Adsorption properties of modified *Saraca asoca* bark powder towards Ni(II) ions

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Abstract.

In this present study, phosphate-modified *Saraca asoca* bark powder was used to bring out an efficient bioadsorbent for nickel (II) ion uptake from wastewater. The bioadsorbent was characterized using scanning electron microscopy and Fourier transform infrared spectroscopy. The effect of various parameters such as pH, contact time, initial concentration and temperature were examined using the batch experimental process. The adsorption of nickel(II) was found to increase with the increase in concentration. The used bioadsorbent proved to be highly efficient and adsorbed 96% of nickel(II) from the aqueous solution at pH 4-6. The adsorption equilibrium data were described by using various isotherm models i.e. Langmuir, Freundlich, Temkin and Dubinin–Radhuskevic. Langmuir and pseudo-second-order-kinetic were found to be best-fitted isotherm and kinetic models, respectively. Thermodynamic parameters showed the spontaneous and endothermic nature of the adsorption process. Succesful desorption of Ni(II) confirms the reusability of the bioadsorbent. The samples from real electroplating wastewater were collected to better examine the applicability of phosphate-modified *Saraca asoca* bark powder towards nickel(II) ions.

Keywords: bioadsorbent, desorption, isotherm, kinetics, modified plant material

1. Introduction

The existence of heavy metals in the water streams always is a potential health problem because of their noxiousness and non-biodegradable nature. Nickel, Ni (II), is the most common chemical element in the list of transition metals and found in small quantities in the environment. It used mainly to make stainless steel, coin, jewellery and other alloys. Ni (II) compounds are used in electroplating, pigment, chemical, mining and battery manufacturing industries (Cempel & Nikel, 2006). Ni (II) is considered safe at low concentrations but when it increases in the environment, it has toxic effects on the ecosystem. As per world health organization (WHO) standards, the regulatory limit of Ni(II) in drinking water is 0.020 mg/L (Uddin, 2017). It is necessary to curb Ni(II) pollution in the environment and various technologies such as ion exchange resin (Tan et al., 2017), electrocoagulation (Djaenudin et al., 2018), membrane filtration (Borbély & Nagy, 2009) have been developed for Ni (II) removal from wastewater but the highly used method is adsorption process (Malamis &

Katsou, 2013). The biosorption process is the ability of biologically derived materials to adsorb the toxic aqueous pollutants. Both adsorption and biosorption are economically feasible methods and have many advantages in comparison to other available water treatment techniques (Kashif Uddin & Fazul Rahaman, 2017). Chao et al. used three different agricultural wastes to prepare biosorbents for heavy metals removal in a fixed bed column (Chao et al., 2014). Activated carbon prepared from H_3PO_4 treated lotus stalks has been investigated for Ni (II) adsorption (Huang et al., 2011). The produced adsorbent was found to be highly porous with a surface area of 1220 m^2/g and removed Ni (II) ion rapidly. Torab-Mostaedi et al. studied the biosorption of nickel with the uptake of 46.13 mg/g onto grapefruit peel and the recovery of Ni(II) was more than 97% (Torab-Mostaedi et al., 2013). The binary and ternary adsorption system was used by Liu et al. for the adsorption of Cd(II), Zn(II) and Ni(II) ions using tourmaline and the study reported that multivalent solution at acidic pH values had a very good adsorption capacity for studied metal ions (Liu et al., 2013). Silica/activated carbon composite found to be very effective in the removal of Ni(II) at all studied concentrations (Karnib et al., 2014). The ash-palm thread which has a larger specific surface area and contained many active groups found to be quite an effective biosorbent for removing various heavy metals from wastewater (Lv et al., 2013). Characterisation revealed that an enormous quantity of active SiO_2 was present in the basic structure of the palm thread and was responsible for high Ni(II) adsorption. Recently, modified dry duckweed (Qu et al., 2021), carbon-based adsorbents (Dhaouadi et al., 2021), chitosan derivative (Liakos et al., 2021), olive stone (Corral Bobadilla et al., 2020), fungal biomass (Sundararaju et al., 2020) and seaweed biomass (El-Naggar & Rabei, 2020) are used as a natural adsorbent for Ni(II) removal.

Saraca asoca tree belongs to the Detarioideae subfamily and is commonly found in East Asian countries. *Saraca asoca* has antimicrobial, anticancer, antihemorrhagic and antimycotic properties (Pradhan et al., 2009). As an adsorbent, *Saraca asoca* has been successfully utilised to remove cationic dyes (Gupta et al., 2012) and lead ion (Goyal et al., 2008). The present work shows the adsorption efficiency of modified *Saraca asoca* and its potential role in Ni(II) removal from wastewater. This bioadsorbent is first time tested for Ni(II) adsorption in this study. The adsorptive performance, ease of availability, non-hazardous nature and low cost of *Saraca asoca* shows its significance for Ni(II) adsorption.

2. Materials and Methods

Saraca asoca is abundantly available in India and was collected, washed, dried and ground to powder size (50-100 μm). The preapared bioadsorbent was then treated with a 50 mL of 0.1 N aqueous solution of trisodium phosphate dodecahydrate ($Na_3PO_4.12H_2O$) for 24 hrs. After washed several times by deionized water, its dried form was used as a phosphate modified bioadsorbent for Ni(II) ion. Nickel nitrate, $Ni(NO_3)_2$, of analytical reagent grade in its necessary amount, was used to prepare the Ni(II) solution for adsorption experiments. The % adsorption of Ni(II) was increased from 80 to 95% after phosphate and therefore, it was chosen for further studies. The modified bioadsorbent was characterized by scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) techniques to observe the morphology and functional groups of *Saraca asoca* prior and subsequent Ni(II) adsorption.

2.1 Adsorption experiments

The adsorption experiments were conducted by the batch process. The amount of 0.5 gm bioadsorbent was dispersed in 50 mL Ni(II) solution and the combined solution was stirred, filtered and analysed to determine the final concentration of Ni(II) ion by atomic absorption spectrophotometer. The following formula was used to calculate the % Ni(II) adsorption:

$$\%\ adsorption = \frac{Concentration_{initial} - Concentration_{final}}{Concentration_{initial}} \times 100$$

The effect of various pH ranges (pH 2-pH 9) on Ni(II) adsorption was tested. The pH was regulated by adding 0.1 M HCl or NaOH solution. The point of zero charge (pzc) of the bioadsorbent was also noted. The effect of various contact time intervals (1- 120 min), concentration levels (10–100 mg/L) and temperatures (30–50°C) were also determined by the process as described earlier.

2..1.1 Breakthrough and desorption experiments

In a breakthrough study, a glass column is used in which 0.5 gm of bioadsorbent was put onto the glass wool support and then 1000 mL of 50 mg/L Ni(II) solution was distributed over it at the periodic interval of 1 mL/min. The 50 mL run-off was collected firstly in 10 mL fractions. The samples were then analyzed for Ni(II) concentration. 50 mL of 0.1M HCl was used as a desorbing agent for desorption experiments.

3. Results and discussion

3.1 Characterization of bioadsorbent

SEM images display the porous honeycomb-like structure with large holes present on the surface of modified *Saraca asoca* bioadsorbent (Fig. 1a). The surface seems smooth and offers more support to the adsorption movement of Ni(II) over it. Fig. 1b clearly shows the effective accumulation of Ni(II) as the holes were successfully filled by Ni(II) and the surface became extremely bright after Ni(II) deposition on it.

The peaks at 3,427-3435 cm^{-1} in FTIR spectra (Fig. 2, ab) were assigned to the hydroxyl (-OH) group due to inter and intramolecular attractive forces (Rao & Kashifuddin, 2014). These -OH stretching vibrations indicate the occurrence of a free OH group and carboxylic acid (Gnanasambandam, 2000). A peak at 3786 cm^{-1} in modified bioadsorbent corresponds to the –OH group of PO_4^{3-} (Arai & Sparks, 2001). Most peaks in both the spectra almost occurred at the same place, however, some shifting in the peaks can be observed. A peak as present in bioadsorbent at 1450 cm^{-1} belongs to carboxylate group was loosened to 1423.6 cm^{-1} after Ni(II) adsorption representing the strong interface of Ni(II) with the modified bioadsorbent. Another peak that appeared at 1157 cm^{-1} belongs to the cyano group (C≡N). After adsorption, it was shifted to 1180 cm^{-1} which suggests the presence of nickel compound. The peaks at 1023 cm^{-1} indicative of carbonyl and carboxyl groups which may have a part in the adsorption mechanism (El-Sadaawy & Abdelwahab, 2014).

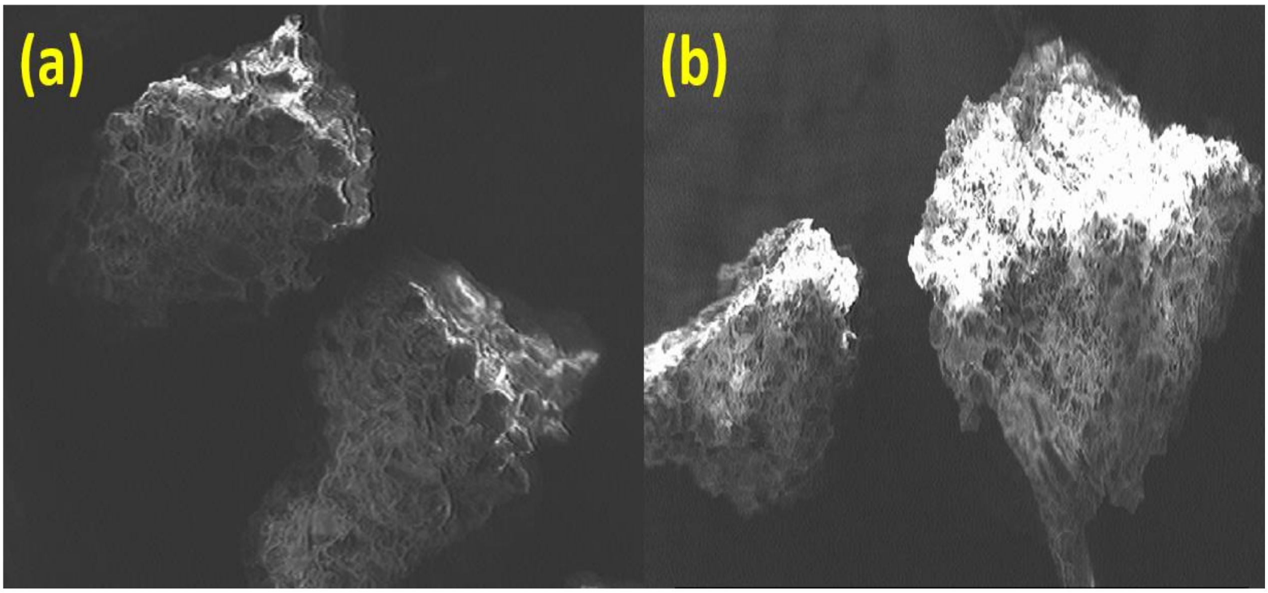


Figure 1: SEM images of modified biaosdorbent (a) and Ni(II) adorsbed modified bioadsorbent (b)

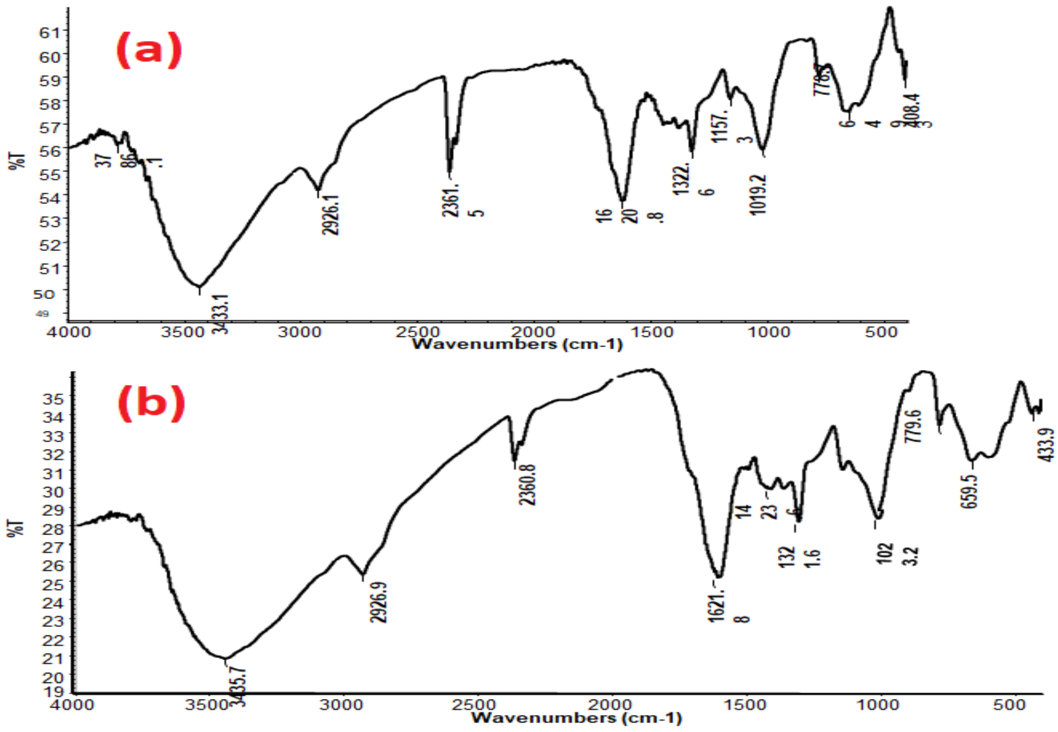
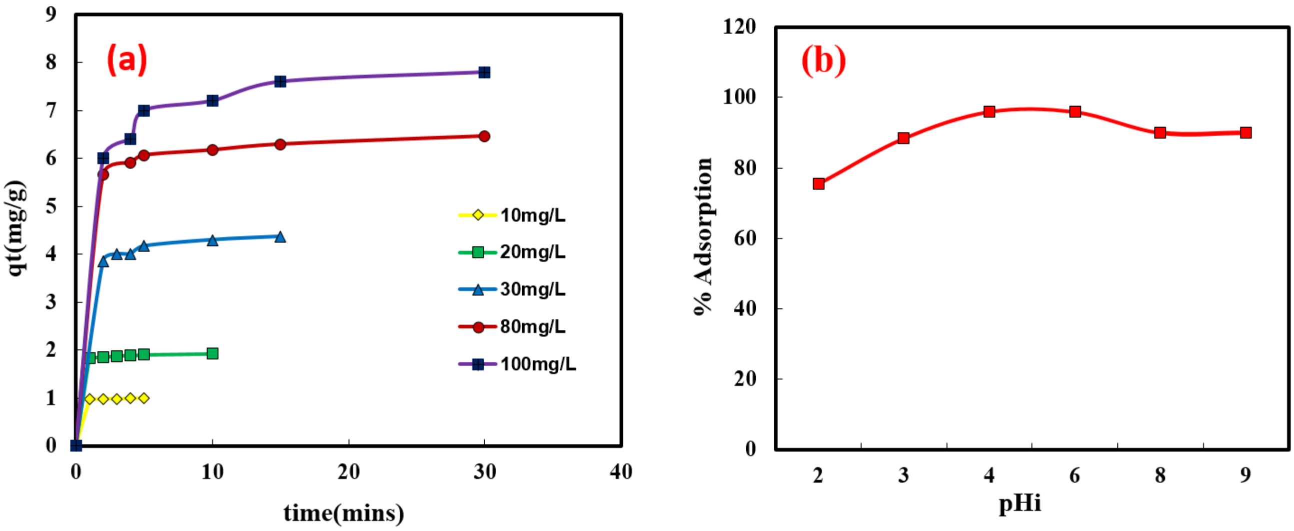


Figure 2: FTIR spectra of modified biaosdorbent (a) and Ni(II) adorsbed modified bioadsorbent (b)

3.1.1 Adsorption experiments

Fig. 3a shows that with the increase in Ni(II) concentrations (10-100 mg/L), the uptake capacity of the bioadsorbent was also increased (0.99-7.90 mg/g). The equilibrium adsorption capacities at 10-100 mg/L Ni(II) concentrations were found to be 0.99, 1.97, 4.42, 6.85 and 7.90 mg/g, respectively This increase is obvious because, with the rise in concentration, the formation of the bond between the Ni(II) compound and bioadsorbent improved. To know the effect of pH values (2-9) on the % adsorption, a series of experiments were carried out. Fig. 3b shows that the adsorption of Ni(II) was intensely exaggerated by pH values. At pH 2, 75.4% adsorption was recorded and then the value increased by an increase in pH up to 96% at pH 4. This rise was because at pH < 6, nickel ion existed as Ni^{2+} while the decline of % adsorption at low pH values was because of the struggle between Ni(II) and H^+ for binding sites on the bioadsorbent surface. At pH> 6, the nickel got precipitated because, at high pH values, $Ni(OH)_2$, $Ni(OH)_3$ are the dominant species (Jalali & Najafi, 2018) that reduce the solubility of the metal and cause precipitation.



Langmuir, Freundlich, Temkin and Dubinin-Radushkeuich isotherm models were used to adequate the experimental data as obtained at various temperatures. Table 1 shows the linear forms of the applied isotherm models while Table 2 shows the adsorption isotherm results. The linear fitting of data and high R^2 values confirmed the monolayer and homogenous distribution of Ni(II) on the bioadsorbent. The calculated values of mean free energy (E) are in the range of 9-11 kJ/mol indicate that adsorption was chemical. Table 3 shows the comparison of maximum adsorption capacity of phosphate-modified *saraca asoca* with other plant-based natural adsorbents as reported earlier. The results prove its effectiveness as a bioadsorbent for the removal of Ni(II) from the aqueous solution.

Table 1 *Linear equations and various adsorption isotherm parameters for the adsorption of Ni(II) on phosphate-modified bioadsorbent*

Isotherm model	Linear equation
Langmuir	$\frac{1}{q_e} = \frac{1}{q_m \times b} \times \frac{1}{C_e} + \frac{1}{q_m}$ Where b refers to Langmuir constant
Freundlich	$\log q_e = \log K + \frac{1}{n} \log C_e$ Where K refers to Freundlich constant

Temkin	$$q_e = \left(\frac{RT}{b}\right) \times \ln At + \left(\frac{RT}{b}\right) \times \ln C_e$$ Where (RT/b) = B_t, R is a gas constant, T is temperature, A_t and B_t are Temkin constants.
D-R	$$\ln qe = \ln q_{dr} - \beta\varepsilon^2$$ Where β is a D-R constant which gives mean free energy (E) which can be find by following relattion: $$\varepsilon = RT \ln(1 + \frac{1}{ce})$$

***Table 2** Adsorption isotherm results for the adsorption of Ni(II) on phosphate-modified bioadsorbent*

Isotherms	Parameters	30^0C	40^0C	50^0C
Langmuir	qm (mg/g)	22.96	12.90	11.41
	R^2	0.99	0.99	0.99
Freundlich	Kf $(L/mg)^{1/n}$	1.74	2.33	2.68
	n (g/L)	1.52	1.73	1.81
	R^2	0.98	0.99	0.99
Temkin	A (L/mg)	0.69	1.38	2.28
	B (J/mol)	4.44	3.31	2.93
	R^2	0.99	0.98	0.98
D-R	E (kJ/mol)	9.25	10.80	11.35

***Table 3** Comaprison of adsorption capacity between various adsorbents used for Ni(II) removal*

Adsorbent	Adsorption capacity (mg/g)	References
Peat	8.52	(Helen Kalavathy & Miranda, 2010)

Modified pine bark	9.50	(Argun et al., 2009)
Wheat Straw	7.90	(Hajahmadi et al., 2015)
Coir pith	9.50	(Ewecharoen et al., 2008)
Activated carbon	4.52	(Kavand et al., 2016)
Orange peel	9.82	(Feng et al., 2011)
Tea factory waste	15.26	(MALKOC & NUHOGLU, 2005)
Grape stalks waste	10.66	(Villaescusa et al., 2004)
Beech sawdust	4.50	(Božić et al., 2013)
Banana peel	6.80	(Annadurai et al., 2003)
Hazelnut shell	10.10	(Demirbas, 2002)
Alternanthera Philoxeroides biomass	9.73	(Wang & Qin, 2006)
Doum-palm seed coat	4.93	(El-Sadaawy & Abdelwahab, 2014)
Phosphate-modified *saraca asoca*	22.90	Present study

Thermodynamic parameters such as free energy change (ΔG), enthalpy change (ΔH) and entropy change (ΔS) were calculated using the following relations:

$$Kc = \frac{C_{adsorption}}{C_{equlibrium}}$$

$$\Delta G = -RTlnKc$$

$$lnKc = -\frac{\Delta H}{RT} + \frac{\Delta S}{R}$$

The positive value of ΔH indicates that the adsorption process was endothermic, the negative values of ΔG affirm the feasible Ni(II) adsorption while the positive value of ΔS specifies greater randomness on the bioadsorbent's surface (Table 4).

Table 4 *Adsorption thermodynamic results for the adsorption of Ni(II) on phosphate-modified bioadsorbent*

Temperature (˚C)	Kc	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (kJ/mol/K)	R^2
30	19.00	-7.42	55.59	12.47	0.97
40	30.30	-8.87			

50	40.70	-9.95

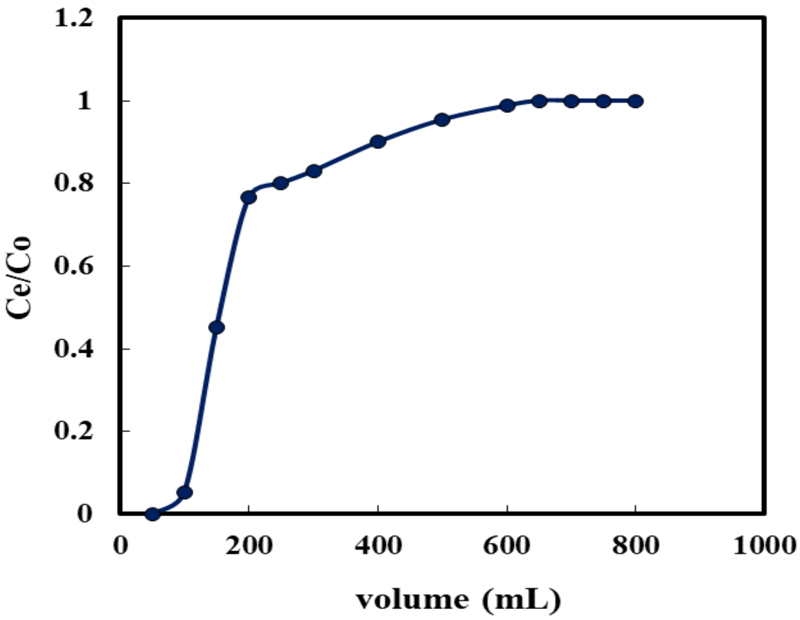
Table 5 shows the data of the parameters as obtained by kinetic study. The high correlation coefficients (R^2) and the close values of calculated adsorption capacity (qe_{cal}) and experimental adsorption capacity (qe_{exp}) confirm that the pseudo-second model was better employed with adsorption results.

Table 5 *Adsorption kinetic results for the adsorption of Ni(II) on phosphate-modified bioadsorbent*

Concentration (mg/L)	Pseudo-first-order				Pseudo-second-order			
	q_e (exp) (mg/g)	q_e (cal) (mg/g)	K_1 (mol/L/s)	R^2	q_e (cal) (mg/g)	K_2 (g/mg/min)	h (mg/g/min)	R^2
10	0.99	0.05	0.74	0.86	0.99	28.36	27.80	1.00
40	1.97	0.14	0.10	0.95	1.98	10.20	40.00	0.99
50	4.42	2.14	0.05	0.90	4.43	0.60	11.83	0.99
80	6.85	1.05	0.03	0.90	6.54	0.32	15.46	0.99
100	7.90	1.96	0.10	0.95	8.00	0.14	9.20	0.99

3.1.2. Desorption, Breakthrough curve and application to electroplating wastewater

Fig. 8 shows the breakthrough point that occurred after passing 50 mL Ni(II) and the breakthrough and exhaustive capacities were 5 and 65 mg/g, respectively. The desorption results showed that 99.1% Ni(II) amount was recuperated which indicate that modified bioadsorbent can be used frequently. The electroplating wastewater was also collected and analysed. The presence of several cations (Na^+, K^+, Ca^{2+}), and metal ions (Cu^{2+}, Zn^{2+}, Ni^{2+}) was found in it. After using *saraca asoca* bioadsorbent, almost 70% Ni(II) was removed which shows that even with the existence of other pollutants, the bioadsorbent worked well.



Conclusion

The adsorption of Ni(II) from contaminated water by *saroca asoca* bioadsorbent was examined. The adsorption outcomes verified that the maximum removal was found at pH 4-6. The adsorption was best reported by the pseudo-second-order kinetic model and was perfectly fitted by the Langmuir isotherm. The adsorption processes were endothermic. The obtained results indicate that the biomass has highly useful in Ni(II) adsorption and can be reused and applied as a promising nontoxic material.

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