

Electrochemical Regeneration of Chromium Containing Solution from Metal Finishing Industry

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The electrochemical regeneration of chromium containing solution obtained from metal finishing industry was carried out using different anode materials (lead & platinized titanium) and cathode materials (lead and stainless steel). Effect of different parameters like anode current density, catholyte concentration, temperature, and anolyte flow rate on current efficiency was studied. At current densities of 2–2.5 A/dm² using lead as anode at temperature of 303–313 K and anolyte flow rate of 130 – 300 cm³/min, the current efficiency of 68 – 76% with an energy consumption of 3.0 to 3.2 kWh/kg CrO₃ was achieved.

Introduction

Chromium plating is a major electroplating process not only for decorative applications but also for wears resistance, such as the hard chromium plating of hydrolytic rams and in automobile engines. In the chromium plating industry, apart from the plating bath, chromic acid is used in pickling and electropolishing. The disposal of such baths is problematic and the recycling and regeneration is always preferable from the point of view economy and environment. The effluent treatment processes are based on the chemical, biological and electrochemical systems. It has been well-established that electrochemistry has much to offer to the prevention of pollution and protection of environment.^{1–26} The regeneration of spent chromium plating solution is an industrially important process that has been given much attention in the past. It is well-known that, the Cr³⁺ concentration builds up from 9 to 15 g l⁻¹ in the electrolyte during the process of chromium plating, resulting, the quality of the plating deteriorates. In order to maintain the Cr³⁺ concentration (in the range of 9–9.5 g l⁻¹) in the electrolyte, installation of regeneration loop in the process scheme is necessary. It is reported that^{27–37} the oxidation of Cr³⁺ to Cr⁶⁺ is carried out in a two-compartment cell with lead electrodes. The anolyte and catholyte are separated by a diaphragm to prevent cathodic reduction of Cr⁶⁺. Diaphragms used are ceramic (porous pot) or blue asbestos. The asbestos has been fitted either cloth separators or bags or asbestos ropes wound over the cathode. However, Cr³⁺ oxidation at the Pb anode was not as efficient in their experiments and the ceramic separators used were sometimes clogged probably by metal hydroxides resulting in high cell voltage. It is also reported that the regeneration of spent chromium plating solutions by low-cost chemical method,³⁸ by membrane technology^{39–41} and by electrodialysis.⁴² More recently, studies were focused on the removal of metallic impurities with simultaneous regeneration of spent plating solutions by ceramic membranes⁴³ and not much work was done with Nafion as the separator material. Therefore, the main objective of this study is to regenerate Cr⁶⁺ from spent chromium plating baths using Nafion as separator material and to optimize the parametric conditions, the cells were operated at different anode current densities, catholyte concentration, flow rate, temperature and electrode materials.

Materials and Methods

Cell Design and Electrolysis. Electrochemical regeneration of hexavalent chromium was carried out in a two-compartment flow cell using Nafion 324 (Dupont, USA) as separator. On either side of the Nafion membrane a PVC flow distributor was placed and held leak-proof with suitable polythene gaskets. Lead (Commercial grade, Lead (India) Ltd, India) and stainless steel (SS304; SAIL, India)/lead is used as anode and cathode respectively. The electrodes [(0.15 m (l) × 0.10 (b))] were positioned in between the flow distributor and the PVC back plates. PVC nozzles for inlet and outlet ports were fitted through the end plates and the electrodes on either side. The total dimension of the cell was 0.20 m × 0.15 m × 0.025 m. The flow of anolyte and catholyte were maintained at desired rate with the help of peristaltic pumps (pp 20, miclines, India), from separate reservoirs. While the catholyte (10% H₂ SO₄) (Ranbaxy, India) was re-circulated from the reservoir through the catholyte compartment, the anolyte passed only once through the cell. Figure 1 shows a sketch of the flow cell. Solutions obtained from Metal finishing industry and synthetic solution were the anolytes used for the study. Composition of the plating bath is shown in Table 1. At the commencement of each experiment the lead anode was cleaned thoroughly by emerying and dipping in 10% nitric acid (Ranbaxy, India) polarized anodically for 30 min at 2.5 A.dm⁻², by a rectifier (5A, 0-10V, Aplab Model), in the Cr(VI) – Cr(III) – H₂ SO₄ solution to obtain an initial coating of lead dioxide. Current efficiency and energy consumption were calculated as a function of experimental variations, such as anode material, current density, flow rate, temperature, etc. Regulated direct current was supplied from a rectifier (25A, 0-25V, Aplab Model).

Analysis

Chromic Acid. Chromic acid was estimated iodometrically, using 0.1 N sodium thiosulfate (analar grade, Ranbaxy, India) and starch (analar grade, Ranbaxy, India) as an indicator.⁴⁴

Chromium (Cr³⁺). Chromium(III) ion (Cr³⁺) was oxidized by sodium peroxide to chromate (which contained hexavalent chromium (Cr⁶⁺)) and the total chromate was determined by iodometrically, using 0.1 N sodium thiosulfate (analar grade, Ranbaxy, India) and starch (analar grade, Ranbaxy, India) as an indicator.⁴⁴ The concentration of chromium (Cr³⁺) was calculated by difference between the total Cr⁶⁺ and the unoxidized Cr⁶⁺ in the solution.

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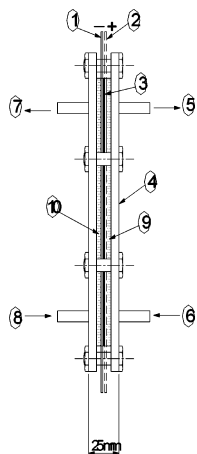


Figure 1. Sketch of the flow cells. Legend: (1) cathode, (2) anode, (3) membrane, (4) end plate, (5) anolyte outlet, (6) anolyte inlet, (7) catholyte outlet, (8) catholyte inlet, (9) anolyte, and (10) catholyte.

Table 1. Composition of the Spent Chromium Plating Bath

element	concentration (g/L)
Cr ⁶⁺ (as CrO ₃)	243.0
Cr ³⁺	14.4
sulfate (as SO ₄ ²⁻)	6.75
iron (as Fe ₂ O ₃)	67.5

Sulfate. Sulfate was estimated via gravimetric methods (by precipitation as barium sulfate), using 10% barium chloride (analar grade, BDH, India).⁴⁴

Iron. The iron content in the bath was determined by precipitating it as ferric hydroxide under oxidizing (using 5 cm³ of 30% H₂O₂ (analar grade, Ranbaxy, India) and alkaline (using 3 g of KOH (analar grade, Ranbaxy, India) conditions and converting the hydroxide to Fe₂O₃ by igniting the hydroxide.⁴⁴

Results and Discussions

Effect of Anode Current Density. Table 2 shows the effect of anode current density on the oxidation of chromium(III) to chromium(VI). The result shows that lower current densities, on the order of 1–2.5 A/dm² are the most optimum. Higher current densities result in the evolution of oxygen (as seen from the vigorous evolution of gas bubbles from the anode), which reduces the anode efficiency. This is because, at higher current

densities, the oxidation potential of chromium(III) and that of oxygen evolution become closer to each other, especially in a low chromium(III) electrolyte. The increase in current density also increases the energy consumption. Because of the fact that operation at a lower current density (such as 1.0 A/dm²) will require anodes and cells of larger size and volume, and the variation of efficiency is not as wide in the range of 1–2.5 A/dm², a current density of 2.5 A/dm² was chosen as being optimum for the remaining experiments.

Effect of Catholyte Concentration. The effect of concentration of catholyte was evaluated, and the results, as shown in Table 3, indicate that the current efficiency increases from 76% with 10% H₂SO₄ to 80% with 30% H₂SO₄, and the voltage also is reduced very slightly. Although this is a favorable situation, if the concentration of acid in the catholyte is very much higher than that in the anolyte, then the transport of water from the anolyte to the catholyte may occur because of osmotic flow. Moreover, the handling of concentrated acid may become difficult. Second, higher catholyte acid concentrations may damage the Nafion membrane. In view of this possibility, further experiments were conducted with only 10% H₂SO₄ as the catholyte.

Effect of Flow Rate of Anolyte. Table 4 shows the effect of the flow rate of the anolyte on the current efficiency. The table shows that the current efficiency is low at lower flow rates and increases gradually as the flow rate increases. Above a flow rate of 300 cm³/min, the efficiency is almost constant. The lower efficiency at flow rates of <300 cm³/min must be due to the inadequate supply of chromium(III) that might have caused oxygen evolution. This is also evidenced from the decreasing cell voltage as the flow rate is increased.

Effect of Temperature. The effect of temperature on the current efficiency for the oxidation of chromium(III) in the chromium plating solution is shown in Table 5. From the results, it is observed that, as the temperature increases, the current efficiency decreases. Operation at higher temperature reduces the oxygen evolution overvoltage, whereas that for chromium(III) oxidation seems to be less affected. When these potentials approach each other, the oxygen evolution rate increases and, thus, an increase in temperature reduces the chromium(III) oxidation rate. The operation at higher temperature reduces the cell voltage. The current efficiency for the oxidation of chromium(III) is dependent on the catalytic nature and the

Table 2. Effect of Anode Current Density on the Oxidation of Cr(III) to Cr(VI)^a

sample	anode current density (A/dm ²)	cell voltage (V)	Final Concentration (±1 g/L)		current efficiency (%)	energy consumption (kW/kg CrO ₃)
			Cr ⁶⁺ (as CrO ₃)	Cr ³⁺		
1	1.0	2.92	251.0	10.4	74	3.29
2	2.0	3.02	250.6	10.2	71	3.24
3	2.5	3.08	253.0	9.2	76	3.57
4	3.0	3.11	250.2	10.6	67	3.76
5	4.0	3.30	248.0	11.8	47	5.74
6	5.0	3.35	247.4	12.1	41	6.62

^a Conditions: anolyte, Cr³⁺, 14.4 g/L, and Cr⁶⁺, 243.0 g/L; catholyte, 10% H₂SO₄; separator, Nafion 324; temperature, 303 K; anode and cathode, lead; cathode current density, 2.5 A/dm²; flow rate, 300 cm³/min; duration, 5 h; current passed, 3.75 A.

Table 3. Effect of Catholyte Concentration on the Oxidation of Chromium(III) to Chromium(VI)^a

sample	catholyte concentration	voltage (V)	Final Concentration (±1 g/L)		current efficiency (%)
			Cr ⁶⁺	Cr ³⁺	
1	10% H ₂ SO ₄	3.08	253	9.2	76
2	20% H ₂ SO ₄	3.02	254.6	8.3	78
3	30% H ₂ SO ₄	3.02	255	8.2	80

^a Conditions: anolyte, Cr³⁺, 14.4 g/L, and Cr⁶⁺, 243 g/L; separator, Nafion 324; temperature, 303 K; anode and cathode, lead; current density, 2.5 A/dm²; flow rate, 300 cm³/min; duration, 5 h; current passed, 3.75 A.

Table 4. Effect of Anolyte Flow Rate of the Anolyte on the Oxidation of Chromium(III) to Chromium(IV)^a

sample	anolyte flow rate (cm ³ /min)	cell voltage (V)	Final Concentration (±1 g/L)		current efficiency (%)
			Cr ⁶⁺	Cr ³⁺	
1	65	3.07	259	6.0	30
2	95	3.05	260	5.6	44
3	150	3.04	260.2	5.5	68
4	250	3.03	254	8.7	70
5	300	3.08	253	9.2	76
6	600	3.02	248	12.0	77

^a Conditions: anolyte, Cr³⁺, 14.4 g/L, and Cr⁴⁺, 243 g/L; catholyte, 10% H₂SO₄; separator, Nafion 324; temperature, 303 K; anode and cathode, lead; current density, 2.5 A/dm²; duration, 5 h; current passed, 3.75 A.

Table 5. Effect of Temperature on the Oxidation of Chromium(III) to Chromium(IV)

sample	temperature (K)	cell voltage (V)	Final Concentration (±1 g/L)		current efficiency (%)
			Cr ⁴⁺	Cr ³⁺	
1	303 ± 1	3.08	253	9.2	76
2	318 ± 1	2.92	254	8.9	71
3	333 ± 1	2.80	253	9.0	69.5

^a Conditions: anolyte, Cr³⁺, 14.4 g/L, and Cr⁴⁺, 243 g/L; catholyte, 10% H₂SO₄; separator, Nafion 324; anode and cathode, lead; current density, 2.5 A/dm²; flow rate, 300 cm³/min; duration, 5 h; current passed, 3.75 A.

Table 6. Effect of Electrode Materials on the Oxidation of Chromium(III) to Chromium(VI)^a

sample	anode	cathode	cell voltage (V)	Final Concentration (±1 g/L)		current efficiency (%)
				Cr ⁶⁺	Cr ³⁺	
1	lead	lead	3.08	253	9.2	76
2	platinized titanium	lead	3.00	257.1	10.2	62
3	platinized titanium (Pb ions added into the electrolyte)	lead	3.05	251.3	10.0	64
4	lead	stainless steel	2.98	255	8.2	82

^a Conditions: anolyte, Cr³⁺, 14.4 g/L, and Cr⁴⁺, 243 g/L; catholyte, 10% H₂SO₄; separator, Nafion 324; temperature, 303 K; current density, 2.5 A/dm²; flow rate, 300 cm³/min; duration, 5 h.

history of the lead dioxide coating that is formed on the lead anode during the initial period of the electrolysis. The structure of the lead dioxide coating is determined by the operating current density and the temperature of formation.⁴⁵ Probably, the low anode current density and room temperature under which the experiment was performed may be responsible for the decrease in the current efficiency with increasing temperature. For optimum conversion, a temperature of 303–313 K may be used, which is an expedient temperature for industrial application.

Effect of Anode and Cathode Material. Table 6 shows the effect of anode and cathode material on the oxidation of chromium(III) to chromium(VI). From the table, one can see that the lead anode and lead cathode exhibit a current efficiency of ~76% and the lead anode and stainless steel cathode exhibit a current efficiency of ~82%. The oxidation of chromium(III) requires an anode potential of more than 1.8 V, and the anode materials that can sustain such higher potentials are limited to lead dioxide and platinum.^{34,45} While using platinum as the anode and lead as the cathode, the current efficiency obtained is 62%. Improved efficiency (64%) is achieved by the addition of Pb ions into the solution. This may be due to the formation of lead dioxide over the surface of the anode. A current efficiency of ~82% is achieved when lead and stainless steel were used as the anode and cathode, respectively. The table also shows that, while using a stainless steel cathode, the cell voltage is slightly less (0.1 V) than that of lead cathode. The cathodic reaction in the regeneration cell is hydrogen evolution and the reduction of chromium(VI) is a secondary reaction that is dependent on the cathode material and the operating current density. The hydrogen evolution efficiency of the cathode can be increased using materials that exhibit a lower hydrogen overpotential than lead. This has been determined to be true when stainless steel is used as the cathode.

Conclusion

The electrochemical oxidation of chromium(III) from the plating bath has been performed. The following conclusions have been inferred from the investigations:

- (1) Lead anodes show good current efficiency, compared to bare platinum anodes.
- (2) If platinum anodes must be used, then a coating of lead dioxide must be formed on them prior to installation in a chromium(III) oxidation cell.
- (3) Stainless steel cathodes show better efficiency than lead cathodes.
- (4) The optimum parameters for the oxidation of chromium(III) are a current density of 1–2.5 A/dm², a temperature of 303–313 K; and an anolyte flow rate of 300 cm³/min.

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