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Study of Biosorption and Desorption Process of Cu(II), Cr(VI), Pb(II) and Zn(II) ions by using peels of Citrus aurantifolia

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Abstract

Heavy metal pollution in aquatic water bodies from the release of toxic heavy metals from factories is a major problem in the modern world. Heavy metal pollution can be fixed in a number of ways, both physically and chemically. Biosorption is seen as a possible alternative to other methods for removing heavy metals from waste water. In this study, the peels of Citrus aurantifolia were used to remove Cu(II), Cr(VI), Pb(II), and Zn(II) ions from aqueous solutions. All four of the metal ions that were studied could be taken up by the peels. It was found that the biosorption process was affected by things like contact time, temperature, pH, turbidity, and the amount of biosorbent used. E-SEM, EDAX, and FT-IR analysis were also used to look at how Citrus aurantifolia changed after the biosorption process. The studies of adsorption isotherms showed that the Freundlich isotherm model fit experimental data better than the Langmuir isotherm model did. From a statistical point of view, the results were found to be important. By using certain eluents in a desorption study, biosorbent was brought back to life.

Keywords: Citrus aurantifolia, biosorption, traits, adsorption isotherm

Introduction

When industrial, household, and agricultural wastes are dumped into water bodies, pollutants, especially heavy metals, build up in the sediments and harm the health of living things. When there are a lot of these heavy metals, they can be toxic because of the metal itself, its inorganic salts, or organic compounds from which the metal is easy to separate or get into cells. Lead, mercury, cadmium, arsenic, copper, zinc, and chromium are all dangerous heavy metals for people [1]. Since most heavy metals don't break down into non-toxic products and/or stay in the environment for a long time, their concentrations must be brought down to safe levels before they can be released into the environment.
Over the years, people have come up with different ways to treat and get rid of heavy metals in industrial effluents. Some of these methods are chemical precipitation, ion exchange, ion exchange, and reverse osmosis. Due to the problems with traditional treatment methods, it is important to find a cheaper way to treat wastewater, such as using a biological method to remove heavy metals from aqueous effluents [2-4].
In the 1970s, as people became more aware of and concerned about the environment, they looked for new, cheap ways to clean up heavy metal-polluted water. Heavy metals can be taken out of water solutions by putting pollutants on solid adsorbents, which works well [5].

Environmental biotechnology studies from the past have shown that many organic materials found in the environment can remove heavy metals from solutions through a process called biosorption [6]. Biosorption is the process of taking substances out of a solution by using living things. These things can be either organic or inorganic, and they can be soluble or not soluble [7]. Compared to other methods, biosorption has benefits like being cheap, selective for metals, regenerative, not making sludge, and the possibility of metal recovery [8].

Biosorbents could be made cheaply from natural materials that are easy to find in large amounts or from waste products from industrial or agricultural processes [9]. Different attempts have been made to use biosorbents like rice husk [10], coconut shell [11], peanut shell [12], orange peel [13], pineapple crown [14], etc. to remove heavy metals from water.

For practical or dynamic adsorption capacity to be estimated, you need to know enough about adsorption equilibrium, which is shown by adsorption isotherms. Adsorption isotherms are mathematical models that show how adsorption species behave when they move from the liquid phase to the solid phase. Two important and often-used isotherm models are the Langmuir isotherm model and the Freundlich isotherm model [15]. The process of desorption, which concentrates the metal ions that biosorbent has already taken up, is also important for reusing biosorbent and getting the metal ions back. Various eluents, such as acids, alkalis, chelating agents, etc., can be used to remove metal ions from biosorbents that are already loaded with metals [16–18].

In this study, scientists tried to find out how well Citrus aurantifolia peels could remove Cu(II), Cr(VI), Pb(II), and Zn(II) ions from water solutions. Researchers looked at how different things affected the removal of metal ions from aqueous solution. The Freundlich and Langmuir isotherm models were also used to study adsorption isotherms. Various eluents were used to get the metal ions out of the biosorbent during the desorption process.

Materials and Methods

Plant material collection, authentication and preparation

The plant parts were taken from a fruit juice center in the area. The sample was checked at the Agarkar Research Institute in Pune, India, to make sure it was from the right species. The peels were washed several times with tap water and then washed again with distilled water. It was completely dried in an oven at 60oC to get rid of all the water. Also, the peels were ground into a powder (which will be called biosorbent from now on) and stored in an airtight container for later use. Color, pH, bulk density, iodine index, moisture content, and ash content were some of the things that were looked at about the biosorbent..

Preparation of metal salt solution

All the chemicals used in the study were of analytical grade. Stock solutions (1000 mg/L) of metal ions viz. Cu(II), Cr(VI), Pb(II) and Zn(II) were prepared by dissolving required amount of respective metal salts in deionized water. The working standard solutions were prepared by diluting the stock solutions to appropriate volumes.

Biosorption process

Cu (II), Cr(VI), Pb(II), and Zn(II) ions in a solution of 50 mg/L were treated with 1% biosorbent in a 250 mL conical flask at 30°C. The solution was then shaken at 150 rpm in an orbital shaker. After an hour, Whatmann filter paper No. 1 was used to filter the solution. As a blank, the aqueous solution of the biosorbent being studied was used. ICP-AES was used to figure out how much of each metal ion was in the filtrate (ARCOS, Simultaneous ICP Spectrometer, IIT-SAIFBombay).

Calculation of biosorption capacity and percentage biosorption

The biosorption capacity (qe) (Kariuki et al., 2017) shows how many metal ions can be absorbed per gram of biosorbent. It was calculated as follows:

qe (mg/g) = (Co - Ce) * V (1) m

Here's how the percentage of biosorption of metal ions was worked out:

Biosorption (%) = (Co - Ce) * 100 (2)

Co

where Co is the amount of metal ions in the water at the start (mg/L).

Ce is the final amount of metal ions (mg/L) after the biosorption process. V is how much solution there is (L)

m is the biosorbent's weight (g)

Factors affecting biosorption process

Using batch experiments, the effect of different factors on the biosorption process using peels of Citrus aurantifolia was studied. These factors included pH, temperature, biosorbent dosage, contact time, and turbidity of aqueous solutions.

To find out how pH affects the biosorption process, 1 g of biosorbent was added to 100 mL of aqueous solutions containing 50 mg/L of each ion: Cr(VI), Cu(II), Pb(II), and Zn(II) in 250 mL conical flasks. The biosorption process was then studied at different pH levels: 2, 4, 6, 8, and 10 for 60 minutes at 30°C. With 1N NaOH and 1N HCl, the pH of the solution was changed.

The process of biosorption of Cr(VI), Cu(II), Pb(II), and Zn(II) ions by Citrus aurantifolia was studied at different temperatures by adding 1 g of biosorbent to aqueous solutions containing 50 mg/L of the four metal ions under study and incubating the solutions at 10, 30, and 50 oC for 60 minutes at pH 8.

The effect of biosorbent dose was studied by adding different amounts of biosorbent (0.5%, 1%, 2%, and 3%) to 250 mL conical flasks with 100 mL solutions of 50 mg/L of Cr(VI), Cu(II), Pb(II), and Zn(II) at pH 8. The biosorption process was run for 60 minutes at 30°C.

The effect of contact time on the biosorption process was studied by adding 1 g of biosorbent to 100 mL of aqueous solutions with 50 mg/L of Cr(VI), Cu(II), Pb(II), and Zn(II) ions at pH 8 and running the biosorption process for 60, 120, and 180 minutes at 30 oC.

100 mL of aqueous solutions with different levels of turbidity and 50 mg/L of Cr(VI), Cu(II), Pb(II), and Zn(II) were mixed with 1 g of biosorbent, and the biosorption process was done for 60 minutes at 30 oC. With a mixture of hexamine and hexamethylene sulphate, the turbidity of the solution was changed to 10, 50, and 100 NTU.

In all of the above experiments, the aqueous solutions with the biosorbents (blank) were kept under the same conditions so that you could compare them. All of the solutions were put through an orbital shaker with a speed of 150 rpm. After the biosorption process, all of the solutions were filtered with Whatmann filter paper No. 1, and ICP-AES was used to check the filtrates for any leftover metal ions (ARCOS, Simultaneous ICP Spectrometer, IIT- SAIF Bombay). All of the experiments were done three times, and the average percent biosorption was reported.

Characterization of biosorbent:

Researchers looked at the physical and chemical properties as well as the surface properties of biosorbent that had not been treated. The following was done to study how the surface properties of biosorbent changed after it was treated with metal salt solutions:

For Environmental Scanning Electron Microscope (E-SEM) analysis, the biosorbent was put on the sample stub and dried under IR light for two to three minutes. Then, the JEOL JFC-1600 Auto fine Coater was used to coat the sample with platinum for 600 seconds. The biosorbents were then scanned with E-SEM (FEI QUANTA 200 E-SEM, IIT-SAIF Bombay) at 15 kV and different magnifications.

Energy Dispersive X-ray Analysis (EDAX) and a Scanning Electron Microscope were used to look at the biosorbent's elements. The beam's power was in the range of 10 to 20 keV. This makes the material that has been irradiated give off X-rays. The X-rays came from a place that was about 2 microns deep. By moving the electron beam across the biosorbent, a 2-D image of each element in the biosorbent was made. At IIT-SAIF Bombay, biosorbent was put through an EDAX test.

Fourier Transform InfraRed (FT-IR) Spectroscopy was used as a qualitative method to check if the surface of the biosorbent had any functional groups. The potassium bromide (KBr) disc method was used to do the study. Wavenumbers from 400 to 4000 cm1 were used to measure the biosorbent's spectrum. The biosorbent was mixed with KBr in a ratio of 1:10 to make pellet. Using a standard light source and a TGS detector, an FT-IR spectrophotometer (FT/IR 4100 type A C208161016, Nanotechnology Research Centre, B. K. Birla College, Kalyan) was used to record the FT-IR spectrum.

Study of adsorption isotherm

Langmuir and Freundlich adsorption isotherm models were used to study the relationship between the concentration of metal ions in the aqueous solution and on the biosorbent at 30 oC. For each concentration of metal ions, a graph was made of the amount of adsorbate (metal ions) per unit weight of adsorbent (biosorbent) against the equilibrium concentration of the adsorbate still in solution. For the Freundlich isotherm, a linearized graph of log qe against log Ce was made, and for the Langmuir isotherm, a linearized graph of Ce/qe against Ce was made. Microsoft Excel 2010 was used to figure out the parameters for both isotherms.

The Langmuir equation can be written in linear form like this:

Ce/ qe = (1/ qm) * Ce + 1/ (b qm) (3)

Based on more analysis of the Langmuir equation, the separation factor (RL), which is a parameter of the equilibrium that has no units, can be written as:

RL = 1 / (1 + b Co) (4)

where Co is the amount of metal ions in the water at the start (mg/L).

Ce is the amount of metal ions in a given volume of water.

qe is the amount of metal ions adsorbed (in mg/g) per unit mass of the biosorbent. qm and b are Langmuir constants calculated from the slope and the intercept, respectively.

The RL parameter is a good way to tell how the adsorption process is going. If the RL value is between 0 and 1, it means that the conditions are good for adsorption [17,19].

This is how the linearized Freundlich equation can be written:

log qe = log Kf + 1 log Ce (5)

n

where Kf is a measure of the amount of adsorption and n is the amount of adsorption.

The constant 'n' gives an idea of how unevenly the energetic centers are spread out and is related to the size of the driving force for adsorption. Good adsorption is shown by 'n' values between 2 and 10 [20–23]. Analysis of the numbers

The data from the experiments done to remove metal ions from metal salt solutions with an initial concentration of 10–50 mg/L of metal ions by using 1% biosorbent dose at pH 8 were put through the Chi-square test. In this test, the two most important parameters were the experimental value for biosorption capacity (qexp) and the biosorption capacity calculated from the model (qcalc).

Here's how to figure out the results of the Chi-square (2) test:

χ2 = (qe exp − qe cal)2 (6)

qe cal

where qe exp (mg/g) is the experimentally found equilibrium capacity
The model is used to figure out the equilibrium capacity, which is written as qe cal (mg/g).
If the experimental data and model data are similar, the value of 2 will be small, but if they are different, the value of 2 will be larger [24,25].

Desorption process

For the study of the desorption process, 2 g of air-dried biosorbent that had been loaded with metal ions like Cu(II), Cr(VI), Pb(II), and Zn(II) was put into flasks with 100 mL of an eluant like 0.1 M HNO3, 0.1 M EDTA, 0.1 M NaOH, and deionized water. The solutions were stirred at 100 rpm in a rotary shaking incubator (Remi Orbital Shaking Incubator, CIS 24 BL) at 30 oC for 2 hours, then filtered with Whatmann filter paper no. 1. ICP-AES (ARCOS, Simultaneous ICP Spectrometer, IIT Bombay) was used to measure the amount of metal ions released by the desorbing agent (M. S.).

2. Results and Discussion

The plant parts were tested, and the peels of Citrus aurantifolia were found to be what they said they were. Figures 1a and 1b show that the peels of Citrus aurantifolia can be dried or ground into a powder.

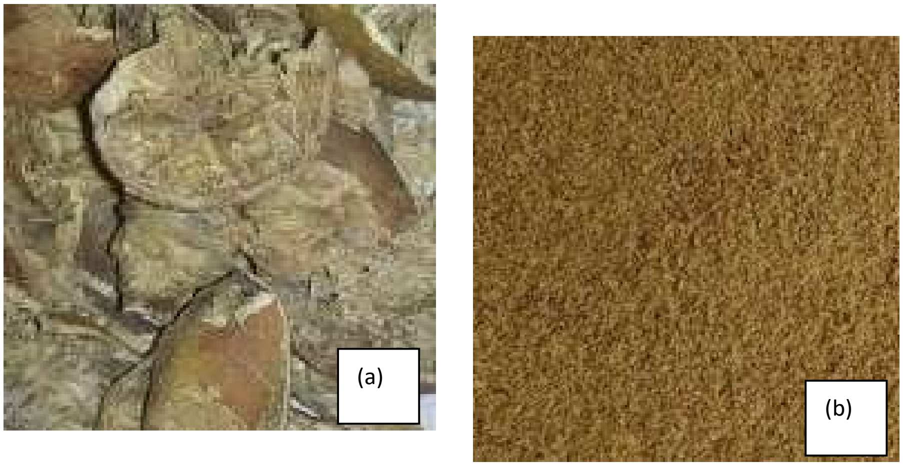


Figure 1. Peels of Citrus aurantifolia in (a) dried (b) powder form

It has been reported that the pH of biosorbents in the range of 6-8 can be used for many applications [26-27]. The bulk density less than 1.2 g/mL represents that biosorbents are fine in nature [28]. Iodine Index for commercial adsorbent ranges from 300 to 1200 mg/g [29]. By comparing the above data with the values of characteristics shown in table 1, it can be observed that peels of Citrus aurantifolia may have good adsorption properties.

Table 1: Characteristics of powder peels of Citrus aurantifolia

Characteristics	Values
Colour	Yellow
Moisture content (%)	6.36
pH	6.21 ± 0.25
Ash content (%)	6.6667
Bulk density (g/mL)	0.3233 ± 0.13
Iodine Index (mg/g)	442.69

The percent biosorption and biosorption capacity (mg/g) by the biosorbent are shown in table 2. The peels were found to be more efficient in the biosorption of Pb(II) and Cr(VI) ions as compared to Zn(II) and Cu(II) ions from the aqueous solution.

Table 2: Percent biosorption and biosorption capacity of peels of Citrus aurantifolia

Metal ions	% biosorption	Biosorption capacity (mg/g)
Cu(II)	55.79	2.30
Cr(VI)	80.2	5.31
Pb(II)	89.83	6.12
Zn(II)	61.26	2.55

The biosorption process was found to be affected by various factors.

Effect of pH:

As the solution's pH goes up, the number of H+ ions in the solution goes down. So, when the pH moves toward the alkaline range, it means that there are less H+ ions, which makes it easier for protons and metal ions to get along. So, a higher pH means that the ligand is available to bind to metal ions, which makes biosorption better [30]. In this study, it was found that the biosorption process by peels went up as the pH of the aqueous solution went up from 2 to 8. However, when the pH went up to 10, the biosorption process of all four metal ions studied went down (Figure 2).

Effect of temperature:

According to the theory of adsorption, the rate of adsorption goes down as the temperature goes up, and molecules that were absorbed earlier tend to leave the surface when the temperature goes up [31]. This could be because when the temperature of the solution goes up, the attractive forces between the surface of the biomass and the metal ions weaken, which slows down the adsorption process.

[32] showed that as the temperature went from 35 to 50 oC, the biosorption process of Cd(II) and Ni(II) ions using Glycine max pods went down. In this work, it was found that the biosorption process got better as the temperature went up from 10 to 30 oC. However, the biosorption process by the biosorbent under study didn't change much when the temperature went up to 50 oC. (Figure 3).

Effect of biosorbent dose:

The biosorption process was found to be affected by the amount of biosorbent used (0.5 to 3 percent). It was seen that the biosorption process sped up as the amount of biosorbent went from 0.5 percent to 1 percent. This could be because as the dose of biosorbent goes up, the number of active sites that can bind to metal ions also goes up. However, if the dose of biosorbent goes up even more, there could be too many biosorbents and they won't be able to bind to metal ions (shell effect). When the biosorbent dose went up to 3 percent, biosorption went down. This might have been because of the shell effect (Figure 4).

Effect of contact time:

The metal ions Cu(II), Cr(VI), Pb(II), and Zn(II) were more easily taken up by the biosorbent as the contact time went up from 60 to 180 minutes (figure 5). This could be because the metal ions need more time to attach to the active sites on the biosorbent. Similar results were seen by [33], who showed that the biosorption process got better as the contact time went up..

Effect of turbidity:

It was found that the biosorption process worked less well as the turbidity of an aqueous solution went from 10 NTU to 100 NTU. The biosorption process may have slowed down because positively charged metal ions are sticking to the negatively charged ions in the turbid solution. This makes the positively charged metal ions unavailable for the biosorption process (Figure 6). Geoffrey et al. [34] found that the biosorption process of Ni(II) and Pb(II) ions using Moringa oleifera seed became less effective as the turbidity of the water solution went from 20 to 50 NTU.

Figure 2. Effect of pH on Biosorption process

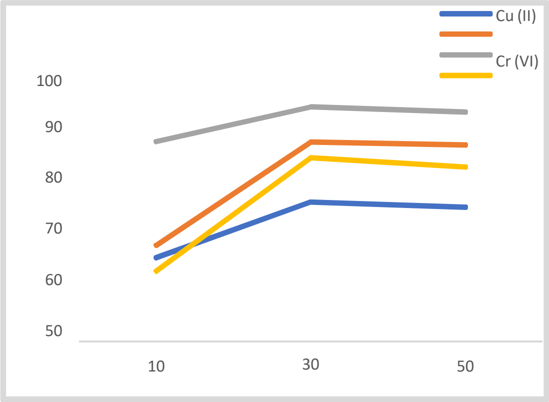


Figure 3. Effect of temperature on biosorption process

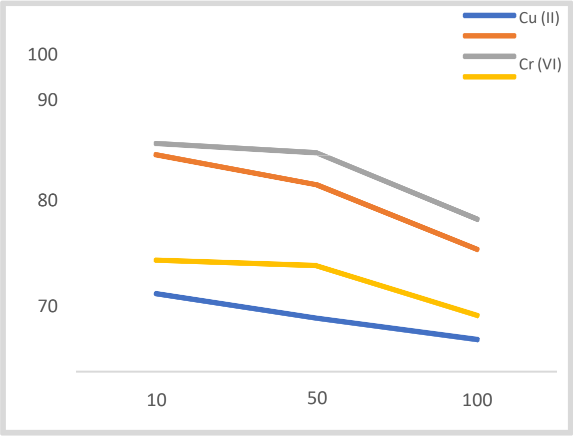


Figure 4. Effect of biosorbent dose on biosorbent process

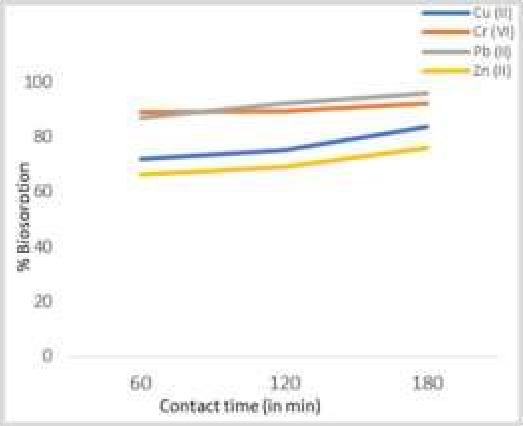
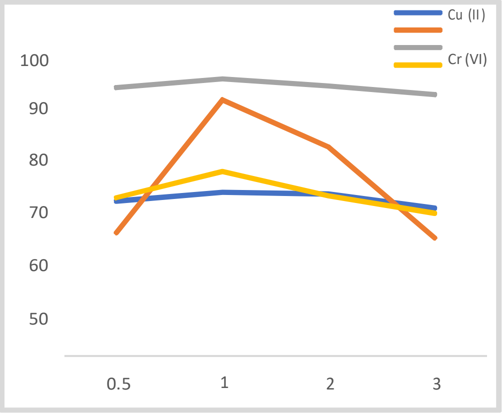


Figure 5. Effect of contact time on biosorption process

Figure 6. Effect of turbidity on biosorption process



The surface of the biosorbent was rougher after the biosorption process (Figure 7b) than it was before the process (Figure 7a). The roughness could be caused by metal ions interacting with biosorbent and sticking to its surface. So, after being treated with metal salt solutions, the surface structure of biosorbents

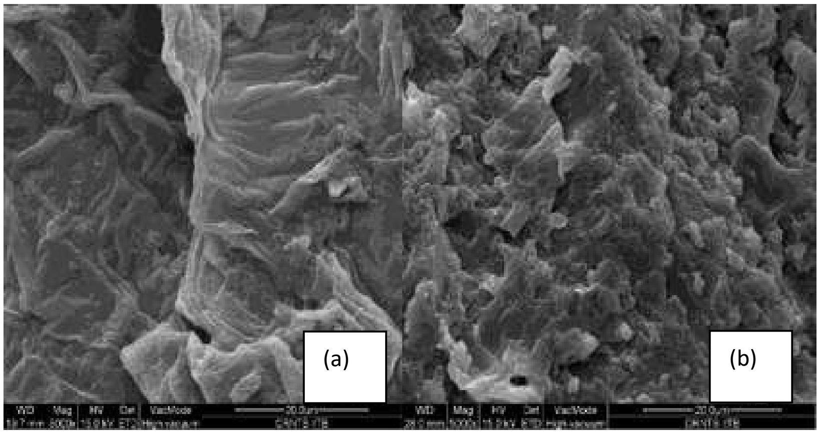
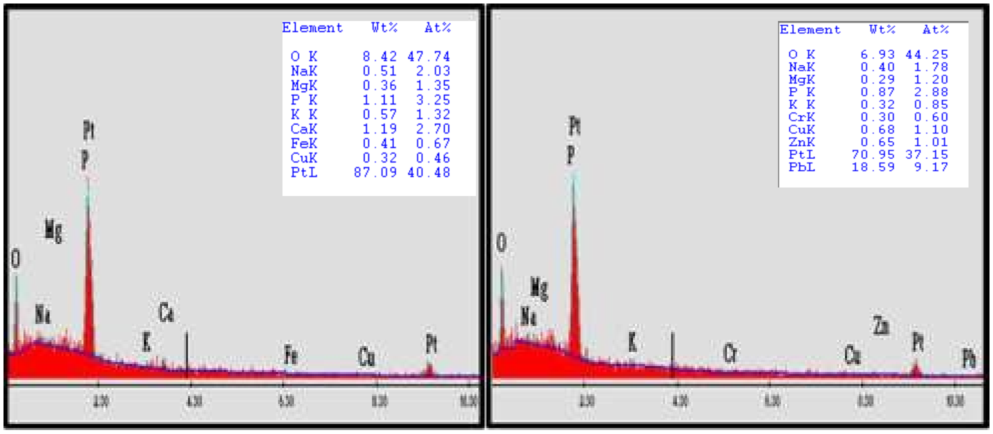


Figure 7. E-SEM images of biosorbent (a)before (b)after treatment

After the biosorption process, the weight (percent) of the heavy metals Cu, Cr, Pb, and Zn was found. This showed that metal ions had gathered on the surface of the biosorbent (Figure 8b). Figure 8a shows that there was 0.32 percent Cu on the biosorbent before the biosorption process. After the biosorption process, the amount of Cu on the biosorbent went up. This shows that there has been biosorption. Also, lead had the most weight (percent) compared to the other three heavy metals that were studied.



Element	Wt%	At%
O K	8.42	47.74
NaK	0.51	2.03
MgK	0.36	1.35
P K	1.11	3.25
K K	0.57	1.32
CaK	1.19	2.70
FeK	0.41	0.67
CuK	0.32	0.46
PtL	87.09	40.48

Element	Wt%	At%
O K	6.93	44.25
NaK	0.40	1.78
MgK	0.29	1.20
P K	0.87	2.88
K K	0.32	0.85
CrK	0.30	0.60
CuK	0.68	1.10
ZnK	0.65	1.01
PtL	70.95	37.15
PbL	18.59	9.17

Figure 8. EDAX images of biosorbet (a)before (b)after treatment

FT-IR spectroscopy was used to find out more about the biosorbent's functional groups. Figures 9a and 9b show the FT-IR spectra of biosorbent that has not been treated and biosorbent that has been treated. Table 3 shows the wavenumbers, vibrations, and functional groups at each location on the biosorbent..

Table 3: FT-IR spectrum analysis of biosorbent

Sr. No.	Location wavenumber (cm^{-1})	Vibration	Functional group
1	3845.36	O-H stretching	hydroxyl groups and alcohol
2	3773.05	O-H stretching	hydroxyl groups and alcohol
3	3435.56	O-H stretching	hydroxyl groups and alcohol
4	2925.48	C-H stretching asymmetrical	alkane

5	2347.91	N-H	ammonium ions
6	2227.38	-C≡C- Stretching	alkynes
7	1640.16	C=O stretching COO- stretching	amide I, carboxylates aromatic ring modes, alkenes
8	1563.02	$-NO_2$ groups	nitro-groups
9	1412.6	C-C stretching (in ring)	aromatic
10	1020.16	-C-H plane bending	aromatic
11	928.557	O-H bend	carboxylic acids

Figure 9. FT-IR spectra of biosorbent (a) untreated (b) after treatment

After the biosorption process, most of the time there were no big changes in the wavenumbers. It could be because the electrostatic interactions between the metal ions and the functional groups on the surface of the biosorbent were not strong enough to change the absorption band frequencies or wavenumbers. It showed that there might be weak coordination between the metal ions and functional groups on the surface of biosorbents [35].

It is very important to look at equilibrium data in order to come up with an equation that accurately represents the results and can be used for design [36]. In this work, the Freundlich and Langmuir isotherm models were used to look into the experimental equilibrium data (qe values). With Microsoft Excel 2010, the line regression equations of both isotherm models for Cr(VI), Cu(II), Pb(II), and Zn(II) ions were found and are shown in Table 4. Table 5 shows how the equilibrium parameters for both models were calculated.

Table 4: Line regression equations for Freundlich and Langmuir adsorption isotherms by using lemon peels as biosorbent

Metal ions	Line regression equation for isotherm models	
	Freundlich isotherm	Langmuir isotherm
Cu(II)	y = 0.307x + 0.4762	y = 0.0922x + 0.6735
Cr(VI)	y = 0.4138x + 0.4187	y = 0.0623x + 0.8318
Pb(II)	y = 0.416x +0.3569	y = 0.0797x + 0.845
Zn(II)	y = 0.2129x + 0.64	y = 0.121x + 0.0607

Table 5: Equilibrium parameters for Freundlich and Langmuir isotherm models

Metal ions	Freundlich isotherm model				Langmuir isotherm model			
	K_F (L/g)	1/n	n	R^2	q_m	b (L/g)	R_L	R^2
Cu(II)	2.9936	0.307	3.2573	0.911	10.8460	0.1369	0.2321	0.905
Cr(VI)	2.6224	0.4138	2.4166	0.7838	16.0514	0.0749	0.3420	0.5929
Pb(II)	2.2746	0.416	2.4038	0.8366	12.5471	0.0943	0.2998	0.7949
Zn(II)	4.365	0.2129	4.9670	0.7145	8.2645	1.9934	0.0222	0.9885

Since the value of 'n' was between 2 and 10 and the value of 'RL' was between 0 and 1, this means that the peels of Citrus aurantifolia did a good job of absorbing all of the metal ions under study. The Chi-square test gave statistical support for the analysis. In table 6, the results of the Chi-square test are shown..

Table 6 : Chi-square test values for metal ions under study

Metal ions	Isotherm model

	Freundlich	Langmuir
Cu(II)	0.3014	1.4564
Cr(VI)	1.8271	3.9638
Pb(II)	0.9283	2.2396
Zn(II)	0.7552	0.8822

We found that the Chi-square values of the Freundlich isotherm model for Cu(II), Cr(VI), Pb(II), and Zn(II) were lower than the Langmuir isotherm model values. This shows that the Freundlich isotherm model fits well with the experimental data for the biosorption of metal ions being studied by using the peels of Citrus aurantifolia as the biosorbent. In the adsorption process, the desorption step is very important because it can improve how quickly and cheaply metal ions can be taken out of wastewater. We watched and figured out how well 0.1 M HNO3, 0.1 M NaOH, 0.1 M EDTA, and deionized water (D/W) removed metal ions from a biosorbent that was loaded with Cu(II), Cr(VI), Pb(II), and Zn(II). In figure 10, it was seen that deionized water removed almost none of the four metal ions when compared to the other desorbing agents that were being studied. So, deionized water was thought to be a bad desorbing agent (eluant). Most metal ions, like Cu(II), Cr(VI), Pb(II), and Zn(II), came off of the biosorbent when EDTA was added. The Pb(II) ions were found to desorb more than the other three metal ions being studied. Also, the desorption process with NaOH was found to be better than with HNO3. Researchers [37] found the same thing when they looked at the desorption process: EDTA worked well as a desorbent to get Pb(II) ions out of the green algae Cladophora fascicularis.

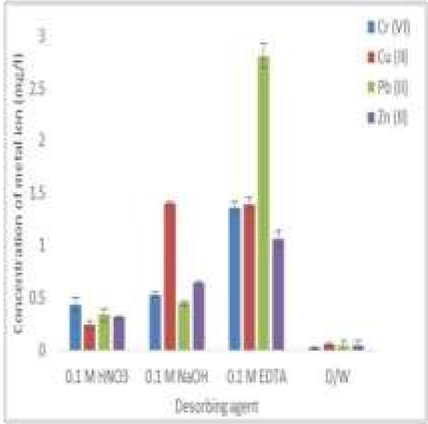


Figure 10. Desorption of metals ions from peels of Citrus aurantifolia

4.0. Conclusion

Citrus aurantifolia peels, which are a waste product from farming, can be used as a good biosorbent to remove Cu(II), Cr(VI), Zn(II), and Pb(II) ions from water. Adsorption isotherm studies showed that the result was correct. The desorption process showed that the biosorbent under study can be regenerated for more cycles of adsorption and desorption, and that it may also be possible to recover metal ions.

Using the biosorbent we have now to remove metal ions from industrial effluents can make a process that is good for the environment and saves money. This is what we need right now.

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Using the biosorbent we have now to remove metal ions from industrial effluents can make a process that is good for the environment and saves money. This is what we need right now.

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