Determination of recovered Cadmium and Nickel from spent alkaline batteries using acidic solutions and AAS measurements

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Abstract

This research include the recovery study of cadmium and nickel from spent alkaline batteries was conducted by two different acidic solutions. The first one is nitric acid, which was used for cadmium recovery; the other is sulfuric acid, which was used for nickel recovery. Many factors affected the recovery process were studied, such as concentration, volume, time, weight and temperature. The parameters which effect on the precipitation of metal ion from leaching solution were also studied, such as pH, time, concentration and temperature. The recovery percentages of metal ion were found to be 98.5% of cadmium using optimum conditions (5 M HNO_3, 2 gm sample, 2 hr, and 20 ml acid volume) and 99% for nickel using (5 M H_2SO_4, 2 gm sample, 2 hr, and 20 ml acid volume). The precipitation processes were carried out using (2 M NaOH) solution. The precipitation percentages were found to be 98.6% and 99.1% of cadmium and nickel respectively. The purification processes were conducted using double distilled water. The purity of products Cd $(OH)_2$ and Ni $(OH)_2$ was found to be 98.08% and 98.23% respectively. The concentration of metal ion in the solutions was measured using AAS; the method was linear with R^2 of (0.9995 and 0.9993) for Ni and Cd respectively.

Key words: Determination, Alkaline batteries, AAS, Cadmium, Nickel.

تقدير الكادميوم والنيكل المستردة من البطاريات القاعدية المستهلكة باستخدام محاليل حامضية وقياسات الامتصاص الذري

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الخلاصة

هذا البحث يتضمن دراسة عملية استرداد الكادميوم والنيكل من البطاريات القاعدية المستهلكة باستخدام نوعين من المحاليل الحامضية المختلفة. النوع الاول هو حامض النتريك والذي تم استخدامه لعملية استرداد الكادميوم؛ اما النوع الثاني فهو حامض الكبريتيك والذي تم استخدامه لاسترداد النيكل. لقد تم دراسة عدد من العوامل التي تؤثر على عملية الاسترداد مثل تركيز الحامض؛ الحجم؛ الزمن؛ الوزن و درجة الحرارة.كذلك تم دراسة العوامل التي تؤثر على عملية ترسيب العناصر من محلول الاسترداد مثل الدالة الحامضية؛ زمن الترسيب؛ التركيز و درجة الحرارة. لقد كانت نسب الاسترداد للعناصر بحدود 98.5% للكادميوم باستخدام الظروف المثلى (تركيز حامض النتريك 5 مولاري؛ وزن النموذج 2 غرام؛ زمن الاسترداد 2 ساعة وحجم الحامض 20 مللتر)؛ كما ان نسبة الاسترداد للنيكل كانت 99% عند استخدام الظروف المثلى (تركيز حامض الكبريتيك 5 مولاري؛ وزن النموذج 2 غرام؛ زمن الاسترداد 2 ساعة وحجم الحامض 20 مللتر). لقد تم اجراء عملية الترسيب باستخدام هيدروكسيد الصوديوم بتركيز 2 مولاري. لقد كانت نسب الترسيب للعناصر هي 98.6% و 99.1% للكادميوم والنيكل على التعاقب. تم اجراء عملية التنقية باستخدام الماء المقطر. كانت نسبة النقاوة للنواتج هي 98.08% و 98.23% لكل من هيدروكسيد الكادميوم وهيدروكسيد النيكل على التعاقب. تم قياس تراكيز العناصر باستخدام تقنية الامتصاص الذري وكانت قيمة R^2 للطريقة هي (0.9993 و 0.9995) للكادميوم والنيكل على التعاقب.

الكلمات المفتاحية : تقدير، البطاريات القاعدية، الامتصاص الذري، الكادميوم ، النيكل.

Introduction

Cadmium – Nickel power supplies account for about 10% of all chemical current sources in use [1]. Various conventional methods for the disposal of the spent batteries which were used in the recent past involve methods such as disposition, stabilization and recycling processes. [2]. Recycling that constitutes the most generally acceptable environmentally friendly method of managing these wastes must be taken seriously, to minimize environmental toxicity, for economic gains and a reduction in dependence on foreign resources or on virgin materials for productions in the industry as well as for the sustainability of the natural resources [3]. Any recycling technology that is developed should therefore be designed so as to fully recover these metals in the finished product, thus preventing the formation of gaseous emissions and keeping these metals out of waste water [4]. Hydrometallurgical routes are commonly found more economical and efficient than pyrometallurgical ones. Hydrometallurgy has the advantage of working at low temperature for use of aqueous solutions generates little waste gas. The hydrometallurgical technologies that can be used for recycling mainly entail converting metals to a solution by basic or acid leaching. The metals can be recovered from the solution by precipitation, electrolysis, or liquid extraction [5 - 9]. In recent years, numerous studies on the treatment, recycling and determination of metals in batteries by hydrometallurgical processes and AAS mesurements have been performed [10 - 22]. In the present study a hydrometallurgical method for cadmium and nickel recovery from spending batteries was developed using an acidic medium for leaching process and basic medium for precipitation of recovered metals from leaching solutions as metal hydroxide. The obtained precipitates were purified using an aqueous solution.

Experimental

1 – Apparatus:

Sartorius balance (Germany), pH meter (Germany), Stirring Hotplate (Korea), Sonic bath (Korea)، Shaking water bath (Taiwan), Furnace (Germany) and Atomic absorption (AURORA – Canada) were used through this study.

2 – Materials and reagents:

Ni-Cd batteries were supplied from Iraqi electrical power stations. All other chemicals used in the present study were of analytical grade reagents unless otherwise stated. Sulfuric acid (BDH), Hydrochloric acid (BDH), Nitric acid (BDH), Sodium hydroxide (GCC), Potassium hydroxide (GCC), Sodium carbonate (GCC), Sodium bicarbonate (GCC) and Ammonium hydroxide (BDH).

3 – Stock solutions (6M, 2M, 20%) of acid and base:

These solutions were prepared in volumetric flask of 1L volume for each solution by the gradual dilution of concentrated solutions of acid and base by dilution law. These solutions are (6 M sulfuric acid, 2M nitric acid, 2M hydrochloric acid, 2M sodium hydroxide, 2M potassium hydroxide, 2M sodium bicarbonate, 2M sodium carbonate and 20% ammonium hydroxide). The more dilution solutions were preparation by dilution law.

4 – Recovery of electrode materials:

The components of spent alkaline batteries were separated manually. An anode (negative electrode) which contains cadmium was leaching with nitric acid, while the cathode (positive electrode) which contains nickel (Table 1, illustrate the chemical analysis of electrode powder) was leaching using sulfuric acid. The leach solutions which contain (cadmium and iron) or (nickel and iron) were filtered and diluted. After dilution the pH of the solutions was adjusting up to 3 to remove iron from mother liquor solutions. The leach solutions which are iron free and contain cadmium or nickel were further used for the precipitation of Cd and Ni as hydroxide at pH 10.5 and 11 respectively, using 2 M sodium hydroxide solution. The hydroxides of metals were purified with double distilled water to remove sodium nitrate and sodium sulfate from precipitates.

Table. 1: Chemical analysis of electrode powder.

Percentage of metals / Type of sample	Ni %	Cd %	Fe %
Nickel sample	43.00		0.50
Cadmium sample		50.00	1.50

5 – Calibration graph and linearity study:

For determining the linearity, a series of solutions have a different metal ions concentration range of (1, 2, 3, 4, 5, 6, 8, 10, 12 and 15 ppm) were prepared by simple dilution of stock solutions. The absorbance of these solutions was measured at (228.8 and 236.0) nm for Cd and Ni respectively. The calibration curves were obtained by plotting absorbance versus known concentrations in ppm. Figure 1, illustrate the calibration graph Ni and Cd by Atomic absorption spectroscopy (AAS). The method were linear with an R^2 of (0.9995 and 0.9993) for Ni (II) and Cd (II) respectively. Linearity was determined by the regression analysis. The obtained results were tabulated in Table 2, shows that the value of t_{cal} is larger than t_{tab} value, and R^2 values are(0.9995 and 0.9993), which indicating that there is a strong correlation between the variation of concentration and response.

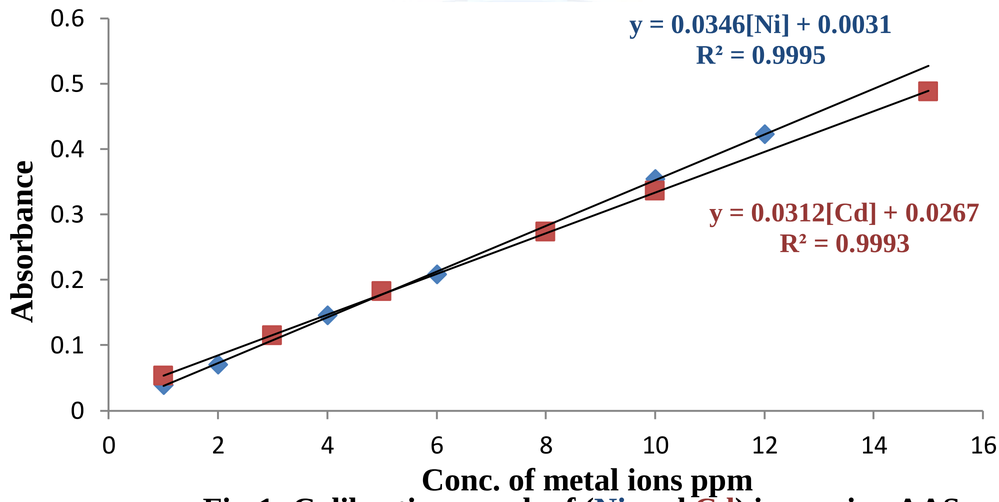

Fig:1: Calibration graph of (Ni and Cd) ions using AAS

Table.2: Summary of linear regression for the variation of absorbance with metal ions concentration using first degree equation of known form y = b[X] + a.

Type of metal ion	**Linear range ppm**	**Straight line equation Abs. = b[X] + a**	**Correlation coefficient (r)**	**Percentage linearity (r^2%)**	**Calculated (t) values** $t_{cal.} = \frac{/r/\sqrt{n-2}}{\sqrt{1-r^2}}$
Ni (II) ion	1 - 12	y = 0.0346[Ni] + 0.0031	0.9997	99.95	89.26 ≫ 2.78
Cd (II) ion	1 - 15	y = 0.0312[Cd] + 0.0267	0.9996	99.93	79.89 ≫ 2.78

Results and Discussions

1 – Recovery studies:

1.1 – Study of the leaching solution type:

In this set of experiments the investigation of the leaching solution type was studied. Different types of solutions were used to carry out this study. The solutions have a concentration of 2 M (H_2SO_4، HNO_3، HCl ،NaOH، $NaHCO_3$, KOH، Na_2CO_3) was used for recovery of metal ion as shown in Table 3 and Figure 2. The results obtained revealed that the highest recovery values were occurring with the acidic solution, while the lowest values occurred with the basic solution. The highest recovery percentage of cadmium was found to be 61.2% of nitric acid solution, while the highest recovery percentage of nickel was 92.56% of sulfuric acid solution.

The recovery percentage was calculated as follows:

$$\% \text{ recovery} = \frac{\textit{metal in solution (mg)}}{\textit{metal in sample (mg)}} \; x\, 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Table. 3: Recovery percentage of Nickel and Cadmium for several type of solution**.**

Type of solution	% Ni recovery	% Cd recovery
H_2SO_4	92.56	52.00
HNO_3	85.58	61.20
HCl	84.69	61.00
NaOH	0.120	3.64
KOH	0.016	3.16
$NaHCO_3$	0.002	2.80
Na_2CO_3	0.001	0.24

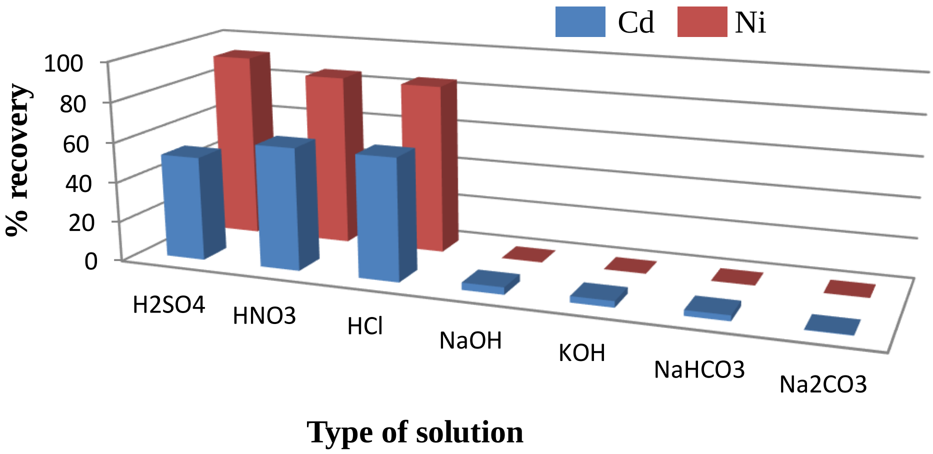

Fig. 2: relationship between type of solution Vs. recovery

1.2 – Effect of leaching solution concentration:

In this study the effect of solution concentration on recovery of Cd and Ni was investigated. Different solutions of nitric acid and sulfuric acid have a concentration of (1, 2, 3, 4, 5, 6) M were used for the recovery of Cd and Ni respectively (table 4 and figure 3). The obtained results indicate that the recovery percentages were increased as the concentration of solutions of acid was increased until reaching 5 M, after that the value of increasing being small or near constant value.

Table. 4: the obtained results from solution concentration study.

Acid conc. mol. / l	% Ni recovery	% Cd recovery
1	18.90	5.50
2	50.00	10.00
3	68.30	26.50
4	80.10	53.50
5	89.10	78.00
6	89.20	78.30

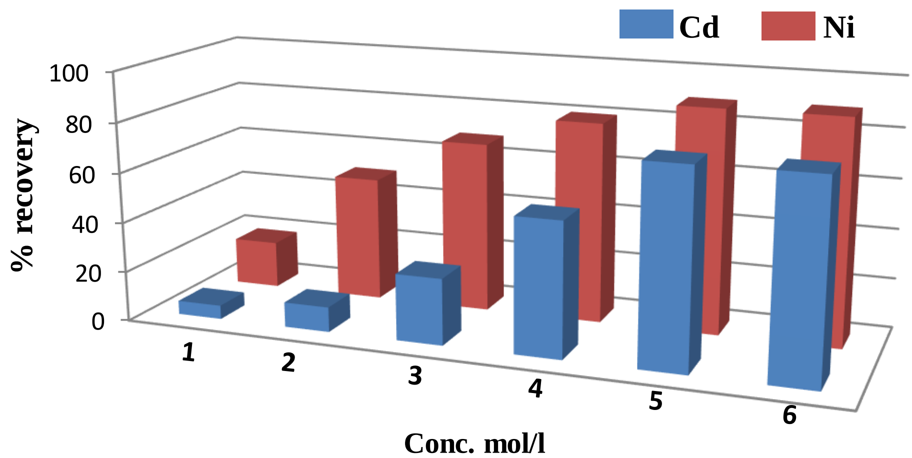

Fig: 3: relationship between conc. of acid Vs. recovery

1.3 – Effect of solution volume:

This study was carried out using different volumes of acid (5, 10, 15, 20, 25 ml) which have a concentration of 5M. The results obtained are tabulated in table 5, which revealed that the recovery percentages were increased as the volume of solutions of acid was increased until reaching 20 ml, after that the value of increasing being small or near constant value as shown in figure 4.

Table. 5: the obtained results from solution volume study.

Acid volume. ml.	% Ni recovery	% Cd recovery
5	18.75	13.00
10	64.76	34.00
15	79.66	67.60
20	89.18	90.20
25	89.30	90.00

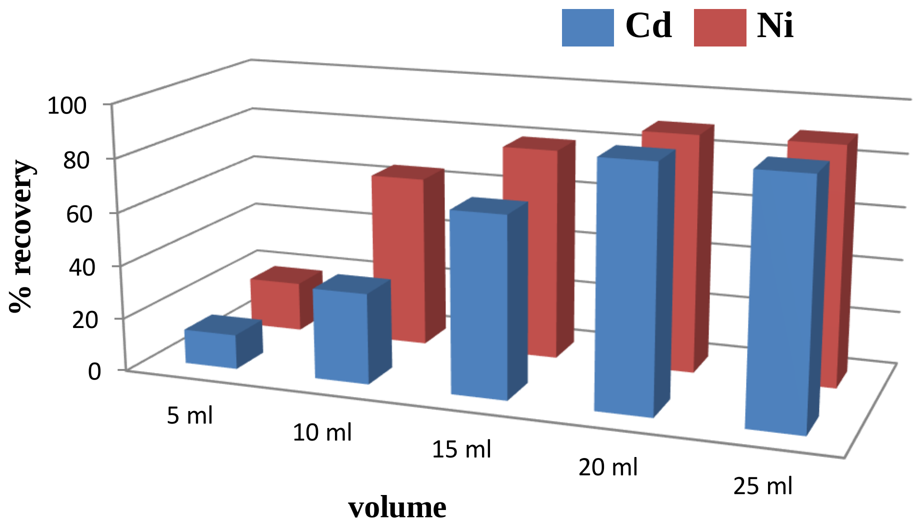

Fig: 4: relationship between acid volume Vs. recovery

1.4 – Effect of leaching time:

In this study the effect of leaching time on recovery of Cd and Ni was investigated. The solutions of nitric acid and sulfuric acid have a concentration of 5 M were used for the recovery of Cd and Ni respectively, at different time (table 6 and figure 5). The obtained results indicate that the recovery percentages were increased at the time of leaching was increased until reaching 120 min., after that the value of increasing being small.

Table. 6: the obtained results from leaching time study.

Leaching time min.	% Ni recovery	% Cd recovery
15	25.53	28.00
30	66.72	46.50
60	89.18	77.50
90	92.32	83.00
120	95.58	90.50
180	95.69	90.30

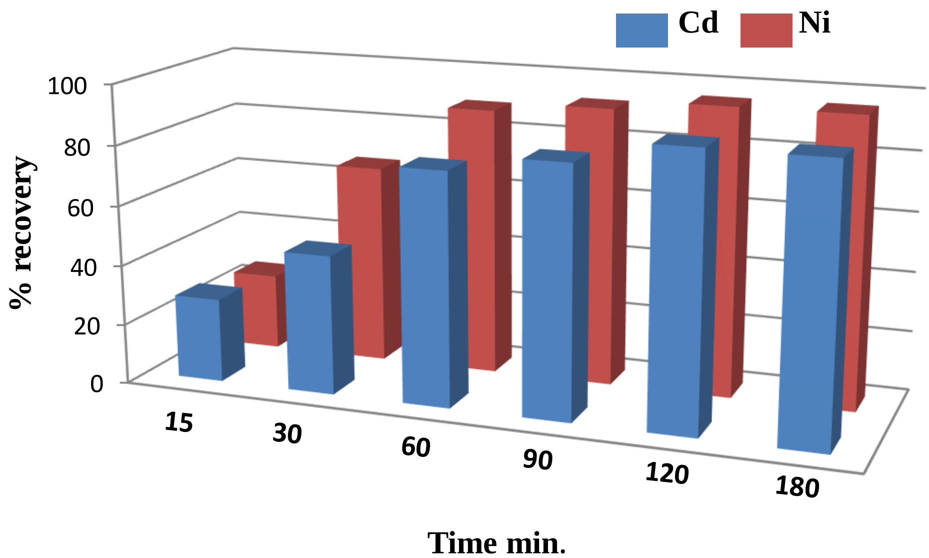

Fig: 5: relationship between leaching time Vs. recovery

1.5 – Effect of sample weight:

This study was carried out using different weight of sample (0.1, 0.5, 1, 1.5, 2, 3 gm) using 20 ml of 5 M acid solutions. The results obtained are tabulated in table 7, which revealed that the metals in samples were recovered with high recovery percentages until reached 2 gm weight of sample and then the recovery percentages were decreased as samples weight increase, that may be due to the volume of acid is not efficient enough to dissolve the sample. So the considered ratio of weight to volume in this research was 2 gm / 20 ml.

Table. 7: the obtained results from sample weight study.

Sample weight gm.	% Ni recovery	% Cd recovery
0.10	98.83	98.00
0.50	99.06	98.00
1.00	99.0	97.8
1.50	98.75	97.3
2.00	97.50	97.00
3.00	64.72	63.73

1.6 – Effect of temperature:

In this study the effect of leaching temperature on recovery of Cd and Ni was investigated. The solutions of nitric acid and sulfuric acid have a concentration of 5 M were used for the recovery of Cd and Ni respectively, at different temperatures (table 8 and figure 6). The obtained results indicate that the recovery percentages were increased as the temperature of leaching was increased until reaching 70 °C, after that the value of increasing being small or decreased due to the re-precipitation process which occurs at high temperature.

Table. 8: the obtained results from temperature study.

Temperature °C	% Ni recovery	% Cd recovery
25	95.50	90.10
40	96.00	91.00
50	97.10	93.00
60	98.20	95.50
70	99.00	98.50
80	99.01	98.30

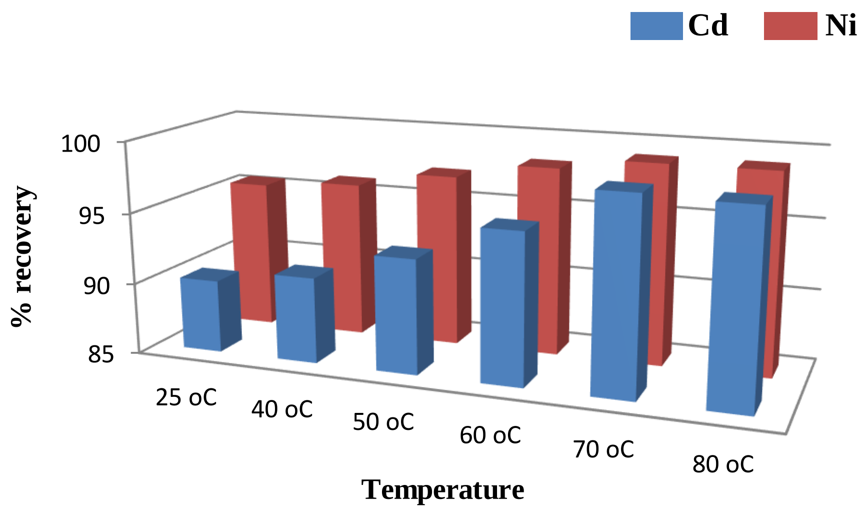

Fig: 6: relationship between temperature Vs. recovery

2 – Precipitation studies:

2.1 – Precipitation of iron:

In this set of experiments the precipitation of iron from leaching solution was investigated. Different types of solution were used to carry out this study. The solutions have a concentration of 20% NH_4OH and 1 M NaOH were used for selective precipitation of iron ion at different pH (table 9 and figure 7). The results obtained revealed that the precipitation of iron from leaching solutions by using ammonium hydroxide was reaching its highest precipitation percentage values at pH 3 for nickel solution and pH 4 for cadmium solution. Also the results indicating that the precipitation percentages were reached its highest values at pH 4 for (Ni and Cd) using sodium hydroxide as precipitating agent. The lose percentages of the metals were reached its highest value at pH 4, as shown in figure 6. The acidity of precipitation of iron from the leaching solution was considered at pH 3, using a sodium hydroxide solution as a precipitating agent for these reasons:

1- To avoid the uses of ammonium hydroxide, which is need special equipment and conditions, due to the production gases?
2- To avoid the precipitation of [$NiSO_4$ $(NH_4)_2$. 6 H_2O] salt [23].
3- To minimize the lose percentage value of Ni and Cd.
4- To minimize the addition of a large quantity of solution, this causes impurities in the solutions.

Table. 9: the results obtained from iron precipitation study.

pH	Nickel precipitation results				Cadmium precipitation results			
	20% NH_4OH		1 M NaOH		20% NH_4OH		1 M NaOH	
	% Fe precipitate	%Ni loses	% Fe precipitate	%Ni loses	% Fe precipitate	%Cd loses	% Fe precipitate	%Cd loses
1	57.30	1.80	70.79	2.00	19.23	1.90	51.54	2.00
2	68.54	2.00	77.53	2.70	32.31	2.10	72.31	2.40
3	83.15	3.00	85.20	3.50	61.00	2.50	87.69	2.50
4	83.15	5.00	86.52	5.20	86.15	3.00	89.23	3.00

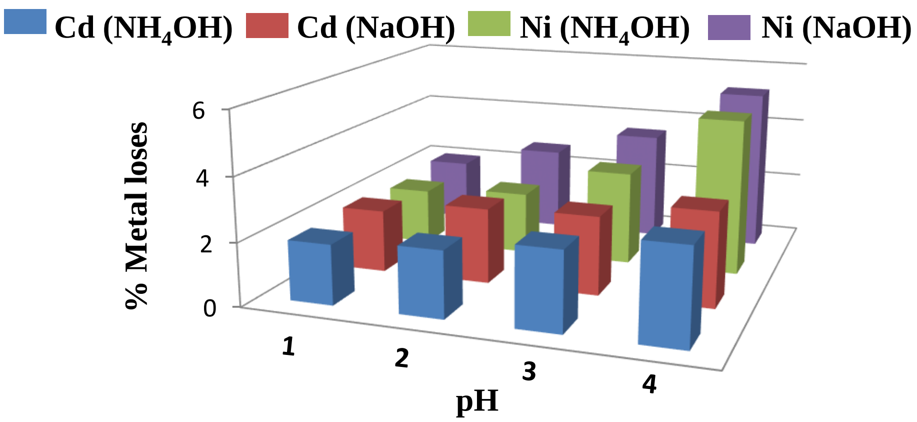

Fig: 7: releatioship between pH Vs. metals loses

2.2 – Precipitation of metals (Ni and Cd) studies:

2.2.1 – Effect of pH on precipitation:

In this set of experiments the precipitation of Ni and Cd from free iron leaching solution was investigated. A solution has concentration 2 M NaOH was used for selective precipitation of iron ion at different pH (Table 10). The results obtained indicated that the precipitation percentages of Ni and Cadmium from leaching solutions were reached its highest precipitation values at pH 11 and 10.5 respectively. The lose percentages values of the metals were decreased as value of solution pH was increased, as shown in figure 8.

Table. 10: the results obtained from pH study.

pH	%Ni precipitate	%Ni loses	%Cd precipitate	%Cd loses
7	66.90	33.10	60.00	40.00
8	67.40	32.60	65.40	34.60
9	81.10	18.90	72.90	27.10
10	91.10	8.90	82.00	18.00
10.5	92.30	7.70	90.20	9.80
11	94.00	6.00	90.60	9.40

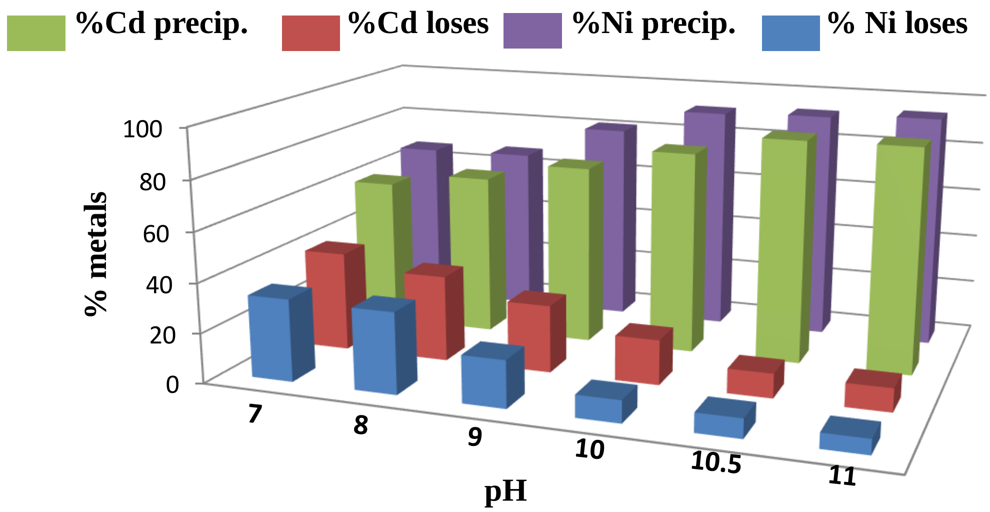

Fig: 8: relationship between pH Vs. % metals precipitate or lose

2.2.2 – Effect of precipitation time:

In this study the effect of precipitation time on the precipitating process of Cd and Ni was investigated. A solution of sodium hydroxide has a concentration of 2 M was used for the precipitation of Cd and Ni at different time (Table 11 and figure 9). The obtained results indicate that the precipitation percentages were increased as the time of precipitation was increased until reached 120 min., after that the value of increasing being small or near constant.

Table. 11: the obtained results from precipitation time study.

Precipitation time min.	% Ni precipitate	% Cd precipitate
15	88.70	66.70
30	91.50	80.40
60	94.20	90.60
120	97.00	94.50
180	97.00	93.70

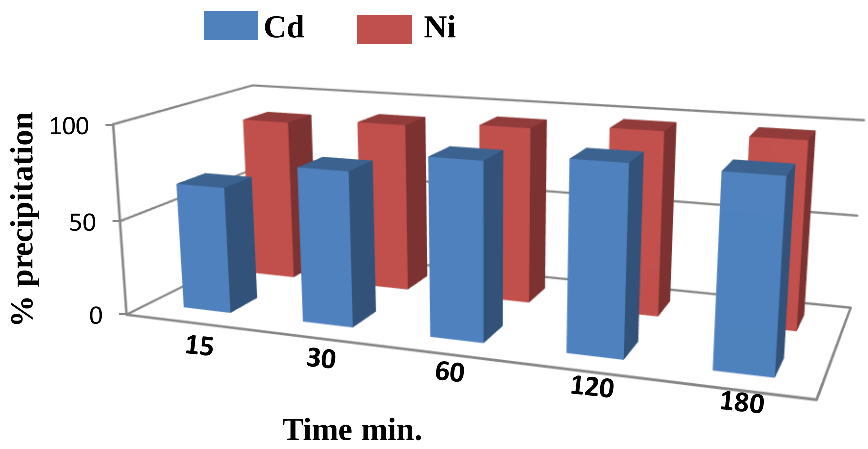

Fig: 9: relationship between time Vs. % precipitation

2.2.3 – Effect of temperature:

In this study the effect of precipitation temperature on precipitation of Cd and Ni was investigated. A solution of sodium hydroxide has a concentration of 2 M was used for the precipitation of Cd and Ni at different temperatures (table 12 and figure 10). The obtained results indicate that the precipitation percentages were increased as the temperature of precipitation was increased until reaching 25 ^oC, after that the value of increasing being small or decreased due to the re - solvation process which occurs at high temperature.

Table. 12: the obtained results from temperature study.

Temperature ^oC	% Ni precipitate	% Cd precipitate
15	96.50	94.40
20	97.50	96.50
25	99.30	98.60
30	99.10	98.20

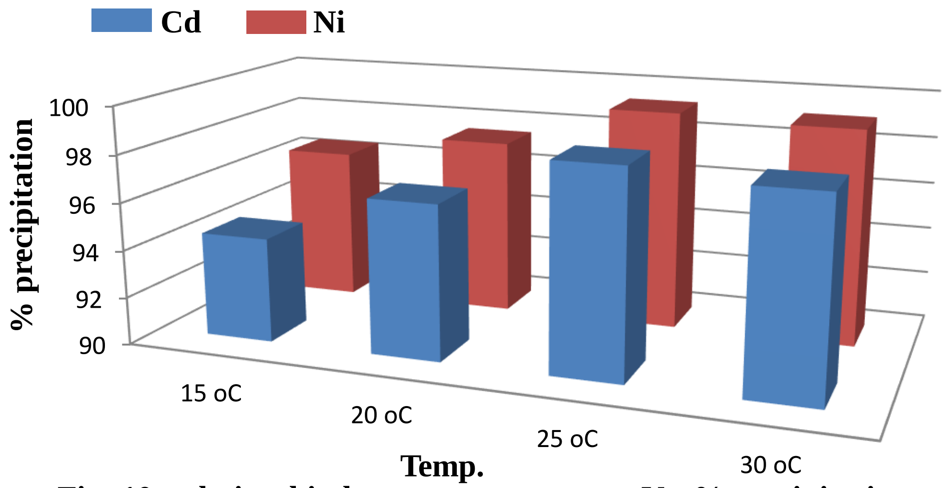

Fig: 10: relationship between temperature Vs. % precipitation

3 – Purification studies:

These studies were carried out using double distilled water to remove impurities such as sodium sulfate from nickel precipitate and sodium nitrate from cadmium precipitate. Many parameters affected the purification process such as (time, temperature and number of washing process) were studied. The results obtain are tabulated in table 13. The purity of products Cd $(OH)_2$ and Ni $(OH)_2$ was found to be 98.08% and 98.23% respectively.

Table. 13: the obtained results from purification study.

Parameters	Ni results	Cd results
Time of washing (min.)	60	30
Volume of water (ml)	25	25
Weight of precipitate (gm)	2	2
Temperature of washing °C	40	50
Number of washing process	4	3
Purity of metal hydroxide	%98.23	%98.08

Conclusions

This paper discusses the possibility of nickel, cadmium leaching with sulfuric acid and nitric acid as the first step for recovery of metals from nickel-cadmium spent batteries. The results of the experimental study indicate that leaching with sulfuric acid has the concentration of 5 M H_2SO_4 and 5 M nitric acid, as well as liquid/solid ratio of 1:10 and the temperature of 70 ºC could be applied for this purpose. Iron salts in leaching solutions were successfully precipitated with sodium hydroxide in acidic medium (pH 3). The metals were successfully precipitated as hydroxide in basic medium using sodium hydroxide. The studies showed that the high recovery degree of cadmium and nickel from spent Ni-Cd batteries is achievable by this hydrometallurgical treatment method. The discussion of results is detailed presented in sections 3 in this paper.

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