



Nickel recovery from synthetic Watts bath electroplating wastewater by homogeneous fluidized bed granulation process



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ABSTRACT

This study determined the removal of nickel from a synthetic Watts bath electroplating composition ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, and H_3BO_3) by homogeneous fluidized-bed granulation process. The effects of adding calcium ions present in an electroplating bath while varying influent nickel concentration (200 $\text{mg} \cdot \text{L}^{-1}$, 300 $\text{mg} \cdot \text{L}^{-1}$, and 400 $\text{mg} \cdot \text{L}^{-1}$), $[\text{CO}_3^{2-}:\text{Ni}^{2+}]$ molar ratio (1.0, 1.5 and 2.0) and pH of carbonate precipitant (10.5, 10.8, and 11.0) were evaluated. Calcium ion addition was varied at 20 $\text{mg} \cdot \text{L}^{-1}$, 50 $\text{mg} \cdot \text{L}^{-1}$, and 100 $\text{mg} \cdot \text{L}^{-1}$. The best operating conditions were at 300 $\text{mg} \cdot \text{L}^{-1}$ influent nickel concentration, $[\text{CO}_3^{2-}:\text{Ni}^{2+}]$ molar ratio of 1.5, and pH of 10.8 of the precipitant from different types of water source. The removal of nickel from synthetic Watts bath electroplating wastewater using tapwater was 84.93%, and 92.66% with the addition of Ca^{2+} ion at 20 $\text{mg} \cdot \text{L}^{-1}$. For the synthetic Watts bath electroplating wastewater using reverse osmosis water, nickel removal was 97.08% and 98.51% when pure nickel ion in the solution was used. The characterized granules has low symmetry that confirmed the characteristics of a nullaginite mineral of $\text{Ni}_2(\text{CO}_3)(\text{OH})_2$.

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1. Introduction

Nickel is a silvery-white, lustrous, fifth most common metallic element that occurs naturally, though it is unreachable in the central part of the earth. Nickel is used for catalysis, coins, jewelry, batteries, industrial plumbing, nickel alloys, spark plugs, electroplating, machinery parts, stainless-steel, nickel-chrome, and resistance wires [1–3]. According to Nickel Institute in Europe [1], the manufacture of stainless steels can consume about 65% of the total nickel production, for nickel plating, 9%, and for coins, batteries for portable equipment, electronics and hybrid cars uses, 6%. Due to the demands of nickel in various industries, much consideration is placed on it. According to studies, about 62% of nickel found in wastewater came from electroplating industries, 25% came from residential wastewaters, 9.8% from runoff and the remaining 3.2% came from other industrial units [4]. At high concentrations, nickel can be very toxic [5–8]. According to the International Agency of Research on Cancer in 1990, nickel was evaluated as carcinogenic that poses adverse effects in human, animal health, and environment [9]. Exposure above 1 $\text{mg} \cdot \text{m}^{-3}$ soluble to 10 $\text{mg} \cdot \text{m}^{-3}$ less soluble nickel concentration was said to be carcinogenic.

Nickel is a well-known chemical used for coating different materials. Moreover, it improves the corrosion resistance of materials causing them to last longer. Watts bath is the most commonly applied nickel-electroplating bath which is composed of nickel chloride (40–60 $\text{g} \cdot \text{L}^{-1}$), nickel sulfate (240–310 $\text{g} \cdot \text{L}^{-1}$), and boric acid (30–45 $\text{g} \cdot \text{L}^{-1}$). The compounds in the bath are used to improve the surface finishing of the materials, to add brightness of the deposits, to serve as corrosion-proofing material, and to provide decorative characteristics [10–12]. Nickel sulfate is the principal source of nickel ion in the bath. Nickel chloride improves anode dissolution and increases solution conductivity. Conversely, boric acid helps control the pH of the bath, and produces smoother and more ductile deposits [11,12].

Various methods were studied on nickel recovery from wastewater namely: the electrochemical processes such as electrodialysis [10], electrocoagulation [13], electrodeionization [14], ion-exchange [15] and adsorption [16–19]. Chemical precipitation is the most commonly used technique for heavy metal removal in wastewater, but due to high sludge formation, various studies were done for its alternate. Another popular technique is the use of fluidized-bed reactor (FBR) [20–22] with seeding material of choice. Many recent studies were done on the use of fluidized bed granulation process to recover the heavy metals or inorganic acids such as As, Cu, Ni, Zn, boric, fluoride and phosphorous acid

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[23–27]. The latest was the recovery of lead carbonate from wastewater with seeding material and compared it without using the seeding material by fluidized-bed homogeneous crystallization system. In a homogeneous fluidized-bed process, the influent metal salts collect sufficient active sides which later produced homogeneous granules. Then, nucleation and particle growth occurred, making particles of high-purity at increasing mass. FBR consist of water interactions that are similar to that involved in the traditional precipitation process, but the nuclei development and granulation mechanisms inside can be empirically estimated with the hydraulic conditions in the metastable region, to form dense granules instead of soft sludge [28].

This study investigated the factors affecting the removal and granulation efficiencies by understanding the mechanisms leading to production of granules by adjusting influent nickel concentration, molar ratio $[\text{CO}_3^{2-}:\text{Ni}^{2+}]$, and pH values of the precipitant. The best operating conditions that resulted to low nickel effluent concentration in a homogeneous fluidized-bed granulation process, the effect of adding calcium ions in its waste streams, on granule formation and removal performance were determined. Moreover, the granules formed were characterized to validate its composition using Scanning Electron Microscope (SEM) for the surface morphology of the granules, Energy Dispersive Spectrometer (EDS) for the elemental distribution, X-ray Diffraction Spectrometry (XRD) for the elemental composition and Fourier Transform Infrared (FTIR) for determining the functional groups.

2. Materials and methods

2.1. Chemicals

All chemicals were reagent grade and used without further purification. The synthetic Watts electroplating compositions were prepared by dissolving 240–310 $\text{g}\cdot\text{L}^{-1}$ nickel (II) sulfate hexahydrate ($\text{NiSO}_4\cdot 6\text{H}_2\text{O}$, 99%, Shimakyu's Pure Chemicals, Osaka, Japan), 40–60 $\text{g}\cdot\text{L}^{-1}$ nickel chloride hexahydrate ($\text{NiCl}_2\cdot 6\text{H}_2\text{O}$, 98%, Panreac, Spain) and 30–45 $\text{g}\cdot\text{L}^{-1}$ boric acid (H_3BO_3 , 99.5%, Shimakyu's Pure Chemicals, Osaka, Japan) in a laboratory-grade reverse osmosis (RO) ultrapure water system (18.2 $\text{M}\Omega$ resistance). The influent nickel concentrations were prepared at 200 $\text{mg}\cdot\text{L}^{-1}$, 300 $\text{mg}\cdot\text{L}^{-1}$ and 400 $\text{mg}\cdot\text{L}^{-1}$ [6,10–12]. Sodium carbonate (Na_2CO_3 , Panreac, Spain) was used as the precipitant solution. Calcium chloride dehydrate ($\text{CaCl}_2\cdot 2\text{H}_2\text{O}$, 75.9% Choneye Pure Chemicals, Taipei, Taiwan) was used for the addition of calcium ions in the synthetic wastewater solution.

2.2. Nickel carbonate removal and granulation in FBR

The upper part of a 1.35 L glass laboratory-scale fluidized-bed reactor (FBR) with 4.0 cm inner diameter and 20 cm height is the effluent region. The reaction region is on the lower part with 2.0 cm diameter and has 80 cm height. Glass beads (0.5 mm diameter) were packed at a 4.0 cm static height. There were three inlets at the bottom part of the reactor: two for the reactants (wastewater and precipitants) entering on both sides and the other one for the recycled solutions. On top of it were two outlets, one for the treated wastewater and the other for the recycled solutions. Constant monitoring of pH values of the effluent solutions using a pH/ORP Transmitter/Controller (Shin-Shang Tech Instruments Co., Ltd. PC-310) with ± 0.05 accuracy in the FBR was done. The flow rate of the solutions was controlled by a Masterflex L/S digital economy drive with easy-load peristaltic pumps (Cole-Parmer Instrument Co., USA).

2.3. Experimental procedures

In an upward direction, the synthetic Watts bath electroplating wastewater and the precipitant solution were introduced as inflow at the bottom part of the reactor in two opposite inlets as shown in Fig. 1. The inflow rates were controlled to achieve the desired hydraulic retention time. The desired pH values of the precipitant were adjusted by adding the 0.12 M nitric acid (HNO_3 , 69%, Panreac, Spain) and 4.0 M sodium hydroxide (NaOH 98%, Shimakyu's Pure Chemicals, Osaka, Japan). Operating conditions varied are the influent nickel concentration, molar ratio and pH value of the precipitant. Two parameters were kept constant while the chosen parameter was changed progressively. The entire process was continuous at room temperature, while the reflux flow rate was adjusted to retain the granule formation in a fluidized state.

To prepare the concentrated metal solution, $\text{NiSO}_4\cdot 6\text{H}_2\text{O}$ (pH 7.5–7.8) was dissolved in RO water (pH 8.50) and varied at different conditions. The synthetic solution simulating the rinse water in an electroplating process corresponds to the desired nickel concentration of effluent wastewater to be treated. The effect of using tap water was evaluated to determine the possible outcome of granule production with the presence of calcium ion in the solution. Synthetic wastewater was also evaluated to compare the effects of other compounds in the nickel bath wastewater in the reaction.

Forty (40) mL of effluent samples were withdrawn twice at the reflux level at the upper part of the reactor during the continuous operation for 72 h. Twenty (20) mL effluent sample was filtered using 0.45 μm membrane filters (GHP membrane, Pall) to achieve the dissolved nickel effluent concentrations, whereas the other twenty (20) mL was left as drawn. The suspended nickel effluent concentrations was the difference between the nickel concentration of the unfiltered samples and the dissolved nickel effluent concentrations. Both samples taken were treated completely by acid preservation and digestion [29].

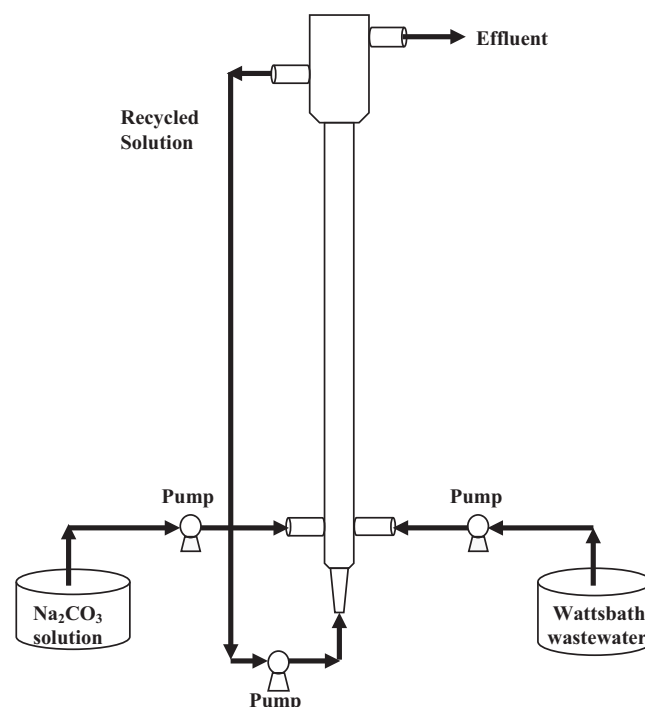


Fig. 1. Diagram of the fluidized-bed reactor set-up.

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