

Electrogenerative Process as an Alternate Technique for Nickel Recovery from Its Sulphate Solution at Low Concentration

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Abstract In this study, a static batch reactor equipped with three-dimensional (3-D) carbon felt (CF) as cathode material was fully optimized to recover a low nickel concentration from its sulphate solution. Nickel recovery was performed using several nickel concentrations ranging from 50, 100, 250, 400 and 500 mg/L. Moreover, within 30 min of electrogenerative operation, it was discovered that the recovery of nickel achieved > 90 % in acidic conditions. At last, the deposition of nickel into CF was further investigated by scanning electron microscopy (SEM) to visualize the surface morphology of nickel on CF.

Keywords: Electrogenerative system; nickel recovery; carbon felt electrode; scanning electron microscopy.

Introduction

Heavy metals (HMs) are described as a group of elements that exist in metal form having comparably high-density levels compared to water [1]. HMs can be categorized into two parts: essential HMs and non-essential HMs. Essential HMs are metals that living organisms need to carry out processes such as growth and even play an essential role in specific organs. For example, iron (Fe) is important in forming an essential part of hemoglobin, one of the blood's main proteins to transport oxygen along the body [2]. Moreover, the body needs other metals such as copper, manganese and zinc as an important micronutrients and possess a biochemical function in living organisms [3]. In contrast, non-essential HMs such as mercury, cadmium and chromium are not required by living organisms and are considered toxic [4-6]. Usually, the necessary amount of HMs required is a small amount. Excessive consumption of HMs makes HMs poisonous and will adversely affect the health of living organisms and the environment. In this era, there is raising concern among the community about the effects resulting from the increase of heavy metal content in the environment.

Among the HMs, nickel is a nonferrous metal widely used in various industrial applications, such as producing stainless steel, electroplating, nonferrous alloys, and nickel-based superalloys [7, 8]. Nickel is considered an odourless and tasteless material [9]. Nickel is an essential element with significant importance in enzymes responsible for metabolic pathways such as acidogenesis, hydrogen metabolism, methane biogenesis, and ureolysis [10]. According to USEPA, nickel has an acceptable maximum of 0.02 mg/L in drinking water [11]. The immense use of nickel in elevated high dosage, however, will have health effects on living organisms as well as biological processes in wastewater treatment.

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Various health and environmental issues because of exposure to nickel have been reported due to its highly toxic, non-biodegradable, and also tends to accumulate in living organisms and influence the health of the living organisms and the environment [12]. Excessive nickel intake can result in severe lung and kidney problems, pulmonary fibrosis, and skin dermatitis [13-15]. In addition, there have also been cases of death by respiratory distress syndrome (ARDS) recorded due to exposure to nickel among spray painting workers [16]. At the same time, the use of nickel is also closely related to risks and issues to the environmental impact where it is linked to nickel-containing materials produced in the nickel industry. Nickel is one of the essential components in the alloying addition of the stainless steel made today. The production of nickel metal or nickel alloys involves processes such as pyrometallurgical and hydrometallurgy, where the result of this process will produce large amounts of solid and liquid wastes (slags and leaching residue) in the environment. Furthermore, nickel manufacturing through pyrometallurgy also releases large amounts of greenhouse gases (GHGs) into the air, water and soil [17]. In this situation, the degree of nickel contamination depends on several factors, such as the chemical composition, characteristics of the wastes produced, the quality and type of nickel ore utilized, and the extraction method used. Therefore, before discharging industrial waste into natural water bodies, it is essential to eliminate the number of nickel ions from industrial waste.

Various techniques and methodologies, including ion exchange [18], precipitation [19], ultra-filtration [20], phytoextraction [21], sequential extraction [22], reverse osmosis [23], and adsorption [24] have been used to remove nickel from the solution. However, at low metal ion concentrations, most of these methods are costly, complex, and less efficient, which does not apply to extensive pollutant ranges. Another alternative method that can be applied to recover HMs at low concentrations with a much greener approach is introduced to overcome this problem. The electrogenerative method is a redox reaction, whereas two distinct cell compartments undergo a spontaneous electrochemical reaction. The main idea of the electrogenerative method is that it is a spontaneous oxidation-reduction reaction where more electropositive ions will reduce at the cathode while more electronegative metal will oxidize. The byproduct of this process will produce energy corresponding to the chemical reaction in which it is converted into an external current potential [25]. One of the unique features of this method is that it does not require any power supply to generate the reaction compared to other methods in metal recovery. In addition, the operating cost of this method is also lower due to the simplicity and straightforwardness when experimenting. It also has more benefits in reducing effluents and side reactions resulting from the reaction. Due to its unique characteristics, electrogenerative is seen as a potential developing method for utilizing greener and environmentally friendly technologies for recovering HMs.

Despite the high constancy of nickel pollution, there have not been specified research or studies on recovering low-concentration nickel from its sulphate solution using an electrogenerative method. In addition, there has been a limited number of studies for the more methodical database that utilized the electrogenerative method in providing a more excellent perception of the importance of the recovery process in wastewater treatment. Therefore, this study was conducted to discover and experiment in obtaining information from the recovery of low-concentration nickel from its sulphate solution. In addition, the optimum conditions required to recover nickel were also observed and determined.

Materials and Methods

Chemicals and Materials

As stated in the experiment, all solutions were prepared from analytical-grade reagents purchased from Sigma Aldrich (Steinheim, Germany). The chemicals used in this study were nickel (II) sulphate (NiSO_4), sodium chloride (NaCl), nitric acid (HNO_3), and ethanol ($\text{C}_2\text{H}_5\text{OH}$) were obtained from Merck (Darmstadt, Germany). Other than that, the materials utilized throughout the study were ion exchange membrane Neosepta® AM-01 from Tokuyama Corp (Tokyo, Japan). The cathode material used for nickel recovery was carbon felt (CF) purchased from Hangzhou Nature Technology Co. Ltd (Hangzhou, China) with the dimension of 2.0 cm x 5.5 cm x 0.3 cm, and the anode material used was pure zinc foil (99% purity) with the measurement of 2.0 cm x 4.5 cm x 0.05 cm purchased from the local hardware store. Lean Hong Hardware Trading Company (Penang, Malaysia) supplied nitrogen gas. A 1000 mg/L standard nickel solution was diluted to a few concentrations for sample analysis.

Instruments

Two digital multimeters used were digital multimeters (Sanwa CD800A) purchased from Sanwa Electric Instrument Co., Ltd (Tokyo, Japan) which are modern, reliable measuring devices characterized by high measurement accuracy and various functional capabilities. The multimeters were attached to the anode and cathode to measure and detect voltage or electrical potential (volt) and the cell's electrical current (ampere) flow during the experiment. The reading of both current and voltage was recorded every 30

minutes during the 4 h of the experiment. The electrolytes' pH was measured using a pH meter modelled Thermo Orion model 720 A⁺ obtained from ThermoScience (Waltham, MA, USA). The nickel determination was performed using Atomic Absorption Spectroscopy (AAS) with the model of Perkin Elmer Analyst 200 (Waltham, MA, USA) with an air acetylene flame at a wavelength of 232 nm, the acetylene flow rate at 2.50 L/min, the oxidant flow rate at 10.00 L/min and slit 1.8/1.35 mm. Quanta FEG 650 scanning electron microscopy (SEM) obtained from Oxford Instruments (Oxfordshire, UK) was utilized to visualize the morphological surface.

Electrogenerative Static Batch Cell Design

The static batch cell comprises two electrolyte cell compartments made from Plexiglass, whose materials are strong, tough, and lightweight with a higher impact strength than glass. The dimensions for the cell compartments were 4.7 cm x 5.3 cm x 9.3 cm, respectively and are detached by an ion exchange membrane Neosepta® AM-01. Nine bolts and nuts were used around the cell to hold both cell components together. The distance between the anode and cathode was 2.0 cm, while the distance between the electrodes and the membrane was 1.0 cm, respectively. The current collectors were attached to a digital multimeter by external conducting wires to complete the circuit. A magnetic stirrer was placed in the middle of the catholyte compartment to stir the catholyte, and the speed was set at 400 rpm throughout the experiment. The experiment was carried out at room temperature. The arrangement of the experiment (Figure 1) corresponds to the experimental setup [26].

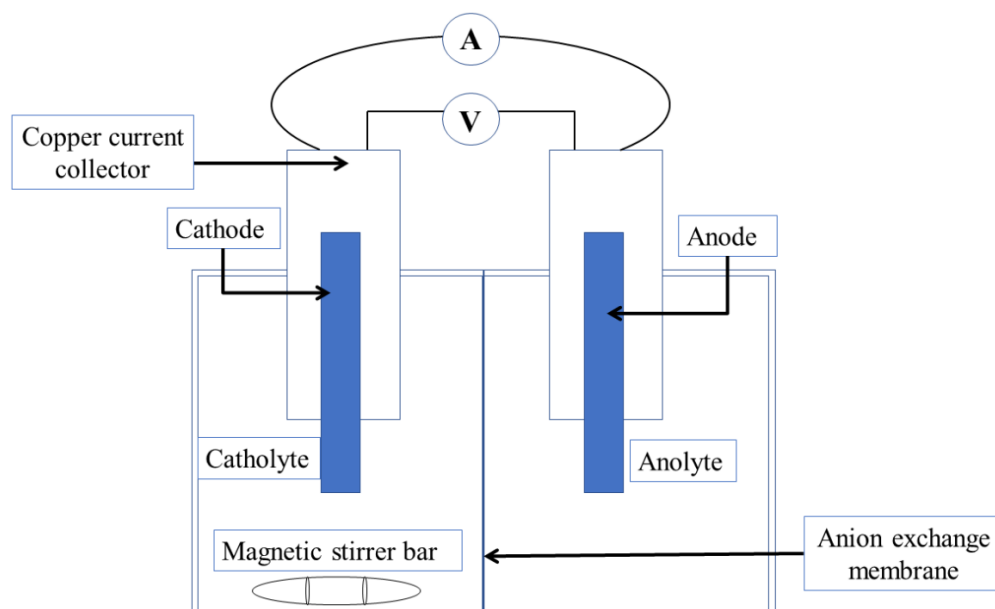


Figure 1. Illustrative diagram of a static batch cell

Electrodes

In this study, the cathode used to recover low-concentration nickel was a CF with higher porosity and higher conductivity [27]. The cathode must go through a pre-treatment procedure first before it is used. Meanwhile, the anode used was zinc foil due to zinc having a lower standard reduction potential than nickel. The dimensions of the electrodes are displayed in Table 1.

Table 1. Details of experimental conditions

Anode component		Cathode component	
Anolyte	250 mL of 0.2 M NaCl solution	Catholyte	250 mL of 500 mg/L Ni ²⁺ in 0.2 M NaCl solution
Anode terminal	Zinc foil (2.0 cm x 4.5 cm x 0.05 cm)	Cathode terminal	Carbon felt (CF) (2.0 cm x 5.5 cm x 0.3 cm)

Ion exchange Membranes (IEMs)

The anion exchange membrane was cut to a dimension of 8.0 cm x 9.0 cm and soaked in distilled water before any use to prevent drying. The membrane was sandwiched between two rubber gaskets in the cell compartment to prevent the electrolyte from leaking. After each experiment, the cell compartment was filled with distilled water to keep the AEM moist.

Pre-treatment of Anodes and Cathodes

For the anode, zinc foil was cut into the dimension, as stated in Table 1, and they were polished with sandpaper to remove the oxide layer formed on the surface of the foil. To ensure the cleanliness of the surface, the zinc foil was rinsed with distilled water and ethanol each time after polishing. Pre-treatment of CF is required to remove contaminants and enhance the surface moisture properties. The pre-treatment procedure for this experiment's cathode corresponds to the suggested experimental setup [26]. The cathode was activated for eight hours before use. Next, the cathode was immersed in ethanol and distilled water mixture with the portion (95:5) for 4 h. Then, the cathode was rinsed with distilled water and dipped again in 0.1 M nitric acid (HNO_3) for another 4 h. At last, the cathode was rinsed again with distilled water and was stored in distilled water to keep the moisture until future use.

Preparation of Electrolytes

All the electrolytes were prepared from analytical-grade reagents and distilled water. The anolyte used in the experiment was 0.2 M NaCl. It was designed by diluting the distilled water with the amount of NaCl needed in a 250 mL volumetric flask. Meanwhile, the catholyte solution was prepared by diluting the distilled water with the amount of anhydrous nickel sulfate, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, in a 1 L volumetric flask. Subsequently, different Ni(II) concentrations were prepared in a 250 mL volumetric flask.

Optimization of the Batch Reactor Conditions

The effect of initial Ni concentration

Several Ni concentrations were investigated to find the optimum concentration for Ni recovery in a sulphate medium. The concentrations ranged from 50 -500 mg/L of the initial Ni^{2+} solution were chosen.

The effect of nitrogen gas purging

The presence of oxygen in the solution might affect the recovery of nickel. Therefore, two conditions were investigated in this study with and without the presence of oxygen. Both anolytes and catholytes were purged with the nitrogen gas in the first place, and next, no action was taken to purge nitrogen gas in the anolyte and catholyte.

The effect of pH

Changes in pH had a significant impact on nickel deposition. The electrolytes' pH was manually adjusted between pH 3 and 5 using 0.1 M sodium hydroxide (NaOH) and 0.1 M hydrochloric acid (HCl).

Electrogenerative Process Experiment for Ni^{2+} Recovery

In the static batch cell, the compartment on the right side was labelled as a cathode, and the compartment on the left side was labelled as an anode. A volume of 170 mL of the anolyte and catholyte was filled into the anode and cathode compartments. Next, the anode and cathode were immersed in the compartments fixedly. Subsequently, a magnetic stirrer was placed into the cathode compartment to stir the catholyte along the electrogenerative process. Meanwhile, two multimeters were linked to the anode and cathode to measure the current (stream of electric flow) and voltage (electrical potential) along the process. The initial value of current and voltage was recorded. Along the electrogenerative process, any current and voltage changes were observed and recorded. Next, 500 μL aliquots of the anolyte and catholyte were collected every 30 minutes of the experiment using an adjustable micropipette to monitor the amount of nickel left in the solution. Distilled water was used to dilute the collected aliquot until 30 mL. The nickel concentration was evaluated by using AAS. At last, the nickel that accumulated on the cathode was washed using 1.0 M nitric acids (HNO_3). The cathode was then rinsed with distilled water and stored in the distilled water until future use. For the experiment condition without the presence of oxygen, nitrogen gas (N_2) was used to purge both anolyte and catholyte for 10-15 minutes before experimenting.

Results and Discussion

Electrogenerative Studies

In the electrogenerative method, the chemical reaction involves a spontaneous oxidation-reduction response where more electropositive ions will reduce at the cathode while more electronegative metal will oxidize. Oxidation reaction occurs where electrons will lose at the anode, while reduction will appear at the cathode, where it will gain electrons. The following equation represents the reaction that may happen in the batch cell:



Equations (1) and (2) represent the half equation that occurred in the anode and cathode. The positive standard reduction potential of the cell, E_{cell}° proved that the reaction involved in this study was spontaneous. Therefore, from the standard reduction potential of the cell, the Gibbs free energy involved in this study can be determined through the formula.

The Gibbs free energy obtained in this study was -97.64kJ/mol. The negative value obtained further reinforces the statement related to spontaneous reaction. The percentage of Ni(II) recovery can be determined from the following equation:

$$\Delta G^{\circ} = -nFE_{\text{cell}}^{\circ} \quad (4)$$

$$\Delta G^{\circ} = -(2)(96485 \text{ C/mol})(0.506 \text{ V})$$

$$\Delta G^{\circ} = -97642.82 \text{ J/mol}$$

$$\Delta G^{\circ} = -97.64 \text{ kJ/mol}$$

$$\text{Percentage of Ni(II) recovery} = \left[\frac{(C_o - C_t)}{C_o} \right] \times 100 \%$$

where C_o is the initial concentration of Ni(II) in mg/L and C_t is the Ni(II) concentration in mg/L at a specific time.

The effect of initial Ni concentration

The initial concentrations for low-concentration nickel recovery were 50, 100 and 250 mg/L. Based on Figure 2, the highest recovery of nickel was achieved at 250 mg/L within 210 min of operation, which is $98.21 \pm 0.221 \%$. Next, the recovery of nickel completed for 50 and 100 mg/L was $97.72 \pm 0.300 \%$ and $94.26 \pm 0.168 \%$, respectively. It was successfully recovered within 30 and 240 min of operation. In addition, it can be seen that at the first 30 min of the operation, the recovery of nickel in every concentration already reached $> 90 \%$ recovery. This further concludes the use of CF as one of the 3-D electrodes that reduce time-consuming cathode material compared to other electrodes [25]. Therefore, this study's optimum concentration for recovery of low-concentration nickel was 250 mg/L respectively.

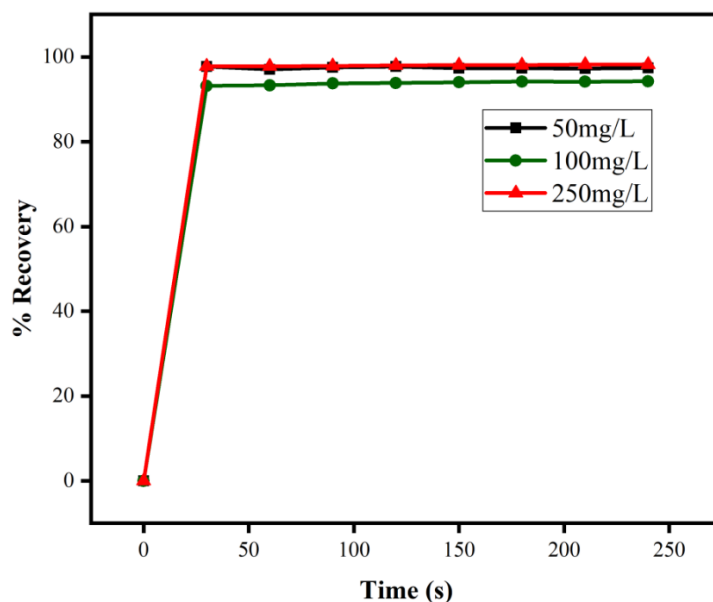


Figure 2. The effect of different initial concentrations of Ni(II) for recovering low-concentration nickel from its sulphate solution within 4 h of electrogenerative performance. The experiment condition; without N₂ gas purging and pH-independent

The effect of the presence of oxygen

The presence of oxygen in the electrolytes affects the nickel deposition rate. The effect of oxygen was further investigated and conducted in two different conditions during the study. The electrolytes were purged with gas nitrogen as the first condition, while the second condition was when the electrolytes were not purged with gas nitrogen. Based on the result, nickel's deposition rate into the cathode increased and then decreased. As reported [28], the change in deposition rate was reflected in the microstructure of the deposits.

With the purging of nitrogen gas, the percent recovery of nickel achieved 97.72 ± 0.300 % compared to 97.15 ± 0.112 % without purging nitrogen gas at 50 mg/L. The recovery of nickel at 100 and 250 mg/L also achieved a higher recovery with the purging of nitrogen gas which was 94.26 ± 0.168 % and 98.21 ± 0.221 %, respectively. Without purging of gas nitrogen, the recovery of nickel achieved a total of 93.92 ± 0.609 % and 96.63 ± 0.114 %, respectively, at 100 and 250 mg/L. These results can be represented by Figure 3(a) - (c).

However, there is little comparison between the total recovery of nickel achieved in purging and without purging with nitrogen gas. Therefore, it can be concluded that oxygen in this study does not affect the performance of nickel deposition into CF.

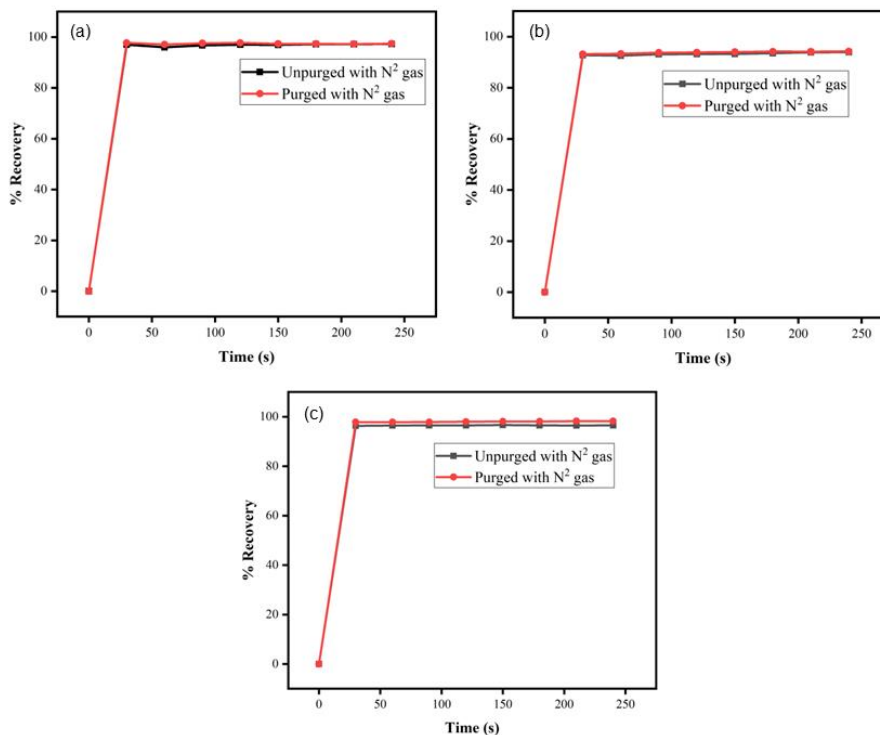


Figure 3. The effect of the presence of oxygen on recovery of low-concentration nickel from its sulphate solution at a) 50 mg/L; b) 100 mg/L; and c) 250 mg/L. The experiment condition; pH-independent

The Effect of pH

The pH condition of the catholyte is one of the crucial parameters in achieving a higher rate of Ni(II) recovery in the electrogenerative method. This is because pH affects the deposition and composition of metal deposited in the cathode [29]. Therefore, this study utilized an analysis to find the optimum pH condition for the recovery of low-concentration nickel. The cathodic nickel reduction is thermodynamically possible within the pH range of 4.9 - 8.3. In the situation where pH was not controlled, the total recovery achieved (96.29 ± 0.21 %) is slightly lower than the recovery in which the catholyte was held (98.42 ± 0.07 %). This may be due to the free acid concentration in the cell, causing decreased pH, eventually hindering the electrolytic reduction.

At $\text{pH} < 3$, hydrogen ions, H^+ would be prevented from accessing the binding sites of the sorbent due to the repulsive forces if H^+ ions were to compete with Ni(II) ions for the surface of the adsorbent. However, the minimal metal removal is most likely caused by increased competition between nickel ions and protons for ligand binding sites and complex formation. When the $\text{pH} > 5$, Ni(II) ions precipitate due to hydroxide anions, OH^- forming a nickel hydroxide precipitate. Therefore, it was determined that this study's optimum pH is pH 4, where catholyte solution with concentration at 250 mg/L generates the highest recovery (98.42 ± 0.07 %) of Ni(II) within 3 h of operation.

Surface Morphological Study using Scanning Electron Microscope

The surface morphology of CF was carried out to confirm the electrodeposition of Ni(II) deposited from 250 mg/L of Ni^{2+} for 3 h. Based on Figure 4(a) - (c) at different magnification scales, it can be seen that the surface of CF resided with nickel after the electrodeposition process. The deposited particles were rough and lumpy structures corresponding to Ni(II) deposition.

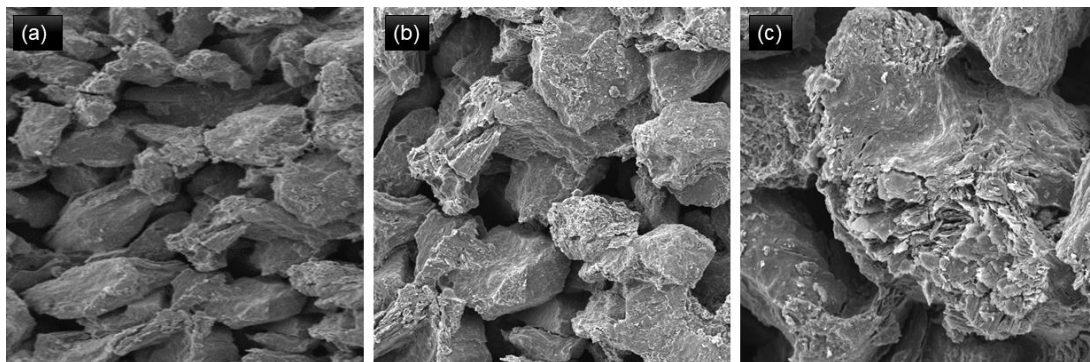


Figure 4. Micrographs of Ni(II) deposition onto CF: (a) micrograph of 100x magnification; (b) micrograph of 200x magnification; (c) micrograph of 500x magnification

Conclusions

Overall, the study that has been conducted in this study has succeeded in obtaining the main objective of this study. Through the electrogenerative method, nickel has been successfully recovered by utilizing the carbon felt material as a cathode. The recovery of low-concentration nickel was achieved > 90 % within 30 min of operation. The highest recovery of low concentration determined is 98.21 ± 0.221 % in 250 mg/L, obtained within 210 min operation. However, the presence of oxygen does not affect the performance of nickel deposition into carbon felt. This is because there is only a slight difference between the recovery percentage with and without the presence of oxygen. Therefore, the optimum concentration for recovery of nickel was determined at 250 mg/L. Subsequently, the optimum pH condition for this study was determined at pH 4 as it generates the highest recovery of Ni(II). This further support the information on the reduction of cathodic nickel, possibly within the pH range of 4.9-8.3. Next, the surface morphological study of carbon felt visualized by scanning electron microscopy (SEM) further supports the deposition of nickel into the cathode. Lastly, the electrogenerative method has been successfully proven as one of the alternative methods that can be applied in recovering low-concentrated HMs and is much more environmentally friendly. The unique features of this method as it does not require an external power supply and the ability to generate its electricity, proving it is one of the promising technologies that can be used in the recovery of the HMs in industry. Some future recommendations can be considered to expand the experimental framework, such as using different electrode materials to determine their impact on the efficiency and selectivity of nickel recovery, including platinum, graphite, and suitable electrode materials. Next, various ion exchange membranes can be considered to replace Neosepta® AM-01 membrane, such as Nafion®, Selemion®, and CMI-7000 Series. These ion exchange membranes offer a range of cation and anion exchange membranes with varying ion selectivity, chemical resistance, and operating temperature ranges. This can enhance the selectivity and stability of membranes used in electrogenerative studies for nickel recovery.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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