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Recovery And Reuse Of Chromium From Spent Chrome Tanning Liquor By Precipitation Process

Kamruzzaman Khan[1], Md. Istiaq Habib Khan[2],Iftakharul Islam Khan[1],Abdullah Al Mahmud[1], Md. Dalour Hossain[1]

[1]*Department of Leather Engineering, University of Dhaka, Bangladesh*
[2]*Bangladesh University of Engineering and Technology, Bangladesh*
Corresponding Author: Kamruzzaman Khan

Abstract: *Leather is a resilient and durable material, rich and unique in quality. It is used in shoe, bag, saddle and other product manufacturing. Nonetheless, the processes of leather manufacturing from raw hides and skins are responsible for serious environmental pollution. There are several steps associated with the leather manufacturing, among all the steps, chrome tanning is the most significant process which brings permanent unique physical characteristics to leather, however, it is one of the most environmentally polluting steps as well. In this work, our aim was to recover the chromium from spent chrome tanning liquor and reuse the chrome liquor in subsequent chrome tanning processes. It was found that the recovered chrome liquor contained 3.44%* Cr_2O_3. *A freshly made chrome tanning bath requires 8% Basic Chromium Sulfate salt. The leather produced from the mixture of fresh chrome and recovered chrome liquor at the ratio of 0:10, 5:5 and 5:3 exhibited better physical characteristics in ball bursting and lastometer test; standard physical characteristics in stitch tear strength, shrinkage temperature and boil test and marginal physical characteristics in tensile strength compared to traditionally prepared chrome tanning bath.*

Keywords: *Chrome liquor,Chrome recovery & reuse,Environmental pollution,Tanning*

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I. INTRODUCTION

Chromium solutions are widely used in many industrial processes such as varieties chromium based chemical production, chrome plating, wood preserving, textile dyeing, pigmenting, pulp, paper and tanning industries [1]. The wastewater resulting from these processes contains high amount of chromium metal which is harmful for the environment and human health.Tanning is the chemical process that converts animal hides and skin into stable and imputrescible products called leather [2]. It is a vital industrial process to produce leather from raw hides and skins. Yet, the process costs a great toll to environment. The transformation of hides into leather is usually done by means of tanning agents and the process generates highly turbid, colored and foul-smelling effluent [3]. One estimate of the carbon footprint of leather goods is 0.51 kg of CO_2 equivalent per £1 of output at 2010 retail prices, or 0.71 kg CO_2eq per £1 of output at 2010 industry prices [4]**.** One ton of hide or skin generally produces 20 to 80 m^3 of waste water, including chromium levels of 100–400 mg/l, sulfide levels of 200–800 mg/l, high levels of fat and other solid wastes, and notable pathogen contamination. Producers often add pesticides to protect hides during transport. With solid wastes representing up to 70% of the wet weight of the original hides, the tanning process represents a considerable strain on water treatment installations [5]**.** Leather biodegrades slowly—taking 25 to 40 years to decompose [6]**.**Leather is produced in three different steps namely beam house operations, tan yard operations, post tanning operations and finishing operations [7]**.**Among all the processes tanning is the most important process and chrome is the most widely used tanning agent across the world. Despite the importance of the process, this very process is the reason for extreme water pollution, especially when discharged outside tanneries. Lots of researches have been conducted to find out the characteristics of wastes produced in tanning process and their impact on environment as well as on workers involved in the process [8-12].Chemical used in tanning and different processes that causes severe health hazards and environmental problems to the entire ecosystems in all stages [13]**.**

In chrome tanning process about 60% - 70% of chromium reacts with the hides. In the other word, about 30%-40% of the chromium amount remains in the solid and liquid wastes especially spent tanning solutions [14]. Hence, the wastewater of the tanning process is a noteworthy source of pollution which adds chromium to the environment. One study showed that the concentration level of Chromium in the spent Chrome liquor is 2656-5420 mg/L which is extremely high [15]. In addition, the cost of the chromium metal is also important and it is possible to be recovered from the wastewater. About 90% of all the leathers that are produced worldwide adopt chrome tanning process where chromium (III) salts are used [16, 17]. The trivalent chromium is converted to hexavalent state when discharged through oxidation [18] and mixed with water. Chronic exposure of tannery workers from a period of five months to fourteen years represents a relevant risk factor for the development of diseases associated with genetic damage. The temporary effects of tannery waste and effluent are such as dizziness, headache, irritation of eyes, skin or lungs, allergic reactions, poisoning of liver, kidney or nervous system or collapse due to lack of oxygen [19], gastro-intestinal ulcer, nasal septum [16], as well as long term illness like occupational asthma, ulcers, bronchitis, genetic defects and dermatitis in humans and animals health [20]. Chromium is an essential metal that is involved in the metabolism of glucose in humans and animals. Beside human and animal, plants (root and shoots area exclusively) are also being affected by tannery effluent [21], but its Cr(VI) form is very toxic, mutagenic, and carcinogenic [22,23]. Cr(VI) affects enzyme amylase in plant (which plays an important role during seed germination through hydrolysis of reserve starch and release in the energy) and shortens seed sprouting of plants. Normally chromium is known to have chronic toxicity (above 5 mg/L) in drinking water [19]. Hence, to minimize pollution, we should consider recycling chromium used in tanning industry. Several methods have been used for removing toxic metal ions from aqueous solutions. These include chemical precipitation, ion exchange, reverse osmosis, membrane processes, evaporation, solvent extraction and adsorption. Of these, chemical precipitation is the usual way for this purpose. Many factors affect the process of chemical precipitation including the type of precipitation agent, pH and velocity of precipitation, sludge volume, time of mixing and complexing agents [14].
The purpose of this work is to establish a recovery method of chromium and comparison of different physical and chemical characteristics between freshly chrome tanned leather and recovered chrome tanned leather.

II. Materials & Method

2.1 Chromium recovery from spent liquor

Chromium recovery procedure from spent chrome tanning liquor is given below:

Step-1: Collection of spent Tan liquor whose pH is 3.9. For the experiment purpose 20 liters of spent chrome liquor were collected from Reliance Tannery Ltd.

Step-2: Maintenance of pH of spent liquor at 8.0 to 8.5 by adding $NaHCO_3$. To facilitate precipitation of chromic hydroxide, a flocculent such as those which are widely known in the art (e.g. hydrolyzed polyacryl amide flocculent) can be used. Removal of chromic hydroxide can be affected by filtration or centrifugation, and the process of the invention has been found particularly suitable where rapid sedimentation, such as that that is achievable using a lamella separator, is desired [24].

Step -3: The mixture was stirred and allowed to settle down for hours. The chrome in the form of Chrome Hydroxide got settled.

Step-4: The Supernatant containing insignificant quantity of chromium concentration was liable to be discarded.

Step-5: The chrome sludge was dissolved in 98% concentrated Sulfuric Acid until a pH of 2.5 was reached. This mixture was worth of blending with freshly made Chrome tan liquor.

Step-6: It was found that recovery of Chromium in a properly designed and operated recovery plant could be around 98% of the Chromium in the spent tanned liquor [25, 26].

2.2 Chromic oxide content determination

Chromic oxide content was measured using the method SLC 208.According to this method a suitable volume of liquor was oxidized to the hexavalent state using Perchloric acid. Then the solution was titrated with standard $Na_2S_2O_3$solution [27]. The calculations and results are discussed in the following section and titration data are given in the Table-1.

2.3 Chrome Tanning

Chrome Tanning is the tanning process using Chromium (III) salts by which putrescible hides and skins are converted into leather. It is achieved by Chromium (III) as tanning matters which cross link with the collagen through octahedral carboxylic covalent compound [16] without modifying the natural fiber texture.
To process leather with recovered chrome liquor, 4 pieces of wet salted goat skin were collected. The raw weight of 4 pieces of goat skins was 6 kg. The manufacturing process for leather preparation can bedivided into three basic sub-processes: preparatory stage/beamhouse stage, tanning stage and crusting stage [28]. These operations also include chemical and mechanical treatment and processing respectively.

2.4 Leather Physical Testing

The finished leather samples under the experiments were tested for their various physical properties. These properties indicated about the quality of the finished leather produced. Selected physical tests were accomplished to determine the quality of produced leather and these tests are briefly discussed here.

2.4.1 Shrinkage temperature determination

Leather has the unique property of shrinking abruptly at a particular critical temperature. While subjected to hot water of gradually increasing temperature, it suddenly shrinks at a particular temperature defined as shrinkage temperature (T_s) [29]. It was measured following the method SLC 406.Each sample was cut into 50 mm X 12 mm size. A 3 mm hole was punched following the method and then a sample was attached to the apparatus.350 ml NaCl solution was used to immerse the sample. A stirrer distributed the heat uniformly while a thermometer denoted the temperature of the bath. When the sample shrunk to such an extent so as to move the pointer half a division from the position corresponding to the maximum length of the sample, the temperature was recorded [27]. The results are given in the Table-2 in the result and discussion section.

2.4.2 Tensile strength & Percentage elongation at break

The Tensile strength and elongation at break was determined by an instrument called 'Tensile Strength Tester'. The results are given in the Table-3 in the result and discussion section.

Tensile Strength: Tensile strength indicates the overall strength of the leather. It was measured following the IUP-6 method. According to this method the sample was cut into dumbbell shaped specimen measuring 110mmX25mm and attach to the tensile strength testing machine (SATRA, Tensile Tester, Leeds, UK). External force was applied to the specimen and was observed in what amount of force the specimen has broken into two parts. The strength depends on the moisture and fat content of the leather sample [30].

$$\text{Tensile Strength} = \frac{\text{Breaking Load (kg)}}{\text{Thickness (cm) x Width (cm)}}$$

Percentage Elongation at break: Elongation at break for this specimen was calculated from the distance of the jaws after breaking was occurred.

$$\text{\% Elongation at Break} = \frac{\text{Distance increa sed by breaking}}{\text{Distance of the two jaw in normal}} \times 100$$

2.4.3 Stitch Tear strength determination

This strength is determined to know the strength of small seams of leather. This test was carried out by the DIN 53331 method. According to this method the sample size was 50mmX25mm and two holes were made each has 2mm diameter. A soft 1mm diameter steel wire bent to U-shape and inserted through the holes, then attached to the tensile strength testing machine (SATRA, Tensile Tester, Leeds, UK) and load was applied until the sample specimen tore down between two cut holes [30].

$$\text{Stitch Tear Strength} = \frac{\text{Tearin g Load (kg)}}{\text{Thickness (cm)}}$$

The results are given in the Table-4 in the result and discussion section.

2.4.4 Lastometer test

The *Bursting Strength* is an index of the overall strength of the leather and is mainly determined for the shoe upper leather. It was carried out by the IUP-9 method. A circular specimen of 25mm diameter was cut from the sample and attached to the Lastometer (STM 104, SATRA, Leeds, UK) and load was applied until the grain of leather was cracked; the test was further extended until the sample burst due to high pressure [30] and the strength is calculated from the formula:

The Grain Crack Strength was determined by following formula,

$$\text{Grain crack strength (Kg/cm)} = \frac{\text{Grain Cracking Load (kg)}}{\text{Thickness (cm)}}$$

The results are given in the Table-5 in the result and discussion section.

III. RESULT & DISCUSSION

3.1 Determination of Cr_2O_3 Content

Table-1: Titration Data

SL. No.	Burette Reading of N/10 $Na_2S_2O_3$ solution			Standard deviation	Average
	Initial	Final	Difference		
1.	0	13.6	13.6	0	13.6
2.	13.6	27.3	13.7	0.05	
3.	27.3	40.7	13.4	0.11	

IV. CALCULATION

The chromic oxide in the recovered chrome liquor is calculated according to SLC 208 as follows:
1 ml 0.1 N titrant is equivalent to 0.00253 gm Cr_2O_3
Chromic Oxide of the Recovered Chrome Liquor is 3.44%.

Calculation of Recovered Chrome Liquor to Equivalent Basic Chrome Sulfate from spent chrome tanning liquor
Sample A was manufactured from freshly made chrome bath where 8% basic chromium sulfate was used.
For Sample – B (1.4 kg):
For Sample – B, 10% recovered Basic Chrome Sulfates (BCS) was used.
The pelt* weight was 1400 gm.
Hence, 1400 gm pelt required (10x1400)/100 gm Basic Chrome Sulfate
= 140 gm Basic Chrome Sulfate

Since, 100 gm Basic Chrome Sulfate contains 25gm Cr_2O_3 [31]

140 gm Basic Chrome Sulfate contains [(140x25)/100] gm Cr_2O_3
= 35 gm Cr_2O_3
But it was determined that, recovered chrome liquor contained 3.44% Cr_2O_3.

35 gm Cr_2O_3 present in [(100x35)/3.44] ml Recovered Chrome Liquor
= 1017.44 ml Recovered Chrome Liquor

140 gm Basic Chrome Sulfate (powder) is equivalent to 1017.44 ml Recovered Chrome Liquor

*The hides or skin under processing from liming to bating is called pelt

For Sample – C (1.5 kg):
For Sample – C, 4% recovered and 4% fresh Basic Chrome Sulfates (BCS) were used.
The pelt weight was 1500 gm.
1500 gm pelt required [(4x1500)/100] gm Basic Chrome Sulfate
= 60 gm Basic Chrome Sulfate
Similarly calculated like Sample B, 60 gm Basic Chrome Sulfate (powder) is equivalent to 436.05 ml Recovered Chrome Liquor
For Sample – D (1.6 kg):
For Sample – D, 3% recovered and 5% fresh Basic Chrome Sulfates (BCS) were used.
The pelt weight was 1600 gm.
1600 gm pelt required [(3x1600)/100] gm Basic Chrome Sulfate
= 48 gm Basic Chrome Sulfate
Similarly calculated like Sample B, 48 gm Basic Chrome Sulfate (powder) is equivalent to 348.84 ml Recovered Chrome Liquor

3.2 Physical Testing Results

The quality of tanned leather with recovered chromium is determined by means of physical testing of the leather. Four different physical testing were carried out and their results are given following.

3.3Determination of Shrinkage Temperature

The quality of wet blue leathers produced were tasted regularly for Boil Test and Shrinkage Temperature and met the standard requirements which are given in the following table:

Table-2: Shrinkage Temperature

Sample	Shrinkage Temperature(T_s)	Standard (T_s)	Boil Test
Sample-A (Using 8% Fresh Chrome)	105°C		Stand
Sample-B (Using 10% Recovered Chrome)	104.5°C		Stand
Sample-C (Using 4% Fresh Chrome + 4% Recovered	106°C	100°C	Stand

Chrome)		
Sample-D (Using 5% Fresh Chrome + 3% Recovered Chrome)	105°C	Stand

3.4 Measurement of Tensile Strength and Percentage of Elongation at Break

The result of tensile strength and percentage of elongation at break are given following.

Table-3: Tensile Strength and Percentage Elongation at Break

Test	Sample Direction	Sample Name				ISO Standard for Shoe Upper
		A	B	C	D	
Tensile Strength (kg/cm^2)	Perpendicular*	319.15	236.84	242.71	296.29	Min. 250 kg/cm^2
	Parallel**	329.79	243.42	252.42	300.93	
% Elongation At Break	Perpendicular	57.14	68.21	67.42	64.07	Max. 70%
	Parallel	53.57	65.64	63.85	59.28	

*Perpendicular: The considered sample was cut perpendicularly from the backbone line
**Parallel: The considered sample was cut parallel from the backbone line

3.5 Measurement of Stitch Tear Strength

The result of stitch tear strength is given following.

Table-4: Stitch Tear Strength

Sample No	Sample Direction	Thickness (cm)	Tearing Load (kg)	Stitch Tearing Strength (kg/cm)	ISO Standard for Shoe Upper
A	Perpendicular	0.11	17	154.54	Stitch Tear Strength = Min. 60-120 kg/cm
	Parallel	0.112	18	160.71	
B	Perpendicular	0.088	12	136.37	
	Parallel	0.09	12.5	138.89	
C	Perpendicular	0.108	15	138.88	
	Parallel	0.11	16	145.45	
D	Perpendicular	0.095	14	147.37	
	Parallel	0.098	16	163.26	

3.6 Measurement of Lastometer Tests

The result of ball bursting test is given below.

Table-5: Data for Lastometer Test

Sample No	Grain Crack Strength (kg/cm)	Distension at Grain (mm)	ISO Standard for Shoe upper
A	20.588	6.08	Cracking load = Min. 20 kg Distention = Max.6-7 mm.
B	20.430	7.34	
C	20.792	6.95	
D	20.833	7.12	

Percentage of Chromic Oxide in Recovered Chrome Liquor was 3.44%. The Shrinkage Temperature of Sample A, B, C and D was more than 100°C and all the samples stood to boil test, which fulfilled all the requirement of the standard value. The leather produced using recovered chromium meets standard qualities of requirements. A comparison between leather produced from 10% Recovered Chrome (sample B); mixture of 4% fresh chrome & 4% recovered chrome (sample C) and 5% fresh chrome & 3% recovered chrome (Sample D) with the leather produced using 8% fresh chromium salt showed the equal quality in terms of physical and chemical properties. Even though the tanned leather Sample-B, where 10% Recovered Chromium was used, gave considerably good shrinkage temperature and stood at boil test also. Considering the results with other scholarly works, it was found that the tensile strength, stitch tear strength and ball bursting strength (Lastometer test) was better than the author's previous work [30], in another work, where Magnesium oxide, Alum and PAC-18 were used to recover the chromium from chrome tanning liquor; nonetheless, the quality of recovered chrome were not tested [32]. From different research works it was found that most of the case magnesium oxide was used to recover chromium from the liquor and the recovery rate was over 90% depending on the process followed and other physical and chemical conditions; however, after recovering the chromium salt that was not used to tan leather, therefore we could not determine the actual quality of recovered chrome liquor [14, 33-36]. Since, leather is measured; sold and leather products are manufactured on the basis of the physical properties of produced leather. Ultrasound method was applied to recover chromium with addition to MgO, but the process is not feasible for large scale application [37]. This project work did not focus highly on quantity and cost which were focused on other scholarly research works [38, 39]. Looking at all the available options and technology, chrome recovery using $NaHCO_3$ was found applicable, especially for the tannery settings of Bangladesh.

V. CONCLUSION

The quality of recovered chromium liquor was tested for percentage of Cr_2O_3. The final wet blue leathers (Sample-A, B, C & D) produced from fresh and recovered chrome liquor were tested for chromic oxide content, shrinkage temperature, boil test, tensile strength, stitch tear strength, ball bursting strength and found better physical characteristics in ball bursting and lastometer test; standard physical characteristics in stitch tear strength, shrinkage temperature and boil test and marginal physical characteristics in tensile strength compared to traditionally prepared chrome tanning bath. Hence, it can be claimed that the economic value of leather is not affected if the leather is tanned with 10% Recovered Chrome. Finally, it is expected that in future all the tanneries will come forward to setup chrome recovery plant to ensure relatively safe environment with a profitable chrome tanning process.

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