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Toxic Metal Pollution Abatement Using Sour Orange Biomass

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Abstract

In this work, Sour orange (SOR) peels waste was used as a solid adsorbent for removal of Cobalt (III) ions from aquatic ecosystem through batch techniques, the influence of biosorbent dose, initial concentration, temperature, contact time and pH on the Co (III) ions removal percentage were investigated. Optimum removal of Co(III) ions was achieved at biosorbent (2 gm),initial Co(III) ions (5 mg/l), temperature(30 C°), contact time(90 min.) and pH(2). Desorption experiments were performed due to the importance for the financial competitiveness of biosorption with other process, indicating that (0.1 M) Ammonium chloride is the most promising desorbing agent for efficient sorbent regeneration. The adsorbent was characterized using FT-IR spectroscopy detecting the changes in hydroxyl and carboxyl groups before and after biosorption confirming their involvement in Co (III) ions binding. Various parts of SOR were selected for comparative studies on the Co (III) ions removal percentage indicating that SOR peels has the highest Co (III) ions removal percentage, followed by the other parts. Bioactivity inquires revealed that the SOR peels has a bioactivity against *Pseudomonas aeruginosa*. The results of this study confirm that SOR peels waste can be used beneficially in treating aquatic ecosystem containing Co (III) ions.

Keywords: Biosorption, heavy metals ions removal, sour orange peels.

Introduction

Tremendous release of heavy metals into aquatic ecosystem due to industrialization and urbanization has intensified environmental issue worldwide. Unlike organic pollutants, the majority of which are susceptible to biological degradation, heavy metals ions do not degrade into harmless end product, but they tend to be absorbed by living organisms in combined forms as well as their elemental form. Once they enter the food chain, large concentrations of heavy metal ions may accumulate in human body; consequently causing sever dysfunction in kidneys, reproductive system, liver, brain and central nervous system thus threateningly the well being of humans and ecosystem [1].

The ecological problems result from the existence of heavy metals ions motivated many physic-chemical methods for their removal from aqueous solution. These include Liquid-Liquid extraction, co-precipitation, resin chelating, electro-chemical deposition, ion exchange, coagulation or flocculation and solid phase extraction [2]. However, those techniques have their own inherent limitation such as incomplete metal removal, requirements for expensive equipment and generation of toxic sludge or other waste products that require disposal or treatment [3]. These disadvantage, together with the need for more effective, affordable and safe methods for the elimination of heavy metals from contaminated towards waters has necessitated research interest towards the development of alternative economical separation technologies.

Biosorption of heavy metals from aqueous solutions is a relatively an innovative process that has emerged as a cost effective and efficient alternative to conventional method for water and waste water treatment utilizing naturally occurring and agricultural waste materials as biosorbents as these are cheaper, renenable, abundantly, available, economical nature and ecofriendly behavior [4]. Sorption field has been enriched by a vast amount of researchers including a variety of citrus biomaterials such as sour orange [5], orange [6, 7, 8, 9, 10, 11, 12, 13, 14, 15], lemons [8, 15, 16, 17], durian [18], grapefruit [6] and Ponkin mandarin [19].

The mechanisms of biosorption are generally based on physic-chemical interactions between metal ions and the functional groups present on the cell surface, such as electrostatic interactions, ion exchange and metal ion chelation or complexaction. Functional groups most commonly implicated in such interactions include carboxyl, hydroxyl, amine and phosphoryl groups present within cell wall components such as polysaccharides, lipids and proteins [20].

Sour orange (Citrus auratium) also named Bitter orange,Seville orange and Marmalade orange is a member of Rutaceae family and one of the famous fruit commodities in Iraq and other mediturnean region. It spiny every green tree cultivated for the essential oil expressed from the fruit, herbal medicine as a stimulant and appetite suppressant [21].

Sour orange peels principally consists of pectin substances, cellulose, hemi cellouse, chlorophyll pigments and other low molecular weight compound including limonene. The pectic substances are the predominant type of polysaccharides identified for the cell wall of citrus aurantiumn residue whose main structural features are linear chain containing more than 100 (1-4) linked D-galacturonic acid residue bear various polar functional groups including carboxylic and hydroxylic groups that are known to bind strongly metal cations in aqueous solution [22].

The choice of grinded and dried Sour orange peels selected for this study was based on their high content of pectin (galacturonic acid), their abundance in various regions in Iraq and their consideration as a solid waste which are often thrown away to litter the environment.

The present study undertaken to evaluate the efficiency of Sour orange peels for the removal of Co (III) ions in aqueous solutions by conducting batch experiments at variation of Biomass dose, initial Co(III) ions concentration, temperature, contact time and pH. This will offer Sour orange peels as an alternative to high cost commercial adsorbent materials.

Materials and Methods

Biomass preparation:

Native or original Sour orange peels was collected from local plantation zone(Al-Bunnok-Baghdad-Iraq), 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, for preservation, it was kept in plastic stopper bottles and to minimize contact with humidity, all these bottles were preserved in desiccators. The fine powder was used without any chemical or physical pre-treatment.

Simulated wastewater preparation:

Simulated wastewater containing Co(III) ion was prepared from $CoCl_3.3H_2O$ (supplied by Merk as analytical grad).Stock solution (1000mg/L) was prepared, other concentrations were obtained from this stock solution by serial dilution.0.1 mol/L HCl (BDH chemicals LTD Pool, England) and 0.1 mol/L NaOH (S.D.FINE-CHEM LIMITED) were used for pH value adjustments using a pH meter (WTW Terminal 740, Germany).

Batch Biosorption Experiments:

Batch biosorption experiments were conducted by mixing a fixed dose of citrus aurantium peels with (50 ml) of Co (III) ions solutions of desired concentrations in (100 ml) for a specified contact time, pH and temperature using a thermostatic rotary machine (BS-11). Batch biosorption studies carried out to evaluate the influence of biomass dose, initial Co (III) ions concentration, temperature, contact time and pH in order to achieve the maximum Co (III) ions removal percentage. After equilibrium, samples were filtered through (CNABO 44-Q, China) filter paper (No.18.0 cm) and the metal ion concentration were analyzed by atomic adsorption spectrophotometer (Shimadzu (AA-680) Atomic Absorption Spectrometer). The amount of metal ion removal by untreated Sour orange peels during the series of batch investigations was determined using the following equation expressed as:

$$\text{Removal\%} = \frac{C_0 - C_e}{C_0} \times 100$$

Where C_0 and C_e are the initial and equilibrium concentrations of Co (III) ions in solution (gm/L), and the amount of Co (III) ions adsorbed (qe in mg/g) was calculated as follows:

$$q_e = \frac{(C_0 - C_e)V}{m}$$

Where V is the volume of solution (L) and m is the mass of adsorbent (g).

Desorption experiments:

In order to test the ability of repeated use of biosorbent,consective batch biosorption and desorption experiments were carried out. (2 gm) of dried metal loaded biomass was contacted to (100 ml) of desorbing agent (NaOH, HCl, NH_4Cl, EDTA and $CaCl_2$) at various concentrations (0.05, 0.1, 0.2, and 0.3 M) on a rotary shaker for (45 min.). The samples were filtered on a filter paper and were subjected to residual metal concentration determination. After the optimizing the desorption efficiency of the selected desorbing agent the adsorption-desorption cycle was repeated four times using the same biosorbent. Desorption ratio was calculated from the following equations:

$$q_{des.} = \frac{C_{des.}V}{m}$$

Where $q_{des.}$ is eluted metal content (mg/g) and $C_{des.}$ is metal concentration in eluent solution (mg/dm^3). The percentage of desorbed metal was evaluated as :

$$\%\ \text{desorption} = \frac{q_{des.}}{q} \times 100 \times 100$$

FT-IR spectroscopy:

To characterize the Sour orange peels functional groups biomass surface, samples of biomass before and after Co (III) ions biosorption were analyzed with a FT-IR (830 Shimadzu spectrophotometer) spectrometer in the range of 400-4000 cm^{-1} under ambient conditions.

Comparatives studies:

Ten Sour orange fruits were randomly chosen from different trees. Each whole fruit was divided into five different parts: flavedo, albedo, segment membranes, juice and seeds. All tissue was homogeneously blended in a blender and dried in sun light. These compounds were then grounded and sieved. Using the same batch adsorption mentioned above to determine the optimal Sour orange fruit part for the Co(III)ions removal percentage.

Study of biological activity:

The biological activity for Sour orange peesl before and after adsorption with Co(III) ions were studied against selected types of bacteria which include (*Escherichia coli, Klebsiella ssp.,Pseudomonas aeruginosa* and *Proteus ssp.*) as gram negative, (*Staphylococcus aureus, Staphylococcus epidermidis, Streptococcus*) as gram positive and against fungus (Candida albicans) by agar diffusion technique. All isolates were streaked on Mueller Hinton Agar (MHA). The MHA plates were prepared by pouring 15ml of molten media into sterile Petri dishes. The Plates were allowed to solidify for 10 minutes. All isolates were suspended in sterile water and diluted to 107 CFU/ml. The suspension (100 µl) was spread onto the surface of MHA. Wells (6 mm) are aseptically punched on the agar using a sterile cork borer allowing at least 30 mm between adjacent wells, plates were incubated at 37 C° for 24 hours (1).The anti-microbial activity was evaluated by measuring the diameter of the inhibition zone formed around the wells [23].

Result and Discussion

Influence of operating variables:

The effect of different operating conditions (biomass dose, initial metal ion concentration, temperature, contact time and pH) on the removal of Co (III) ions by adsorption onto Sour orange waste peels were investigated. The metal removal studies were illustrated graphically in Fig.(1,2,4,5,6) which showed their removals were strongly affected by these different operating conditions:

Influence of Biomass dose:

To study the effect of citrus waste biomass dosage on the removal of Cobalt(III) ions different dosage in the range of 0.5 to 2.5 gram. As shown in Fig.(1) the biosorption capacity increased with the increase of biosorbent dose up to (2 gm) then there was a decrease in Co(III) ions removal percentage this was due to increased surface area of the biosorbent,which in turn increased the number of binding sites [24].

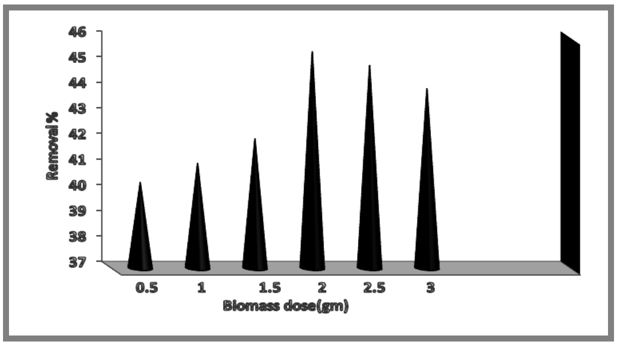

Fig.(1) Influence of biomass dose variation on Co (III) ions removal percentage.

Influence of Initial metal ion concentration:

The influence of Initial metal ion concentration plays an important role in the process of removal of pollutants from water and waste water by adsorption. Adsorption experiments were carried out at different concentration ranging from 2 to 100 ppm as cited in Fig.(2). It was observed as a general trend that there is a decrease in removal percentage with increase in initial concentration (the higher removal percentage was at 10 ppm) this may be attributed to the greater number of ions in solution thus leading to desorption of the metal ions from the binding sites of adsorbent particals.Wherease with limited number of ions there is limited number of collisions between the metal ions that is why low concentration of metal ions showed higher adsorption rate [25].

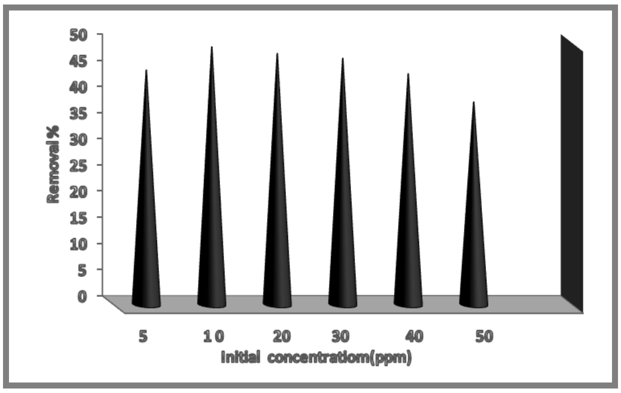

Fig.(2) Influence of initial concentration variation on Co (III) ions removal percentage.

The equilibrium adsorption isotherms are one of the promising data to understand the mechanisium of the adsorption. Numerous theoretical equations where used including the Langmuir equation which represents one of the first empirical equations of non-linear sorption and assume that uptake occurs on a homogenous surface by monolayer sorption without interaction between adsorbed molecules. The mathematical description of this model is as follow [26]:

$$\frac{Ce}{Qe} = \frac{1}{ab} + \frac{Ce}{a}$$

Where Ce is the equilibrium concentration of Co (III) in solution (ppm), Qe is the amount of Co (III) adsorbed in mg/g,a and b are the Langmuir isotherm constant.

The other isothermal equation is Freundlich equation which was applied for non ideal sorption onto heterogeneous surface and multilayer sorption of the sorbent. This model is expressed by [26]:

$$\text{Log Qe} = \text{Log } K_f + \frac{1}{n}\log \text{Ce}$$

Where Ce and Qe are the same as in Langmuir equation, K_f and n represent the Freundlich constants.

The linearization of Langmuir and Freundlich isothermals for the biosorbent from Sour orange peels are presented in Fig.(3) repectively. The model parameters are tabled in Table (1). The Freundlich equation was found to be more suitable than Langmuir equation depending on the regression coefficient obtained from these models as the appropriate criteria. The linearity of Freundlich plot suggested the formation of homogenous monolayer of Co(III) ions on the outer surface of the sorbent, the adsorption coefficient K_f of Co(III) ion on Sour orange peels was found to be (53.296) L/gm(the higher K_f values indicate that the saturation time for adsorption of metal ion is attained quickly due to high affinity of Sour orange peels towards adsorbate) and the value of n was (0.935) mg/g for Co(III) ions (the high value of n signifies that the forces which are exerted on the surface of Sour orange peels during metal ion adsorption are strong [27].

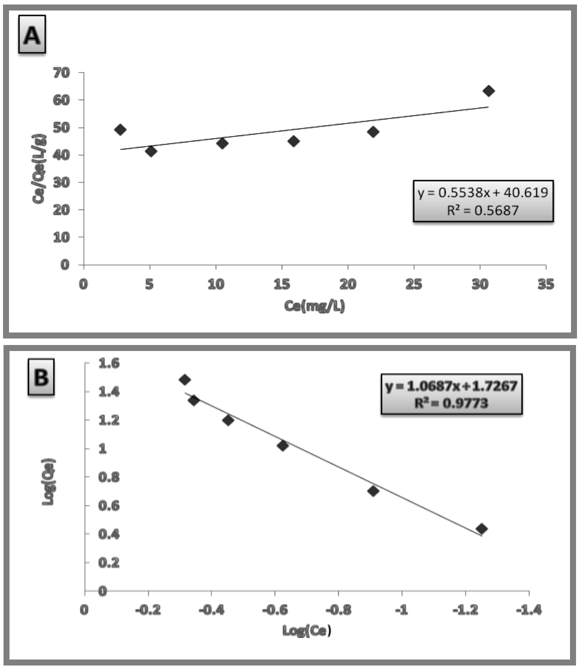

Fig.(3) Langmuir(A) and Freundlich (B) isotherms plots for the biosorption of metal ions by SOR peels.

Table (1)
Parameters of Langmuir and Frenundlish adsorption isotherm models for Co(III) ions adsorbed by SOR peels.

Langmuir isotherm parameters			*Frenundlish isotherm parameters*		
a	b	R^2	n	K_f	R^2
1.808	0.0136	0.5687	0.935	53.296	0.9773

Influence of Temperature:

The influence of temperature on the biosorption capacity of citrus waste biomass was studied within the temperature range 20 to 60. As can be seen from the Fig.(4), the biosorption capacity decreased with increase in the temperature, indicating that the biosorption of Co(III) ions on the citrus waste biomass was exothermic. Maximum biosorption capacity was observed at 30 C°. The decrease in biosorption capacity with increasing temperature might be due to the decreased surface activity. Moreover, at higher temperature the thickness of the boundary layer decrease due to increased tendency of the metal ion to escape from metal ion to escape from metal surface to the solution phases, which results in a decrease in the biosorption capacity [28, 29].

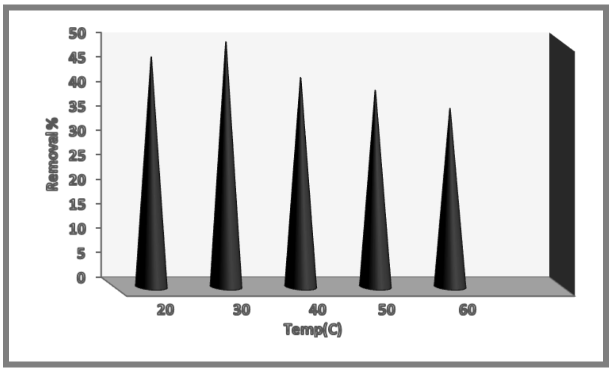

Fig.(4) Influence of Temperature variation on Co (III) ions removal percentage.

Influence of contact time:

Equilibrium time is fundamental parameters for an economical waste water treatment system. Consequently it is important to study its influence on the capacity of retention of Cobalt (III) ions by citrus biomass over time intervals from 30 to 240 min. Fig.(5) shows that the amount of Cobalt (III) ions adsorbed by the substrate initially increased with contact time up to 120 min., therefore, in subsequent experiments 120 min. was deemed more than sufficient to establish equilibrium and used in all subsequent study.

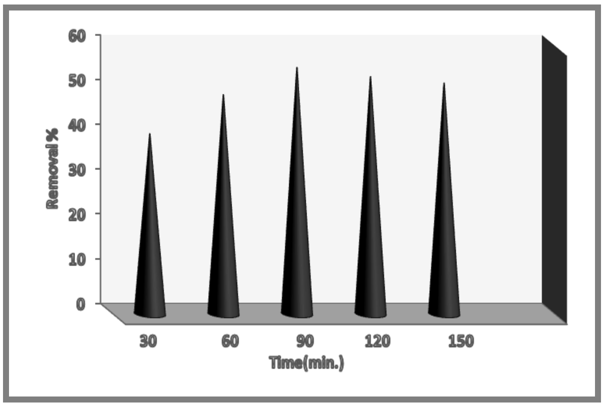

Fig.(5) Influence of contact time variation on Co (III) ions removal percentage.

Influence of pH:

The result of the pH study shows that maximum sorption occurred at pH = 2 for Cobalt (III) metal ions as shown in Fig.(6). The decrease in the adsorption with increase of pH may be due to the decrease in electrostatic force of attraction between the sorbent and sorbate ions.

At lower pH ranges, due to the high electrostatic force of attraction, the percentage of Cobalt (III) ion removal is high. At very low pH value, the surface of sorbent would also be surrounded by the hydronium ions which enhance the Co (III) interaction with binding sites of the adsorbent by greater attractive forces. Sharp decrease in adsorbents above pH 2 may be due to occupation of the adsorption sites by anionic species which retards the approach of such ions further towards the sorbent surfsce. The decrease in adsorption at higher pH values may be due to the competitiveness of the oxy anion of Co (III) and OH ions in the bulk. As the pH increased, the overall surface charge on the citrus peels became negative and adsorption decrease [25].

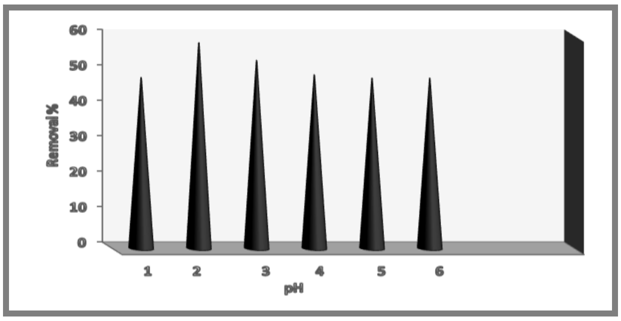

Fig.(6) Influence of pH variation on Co (III) ions removal percentage.

Desorption and Recovery:

Efficient desorption of the Cobalt (III) ions loaded biosorbent for repeated use is an important attribute for any successful development of a continuous flow effluent treatment system. Desorption studies were examine using different desorption agents, and as shown in Fig.(7), 0.1 M NH_4Cl has the highest desorption percentage (95.12%), followed by Sodium chloride (89.02%), EDTA(81.30), Hydroc-hloric acid (74.79 %) and Calcium chloride has the lowest desorption percentage (59.75 %). Thus 0.1 M NH_4Cl is considered as the most promising desorbent.

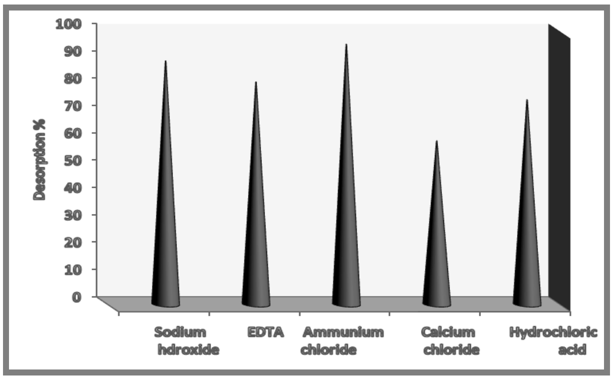

Fig.(7) Recovery of Co(III) by different desorption agent.

In order to examine the reusability of the biomass, the adsorption-desorption cycle was repeated four times using the same biomass. The obtained results show that after the first cycle the removal efficiency by biosorption decreased (12%) and remained approximately constant for all consecutive cycles Fig.(8). These results showed that sour orange residue can be repeatedly used in biosorption process without detectable loss in the total removal efficiency.

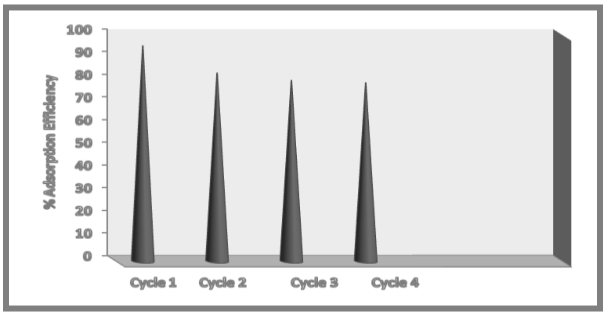

Fig.(8) Cyclic study of Co(III) adsorption-desorption on citrus waste biomass.

FT-IR characterization:

The FT-IR spectra of citrus waste biomass was recorded before and after Co(III) ions adsorption in order to ascertain the excesstance of the biomass surface functional groups and its binding sites with metal ion. These spectra were obtained from scanning in the range of 400-4000 cm^{-1}. The spectrum shown in Fig.(9) have a typical citrus peel peaks at 3416.05 cm^{-1} correspond to O-H vibration, 2929.00 cm^{-1} peak which can be attributed to the C-H stretching vibrations of CH,CH_2,CH_3 groups. The peaks at 1734.06, 1630.87 and 1617.37 cm^{-1}in the Sour orange peels spectrum indicate the carboxylic acid (C=O) groups and carboxylate ion stretching band (COO^-), respectively. The appearance of a peak at 1325.14 cm^{-1} denotes the presence of carboxylic acid salt also the band at 1064.74 cm^{-1} and 1056.06 cm^{-1} can be assigned to the (C–O) stretching of carboxylic acids and alcohols.

FT-IR Comparison between original Sour orange peels and Sour orange peels after Co(III) ions binding revealed the overall reduction in intensity of peaks confirming that these functional moieties were involved in the biosorption of Co(III)ions on Sour orange peels biomass [5].

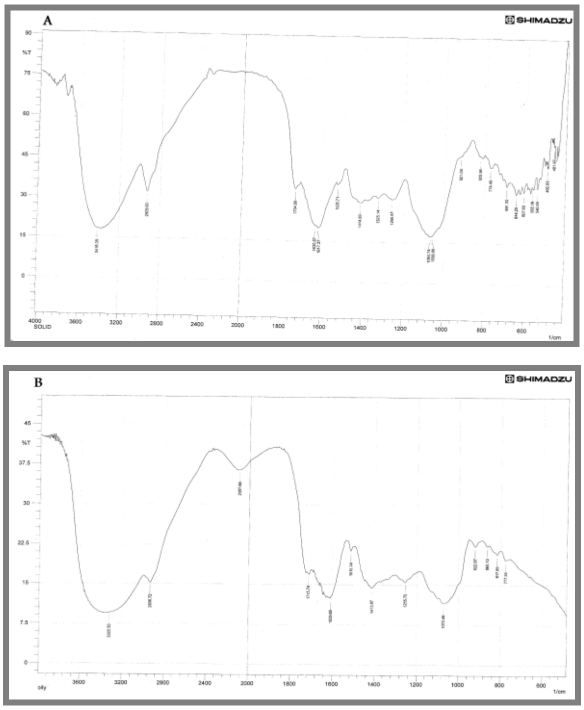

Fig.(9) Sour orange peels FT-IR chart before(A) and after(B) biosorption.

Fruit parts comparative studies:

In this study, Sour orange was selected for comparative studies on the removal of Co (III) ions from water and waste water in various parts of the fruit. Each fruit part: Flavedo (peel), Albedo (white inner skin), Segment membranes, juice and seeds were analyzed for Co (III) ions removal. Fig.(10) demonstrate the cross section of Sour orange indicating the Co (III) ions removal percentage in each part. The highest removal percentage was as follow: Albedo (57.99%), Juice (15.63%), Segments membranes (20.9%), Albedo (20.11%) and Seeds (10.06%) respectively. It is obvious that the choice of Sour orange peels was the best for this study due to high removal percentage.

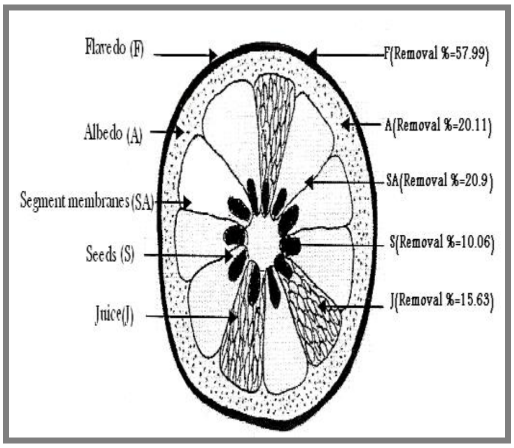

Fig.(10) Cross section of SOR showing Removal percentage of Co(III) ions.

Biological activity:

For antimicrobial activity of seven different isolates of gram negative and gram positive bacteria and fungus were tested. All isolates showed resistant to Sour orange peels and Sour orange peels with Co(III) ions except *Pseudomonas aeruginosa* isolate showed inhibition zone (12mm) against Sour orange peels and (16mm) inhibition zone against Sour orange peels with Co(III)ions as indicated in Fig.(11), which agree with a study performed earlier found that the Citrus peel oils showed antimicrobial activity against the test organisms including *Pseudomonas aeruginosa* [30].

Fig.(11) Inhibition zone of Citrus aurantium(SOR)peels before and after biosorption.

Conclusion

The results of present study indicating that: i) easy and simple pretreatment of the inexpensive biosorbent consist of SOR peels biomass show high-sorption performance of Co(III) ions and feasibility of desorption and recovery sorption capacity. ii) a comparison of various SOR parts revealed that SOR peels is perfectly preferred due to highest Co(III) ions removal percentage data compared with other parts. iii). The interaction between Co(III) ions and functional groups on the cell wall surface of the biosorbent cells were confirmed by FT-IR analysis and the spectra showed that carboxyl and hydroxyl groups were involved in Co(III) binding to the SOR peels. iiii) *Pseudomonas aeruginosa* isolate has a bioactivity against SOR peels loaded with Co(III) ions, this was concluded from inhibition zone.

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الخلاصة

تم دراسة قشور النارنج كسطح ماز لازالة ايون الكوبلت الثلاثي من النظام البيئي المائي بتطبيق تقنية الدفعات.تم دراسة الظروف المثلى وكانت النتائج كالتالي:وزن السطح الماز (٢ غرام)، التركيز الابتدائي لايون الكوبلت الثلاثي (٥ مليغرام/لتر)، درجة الحرارة (٣٠ درجة سليزية)، زمن التماس (٩٠ دقيقة) و الدالة الحامضية الامثل كانت (٢)، كذلك تم أجراء تجارب لبيان قابلية الاستخدام المتكرر لقشور النارنج لما لها من اهمية تنافسية مالية مع بقية المواد ووجد ان (٠,١ مولاري) كلورايد الامونيوم من افضل المواد المسترجعة للعنصر الثقيل، كما وعند تشخيص قشور النارنج قبل وبعد الامتزاز بتقنية تحول فوريه للاشعة تحت الحمراء دلت النتائج على ارتباط مجاميع الهيدروكسيل والكاربوكسيل مع ايونات الكوبلت الثلاثية.اما الاجزاء المختلفة للنارنج فقد تم اختياره لدراسات مقارنة حول الافضلية في ازالة العنصر الثقيل (الكوبلت) مشيرة النتائج الى افضلية القشور على بقية اجزاء النارنج، وقد وجد ان لقشور النارنج فعالية بايولوجية ضد *Pseudomonas aeruginosa*. يمكن االاستنتاج من هذه الدراسة ان قشور النارنج يمكن ان تستخدم بفعالية في معالجة النظام البيئي المائي من ايونات العناصر الثقيلة (ايونات الكوبلت الثلاثية).