Desorption and Regeneration Characteristics for Previously Adsorbed Indium Ions to Phosphorylated Sawdust

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Abstract

The desorption characteristics of previously adsorbed indium ions on phosphorylated sawdust were tested by various chemical reagents such as HCl, HNO3, NaCl, ethylenediaminetetraacetic acid, and nitrilotriacetic acid. Among them, HCl was chosen as the best desorbing agent in terms of economics. The desorption efficiency of HCl for indium ions was about 97% at a concentration of 0.5 M. The desorption efficiency for indium ions was very high at about 94% even at a solid/liquid ratio of 10.0, and the desorption process was quickly performed within 60 min. The removal efficiency of indium ions in recycled phosphorylated sawdust could be maintained at 85% in the 4th cycle.

Keywords: Desorption, Indium ions, Recovery, Regeneration, Sawdust

1. Introduction

The control of metal-loaded and used adsorbent after adsorption process is very important and gives useful information to allow for the economic design of an overall operation. In this sense, desorption and reutilization of the adsorbed metals in metal-recovery processes from water and wastewater are also important. In general, desorption can be performed by proton exchange using acids, chelating agents (ethylenediaminetetraacetic acid [EDTA], nitrilotriacetic acid [NTA]) or exchange with other ions (i.e., NaCl, CaCl2) [1, 2]. The choice of desorbing agent depends on the kind of adsorbent used and the metals adsorbed. An effective desorbing agent is one that desorbs the metal entirely without degradation of the properties of the adsorbent. After metal desorption by means of some treatment, a regeneration step can be used to prevent adsorbent malfunction or loss of adsorption capacity. Also, the ability to regenerate the sorbent is very important in both batch and continuous processes, especially when expensive and selective sorbents are used.

Recently, concern for recycling valuable metals is increasing. Therefore, the recovery of indium ions mainly discharged from indium-tin oxide (ITO) has been widely studied. We have already shown that indium(III) ions could be selectively adsorbed by phosphorylated sawdust, which has economic merits compared to other adsorbents [3]. Unfortunately, however, there is little study on the recovery of the indium(III) ions and the regeneration of the adsorbent. In the present study, the characteristics for desorption and regeneration of indium(III) ions adsorbed onto phosphorylated sawdust was investigated with various desorbing agents.

2. Materials and Methods

The manufacturing methods, physical and chemical characteristics for phosphorylated sawdust are described in the references [3]. For the desorption experiment, previously adsorbed indium ions on phosphorylated sawdust were transferred to a flask containing 100 mL of desorbing agent such as HCl, HNO3, EDTA, NTA, or NaCl. The mixture was shaken at 250 rpm using a rotary shaking incubator (SI-600R; JEIO TECH, Seoul, Korea) at room temperature for 24 hr. Twenty-four hr was long enough to achieve an equilibrium state. After choosing the best desorbing agent, experiments to determine the optimal desorption conditions were performed. The effects on desorption efficiency of concentration, solid/liquid (S/L) ratio (the ratio of the added amount of adsorbent to the volume of desorbing agent), and desorption time were investigated. The desorption efficiency of indium(III) ion from the phosphorylated sawdust was calculated as the ratio between the amount of indium ions desorbed and the amount of indium ions adsorbed. In the regeneration experiment, the adsorbent metal that was eluted by the desorbing agent was thoroughly washed three times with deionized water to remove any traces of desorbing agent, and then mixed again.
On the other hand, HCl could desorb 72% of the indium ions. Although the desorption efficiency of HCl was lower than that of NTA and EDTA, the best desorbing agent for indium ions was chosen as HCl in terms of economic feasibility. In general, EDTA is well known as a very strong chelating agent for metal ions [4]. The investigation for the optimal concentration of HCl was also carried out. HCl concentrations of 0.1, 0.3, 0.5, 0.7, and 0.9 M were tested for elution of adsorbed indium ions. The initial indium loading for phosphorylated sawdust was 1.075 mg/g dry mass [3]. Fig. 2 shows that the desorption efficiency for indium ions increased as the concentration of HCl increased. However, changes of desorption efficiency did not occur at HCl concentrations over 0.5 M. Therefore, with 97% desorption efficiency, the 0.5 M was chosen as the optimal concentration for HCl in terms of cost and efficiency. As mentioned, the S/L ratio is a very important operation parameter in batch processes [5]. Various S/L ratios including 0.01, 0.1, 1.0, and 10.0 were tested. Generally, it is well known that as the S/L ratio decreases, desorption efficiency increases [6]. But in terms of the cost, there is an ideal value. Fig. 3 shows that as the S/L ratio increased, desorption efficiency decreased. At very low S/L ratio, the desorption efficiency approached 100% because much desorbing agent was used for comparatively less phosphorylated sawdust. The desorption efficiency for indium ions was very high at about 94%, even at an S/L ratio of 10.0. However, to apply a desorption operation to real processes, the concentration factor (data not shown), which is defined as the ratio of desorbed metal concentration in a desorbing agent solution to the initial metal concentration in aqueous solution, must be also considered as another important parameter [7]. The concentration factor generally increased with the S/L ratio. Fig. 4 shows that the desorption efficiency for indium ions increased with time. However, most of the desorption process was completed within 60 min. It means that desorption process was performed very quickly. Generally, it has been reported that most of the desorption processes finish within 180 min [5]. To develop a batch treatment process, it is essential to describe regeneration aspects of the process in order to improve its cost-effectiveness by recycling the adsorbent for reuse in multiple cycles. In order to investigate regeneration ability for phospho-

in wastewater containing indium(III) ion for the next adsorption cycle. This procedure was employed for four consecutive cycles. The removal efficiency of indium ions for each cycle was calculated using the following equation:

\[ R.E = \left( \frac{C_i - C_f}{C_i} \right) \times 100 \]

where R.E is the removal efficiency of indium ions (%), \( C_i \) is the initial concentration of indium ions (mg/L), and \( C_f \) is the final concentration of indium ions (mg/L). The concentration of indium ions was analyzed by atomic absorption spectroscopy (AAnalyst 100, AAnalyst 700; PerkinElmer Inc., Waltham, MA, USA). All experiments were performed three times, and the average values are presented.

3. Results and Discussion

As shown in Fig. 1, in the case of EDTA and NTA, desorption efficiencies for indium ions were about the same at around 79%. On the other hand, HCl could desorb 72% of the indium ions. Although the desorption efficiency of HCl was lower than that of NTA and EDTA, the best desorbing agent for indium ions was chosen as HCl in terms of economic feasibility. In general, EDTA is well known as a very strong chelating agent for metal ions [4]. The investigation for the optimal concentration of HCl was also carried out. HCl concentrations of 0.1, 0.3, 0.5, 0.7, and 0.9 M were tested for elution of adsorbed indium ions. The initial indium loading for phosphorylated sawdust was 1.075 mg/g dry mass [3]. Fig. 2 shows that the desorption efficiency for indium ions increased as the concentration of HCl increased. However, changes of desorption efficiency did not occur at HCl concentrations over 0.5 M. Therefore, with 97% desorption efficiency, the 0.5 M was chosen as the optimal concentration for HCl in terms of cost and efficiency. As mentioned, the S/L ratio is a very important operation parameter in batch processes [5]. Various S/L ratios including 0.01, 0.1, 1.0, and 10.0 were tested. Generally, it is well known that as the S/L ratio decreases, desorption efficiency increases [6]. But in terms of the cost, there is an ideal value. Fig. 3 shows that as the S/L ratio increased, desorption efficiency decreased. At very low S/L ratio, the desorption efficiency approached 100% because much desorbing agent was used for comparatively less phosphorylated sawdust. The desorption efficiency for indium ions was very high at about 94%, even at an S/L ratio of 10.0. However, to apply a desorption operation to real processes, the concentration factor (data not shown), which is defined as the ratio of desorbed metal concentration in a desorbing agent solution to the initial metal concentration in aqueous solution, must be also considered as another important parameter [7]. The concentration factor generally increased with the S/L ratio. Fig. 4 shows that the desorption efficiency for indium ions increased with time. However, most of the desorption process was completed within 60 min. It means that desorption process was performed very quickly. Generally, it has been reported that most of the desorption processes finish within 180 min [5]. To develop a batch treatment process, it is essential to describe regeneration aspects of the process in order to improve its cost-effectiveness by recycling the adsorbent for reuse in multiple cycles. In order to investigate regeneration ability for phos-
Previously adsorbed indium ion on phosphorylated sawdust was effectively desorbed by HCl. Desorption efficiency for indium ion was about 97% when a concentration of 0.5 M was used. Also, the desorption efficiency for indium ions was very high at about 94%, even at an S/L ratio of 10.0, and most of desorption process was completed within 60 min. In addition, the desorption efficiency of recycled phosphorylated sawdust for indium ions can be kept at 85% through 4 cycles.

**4. Conclusions**

Previously adsorbed indium ion on phosphorylated sawdust was effectively desorbed by HCl. Desorption efficiency for indium ion was about 97% when a concentration of 0.5 M was used. Also, the desorption efficiency for indium ions was very high at about 94%, even at an S/L ratio of 10.0, and most of desorption process was completed within 60 min. In addition, the desorption efficiency of recycled phosphorylated sawdust for indium ions can be kept at 85% through 4 cycles.

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