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ABSTRACT

The discharge of toxic metals from industries is a major environmental problem. It is, therefore, crucial to find efficient methods for their removal. The present study analysed the *Lagenaria siceraria* (LS) peel biomass for the elimination of chromium, cobalt, copper, iron, mercury, silver and zinc from wastewaters. Formaldehyde treated and untreated biomass was also tested. The ability of the biomass to adsorb metal ions was found to depend on pH, the original concentration of ions, and the biomass treatment. With untreated biomass, the order of adsorption was copper > zinc > cobalt > iron > silver > chromium > mercury, with treated biomass, the order was copper > mercury > iron > chromium > cobalt > silver > zinc, and at pH 4–5, the order was copper > iron > cobalt > chromium > silver > zinc > mercury. The biomass had remarkable affinity for copper under all the conditions. Since LS is a vegetable cultivated on a large scale, adsorption strategies based on its biomass are expected to be convenient, eco-friendly and cost effective.

KEYWORDS

Wastewater; effluents; metals; biosorbent; *Lagenaria siceraria*

Introduction

Many industrial effluents contain hazardous metals. It is therefore necessary to investigate safe and cost effective methods for the removal of toxic metals from contaminated waters and soils [1]. There is a wide range of concentrations of heavy metals in industrial waters and contaminated soils [2]. These heavy metals have adverse effects on the natural environment and human health [3]. They also affect the production capacity of soil [4]. These metals have diverse origins. Zinc (Zn) has applications in many industries including tyre-tread materials [5]. It can be released into the air and contaminate water and soil. It affects eyesight and memory [6]. Refineries are a major source of cobalt (Co), which is unsafe for health and causes disorders related to lungs and heart including carcinogenic diseases [7]. Chromium (Cr) is released from tanneries in large amounts and pollutes water and soils. Prolonged exposure adversely affects the human body and can destroy lungs, kidney, heart, brain, liver, eyes, nasal cavity, skin, teeth and eardrums [8]. Excessive amounts of iron (Fe) in water are also harmful. It attacks joints, liver and heart and can cause gene mutations [9]. Copper (Cu) is widely used for electroplating, electrical wires and electrical cells. Small

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amounts can seriously affect metabolism causing various disorders such as growth inhibition and anaemia [10]. Mercury (Hg) is a highly toxic metal, which is present in the environment either as the free element or as highly toxic organic compounds as well as inorganic compounds [11]. It can cause serious health issues including hypertension, hearing and vision problems, and Minamata disease [12,13]. It has a very low excretion rate from the body which makes it a matter of further concern [14]. Silver (Ag) is found in the form of salts in wastewaters from various industries. Its toxic effects include impairment of tissues, stomach and lungs, and creation of grey and black spots on skin [15].

Several different methods, both physical and chemical, are used to remove these contaminant metals from polluted waters and soils. Adsorption by a suitable adsorbent is a very common method for removal of heavy metals from contaminated waters [1]. The use of plant based biosorbent for the removal of toxic metals from contaminated waters is being extensively studied [16,17]. The present study investigates *Lagenaria siceraria* (LS) as a possible biosorbent for various toxic metals. The plant is easily available in abundance as it is a common vegetable and is cultivated on a large scale. In this research task, the effect of pH, adsorbent dose, contact time, and initial metal concentration on adsorption efficiency were studied.

Materials and methods

Reagents

Various chemicals used for the study were of analytical grade. HCl and KOH were used to adjust the pH of metal ion solutions, while HNO_3 and HCHO were used to treat the biomass. Distilled water was used to prepare standardised solutions of metal salts including $AgNO_3$, $CoCl_2$, $HgCl_2$, $CrCl_3$, $CuSO_4$, $ZnCl_2$ and $FeCl_2$.

Biomass preparation

Fruit of LS was purchased from an agricultural farm of Pattoki (Pakistan). The specimens were washed with distilled water and dried with a piece of muslin cloth. The peel of the fruit was separated carefully with the help of a knife. It was allowed to dry under shade for 15 days. The dried material was crushed and ground to obtain a powder. A weighed amount of the powder was macerated in methanol for two weeks. Then, it was filtered to remove the methanol soluble components. The residue was washed with methanol twice and dried in an oven at 80 °C for one day. The resulted residual biomass of the peel of LS was ground with a domestic grinder to obtain a fine powder, which was stored in airtight polyethylene bags until used for the adsorption study.

Biomass treatment

A method reported by Alves et al. was used for treatment of biomass [18]. An amount of 5 g LS peel biomass powder was mixed with 150 mL nitric acid (3%) and 0.125 mL formaldehyde (40%). The mixture was boiled in a water bath for 15 min at 100 °C. Afterward, the biomass mixture was allowed to cool to room temperature. It was filtered and the residue

(treated biomass) was washed with distilled water. Washing was continued until the pH of the residue came to 7 [19]. This is referred to as treated biomass in this paper.

Determination of metal content in solutions

The concentration of each of the metal ions before and after adsorption on the biomass was determined with an atomic absorption spectrophotometer (AAS; Varian AA240FS). Standard solutions of the metals salts were prepared separately. They were brought in contact with the biomass by soaking for certain periods of time. The concentration of each metal absorbed, C_t, was calculated with the help of the following formula:

$$C_t = C_i - C_e$$

where, C_i is the initial concentration of a metal, while C_e is its equilibrium concentration after the adsorption has taken place. To determine the adsorption capacity, the following formula was used:

$$q = \frac{V(C_i - C_e)}{W}$$

where, q is adsorption capacity, V is volume of the metal ion solution and W is mass of biomass taken.

Preparation of metal ion solutions

A quantity of 1000 ppm stock solution of each metal salt (500 mL) was, separately, prepared in distilled water. Five dilutions (100 mL each) of each metal salt were prepared with concentrations 2, 4, 6, 8, and 10 ppm. Silver solutions were preserved in bottles covered with aluminium foils.

Untreated method (Method 1)

To 100 mL of a salt solution, 0.2 g untreated LS biomass was added, and mixed to obtain a suspension. The mixture was agitated on an orbital shaker for 16 h. The biomass was then filtered with Whatman filter paper 42 and the filtrate was used to analyse the quantity of metal ion using AAS. The same procedure was followed for all the dilutions of each of the salt solutions used for the study.

pH adjustment method (Method 2)

Five dilutions of stock solution were prepared to yield 2, 4, 6, 8 and 10 ppm of each metal in 100-mL volumetric flasks. The pH of each solution was adjusted to be 4–5 by adding 0.1 M HCl or 0.1 M KOH. Subsequently, 0.2 g (adsorption dose) LS were added. The solutions were shaken on an orbital shaker for 16 h. They were then filtered with Whatman filter paper 42. The adsorption capacity of LS biomass was determined.

Treated method (Method 3)

Five dilutions of each metal salt were prepared with concentrations of 2, 4, 6, 8 and 10 ppm. Then, 0.2 g of treated biomass was added to each of the solutions. Each mixture was agitated on an orbital shaker for 16 h. After agitation, the mixtures were filtered and the filtrates analysed to determine the quantity of unadsorbed metal ions.

Statistical analysis

All determinations were conducted in triplicate and the statistical average was calculated with SEM. Microsoft Excel 2013 was used for calculations and data presentations.

Results and discussion

L. siceraria peel residual biomass was used as an adsorbent for the removal of seven heavy metals from water, viz., chromium, copper, cobalt, iron, mercury, silver and zinc (Figures 1–3). Three methods were employed, namely, untreated method without pH adjustment (method 1), untreated method with pH adjustment (method 2), and treated method without pH adjustment (method 3).The study was planned to evaluate the usefulness of the *L. siceraria* biomass for the removal of the above-mentioned heavy metals from contaminated waters. It was a laboratory scale study using standard single metal solutions. Actual polluted waters would be more complex since they would be expected to contain a variety of organic and inorganic pollutants which would possibly be competing for the adsorbent sites. The present investigation is valuable because it explored the adsorption ability of the metals under study for the *L. siceraria* biomass using a number of variables.

The results indicated that overall copper showed maximum adsorption (99%), followed by iron and silver metal ions with 95% adsorption. Zinc displayed good adsorption in the untreated method in a dose dependent manner. The adsorption of silver, iron, copper, and cobalt was dose dependent in all the three methods. The adsorption behaviour of chromium was dose dependent in methods 2 and 3. Mercury displayed appreciable adsorption only in method 3. Zinc in methods 2 and 3, and mercury in method 1 and 2 did not show any adsorption. This may be because of pH change and interaction of functional groups in the biomass such as hydroxyls and carbonyls, which are known to have a strong attraction for metal ions [20].

Biosorbents from plant sources are considered to be promising adsorbents to remove heavy metals from waste water. Numerous studies have been conducted on the biomasses of different origins. They include the biomass of pomegranate [21], rice husk, straw, fly ash, bamboo dust, coconut shell, coal sawdust, Babhul bark, rice husk ash, boiler bottom ash, wood coal, corncob carbon, coconut oil cake and peel of orange and banana [22–24]. Nevertheless, adsorption is still not in common use for the treatment of industrial effluents. The reasons include the difficulty in separating adsorbates from adsorbents, and the high capital cost [25]. According to the United States Environmental Protection Agency (USEPA), 13 of the most common trace metals present in polluted water are Ag, As, Be, Cd, Cu, Cr, Hg, Ni, Pb, Sb, Se, Tl, and Zn [26], which must be removed through viable strategies.
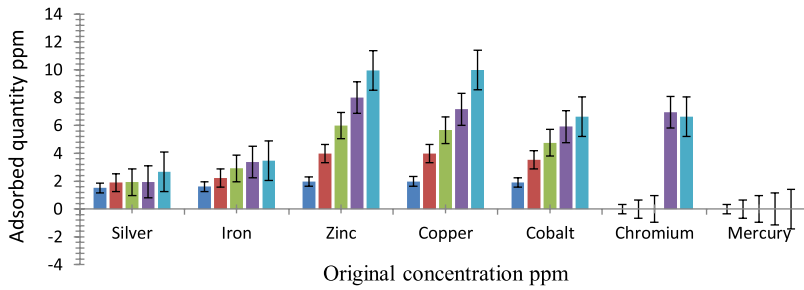
Figure 1. Adsorption of metals under untreated biomass of *L. siceraria* (method 1). Original concentrations for each metal were from left to right 2, 4, 6, 8 and 10 ppm.
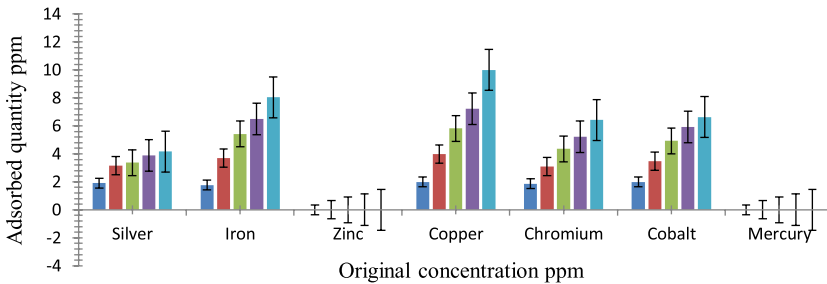
Figure 2. Adsorption of metals by *L. siceraria* biomass when pH of salt solutions was adjusted to 4–5. Original concentrations for each metal were from left to right 2, 4, 6, 8 and 10 ppm.

## Analysis of metal ions adsorption under method 1

LS peel residual biomass was used for the removal of metal ions from their solutions and the results are shown in Figure 1.

Method 1 employed untreated biomass. For most metals, adsorption was dose dependent and increased with the increase of concentration of the given salt. Copper showed the highest adsorption followed by zinc. The biomass was also efficient for cobalt in a dose dependent manner. For chromium the method was successful only at high concentrations. Mercury showed no affinity for the biomass under this method. The method may be recommended to remove copper and zinc from polluted waters.

Analysis of metal ions adsorption under method 2

Removal of metals by LS biomass was evaluated by method 2 (untreated biomass and salt solutions adjusted to pH 4–5). Results are shown in Figure 2.

Figure 2 shows a comparison of the adsorption of metals by untreated biomass when the pH of salt solutions was adjusted to be 4–5. The pH of a salt solution is an important factor to affect adsorption of the salt's ions on an adsorbent because it affects the degree of ionisation of the salt [27,28]. Under the conditions used, copper displayed excellent adsorption followed by iron and cobalt. For copper, this method was as efficient as method 1. This method was also good for chromium in a dose dependent manner. Significantly, the extraction of metal ions increased progressively with the increase in concentration of a salt,
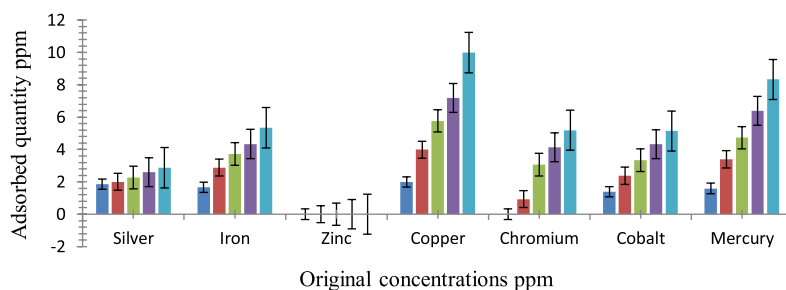
Figure 3. Adsorption of metals on treated biomass (method 3).Original concentrations for each metal were from left to right 2, 4, 6, 8 and 10 ppm.

which is a common phenomenon [29]. Interestingly, the method totally failed to remove zinc, which exhibited excellent affinity for the biomass under the conditions of method 1.

Under the condition of acidic pH, adsorption of a metal ion on the biomass competes with the protonation of the biomass. The lower adsorption of Zn(II) on the biomass at low pH might therefore be caused by the higher affinity of the biomass for hydrogen ions in comparison to the metal ion at equilibrium [30,31]. For mercury, this method proved to be ineffective. At low pH, a large amount of H$^+$ ions was present in solution which protonated the functional groups of the biomass and rendered them unavailable for metal ions. It has been suggested that low or no adsorption of ions occurs on biomass at low pH for many metals [32,33].

Analysis of metal ion adsorption using method 3

LS biomass was treated with nitric acid and formaldehyde according to a method reported in the literature [18]. The treated biomass was then used for the removal of metal ions from their salts. The results are shown Figure 3.

Comparison of adsorption of metals under the treated biomass method (method 3) revealed that in this method also copper ions exhibited maximal adsorption. The finding indicated that copper has very high affinity for LS peel biomass, which remained almost constant with all the three methods. Method 3 proved to be very effective for mercury as well, where it showed very good adsorption in contrast to methods 1 and 2, which displayed no adsorption. Method 3 was also efficient for iron, cobalt and chromium in a dose dependent manner. Zinc did not show adsorption under this method. Silver, the only uni-positive metal used in the present study, showed medium but consistent affinity for LS biomass under all the conditions.

The present study through various methods of investigation demonstrated LS residual peel biomass to be highly effective for the removal of heavy metals via method 2 (pH variation method with untreated biomass). A pH of 4–5 worked best for the removal of heavy metal contaminants using our experimental methods. In methods 1 and 2, the biomass was used without treatment; but in method 3, it was used after pre-treatment. The pre-treatment was done to remove soluble organic substances from the biomass and increase its porosity to enhance its efficiency [34].

Based on the results of the study, method 2 findings were selected for thermodynamic analysis.
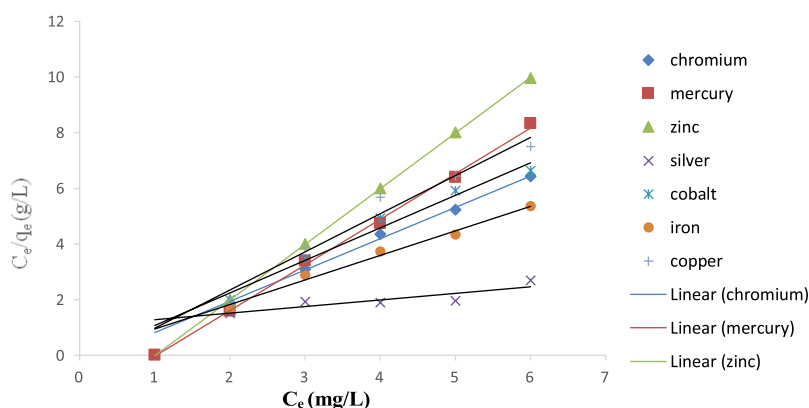
Figure 4. Langmuir isotherm model for the biosorption of metals by *Lagenaria siceraria* peel biomass.

Adsorption isotherms

The adsorption phenomena are generally explained through adsorption isotherms. Langmuir and Freundlich isotherms are commonly used for this purpose. With the Langmuir isotherm, adsorption is considered to be a surface phenomenon that occurs only at the first layer of an adsorbent. The linear Langmuir isotherm model is expressed in the following equation:

$$\frac{C_e}{q_e} = \frac{1}{q_{max}K_L} + \frac{C_e}{q_{max}}$$

where, q_e is the amount of a metal adsorbed on the adsorbent at equilibrium (mg/g), C_e is the equilibrium concentration of the metal (mg/L), q_{max} represents maximum adsorption capacity of the adsorbent (mg/g) and K_L is Langmuir adsorption constant (mg/L). The values of all these parameters are determined from graphs (Figure 4).

In the Freundlich isotherm, the adsorption process is thought to occur at many layers of an adsorbent. The isotherm is expressed by the following equation:

$$\log q_e = \frac{1}{n} \log C_e + \log K$$

where K and n are Freundlich constants. The Freundlich isotherms are shown in Figure 5.

The parameters of Langmuir and Freundlich isotherms calculated according to the equations given above are presented in Tables 1 and Table 2. From the obtained values of K, q_{max} and R^2, it can be concluded that, in general, the two models are comparable. According to the results, the Langmuir isotherm better approximates the adsorption of many heavy metals. As Table 1 shows, the values of the constant for the Langmuir isotherm, K_L, for these metals were in the order of Ag > Cu > Co > Cr > Fe > Zn > Hg. The trend indicated that silver had the highest binding affinity with the surface of this biomass and mercury the least. The value of q_{max}, which represents the saturated monolayer sorption capacity, is highest for iron, followed by chromium, followed by cobalt. It is low for silver and zinc.
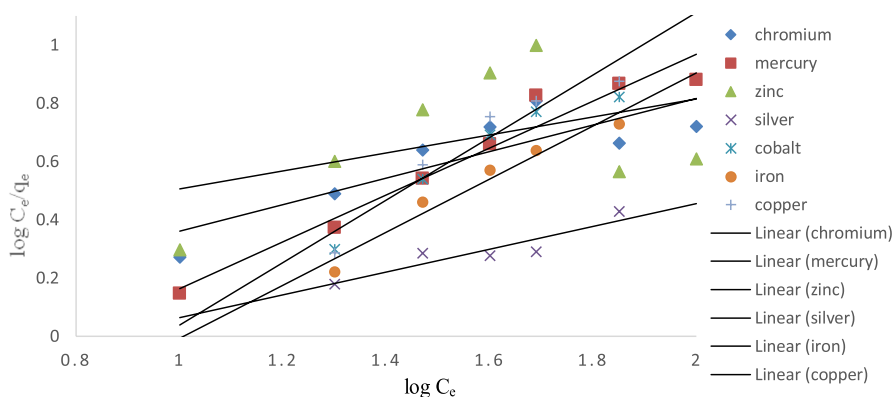
Figure 5. Freundlich isotherm model for the biosorption of metals by *Lagenaria siceraria* peel biomass.

Table 1. Parameters obtained using the Langmuir isotherm.

	Metals						
Parameters	Cu	Co	Zn	Cr	Hg	Ag	Fe
q_{max}	7.340	8.547	5.032	8.927	6.057	4.347	11.363
K_L	0.134	0.124	0.004	0.091	0.009	0.294	0.083
R^2	0.968	0.979	1.003	0.996	0.996	0.770	0.944

Table 2. Parameters obtained using the Freundlich isotherm.

	Metals						
Parameters	Cu	Co	Zn	Cr	Hg	Ag	Fe
K	1.625	1.753	1.565	2.182	4.365	5.051	2.792
$1/n$	0.589	0.603	0.614	0.646	0.805	0.746	0.686
R^2	0.842	0.934	0.743	0.962	0.954	0.740	0.964

As the values of R^2 (coefficient of determination) show, the Langmuir isotherm explained well the adsorption of zinc, chromium, mercury, cobalt, copper and iron. Hence, these metals, most probably, involve a monolayer adsorption process as predicted by the Langmuir isotherm. On the other hand, the Freundlich isotherm successfully explained the adsorption of iron, chromium, cobalt, mercury and copper. As Table 2 indicates, the Freundlich isotherm successfully applies to the adsorption of iron chromium and mercury as suggested by their R^2 values. The adsorption of silver and zinc was less adequately explained. Silver presents a unique case. Its adsorption was not explained by either of the isotherms. The order of the values of K was Ag > Hg > Fe > Cr > Co > Cu > Zn indicating how tightly the metal ions are associated with the biomass. Copper, cobalt and zinc are predicted to have similar affinity.

Since R^2 is an important parameter that indicates which model is fitted best, we may conclude that most of these metals predominantly follow an adsorption mechanism predicted by the Langmuir equation. This is particularly true for zinc, chromium and mercury. The sorption of iron, however, is better explained by the Freundlich isotherm.

Conclusions

Lagenaria siceraria peel biomass can be used to remove different heavy metals from polluted waters. The adsorption efficiency depends on various parameters, and was highest for copper under all the conditions used in this study. Untreated biomass was also efficient for zinc, while treated biomass showed excellent affinity for mercury as well. As the plant is grown on a large scale, its use as a biosorbent for removal of toxic metals from industrial discharge would provide an efficient low cost adsorbent material.

Authors' contributions

DA conceived of the study, provided basic guidance and oversaw the submission of the resulting manuscript; HA supervised the research and prepared the manuscript; AR conducted the research and presented the data for the first draft of the manuscript. All authors read and approved the manuscript.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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