Recovery of Metal Values from Secondary Resource

Mahmoud A Rabah [1, *], Omaima A El Qady [2], Shaimaa A Abou El Kassem [2]

[1] Chemical and Electrochemical Lab., Mineral Processing Dept. Central metallurgical Research and Development Institute (CMRDI) P.O. Box 11421, Helwan, Cairo Egypt.
[2] Powder Metallurgy Lab., Central metallurgical Research and Development Institute (CMRDI) P.O. Box 11421, Helwan, Cairo Egypt.
*Corresponding authors email: mrabah010@gmail.com

Abstract: The input waste was KanthalA4 grade alloy spired in 8-25 mm outer diameter. A sample weighing ≈250 g was leached in HCl or aqua regia at room temperature. The acidic leachate was filtered and the pH value was increased to *4.0*-9.2 with the addition of 3 M ammonia solution. The leachate was freed from iron by H_2O_2 addition. Chromium ions were separated from nickel ions by pH adjustment and solvent extraction using Lewatite MP 600 ionic exchanger. Ammonium hydroxide gave insoluble hydroxide ppt that was filtered and washed. Different organic salts of acetate Ni $(OCOCH_3)2$, oxalate C_2O_4, citrate $C_6H_6O_5$ with ∆E of 74.16 KJ/ mol with Ni and 139.33 KJ/ mol with Cr. Inorganic salts were also prepared like Ni $(NO_3)_2.6H_2O$, dichloride $NiCl_2$, basic carbonate $NiCO_3.2Ni(OH)_2$ as well as free metals of nickel and chromium. The effect of the process parameters was studied. The time of synthesis decreased by stirring at 300 rpm by about 11 %. Ascorbic acid solution reduces the Ni and Cr hydroxide to their respective free metal *to give* spherical nanoparticles having the molecular size of 15-35 nanometer. SEM showed that Ni particles are spherical in morphology. XRD and XDS showed Ni and Cr are pure.

Keywords: Nickel and Chromium, Heating Element Scrap, Recovery of Nonferrous Metals, Solvent Extraction, Acid Leaching of Metal Scrap

1. Introduction

Cyanidation process may be the main technique for precious metals recovery of the mining industry. That technique consisted in an electrochemical process that oxidizes gold and reduces oxygen with cyanide solutions at pH values above 10.5. However, Tilley and Jefferson (2002) reported the recovery of nickel and chromium metals by thermal reduction of their salts using hydrogen gas at 300 °C. Nickel hydroxide Ni (OH)2 nickel oxide are used as catalyst intermediate and in the manufacture of Ni-Cd batteries given [1]. The chemical properties of nickel make it suitable to be combined with other elements to form many alloys [2, 3]. It imparts desirable properties as corrosion resistance, heat resistance, hardness, and strength are recommended. A process was reported [4]. in which chromium can be conveniently and inexpensively removed from chromium bearing metal scrap containing nickel and/or cobalt. It relates to scraps derived from Ni and Co-based superalloys containing Cr., Co and/or Ni and possibly Fe as the principal constituents. [5, 6] studied a hydrometallurgy process to dissolve metal values, followed by chemical purification and/or metals recovery by some methods. Pretreatment before leaching such as calcination can be used to increase leaching rate. These methods were described complex and only successful with fine scrap material such as grindings or atomized metal. Fletcher showed that dealing with metals may provide data on tonnages treated, description of scrap arisings and the processes operated for metal extraction and refining copper, aluminum, lead, zinc, tin and other precious metals [7]. Reference is made to difficulties encountered and the efficiency of reclamation, such as the small amount of zinc recycled as metal. The future developments, wider use of oxygen in copper refining and hydrometallurgical processing of high value and complex scrap are literature. The potential for metal recovery for domestic refuse and the role of the Waste Management Advisory Council described. a technique using CYANEX 923. [8] reported that oxalic acid was employed to precipitate the Ferron, nickel, cobalt and manganese from the leaching sulfate solution of ferrochrome alloy. The authors showed that the deposition efficiency of Ferron reaches 99% and the loss rate of chromium is less than 1% when 120% theoretical quantity of oxalic acid is used under 30°C and terminal pH = 3. At the same time, the A method was given to employ precipitation, solvent extraction, and electrowinning steps [9]. A toluene solution of Cyanex 923 is used as an extractant for the separation of Cr(III)

and Ni(II) from some commonly associated metal ions such as Al(III), Fe(III), Mn(II), Co(II), Cu(II), Zn(II), and Pb(II). The experiments conducted up to 10 cycles indicate a negligible loss in extraction efficiency. The electrodeposition of Cr (III) and Ni (II) offers average cathode efficiencies around 40 and 75%, respectively. The recovered metals are 99.9% pure. [10] reviewed methods of separation of valuable metals from pickling solution containing nickel frum the spent liquor by solvent extraction deposition efficiencies of nickel, cobalt and manganese in the solution were 98.4, 92.2, 97.5%, respectively. [9] recovered nickel from spent lithium and N-Me hydride batteries by leaching with aqua regia digestion. The author reported that after leaching, >97% of rare earth elements (REEs) were recovered with the addition of NaOH and Na_2SO_4 precipitants. The REEs-free solution was then further processed to separate and recover the battery metals present: Mn, Co, Ni, and Li as a marked improvement over the previously reported. Lithium recovery levels of 60–80% extent was reported. [11] claimed recovery of chromium, nickel, and other alloying elements in stainless and heat-resisting steel. Conventional nickel electrolysis processes were particularly attractive techniques. The use of side-stream purification of process solutions was to improve the purity of nickel metal. Moreover, nickel purification by electrolysis was effective in a variety of electrolyte systems, including sulfate, chloride, and nitrate. Conventional electrorefining processes typically employed a mixed electrolyte which included sulfate, chloride, and borate. The use of an electrorefining or electrowinning system for scrap nickel recovery could be combined effectively with a variety of other processes, such as cementation, solvent extraction, ion exchange, complex-formation, and surface sorption, developed for uranium and transuranic purification.

The aim of this study is to recover metal values and some salts from the spent heating coils made of KanthalA4 grade. The suggested experimental procedure involves breaking the scrap coils to separate rings followed by leaching with mineral acids. The leachate was partially neutralized with ammonia solution to a pH 4. Chromium was precipitated as a-Cr (OH)₃ at pH 4.57 to 6.576. Ni hydroxide precipitates at pH 9.3. Part of. neutralized leachate reacted with 1.0 M ascorbic acid to reduce nickel and chromium ions to their free metal particles. The remaining part of the leachate was treated with ammonia to a pH range 6-9.5 to precipitate insoluble hydroxide the ppt was washed with distilled water, and reacted with the acid of concern to obtain citrate, acetate, oxalate, chloride, nitrate and sulphate salts. Parameters affecting the recovery quality and extent were investigated.

2- Experimental

2.1 Materials and Methods

A sample of the spent heating coil scrap weighing about 2 Kg was obtained from the scrap stores of the CMRDI institute. The sample was washed with distilled water to get rid of the dust and/or foreign ingredients and dried. It was spilled to separate rings annually on basis of its brittleness. Figure 1 shows a photograph of the coil as received and the spilled separate rings.

Figure 1. Photograph of the heating coil as received Spilled rings of the scrap coil

2.2 The Chemicals Used in This Work

The chemicals used in this work are given in Table 1.

2.3 The process of preparation of the products

Figure. 2 shows a sequential process of the method used in this study for the preparation of the end products.

2.4 Determination of the physical and chemical properties

2.4.1 Determination of density of metal particles

Determination of density of metal particles was carried out by liquid displacement technique with the help of a pycnometer bottle 50 ml size.

2.4.2 Determination of nickel and chromium

Nickel and chromium and other metals content in the scrapped source were determined using ATI UNICAM 929 flame atomic absorption Spectro-photometer (FAAS) (Unicam Ltd., Cambridge, England). A standard blank solution containing exactly 0.1- 10 g /L metal of nickel and chromium nitrate was measured at a wavelength, 357.9 and 232.0 nm; HCL current, 11.5 and 7.5 mA; acetylene flow rate, 4.2 and 0.5 L/min; N_2O flow rate 4.7 mL/min (for Cr), air flow rate, 4.0 mL/min (for Ni); slit width, 0.5 and 0.2 nm; for Cr and Ni, respectively. The straight line in the plot was taken as a reference for measuring the unknown sample.

Table 1. Properties of the chemicals used in this study

| Material	properties	Purpose of use	supplier
sup CH₃COOH	90 % SP. Gr. 1.044 – 1.049		Riedel-de Hein
	SP.GR.1.18 (AR)		ADWIC
	Min. assay 36 %	Leaching	Riedel-de Hein
Nitric acid	Fuming 69 %	Process	
H₂SO₄	H₂SO₄ 95-97%		ADWIC
HCl	SP.GR.1.18 (AR)		
Oxalic acid	Pure crystals	Precipitation	
Acetic acid	60%	Synthesis of salt	Sigma, Aldrich
Citric acid	Colorless crystals	Synthesis of salt	Sigma Aldrich
Ascorbic acid	White solid	Chemical reduction	China
		Solvent extraction	
Ethyl alcohol	96% Pure grade	Solvent extraction	ADWIC
Acetone	Pure grade	Solvent extraction	ADWIC
DMSO	Pure grade	Solvent extraction	ADWIC
DMF	Pure grade		ADWIC
Ca carbonate, EJSF2	Extra pure	Synthesis process	Green Egypt
			Sigma Aldrich
NaOH (Sodium hydroxide)	Pure reagent for analysis	United Co. for chemicals &Med.	
Ammonium hydroxide	25 % Pure reagent for analysis	Preparations	Merck
AgNO₃ (Sliver Nitrate)	Pure reagent for analysis	Chlorideion determination	Merck
Mono-distilled water		Chemical reactions	
Tap water	Ultra-Filtered	Other purposes	
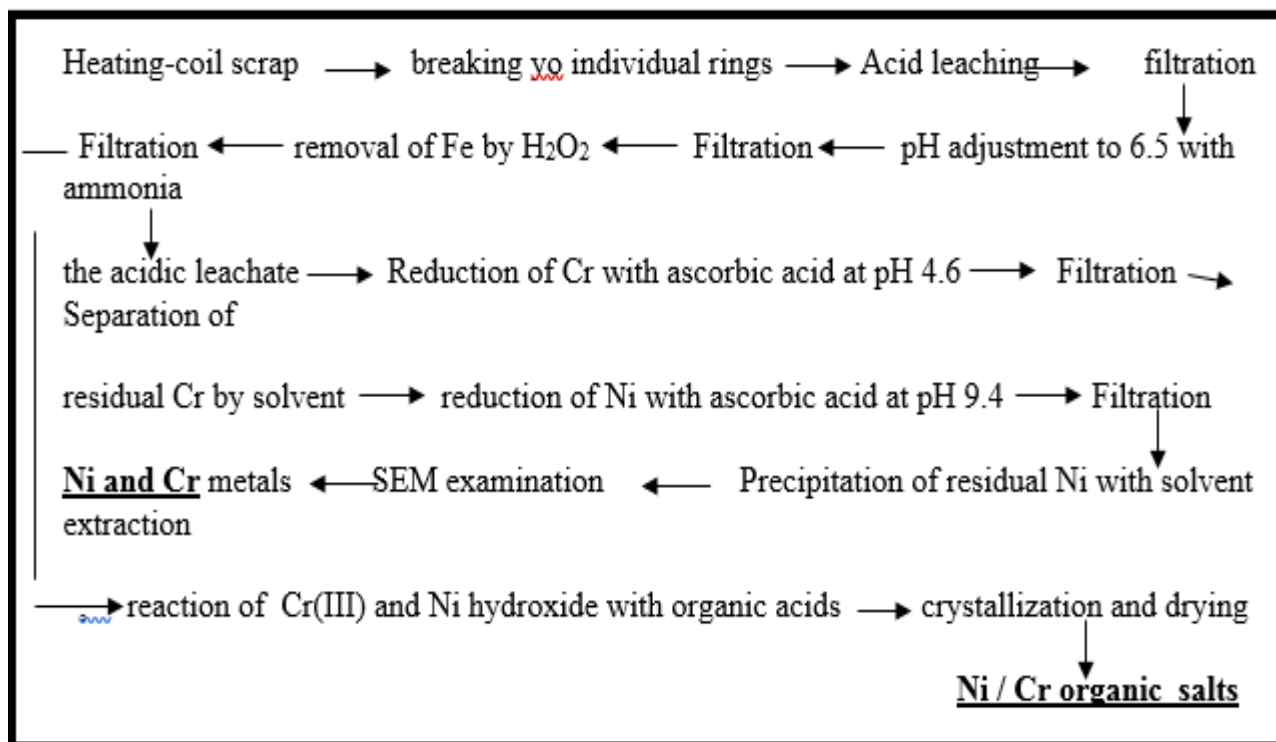
Figure 2. Sequential process of the method used in this study for the preparation of the end products.

2.4.3 Removal of Iron from the Leachate

Ferrous ions were removed from the acid leachate as ferric basic oxide Fe $(OH)_2O_2$ with the help of H_2O_2 addition to the acidic leachate. The filtrate contains nickel and chromium ions in nitric acid with other minor elements.

2.4.4 Oxidation of Chromium (III) to Chromium (VI)

Oxidation of chromium (III) to chromium (VI) using 30% hydrogen peroxide was conducted according to the method given by I. Bojanowska [12].

2.4.5 Separation of Chromium from Nickel-Chromium Nitrate Solution

Nickel was separated from chromium in the nitrate solution by adjusting the pH value using 30% ammonium hydroxide solution. Alternatively, the excellent nickel removal is performed by combining carbonate and hydroxide precipitations probably to benefit a coprecipitation phenomenon., the pH was adjusted by dropwise addition of IN NaOH and an equivalent amount of IN CaCI2 solution to simulate lime addition. Nickel selectively precipitates at pH of 10.3 – 11 (McAnally, [13]. The slurry of chromium hydroxide that separates at lower pH ($\approx$ 6) was filtered using vacuum suction filter fitted with a filter paper # 42 Whatman. Remaining Chromium was separated by solvent extraction. A strongly basic anion exchange lewatit MP 600 resin was studied to separate Cr from the filtrate. An azo resorcinol 4-(2-pyridylazo) dyestuff was used as a complexing agent. Adsorption isotherms were modelled in an ethanol/acetic acid/water media. The loaded resin is regenerated using 4 M HCl whereby the eluted metal species are recovered. Mere anion exchangers could not uptake metal ions unless they reacted with the dye to form negatively charged complexes. Trivalent chromium showed significant uptake at pH range of 3.5-4.5. Cadmium, zinc and nickel ions formed complex compounds in the alkaline medium. The electrostatic interaction and formation of complex compounds identified the major adsorption mechanisms.

2.4.6 Determination of the equilibrium constant K_d of the solvent extraction process

The equilibrium constant **K_d** value of the separation of residual chromium remaining after pH separation step from Ni-Cr iron-free solution was calculated from the relation of the weight of Cr in the organic and liquid phases [14].

2.4.7 SEM and XRD examinations

Malvern Panalytical's X-ray diffractometer was used to determine the article shape, size, Crystal structure and Phase quantification of the prepared nickel and chromium metals. SEM images were performed using Philips XL30 Scanner electron microscope.

3- Results

Table 2 shows the metals content in the scrap sample.

Table 2. The metals content in the scrap sample

Metal	The acid leachate	Fe-free solution
	Wt. %	Wt. %
Nickel	58.8	77.95
Chromium	16.62	22.01
Iron	24.58	0.02
Other minor		0.01

Figure 3 shows the extent of leaching the spent heating coil materials (HC) using different concentrations of HCl acid and aqua regia as a function of time at room temperature It can bwa seen that the leaching extent increases regularly with time whereby the maximum extent of leaching amounts to 44%.

Figure 3 shows the extent of leaching the spent materials using aqua regia as a function of time at room temperature It can be seen that the leaching extent increases regularly with time whereby complete leaching takes place at $\geq$ 60 minutes. Finding given in Fig. 4 shows that the internal temperature of the leaching process increased due to the exothermic nature of the reaction. Figure 5 shows the effect liquid: solid mass ratioof on the extent of acid leaching of the scrap at room temperature. It is seen that liquid: solid mass ratio becomes significant with $\geq$ 1.15 :1 mass ratio. Figure. 6 shows the effect of reaction temperature in °C on the extent of formation of the different organic salts of Ni and Cr. Ni acetate and citrate are readily obtained as compared to the formation of the same salts with chromium.

Figure. 7 shows the Arrhenius plot of 1/T °K x10-4 against ln weight of formation of the different salts of Ni and Cr. The calculated ΔE value with nickel amounts to 74.16 KJ/ mol and 139.33 KJ/ mol with chromium. Fig 8 shows SEM of Ni and Cr particles as obtained by reducing Ni nitrate with 1 M ascorbic acid.The particle size of nickel amounts to 15-25 nm and about 35 nm with chromium. The global recovery extent amounts to98.6% with nickel and 96.4% with chromium

SEM investigation proved that nickel particles as explored by JCPDS file no 04 -850 are spherical in shape with smaller size typically 10 - 40 nanometers as compared to chromium particles that are octahedral in shape.
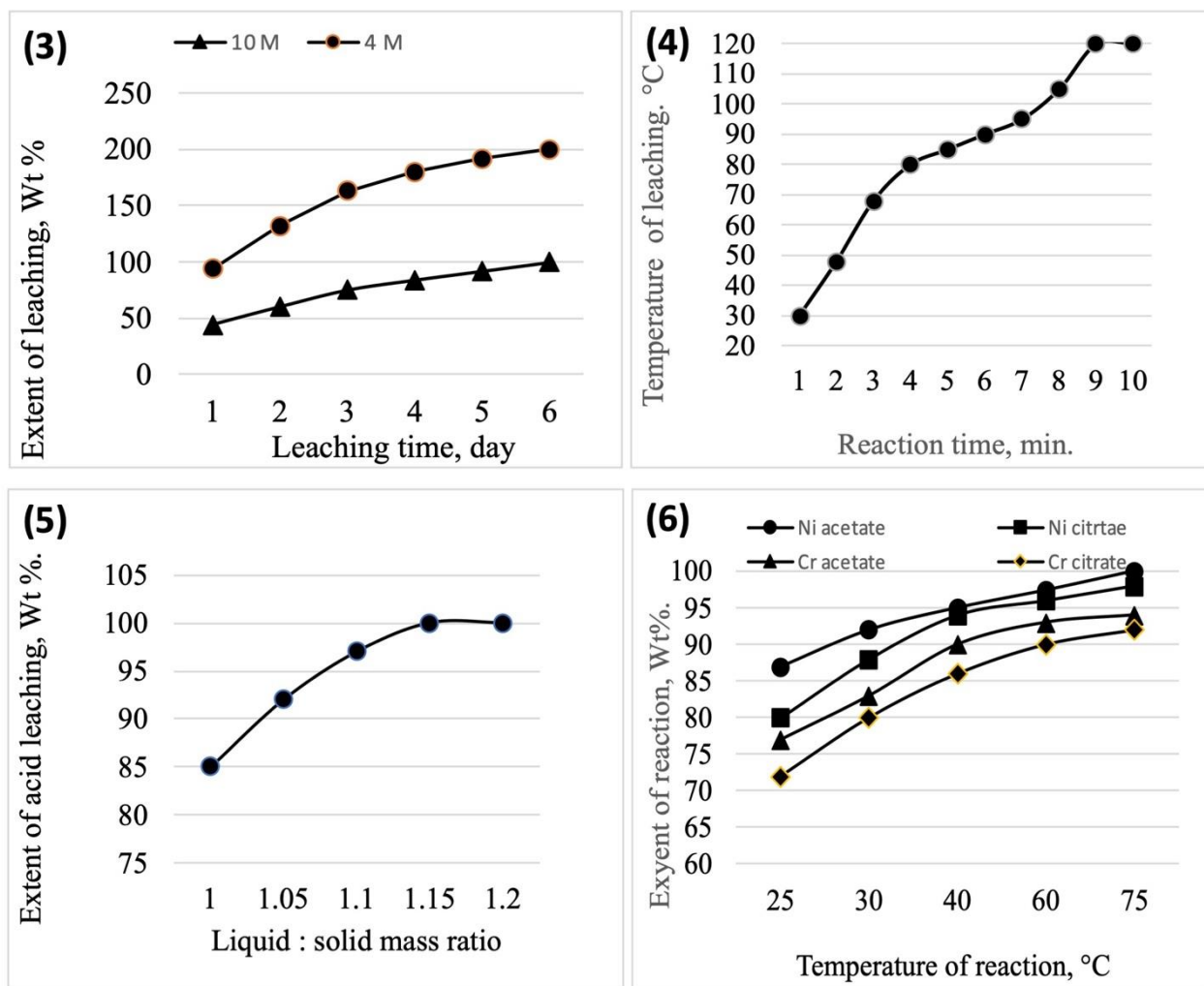
Figure 3-6. 3. Leaching extent of spent HC with 10M and 4M HCl at room temperature **4.** Temperature change of the leaching process during the reaction with aqua regia **5**. the extent of leaching the scrap material with different liquid: solid mass ratio using 10 M HCl at room temperature **6.** Effect of temperature on the extent of formation of the different salts of Ni and Cr.
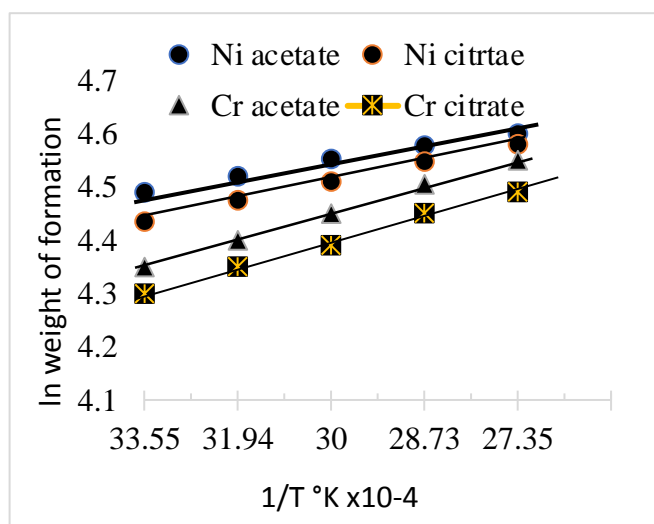

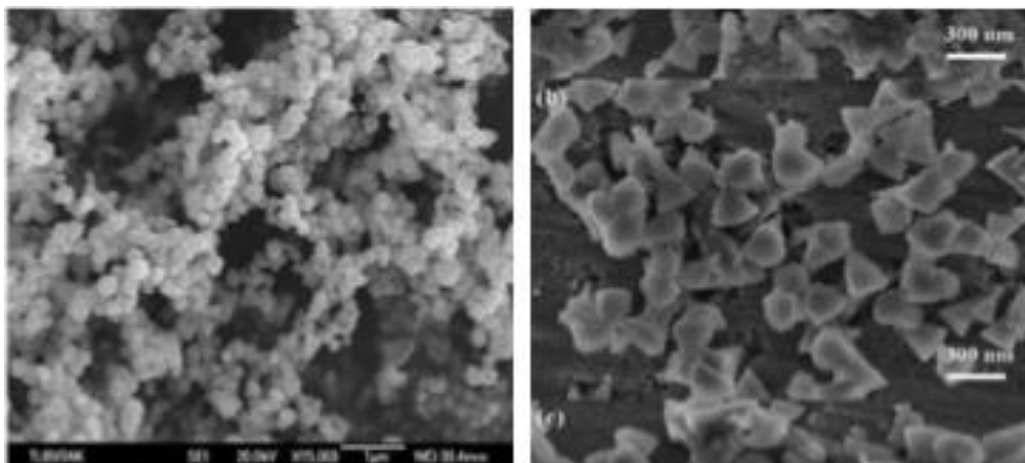**Figure 7.** The Arrhenius plot of the formation of nickel and chromium salts

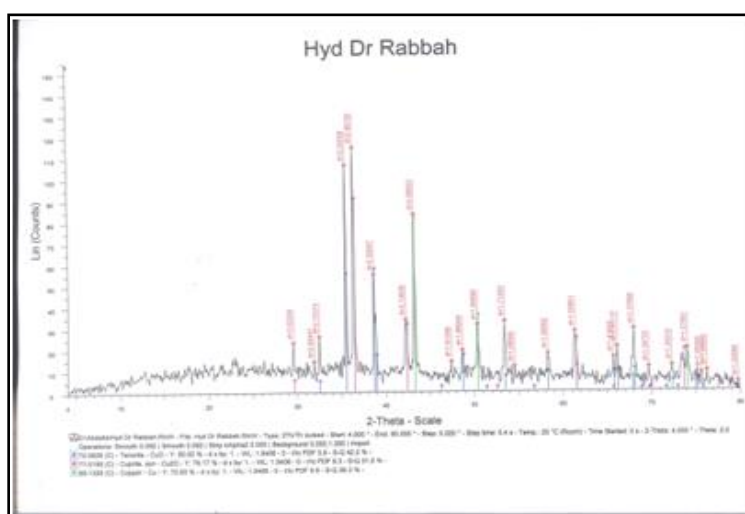**Figure 8.** The SEM images of Bi (JCPDS file no 04 -850) and Cr particles

Figure 9. The XRD pattern of the recovered Ni

In the XRD pattern of the recovered Ni and Cr particles (Fig.9) , the main peaks appeared at 2θ 48, 54 and 75 with Ni. The corresponding peaks with chromium appeared at 2θ of 28, 33, 35, 58 and 74, the XRD pattern indicated that that Ni is crystalline. The global recovery efficiency of nickel (metal + salts) amounts to 98.6 Wt. % and amounts to 96.4 Wt. %. with chromium.

4. Discussion

Nickel-chrome Ni-Cr may be alloyed with iron together with some minor elements used as resistance wire, space heaters, electric furnaces and other similar applications. The alloy shows that chromium is soluble in nickel at a maximum weight pct of 47% at its eutectic temperature and decreases to about 30% at room temperature. Such nickel-chrome alloys have excellent electrical resistance with simultaneous generation of temperature. The alloy has good oxidation, corrosion and wear resistance. The scrap used in this study contains 16.62 wt. pct of chromium as revealed in Table 1.

With repeated heating and cooling-cycles the Ni-Cr spiral is subjected to structural changes and rend brittle. Fig. 2 shows a photograph of the spent Ni-Cr coil as received and after splitting to single rings to make leaching with mineral acids easier Ni and Cr metals are present in the leachate together with iron. The process of recovering Ni and Cr starts by getting rid of iron by conversion to insoluble ferric hydroxide by H_2O_2

$$2\ Fe^{+2} + H_2O_2 + 4\ OH^- \rightarrow 2\ Fe\ (OH)_3 \qquad (1)$$
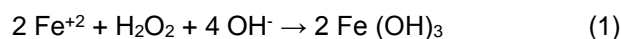
The ppt of ferric hydroxide is filtered and the leachate contains Ni and Cr chloride or nitrate

recovered in nanoparticle size amounting to 15-25 nm and about 35 nm with chromium. Figure 2 represents a sequential process route to achieve the target from the waste material. The leaching step was performed using HCl with different concentrations and with aqua regia. The process of leaching is friendly environment safe with low cost.
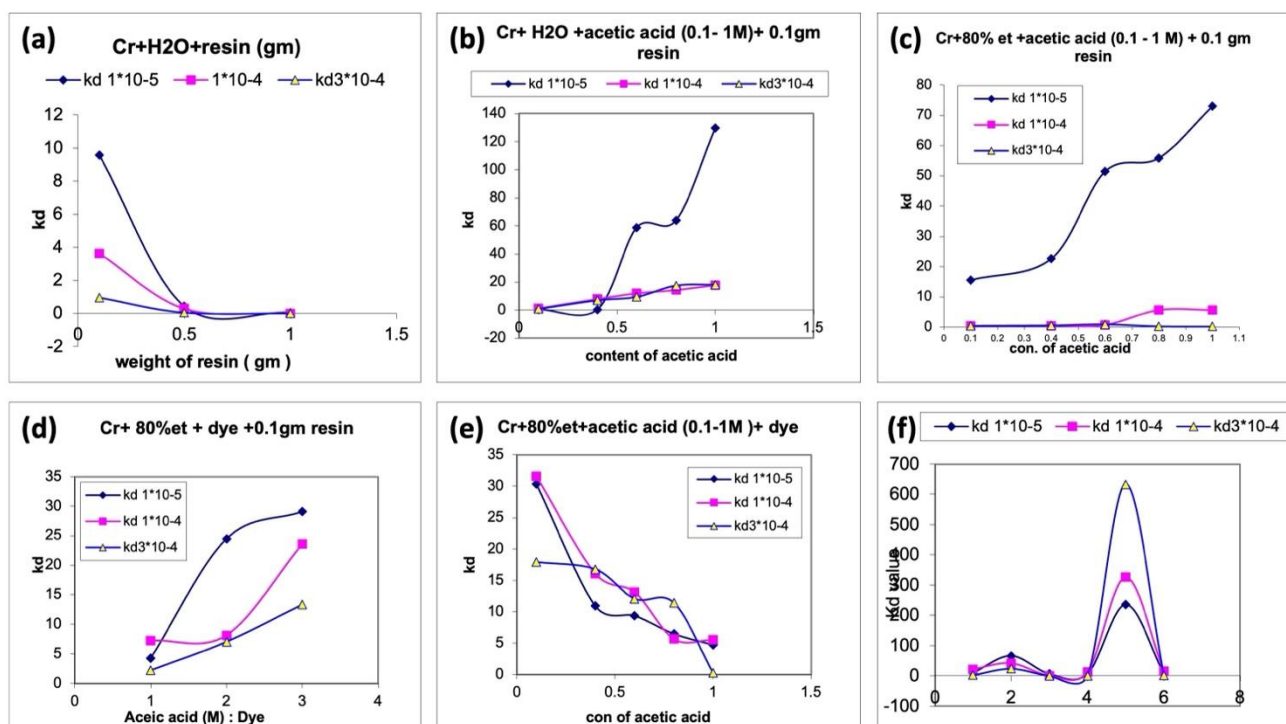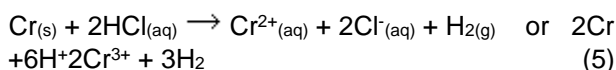
$$Ni + 2HCl \longrightarrow NiCl_2 + H2 \qquad (2)$$

Figure10a. Effect of resin weight on the Kd value of K_d Cr uploading Cr in Cr Acetic +H_2O+resin system **b.** Effect of acetic acid addition on the value of uploading Cr from Cr +H2O + acid + resin system **10c.** Effect of resin weight on the Kd value of Cr uptake from Cr+80% et +acetic acid **10d.** Effect of acetic acid addition on the K_d value of Cr+80%et+.1gm resin/20ml+dye 0.1 - 1 M) + 0.1 gm resin **10e.** Effect of acetic acid concentration on Cr Uptake Cr+80% et +acetic acid (.1-1M)+ from cr+.1gm resin+80%et 80% +. **10f.** Effect of different solvents (ethanol, DMSO, DMF on K_d value of Cr uptake 1 gm resin/20ml dye resin/20ml+dye

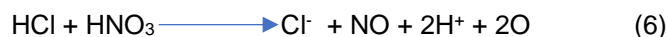It is usually come upon as the green hexahydrate, the formula of which is frequently written NiCl2·6H2O

$$NiCl_2 + 6\ H_2O \longrightarrow NiCl_2 \cdot 6H_2O \qquad (3)$$

and the metal hexahydrate forms metal aquo complex [Ni(H2O)6]$^{2+}$ Dehydration of the NiCl2·6H2O or NiCl$_{x2}$·6H$_2$O gives the yellow anhydrous NiCl$_2$. Dehydration successively causes passivation of the nickel metal surface. The latter phenomenon explains why 10M HCL is less reactive as compared to less concentrated 4M acid as confirmed by the results given in Figure 3b.

The same reaction of HCl takes place with chromium as with nickel provided that Cr(II)Cl2 is formed.

$$Cr_{(s)} + 2HCl_{(aq)} \longrightarrow Cr^{2+}_{(aq)} + 2Cl^-_{(aq)} + H_{2(g)} \quad \text{or} \quad 2Cr$$
$$+6H^+2Cr^{3+} + 3H_2 \qquad (5)$$

Chromium metal dissolves in dilute HCl acid to form Cr)II) ion together with hydrogen gas provided that some metal samples may be resistant to the acid attack.

Leaching the scrapped source was also tested using aqua regia. Results depicted in Figure 4 shows complete acid leaching is scored after 1 hour. The main disadvantage of using this strong corrosive solution is that leaching proceeds vigorously with the evolution of hazardous nitrogen dioxide. The reaction involved the following sequence.

$$HCl + HNO_3 \longrightarrow Cl^- + NO + 2H^+ + 2O \qquad (6)$$

$$M_{(NI/Cr)} + 2O \longrightarrow MO \uparrow \qquad (7)$$

$$NO + O \longrightarrow NO_2 \qquad (8)$$

$$MO + NO_2 \longrightarrow MNO_3 \qquad (9)$$

Where M is for Cr. It is worthy to note that the acid leachate produces metal hydroxide ppt after reaction with ammonia. Careful analysis revealed that some of the chromium is still remaining in a low concentration of about 0.0x mol. Basic carbonate of nickel and chromium can be synthesized from their nitrate salt by reacting with ammonium carbonate.

$$M(NO_3)_2 + (NH_4)_2CO_3 + H_2O \longrightarrow MCO_3.2H_2O + 2\ NH_4NO_3. \quad (10)$$

The study includes determination of the K_d value of chromium uptake as affected by addition of resin, acetic acid, ethanol + acetic acid, ethanol + dye, acetic acid from Cr+. +1gm resin+80%ethanol 80%+dye without resin (Figure. 10 a-f) and using different solvents ; ethanol, dimethyl-sulphoxide DMSO, acetone and dimethyl furane. Results revealed that mere anion exchanger could not uptake Cr metal ions unless they reacted with the dye to form negatively charged complexes. The residual metal was separated by solvent extraction using LEWATIT MP 600 ion exchange resin. Results are graphically represented in Figure 10a through Figure 10f.
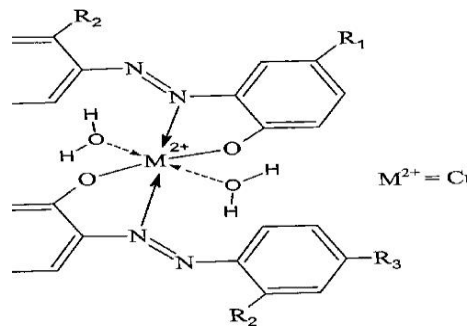
Figure 11. The structure of the Azo-metal complex

Nickel hydroxide   + 2 ascorbic acid   water   Nickel metal   oxidized ascorbic acid

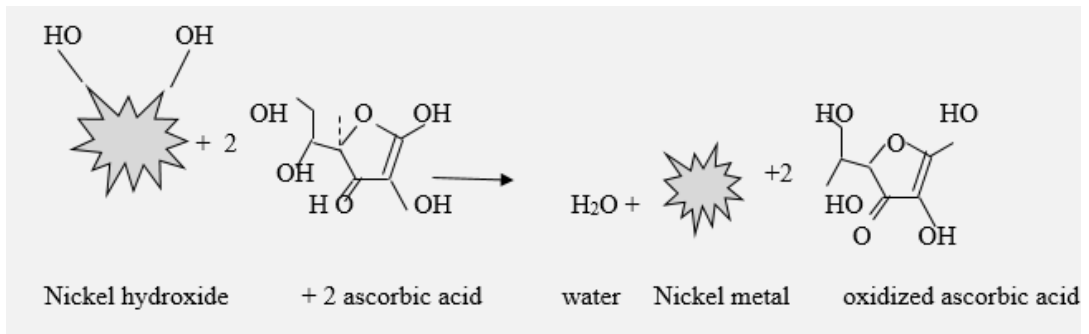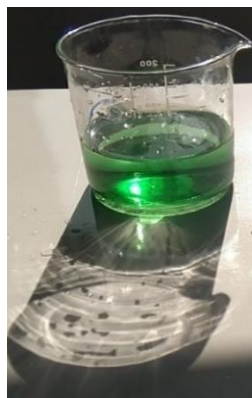**Figure 12.** shows the global recovery efficiency of Ni and Cr recivery extent

Ni/Cr leachate nitrate solution   Ni nitrate solid   Nickel acetate   Ni oxalate

Chromium chloride   Chromium chloride hexahydrate   chromium chloride anhydrous

Figure 13. shows the global recovery efficiency of Ni and Cr recivery extent

Nickel metal present in the heating coil structure alloyed with chromium metal as confirmed with SEM image given in Figure 8.

Preparation of chromium oxalate in solution can form complex, metals such as nickel can be precipitated at the same time. The reactions involved are as follows:
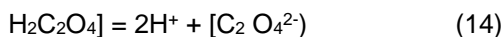
$$Me^{2+} + [C_2]O_4^{2-} = MeC_2O_4 \qquad (11)$$
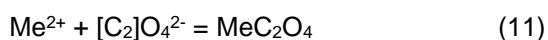
Me is Fe, Ni, Cr in the solution. When the reaction reaches the balance:

$$K_{sp} = [Me^{2+}][C_2]O_4^{2-}] \qquad (12)$$

$$[C_2]O_4^2 = \frac{K^0{}_{SP}}{c(Me^{2+})/C_0} \qquad (13)$$

C (Me^{2+}) is the concentration of metal (Fe, Ni, Cr) in the solution

$C_2O_4^2$ in the solution is determined by the following reaction:

$$H_2C_2O_4] = 2H^+ + [C_2O_4^{2-}) \qquad (14)$$

The ionization constant: $K^0{}_{sp1} = 3.5 \times 10^{-2}$, $K^0{}_{a2} = 4.0 \times 10^{-6}$

So:

$$C(H^+) = \frac{\{K_{al\theta}\,K_{a2\theta}\,C(H_2C_2O_4)/C^0\}^{1/2}}{C(C_2O_4{}^{2-})/C^0} \qquad (15)$$

precipitation initial pH value is calculated as:

$$pH = -\log(C\,H+) \qquad (16)$$

Therefore,

$$p^{H=} -\log)\,\frac{\{K_{al\theta}\,K_{a2\theta}\,C(H_2C_2O_4)/C^0\}^{1/2}}{C(C_2O_4{}^{2-})/C^0} \qquad (17)$$

When $[C_{r2}O_4^2]$ in equation (13) substitutes to equations (14) and (15), the precipitation counterparts at the initial pH value can be calculated by equation (17). According to the initial concentration of the materials in the solution, and applying equation (15), we can calculate the critical pH value for the metal ions of concern. Accordingly, the precipitation pH value for iron in the solution with hydrogen peroxide would be manifested at low pH whereas the other metals such as chromium precipitates at higher pH value of 4.6-6.2 and 10.3 - 11 with nickel. Citric acid proved more aggressive than acetic acid due to higher metal complexation and because the ionization constant of acetic acid is incomplete [15]. The prepared organic salts of nickel and chromium exhibit complex formation of these compounds.

Figure 13 shows photographs of Ni nitrate, citrate and oxalate and chromium chloride solid salts

The different colors of nickel and chromium salts as shown in Figure 13. may be ascribed to the displacement of electrons of the metal ionic configuration from the d orbital to a higher or lower level. Certain wavelengths are absorbed in the process of complex formation so that subtractive color takes place and the coordination complex solution becomes colored [16-18].

5. Conclusion

The outmost findings of this study conclude that spent Ni-Cr heating alloy contains appreciable amounts of nickel and chromium that cannot be discarded without making effort to recover its content of metal values. The suggested process revealed a successful way to achieve the target with no need for sophisticated tools, chemicals or experimental techniques. The method is a friendly environment and simple. It involves breaking of the useless coil to single rings, leaching with mineral acids. Part of the leachate reacts with ascorbic acid in slightly acidic medium to give nickel and chromium nanoparticles. The average particle size of nickel amounts to 15-25 nm and about 35 nm with chromium. The other part of the leachate reacted with ammonia to form hydroxide ppt of nickel and chromium at different pH values. The hydroxide compound reacts with some organic and inorganic acids to form the respective salts. The global recovery extent amounts to 98.6% with nickel and 96.4% with chromium.

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Conflict of interest
The Authors have no conflicts of interest to declare that they are relevant to the content of this article.

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