# Removal of Chromium from Tanning Wastewater by Chemical Precipitation and Electrocoagulation

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## Abstract

Chromium, used in the form of basic chromium sulfate (as $Cr_2O_3$) for processing of hides, has well-known adverse effects when improperly disposed of in the environment, because it is highly toxic, highly persistent in the environment and not biodegradable. Tanneries spend large amounts of water for processing of hides; hence, chromium recovery from tanning wastewater is an environmentally friendly and economically viable alternative, as it can prevent a greater amount of chromium-containing sludge from being disposed of in industrial hazardous waste landfills. Compliance with environmental legislation also poses a challenge to the leather industry, as the parameters for discharging treated wastewater into receiving water bodies are increasingly stringent, which encourages the practice of techniques for recovery and reuse of chemical inputs in the tanning of hides. This study focuses on the removal of the chromium present in tanning wastewater through chemical precipitation and electrocoagulation. In both methods, chromium is separated in the form of an insoluble precipitate either by the addition of alkali or by the oxidation and reduction of metal anodes. Chemical precipitation resulted in 99.74% of removal efficiency, while in electrocoagulation with aluminum, copper and iron electrodes, removal efficiency was 97.76%, 69.91% and 90.27%, respectively. After these processes, chromium may be used again as a tanning agent in leather processing.
**Keywords:** chrome, tanning, chemical precipitation, electrocoagulation.

## 1. Introduction

Tanning of hides is a very important economic activity in Rio Grande do Sul and in Brazil. The leather industry has been giving increasing attention to the outcomes of such activity and undertaking efforts to treat wastewater and properly dispose of the sludge and wastewater which are generated in the tanning process (Mella *et al.* 2012). Some of the main characteristics of the effluents generated in tanneries are high biological oxygen demand (BOD), chemical oxygen demand (COD) and high concentrations of suspended solids, organic nitrogen, sulfide and chromium in the effluents resulting from tanning. Most of these compounds are removed when efficient processes are implemented in wastewater treatment plants.

Nowadays, there is great concern about the level of treatment and the final destination of waste, and also about the consequences on the environment as regards the quality of receiving waters. Taking these aspects into account, special care should be taken with studies, criteria and projects focused on the treatment and disposal of effluents in order to ensure adequate disposal of effluents as well as maintain and improve the quality and use of receiving bodies (Jordan *et al*. 2011).

In Brazil, there are nationwide regulatory standards. CONAMA Resolution no. 357/2005 establishes standards for discharging effluents into Brazilian water bodies and determines that the release of these effluents do not modify the original character of the receiving body, i.e., there can be no visual change. At the state level, CONSEMA Resolution no. 128/06 sets emission standards for wastewater emission sources to discharge their effluents into surface waters in the state of Rio Grande do Sul. In May 2011, CONAMA Resolution no. 430 came into force and determined patterns of discharge of effluents containing hexavalent chromium and trivalent of 0.1 mg $Cr^{+6}$. $L^{-1}$ and 1.0 mg $Cr^{+3}$. $L^{-1}$, respectively.

CONSEMA Resolution no. 129 provides for emission standards for toxicity of effluents released to surface waters of the state of Rio Grande do Sul, where analyses have to be made of effluent toxicity tests with organisms of three (3) different trophic levels. These tests will enable the identification of possible toxic substances that pose threats to public health and the environment.

Generally, the chromium used in leather processing is in the form of basic chromium sulfate ($CrOHSO_4$) and it is analyzed in the form of chromium oxide ($Cr_2O_3$). In a study by Aquim (2009), 9 tanning tests with whole hides on an industrial scale gave a mean value of 3.19 ± 0.71 g/L $Cr_2O_3$ in residual baths. Considering this level of chromium in the baths, it is crucial to develop appropriate techniques that aim to recover and recycle the chromium present in wastewater tanning baths.

Many methods are used to remove heavy metals from wastewater e.g., chemical precipitation, ion exchange, adsorption, reverse osmosis, coagulation-flocculation, electrocoagulation, flotation, membrane filtration, etc. (Fu *et al.* 2011; Kurniawan *et al.* 2006).

Chemical precipitation is widely used for heavy metal removal in inorganic wastewater (Kurniawan *et al.* 2006). After pH adjustment to basic conditions, the dissolved metal ions are converted to an insoluble solid through a chemical reaction by adding alkali. Generally, the metal (M) precipitates as a hydroxide as shown in Equation (1):

$$M^{2+} + 2(OH)^- \leftrightarrow M(OH)_2 \qquad (1)$$

In the case of chromium, it precipitates as chromium hydroxide (III). After sedimentation, and after being redissolved with strong acid under controlled addition, such chrome can be used again as input in the tanning process.

Electrocoagulation occurs when the sacrificial anode undergoes oxidation, releasing metal ions, and the cathode is reduced, with hydroxyl ions being formed by hydrolysis of water. Metal ions combine with the hydroxyl ions to form metal hydroxide compounds that favor the formation of flocs by destabilizing suspended particles. The resulting flocs can be separated from the liquid by flotation or sedimentation depending on the density thereof (Bensadok *et al.* 2007).

To release the coagulating agent, iron ions and hydroxyl radicals, a potential difference has to be applied to the electrodes. As the sacrificial anode corrodes, the active cation is released to the solution (Wimmer 2007).

Using iron as constituent material of the electrode, the following reactions occur, according to Equations (2) to (7):

Oxidation of Iron:

$$Fe \rightarrow Fe^{+2} + 2e^{-} \quad (2)$$

Anode:

$$2Fe^{+2} + 5H_2O + 1/2O_2^{-} \rightarrow 2Fe(OH)_3 + 4H^{+} \quad (3)$$

$$Fe^{+2} + 2OH^{-} \rightarrow Fe\,(OH)_2 \quad (4)$$

Cathode:

$$2H_2O + 2e^{-} \rightarrow 2OH^{-} + H_2 \quad (5)$$

Overall Reaction:

$$2Fe + 5H_2O + 1/2O_2 \rightarrow 2Fe(OH)_3 + 2H_2 \quad (6)$$

$$Fe + 2H_2O \rightarrow Fe\,(OH)_2 + H_2 \quad (7)$$

Electrocoagulation is an alternative to conventional chemical precipitation, because it has shown satisfactory results for color reduction, BOD, COD, oil and total chromium tannery effluents (Feng *et al.* 2007; Babu *et al.* 2007).

Electrocoagulation may be considered as an alternative treatment because it works without the addition of chemicals, has proved promising in the treatment of effluents from various industries e.g., tannery effluents (Feng *et al.* 2007; Sirajuddin *et al*. 2007; Fornari *et al.* 2009; Sengil *et al.* 2009; Benhadji *et al*. 2011; Módenes *et al.* 2012), effluents from the textile industry (Palace *et al.* 2009; Zongo *et al.* 2009), effluents from the metal industry (Akbal *et al.* 2011), removal of heavy metals (Aji *et al.* 2012), removal of COD in the treatment of alcohol distillery wastewater (Yavuz 2007); discoloration of dyes (Daneshvar *et al.* 2003).

Electrocoagulation has a number of advantages over conventional chemical precipitation because it is a simple operating process where the dosing of chemicals is not necessary, and it can remove colloidal particles more effectively (Benhadji *et al.* 2011).

Water reuse in tanning not only saves clean water but also proposes reusing a wastewater that would be handled and disposed of to the environment; thus the use of this input is maximized. The proposed direct reuse of chrome baths is even more important because chromium is a chemical of great environmental impact (Aquim 2009).

The system of an industrial recycling plant of chromium contained in tanning baths, to be used in the pickle, was tested in a tannery that processed 500 hides/day. The chromium present in the baths was precipitated as hydroxide and diluted as sulfuric acid and then reused in the pickling bath. This system saves chromium sulfate, sodium chloride, water and sodium formate (Salazar, 2008). The technology has met the requirements of the process e.g., smooth and clear color, no blemishes and concentration of oil and grease into the leather below 0.5%.

Chromium is an essential element in human nutrition. The deficiency of this element causes ingestion disorders associated with glucose metabolism as this acts as a potentiator of insulin. On the other hand, ingesting higher doses than those recommended may cause intoxication and a series of diseases (Silva *et al.* 2001).

In a previous study, Mella *et al.* (2012) visited four tanneries in the State of Rio Grande do Sul in order to analyze, characterize and compare the unit operations involved in wastewater treatment. They found that the tanneries had attempted to deploy clean technologies in order to reduce their water demand and, thus, the volume of effluents to be treated before disposal. As for the unit operations involved in wastewater treatment plants (WWTPs), the processes employed in wastewater treatment in tanneries were similar (there were physical-chemical and biological treatments, each followed by sludge sedimentation). Sludge samples from two tanneries resulting from the treatment of leather tanning baths had values of chromium oxide content of 11.58% and 28.48%, in which case the tanneries segregated the effluents in the tanning step. In comparison, for the other two tanneries with combined treatment of all effluent streams, the values of chromium oxide content for the mixed sludge were 1.98% and 3.62%.

The objective of this study is to evaluate the physicochemical removal of chromium present in wastewater tanning baths by adding sodium hydroxide (12M) and conducting electrocoagulation tests through oxidation and reduction of metallic anodes, where copper plates, aluminum and iron were used.

## 2. Materials and Methods

### 2.1. Wastewater Sample

The tanning liquor was collected directly from the tanning drum (without any pretreatment) of a tannery that basically performs beamhouse and tanning operations, a couple of post-tanning procedures until leather finish. The tannery can process 500 hides/day, generating a total average effluent flow of 200 $m^3$/day and a flow rate of 60 $m^3$/day of chromium-containing wastewater.

The wastewater was characterized by the analysis of the following parameters: pH, electrical conductivity, temperature, BOD, COD, total chromium, suspended solids, turbidity and sulfides, as shown in Table 1:

Table 1: Characterization of the tanning liquor.

| Parameter | Unit | Value |
|---|---|---|
| **pH** | | 4.07 |
| **Temperature** | $^{o}C$ | 20.00 |
| **BOD** | mg $O_2$/L | 116.20 |
| **COD** | mg $O_2$/L | 387.20 |
| **Conductivity** | mS/cm | 103.30 |
| **Total Chromium** | mg Cr/L | 2000.00 |
| **Turbidity** | NTU | 14.56 |
| **Suspended Solids** | mg/L | 456.00 |
| **Sulfides** | mg/L | 0.05 |

Figure 1: Raw Effluent

The effluent showed a strong blue color, which is typical of the presence of chromium, as can be seen in Figure 1.

### 2.2. Chemical precipitation

The alkali agent used was sodium hydroxide (NaOH) 12M concentration.

The jar test was performed with the same sample; 1000 ml of effluent were added in each of the 12 vats of the equipment; a medium speed of 50 rpm was applied, and stirring was performed for 1 hour. In each vat, 2.0ml to 24.0mL NaOH (12M) were measured in order to determine the influence of pH on the precipitation of metals in the form of hydroxides. After stirring, the system was allowed to rest for sedimentation of the sludge to occur.

### 2.3. Electrocoagulation Reactor

The laboratory-scale electrocoagulation reactor was made of glass and measured 8.5 cm in diameter and 13 cm in height. The experiment was performed according to the design shown in Figure 2:

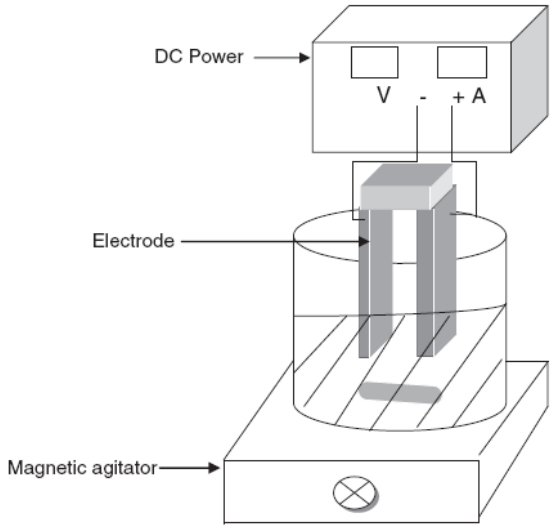


Figure 2: Design of electrocoagulation

The experiment used iron, copper and aluminum electrodes measuring 150 mm x 51 mm x 0.9 mm, 137 mm x 53 mm x 1.0 mm and 143 mm x 50 mm x 1.5 mm, respectively. These electrodes were spaced at 2.0cm and each one was submerged at 52cm². An Iminipa MPL-1303M power supply with adjustable settings was used (voltage = 0 - 30V; amperage 0 - 3A).

500 mL of tanning bath were added in the electrocoagulation reactor; the voltage was adjusted, and a sample was collected in duplicate every 10 minutes.

### 2.4. Analytical Methods

The content of trivalent chromium oxide ($Cr_2O_3$) was determined in the laboratory in accordance with standard NBR 13341:2010, which consists of a digest from the addition of sodium hydroxide (NaOH) 4%, hydrogen peroxide ($H_2O_2$) 6%, and sulfate and nickel ($NSO_4$) 5%. After this step, hydrochloric acid (HCl) and 50% potassium iodide (KI) 10% were added, and then a titration was carried out with sodium thiosulfate ($NA_2S_2O_3$) 0.1 N.

The experiments were performed in duplicate to keep the margin of error below 5%.

## 3. Results and Discussion

### 3.1 Chemical precipitation

In the first six (6) tests, samples 5 and 6 were the ones that had the best visual results, with clear supernatant appearance and rapid sedimentation, after 3 hours (Figure 3).

Figure 3: Testing of samples 1 to 6.

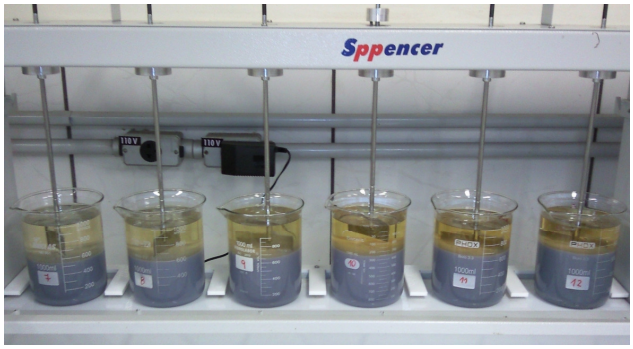


Figure 4: Testing of samples 7 to 12.

In tests 7 to 12, the supernatants of samples 10, 11 and 12 looked yellowish, the sludge formed was thick, and sedimentation took longer, around 6 hours (Figure 4).

According to the final visual evaluation of the samples, the best results were obtained in samples 5 to 9. This preliminary assessment can be confirmed with the lowest values of $Cr_2O_3$ concentration (mg / L) in the supernatants and improved removal efficiencies obtained in samples 6 to 12 (Table 2).

Table 2: Results of physical and chemical precipitation of the residual tanning bath.

| Sample | Final pH | Vol. NaOH used (mL) | Appearance | $Cr_2O_3$ Concentration (g/L) | Removal efficiency (%) |
|---|---|---|---|---|---|
| **Bruto** | 4.07 | 0 | **Cloudy** | 2.0000 | 0.00% |
| **1** | 5.37 | 2 | **Cloudy** | 1.8379 | 8.11% |
| **2** | 6.78 | 4 | **Transition** | 1.0543 | 47.29% |
| **3** | 7.71 | 6 | **Transition** | 0.0293 | 98.54% |
| **4** | 9.08 | 8 | **Transition** | 0.0257 | 98.72% |
| **5** | 9.97 | 10 | **Clear** | 0.0217 | 98.92% |
| **6** | 10.1 | 12 | **Clear** | 0.0196 | 99.02% |
| **7** | 9.98 | 14 | **Clear** | 0.0053 | 99.74% |
| **8** | 10.24 | 16 | **Clear** | 0.0181 | 99.10% |
| **9** | 10.34 | 18 | **Clear** | 0.0177 | 99.12% |
| **10** | 10.62 | 20 | **Cloudy** | 0.0122 | 99.39% |
| **11** | 10.69 | 22 | **Cloudy** | 0.0106 | 99.47% |
| **12** | 10.88 | 24 | **Cloudy** | 0.0098 | 99.51% |

There was a significant influence pH on chromium removal; the best results were obtained above 9.98, as can be seen in Figure 5, and the greatest chromium removal was 99.74% for sample 7.

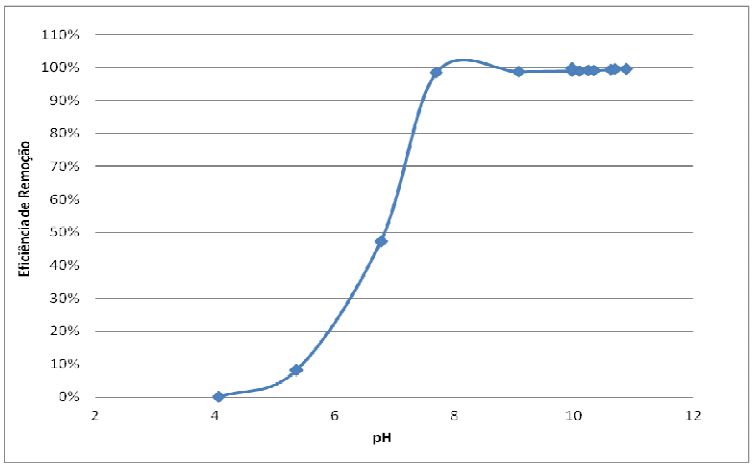


Figure 5: Influence of pH on the removal of $Cr_2O_3$ from the residual tanning bath.

### 3.2 Electrocoagulation

Tests for the three plates were performed with the raw effluent and current variance of 0.5V - 3.0V for 60 minutes. Samples were collected in duplicate every 10 minutes during electrocoagulation in order to determine the optimal range of chromium removal.

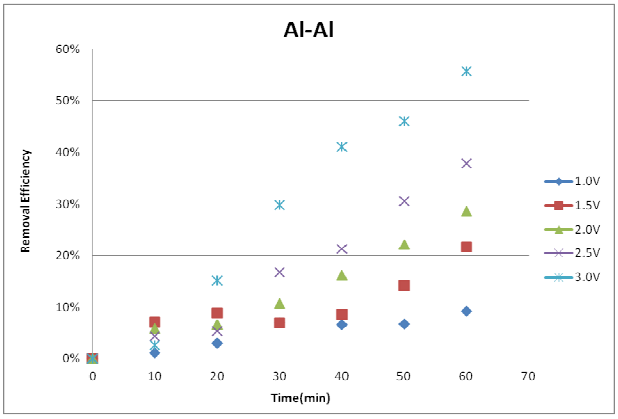


Figure 6: Time variations for removal efficiency with aluminum electrodes at different voltages.

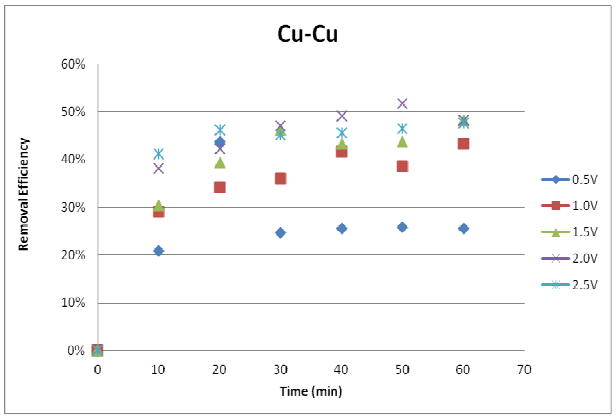


Figure 7: Time variations for removal efficiency with copper electrodes at different voltages.

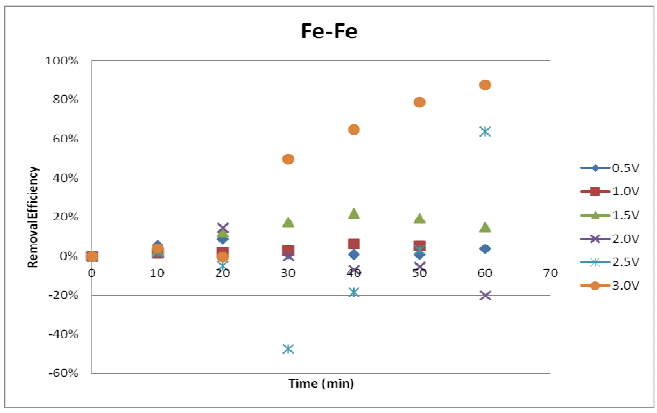


Figure 8: Time variations for removal efficiency with iron electrodes at different voltages.

For aluminum and iron plates, a better removal of chromium was obtained at voltages of 2.5V and 3.0V, and for copper, at 1.5V and 2.0V.

After the optimum voltages were chosen for each electrode, tests were conducted again, prolonging electrocoagulation for more than 100 minutes to achieve best chromium removal efficiency. The tanning liquor had been previously filtered with glass wool, according to standard NBR 13336:1995, which determines the conditions required for sampling wastewaters.

For iron electrodes (Figure 9), the voltage of 2.5V achieved 90.27% removal, and the best removal efficiency (80.57%) was achieved at 3.0V, both at 100 minutes. Therefore, it can be concluded that an increase in voltage does not influence chromium removal.

The copper electrodes (Figure 10) showed no efficiency values for chromium removal; 68% removal was obtained for a period of 50 minutes at 1.5V and 69.91% removal, for 100 minutes at 2.0V.

For aluminum electrodes (Figure 11), removal efficiency above 97.76% was obtained at 3.0V and 110 minutes, but the efficiency values were very similar at the voltage of 2.5V.

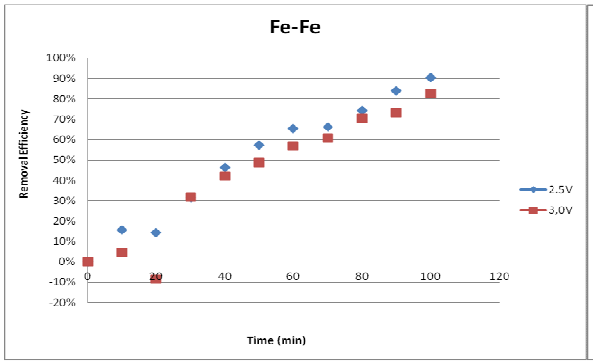


Figure 9: Removal efficiency with time for iron electrodes at different voltages.

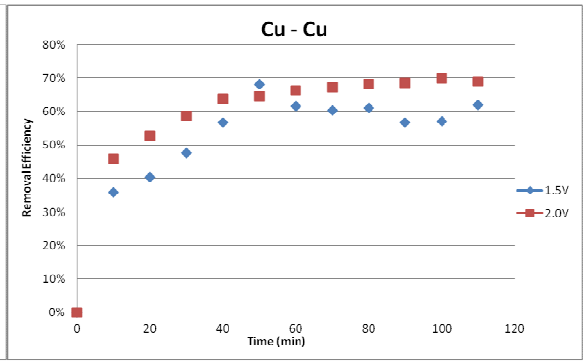


Figure 10: Removal efficiency with time for copper electrodes at different voltages.

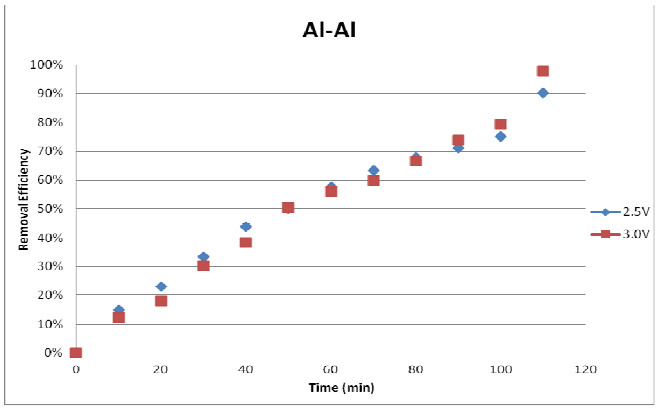


Figure 11: Removal efficiency with time for aluminum electrodes at different voltages.

## 4. Conclusions

In chemical precipitation tests, efficient chromium removal was observed in the samples of wastewaters, reaching values up to 99.74%, as in sample 7, where a final concentration of 5.3 mg/L of $Cr_2O_3$ was obtained.

In electrocoagulation tests, the best removal efficiency (97.76%) was obtained with aluminum electrodes at 3.0V, followed by iron electrodes at 2.5V (90.27%) and copper electrodes at 2.0V (69.91%).

In both processes, compliance with the environmental legislation in Brazil would require further treatment in order to reach the limit determined by CONAMA Resolution no. 430, which determines that the pattern of discharge of chromium-containing effluents has to be 1.0mg $Cr^{+3}.L^{-1}$. Because this effluent results from the tanning process, it has a higher concentration of chromium, and when the effluent is sent to the treatment plant, it is diluted by other currents. However, this was not the goal of this study.

Removing chromium directly from tanning liquors allows the reuse of these waters in the process, and they can be used in the baths or the tanning pickle solution. Precipitated as hydroxide, chromium can be recovered and used again as raw material in the processing of hides, avoiding the generation of hazardous sludge classified as Class I, according to standard ABNT NBR 10.004, published by ABNT (Brazilian Association of Technical Standards).

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