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Adsorption of Heavy Metals from Industrial Wastewater in Ranipet District using Agricultural Wastes as Biosorbents

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Abstract: *The contamination of the natural environment by heavy metal is an emerging problem in the world. Remediation of these contaminants from our environment is posing a technological challenge for us. Various biosorbents were examined and their maximum adsorption capacities for lead(II) and nickel(II) usually found in Industrial wastewater samples from Common Effluent Treatment Plant. The results emphasized that the primary adsorption mechanism is the complexation of metal ions at the biosorbent surfaces. Adsorption Isotherm indicate that Pb(II) has higher adsorption property than Ni(II), on the selected biosorbents. Among the selected biosorbents, castor leaves (CL) collected from roadside possess higher adsorption capacities, which can be further developed as a lead(II) selective biosorbents*

Keywords*:* *natural environment*

I. INTRODUCTION

The contamination of the natural environment by heavy metal is an emerging problem in the world. Remediation of these contaminants from our environment is posing a technological challenge for us. According to the European Union's Restriction of Hazardous Substances (RoHS) Directive, application of lead, cadmium, and mercury in the manufacturing of electrical and electronic equipment are banned as they impart a hazardous effect on our health (CitationEuropean Parliament, 2002). These metal pollutants accumulate in the environment due to their non-biodegradable nature resulting in the contamination of food and water (CitationVries et al., 2007). Therefore, only permissible limits of these metal toxicants defined by World Health Organization (CitationWHO, 2008) are strictly allowed in the drinking water. Some heavy metals like nickel and lead are purely toxic and are frequently found in industrial wastewater. The permissible values for Ni in serum is 0.2 µg/L and in urine is 1–3 µg/L for a healthy adult, as defined by Agency for Toxic Substances and Disease Registry (ATSDR) of United States (CitationASTDR, 2005). The most adverse health effect associated with Ni exposure is dermatitis which occurs through dermal contact with Ni. Conventional methods employed for heavy metal remediation are solvent extraction, precipitation, coagulation, reverse osmosis, adsorption, chemical oxidation and reduction, electro-dialysis and ion-exchange (CitationMeunier et al., 2006; CitationDjedidi et al., 2009; CitationKurniawan et al., 2006; CitationLee et al., 2006) But at metal ion concentration of 100 ppm or less, these are costly and ineffective too (CitationInbaraj et al., 2009). Bioremediation is a newer approach for the eradication of these heavy metals (CitationGaur et al., 2014) in which the removal of metal toxicants employ application enzymes, microorganisms, and plants which are eco-friendly in nature. It is one of the most favourable methods as it offers better results by utilizing low-cost materials in comparison to the conventional expensive means. A large number of literature studies indicate that the bioremediation of heavy metal pollutants (CitationAksu, 2005) through agricultural wastes by-products and industrial waste products can be an alternative to conventional methods. In these materials, mechanism of contaminant removal through biosorption process is based on ion exchange, surface complexation, adsorption, absorption, and precipitation. Application of biosorption for the eradication of heavy metals (lead, arsenic, cadmium, uranium, mercury) along with dyes, phenolic compounds, and pesticides present in the aqueous wastewater has been

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carefully studied (CitationGadd, 1992). Frequently used biosorbents for the remediation of heavy metal toxicants in the solution are: agricultural wastes, including fruit peels, straw, coconut coir, and so on (CitationRocha et al., 2009; CitationZheng et al., 2009; CitationReddy et al., 2011) and microorganisms, such as bacteria, yeasts, fungi, and algae which are attached with mediums to form biosorbents (CitationDeng et al., 2006, Citation2007; CitationDursun, 2006; CitationVilar et al., 2007; CitationHanif et al., 2007; CitationPreetha and Viruthagiri, 2007; CitationBhainsa and D'Souza, 2008). As the non-living biomass does not require any special care for its storage and application, it is preferred more than the living biomass for the biosorption process. Additionally, the biomass is obtained from industrial waste which makes the process cost-effective. Biosorbents from agricultural wastes can be easily obtained through a simple pretreatment process and they frequently possess specific functional groups, such as –OH or –COOH, which may generate adsorption affinity toward heavy metal ions. Therefore, the surface functional groups present on the biosorbent and the affinity of biosorbent to bind metal ions significantly affects the adsorption of metal contaminants (CitationChao and Chang, 2012).

In this study, fibrous materials from industrial waste i.e. sugarcane bagasse (SB), agricultural waste, i.e., rice husk (RH) and natural residues i.e. castor leaves (CL) have been selected to produce biosorbents. The adsorbents obtained after simple pretreatment were characterized using scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. The adsorption capacity for heavy metal ions (Pb^{+2}, Ni^{+2}) were studied. The results obtained can offer a reference to determine the appropriate biosorbents for heavy metal pollution abatement.

II. METHODS

2.1 Preparation of test solutions of heavy metal cations

The water soluble salts from CDH Fine Chemicals, India ($PbSO_4$, $NiCl_2$) were used to prepare a stock solution of test metal containing 1000 ppm of Pb(II) and Ni(II) separately in double distilled water. The experiment was performed using single test metal ions (Pb^{+2}/Ni^{+2}) to prevent competitive adsorption. The concentrations of test metal ions were always below 1000 mg/L to rule out any system error. All the solutions were stored at room temperature.

2.2 Preparation and characterization of adsorbents

The materials selected for the preparation of biosorbents consisted of, sugarcane bagasse (SB), rice husk (RH) and castor leaves (CL) shown in Fig. 1(A–C). They were collected from different places as waste material. Step-wise simple pretreatment process comprising drying, grinding, heating, and degreasing was employed on the raw materials, based on the characteristics of the materials and adsorbents were obtained. The flow chart of the preparation processes of SB, RH and CL is shown in Fig. 2. In brief, the dirt particles at the surfaces of materials were removed by washing with distilled water and the materials were then sun-dried until the water evaporated. The non-living masses were further dried in a 50 °C oven for 12 h and grounded to obtain uniform size particles which can pass through a 45 mesh filter. The obtained adsorbents were treated with a mixture of organic solvents [hexane:ethanol; 1:1 (V/V)] in a Soxhlet extraction to remove fine, greasy particles of adsorbents. After the removal of hydrophobic dirt with organic solvents, distilled water was used to rinse the adsorbents. It was further heated at 50 °C in the oven for overnight (12 h) for drying. The obtained biosorbent particles were stored in a brown bottle until use. SEM instrument (Model S-4800 microscope, Hitachi, Tokyo, Japan) and Fourier transform infrared spectroscopy (FTIR, Perkin-Elmer) were used to study the surface properties of the 3 biosorbents.

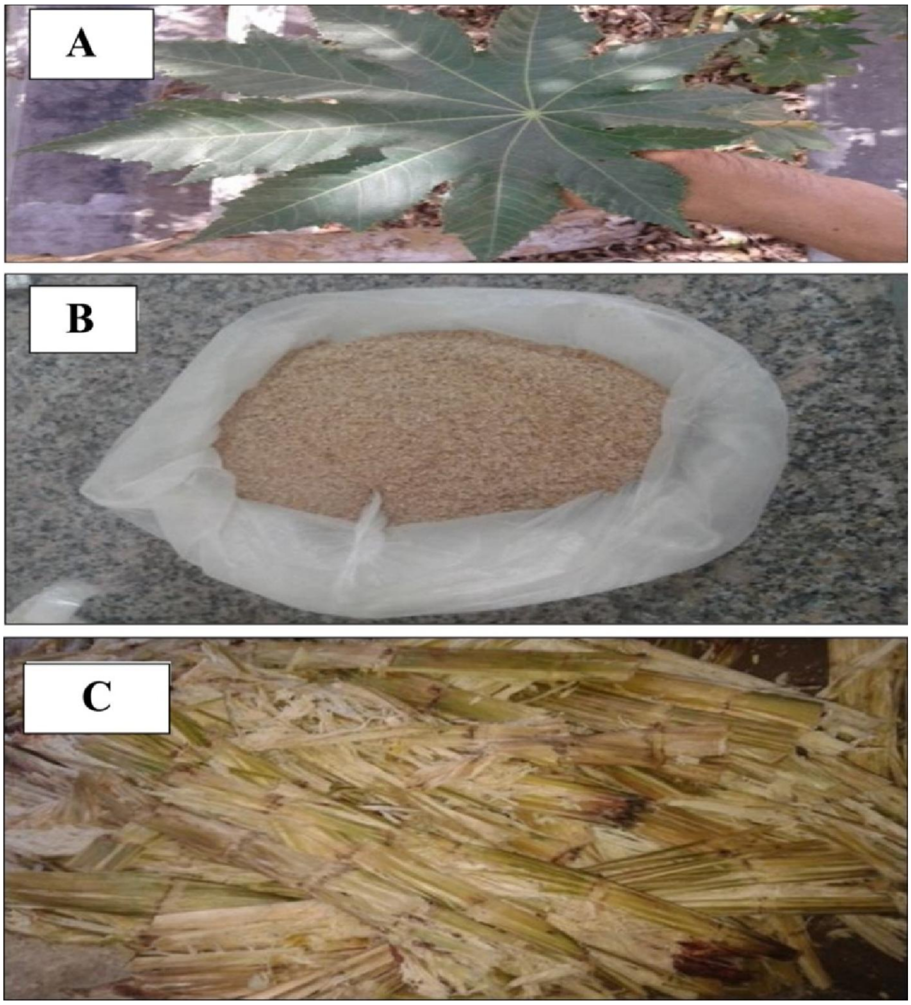

Fig. 1 Biosorbent in natural condition from the source: (A) fresh castor leaves, (B) rice husk, (C). sugarcane bagasse.

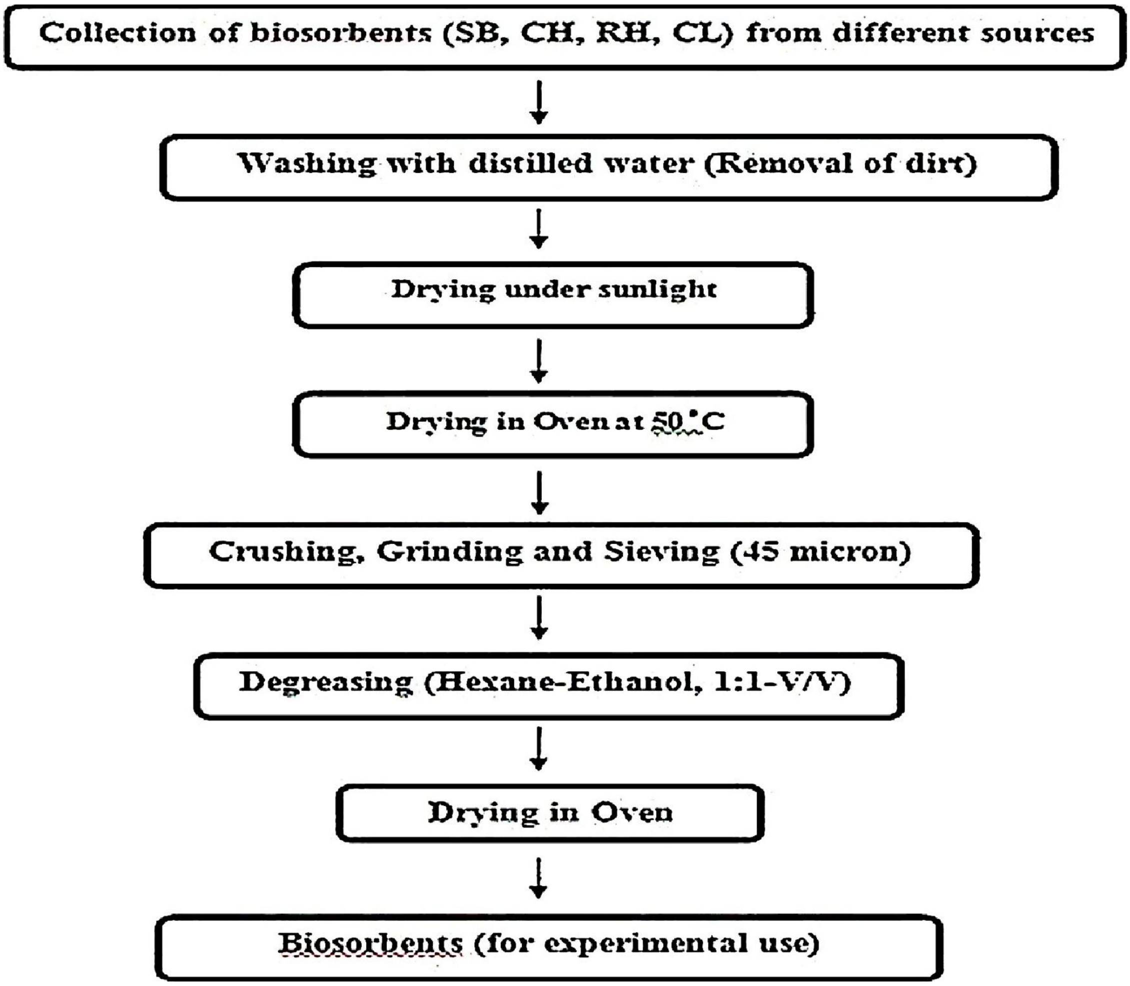

Fig. 2 Preparation process of biosorbents (flow chart).

2.3 Equilibrium experiments

A 0.2 g of non-living biosorbent was placed in a 25 mL Teflon centrifuge tube. Different concentrations of metal ion solutions (10 mL) were added into the tube. As reported in the literature, the heavy metal adsorption is pH dependent showing maximum adsorption capacities at pH 5–6 (CitationVaghetti et al., 2009). Hence, the pH of the solution was adjusted at 5.0 ± 0.1 before the adsorption process. The centrifuge tubes were agitated in an incubator-shaker at 200 rpm at 25 °C for 24 h to attain complete adsorption of metal ions. Each tube was further centrifuged for 30 min at 10,000 rpm to sediment heterogeneous mixture of adsorbent and adsorbate. Then it was passed through 0.40-µm filter. By using atomic absorption spectrometry, the concentrations of heavy metal ions present in the filtrate were analyzed. The amount of metals adsorbed on the biosorbent were calculated by the difference between the metal concentrations in the solution before and after equilibration. Each experiment was performed in triplicates and average was calculated. In the control experiments, no biosorbents were used in the adsorption process. The recoveries of metal ions in the control experiments ranged from 90 to 100%. The measured equilibrium concentrations were not adjusted for the recoveries. The maximum adsorption amount in the batch experiment is less than 80% of the initial concentrations of heavy metals in the solution. The characteristics of adsorbates and the adsorbents were described by the Freundlich and Langmuir adsorption equilibrium equations. The Freundlich equation can be expressed as in Eq. (Equation1):$Q=x/m=K\quad C1/n(1)$ where 'x' is the amount of the metal cations sorbing onto the biosorbent; m is the biosorbent weight; K is the equilibrium constant; C is the concentration of metal ions (mg/L) in the solution at equilibrium, and n is an empirical constant. When n is greater than 1, the isotherm is concave downward; when n is less than 1, the isotherm is concave upward; and when n was equal to 1, the isotherm is linear.

The Langmuir isotherm describes the adsorption of molecules or ions on a solid surface at fixed temperature. It is derived assuming a uniform surface with finite identical sites and monolayer adsorption of the adsorbate (CitationGokhale et al., 2008). The Langmuir equation can be expressed as in Eq. (Equation2):$Q=xm=K \quad b \quad C1+K \quad C(2)$ where x, m, and C are same as defined in Eq. (Equation1), K is the Langmuir adsorption constant (L/mg), and b is the maximum sorption capacity (mg/kg). When an adsorbate with a low concentration exists in the system, the Langmuir equation appears in the linear adsorption isotherm x/m = KHC. The KH is the Henry adsorption constant. The Henry adsorption constant is commonly used to indicate the affinity of an adsorbate towards a solid surface. Commonly, Freundlich and Langmuir's equations are applied to obtain the characteristics of an adsorbate relative to an adsorbent in solution but complex adsorption behavior may lead to inadequate data approximation. Herein, the adsorption characteristics of three different biosorbents are discussed according to the obtained isotherms and adsorption capacities.

III. RESULT AND DISCUSSION

3.1 Characterization of biosorbents

The bio sorbents obtained from different sources are shown in Fig. 1(A–C). After grinding and processing they were converted into powdery mass. In order to get insight into the morphology and dimensions of biosorbent particles, we examined the smears or films of materials by SEM. As can be seen from Fig. 3(A–C), castor leaf surface is very rough and some pores are visible, while the biosorbents RH and SB lack the pores and their surfaces are smooth. The fewer number of exchangeable cation sites are present at the non-porous biosorbents (RH, SB) which limit the adsorption of metal cations. The biosorbent surface might generate the exchangeable cation by the dissociation of a proton (H^+) or other cation from the functional groups present on the biosorbents under the specific pH condition. Additionally, functional groups, such as carboxyl (–COOH), amino (–NH_2), and hydroxyl (–OH) may be present at the surface of the biosorbents which can provide the site for complexation with the metal cations (CitationZheng et al., 2009). The oxygen or nitrogen atoms present at the surface functional groups can form a coordinate bond with the divalent metal cations by using non-bonding electrons. In other words, the presence of coordination sites or functional groups on the surfaces of biosorbents acts as a dominant factor in determining the adsorption capacities of these materials.

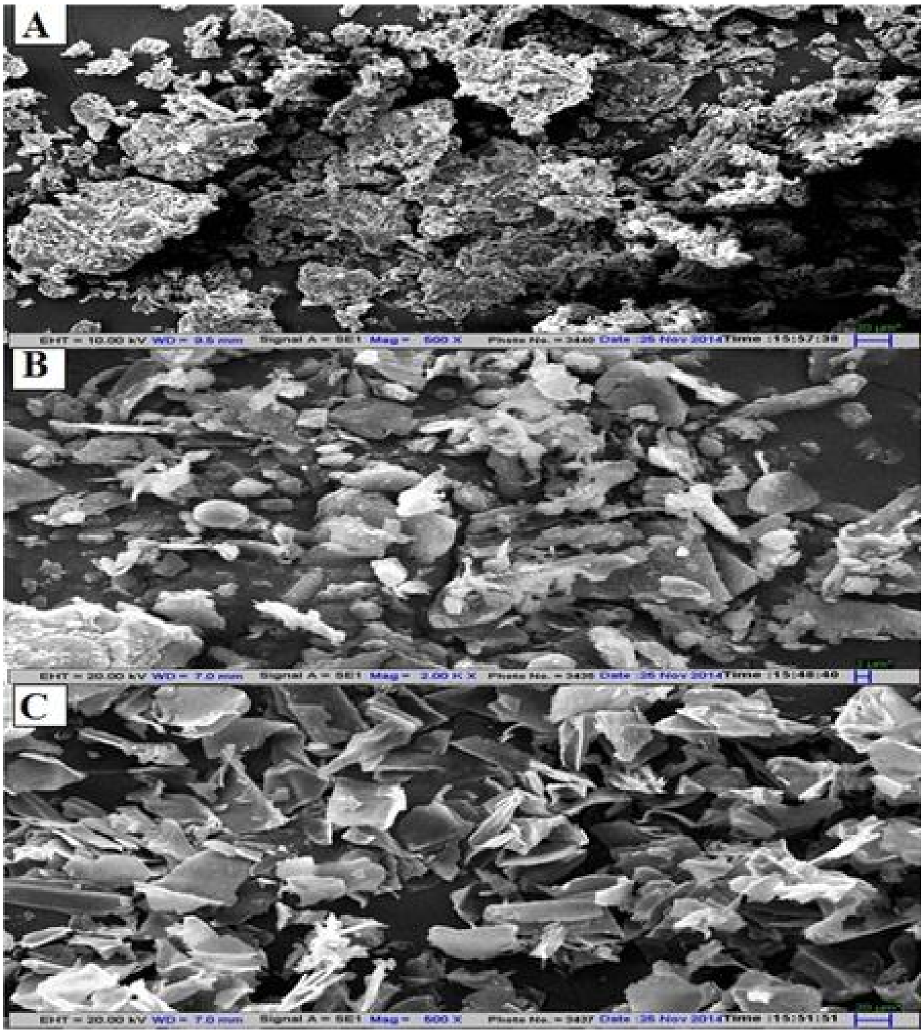

Fig. 3 SEM image of the biosorbent: (A) castor leaves (B) rice husk (C) sugarcane bagasse.

Biosorbents are analyzed using an FT-IR spectrophotometer to confirm the possible interactions of heavy metal ions onto biosorbents (CL, RH, SB) and its effect on the biosorption behavior. Fig. 4(A–C) shows the most relevant spectral bands assigned to the corresponding functional groups. The spectra of the three biosorbents used in our study are quite similar which indicates that the biosorbents possess similar functional groups on their surfaces. The spectral bands present between 3400–3300 cm^{-1} show the presence of –OH and $–NH_2$ groups on the biosorbent surface. The spectral peaks at around 2900 cm^{-1} and 600 cm^{-1} indicate the presence of C–H group while the spectral peak observed at ~1600 cm^{-1} corresponds to the C=O stretching band of the carboxyl group. The presence of a spectral band at approximately 1050 cm^{-1} corresponds to the C–O stretching vibrations of primary alcohol. Thus the outcomes of FTIR study suggest that the biosorbents may possess –COOH groups, –OH groups or $–NH_2$ functional groups on their surfaces. Consequently, the obtained results revealed that the complexation was a potential adsorption mechanism between the test heavy metals and the selected biosorbents.

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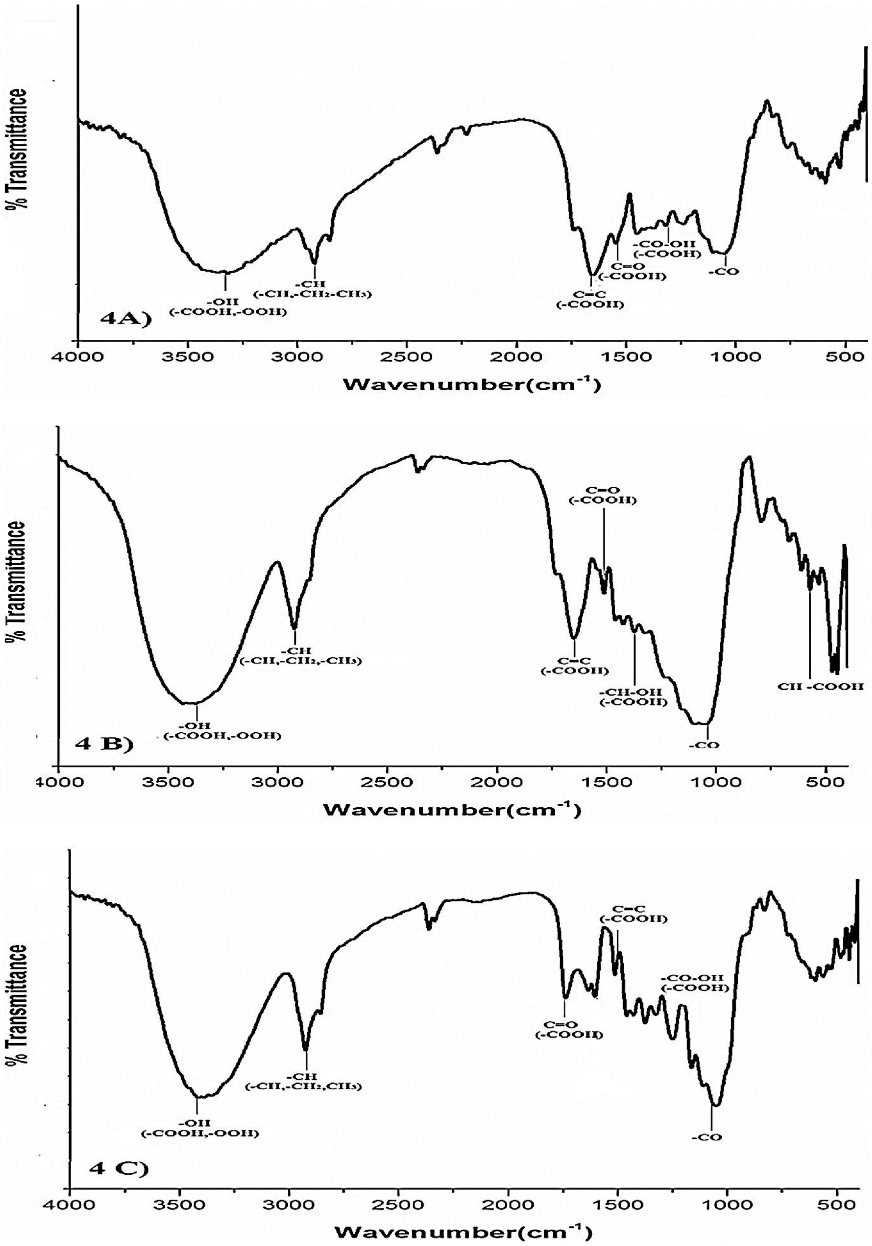

Fig. 4 The FTIR spectra of the biosorbents: (A) castor leaves, (B) rice husk, (C) sugarcane bagasse.

3.2 Biosorption isotherm

Fig. 5(A–C) shows the biosorption isotherms of heavy metal ions (nickel, lead) on the selected biosorbents measured at pH 5.0. The biosorption isotherms curves of both cations (Pb^{+2}, Ni^{+2}) can be described by an initial step with the increase in biosorption capacity followed by saturation which results in a convex shape graph. The presence of large biosorbent-cation affinity and numerous readily accessible sites causes the initial step with the increase in biosorption capacity while the formation of a monomolecular layer of the cations on the biosorbent surfaces and saturation indicates towards convex shape.

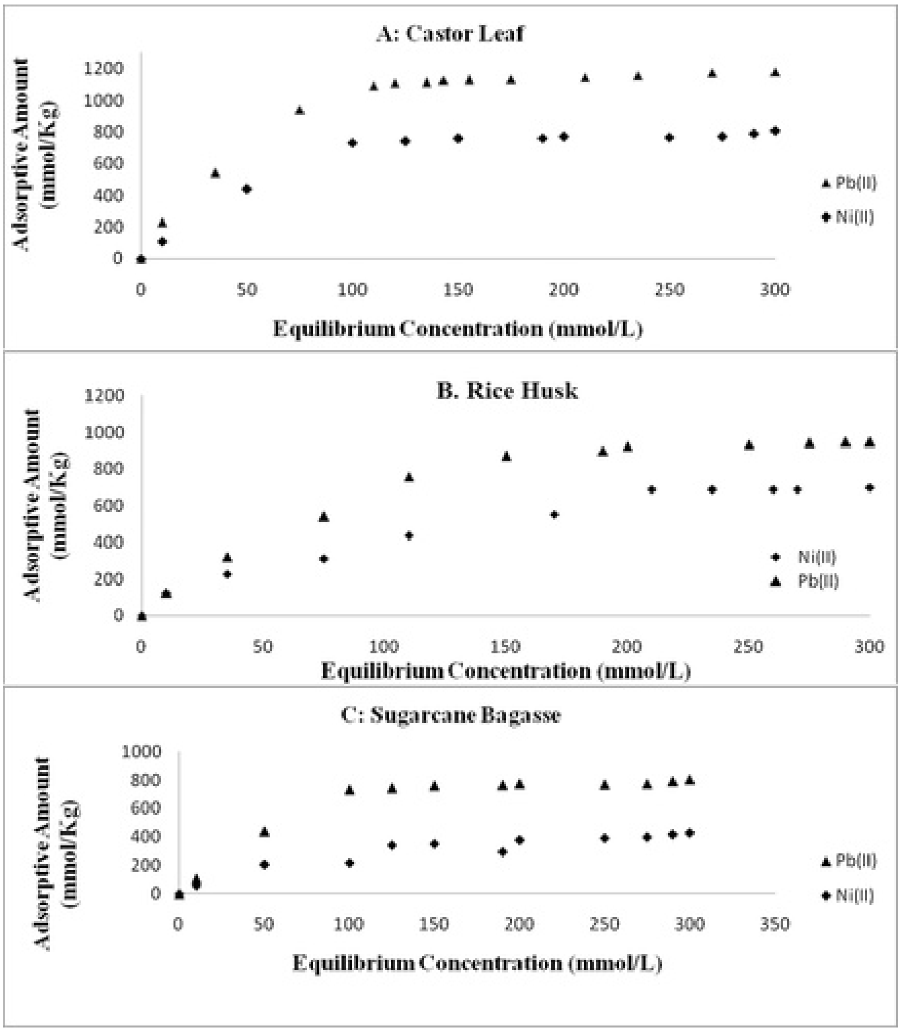

Fig. 5 Adsorption isotherms of the biosorbents towards the test heavy metal: (A) castor leaf; (b) rice husk; (C) sugarcane bagasse.

The isotherms (Fig. 5A–C) indicate the order of adsorption capacities for the tested heavy metals as CL > RH > SB. And in all these biosorbents, the adsorption of Ni(II) was lower than the adsorption of Pb. The complexation abilities for the heavy metals are a function of the properties of heavy metals, functional groups, and of the pH of the solution. Generally, copper(II) and nickel(II) possess a relatively higher potential to form such complexes. Conversely, lead(II) and cadmium(II) generate relatively higher adsorption amounts through the ion exchange (CitationChao and Chang, 2012). The comparatively higher adsorption of Pb(II) suggests that ion-exchange is the primary mechanism of metal ion adsorption in these biosorbents. Table 1 shows the maximum adsorption capacity of the adsorbents at equilibrium condition calculated from Langmuir adsorption equilibrium equation. Biosorbents developed from castor leaves showed the maximum adsorption of 1180 mmol/Kg of Pb(II).

Table 1 The adsorption capacities of the heavy metals on different bioadsorbents.

Cation	Adsorbents	Q (max) (mmol/Kg)
Pb(II)	Sugarcane bagasse	1180

	Rice husk	948
	Castor leaves	802
Ni(II)	Castor leaves	810
	Rice husk	698
	Sugarcane bagasse	432

IV. CONCLUSION

In this study, we selected three non-living biomass/wastes to develop biosorbents and examined their maximum adsorption capacities for lead(II) and nickel(II) usually found in Industrial wastewater samples from Common Effluent Treatment Plant. The results emphasized that the primary adsorption mechanism is the complexation of metal ions at the biosorbent surfaces. Adsorption Isotherm indicate that Pb(II) has higher adsorption property than Ni(II), on the selected biosorbents. Among the selected biosorbents, castor leaves (CL) collected from roadside possess higher adsorption capacities, which can be further developed as a lead(II) selective biosorbents.

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