and 2 mg Grapefruit/orange and sour lemon peels treated by 2% coffee waste beans /100 ml of *waste water* respectively, temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.2

The results of the increasing adsorbent to 0.5% in figure 2, showed significant differences in Lead up -taking by bio-adsorbent after 2 weeks. But despite of other previous concentration it can be observed that passing time up to 1 week has no significant difference by 72 hours ($p \geq 0.05$) and also 7 days (1 week) with stirring by 2 weeks without stirring too. The data showed that accomplishing of agitation speed= 200 rpm during 1 week and 14 days (2 weeks) with stirred have significant differentiate (p <0.001). Lead content in untreated sate 20.004 mg/Lit after 2 weeks decreased to 0.001 mg/Lit (without stirring process) which has significance difference by 0.2% concentration of biomass. This content of adsorbent can reduce 20.004 mg/Lit Pb to 0.000 mg/Lit (ND= not detectable) in pharmaceutical effluent which means removal of 100 % of heavy metal.

When the ratio of Citrus peels / Coffee waste is less than 0.1, the addition of citrus tangeretin derivative, 5-acetyl-6,7,8,4 ' –tetramethylnortangeretin is impractical, and at a ratio of more than 0.6 the degree of adsorption increases less noticeably, and the concentration of adsorbed metals in the spent adsorbent decreases, so more ratio is technologically impractical. Thus, the proposed method of purification of water from heavy metal ions is as follows:

According to our research, the dependence of the adsorption capacity on the concentration of water in the amount of adsorbent for metals such as Cadmium and Lead are different from the capacity for such as Cobalt, Chrome and Nickel. For the former, the greatest effect is the concentration of coffee waste in solution.

It was found that the minimum concentration of citrus peel treated by coffee waste in water for its complete purification from metal ions with an initial concentration of 100 mg/l in 48 hours should be 2%. With smaller values, the percentage of removing heavy metals would be decreased.

4. Conclusion

Results showed that low-cost and available bio- adsorbents can be productively, used for the removal of heavy metals: lead and Cadmium with a high concentrations. It also was found that the percentage removal of heavy metals was dependent on the dose and particle size of low cost adsorbent concentration. The contact time necessary for maximum adsorption was found to be 48 hours. The optimum pH range for Cadmium adsorption was in Acidic range and best results obtained in 4.1-5.3 which the best results comes from pH= 4.2 in current study. The most advantage of this method is the applied adsorbent used in the experimental work is a home-made waste and also commercially available, and they are the waste of fruit that could be collected in every house with no cost in fact. We highly recommend that to study the mechanism, which is necessary to have the exact information about the cell wall structure of the biomass as well as the solution chemistry.

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