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Selective separation of Ni(II)/Co(II) ions from dilute aqueous solutions using continuous electrodeionization in the presence of EDTA

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ABSTRACT

The selective separation of Ni^{2+}/Co^{2+} ions from their dilute solution using continuous electrodeionization process, facilitated with complexing agent, was investigated in this study. In order to increase the selective removal of Ni^{2+} ion, EDTA was used as a complexing agent and Taguchi method was implemented for systematic design of the experiments. The statistical analysis of the experimental results showed that EDTA:Ni mole ratio and feed flow rate were the most influential parameters on performance of the process with contributions of 55.3% and 28.1%, respectively. The comparative experiments proved that the effective separation of Ni^{2+}/Co^{2+} in the absence of EDTA was impossible. Also, electrodeionization process was more effective than electrodialysis process for selective separation of Ni^{2+}/Co^{2+} when EDTA was used in both methods. Ni^{2+}/Co^{2+} ions molar ratio increased from its initial value of 3 to a final value of 154.6 at optimum operating condition, as predicted by Taguchi method. Finally, obtained results showed that the applied procedure can be considered as a promising method for selective separation of Ni^{2+}/Co^{2+} ions from their dilute aqueous solution.

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1. Introduction

The separation of nickel and cobalt ions from each other is generally a difficult task because of their relatively similar properties. The methods in use for this purpose usually tend to be more efficient in removing cobalt ion from aqueous solution in the presence of nickel ion in comparison with separation of nickel ion in the presence of the other one. Most of these methods are based on differences in solubility, kinetic, or thermodynamic stability of the chemical compounds of these ions such as their hydroxides, oxides, carbonates, amine complexes, etc. Also these ions can be separated from each other by methods based on different cementation rates of the dissolved metal ions when reduced by hydrogen or other metals such as zinc. As well as those methods the physicochemical separation methods including ion exchange, solvent extraction and liquid membrane technology are used for this purpose [1–3].

The separation of cobalt and nickel ions has been done by using electrodialysis (ED) in the presence of EDTA, exploiting the greater stability of the EDTA complex with nickel [3–5]. Using electrodialysis process coupled with complexing process has been reported by other researchers for the separation of different metal ion systems [6–11]. However, concentration polarisation and low concentration range of solutions used in ED process are two important limiting parameters always encountered in this process [12].

To overcome these problems, electrodeionization (EDI) has been suggested as a promising alternative. In this hybrid process a fixed bed of ion exchange resins is placed into ED cell to increase the overall conductivity of the system which helps overcome the problems encountered in ED process and dilute solutions can be treated with lower cell voltage to reach acceptable separations [12–14]. In its main application, ultrapure water production, a mixed bed of cation and anion exchange resins is used in continuous electrodeionization (CEDI) process. However, this configuration of EDI process cannot treat the solutions containing heavy metal ions because of metal hydroxides precipitation [15–17]. In a different configuration, a bed of cation exchange resins is placed between two cation exchange membranes for metal ions removal from electro-plating waste streams [18–21]. In this configuration, the cation exchange resin bed is continuously regenerated electrochemically by H^+ ion produced in the anode compartment.

So far, only a few studies have been done on the application of EDI process coupled with complexing process for the selective separation of metal ions [22]. In this study, EDI was used in the presence of EDTA, as a complexing agent, for selective separation of Ni^{2+}/Co^{2+} ions from a dilute solution containing total metal ions concentration of 100 ppm. EDTA was selected because its 1:1 overall formation constants with Ni^{2+} and Co^{2+} are high enough, but still a certain difference exists between these two constants [23].

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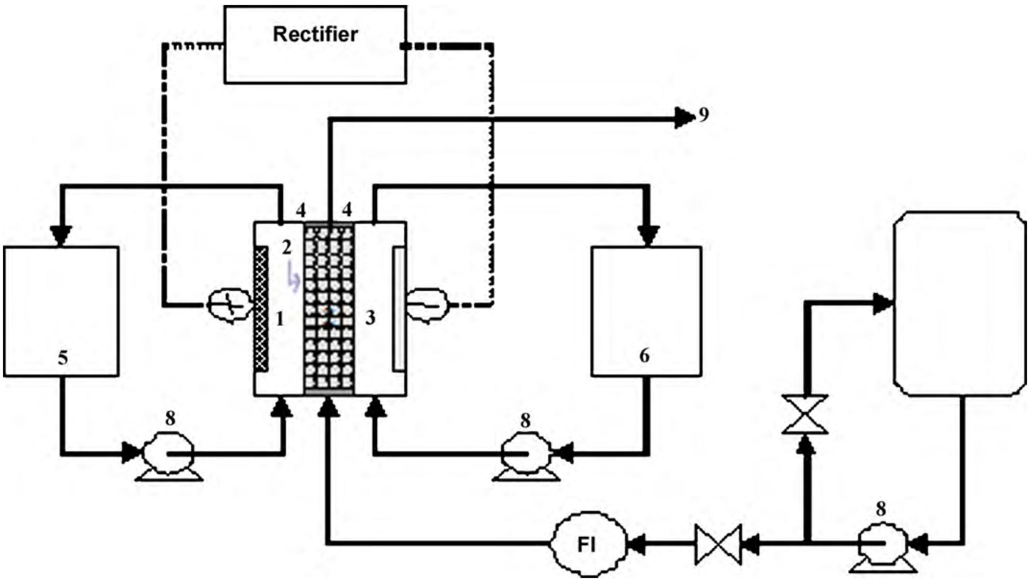

Fig. 1. Experimental set up of the continuous EDI system: (1) anode compartment; (2) ion exchange bed; (3) cathode compartment; (4) cation exchange membranes; (5) anolyte reservoir; (6) catholyte reservoir; (7) feed solution reservoir; (8) pump; (9) product (to waste container); FI: flowmeter.

These constants ($\log k_f$) for Ni^{2+}–EDTA and Co^{2+}–EDTA are 18.56 and 16.31, respectively [24].

During the experimental study of the process, the effects of independent parameters such as feed flow rate and pH of the feed solution, cell voltage, the initial molar ratio of the metal ions, and EDTA:Ni mole ratio (0.5, 0.7, 1.0) on separation factor were investigated. The Taguchi experiment design method was used for lowering the number of required experiments and estimation of optimum conditions. Also the relative importance of independent parameters can be determined by this method. To evaluate the success of the applied process and make a relative comparison between this process and others, some simple EDI (without EDTA) and ED (in the presence of EDTA) experiments were carried out at similar operating conditions.

2. Experimental

2.1. Experimental set up

The experimental set up used in this work is shown in Fig. 1. The key part of the set up was a three-compartment plexiglas cell. The middle compartment of the cell was separated from the other two by two cation exchange membranes (CR67 MKIII, Ionics, USA). This compartment was filled with strong acid cation exchange resins (Purolite, C100E), initially in the H^+ form. The effective area of the membranes was 90.25 cm^2 and the thickness of all compartments was 8 mm. The specifications of the membrane and resins are listed in Tables 1 and 2, respectively. Prior to use, the membranes were treated with HCl (1 M) for 2 h and then washed with distilled water.

Platinum coated titanium mesh and stainless steel electrodes were used for anode and cathode, respectively. The area of each electrode was equal to 69.6 cm^2. Two H_2SO_4 (0.05 M) reservoirs, 2000 cm^3 each, were connected to the electrode compartments and acid solutions were pumped into these compartments by two submersible centrifugal pumps (Model YJ200S-2X CW, Electrogen Co., Iran). Besides acting as electrode rinse, these two solutions gave enough conductivity to the electrode compartments. The feed solution passed through the middle compartment using a diaphragm pump (Model CAPEX 30, Charles Austen Pumps Ltd., England). An adjustable power supply (Model STAR 305, Iran) was used to supply direct current (DC). It was able to supply voltage and direct current in the range of 0–40 V and 0–4 A, respectively.

2.2. Feed solution and reagents

The analytical grade of EDTA disodium salt and other inorganic chemicals such as HCl, NaOH, H_2SO_4, $CoSO_4$, and $NiSO_4$ were purchased from Merck Co. The feed solution was prepared by dissolving proper amounts of $CoSO_4$, $NiSO_4$ and EDTA in distilled water. Sodium hydroxide (0.1 M) or dilute hydrochloric acid solutions were used to adjust the initial pH of the feed solution. The solution pH was measured with a digital pH meter (HANNA Model HI 1230B, Italy). The feed solutions used in these experiments had various pH values. Also molar ratio of the metal ions (Ni:Co) was changed while the initial total molar concentration of them was kept constant at 100 ppm for all experiments.

Table 1
Properties of the CR67, MKIII cation exchange membrane (Ionics, Watertown, MA, USA).

Specification of the membrane	
Matrix structure	Homogeneous polystyrene/DVB copolymer
Functional group	Sulfonic acid
Specific weight (mg/cm^2)	13.7
Thickness (mm)	0.5
Burst strength (kg/cm^2)	7.0
Water content	46% of wet resin only
Capacity (mequiv./dry gram resin)	2.4
Chemical stability, pH	1–10

Table 2
Properties of the Puroulite C100E cation exchange resin.

Specification of the resin	
Matrix structure	Polystyrene/DVB copolymer
Functional group	Sulfonic acid
Particle size	0.3–1.2 mm
Chemical stability, pH	0–14
Moisture content	46–50%
Total capacity:	
Na^+ form, dry	4.5 equiv./kg
Na^+ form, wet	1.9 equiv./L

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