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Leaching of Heavy Metals from Electrode Materials of Spent Ni-Cd Batteries in Different Acidic Solutions

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Abstract:
The Nickel-Cadmium (Ni-Cd) batteries are attracted much attention in many applications such as railway engines, mines, armored vehicles, and aircraft engines. This requires more production of these valuable metals from their conventional resources. Meanwhile, the spent types of these batteries are considered to be as a source of contamination of the environment due to the presence of Cd in the electrode material. On the other hand, the other metals such as Ni in these types of batteries has a considerable economic value. To achieve effective utility of resources and environmental conservation, it is necessary to apply effective and inexpensive methods to recycle these batteries. The current research focused on recovering Cd and Ni from spent Ni-Cd batteries. One of the attractive method for that purpose is the hydrometallurgical method. The leaching behavior of nickel and cadmium metals and their compounds of interest was evaluated under different conditions such as acid solution types (sulfuric, nitric, and hydrochloric acids), acid concentration (1,2,3,4, and 5 M), leaching temperature (25,35,45,55, and 65 ˚C), and leaching time (60,90,120,150, and 180 min). The sulfuric acid showed better Ni leaching rate than other acids, while the Cd was efficiently leached by nitric acid compared to sulfuric and hydrochloric acids. The results showed that the best recovery rate for Ni and Cd were reported to be 85.46% and 79.64% in 5 M sulfuric and nitric acids for 2 hours and 65 oC, respectively.

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I. INTRODUCTION

Nickel-cadmium battery is specifically suitable for various applications in cellular and cordless telephones, video cameras, portable power tools, and laptop computers due to its ability to deliver high currents. After several cycle of uses, these batteries can no longer hold charge and become spent. Increase in the consumption of batteries for household appliances, implementation of strict environmental regulations and promoting the treatment of Ni-Cd household batteries have turned this kind of battery waste into an increasingly representative material to be recycled. A major concern with Ni-Cd batteries is the environmental pollution problem due to the presence of toxic metals such as cadmium at the same time spent batteries contain high value metals such as nickel and cobalt. This has led to an increased interest in the recycling of used household batteries such as alkaline, zinc-carbon, mercuric oxide [1] , lead and nickel–cadmium Ni-Cd batteries which will not only generate revenue but also prevent environmental pollution [2] . There are several recycling processes of Ni-Cd batteries which are pyrometallurgy process, hydro-metallurgical process, and thermal separation process. The process of recycling

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should be relatively clean and secure. High percentages of the valuable components in the battery must be restored. The method should be as simple as possible, low cost, and the waste resulting from the recycling process is minimum and has no negative impact on the environment and easy handling [3 , 4].

After checking the available literature [5 -23], it can be concluded that the recycling of Ni-Cd batteries using hydro-metallurgical process is preferred since it is considered to be more economical, eco-friendly and efficient approach than both pyro-metallurgical and thermal separation processes. Metal separation routes based on hydrometallurgical operations are characterized by lower energy consumption, higher metal selectivity, and no air pollution. The hydrometallurgical process consists of two steps which are leaching followed by electrochemical process [5,6,10,17,18,25], or solvent extraction [8,9,11,12,14,15,16,20,23,24] or chemical process [21]. The leaching of Ni and Cd is considered to be a very important, simple, and inexpensive step that can be used to recover Ni and Cd in a large quantities and several studies [7,13,19] were carried out in order to find the best conditions which give a higher leaching efficiency.

In (2006), Carlos A. Nogueira and Fernanda Margarido [7] recycled spent Ni-Cd batteries by integrated physical-chemical processes such as shredding, sieving, washing, acid leaching, and solvent extraction to recover Ni, Co, and Cd. The acid leaching experiments of the electrode materials were performed in H_2SO_4 and the leaching parameters such as temperature, acid concentration, and liquid/solid ratio (L/S) were optimized. It was concluded that Co and Cd are easily leached under a wide range of conditions compared to Ni. The results revealed that metals can be efficiently recovered (~95%) with minimum consumption of reagents at temperature of 100 oC, acid concentration range of 2.3-2.7 M, and L/S ration of 8 to 10 L/kg. In another study, Jerzy GĘGA et. al. [13] investigated experimentally the first step of the hydrometallurgical route, which is the acid leaching, to recover the Cd and Ni metals from the spent Ni-Cd batteries. The electrode materials were dissolved in sulfuric acids and the effect of acid concentration on the leaching process was systematically evaluated. The results showed that the acid concentration of 2 M and the liquid/solid ratio of 1:20 at room temperature are the condition for leaching process. More recently, nickel and cadmium extraction in Ni-Cd powder sulfuric acid filtration has been enhanced at comparatively low temperatures . The depleted Ni-Cd battery powder comprising approximately 69% Ni, 15% Cd and 0.94% Fe was sprayed with various parameters in sulfuric acid, i.e. acid concentration, moment, temperature and hydrogen peroxide addition , the solubility rate was determined by the chemical reaction of the surface . The novelty of this research was the elevated recovery of nickel (96 percent) and cadmium (99.5 percent) by filtering Ni-Cd batteries produced with dilute sulfuric acid (10vol. percent) at a mild temperature (328 kg), suggesting the potential for enhanced entry routes. The filtration liquid acquired in this research contained roughly 6-7 g / dm3 Cd2 +, 26-28g / dm3 Ni2 + and up to 0.34g / dm3 Fe3 + and could be exposed to a suitable purification and separation method for metal / pure nickel compounds recovery

It should be noted that the above studies carried out the leaching process on the battery materials that mixed together to prepare the samples (anode and cathode electrode materials), while A. M. Saeed et. al. [21] investigated the recovery of cadmium and nickel from spent alkaline batteries by leaching the electrode materials separately in a way that nitric acid was determined to effectively leach Cd from the anode electrode materials and the Ni was effectively leached by sulfuric acid from the cathode electrode materials. This requires more in depth investigation to perform a

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systematic study to determine the best experimental conditions such as the selection of the best acidic solutions to leach the Ni from the cathode and Cd from the anode. Hence, the aim of this work is to carry out a systematic leaching study on the anode and cathode electrode materials separately and find the best leaching conditions.

II. EXPERIMENTAL WORK

The practical part presented the experimental steps and the required chemicals and instruments for Nickel and Cadmium recovery from the spent Ni-Cd batteries . The experimental steps include dismantling of batteries, powder preparation of each electrode, preparation of chemical solutions, and leaching process of Ni and Cd at different conditions. For material characterization, the X-ray diffraction was utilized to determine the chemical composition of the electrode material powder and the concentration of metal ions in solutions were specified by atomic absorption spectroscopy (AAS).

2.1 Preparation of Sample

The nickel-cadmium battery were provided from the battery factory in Baghdad which belongs to the state company for automotive Industry at Al-Eskandria. The work steps were carried out as follows:

- Disassembling, cutting, and removing the plastic cover of the battery.
- Cadmium electrode (negative electrode) plates were separated from the positive cathode plates that contains Nickel . The plastic clamps between the cells were removed and washed to remove the residue of the electrolyte solution in the battery as shown in Fig (1) .
- The iron frame was removed and cut into slices with the removal of the graphite powder inside each cell, then the cells were washed .
- The electrode materials were grounded to prepare powders from each electrode using custom-made grinding machine as shown in Fig. (2).
- The powder for each electrode was analyzed using X-Ray-Diffraction to determine the chemical composition for each electrode shown in Fig (3 and 4). Table (1) contains the chemical composition for anode and cathode powders.

Figure 1: Disassemble the nickel-cadmium battery

Figure 2: Grinding machine (to convert solid to powder)

Table 1: Chemical composition of spent Ni-Cd batteries electrodes

Element	Chemical Composition [%]									
	O	Na	Mg	Si	K	Mn	Fe	Ni	Cd	In

Cathode Sample (Ni)	15.74	0.31	0.08	0.18	8.81	0.15	34.37	40.36	___	__
Anode Sample (Cd)	19.96	0.30	___	0.13	3.60	0.11	26.29	7	42.28	0.33

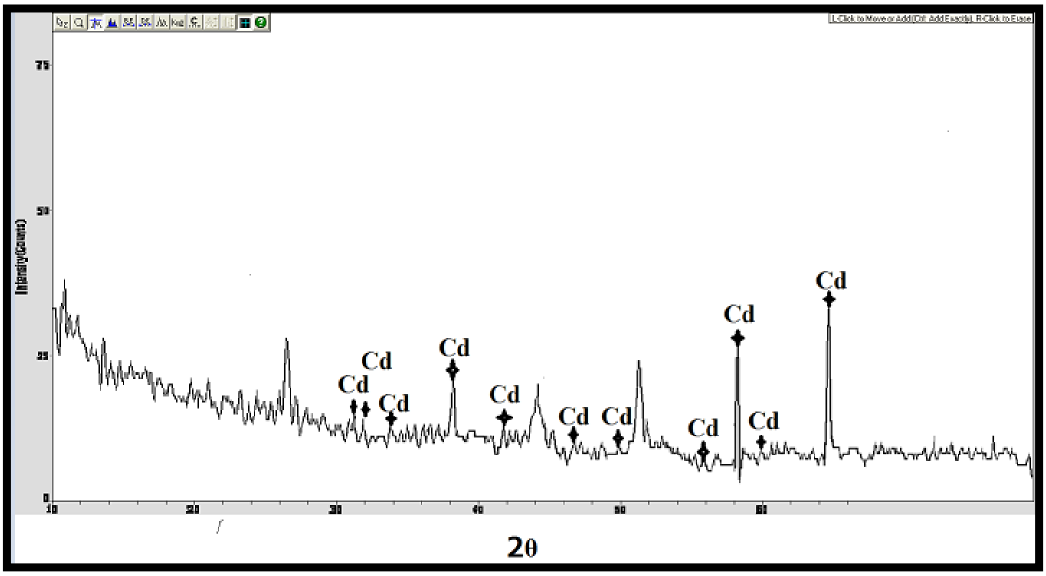

Figure 3: Result of X-ray diffraction analysis for Cadmium powder

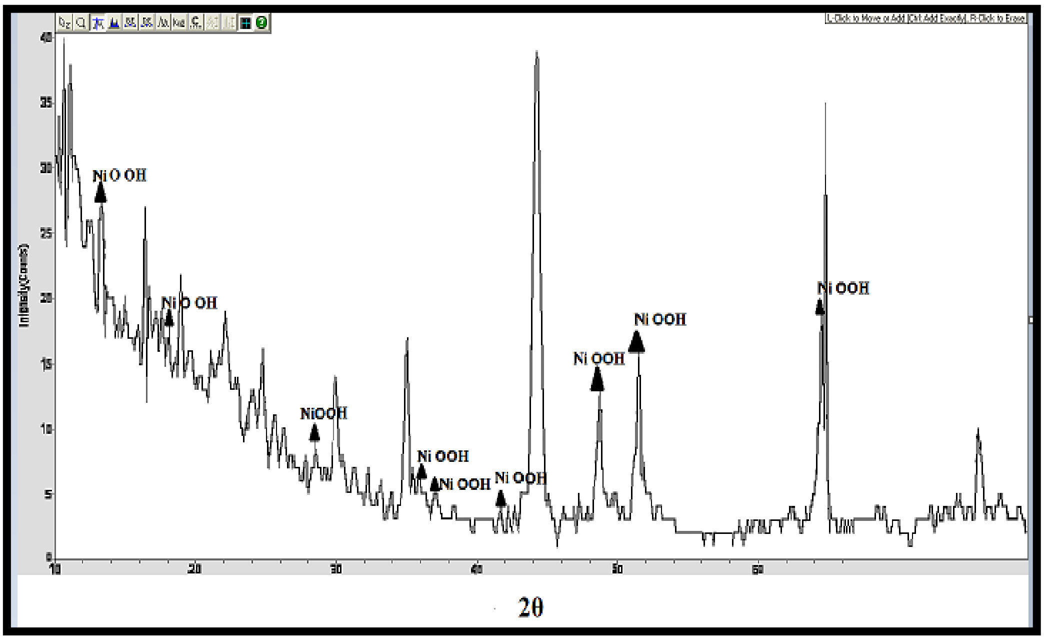

Figure 4.2: Result of X-ray diffraction analysis for nickel powder

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2.2 Leaching of Nickel & Cadmium in Different Acidic Solutions

This experiment was carried out based on the solid to liquid (S/L) ratio of (1/20) by taking (3g) from nickel and cadmium powders and dissolving them in a 100 ml beaker using 2M of different solutions (HNO3, HCl, and H2SO4).The mixture was placed on the vibrating device for a period of (2 h) and at laboratory temperature (25˚C). The recovery solution was filtered by a filtration paper (F2042) and was tested by the atomic absorption spectroscopy (AAS) device for measuring the amount of recovered nickel and cadmium ions and selecting the best acidic solution for Ni & Cd recovery. It should be noted that the fraction of Ni and Cd powders leached in the solution to initial weight taken was used to determine the leaching efficiency.

2.2.1 Effect of Temperature on Ni and Cd Leaching Process

The effect of temperature on the leaching of Ni and Cd was evaluated by taking (5) g of materials powder and dissolving it in a beaker vial of 100 ml based on the ratio of 1/20 (S/L) using 2M H2SO4 solution for Ni and HNO3 for Cd. The leach solution for each electrode powder was placed on the vibrating device for a period of 2 h at different temperatures (25,35,45,55, and 65 ˚C). The recovery solution was filtered by the filter paper (F2042) and was tested by AAS for the purpose of measuring the amount of recovered Ni and Cd and thus, selecting the best recovery temperature.

2.2.2 Effect of Acid Concentration on Ni and Cd Leaching Process

This experiment was carried out by taking (5 g) of Ni and Cd powders and dissolving them in a beaker vial of (100 ml) based on the ratio of 1/20 (S/ L) using H2SO4 for Ni and HNO3 for Cd of different concentration (1,2,3,4, and 5 M). The solution was vibrated for (2 h) at a temperature of (65˚C). The recovery solution was filtered by the filter paper (F2042) and was tested by AAS for the purpose of measuring the amount of recovered Ni and Cd ions and thus, selecting the best recovery concentration of H2SO4 and HNO3 solution for Ni and Cd respectively.

2.2.3 Effect of Time on Ni and Cd Leaching Process

This experiment was carried out by taking (5 g) of nickel & cadmium powders and dissolving them in beaker vial of 100 ml based on the ratio of 1/20 (S/L) using 5M H2SO4 and HNO3 solutions for Ni and Cd, respectively. The vibrator was used to mix the sample at a temperature of (65˚C) for different time (60,90,120,150, and 180 min). After acid leaching, the residue was removed by filtration using the filter paper (F2042). The chemical composition of Ni and Cd in the leach liquor was quantitatively analyzed by AAS.

III. RESULTS AND DISCUSSION

The leaching of Ni and Cd were separately performed using different solution such as sulfuric, nitric, and hydrochloric acids. The concentration of metal ions in leach liquors was determined by AAS test . The results are included in Table (2) , which shows that the amount of nickel recovered has the highest value in sulfuric acid and the lowest value in hydrochloric acid. The recovery ratio increases with the acidity of the solution, because nickel is deposited as nickel hydroxide in acidic analysis due to high dissolution [24], while the amount of cadmium recovered has the highest value in nitric acid and the lowest value in hydrochloric acid .The recovery ratio increases with the acidity of the solution because cadmium is deposited as cadmium hydroxide in acidic analysis due to high dissolution. Figures (3) and (4) show the leaching efficiency of Cd and Ni in various acidic solutions. The metals (M is Ni or Cd) react with the used acids based on the following reactions [21]:

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$$M(OH)_{2(S)} + 2HCL \longrightarrow MCL_{2\,(L)} + 2H_2O$$

$$M(OH)_{2(S)} + 2HNO_3 \longrightarrow M(NO_3)_{2(L)} + 2H_2O$$

$$M(OH)_{2(S} + H_2SO_4 \longrightarrow MSO_{4(L)} + 2H_2O$$

Table 2: Leaching of Cd and Ni in different acidic solutions

Type of solution	The amount of Cd & Ni in sample (g)	The recovered amount of cadmium (g)	The remained amount of cadmium (g)	The recovered amount of nickel (g)	The remained amount of nickel (g)
HCl	3	1.932	1.068	0.812	2.188
H_2SO_4	3	1.547	1.453	1.489	1.511
HNO_3	3	2.0687	0.9313	0.932	2.068

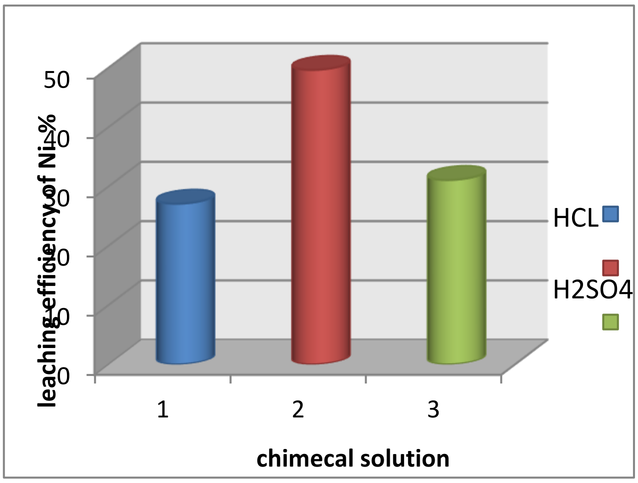

Figure 3: The leaching efficiency of Ni in various acidic solutions

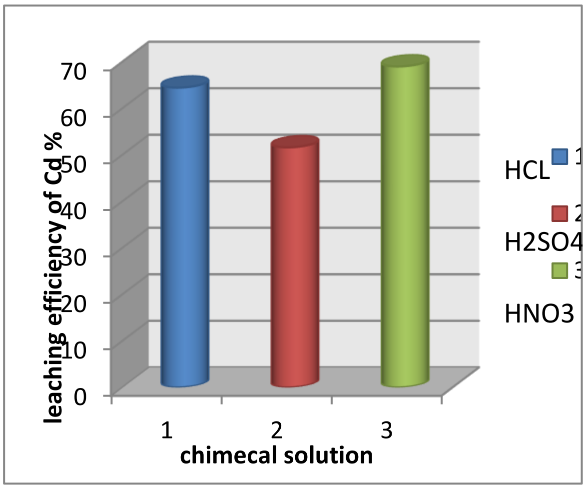

Figure 4: The leaching efficiency of Cd in various acidic solutions

The results in Table(3) revealed the effect of the temperature change on leaching of cadmium and nickel from electrode material powder. It was found that the amount of cadmium and nickel increases with increasing temperature, the highest leaching rate at 65˚ and the lowest ratio at laboratory temperature 25˚C as shown in Figures ((5) and(6)). A temperature of 65˚C was adopted as the best leaching condition. A significant increase in temperature results in the loss of part of the recovery solution as a result of evaporation.

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Table 3: The effect of the temperature on nickel and cadmium leaching

Temperature (C)	The amount of Cd & Ni in the sample (g)	The recovered amount of cadmuim (g)	The recovered amount of nickel (g)	The remained amount of nickel (g)	The remained amount of cadmium (g)
25	5	2.0687	2.5532	2.4468	2.9313
35	5	2.0537	2.6636	2.3364	2.9463
45	5	2.0579	2.8549	2.1251	2.9421
55	5	2.4737	2.9661	2.0339	2.526
65	5	3.0343	3.266	1.734	1.9657

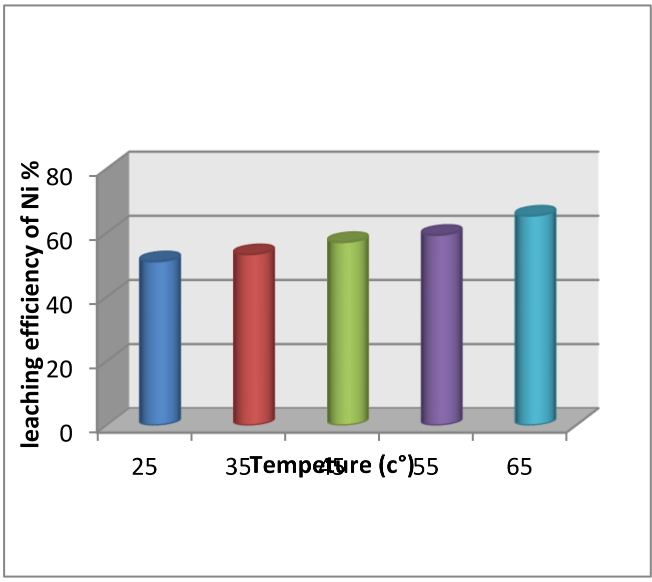

Figure 5: Effect of temperature on nickel leaching

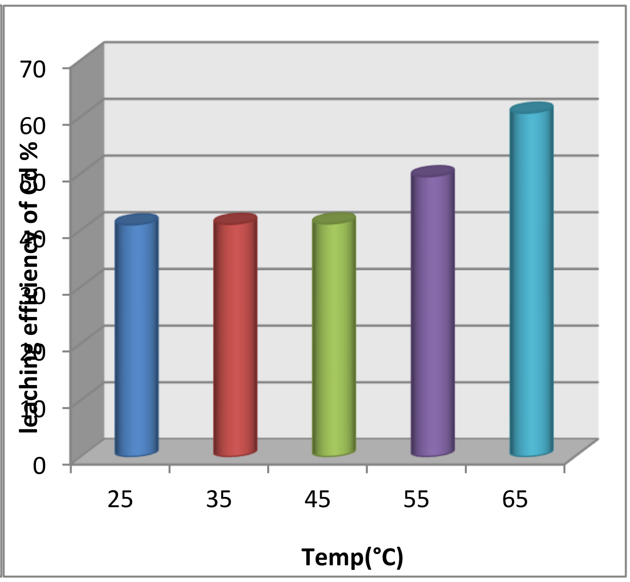

Figure 6: Effect of temperature on cadmium leaching

The highest nickel recovery value was found at a concentration of (5M) and the lowest value at (1M), therefore the concentration (5M) was adopted as the best concentration under the optimal conditions for nickel recovery as shown in Fig (7). It was found that increasing the acid concentration leads to an increase in sulfate ion concentration in the solution with the formation of sodium sulfate that is highly soluble in water [25]. While the highest value of cadmium recovery at concentration of (5M) and the lowest value at (1M), therefore the concentration (5M) was adopted as the best concentration under the optimal conditions for cadmium recovery as shown in Fig (8). Table (4) shows the results.

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Table (4) The effect of sulfuric and nitric acid concentrations on nickel and cadmium leaching process

concentration of sulfuric acid	The amount of cadmium &nickel in the sample (g)	The recovered amount of nickel (g)	concentration of nitric acid	The recovered amount of cadmium (g)	The remained amount of cadmium(g)	The remained amount of nickel (g)
1	5	2.803	1	2.0651	2.9349	2.197
2	5	3.489	2	2.0687	2.9313	1.511
3	5	3.8549	3	3.532	1.468	1.1451
4	5	3.532	4	3.7761	1.2239	1.468
5	5	4.273	5	3.9823	1.0177	0.727

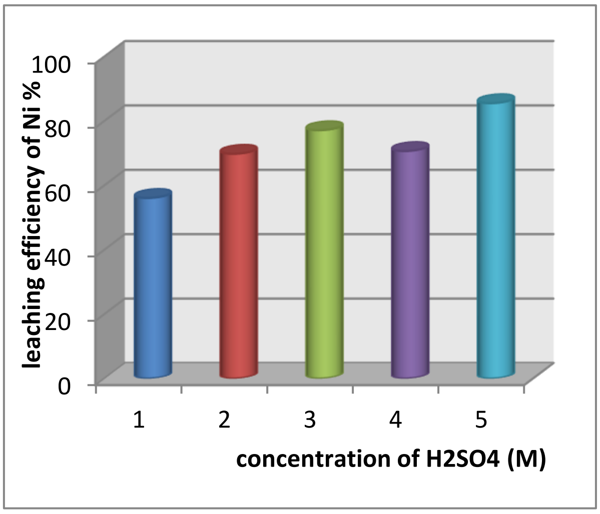

Figure 7: Effect concentration sulfuric acid on nickel leaching

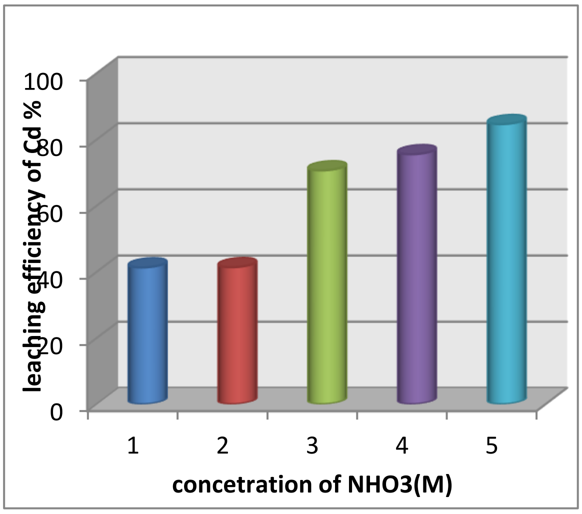

Figure 8: Effect concentration nitric acid on cadmium leaching

Table (5) shows the results where the highest value of nickel and cadmium recovery at time (180) min and the lowest value at time (60) min , therefore the time (180) min was adopted as the best time under the optimal conditions in nickel and cadmium recovery. Figure (9) shows the effect leaching time on nickel and cadmium recovery.

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Table 5: Effect of leaching time on nickel & cadmium recovery

Time (min)	The amount of cadmium & nickel in the model (g)	The recovered amount of cadmium (g)	The recovered amount of nickel (g)	The remained amount of nickel (g)	The remained amount of cadmium (g)
60	5	2.0773	3.832	1.168	2.9227
90	5	3.0661	3.843	1.157	1.9339
180	5	3.9823	4.273	0.727	1.0177
220	5	3.7078	3.783	1.217	1.2922
280	5	3.6859	3.755	1.245	1.3141

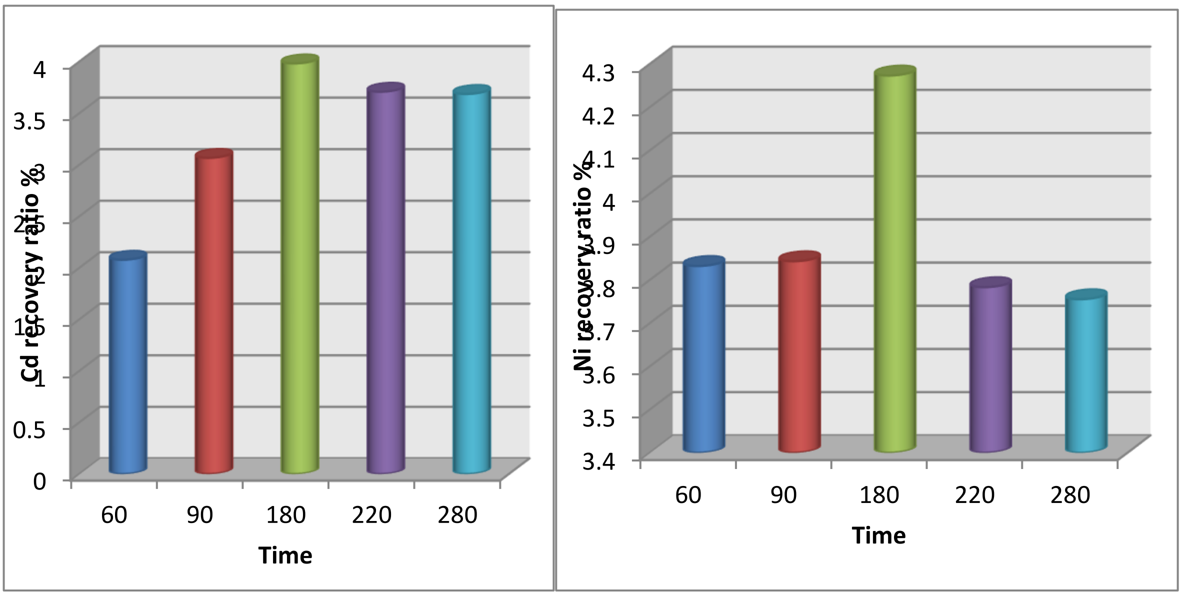

Figure 9: Effect of leaching time on nickel & cadmium recovery

IV. CONCLUSIONS

The following conclusions were drawn after conducting practical experiments and discussing the results, which are summarized as follows: :

1. Among various types of acids, the best acid for the leaching of nickel is sulfuric acid, while nitric acid exhibits better leaching efficiency for cadmium ,

2. The concentration of acids has positive effect on leaching of Ni and Cd; The 5M of sulfuric and nitric acids solutions show high leaching rate for Ni and Cd, respectively.

3. The leaching rate of cadmium and nickel increases as the leaching time increases and the highest rate was reported for 120 minutes.

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4. Like the acid concentration and time parameters, the temperature of leaching process enhances the leaching rate for both Ni and Cd. The best leaching temperature was determined to be 65 oC.

5. These observations are encouraging to continue our work investigating a simple, inexpensive, suitable and economical second step of the hydrometallurgical approach on the leach liquors obtained from anode and cathode electrodes.

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