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Nickel Recovery from Aqueous Solution Using Crab Shell Particles

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ABSTRACT: The potential use of crab shell as a sorbent for the removal of nickel(II) ions from aqueous solution was investigated. The binding of nickel ions by crab shell was found to be affected significantly by pH, with the maximum sorption capacity being observed at pH 4.5. The sorption isotherm was well represented using the Freundlich model. Nickel(II) ion removal by crab shell was mainly influenced by the removal of calcium carbonate, proteins and chitin, indicating the importance of these components in nickel ion binding. Co-ions such as Cu2+, Co2+, Cd2+, Zn2+ and Mg2+ affected the Ni(II) ion removal efficiency of crab shell.

The biosorbed Ni(II) ions were effectively eluted by various mineral acids, EDTA solutions and NH4OH. Of these, the sodium salt of EDTA (0.01 M) in NH4OH appeared to be the best eluant, being capable of desorbing more than 99% of the sequestered Ni(II) ions with insignificant damage to the shell particles. The biosorbent could be regenerated and re-used in five sorption–elution cycles.

INTRODUCTION

Biosorption — a process which utilizes inactive biological materials for the removal of metals from their dilute solutions — has gained in importance during recent years due to its effectiveness, selectivity, low cost and operation over a wide range of pH and temperature. Biosorption usually involves several mechanisms and some of these mechanisms can be sub-mechanisms of other overall mechanisms, such as ion exchange, complexation, coordination, chelation, microprecipitation or adsorption (Volesky and Holan 1995). Heavy metal biosorption by biological materials such as bacteria and fungi presents few problems when operated in a continuous mode; however, of these solid/liquid separation is a major constraint. Even though immobilization may solve this problem, chemical costs and mechanical strength should be taken into consideration (Veglio and Beolchini 1997). For these reasons, recent research has focused on the use of low-cost and waste materials as adsorbents (Bailey et al. 1999). Of these, crab shell which has been used for the removal of lead ions (Lee et al. 1997) and cadmium ions (Evans et al. 2002) has been chosen as the focus of the present study.

Crab shell is mainly composed of calcium carbonate and chitin together some proteins. Chitin and its deacetylated form (chitosan) have been recognized as effective biosorbents for metal removal. Chitin and chitosan are long linear polymeric molecules of β-(1→4)-linked glycans. The repeat unit in chitin is 2-acetamido-2-deoxy-D-glucose-(N-acetylg glucosamine) while chitosan

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consists of a non-homogeneous mixture of chitin with its deacetylated form (glucosamine) (Onsoyen and Skaugrud 1990).

Nickel, which is the most widely used heavy metal frequently employed in electroplating industries, usually shows a low affinity towards biosorption. Investigations have reported different biosorption capacities for Ni(II) ions within the range 0.8–70 mg/g (Veglio and Beolchini 1997). The purpose of the present study was to utilize the shells of *Portunus sanguinolentus* in the removal of Ni(II) ions from aqueous solution. The influence of various factors such as the initial pH of the solution, the initial Ni(II) ion concentration and concentrations of co-ions were investigated. Desorption studies using different elutants at different concentrations and solid-to-liquid ratios were also examined.

**MATERIALS AND METHODS**

Waste shells of *Portunus sanguinolentus*, commonly known as three-spot crabs, were collected from Marina beach, India, sun-dried and crushed to desired particle size ranges using a ball mill. Pre-treatment of the crab shells was undertaken by washing them with 0.1 M HCl for 4 h to remove CaCO3. The treated shells were then washed with de-ionized water and dried naturally; the corresponding weight loss was found to be ca. 50%. In this paper, the pretreated crab shell particles obtained are referred to by the abbreviation “CSP”. Subsequent to washing with de-ionized water, the pretreated shells were soaked overnight in 1% NaOH at room temperature to remove the bulk of their protein content. The residue (chitin) was then rinsed with de-ionized water and dried at 100°C. Chitin was converted to chitosan by soaking in 50% NaOH for 2 h at 100°C to remove some or all of the acetyl residues and then dried naturally (Kim and Park 2001).

The various reagents employed, i.e. HCl, HNO3, H2SO4, NaOH, NaCl, KCl, CaCl2·2H2O, MgCl2·6H2O, CoSO4·7H2O, CuSO4·5H2O, 3CdSO4·8H2O, NiSO4·6H2O, ZnSO4·7H2O, NH4OH, NH4Cl, EDTA (free acid) and EDTA (Na) were all of AnalaR grade and purchased from Ranbaxy Fine Chemicals Ltd., India.

Biosorption experiments were performed in a rotary shaker at 150 rpm using 250 ml Erlenmeyer flasks with 0.5 g CSP in 100 ml solution containing different Ni(II) ion concentrations. After equilibrium had been reached, the reaction mixture was centrifuged at 3000 rpm for 10 min. The Ni(II) ion content in the supernatant was determined using atomic absorption spectroscopy (AAS 6V ARIO spectrophotometer; Analytik Jena, Germany). When necessary, the supernatants were diluted with de-ionized water prior to analysis. The amount of metal ion biosorbed was calculated from the difference between the quantity of metal ions added to the CSP and the metal ion content of the supernatant using the following equation:

\[
q = (C_i - C_f) \times V/M
\]

where q is the amount of metal ion adsorbed (mg/g); \( C_i \) and \( C_f \) are the initial and equilibrium metal ion concentrations in the solution (mg/l), respectively; V is volume of the solution (l); and M is the mass of biosorbent employed (g). The pH of the aqueous solution was adjusted using 0.1 M HCl or 0.1 M NaOH solutions, respectively.

The Langmuir sorption model was chosen for the estimation of maximum Ni(II) ion biosorption by the biosorbent. The Langmuir isotherm can be expressed as:

\[
q = \frac{q_{\text{max}} b C_f}{1 + b C_f}
\]
where $q_{\text{max}}$ is the maximum metal ion uptake (mg/g) and $b$ is the Langmuir equilibrium constant (l/mg). For fitting the experimental data, the Langmuir model was linearized as follows:

$$\frac{1}{q} = \frac{1}{q_{\text{max}}} + \frac{1}{q_{\text{max}} b c_f} \tag{3}$$

Similarly, the Freundlich sorption model may be represented by the equation:

$$q = K c_f^n \tag{4}$$

where $K$ and $n$ are constants.

After biosorption, the Ni(II) ion-loaded CSP was contacted with different elutants at desired concentrations for 3 h on a rotary shaker (150 rpm) to study the removal of the biosorbed Ni(II) ions. The remaining procedure was the same as that employed in the sorption equilibrium experiments.

**RESULTS AND DISCUSSION**

The experimental results obtained for nickel ion biosorption by CSP under different initial pH conditions are shown in Figure 1. The CSP dosage (5 g/l) and agitation speed (150 rpm) were kept constant in all experiments. The Ni(II) ion uptake by CSP was sensitive to pH variation over the examined range of 2 to 5. The uptake increased with increasing pH to reach a maximum at pH 4.5. This was not only because of hydrogen ion competition at low pH, but also may be due to the lyophobic behaviour of the adsorbate. Since the solubilities of many metal ion complexes in solution decrease with increasing pH, the sorption also increased with increasing pH. At pH 2, the maximum Ni(II) ion removal efficiency was 72%, whereas at pH 4.5 the removal efficiency...
increased to 84% for 100 mg/l initial Ni(II) ion concentration. This enhancement was not that much more appreciable than those reported earlier (Kim and Park 2001; Lee et al. 1997). On changing the initial Ni(II) ion concentration from 100 mg/l to 1000 mg/l, the uptake increased from 16.7 mg/g to 136.36 mg/g at pH 4.5. However, the percentage Ni(II) ion removal decreased from 84% to 68% as the concentration increased from 100 mg Ni(II)/l to 1000 mg Ni(II)/l.

The equilibrium biosorption data were modelled using both the Langmuir and Freundlich adsorption models. It should be noted that An et al. (2001) used both the Langmuir and Freundlich models successfully to describe lead ion sorption by crab shell. Table 1 shows the model constants together with the corresponding correlation coefficients, $R^2$, for the biosorption of Ni(II) ions onto CSP. The Langmuir sorption model allowed the maximum Ni(II) ion uptake values to be calculated under situations which could not be attained experimentally. The constant $b$ represents the affinity between the adsorbent and adsorbate. The Langmuir model parameters were largely dependent on the initial pH value of the solution. Both the maximum Ni(II) ion uptake, $q_{\text{max}}$, and the Langmuir equilibrium constant, $b$, increased as the initial pH value increased from 2.0 to 4.5. In addition, the fact that both $K$ and $n$ attained their maximum values at pH 4.5 implies that the binding capacity and the affinity between the CSP and the Ni(II) ions were also higher than at the other pH values investigated. For all the initial pH conditions examined, regression analysis of the data obtained from the Freundlich model resulted in higher correlation coefficients than those obtained from the Langmuir model.

In an attempt to understand the role of calcium carbonate, proteins, chitin and chitosan in Ni(II) ion removal by crab shell, the shell particles were subjected to various chemical treatments. The experimental results depicted in Figure 2 indicate that Ni(II) ion uptake by the resulting biosorbents followed the order: raw crab shell > CSP > chitin > chitosan. Calcium carbonate plays a major role in Ni(II) ion removal by raw crab shell. However, in this case, the maximum removal efficiency (88%) was mainly due to precipitation because of the presence of excess calcium carbonate. The precipitates were then adsorbed onto the chitin located at the surface of the crab shell. In addition, the rapid steep rise in pH from an initial value of 4.5 to a value of 8.5 may be attributed mainly to dissolution of $\text{CaCO}_3$ and the generation of $\text{CO}_3^{2-}$ ions (Lee et al. 1997). After the removal of calcium carbonate from crab shell, the maximum removal efficiency decreased to 84%, while the removal efficiencies of chitin and chitosan fell to 72% and 20%, respectively.

### Table 1. Langmuir and Freundlich Model Parameters Obtained under Different pH Conditions

<table>
<thead>
<tr>
<th>pH</th>
<th>Langmuir parameters</th>
<th>Correlation coefficient, $R^2$</th>
<th>Freundlich parameters</th>
<th>Correlation coefficient, $R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$q_{\text{max}}$ (mg/g)</td>
<td>$b$ (l/mg)</td>
<td></td>
<td>$K$ (l/g)</td>
</tr>
<tr>
<td>2.0</td>
<td>149.25</td>
<td>0.0037</td>
<td>0.9815</td>
<td>0.966</td>
</tr>
<tr>
<td>2.5</td>
<td>151.52</td>
<td>0.0039</td>
<td>0.9847</td>
<td>1.076</td>
</tr>
<tr>
<td>3.0</td>
<td>153.85</td>
<td>0.0043</td>
<td>0.9871</td>
<td>1.238</td>
</tr>
<tr>
<td>3.5</td>
<td>156.25</td>
<td>0.0046</td>
<td>0.9897</td>
<td>1.398</td>
</tr>
<tr>
<td>4.0</td>
<td>161.29</td>
<td>0.0055</td>
<td>0.9918</td>
<td>1.747</td>
</tr>
<tr>
<td>4.5</td>
<td>169.49</td>
<td>0.0065</td>
<td>0.9934</td>
<td>2.225</td>
</tr>
<tr>
<td>5.0</td>
<td>166.67</td>
<td>0.0059</td>
<td>0.9901</td>
<td>1.842</td>
</tr>
</tbody>
</table>
The significant decrease of 20% in the maximum Ni(II) ion uptake when proteins were removed from the shell indicates that proteins also contribute to Ni(II) ion removal. This may be due to the functional groups of the protein or the possible dissolution of compounds in the outer layer of the shell during previous acid treatment. This, in turn, may lead to the generation of additional binding sites. Chitin has been postulated as the main constituent responsible for metal ion coordination (Lee et al. 1997; Tsezos and Volesky 1981). The mechanism involved is usually complex formation between dissolved metallic species and chitin. The deacetylation of chitin resulted in a major decrease in Ni(II) ion removal efficiency, either due to the degree of deacetylation or because of the distribution of acetyl groups along the molecule and throughout the chitosan particle (Onsoyen and Skaugrud 1990). Hence, it may be concluded that CaCO₃, proteins and chitin appear to be the major constituents of raw crab shell responsible for Ni(II) ion removal.

The effect of the presence of co-ions in the solution on the Ni(II) ion uptake capacity of CSP was examined by adding Na⁺, K⁺, Mg²⁺, Cu²⁺, Co²⁺, Cd²⁺ and Zn²⁺ ions, respectively, one at a time. Examinations were carried out at a pH value of 4.5 for different concentrations of co-ions (Figure 3). Of the ions of the lighter metals, Mg²⁺ had an appreciable effect on the Ni²⁺ uptake while it is interesting to note that a positive effect was observed for Na⁺ and K⁺ ions when these were used separately as competing ions in the Ni(II) ion solutions. Kuyucak and Volesky (1989a) similarly observed an enhanced cobalt ion uptake by *Ascophyllum nodosum* when K⁺ ions were used as the competing ion. The possible reason for this increase might be due to the chloride ion in NaCl or KCl combining with the Ni²⁺ ion and being deposited as solid NiCl₂ on the CSP surface. The presence of Co³⁺ ions caused a considerable suppression in Ni(II) ion uptake, while Cu²⁺ and Cd²⁺ ions also resulted in a substantial decrease in Ni(II) ion uptake by CSP. The existence of Zn²⁺ ions (up to 500 mg/l) in the Ni(II) ion solution caused a slightly higher suppression in Ni²⁺ ion uptake than that observed at higher concentrations. Hence, it may be concluded that inhibition by ions of...
the lighter metals (Mg²⁺, Na⁺ and K⁺) of the removal of Ni(II) ions by CSP was less pronounced than inhibition by heavy metal ions (Co²⁺, Cu²⁺, Cd²⁺ and Zn²⁺).

The Ni(II) ion-loaded CSP was subjected to elution by various desorbents, i.e. HCl, H₂SO₄, HNO₃, CaCl₂, NaCl, KCl, NH₄Cl, NaOH, EDTA (free acid), EDTA (Na) and NH₄OH (Table 2). It was necessary that the optimal eluant was effective and non-damaging to the biomass, non-polluting and cheap. The elution efficiency was determined from the ratio of the mass of metal ion in solution after desorption to the mass of metal ion initially bound to the biosorbent (Davis et al. 2000). It was found that washing the Ni(II) ion-laden biomass with mineral acids (0.1 M HCl, 0.1 M H₂SO₄ and 0.1 M HNO₃) led to release of all the metal ions. In contrast, de-ionized water and boiled water were incapable of eluting biosorbed Ni(II) ions, thereby demonstrating the strong affinity of Ni(II) ions towards CSP. Furthermore, in expectation of the potential involvement of an ion-exchange process in the biosorption phenomenon, the Ni(II) ion-laden CSP was contacted with 0.1 M solutions of NaCl, KCl, CaCl₂ and NH₄Cl, respectively. However, these solutions only exhibited a limited ability to elute biosorbed Ni(II) ions, thereby indicating the absence of ion exchange as a major mechanism. The elution efficiency of CaCl₂ improved slightly at a pH value of 2.0, whereas 0.1 M NaOH exhibited only a very low desorption capability when used as an eluting solution.

Although the use of conc. NH₄OH as an eluant led to release of all Ni(II) ions from the Ni(II) ion-laden CSP, dilution of the conc. NH₄OH solution to 2 M resulted in a ca. 15% reduction in elution efficiency. In an attempt to improve the elution efficiency, use of the strong complexing agent EDTA (ethylenediaminetetra-acetic acid) was considered. A 0.01 M aqueous solution of EDTA (Na) eluted 96% of sequestered Ni(II) ion from the CSP, with the elution efficiency of the EDTA solution being enhanced both by decreasing the pH (using HCl) and increasing the pH (using NH₄OH). Cations sequestered physicochemically onto a cell surface are readily coupled to complexing agents, whereas intracellularly accumulated cations are not so easily desorbed from

![Figure 3. Effect of co-ions on the adsorption of Ni(II) ions onto crab shell particles. The data symbols refer to the following co-ions: ●, Na⁺; □, K⁺; △, Mg²⁺; ○, Cu²⁺; ●, Cd²⁺; ○, Co²⁺; +, Zn²⁺. In all experiments, the adsorbent dosage was 5 g/l, the pH value of the solution was 4.5 while the initial concentration of Ni(II) ions was 1000 mg/l.](image-url)
biosorbent materials (Kuyucak and Volesky 1989b). In addition to the above eluants, 0.01 M EDTA (free acid) dissolved in NH₄OH at pH 9.2 also successfully released 99.2% of biosorbed Ni(II) ions from CSP.

Another important parameter in metal ion biosorption is the solid-to-liquid (S/L) ratio defined as the mass of metal ion-laden biosorbent to the volume of eluant (Davis et al. 2000). Thus, upon elution of a metal ion from a biosorbent, it is desirable to use the smallest possible volume of eluant so as to obtain the highest concentration of the metal ion. At the same time, the volume of the solution should be sufficient to provide maximum solubility for the metal ion desorbed. Figure 4 illustrates the effect of the S/L ratio on the Ni(II) ion elution efficiency of the eluants examined.

Among the mineral acids examined, the elution efficiency of HCl appeared to be virtually independent of the S/L ratio up to the examined value of 10 g/l, whereas that of HNO₃ and H₂SO₄ dropped to 97% and 95%, respectively, at an S/L ratio of 10 g/l. The elution efficiency of conc. NH₄OH remained virtually unaltered when the S/L ratio was varied. However, the elution efficiency decreased with increasing S/L value for EDTA solution, whereas both EDTA/HCl and EDTA/NH₄OH solutions retained almost all of their elution efficiencies up to an S/L value of 10 g/l.

The use of crab shell as a potential biosorbent depends not only on the biosorptive capacity, but also on how well the shell particles can be re-used. Thus, to study any changes in Ni(II) ion biosorption efficiency with subsequent re-use, a CSP sample was re-used for five successive biosorption elution cycles. A preliminary examination on the eluants revealed that the application of mineral acids and conc. NH₄OH resulted in damage to the macroscopic appearance of CSP with the weight loss being greater than 60%. Furthermore, use of EDTA solution as an eluant was insufficient to remove the considerable amount of Ni(II) ions loaded onto CSP at high S/L ratios. Such retention of Ni(II) ions could result in a decreased metal ion uptake in the next re-use cycle.

These aspects limited the eluants examined to EDTA (Na)/HCl, EDTA (Na)/NH₄OH and EDTA (free acid)/NH₄OH. Figure 5 shows that it was subsequently possible to re-use CSP for Ni(II) ion biosorption materials (Kuyucak and Volesky 1989b). In addition to the above eluants, 0.01 M EDTA (free acid) dissolved in NH₄OH at pH 9.2 also successfully released 99.2% of biosorbed Ni(II) ions from CSP.

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TABLE 2. Elution of Biosorbed Ni(II) Ions by Various Chemical Agents

<table>
<thead>
<tr>
<th>Eluant</th>
<th>Initial pH</th>
<th>% Elution</th>
</tr>
</thead>
<tbody>
<tr>
<td>De-ionized water</td>
<td>Unadjusted</td>
<td>1.1</td>
</tr>
<tr>
<td>Boiled water</td>
<td>Unadjusted</td>
<td>2.1</td>
</tr>
<tr>
<td>0.1 M HCl</td>
<td>1.2</td>
<td>100.0</td>
</tr>
<tr>
<td>0.1 M H₂SO₄</td>
<td>1.0</td>
<td>99.1</td>
</tr>
<tr>
<td>0.1 M HNO₃</td>
<td>1.2</td>
<td>100.0</td>
</tr>
<tr>
<td>0.1 M CaCl₂</td>
<td>Unadjusted</td>
<td>6.8</td>
</tr>
<tr>
<td>0.1 M CaCl₂/HCl</td>
<td>2.0</td>
<td>8.6</td>
</tr>
<tr>
<td>0.1 M NaCl</td>
<td>Unadjusted</td>
<td>3.1</td>
</tr>
<tr>
<td>0.1 M KCl</td>
<td>Unadjusted</td>
<td>3.4</td>
</tr>
<tr>
<td>0.1 M NH₄Cl</td>
<td>Unadjusted</td>
<td>8.9</td>
</tr>
<tr>
<td>Conc. NH₄OH</td>
<td>Unadjusted</td>
<td>100.0</td>
</tr>
<tr>
<td>2 M NH₄OH</td>
<td>Unadjusted</td>
<td>85.7</td>
</tr>
<tr>
<td>0.1 M NaOH</td>
<td>Unadjusted</td>
<td>4.2</td>
</tr>
<tr>
<td>0.01 M EDTA (Na)</td>
<td>Unadjusted</td>
<td>95.9</td>
</tr>
<tr>
<td>0.01 M EDTA (Na)/HCl</td>
<td>3.5</td>
<td>99.1</td>
</tr>
<tr>
<td>0.01 M EDTA (Na)/NH₄OH</td>
<td>9.8</td>
<td>99.5</td>
</tr>
<tr>
<td>0.01 M EDTA (free acid)/NH₄OH</td>
<td>9.2</td>
<td>99.2</td>
</tr>
</tbody>
</table>

*Ni(II) ion loading on crab shell particles was 136 mg/g; solid-to-liquid ratio = 1:1.*
Figure 4. Effect of the solid-to-liquid (S/L) ratio on the efficiency of Ni(II) ion elution from CSP with different eluants.

Eluant: ▼, HCl; □, HNO₃; ▲, H₂SO₄; △, NH₄OH; ■, EDTA (Na); ×, EDTA (Na)/HCl; ○, EDTA (Na)/H₂SO₄; ●, EDTA (free acid)/NH₄OH.

Figure 5. Re-use of regenerated crab shell particles in Ni(II) ion biosorption. The crab shell particles were regenerated using EDTA (Na)/HCl (E₁), EDTA (Na)/NH₄OH (E₂) and EDTA (free acid)/NH₄OH (E₃), respectively.
sorption in five successive cycles when the selected eluants for regeneration were used at an S/L ratio of 10 g/l. The CSP regenerated with both EDTA (in NH₄OH) solutions exhibited a higher Ni(II) ion uptake capacity than CSP regenerated using EDTA (in HCl) solution. The loss in dry weight of CSP was less than 10% after five cycles for all the EDTA solutions examined. These observations indicate that 0.01 M EDTA (in NH₄OH) appears to be the most efficient and practical eluting agent for the release of sequestered Ni(II) ions from CSP.

CONCLUSIONS

The present investigation resulted in identifying a more potent biosorbent, crab shell, for the removal of Ni(II) ions from aqueous solution. The results obtained indicate the high Ni(II) ion sorption capacity of crab shell. However, the sorption capacity decreased in the presence of Co²⁺, Cu²⁺, Cd²⁺, Mg²⁺ and Zn²⁺ ions. Chitin and proteins were identified as the major constituents responsible for Ni(II) ion removal by CSP. Of the eluting agents examined, an aqueous solution of the sodium salt of EDTA (in NH₄OH, pH 9.8) was chosen as the most suitable for desorbing Ni(II) ions. Low biomass damage, high elution efficiency and low cost made this type of EDTA elution most appropriate for CSP. Thus, crab shell particles can serve as an excellent biosorbent of low cost, high rigidity, high sorption/resorption efficiency and operation over a wide range of pH values. Such behaviour would also favour the use of crab shell particles as an adsorbent in column studies.

REFERENCES
