

Chemical and Electrochemical Recycling of Nickel from the Positive Electrode of Spent Nickel – Cadmium Batteries in Sulphuric Acid

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Abstract

In this research work, The recycling of spent nickel - cadmium batteries is an important issue not only from the viewpoint of treatment of hazardous wastes but also due to the recovery of valuable elements present in such a waste. Chemical and electrochemical recycling metal value by environmentally friendly process is the best option to obtain economical advantage and prevent environmental pollution. The chemical recycling methods for the Ni, Co, and Si from the positives electrodes of spent Ni - Cd batteries were studied using different acidic solutions. The first one is sulphuric acid, which was used for nickel recovery. The chemical compositions of the components of the cell. It was found that nickel was present in both the active electrode materials of the cathode. cadmium was the main constituent of the anode, cobalt and silicon was present in both electrodes. The present paper reports improved nickel recoveries in sulphuric acid leaching of Ni–Cd battery powder at relatively lower temperatures. the positives electrodes of spent Ni–Cd battery powder containing about 55% Ni, 2.2% Cd, Co, 2.4 % and Si 0.30 % was leached in sulphuric acid varying the parameters viz., acid concentration, time, and temperature. The recovery percentages of metal ion were found to be 98.72% of nickel using optimum conditions (5 M H₂SO₄), 2000 mg sample, 180 min, and 25 ml acid volume. Dissolution of various nickel phases present in Ni–Cd battery powder was investigated by SEM, XRD analysis and the concentration of metal ion in the solutions was measured using AAS.

Keywords: pollution, nickel - metal hydride Batteries recycling, SEM, XRD, AAS.

Introduction:

The Ni–Cd metals have been used in applications that require high energy density, long lifetime and high discharge ratios.[12]. Chemical and electrochemical recycling methods for the Ni, Co, Zn and Mn from the positives electrodes of spent Ni-MH batteries were developed [28]. The production of rechargeable nickel-cadmium batteries began in Europe and in United States in 1950. These types of batteries have been used in applications that require high energy density, long lifetime and high discharge ratios[29]. Cadmium – Nickel power supplies account for about 10% of all chemical current sources in use[13] So 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 [3]. The refore, recycling of spent batteries appeared as the most adequate destination for this type of waste. Recycling of wastes is important since it may contribute to the benefit of future generations and preservation of raw materials [1]. Spent nickel cadmium batteries are classified as hazardous waste due to the presence of toxic cadmium in high concentration. Recycling of metal value by environmentally friendly process is the best option to obtain economical advantage and prevent environmental pollution[2]. Therefore,

any recycling technology should be designed for nickel cadmium batteries where it is developed to fully recover these metals into the final product, thus preventing the formation of gas emissions and keeping these metals out [5]. In recent years, several studies have been conducted on the treatment and recycling of metals from nickel cadmium batteries through hydro-metal processes, AAS measurements and Scanning Electron Microscopy (SEM). Various authors investigated in details the individual stages of the hydrometallurgical treatment. Studies of acidic leaching[32, 20, 23]. Elements like nickel, cadmium, tellurium, and rare earths are important raw materials for the manufacture of batteries and energy equipments [17].

The spent Ni–Cd batteries have been ending up in huge piles and waste streams polluting the environment due to considerable cadmium content in the landfills [19]. Leaching of shredded and separated Ni–Cd battery powder in acid lixiviate has been key step in hydrometallurgical developments. Processes based on hydrochloric acid leaching of spent Ni–Cd battery effectively dissolve nickel and cadmium [6, 25, 11]. showed that the hydroxide phases of nickel, cobalt and cadmium were effectively leached in 5.86% v/v H₂SO₄ within 15 min at about 323 K. But overall nickel leaching efficiency at 358 K was about 73% and 93% from anodic and cathodic materials, respectively [26]. The materials were characterized with the aid of X ray diffraction (XRD),

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scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and Atomic absorption (AURORA – Canada) were used through this study. Nickel - cadmium (Ni-Cd) batteries are an example of rechargeable batteries. This technology was proposed by Waldemar Jungner (1869-1924) in 1899. After some technological improvements [7] there was a burst in their consumption in the 1950s followed by another in the 1980s due to the appearance of portable electronic devices [8]. Despite its advantages, cadmium is a very toxic metal (it comprises 15-20 wt.% of the Ni-Cd battery) for humans. Nickel is also a toxic element [18]. In many applications, Ni-Cd batteries have been replaced by Ni-MH and Li-ion systems [27]. recovered nickel and cobalt from waste alloys. A flow sheet comprised of acidic leaching precipitating copper, iron and chromium was tested. The recovery of cobalt amounted to 91.8% and that of nickel to 97.2%. [31]. studied the leaching kinetics of nickel and cobalt in spent cadmium-nickel batteries. The recovery process was controlled by surface chemical reaction. [30]. The effect of factors like temperature, acid concentration, reaction time, stirring speed and grinding of material on the leaching yields of metals contained in anodic and cathode materials (nickel, cadmium and cobalt) using sulphuric acid [21,24]. End-of-life products have to be processed ensuring not only less consumption of natural raw materials but also environmentally sound processing and disposal. Recycling is one of the waste management options for attaining this objective and must be used when economic benefits are expected. This approach can be applied to spent batteries [9,10] which can be considered high-grade metal products with potential economic value. Nickel-cadmium batteries are a particular case, containing nickel with a high economic value together with cadmium, which is environmentally very harmful. Elemental quantitative analysis showed that the electrodes are highly concentrated in metals. The phase identification achieved by X-ray powder diffraction combined with chemical analysis and leaching tests allowed the authors to proceed with the composition of the electrode materials as following: cathode: 28.7% metallic Ni, 53.3% Ni(OH)₂, 6.8% Cd(OH)₂ and 2.8% Co(OH)₂; anode: 39.4% metallic Ni and 57.0% Cd(OH)₂ [22].

Experimental:

Apparatus:

Sartorius balance (Germany), Shaker Device (Germany), Atomic absorption (Germany), X-ray

(Germany), scanning electron microscopy (SEM) (Germany), and Atomic absorption (AURORA – Canada) were used through this study.

Materials and reagents:

The spent Ni-Cd battery powder was obtained were supplied from Hadhramout Valley, Yemen electrical power stations. All of the chemical that are employed substances in this work were of analytical grade and are used without any further purification. the required chemicals involve. Sulfuric acid (BDH), Hydrochloric acid (BDH), Nitric acid (BDH), Sodium hydroxide (GCC), Potassium hydroxide (GCC), Sodium carbonate (GCC), and Sodium bicarbonate (GCC).

Methods of preparation of acid solutions and bases:

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 to 2 M) sulfuric acid, (6 M to 2 M) nitric acid, (6 M to 2 M) hydrochloric acid, 2M sodium hydroxide, 2M potassium hydroxide, 2M sodium bicarbonate, 2M sodium carbonate). The more dilution solutions were prepared by distilled water.

Procedure sample spent of Ni-Cd electrode:

The components of spent Ni-Cd alkaline batteries of electric power stations were separated manually. An cathode (positive electrode) which contains nickel was leaching with sulfuric acid, while the anode (negative electrode) which contains cadmium. The leaching experiments was using different concentrations of sulfuric acid. add it to certain standards to nickel paste and put it in the shaker device using different concentrations, time, size and temperature and then filtered. The same method is used with cadmium paste but using nitric acid at different concentrations. the concentrations of nickel were determined by AAS after required dilution with deionized water. Leaching efficiency was calculated from the fraction of Ni-Cd powder leached in the solution to the initial weight taken. The recoveries of Ni were calculated from their concentrations in the leach liquor. [16]

Results and discussion

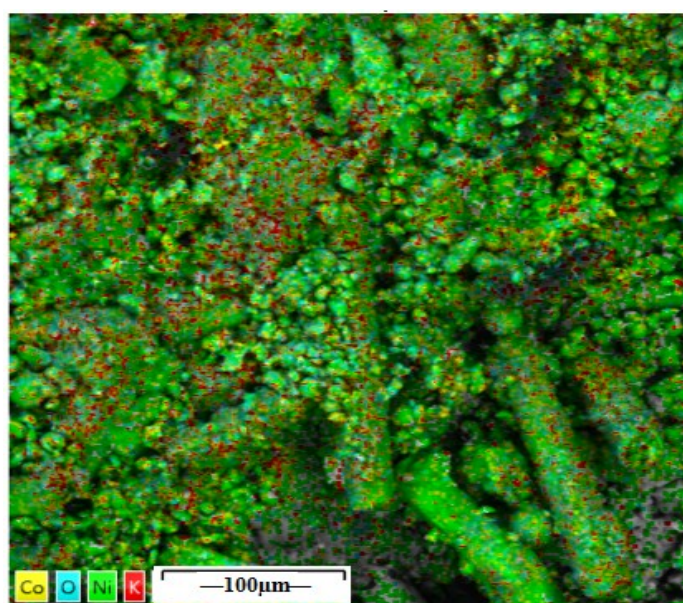
Characterization of the cathode materials of spent nickel and cadmium batteries.

The chemical composition of spent Ni-Cd battery powder. (Fig.1) represents the micrographs of the cathode materials of spent nickel and cadmium batteries. The predominant crystal structure of the Ni-Cd battery cathode material is hexagonal. (Fig.2) shows the energy-dispersive X-ray (EDS) spectrum obtained from the positive

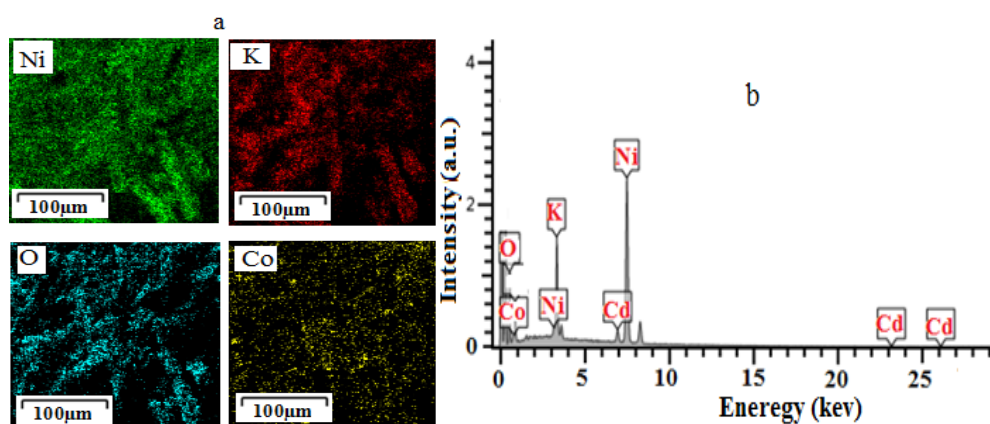
electrode material. The presence of potassium from the KOH electrolyte is detected on the surface of the material. The presence of oxygen is due to Ni and Co oxides. The carbon and cobalt detected are used as additives to improve the electrical conductivity in the positive electrode. (Fig.3) shows the XRD results for the Ni-Cd cathode material. The diffracts gram shows peaks that, when compared with those reported in the JCPDS database, correspond to Ni(OH)₂ [14] and Co(OH)₂ [15]. The structure of the compound Co(OH)₂ is also hexagonal; however. NiOOH exhibits an orthorhombic structure with parameters. The structure of NiO has a cubic

structure [14 - 15]. Additives Worked on that stabilize the a-Ni(OH)₂ phase and improve the capacity of the electrodes respectively. a-Ni(OH)₂ became b-Ni (OH)₂ during the charge-and-discharge cycling [27,31]. Chemical composition of the cathode materials of spent Ni-Cd battery powder is given in (Table.1) shows that nickel and cadmium are the major constituents. Other elements like Si and Co were present in minor amounts.

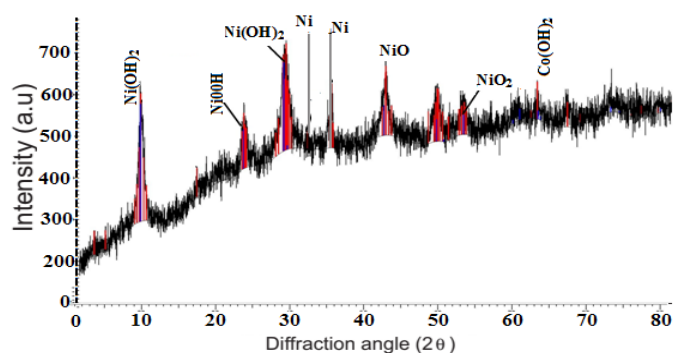
It is noted that nickel content in the present sample is quite higher than that has reported by other researchers in spent Ni-Cd batteries [20,6,26,22].



Figure(1): Scanning electron micrograph of the positive electrode of spent Ni-Cd battery powder



Figure(2): Scanning electron micrograph (a) and Energy dispersive X-ray (EDX) spectrum (b) obtained for the positive electrode of spent Ni-Cd battery batteries.



Figure(3): Typical X-ray diffraction spectrum of a positive electrode from a spent Ni-Cd battery

Table (1): Chemical composition of spent Ni-Cd battery powder of the positive electrode and leached residue

Element	Chemical composition	(Wt.%)
	Spent Ni-Cd battery powder	Leach residue
Ni	55.0	98.4
Cd	2.2	Not found
Co	2.4	1.6
Si	0.1	Not found

Leaching studies:

Recovery of Nickel using different chemical solutions:

This study was conducted using different acidic and alkali solutions 4 M (Na_2CO_3 - KOH - NaHCO_3 - NaOH - HCl - HNO_3 - H_2SO_4) to recover nickel from nickel paste. The results were shown in (Table 2). The recovered nickel was higher when the solution was acidic, and it was lower

when the solution was is alkali. The highest amount of the recovered nickel was resulted when the sulfuric was acid solution, and its least amount was resulted when it was sodium carbonate solution. (Fig. 4) showed the relationship between the nickel recovery rate and the type of solution of the highest nickel recovery rate.

The recovery percentage was calculated as follows:

$$\% \text{ recovery} = \frac{\text{metal in solution (mg)}}{\text{metal in sample (mg)}} \times 100 \dots\dots\dots (1)$$

Table (2): Results of the nickel recovery study using different chemical solutions

Type of solution	Quantity of nickel paste (mg)	Recovery of nickel %
H_2SO_4	2001	90.20
HNO_3	2100	84.50
HCl	2121	79.52
NaOH	2001	3.23
KOH	2000	3.11
NaHCO_3	2100	2.54
Na_2CO_3	2060	1.82

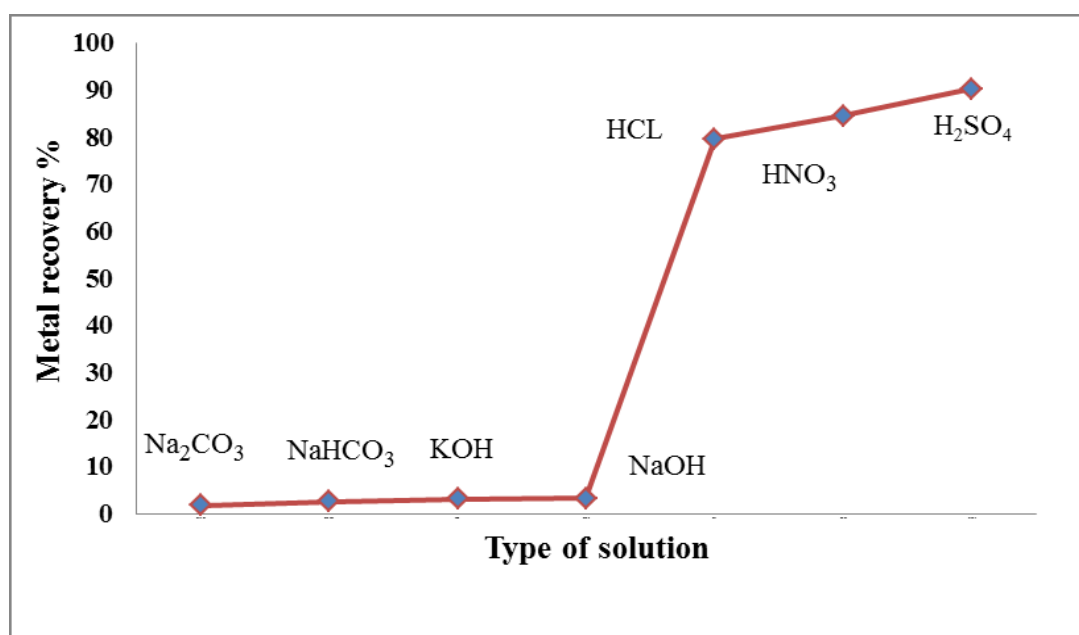


Figure 4: relationship between different chemical solutions

Effect of acid concentration

To study effect of the sulfuric acid concentration on nickel recovery, a sample was leached in (1 , 2, 3, 4, 5 , 6) M. The results were as shown in Table (3) where it was found that the best recovery amount was noticed when the concentration of the sulfuric acid was 5M, and it was less when

the concentration of the sulfuric acid was 1M. The results obtained in this study were compared with other published studies [30]. So when the acid concentration was higher, the recovery ratio was better . Because the difference in the recovery rate is higher at the recovery rate when the acid concentration 6M (Figure 5).

Table (3): Results of the nickel recovery study using different concentration

Acid conc. mol. / l	Quantity of nickel paste (mg)	Recovery of nickel %
1	2021	21.21
2	2002	43.54
3	2091	64.32
4	2003	81.51
5	2024	98.72
6	2016	94.32

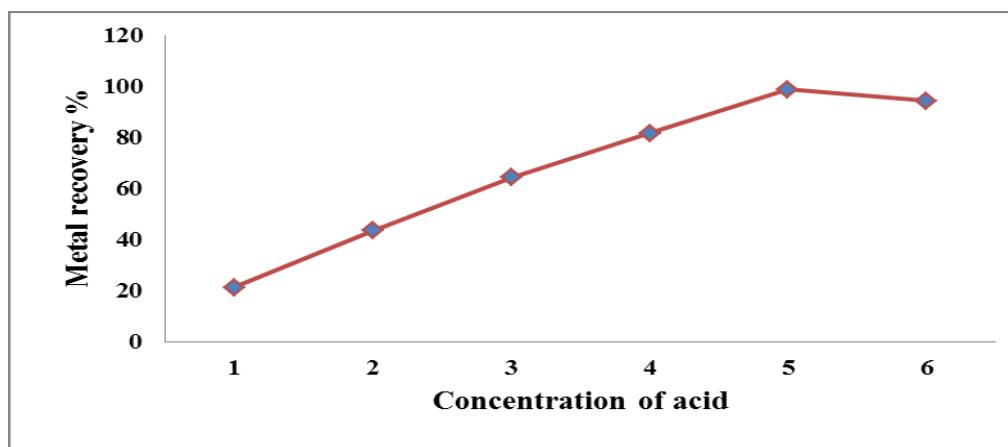


Figure 5: relationship between different solution concentration study

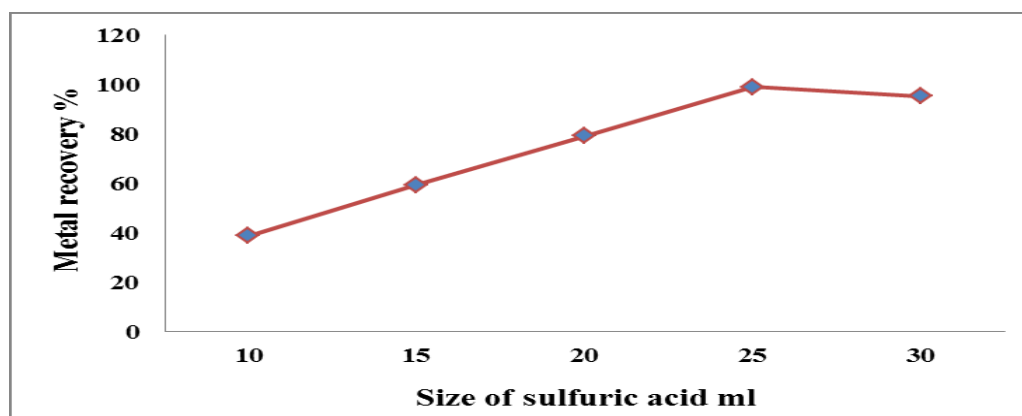
Effect of solution volume:

This study was conducted using a sulphuric acid solution at the concentration of 5 M and different volumes of sulphuric acid (10 ml, 15 ml, 20 ml, 25 ml, 30 ml) to recover nickel from cadmium paste. The results were shown in (table 5). It was found that the recoverable amount of nickel was the highest value when the sulphuric acid volume

was 25ml, and the amount of nickel recovered was less when the volume of sulphuric acid was 10 ml and size 25 ml was adopted as the best size of the recovery ratio. So that increasing the volume led to a dilution in the recovery solution. Because the difference in the recovery rate is higher at the recovery rate at volume 25 ml (Fig.20).

Table (4): Results of the nickel recovery study using different Size

Volume of sulphuric acid (ml)	Quantity of nickel paste (mg)	Recovery of nickel %
10	2091	38.82
15	2089	59.53
20	2000	79.21
25	2005	99.12
30	2017	98.31



Figure(6) : relationship between different between acid volume Vs. recovery

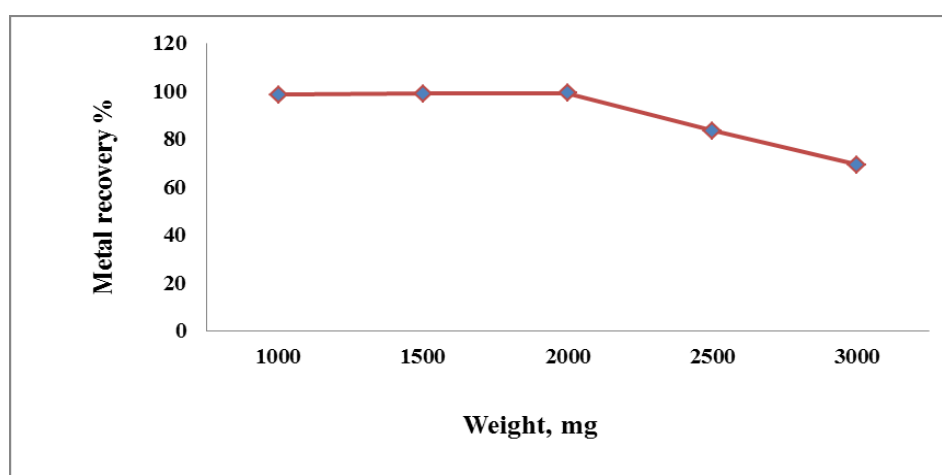
Effect of change of weight of sulfuric acid on nickel recovery process:

The sulfuric acid solution was used at concentration of 5M different weights of the nickel paste were used. The following different weights were taken (1000 mg - 1500 mg - 2000 mg - 2500 mg- 3000 mg) to recover nickel from the nickel paste, and the results were shown in (table 6) which revealed that the metals in samples were recovered with high recovery rates

reached at 2000mg weight of sample and then the recovery rates were decreased as samples weight increased. That might be due to the volume of acid which was not efficiently enough to dissolve the sample. So that the considered ratio of weight to volume in this research was 2000mg / 25 ml. Because the difference in the amount of nickel recovered is higher than the amount of nickel recovered when using the dough weight (3000mg). as shown in (Fig.21).

Table(5) : relationship between different solution weight study

Weight of nickel (mg)	Quantity of nickel paste (mg)	Recovery of nickel %
1000	1001	98.41
1500	1504	98.91
2000	2040	99.21
2500	2506	83.52
3000	3015	69.32

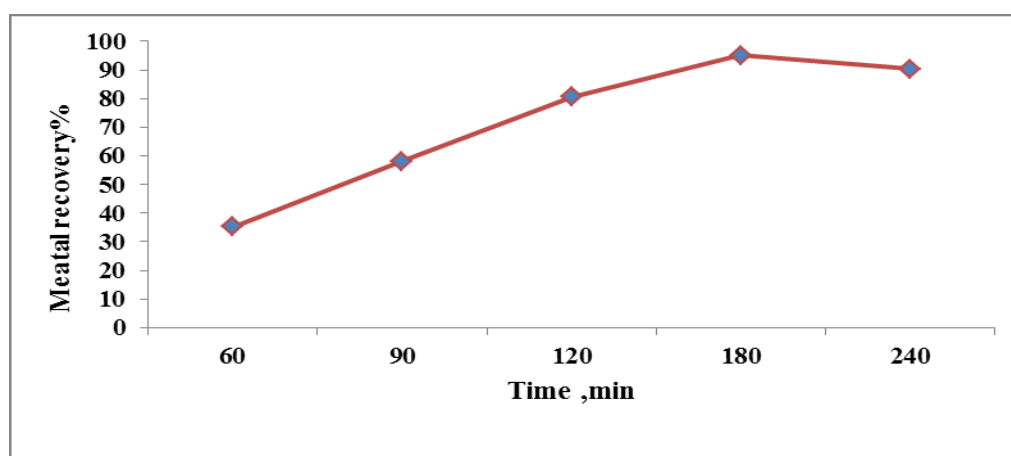
**Figure (7) : Relationship between different between weight****Effect of change of time of sulfuric acid on nickel recovery process:**

The sulfuric acid solution was used at a concentration of 5M at different times ranging from (60,90,120,180,240) minutes to recover nickel from the nickel paste as shown in (table

7). The results showed that the recoverable nickel was at the highest value when the recovery time was 180 minutes and the recoverable amount of nickel reduced when the recovery time was 60 minutes (Fig.22).

Table (6) : Results of the nickel recovery study using different time

Type of time (min)	Quantity of nickel paste (mg)	Recovery of nickel %
60	2037	35.23
90	2016	58.2
120	2000	80.52
180	2009	95.11
240	2020	90.23

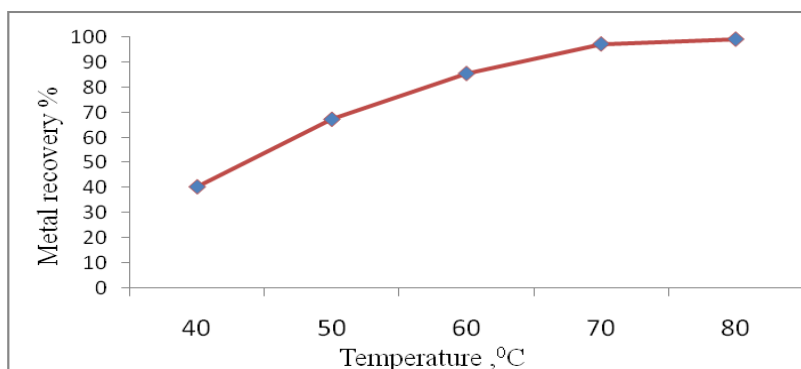
**Figure(8) : relationship between different solution time study****Effect of change of temperature of sulfuric acid on nickel recovery process:**

This study was conducted using a sulfuric acid solution at a concentration of 5 M at a different temperature of (40 - 50 - 60 - 70 - 80) ° C to recover nickel from the nickel paste as shown in

the table (7) The recovered nickel was found to be increasing by increasing the temperature with the highest nickel recovery at 80 ° C and the lowest nickel recovery when the C was below 40 ° C to illustrate this as shown in the Figure 9.

Table(7) : relationship between different solution temperature study

Temperature (° C)	Quantity of nickel paste(mg)	Quantity of recovered nickel (%)
40	2021	40.34
50	2000	67.30
60	2001	85.60
70	2023	97.40
80	2015	99.31



Figure(9) : relationship between different solution temperature study

Characterization of materials recovered by chemical precipitation from cathodes of Ni-Cd batteries:

Characterization of the leaching residues:

The final leach residue obtained at different times was identified by X-ray diffraction presented in (Fig.10, 11, 12.) for phase identification. Apparently, leaching in sulphuric acid dissolves both the cadmium phases i.e. CdCO_3 and Cd(OH)_2 along with $\text{Ni}_2\text{O}_2(\text{OH})_4$ as confirmed by the absence of corresponding peaks in (Fig. 10,11,12). Few very low intensity peaks are attributed to Ni(OH)_2 (JCPDS file: 27-0956) [31].

The morphology of the leach residues is examined by XRD – SEM and shown in (Fig. 13 (a-e)) The final leach residue obtained at different times was identified by X-ray diffraction presented in (Fig. 10,11,12.) and also described elsewhere [22,4] . XRD patterns of leach

residues obtained at different time intervals indicate only the presence of Ni phase. The solid particles before leaching are showing broad size distribution (Fig.13 a) as well as a dense surface. The micrographs of the leaching residues have shown a marked change in morphology (Fig. 13 b and c) that involved progressive reduction in particle size and then an increase in roughness and porosity of the solids. For instance, after about 99% Ni extraction (Fig. 13 b), the particles show mesh type structure formed of small particles that disintegrate along the progress of leaching (Fig. 13 c and d). The nickel containing powdery mass (mesh like formations) progressively has disappeared while the pitted connecting wires comprising Ni and Co have predominated at later stages of leaching. (Fig. 13 .e) shows the final leach residue of (initial sample) that comprises only connecting wires and hard parts, containing nickel (99.00 wt.%).

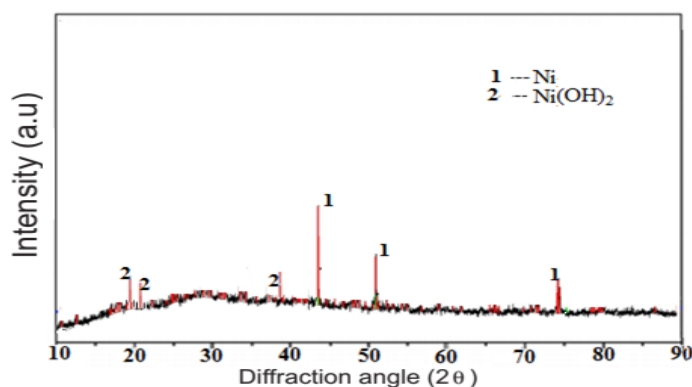


Figure (10): X-ray diffraction pattern of leach residue obtained after leaching of nickel-cadmium battery powder with in 20 ml in 5M (H_2SO_4) and 120 min temperature: 358 K

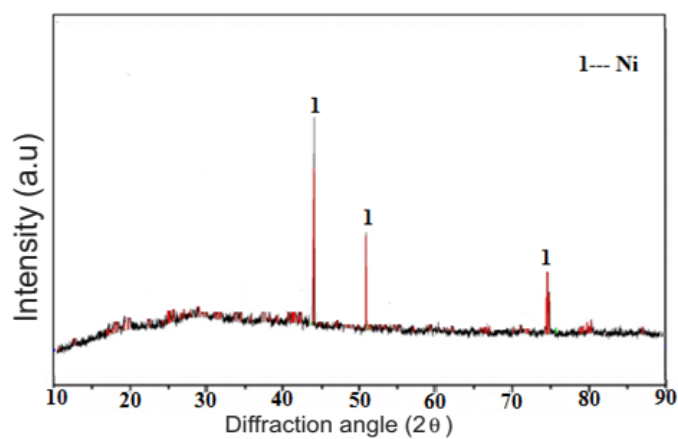


Figure. (11): X-ray diffraction spectrum of leaching residues obtained for the positive electrode of spent Ni-Cd batteries in 25 ml in 5M (H₂SO₄) and 180 min temperature: 378 K

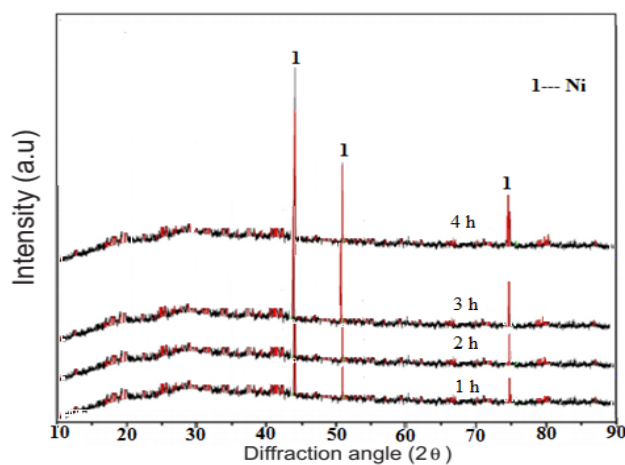


Figure. (12): X-ray diffraction spectrum of leaching residues obtained at different time intervals

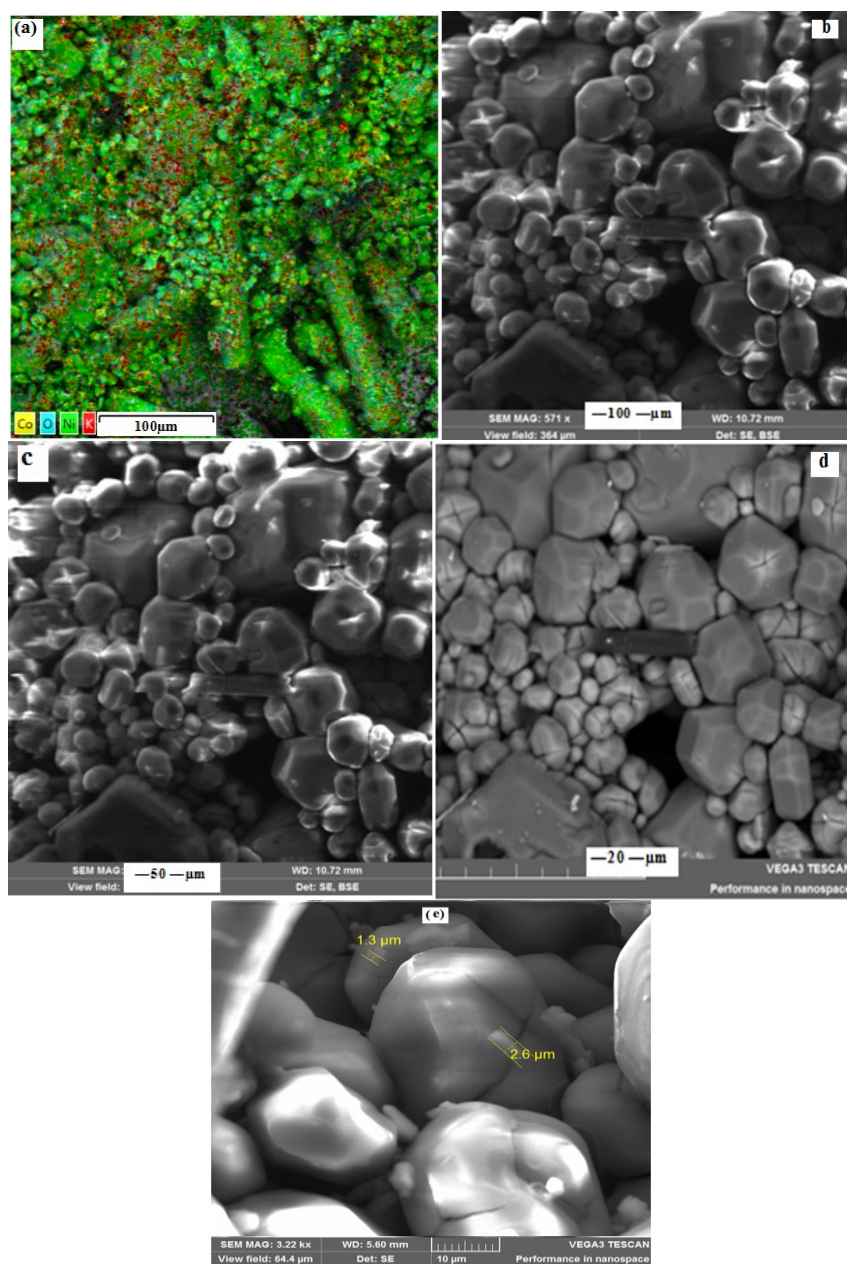


Figure. (13): (a) through (e) SEM photographs of the cathode materials of Ni–Cd battery powder and leach residues: (a) Ni–Cd battery powder, (b) residue — 60 min, (c) residue — 120 min, (d) residue — 150 min, (e) residue—180 min.

Conclusions:

It has been shown in the present study that,

- 1- The studies discussed presented in this paper showed that high degree of recovery of nickel is achievable by the leaching with sulfuric acid leaching in H_2SO_4 of spent Ni–Cd batteries. Sulphuric acid leaching, the first chemical step of an integrated process for recovering metals from spent Ni–Cd batteries, was tested.
- 2- Improved nickel recoveries from nickel –

cadmium battery powder in sulfuric acid leaching were investigated. Sulfuric acid concentration up to (5 M H_2SO_4) had marked effect on nickel recovery; however, nickel recovery.

3- Temperature significantly enhanced the nickel leaching recovering 99.0% within 180 min at 378 K. Addition of hydrogen peroxide improved kinetics of nickel leaching, and the nickel recovery was enhanced to 99% in 4 h leaching time.

4- Nickel leaching increased with the tempera-

ture. SEM and XRD analysis showed a progressive change in particle morphology and size during leaching which supported the application of the shrinking core model to the leaching data.

5- The novel feature of the present study was high recovery of nickel (99. 0%) by leaching of spent Ni–Cd batteries using dilute sulfuric acid (5 M H₂SO₄) at moderate temperature (378 K).

6- Based on the experimental results, it is thus concluded that as well as waste Ni–Cd batteries,

the other solid wastes, which contain the metals, can be effectively recycled by using by different acidic solutions.

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إعادة التدوير الكيميائي والكهروكيميائي للنikkel من القطب الموجب لبطاريات النikkel والكادميوم المستهلك في حمض الكبريتيك

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الملخص

في هذا البحث، تعتبر إعادة تدوير بطاريات النikkel والكادميوم المستهلكة قضية مهمة ليس فقط من وجهة نظر معالجة النفايات الخطرة ولكن أيضاً بسبب استرداد العناصر القيمة الموجودة في هذه النفايات. تعد القيمة المعدنية لإعادة التدوير الكيميائي والكهروكيميائي من خلال عملية صديقة للبيئة هي الخيار الأفضل للحصول على ميزة اقتصادية ومنع التلوث البيئي. تمت دراسة طرق إعادة التدوير الكيميائي والكهروكيميائي، للنikkel، والكوبلت، والسليكون من الأقطاب الموجبة لبطاريات النikkel- الكادميوم المستهلكة باستخدام محاليل حمضية مختلفة. أول واحد هو حامض الكبريتيك ، والذي كان يستخدم لاسترداد النikkel. التركيبات الكيميائية لمكونات الخلية وقد وجد أن النikkel كان موجوداً في كل من المواد القطبية النشطة في الكاثود. كان الكادميوم هو المكون الرئيسي للأنود ، وكان الكوبالت والسليكون موجودين في كلا القطبين. تم تحليل انحلال مختلف مراحل النikkel الموجودة في مسحوق بطارية النikkel - الكادميوم بواسطة (XRD ، SEM) وتم قياس تركيز أيون المعادن في المحاليل بواسطة التحليل باستخدام AAS.

الكلمات المفتاحية: التلوث بالنikkel، إعادة تدوير البطاريات، اشعة اكس، الميكروسكوب الالكتروني الماسح، جهاز الامتصاص الذري.