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PROCESS FOR RECOVERY CHROMIUM FROM ELECTROCHEMICAL MACHINING WASTE BY ACID LEACHING AND CHEMICAL PRECIPTATION

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Abstract
The waste from ECM is hazardous solid wastes due to the presence of specific heavy metals such as chromium, iron, nickel, lead and molybdenum. Hence, recovery of these metals not only saves the environment, but also has the advantages of financial and economic returns. So recovery of chromium from this waste was investigated. Chromium was recovered by using hydrometallurgical method via acid leaching and chemical precipitation. The effect of leaching parameters of time, solution concentration, and leaching temperature were found to be significant. The optimum process operating parameters were established as follows: (7 M) sulfuric acid concentration, (60 min) leaching time, and the temperature was (25 C^o). Leaching solutions at the optimum experiments were used for chemical precipitation, chromium was selectively converted into insoluble component by adding sodium hydroxide {NaOH} at pH (8).

Introduction
Chromium is a metallic material with important properties that has been used since ancient time and it has a significant impact on economic and technological development. Because of chemical properties of chromium, chromium oxide formed protective layer on alloys, and it has high corrosion resistance and hardness so that it is used in the manufacture of stainless steel alloy and in metal plating (electroplating with chromium) [1]. Chromite is the main commercially recoverable known mineral for chromium, although chromium may present in many other minerals in low percentage. Chromite mineral is found in nature as compound of chromic oxide (Cr_2O_3 68%) and ferrous oxide (FeO 32%).In nature; chromite is rarely found in pure form [2, 3]. Pure chromium in the form of chromium metal or ferro-chromium is produced by many methods including; thermal reduction in electric furnace, thermal dissociation, electrolysis, or thermal decomposition. Chromium is used in many metallurgical and chemical industries. Many of these industries, such as; ore processing, stainless steel manufacturing plants, pigments industry, leather tanning, generate a lot of solid and liquid waste containing different proportions of chromium. These industrial wastes are considered as important secondary resources to recover and extract of chromium metal. The continuing need for chromium metal industry makes the recycling of industrial waste and the production of metal a very important task. The process of waste recycling and recovery of metals is not only related to the acquisition of important minerals but also to the conservation of the environment by reducing the percentage of pollutants to the soil, water and air. Industrial wastes containing chromium can be processed recycled and some of which can be used in other industries after treatment such as cement, bricks, glass and paints in addition to environment conservation [4, 5]. One important secondary resource of chromium it was electrochemical machining (ECM). ECM is one of the non- conventional machining processes belonging to electrochemical category. It is called by this term, because electrical energy is used in combination with chemical reaction to accomplish material removal. The process was used for machine of heat resistance and elevated strength alloy. The electrochemical machining process produces waste that is hazardous to the environment and contains minerals that can be recovered, thereby reducing the risk of environmental pollution. One of these metals that can be recovered from the electrochemical machining waste is the chromium metal [6, 7].

Materials and Methods

A. Sample Preparation

Sample to be used in leaching and then in extraction experiments are supposed to be waste left over from the electrochemical machining process to determine the effect of variables of this operation on the results of the extraction operations.

B. Experimental Procedure

The powder obtained from the electrochemical machining waste was leached in acid solution. Leaching solution used for these processes was sulfuric acid (H_2SO_4) CO. (98%).

All leaching experiments carried out in a glass reactor equipped. The leaching reactor was placed on a magnetic heater stirrer. The stirring speed was kept constant at 420 rpm. For each run, Ten grams of waste from electrochemical machining were mixed with 100 ml of sulfuric acid. When the time of the experiments reached the end the slurry was filtered using filtration paper and a sample of the filter is taken for chemical analysis. The experiments were designed by the use of Taguchi approach (Orthogonal Array). Taguchi orthogonal arrays $L9$ (3^3) was established for experiments. The Taguchi design of experiments was done at three variables and three levels: leaching time, 30, 45 and 60 min, concentration, 5, 6 and 7 M and temperature 25, 30, and 40. Each experiment was repeated three times and the average was calculated.

Recover the ions of chromium from leaching solution by chemical precipitation process were carried out by prepared 100 ml of leachate solution. Then Sodium hydroxide (95% pure) of 1 M is prepared and added to the solution from leaching orderly to elevate the pH of the solution to pH (8) and to measure the pH degree using the pH papers.

The precipitant was separated from the solution by vacuum filtration after 1 hour and dried at 110 ^oC for interval of 30 min. chromium oxide (Cr_2O_3) was obtained. It should be reported that the precipitation efficiency has been calculated using the following equation:

$$RY\% = \left[\frac{(C1\,V1) - (C2\,V2)}{(C1\,V1)}\right] 100\%$$

Where:- $RY\%$: Recovery yield, $C1$ &$C2$ are the concentrations of a metal in the solution before and after precipitation in g/l, respectively, V1 & V2 are the volumes of the leach liquor before and after precipitation in L, respectively.

Results and discussion

The values of average leaching efficiency at different levels for the three variables studied are listed in Table 1. The corresponding efficiency of the three variables and their levels is described in Fig 1.

Table 1 the Leaching efficiency of Chromium in H_2SO_4 solution.

No.	Conc. (M)	Time (min)	Temp. (^oC)	LE 1 %	LE 2 %	LE 3 %	Mean %
1	5	30	25	32.5	77.7	51.2	53.80
2	5	45	30	29.4	30.8	26.8	29.00
3	5	60	40	44.6	27.3	25.9	32.60
4	6	30	30	64.2	62.4	83.1	69.90
5	6	45	40	90.5	75.7	75.1	80.43
6	6	60	25	77.5	78.7	84.9	80.36
7	7	30	40	69.4	54.3	91.8	68.83
8	7	45	25	74.5	75.4	92.5	80.80
9	7	60	30	73.4	82.2	90.5	81.93

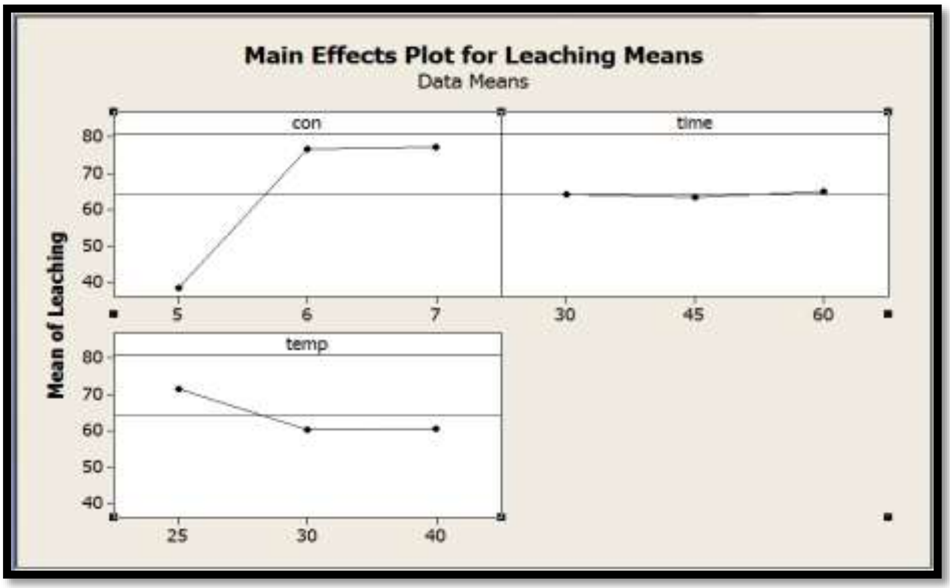
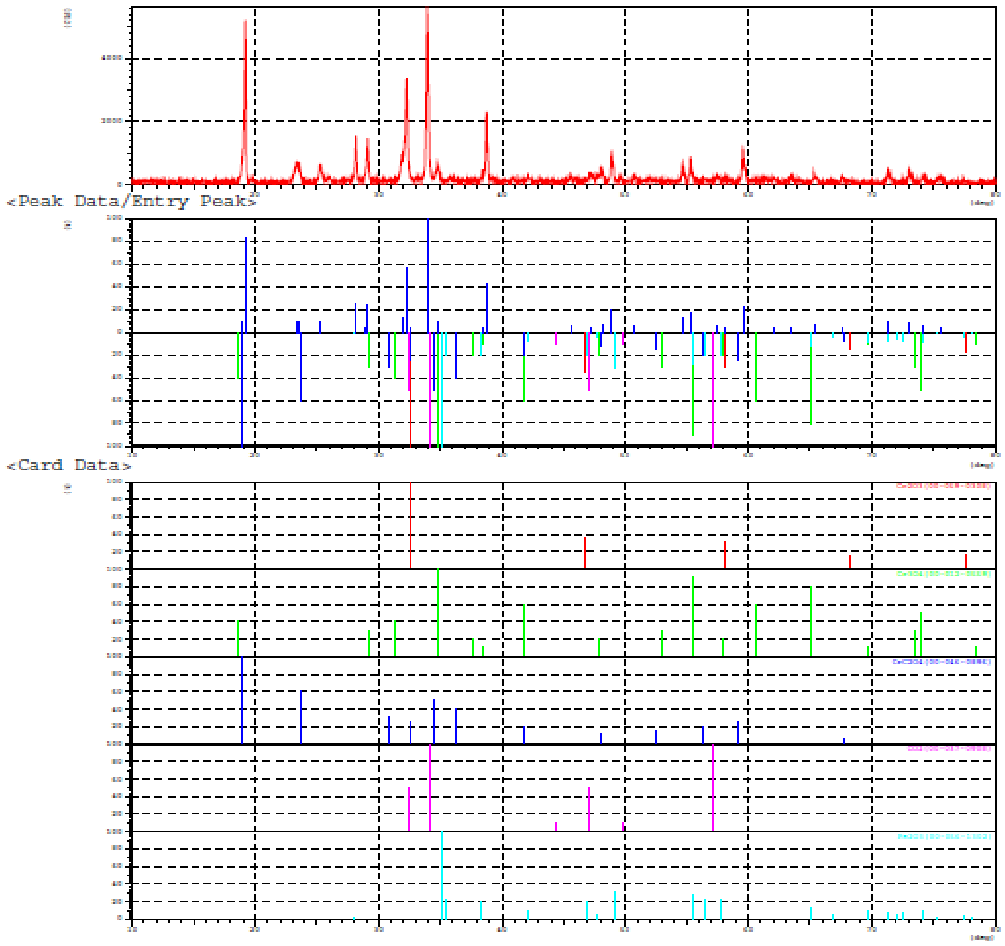


Figure 1 The effect of leaching factors on the leaching efficiency of Chromium.

According to the data of Taguchi design experiments, the solution concentration has the largest effect on the leaching process. It was found that the 7 M concentration is the best for leaching vanadium to 81%. With continues increasing temperature, the leaching efficiency decreases. This may be due to the reaction of waste with H_2SO_4 is exothermal reaction, which means that the reaction creates heat, since with increasing the temperature of exothermal reaction, the solubility decreases [8].
Time has little effect on the leaching efficiency of chromium among the three controlled factors. . Leaching time, as shown in Fig 1 curve, was unstable. The leaching efficiency of chromium decreases when the time was increased until arrive 45 C^o the leaching efficiency will increase. This is because of consumption of the reactant. When the concentration of reactant decreases, the collision among chromium molecule and H+ will be fewer.

According to the above points, it seems that leach time of (60 min) is sufficient in the acid leach process
After determining the optimum conditions for the leaching process, leaching solution resulted from the optimum experiment is used to recover chromium by chemical precipitation as chromium oxide Cr_2O_3. Sodium hydroxide was used to precipitate chromium by adding the proper amount of solution to raise pH to 8. The quantities of the leachate used in precipitate experiments were (100ml). Chromium in the leachate is converted selectively to soluble components of chromium oxide (Cr_2O_3) by adding sodium hydro oxide (NaOH).

After 1 hr, the precipitate is filtered and dried in oven for one hour in 110 C^o. 98.9% of chromium precipitated as chromium oxide indicated by XRD analysis in (Fig. 2).



No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L	d	I	R
				Dx	WT%	S.G.	
1	00-059-0308	Cr2O3 Chromium Oxide	0.072	0.600(3/ 5) 3.05	0.688	0.576 Fm-3m	0.238
2	00-012-0559	Cr3O4 Chromium Oxide	0.196	0.588(10/17) 5.13	0.594	0.564 I41/amd	0.197
3	00-046-0896	CrC2O4 Chromium Oxalate	0.229	0.583(7/12) -----	0.657	0.472 -------	0.181
4	00-037-0908	CO2 Carbon Oxide (Unnamed mineral (NR))	0.377	0.667(4/ 6) -----	0.658	0.289 -------	0.127
5	00-056-1302	Fe2O3 Iron Oxide	0.329	0.545(12/34) 7.37	0.559	0.365 Cmcm	0.111

Figure 2 X-ray Analyses of the precipitant.

Conclusion

It is clear from the experiments that chromium can be easily leached from ECM waste by using H_2SO_4. The results show that, working at 7 M, temperature 25 C°, time 60 min, stirring speed 420, the extraction efficiency of chromium attained about 81%.

Recovery of pure Cr2O3 from the resultant leach liquor is done to establish a good separation method of chromium oxide from electrochemical machining waste.

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