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Preliminary Study on Recovery of Nickel from Electro-Nickel-Plating Solution by Electrowinning

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Abstract

This research aims to study the feasibility of nickel recovery from spent electroplating solution via hydrometallurgy and electrochemical treatment. The spent nickel-plating solution obtained as an industrial waste was found to contain nickel of 89.4 g/l, which was still in high quantity sufficient for prospective recovery. In this research, precipitation of nickel from the spent electro plating solution was first carried out to separate other unwanted impurities. The remained nickel precipitate was then leached by using 1 M or 2 M H_2SO_4 for 24 h. at a solid/liquid ratio of 100 g/l. The leached solution was then adjusted to obtain pH 2 prior to electrowinning. It was found that the maximum weight of nickel cathode is 5.07 g or at 90.7% recovery of 97.46% purity can be achieved in a condition using 1 M H_2SO_4 and electrical potential 3.7 V.

Keywords: Hydrometallurgy; Electrowinning; Electro nickel plating solution; Industrial waste

Introduction

Currently, wastes generated from the coating industry are increasingly significance due partly to accumulative amount of parts that require coating and the valuable metal contents left in the waste solution. In the nickel plating process, the spent electro plating solution contains nickel as the main constituent, which may be as high as 90 g/l of nickel, potentially for waste management and recycling.

However, with the abundant amount of nickel in the solution, nickel extraction for producing pure nickel is considered to be cost effective and also helps to reduce disposal of waste conformed to regulation. The recovery process of nickel of the waste solution itself requires complex operation. Research by H. Y. Lee recovered nickel from spent electroless nickel-plating solutions using feasible hydrometallurgy and electrochemical metallurgy processes [1]. Precipitation of the spent plating solution prior to electrowinning was suggested to be effective for greater recycling potential.

This research therefore aims to study the feasibility of nickel recovery from spent electroplating solution, that was initially in the forms of nickel sulfate and nickel chloride compounds, by using hydrometallurgy and electrochemical methods. Varying the concentration of the solution and the electrical potential have been investigated in order to study the effects of such parameters on efficiency of nickel recycling in the laboratory scale, with the extension to practical use in the industry being expected in the future.

Experimental Section

Spent electro nickel plating

The spent electro nickel plating solution obtained from the electroplating process for surface coating of cutting tools, is classified as industrial waste. The waste solution still contains high amount of nickel after use for 6 months, which is thus valuable for metal recycling. ICP-OES was therefore selected for elemental analysis to quantify the amount of nickel in the solution, as listed in Table 1. The nickel concentration is 89.4 g/l [2].

Recovery of nickel from the spent solution

The extraction of nickel metal from waste generated from the plating process studied in this research can be divided in two processing routes namely the first (1) and second (2) processing routes. The first processing route consists of several steps of waste pretreatment to reduce impurities and refining, starting from precipitation, leaching and electrowinning whereas the second processing route is the direct electrowinning, as illustrated in Figure 1 [3].

For the first processing route, the recovery of nickel from the spent electro nickel plating solution started from precipitation by using sodium hydroxide (NaOH) at a concentration of 2 M and pH ≈ 13, for 24 hours to give precipitation solution as shown in Figure 2a. After filtration, precipitate was oven dried at 120°C for about 4-6 h. as illustrated in Figure 2b. For chemical analysis by ICP-OES, dried precipitate must be ground and made into a form of solution using microwave digestion.

The leaching process was carried out using ground precipitate and sulfuric acid solution (H_2SO_4) at a varying concentration of 1 M and 2 M, by controlling a solid and liquid ratio at 1:10 (100 g precipitate per 1 liter of acid solution) for a duration of 24 h. The leached solution was again analyzed by ICP-OES to obtain elemental analysis.

Filtration of the leached solution later showed no residual. The electrolyte solution was therefore prepared for electrolysis by adjusting

Elements	Concentration (g/l)
Ni	89.42
P	0.0881
Fe	0.0015
Pb	0.071
Cu	n/a
Al	0.0041
Zn	0.0002

Table 1: Chemical analysis of spent electro nickel plating solution by ICP-OES.

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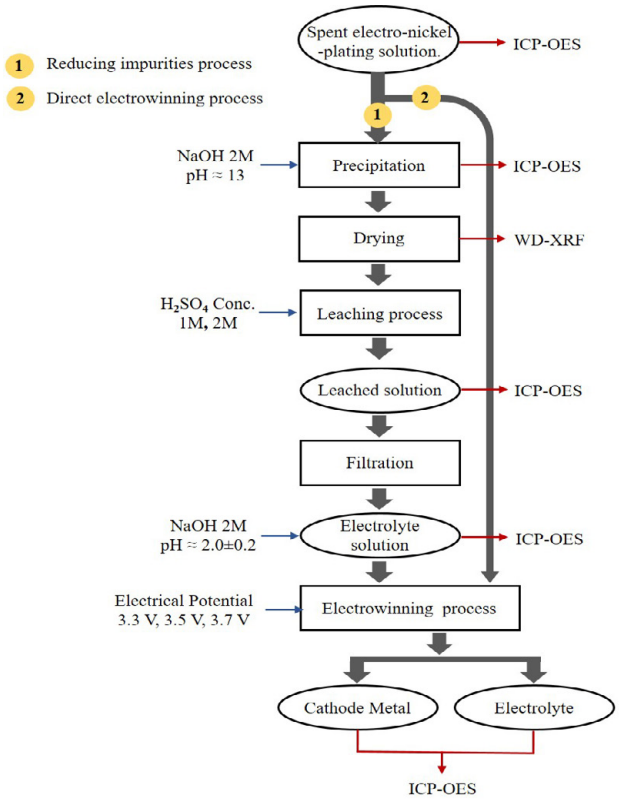


Figure 1: Experimental flow chart of nickel recovery from spent electro plating solution.

a) Precipitation solution b) Dried precipitate

Figure 2: Nickel precipitation process of the spent electro nickel plating solution.

the leached solution to pH ≈ 2 by using NaOH of 2 M concentration and then again analyzed by ICP-OES.

Next, electrolysis cell was set up using the leached solution of pH ≈ 2 as the electrolyte, and SS304 and Pb alloys both having dimensions of 5x5x0.2 cm^3 as cathode and anode respectively, as demonstrated in Figure 3. The electrowinning process was carried out using varying electrical potential of 3.3, 3.5 and 3.7 volt for 24 hours. The desired product received at cathode was nickel metal, which was then analyzed by ICP-OES to obtain the nickel content. The remaining electrolyte was also chemical analyzed by the same technique to determine the nickel left in the solution. The chemical reactions that occur in the electrowinning process are expressed in equations (1) - (3). The weight gain of nickel at cathode was measured in order to give % recovery based on initial amount of nickel in the spent electro nickel plating solution.

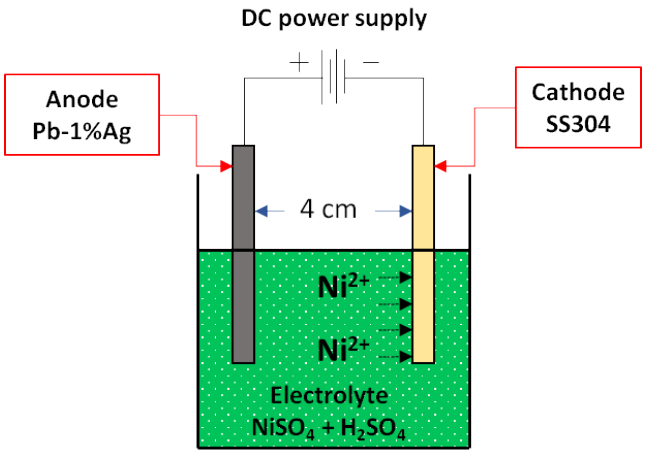


Figure 3: Electrolytic cell for electrowinning of nickel solution.

For the direct electrowinning, the process started from preparing the spent electro nickel plating of 500 ml as an electrolyte for setting up an electrolysis cell, as previously described for the first processing route. Electrowinning parameters, electrical potential and time, were kept similar. The result involving weight gain of nickel, % recovery, % current efficiency and purity are compared with those obtained from the first processing route. The current efficiency can calculate by equations (4) and (5) as follows (Figure 2);

The chemical reactions occurring in the electrowinning process are as follows [4,5];

Cathodic reaction: $Ni^{2+}+2e^- \rightarrow Ni$ (1)

Anodic reaction: $H_2O+2H^+ \rightarrow 1/2O_2+2e^-$ (2)

Overall reaction: $Ni^{2+}+H_2O \rightarrow Ni+2H^++1/2O_2$ (3)

The current efficiency can be calculated following the equations:

$$\text{Current efficiency} = \frac{actual\ mass}{theoretical\ mass} \times 100\% \quad (4)$$

Faraday's laws: Theoretical mass

$$m = \frac{MW * I * t}{n * F} \quad (5)$$

Where

M=theoretical mass (g)

MW=atomic weight (g/mol)

t=time (sec.)

i=electric current (A)

n=number of valence electron

F=Faraday's constant (96500 C)

Results and Discussion

Hydrometallurgy process (Precipitation and Leaching)

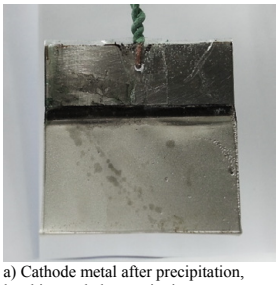
The chemical concentration analyses by ICP-OES of precipitation solution and dried precipitate are listed in Table 2, showing concentration of nickel in the dried precipitate at 559 g/l, in comparison to 0.0207 g/l of that analyzed from precipitation solution. The result indicated separation of nickel by precipitation can be successfully

Elements	Concentration (g/l)	
	Precipitation solution	Dried precipitate
Ni	0.0207	559.18
P	0.0136	n/a
Fe	0.0017	0.0003
Pb	n/a	0.0092
Cu	n/a	n/a
Al	0.0251	0.0189
Zn	n/a	0.0015

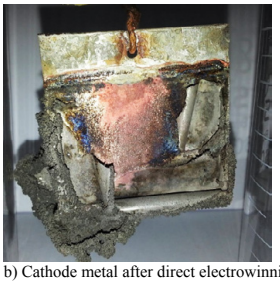
Table 2: Chemical analysis of precipitation solution and Dried precipitate by ICP-OES.

Elements	Concentration (g/l)			
	Leached Sol. 1 M H_2SO_4	Adjusted pH=2.0	Leached Sol. 2 M H_2SO_4	Adjusted pH=2.0
Ni	17.8000	8.5800	16.6700	3.3400
P	n/a	n/a	0.0003	n/a
Fe	0.0008	0.0001	0.0015	n/a
Pb	0.0318	0.0214	0.0263	0.0111
Cu	0.0094	n/a	n/a	n/a
Al	0.0352	0.0298	0.0386	0.0164
Zn	0.0032	0.0015	0.0026	0.0016

Table 3: Chemical concentration of leached solution 1 M, 2 M and adjust pH electrolyte solution by ICP-OES.



a) Cathode metal after precipitation, leaching and electrowinning



b) Cathode metal after direct electrowinning

Figure 4: Cathode metal after electrowinning via a) first processing route and b) second processing route.

carried out by using 2 M NaOH at pH ≈ 13, for 24 h. can then be separated. Moreover, impurities in the solution such as P, Fe, Pb, Cu, Al and Zn can be reduced. Leaching of the solution using both 1 M and 2 M H_2SO_4 for 24 h. offered high leaching efficiency due to no residual left. The results of chemical concentration analyses by ICP-OES after leaching and adjusted pH are shown in Table 3.

Electrochemical process (Electrowinning)

Cathode metal after electrowinning of first and second processing routes are demonstrated in Figure 4a and 4b respectively. The former exhibited nickel deposition of densely smooth and bright surface at the cathode whereas the latter showed nickel deposition with significantly high amount of impurities co-deposited at the cathode. After disposing of these impurities using deionized water, the nickel deposition was measured around 0.86-1.99 g, which is lower than those obtained from the first processing route. The maximum weight of nickel cathode is 5.07 g, which accounted for 90.7% recovery and of 97.46% purity can be achieved in a condition using 1 M H_2SO_4 and electrical potential 3.7 V. Table 4 summarized weight of nickel deposited at cathode, % recovery, % purity of nickel and % current efficiency after electrowinning via the first and second processing routes. Increasing electrical potential from 3.3 to 3.7 V gave comparable result of nickel deposition weight except for in the case of using 2 M H_2SO_4 and 3.3 V electrical potential. This might be due to initially low content of nickel in the solution after adjusting the solution to obtain pH 2. The overall results suggested that by using 1 M H_2SO_4 over electrical potential of 3.3-3.7 V, it offered the optimum values of weight of nickel deposited at cathode, % recovery, % purity and % current efficiency, as illustrated in Figures 5-8. The direct processing route unfortunately gave disappointing result as to confirm that the spent electro nickel plating solution waste required pretreatment via precipitation and leaching prior to electrowinning.

Conclusion

From the experiment of the recycling of spent electro nickel plating solution by hydrometallurgy and electrochemical methods, it can be summarized as follows;

	Ni deposited at cathode (g)	% Recovery	% Purity	% Current efficiency
Direct, 3.3 V	1.93	38.47	95.34	30.57
Direct, 3.5 V	1.99	13.92	93.60	23.67
Direct, 3.7 V	0.86	48.98	96.23	10.66
1 M, 3.3 V	5.07	99.58	95.66	49.66
1 M, 3.5 V	4.67	60.69	97.36	32.57
1 M, 3.7 V	5.07	90.66	97.46	29.88
2 M, 3.3 V	1.11	91.53	93.99	11.73
2 M, 3.5 V	5.05	91.90	94.42	26.33
2 M, 3.7 V	4.60	93.67	97.24	25.06

Table 4: The weight, %Recovery, %Purity and %Current efficiency of the nickel.

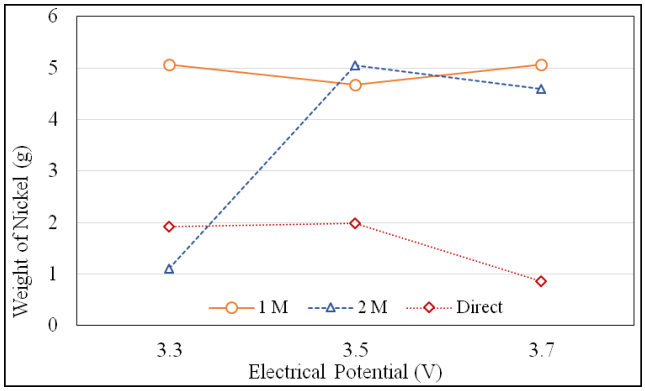


Figure 5: The deposition of the nickel at cathode.

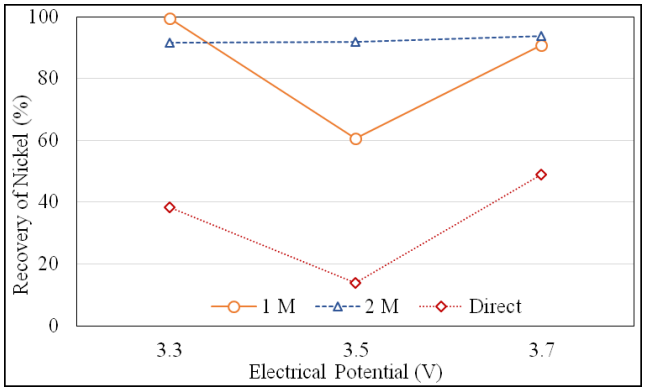


Figure 6: Percentage recovery of the nickel deposited at cathode.

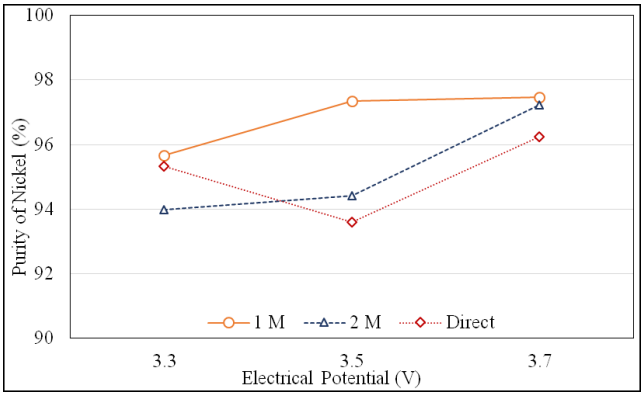


Figure 7: The purity of the nickel deposited at cathode.

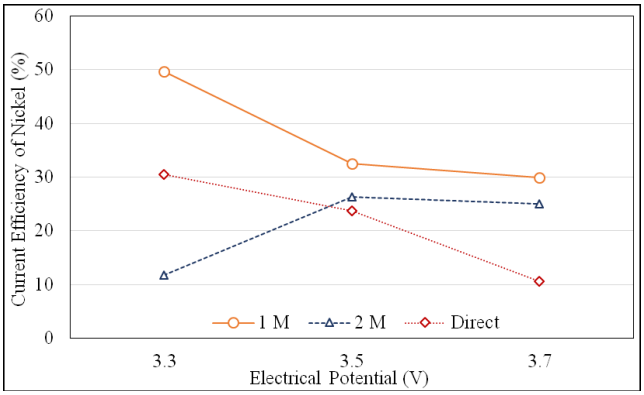


Figure 8: The current efficiency of nickel deposited at the cathode.

1. Precipitation of the spent electro nickel plating solution by using 2 M NaOH can be used to successfully separate nickel into the solution with very few amount of P, Fe, Pb, Cu, Al and Zn as impurities.

2. Leaching by 1 M H_2SO_4 solution is found to be more applicable than 2 M H_2SO_4 solution for electrowinning as the solution over the electrical potential used.

3. The optimum electrowinning condition was observed in a condition using 1 M H_2SO_4 and electrical potential 3.7 V to provide the maximum weight of nickel cathode at 5.07 g or 90.7% recovery of 97.46% purity.

4. Direct electrowinning gave reduced amount of recovered nickel due to high impurities co-deposited at the cathode; thereby, yielding low %recovery and %efficiency, and that to confirm the spent electro nickel plating solution should require pretreatment.

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