

Efficient chromium recovery from tannery sludge for sustainable management

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Abstract The solid waste generated from tannery wastewater treatment is hazardous as it contains high level of chromium (Cr) which is carcinogenic. To reduce its environmental risk and health concern, recovery and recycling is attempted. The recovery of Cr was performed in three steps, viz., extraction of Cr from the tannery waste sludge, oxidation of Cr(III) to hexavalent Cr(VI), and finally reduction to Cr(III). Total chromium in the sludge was determined using EPA3050(B) method and found to be about 8514 to 9130 mg/kg. The first step, i.e. Cr extraction from tannery sludge, was carried out under three different conditions, viz., neutral, acidic, and oxidizing conditions. The acidic extraction using 4% H₂SO₄ (v/v) on raw sludge extracts 90% of total Cr in the sludge. Though extraction under oxidizing condition using piranha solution (H₂SO₄ + H₂O₂ in ratios 3:1) resulted in complete dissolution of sludge, due to its highly corrosivity, strong oxidizer, economy and experimental safety concerns thus acidic extraction was chosen for further experiment. The second step using advanced oxidation process (H₂O₂ + UV) at pH 10 achieved maximum oxidation (83%) to Cr(VI) at 3 h. Finally, the reduction of Cr(VI) to Cr(III) as chromium sulphate [Cr₂(SO₄)₃] was performed using sodium sulphite at acidic pH 2 with overall recovery of about 83%. The study showed promising results for recovery of chromium,

but the main problem associated with this process is the high dilution of chromium sulphate solution obtained.

Keywords Tannery sludge · Chromium · Metal recovery · Extraction · Advance oxidation process (AOP) · Chromium reduction

Introduction

The solid waste discarded by tannery industries, which contains high concentration of chromium (Cr), is a major environmental and health concern due to its toxicity. The wastewater treatment from tannery industries produces sludge containing high COD, salinity, and heavy metals like Cr poses serious threat to human health as it is carcinogenic. In India, tannery industry is an oldest industry and it is one of the major sources of environmental pollution by heavy metal chromium (Cr). There are more than 2000 tanneries in India, and the wastewater generated from the tannery industry at the end of all processes contains chromium concentration of 80–200 mg/l, but the wastewater generated at chroming tanning stage has a concentration of 1500–3000 mg/l (MoEF 2010). The permissible limit of total chromium in industrial effluent is 2 mg/l as per The Environment (Protection) Rules, 1986 (Schedule VI). While in case of potable water, the limit is 0.05 mg/l as per IS 10500:2012 (2012). Aqueous chromium is found mainly in two forms: trivalent Cr(III) and hexavalent Cr(VI). The Cr(VI) is more toxic than Cr(III) due to high water-soluble, and can accumulate in the body and damage the liver, kidney, and other organs.

To reduce this risk on human health and environment, recovery and recycling of chromium is given importance nowadays. The process of selective separation of an

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element of interest from a heterogeneous waste matrix and sending it back into industrial process from where it has been generated is called recovery and recycling. Many methods have been developed for recovering Cr(III) from the tanning wastewater, which include direct recycling, flocculation, chemical precipitation, ion exchange, extraction, electrolysis, and membrane separation (Macchi et al. 1991; Guo et al. 2006; Kiliç et al. 2011; Li et al. 2014; El-Sheikh and Rabah 2015). An environmental friendly removal and recovery of Cr(III) using natural media was also extensively studied (Fabbicino et al. 2013; Fabbicino and Gallo 2010; Cesaro et al. 2008). Among these methods, chemical precipitation is preferred due to its maturity in technique, simplicity in equipment, flexibility in operation, and ability to recover Cr(III) (Mao et al. 2015).

The use of Cr(III) in the form of basic chromium sulphate [$\text{Cr}_2(\text{SO}_4)_3$] as tanning agent is extensive in leather tanning process (Ramteke et al. 2010). About 40% of the chromium salt remains in the liquid without reacting with the hide and only 60% react with the hide (Li et al. 2014). Therefore, the sludge generated due to the treatment of this tanning wastewater containing unreacted chromium is treated as hazardous waste. Sludge contains mostly Cr(III) and only traces of hexavalent chromium Cr(VI). The sludge are mostly disposed off on the soil surface directly with further treatment, the redox proper of soil as well as the presence of manganese oxides Mn(III/IV) can oxidize Cr(III) into Cr(VI) (Trebien et al. 2011). Due to this oxidation of Cr, its toxicity as well as mobility also increases.

Various studies have been conducted for the removal and recovery of Cr from tannery sludge in past such as Macchi et al. (1991) extracted chromium from tannery sludge using sulphuric acid, and 90% extraction was attained at a pH 1. Oxidation of Cr(III) to Cr(VI) yielded 79% oxidation at pH 10 and at a temperature of 60 °C. But the duration of heating required was 30 h, which is time- as well as energy-consuming. Finally, reduction of Cr(VI) to Cr(III) was performed with sulphur dioxide (SO_2). Guo et al. (2006) performed sludge dissolution using H_2SO_4 , and precipitation of Cr(III) was done by using different alkali such as NaOH, MgO, CaO, NaHCO_3 , and Na_2CO_3 , about 60% recovery was achieved. Kiliç et al. (2011) recovered chromium from tannery sludge by oxidation of Cr(III) to Cr(VI) form using 50% H_2O_2 at pH greater than 10 and then reduced to Cr(III). About 70% of chromium recovery was obtained at pH 2. It was observed that the oxidation of Cr(III) would be the governing factor in the recovery processes which is generally followed. Therefore, to achieve higher oxidation of Cr(III) advanced oxidation process (AOP) with H_2O_2 and UV was attempted.

The objective of the study is to extract Cr from the tannery wastewater sludge, and to oxidize the sludge to get to Cr(VI) which is further reduced to Cr(III) and its subsequent removal by precipitation and coagulation. Cr recovery processes from sludge extract is mostly governed by its oxidative capacity to Cr(VI). Therefore, this study further utilizes the effective oxidation procedure using H_2O_2 and advanced oxidation process (AOP— $\text{H}_2\text{O}_2 + \text{UV}$) to get higher concentration of Cr(VI). And finally, Cr(VI) may be effectively reduced to Cr(III), using sodium sulphite, Na_2SO_3 .

Materials and methods

Tannery sludge characteristics

Sludge from tannery wastewater treatment plant near Ranipet area, Vellore, Tamil Nadu, was collected in a sealed polyethylene bag and stored at a temperature of 5 °C. The water content of the sludge was determined by gravimetric method, and the organic matter was determined by loss of ignition (LOI) method. Total chromium concentration in the sludge was determined by as per USEPA 3050 B.

Extraction procedure

The extraction process was carried out at different conditions such as neutral, reducing, and oxidizing conditions using distilled water, 4% H_2SO_4 (v/v), and piranha ($\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ in ratios 3:1), respectively. The various methods used in the extraction procedure are discussed in the following sections for efficient recovery process. All the experiments were carried out in duplicates so as to avoid undue error with acceptable limits of 10% being considered.

Extraction in neutral condition

For neutral condition extraction, distilled water alone was used as the sludge dissolution medium. Dissolution was carried out by adding 1 g of wet sludge to 50 ml of distilled water ($L/S = 50$) which was taken in 250-ml conical flask. This experiment was performed to find the leachability of sludge, which may potentially harm the environment. Then, the flask was placed in an orbital shaker at 250 rpm for 24 h. The supernatant was collected and placed in a centrifuge at 8000 rpm for 10 min and analysed for metal concentration.

Extraction in acidic condition

Acidic extraction was carried out with 4% (v/v) H_2SO_4 for sludge dissolution. 1 g of wet sludge was mixed to 50 ml of 4% H_2SO_4 which was taken in 250-ml conical flask. Similarly, H_2SO_4 was commonly used in the extraction procedure of Cr from the sludge (Macchi et al. 1991; Guo et al. 2006; Kiliç et al. 2011; Li et al. 2014) and reported significant amount of Cr extractions up to 90% of total Cr in the sludge. The resultant supernatant was centrifuged at 8000 rpm for 10 min and then analysed.

Extraction in oxidizing condition

Piranha solution, a strong oxidizing agent, is a mixture of sulphuric acid and hydrogen peroxide in the ratio of 3:1, respectively. Since the reaction is exothermic and produces fumes, the preparation was carried out inside a fume hood by placing the acid in a water bath. The L/S ratio was kept at 50 and similar procedure was followed, and the samples were analysed.

Oxidation of Cr(III) to Cr (VI)

Oxidation of Cr(III) to Cr(VI) was carried out in three ways, viz., chemical oxidation using H_2O_2 , combining $\text{H}_2\text{O}_2 + \text{UV}$ (advanced oxidation process—AOP), and oxidation using piranha solution. Cr(VI) concentration was measured using Cyberlab UV-100 UV spectrophotometer by diphenylcarbazide method.

Chemical oxidation involving 30% H_2O_2 was performed in 1:1 ratio volumetrically. Advanced oxidation process (AOP), i.e. combination of H_2O_2 and UV, was set up in laboratory with UV-C radiation (wavelength 280–100 nm). Kiliç et al. (2011) reported that higher efficiency of oxidation was observed using H_2O_2 at a pH range of 10 and above.

Oxidation of Cr using piranha solution was performed by adding 5 ml of piranha solution to 15 ml of the extract obtained by dissolving sludge in 4% H_2SO_4 . A precipitate formation was noticed in a clear solution after the addition. The solution was analysed for its metal concentration. The precipitate was dried, and it was given gold coating by sputtering process. Then, the gold-coated sample was analysed for the presence of chromium using EVO 18 Research Zeiss scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDS).

Reduction of hexavalent chromium as chromium sulphate

The final step is the reduction of Cr(VI) to Cr(III) as chromium sulphate. For this step, reducing agents such as sodium sulphite (Na_2SO_3), sulphur dioxide (SO_2), and ferrous compounds can be used under acidic condition (Macchi et al. 1991; Erdem 2006). Among these reducing agents, sodium sulphite (Na_2SO_3) and SO_2 are the most preferred (Erdem 2006). This is because reducing agents Na_2SO_3 and SO_2 do not contain foreign ions. As SO_2 is toxic and considering its safety, Na_2SO_3 was used to obtain the recovered Cr in the form of chromium sulphate [$\text{Cr}_2(\text{SO}_4)_3$] which is relatively pure and suitable for re-tanning processes.

Analytical procedure

Usepa 3050b

The total chromium (Cr) content in the sludge was determined using US EPA 3050 (B) sludge digestion methods. 1 g of moist sludge aliquot was taken in a digestion vessel, and 10 ml of 50% (v/v) HNO_3 was added. The vessel was covered with vapour recovery device, heated to $95 \pm 5^\circ\text{C}$, and refluxed for 10–15 min without boiling. The sample was cooled, and then 5 ml of conc. HNO_3 was added and refluxed for 30 min. Then, the sample was cooled, and 2 ml of distilled water and 3 ml of 30% H_2O_2 were added and heated until effervescence got subsided. Finally, 10 ml of conc. HCl was added to the sample and refluxed at $95 \pm 5^\circ\text{C}$ for 15 min. The cooled digestate was filtered and collected in a 100-ml volumetric flask, then a mark was made, and analysed by Varian AA240 atomic absorption spectrometer (AAS). All the experiment of digestion of sludge was performed in duplicates, and most of the results were below 10% variation.

Results and discussion

This section shows the significant result and discussion of the analysis for the recovery of chromium from the tannery sludge. The characteristics of the sludge collected and the various steps involved in the recovery process, viz., chromium extraction, oxidation of Cr(III), and reduction of Cr(VI) as chromium sulphate, are discussed.



Characterization of sludge

The sludge collected from tannery wastewater treatment plant was moist, dark brown in colour. It is alkaline with a pH 8.3. The water content of the wet sludge was about 64%. Organic matter was estimated as per loss of ignition method from the weight loss of a dry aliquot of sludge after washing at 550 °C for 2 h, and organic matter was about 31% of dry mass. Total chromium content in the sludge was 8514–9130 mg/kg of sludge as per USEPA 3050B.

Extraction of chromium from sludge

The extraction of Cr from tannery sludge which is the first step of recovery process was performed under three different extraction conditions, and the supernatant obtained was analysed for metal concentration using atomic absorption spectroscopy (AAS).

Extraction in neutral condition

The neutral extraction by using distilled water was about 145 mg/kg of Cr. This corresponds to about 1.5% (wt.) of total Cr extracted using EPA 3050B. As the sludge contains enormous amount organic matter, the Cr metal which is in Cr(III) form always gets bound to these particles, and there is not much release of chromium into the solution when the sludge was dissolved in distilled water. At a pH around 8.3, iron will be in insoluble ferric (Fe^{3+}) state and might be removed along with the solids filtered. Magnesium (Mg) concentration was 1574 mg/kg and manganese (Mn) 14.05 mg/kg of sludge. The metal concentration obtained by extraction under neutral condition is shown (Fig. 1). Higher Mg in sludge is due to the use of magnesium oxide, to increase the pH thus allowing chromium to bind with the skin protein (Anthony 2009).

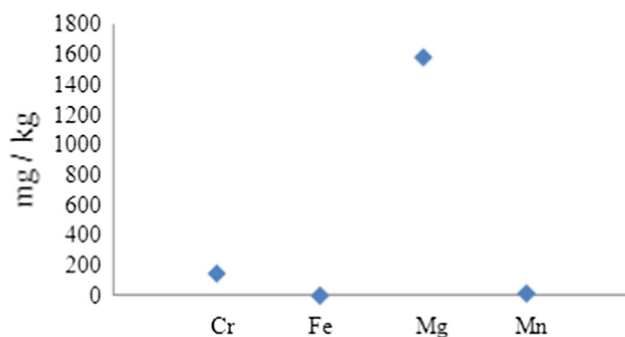


Fig. 1 Concentration of metals under neutral extraction condition

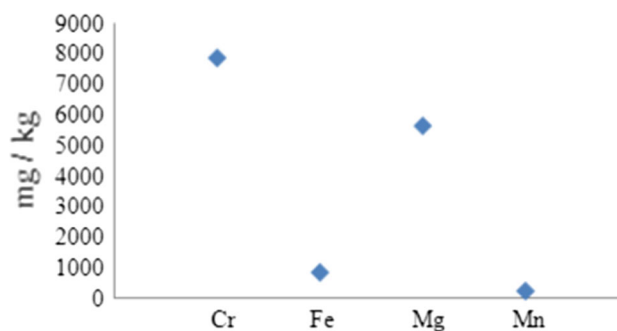


Fig. 2 Concentration of metals under acidic extraction condition

Extraction in acidic condition

The metal extraction carried out under acidic condition using 4% (v/v) H_2SO_4 solution showed Cr concentration of about 7843 mg/kg of sludge at pH 0.4. This comprises about 90% of total chromium present in the sludge, similar results were also reported by Macchi et al. (1991) and Li et al. (2014). The decreased extraction yield of total chromium was due to the metal adsorption and bonding on the insoluble organic matters as well as the formation of metal precipitates (Shen et al. 2001). Fe concentration was 841 mg/kg of sludge, Mg concentration was 5627 mg/kg of sludge, and Mn concentration was 231 mg/kg of sludge as shown in (Fig. 2). Iron in the sludge can be due to the use of iron compounds as coagulants in tannery wastewater treatment process.

Extraction in oxidizing condition

A complete dissolution of sludge was observed under oxidizing condition using piranha solution ($\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ in ratios 3:1). Total Cr extraction with piranha solution at solid solution ratio 1:50 was about 8845 mg/kg of sludge. Piranha solution ($\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$), which is a highly oxidizing agent, oxidizes all the organics by the reactive atomic oxygen formed by the reaction between sulphuric acid and hydrogen peroxide (Koh et al. 2012) as shown in Eq. 3.1.



This atomic oxygen disrupts all the carbon bonding, liberating the bounded chromium into the solution. Fe concentration was 3754 mg/kg of sludge and its co-ions Mn was found to be 877 mg/kg dissolved under this condition as shown (Fig. 3). Iron in the sludge can be due to the use of iron compounds as coagulants in tannery wastewater treatment process. Magnesium was 6437 mg/

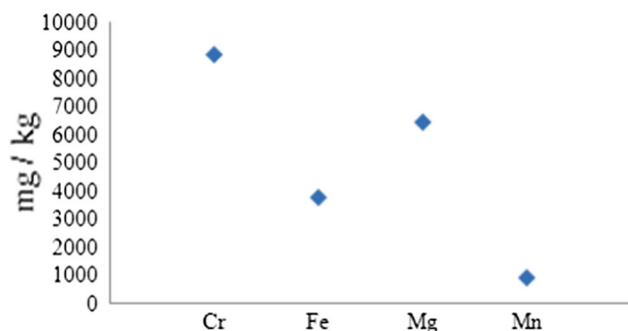


Fig. 3 Concentration of metals under oxidizing extraction condition

kg of sludge and its source is magnesium oxide which is used to increase the pH (Anthony 2009).

Oxidation of Cr(III)

Even though total Cr extraction was achieved using piranha solution, but considering the operational safety of the process, acidic extraction using 4% (v/v) H_2SO_4 yielding significantly 90% of Cr extraction was chosen. The preparation and handling of this solution is easier, safe, and economical.

Oxidation of Cr(III) to Cr(VI) which is the next step after extraction process was carried out in three ways such as chemical oxidation using hydrogen peroxide (H_2O_2), advanced oxidation process ($H_2O_2 + UV$), and oxidation using piranha solution.

Chemical oxidation using H_2O_2

The chemical oxidation of Cr(III) to Cr(VI) using H_2O_2 suggested slow rate and equilibrium was observed at about 2.5 h after the addition of H_2O_2 as shown in (Fig. 4) at 30 °C. Kiliç et al. (2011) reported best recovery involving oxidation of Cr at pH above 10 due to the formation of

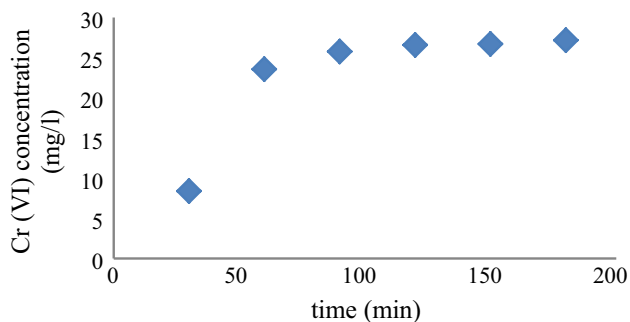


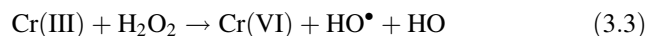
Fig. 4 Chromium oxidation using 30% H_2O_2

hydroxyl radical. Similarly, for this study an alkaline range was chosen to effectively oxidize the tannery sludge with high organic matter where additional heating may be avoided. The oxidation reaction was performed, and the equilibrium concentration of oxidized Cr(VI) was 27 mg/l which constitute about 17% of total Cr in the solution. Under alkaline condition, the oxidation process takes place as shown in Eq. 3.2



As per the equation shown above, peroxide abstracts electron from Cr(III) and converts it into Cr(VI). The variation in oxidation yield with respect to time is shown in Fig. 4.

The graph (Fig. 4) shows high rate initial oxidation up to 60 min, but after this the rate of oxidation reduces till it reaches the equilibrium. Bokare and Choi (2014) reported the oxidation of Cr(III)/ H_2O_2 systems as



Although this equation explains the general oxidation mechanism, the unexpected pH-dependent reactivity is attributed to the formation and oligomerization of Cr(III)-hydroxo complexes. All these oligomers react with H_2O_2 and generate HO^\bullet , but the reaction rate is inversely proportional to the degree of oligomerization which in turn is directly proportional to the solution pH (Bokare and Choi 2011).

Advanced oxidation process using H_2O_2 and UV

Advanced oxidation process (AOP) was similarly performed under alkaline condition of pH 10, and at the end of 3 h Cr(VI), concentration was 131 mg/l which is about 83% oxidation of Cr (Fig. 5). At the end of AOP process, the solution colour was yellowish indicating higher Cr(VI) concentration. The reason mainly attributed for this higher oxidation of Cr(III) ion to Cr(VI) ions is due to the hydroxyl radicals generated by the photolysis of H_2O_2 molecules by the UV radiation (Beltran 2003) as shown in Eq. 3.4



These hydroxyl radicals react typically a 10^6 – 10^9 times faster than ozone and hydrogen peroxide. The hydroxyl radical attacks the Cr(III) by direct electron transfer to fill the electro hole created in it and produces Cr(VI) and hydroxide ions. At the end of advanced oxidation process (AOP), the pH of the solution increased to 12.5 which

indicate the formation of hydroxide ions. This rise in pH also indicates that the oxidation of Cr(III) is due to the electron removal from chromium atom by hydroxyl radical. The reason for 83% oxidation to Cr(VI) may be due to the parallel formation and precipitation of less reactive Cr(III) species (Macchi et al. 1991).

The oxidation of Cr using AOP (Fig. 5) showed very high oxidation of Cr(III). UV-C radiation which has a wavelength in the range of 280–100 nm is used for the photolysis of hydrogen peroxide (H_2O_2), since the energy of the photon to break the O–O bond should be greater than 213 kJ/mol which can be easily produced with radiation of wavelength less than 280 nm. This activates the molecule from ground state to the excited state. As a result of this excitation, the molecules undergo homolytic or heterolytic breakage which might lead to oxidation of Cr(III). In most of the conditions, homolytic rupture occurs resulting in the production of radicals (Litter 2005).

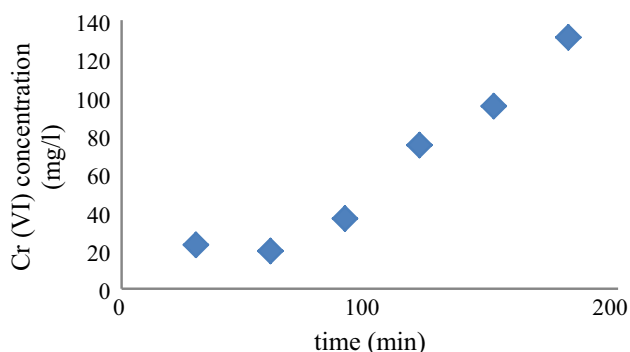
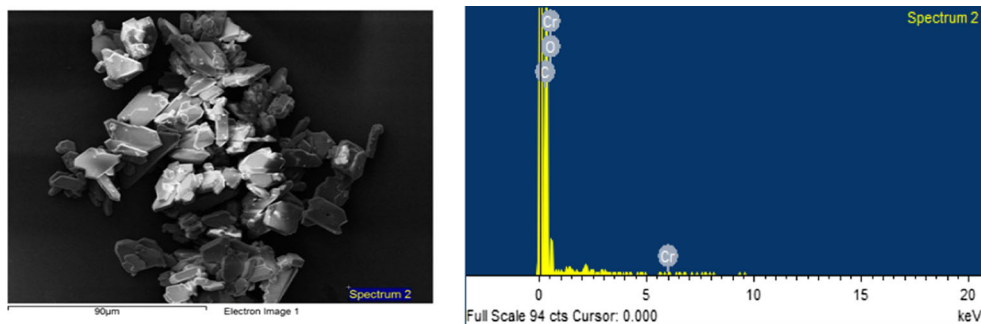


Fig. 5 Chromium oxidation using 30% H_2O_2 + UV (AOP)

Fig. 6 Precipitate analysis using energy-dispersive X-ray (EDX) spectroscopy

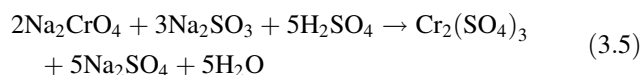


Oxidation using piranha solution

The oxidation of Cr(III) by adding piranha solution to the 4% (v/v) H_2SO_4 extract in the ratio of 1:3 ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$) was most significant. The total Cr concentration in the sample was reduced to 66 mg/l which is about 60% reduction. The negligible concentration of Cr(VI) can be due to the low pH of the piranha solution. A precipitate was formed, and it was analysed for energy-dispersive X-ray spectroscopy (EDS) (Fig. 6). The result of EDS analysis using EVO 18 Research Zeiss scanning electron microscope (SEM) confirmed the presence of Cr in the precipitate.

Chromium recovery as chromium sulphate [$\text{Cr}_2(\text{SO}_4)_3$] using Na_2SO_3

The final step in the recovery process is the reduction of the Cr(VI) ions using a reducing agent. The Cr(VI) from the above process was completely reduced to $\text{Cr}_2(\text{SO}_4)_3$ using sodium sulphite in room temperature at a pH of 2 ± 0.1 (Erdem 2006) as shown in Eq. 3.5. The Cr(VI) concentration was below detection limit. The solution obtained was pale green in colour indicating the presence of trivalent chromium.



The $\text{Cr}_2(\text{SO}_4)_3$ thus obtained is relatively pure and can be directly reused in the tanning operations. As there was 83% of oxidation, almost all the Cr(VI) ions were reduced

to chromium sulphate $[\text{Cr}_2(\text{SO}_4)_3]$, indicating total reduction of oxidized chromium, i.e. Cr(VI). The main disadvantage in this recovery process is the high dilution of $\text{Cr}_2(\text{SO}_4)_3$ solution recovered from tannery wastewater sludge.

Conclusion

This research work focused on the recovery of chromium (Cr) from tannery sludge. To achieve this goal efficiently, three steps, viz., extraction of Cr from sludge, oxidation of Cr(III) into Cr(VI), and finally, reduction of Cr(VI), were investigated and the following conclusions were drawn.

Acidic condition using 4% (v/v) H_2SO_4 solution, comprising of 90% of the total chromium present (USEPA 3050B) in the sludge, was chosen for the further steps of recovery process, since the preparation and handling of 4% H_2SO_4 solution is easier, safe, and economical. Advanced oxidation process (AOP) using $\text{H}_2\text{O}_2 + \text{UV}$ showed dominant oxidation of 83% of Cr(III) to Cr(VI) under alkaline pH 10 at 3 h. Finally, the Cr(VI) was completely reduced to Cr(III) in the form of chromium sulphate $[\text{Cr}_2(\text{SO}_4)_3]$ using sodium sulphite. This indicates recovery is about 83%. Even though about 83% of recovery was reached, high dilution of the recovered chromium sulphate limits the use of this technology in a large scale.

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