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Research Article

Green Method for Cadmium Removal from Pharmaceutical Effluent Laboratories by Grapefruit Peel

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Abstract Progesterone and estradiol are arguably the most important secretory products of ovarian steroid production. Adsorption is a practicable separation method for purification or bulk separation with surface characteristics and pore structures of as the main properties in determining its rate of equilibrium. Heavy metals are natural components of the Earth's crust. They are non-biodegradable and are able to reach our body system through drinking water, air and intake of food. The sustainable removal of toxic heavy metals from pharmaceutical wastewaters; effluent have become the major challenges to scientists and engineers despite there are numerous treatment technologies. This is because most of the explored technologies are too expensive and also have significant disposal challenges. The Bio-adsorption of Cadmium metal ions (Cd+2) have been studied to investigate the effects of controlling parameters such as particle size, doses, and initial concentration of initial toxic heavy metals , contact time, pH and selectivity of the peel. The purpose of current study is to investigate adsorption characteristic of Grapefruit peel for removal of cadmium ions (Cd2+) from aqueous and waste water solution of pharmaceutical effluent academic educational and research centers laboratories in Tehran Medical Sciences, Islamic Azad University in Tehran, Iran. Batch adsorption experiments were carried out to evaluate the effects of pH value of solution, adsorbent particle size, adsorbent dosage, and ionic strength of solution on the adsorption of Cd2+ onto bio-adsorbent that was simply gathered from fruit juice factories or home-made wastes at room temperature and then dried at 700 C and grind to specified sizes. The results revealed that the adsorbent content and percentage in the studied Effluent solution played a major role in adsorption. The adsorptive amount of Cd2+ onto the bio-adsorbents decreased with increasing the adsorbent particle size, and ionic strength, while it increased with increasing the initial pH value of solution and temperature. Cd2+ was removed efficiently and quickly from aqueous solutions by the bio-adsorbents which treated by tartaric acid 1% with a maximum capacity and remove all the 10.01 mg/Lit cadmium in waste water .The adsorption process was well described by the pseudo-second-order kinetic model with the correlation coefficients greater than 0.994. The adsorption isotherm could be well fitted by the Langmuir model. The thermodynamic studies showed that the adsorption of Cd2+ onto the treated bio-adsorbent was a spontaneous and exothermic process. The results indicate that Grapefruit peel can be considered as an efficient adsorbent even when it is not treated by tartaric acid.

Keywords: Cadmium, Pharmaceutical effluent, Bio-adsorbent, Waste water, Remediation.

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Introduction

Although as trace elements some heavy metals are required to maintain the human body metabolism. But, poisoning naturally occurs at higher concentrations of such heavy metals. Heavy metals are dangerous and toxic in nature because they tend to bio-accumulation living cells where the concentration of a specific chemical or a heavy metal increases the non-degradable toxic in nature [1-7]. The use

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of bio-adsorbents for removal of toxic heavy metals from waste offers a relatively low-cost method with potential for metal recovery [8-12]. The metal sequestering ability of adsorption technology offers great advantages as it does not produce sledges requiring further disposal, further it is highly selective, more efficient, easy to operate, can handle large volumes of waste containing low metal concentrations. Both commercial adsorbents and bio adsorbents are used for the removal of heavy metals from wastewater, with high removal capacity [13-18]. However, in the absence of efficient methods of chemical-physical or high-cost of industrial waste treatment necessitates innovative methods using plant wastes as alternative sorbent materials due to greater availability on an operational scale and cost effective to use[19-22]. The increase in environmental awareness and concern led to a search for new techniques capable of inexpensive treatment of polluted wastewaters with metals. The search for new technologies involving the removal of toxic metals from wastewaters has directed attention to bio-sorption, based on binding capacities of various biological materials 17, 18].

Adsorption has come out as effective, low-cost, environmentally and ecofriendly treatment technique [23-25]. It is a process potent enough to fulfill water reuse obligation and high effluent standards in the industries [18]. Generally, adsorption is mainly based on attractive interfaces between surfaces and interfaces which the group is being absorbed [7].

The removal or decreasing of toxic and heavy metals by using low cost agricultural or food waste adsorbent found to be more supportive in prolonged designations as there are considerable components existing locally and profusely such as natural materials, agricultural wastes or Food industrial by-products which can be applied as environmentally and economic adsorbents [7- 8, 26-27].

On the point of a toxic trace element cadmium is highly leading to environmental and occupational hazard. Cadmium is widely used in many industries such as battery and industrial workers dealing with this heavy metal are at high risk of cadmium poisoning through ingestion and inhalation which may damage digestive tract, lungs, liver and urethra and if severe, it can lead to coma and even death [2 , 28-30]. Chronic exposure to cadmium suppresses norepinephrine, serotonin and acetylcholine levels and its inhalation triggers development of the pulmonary adenocarcinoma [31]. Along with direct contact with cadmium would generate prostatic lesions as well as adenocarcinoma.There is evidence indicating that cadmium can damage. However, cadmium is less mutagenic than other metals [32-33].

In many countries such as Austria, Australia, Finland, France, United States of America, Germany, Greece, Italy, United Kingdom, South Africa, Bangladesh, Inner Mongolia, China, Taiwan, India, Iran, Cambodia, Nepal, Pakistan, Thailand, Vietnam, Alaska, Argentina, Chile and Mexico Cadmium concentrations beyond acceptable standards have been detected [2-3, 8-20]. Soil and water contamination can be cleaned up by some conventional methods such as immobilization, verification, soil washing/flushing, precipitation, membrane filtration, adsorption, ion exchange, permeable reactive barriers biological treatment, thermal processes, excavation and disposal process, chemical processes, and phytoremediation [34-36]. Unfortunately most of these conventional and classic methods are found very high-priced. As Metal bio-adsorption is the removal of metal ions by inactive, nonliving biomass due to highly attractive forces present between them. Particularly, it is due to the presence of certain functional groups, such as amine, carboxyl, hydroxyl, phosphate, sulfhydryl etc., on the cell wall of the biomass. In current study, the main goal is finding suitable green method for Cadmium removal from pharmaceutical effluent laboratories by Grapefruit Peel (GP), as a cheap citrus waste in a batch system by considering the effects of various parameters like initial concentration, pH, contact time, agitation speed and bio-adsorbent dose.

Materials and Methods

Material preparation

Grapefruit were purchased from recognized local markets in Tehran in January and February of 2020. The pith on the peel was removed and the peels were cut into small pieces (1-1.5 cm), washed with deionized water to remove external dirt and dried in an oven at 70°C for 48 hours. Thereafter, the peels were subjected to a crusher to reduce particle sizes and then dried again for 4 hours at 85°C . The Grapefruit peels used during the Bio-adsorption study were at natural state with no chemical or thermal treatment. The surface morphology and functional groups of the Grapefruit peels were studied before and after adsorption by scanning electron microscope (SEM) and Fourier-Transform infrared (FTIR) spectroscopy, respectively.

Chemicals

All the chemicals used in this work were of analytical grade and actual Pharmaceutical effluent and waste water samples were collected in 5 groups of 5L bottles from Chemical Laboratories (Inorganic Chemistry, Analytical Chemistry, General Chemistry, Physicochemical Chemistry) from Pharmaceutical Faculty and also Nutrition and Food Sciences Research Center in Tehran Medical Sciences, Islamic Azad University in Tehran, Iran. After collection, the wastewater effluent was instantly transported to the main research laboratory for analysis. Physico-chemical parameters such as pH, Dissolved oxygen, Total Solids, Total hardness, Electrical Conductivity, Total Dissolved Solids, Chloride, Sulphate, Calcium, Sodium, Cadmium, Lead, Nickel, Zinc, Copper, Chrome, Manganese ,Iron and Potassium were analyzed as per the standard methods [17-18,37].

The synthetic Sigma-Aldrich stock solution of Cd^{+2} (1000mg/L) were used for making standard solutions. The desired concentrations of six standard solutions for the metal were prepared by successive dilutions of the stock solution.

Adsorbent particle size

The crusher sieve was used for the reduction of particle size of the dried 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 Grapefruit peel particles retained on each sieve. The particle size range used in this study was 0.15 mm to 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 [17,19-21] .All the experiments were carried out in triplet and the relative standard deviation was less than 2%. In general the sorption consisted of 0.1, 0.2, 03, 0.4, 0.5, 0.6, 1 and 2 percent for the adsorbent dose in one Liter of Pharmaceutical effluent and waste water samples were studied while for 0, 48 and 72 hours, 1 and 2 week(s) without and stirring and for other samples at an agitation rate of 200 rpm with an adsorbent time 1 week and 2 weeks at room temperature (25 ± 3).To study the effect of pH on sorption, the pH of the metal ion solution was adjusted to values in the range of (2–10) prior to the experiment. The Langmuir isotherms were obtained by equilibrating metal ion solutions of different adsorbent doses (1–20) mg/l with different times (0–2 weeks) at equilibrium pH and rpm with an initial metal concentration of 10 mg/l at room temperature.

Statistical Analysis

The values reported in current study are means of three values. Data were tested at different significant levels using student t-test to measure the variations between the contaminations in wastewater and the dose of bio-adsorbent and contact time parameters before and after treated by Grapefruit bio-adsorbents in 2 forms of treated by tartaric acid and not treated . One way analysis of variance (ANOVA) was used for data analysis to measure the variations of metal concentrations using SPSS 22.0 software (SPSS Inc, IBM, Chicago, IL).

Results

The mean content of Cadmium in presence of grapefruit peel samples collected from Tehran market in Iran are shown in figures 1–5 in applied by different percentage and concentrations of bio-adsorbent. The samples were analyzed by wet digestion method and standardized international protocols [37] were followed for the preparation of material and analysis of Cadmium contents and analyzed by Atomic Absorption Spectrophotometer in Research Laboratory of Nutrition and Food Sciences Research Center, in Pharmaceutical faculty, Tehran Medical Sciences. The data obtained from chemical analyses, mean values were calculated and are given in the table 1. The results of characteristics of pharmaceutical effluent from all samples gathering from all laboratories in December 2019 were analyzed are showed in table 1.

The effect of the concentrations of adsorbent on the removal of Cd +2 ions by Grapefruit peel is depicted in figures 1-6 , for varied adsorbent doses of 0.1%, 0.2%,0.3%, 0.4%, 0.5%, 0.6% , 1 and 2% w/v . In the next part of this research for Cadmium removal using grapefruit peel treated by Tartaric Acid 1% and results revealed the potentially increased from 74.38. % to 98.66% which means that treated by chelation agents with the increase of the potential of bio-absorbent, removal ability of heavy metals by Grapefruit peel without treatment by Tartaric acid bio-adsorbents varied from 44.23% to 97.23% depending on different concentrations of bio-adsorbents and also agitation speed and the best results obtain in temperature=25 ± 1 ºC, agitation speed= 200 rpm in pH = 4.5-5.6.

Table 1. Characteristics of studied Pharmaceutical Effluent

Parameters	Concentration Range	Average
pH	0.18 – 6.8	2.5
BOD5 at 208C (mg/L)	1965–3199	2573
COD (mg/L): chemical oxygen demand	1200 – 7200	2384
TSS (mg/L): total suspended solids	30 – 62	45
Total alkalinity as $CaCO_3$(mg/L)	70 – 1200	610
TVA (mg/L)	85 – 2100	784
Lead (mg/L)	1.12 – 15.28	9.86
Tin (mg/L)	0.9 – 11.4	6.5
Cadmium (mg/L)	3.5 – 10.01	5.3
Mercury (mg/L)	0.11 – 0.38	0.19
Zinc (mg/L)	9.19 -49.87	16.51
Cobalt (mg/L)	50-230	118.1
Chromium (mg/L)	5.89 – 10.54	6.09
Chloride (mg/L)	500 – 1360	894
Sulfide (mg/L)	2-18	8
Nitrate (mg/L)	350-1980	1100

As compared to BOD, COD was very high which is normal for effluent of such pharmaceutical laboratories. The minimum and maximum values ranged between 1965–3199 and mean of 2573 mg/L and the average values ranged between 1200 – 7200 and mean of 2384 mg/L for the studied effluent.

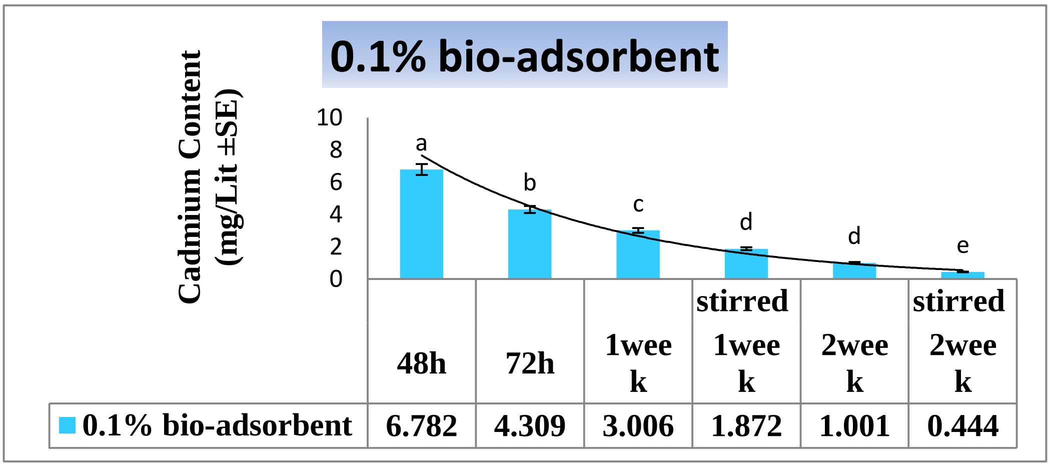

Figure 1. Effect of contact time on the removal of cadmium (initial Cd concentration=10.001 mg/l, bio-adsorbent dose=0.1 mg Grapefruit peel /100 ml of *waste water*, temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.6

Results in figure 1 showed significant difference in Cadmium up -taking by bio-adsorbent after 48 and 72 hours and also 7 days (1 week) without stirring. The data showed that accomplishing of agitation speed= 200 rpm during 1 week and 14 days (2 weeks) without stirred have no significant differentiate ($p \geq 0.05$) , but the potential of taking up Cadmium was increased significantly between 2 week with agitation speed by 2 weeks in statistic state ($p < 0.001$). Moreover, time factor of putting adsorbent in contaminated effluent wastewater by cadmium in the study showed significant effect ($p < 0.05$) and positive correlation with contents of Cd (r = +88 to r = +93), in the contaminated wastewater and Grapefruit peel in one week contact respectively. The amounts of Cadmium adsorbed increased significantly with increase contact time ($p<0.003$).The other factor of stirring solution also showed significant agent to decreasing and removal of heavy metal from waste water ($p<0.001$). The data revealed that utilizing 1% bio-adsorbent of GP can remove 95.56% of cadmium at room temperature.

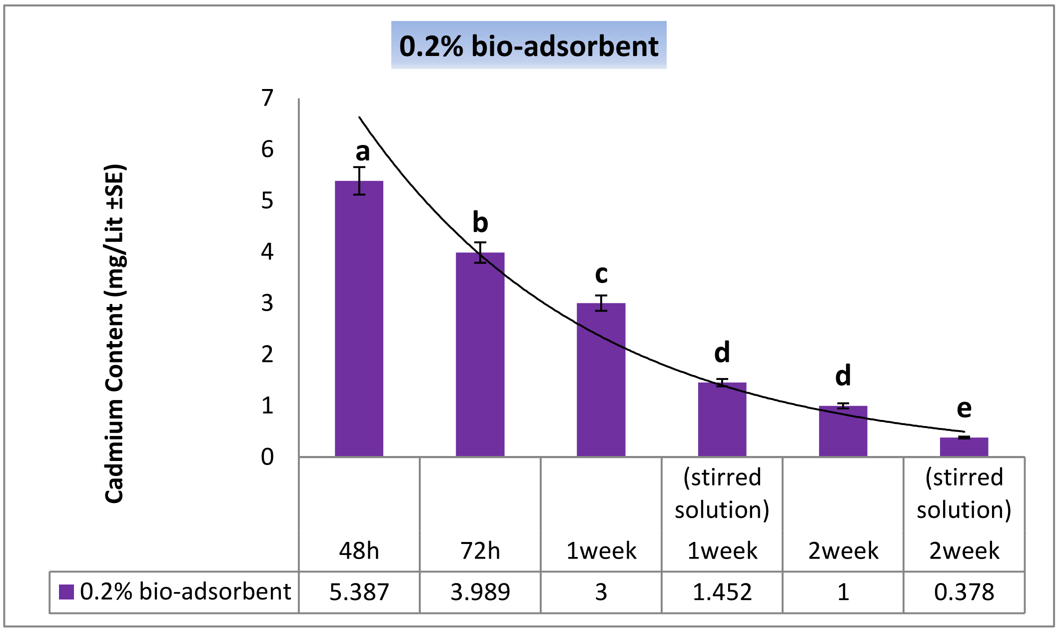

Figure 2. Effect of contact time on the removal of cadmium (initial Cd concentration=10.001 mg/l, bio-adsorbent dose=0.2 mg Grapefruit peel /100 ml of *waste water,* temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.6

Results in figure 2 in presence of 2% Grapefruit Peel showed significant difference in Cadmium up -taking by bio-adsorbent after 48 and 72 hours and also 7 days (1 week) without stirring. The data showed that accomplishing of agitation speed= 200 rpm during 1 week and 14 days (2 weeks) without stirred have no significant differentiate ($p \geq 0.05$) , but the potential of taking up Cadmium was increased significantly between 2 week with agitation speed by 2 weeks in statistic state ($p < 0.001$) and Cadmium content in untreated sate 10.01 mg/Lit after 2 weeks decreased to 0.378 mg/Lit which prove that biomass of adsorbent can reduce 96.22% of heavy metal. Moreover, time factor of putting adsorbent in contaminated effluent wastewater by cadmium in the study showed significant effect ($p < 0.03$) and positive correlation with contents of Cd (r = +89 to r = +94), in the contaminated wastewater and Grapefruit peel in one week contact respectively. The amounts of Cadmium adsorbed increased significantly with increase contact time (p<0.003).The other factor of stirring solution also showed significant agent to decreasing and removal of heavy metal from waste water (p<0.001).

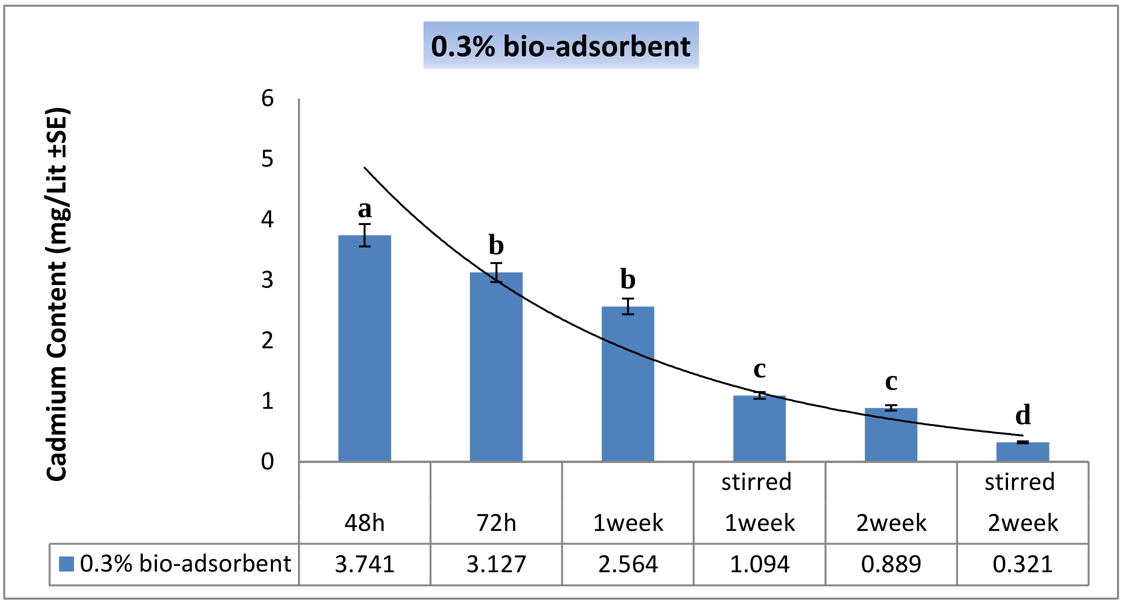

Figure 3. Effect of contact time on the removal of cadmium (initial Cd concentration=10.001 mg/l, bio-adsorbent dose=0.3 mg Grapefruit peel /100 ml of *waste water,* temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.6.

Results in figure 3 in presence of 3% Grapefruit Peel showed almost the same results of presence of 1% and 2% of bio-adsorbents. Moreover, time factor of putting adsorbent in contaminated effluent wastewater by cadmium in the study showed significant effect (p <0.02) and positive correlation with contents of Cd (r = +87 to r = +93), in the contaminated wastewater and Grapefruit peel in one week contact respectively. The amounts of Cadmium adsorbed increased significantly with increase contact time (p<0.003).The other factor of stirring solution also showed significant agent to decreasing and removal of heavy metal from waste water (p<0.001). The finding from this stage of study showed that biomass of adsorbent can reduce 96.79% of heavy metal which no significant differences by lower percentage of studied biomass of GP as bio-adsorbent.

Results in figure 4 in presence of 4% and 5% Grapefruit Peel showed significant difference in Cadmium up -taking by bio-adsorbent after 48 and 72 hours and also 7 days (1 week) without stirring. The data showed that accomplishing of agitation speed= 200 rpm during 1 week and 14 days (2 weeks) without stirred have no significant differentiate (p ≥ 0.05) just like lower concentrations of adsorbents in this study, but the potential of taking up Cadmium was increased significantly between 2 week with agitation speed by 2 weeks in statistic state (p <0.001) and Cadmium content in untreated sate 10.01 mg/Lit after 2 weeks decreased to 0.0.227 and 0.207 mg/Lit respectively which prove that biomass of adsorbent can reduce 97.73 and 97.93 of heavy metal by utilizing of 4% and 5% of biomass.

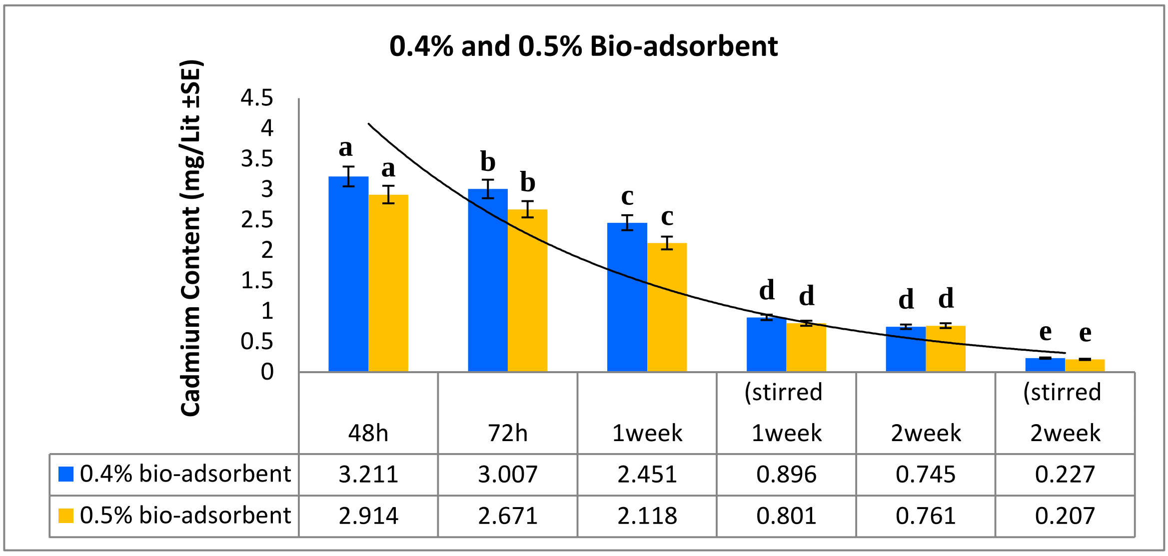

Figure 4. Effect of contact time on the removal of cadmium (initial Cd concentration=10.001 mg/l, bio-adsorbent doses=0.4 and 0.5 mg Grapefruit peel /100 ml of *waste water* respectively, temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.6.

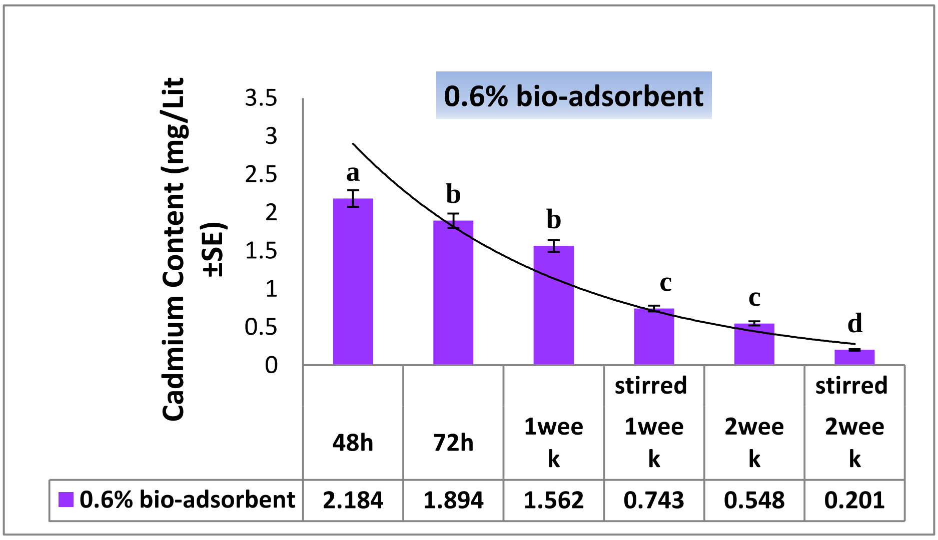

Figure 5. Effect of contact time on the removal of cadmium (initial Cd concentration=10.001 mg/l, bio-adsorbent dose=0.6 mg Grapefruit peel /100 ml of *waste water,* temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.6.

The results of the increasing adsorbent to 0.6% in figure 5, showed significant differences in Cadmium up -taking by bio-adsorbent after 48. 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 <0.03). Cadmium content in untreated sate 10.01 mg/Lit after 2 weeks decreased to 0.0.201 mg/Lit which has no difference by 0.5% concentration of biomass. This content of adsorbent can reduce 10.01 mg/Lit Cadmium to o.201 mg/Lit in pharmaceutical effluent which means removal of 98% of heavy metal.

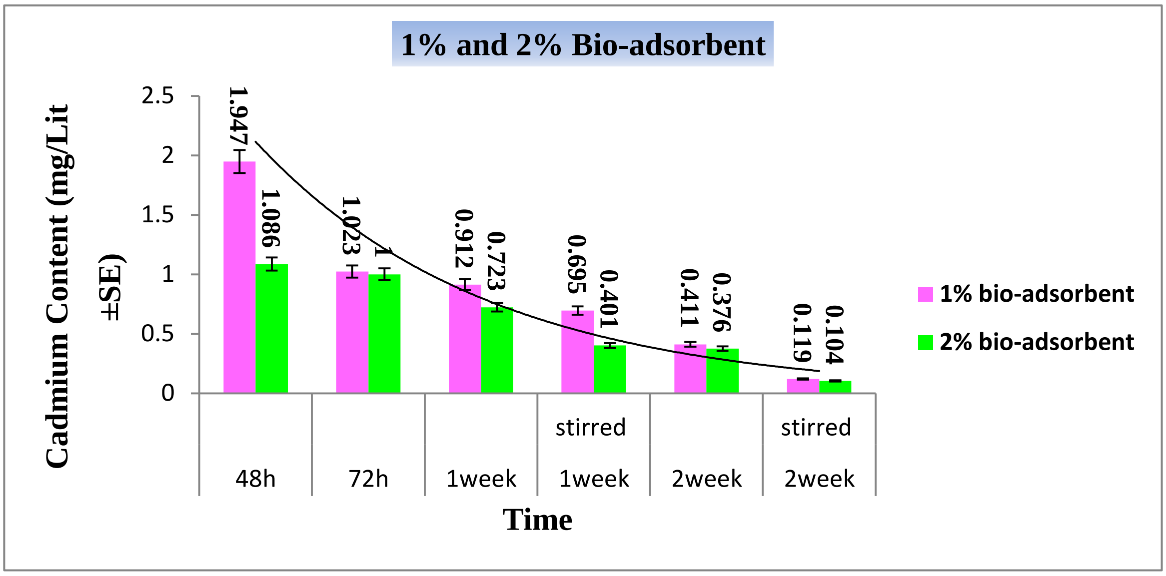

Figure 6. Effect of contact time on the removal of cadmium (initial Cd concentration=10.001 mg/l, bio-adsorbent dose=1 and 2 mg Grapefruit peel /100 ml of *waste water,* respectively; temperature=25 ± 2 ºC, agitation speed= 200 rpm), pH = 4.6.

As Cadmium contents compared between the final results of 2 weeks contact time by 2% bio-adsorbent (0.376 mg/Lit) to the untreated initial concentration (10.001 mg/Lit), the manifest removal trend was observed.

Results in figure 6 in presence of 1% and 2% Grapefruit Peel showed significant difference in Cadmium up -taking by bio-adsorbent after 48 by 2 different doses of bio-adsorbent but at 72 hours and also 7 days (1 week) without stirring no significant differences between removal of heavy metal ($p \geq 0.05$). The data showed that accomplishing of agitation speed= 200 rpm during 14 days (2 weeks) without stirred have significant differentiate ($p < 0.001$) just like lower concentrations of adsorbents in this study,but the potential of taking up Cadmium was increased significantly after 2 week with agitation speed reached the maximum rate of removal ($p < 0.001$) and Cadmium content in untreated sate 10.01 mg/Lit after 2 weeks decreased to 0.0.119 and 0.104 mg/Lit respectively which prove that biomass of adsorbent can reduce 98.81 and 98.96 of heavy metal by utilizing of 1% and 2% of biomass. The higher concentration of biomass or time in the rest of study had no significant and meaningful boosting the cleaning-up the waste water.

Bio-sorption of heavy metals can increase with increasing contact time (Figures 1-7). This could be attributed to the transfer of higher amount of bio-sorbent from solution phase to the bio-sorbent active sites as contact time increases. The results of current project are similar the other studies [17-18]. One of the most important factors in current study was contact time and which could be interpreted for the main reason for increasing sorption of heavy metals with protracts stirring time could be due to the reduced bound covering resistance to the transfer of biomass in solution and enhanced heavy metals kinetic energy [18, 38-41].

The results of current study revealed that cadmium adsorption ranged from 43-98 % after agitation for 48 hours up to 2 weeks (equilibration time after 24 hours), and there was no significant further increase in % sorption of them after the equilibration time ($p \geq 0.05$).

Results from this investigation revealed that Bio-adsorbent dose is one of the most important factors which affect significantly on influence specific uptake of all studied samples of waste water effluent. Generally, for low bio-adsorbent dose, there is an enhanced heavy metals sorption for Cd and Sorption capacity of different bio-adsorbents have been observed no significant effect with increasing bio-adsorbent dose by higher concentrations that 2% Grapefruit peel (figures 2-6).

Salman et al., in 2014 claimed that metal bio-sorption is the removal of metal ions by inactive, nonliving biomass due to highly attractive forces present between the two phases of metal ions and bio-mass [42]. Particularly, the process of adsorption is highly depend on the presence of certain functional groups, such as hydroxyl, amine, carboxyl, phosphate, sulfhydryl etc., on the cell wall of the biomass [43]. The process involves a solid phase (biomass) and a liquid phase containing metal ions (solution of metal ions/waste-water) which in our study was pharmaceutical effluent of different educational or research laboratories in Pharmaceutical faculty. Metal ions are attracted and bound to the biomass by a complex process that comprises of a number of mechanisms like adsorption on the surface and pores, ion-exchange, surface precipitation, complexation and chelation and entrapment in capillaries and spaces of polysaccharide network, due to the concentration causing diffusion through the cell wall and membrane.

Adsorption process are economical, metal selective, regenerative, devoid of toxic sludge generation, metal recovery and most very effective. The removal of heavy metals by low cost adsorbent was encouraging for several natural materials, agricultural and industrial by-products which can be utilized as low-cost adsorbents [18]. Various disorders and damage due to oxidative stress induced by free radical formation of heavy metals and their toxicity mechanisms result in ill-health effects. Heavy metal toxicity is linked to several health risks including biological role where the pseudo element interferes with metabolic processes and gets chronic deposit into the body and food chain. Control, prevention and treatment of metal toxicity at various levels of occupational exposure, accidents and environmental factors are absorbed during the exposure and duration leading to acute or chronic [44]. Monish et al in 2014 published that adsorption performance is good during the removal from dilute waste solutions, despite the fact that the use of common materials such as activated carbon, chitosan, zeolite, clay are completely recognized in order to the high adsorption capacity, they lead to overpriced process, too. The increase in demand to find relatively efficient, low cost and easily eco-friendly and adsorbents for heavy metals therefore is relied on the wastes from agricultural or food industries [7, 45-47]. The results of experimental study by Ziarati et al., in 2019 , revealed that using 0.5 % red tea residue in accompany by 3 % black tea residue can remove and decrease Cadmium and Nickel significantly and for Cobalt decreasing the rate of adsorption is not as much as 2 other metals but remove Cobalt after 40 minutes in remarkable contents [18]. Next, mathematical modeling was carried out based on experimental data. Work on the mathematical model creation was divided into several stages. At the first stage, the general kinetic regularities of the adsorption process were assessed. At the second stage, model coefficients specific values were assessed. At the third stage, the technological parameters impact on technological solutions was assessed. Next, mathematical modeling was carried out based on experimental data. Work on the mathematical model creation was divided into several stages. At the first stage, the general kinetic regularities of the adsorption process were assessed. At the second stage, model coefficients specific values were assessed. At the third stage, the technological parameters impact on technological solutions was assessed [23- 24].

Conclusion

Results showed that low-cost and available bio- adsorbents can be fruitfully used for the removal of heavy metals with a concentration range of 1–20 mg/lit. It also was found that the percentage removal of heavy metals was dependent on the dose and particle size of low cost adsorbent and adsorbent concentration. The contact time necessary for maximum adsorption was found to be five hours. The optimum pH range for Cadmium adsorption was in Acidic range and best results obtained in 4.3-5.6 which the best results comes from pH= 4.6 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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Conflicts of Interest

None of the authors have any conflicts of interest associated with this study.

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