

TREATING LEATHER SHAVING WASTE TO DECREASE ITS ENVIRONMENTAL IMPACT

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Chrome shaving waste (CSW) is one of the largest leather tanning wastes, caused an economical and environmental loss if it did not utilize properly. This study aims to extract chromium from chrome shaving waste, by alkaline hydrolysis or incineration in order to reuse it in leather tanning. Sodium hydroxide and carbonate were used in chemical hydrolysis and compared with incineration method in chrome yielding. Twenty five sheep pelts were tanned with recovered and commercial chromium 33% basicity to compare their effects in tanning. The results showed that among alkaline treatments, caustic soda was more effective than sodium carbonate in extraction, but incineration method was the best extraction method because of its high extraction yield (99.37%). Using sugar as a reducing agent was better than sodium sulphite to prepare tanning liquor. It increased chrome yield and produced chromium sulphate 33% basicity, which are more suitable for leather tanning. Most physical and chemical properties, except fat and ash contents, were similar in leathers tanned with recovered chrome and those tanned with commercial chrome. Thus, all studied tanned leathers were suitable for using in leather manufacturing.

Keywords: chrome, physical properties, recovery, tanning

Leather tanning process transforms raw hides or skins into leather-final product. Chrome tanning is the most widely leather tanning method used in the world. Liquid and solid pollution loads at various processing stages were generated in leather tanning (Beltrán-Prieto et al., 2012). Each one metric ton of raw hide provides 200 kg of a leather-final product, along with 250 kg of non-tanned waste, 200 kg of tanned waste and 50,000 kg of wastewater. Thus, only 20% of the raw material weight is converted into leather (Sanjuán-Herráez et al., 2012).

Chrome shaving waste (CSW) is one of the largest solid tanned wastes, generated after mechanical shaving step (Putshaka et al., 2015). Shaving waste primarily consists of chromium and fiber proteins. Due to the complexation among chromium (III) salts and the carboxyl groups of the collagen, shaving waste is stable biologically and not subject to putrefaction (Lakrafli et al., 2012). In Egypt, shaving waste estimated about 30 thousand tons annually (FAOSTAT, 2014), disposed by deep burial, which does not utilize and causes an economic loss.

On the other hand, Cr (III) in shaving waste can be oxidized into Cr (VI) under the most oxidation states in the natural environment. Cr (VI) is recognized to be highly toxic and carcinogenic, and thus it causes a negative environmental impact (Sutyasmi and Supraptiningsih, 2014).

All previous investigations aimed to valorize shaving waste by transform it to eco-friendly materials, can be used in other useful proposes. Therefore, the only way to achieve this target is separating chromium salts from shaving wastes and reuses both of obtained free chromium protein and extracted chromium again. Usually extracted chromium salts are reusing in leather tanning, while free chromium protein is used in different purposes such as animal feeding, gelatin industry or amino acids and peptides production (Muralidhara et al., 1982; Alves Dos Reis and Beleza, 1991 and Mohamed et al., 2013).

In literature, incineration and chemical treatments are the most common methods for CSW treatment. Incineration has been considered as a means for reducing waste volume and provide energy recovery from the combustion of the organic material, while chemical treatment includes extraction of chromium to be reused in the tanning process and isolation of protein fractions (Kantarli and Yanik, 2010; Beltrán-Prieto et al., 2012 and Putshaka et al., 2015). Alkaline such as caustic soda or sodium carbonate are usually more effective than acids in chemical hydrolysis for CSW (Mohamed et al., 2013 and Sutyasmi and Supraptiningsih, 2014).

Therefore, the main objective of this study is to extract chromium from CSW by alkaline hydrolysis or incineration to reuse it in leather tanning as well as comparing among these extraction methods in order to pick up the most suitable one.

MATERIALS AND METHODS

The experimental work was carried out in the Desert Research Center, Cairo, Egypt. Three consecutive stages were done in this work. The first stage is to determine the best chrome recovery method from chrome shaving wastes in order to obtain the highest extraction yield. In the second stage, different chrome extraction conditions were applied to maximize the quantity of recovered chrome. In the last stage, prepared basic chromium

sulphate (BCS) was used in leather tanning to investigate leather properties were determined.

The chrome shaving wastes used in this study were collected from commercial tannery, located in Cairo, Egypt. After that, it was dried for five days in an open shaded place without using any thermal drying treatment. Characterization of CSW was determined. Moisture, ash, fat, protein, chrome contents, total energy and pH values of CSW were determined according to ASTM (2014).

1. Determination of the Best Chrome Recovery Method

Fig. (1) shows the working steps of this stage, which aimed to compare between alkaline hydrolysis vs. incineration extraction method. Thus, chrome was extracted directly from CSW by alkaline hydrolysis or from chrome shaving ash (CSA) after incineration.

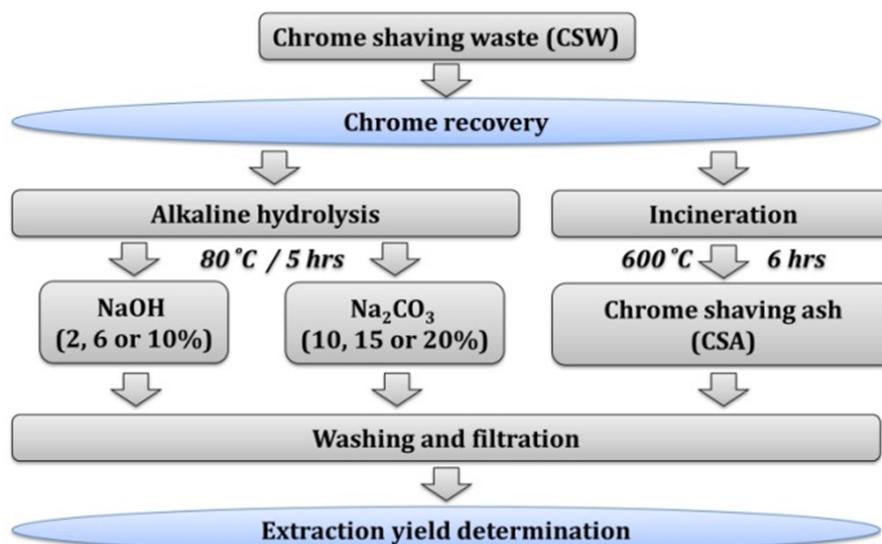


Fig. (1). Schematic diagram for the work plan executed in the first stage of experimental work.

1.1. Chrome shaving waste incineration

Ten grams of CSW were incinerated in muffle furnace at $600 \pm 25^\circ\text{C}$ for 6 hours. After incineration, CSA was washed and filtered through No. 11 Whatman filter paper to dissolve chromium salts and separate it from other residues.

1.2. Alkaline hydrolysis of CSW

Alkaline hydrolysis was done using sodium hydroxide or sodium carbonate, which added as a percentage from CSW weight. Sodium hydroxide was added in three concentrations 2, 6 or 10%, while sodium carbonate concentrations were 10, 15 and 20%. In each concentration, 10 g of CSW were placed in 100 ml of distilled water and then chemical additive was added. The mixture was rotated mechanically at $80 \pm 2^\circ\text{C}$ for 5 hours. Dissolved chromium salts were filtered and sludge was washed after separation.

1.3. Extraction yield

Extraction yield was calculated for each extraction group according to following equation:

$$\text{Extraction yield (\%)} = \frac{\text{weight of chromium salt in extract (g)}}{\text{weight of chromium salt in CSW (g)}} \times 100$$

2. Maximizing the Quantity of Recovered Chrome

Fig. (2) shows the working steps of this stage. The best extraction method from first stage, incineration method, was repeated under different conditions to increase the quantity of recovered chrome. CSA was dissolved in water and then filtered through No. 11 Whatman filter paper. According to Beltrán-Prieto et al. (2012), the recovered chromium was acidified with sulfuric acid then treated with sodium sulphite or sugar in order to obtain BCS solution, which can be used for leather tanning. During the reaction, the color of the liquor changed gradually from orange to green and finally to bluish green indicating the completion of the reduction (Fig. 3). At the end of this stage, extraction yield and chrome basicity of prepared BCS were determined for each extracted group.

2.1. Preparing basic chromium sulphate using sulphuric acid and sodium sulphite

Three treatments were used at three levels of pH. In each treatment, 1 g of CSA was placed in flask and 20 ml of distilled water were added, then sulphuric acid solution was used for adjusting pH values to be 1.00, 1.25 or 1.5. One gram of sodium sulphite was added for reduction and preparing BCS.

2.2. Preparing basic chromium sulphate using sulphuric acid and sugar

According to Salma et al. (2013), 250 g of CSA were mixed with 625 ml water in a beaker; 230 g of concentrated sulphuric acid (Sp. Gr. 1.84) were added carefully and stirred well. The beaker was under cooling with continuous stirring, while 62.5 g of sugar were added slowly in 10 lots over three hours.

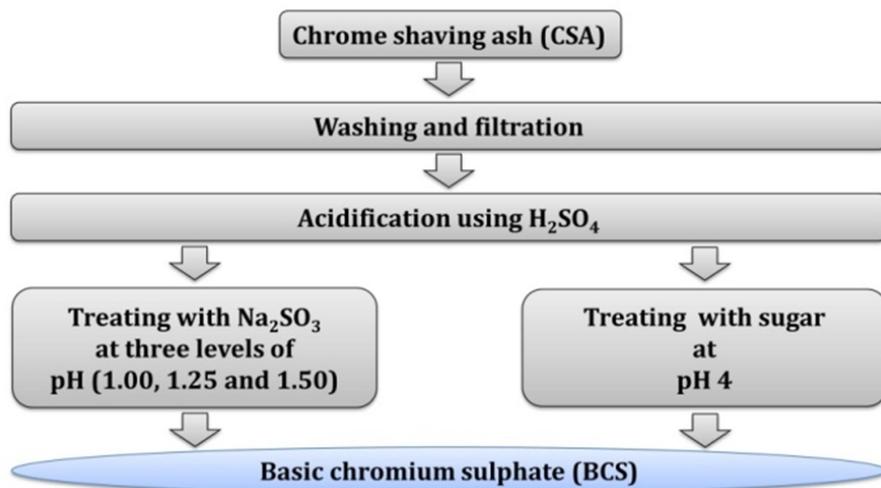


Fig. (2). Schematic diagram for the work plan executed in the second stage of experimental work.

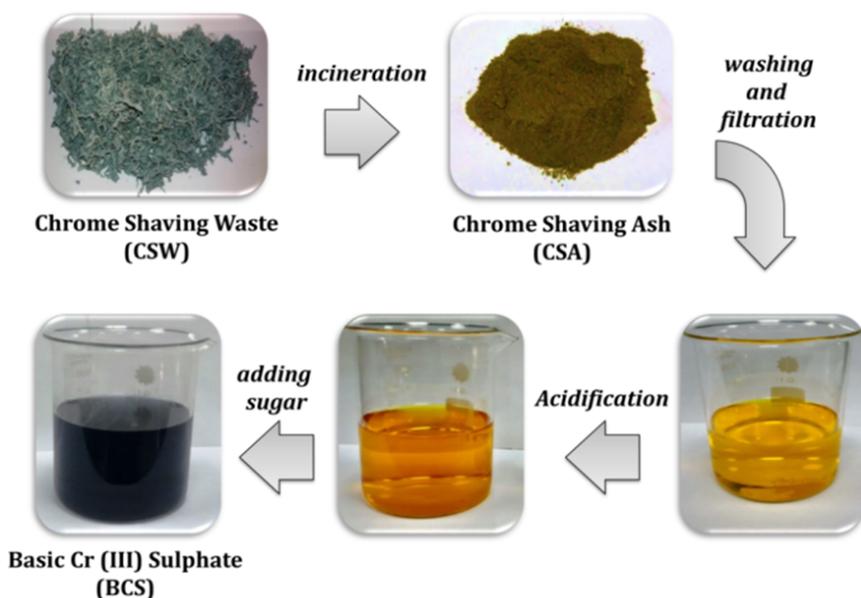


Fig. (3). Producing basic chromium sulphate from shaving wastes.

3. Using of Prepared Basic Chromium Sulphate in Leather Tanning

The prepared BCS was used in tanning pickled sheep skins. Twenty five pickled sheep pelts were tanned in El-Shaar tannery with 8% from pelts' weight of chromium sulphate basicity 33%. Pickled pelts were divided into five groups, five pelts in each group, which differed in the added amounts of commercial and recovered chrome as follows:

- G1: tanned with 100% commercial chromium sulphate and 0% recovered chrome.
- G2: tanned with 75% commercial chromium sulphate and 25% recovered chrome.
- G3: tanned with 50% commercial chromium sulphate and 50% recovered chrome.
- G4: tanned with 25% commercial chromium sulphate and 75% recovered chrome.
- G5: tanned with 0% commercial chromium sulphate and 100% recovered chrome.

Tanning and post-tanning steps were done at all tanned leathers according to the recipe in table (1). Qualitative and operational properties of the obtained leathers were assessed according to indices of chemical analysis, physico-mechanical analysis of the finished leather. Thickness, tensile strength, elongation, split tear strength, water absorption, permeability to water vapor, pH, and contents of ash, chrome and moisture were analyzed according to ASTM (2014).

Table (1). Tanning and post tanning steps recipe.

Step	Description		Time (min)	Notes
	%*	Added		
Tanning	8.0	Chromium sulphate	90	Chrome 33% basicity
Fixation	1.0	Sodium bicarbonate	60	Overnight, pH = 3.8- 4.2
Washing	100.0	Water	60	Drain float
	2.0	Soap		
Naturalization	100.0	Water	60	pH= 5.5 Drain float
	2.0	Sodium bicarbonate		
Dyeing and fatliquoring	150.0	Water	90	Bath temperature 40°C
	3.0	Black dye		
	6.0	Fish oil		
Fixation	0.5	Formic acid	30	Overnight
	0.5	Formic acid	30	
Washing	100.0	Water	10	Horse up - samming - dry hanging

*Percentages were calculated based on the pelt weight of the previous step.

4. Statistical Analysis

Data of physical and chemical properties of tanned leathers were analyzed using GLM procedure of SAS (2008) to evaluate the differences among produced leathers. The following model was used in the analysis:

$$Y_{ij} = \mu + T_i + e_{ij}$$

Where Y_{ij} is the observation taken on finished leather, μ is overall mean, T_i is a fixed effect of the i^{th} tanning group (1=G1, 2=G2, 3=G3, 4=G4 and 5=G5) and e_{ij} is the random error assumed to be normally distributed with mean=0 and variance= σ^2 .

RESULTS AND DISCUSSION

1. Chrome Shaving Waste Characteristics

The chemical properties of collected CSW were determined at the first stage of this study and their results are shown in table (2). The values of chemical characteristics were in agreement with previous literature (Sethuraman et al., 2013; Putshaka et al., 2015 and Abebaw and Abate, 2018). CSW is similar to ordinary chrome tanned leather in chemical characteristics. The reason is due to that CSW is mainly collagen fibers, which produced as a residue of shaving machine when adjusting leather thickness (BASF, 2007).

Regarding high protein content (TKN 12.66%) and low fat content (0.29 %) of CSW, it is for removing flesh, fats and globular proteins from pelts in beam house, before tanning, to purify collagen fibers from other filling materials (Dutta, 2008).

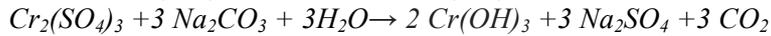
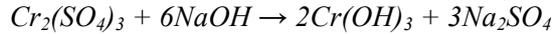
On the other hand, determined total energy of CSW (3515.33 cal/g) is tended to be lower than those found by Muralidhara et al. (1982), Chattopadhyay et al. (2011) and Forero-Núñez et al. (2015). That might be due to the very low content of fat of CSW in this study.

Table (2). Characteristics chrome shaving waste.

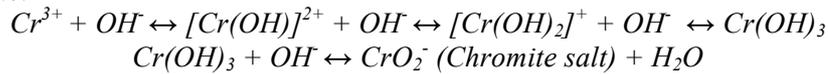
Parameter	ASTM	Value	Unit
Moisture content	D-6403	10.24	%
Ash content	D-2617	12.37	%
Chromium content	D-6714	2.51	%
Fat content	D-3495	0.29	%
Total Kjeldahl Nitrogen (TKN)	D-2868	12.66	%
pH value	D-2810	3.32	ml mol/L
Total energy (dry)	D-5865	3515.33	cal/g

2. Chrome Extraction Yield

Fig. (4) shows the extraction yield of different chrome recovery treatments. The effect of sodium hydroxide and sodium carbonate in hydrolysis is due to the following reactions equations, respectively:



As explained by Covington (2011), chromium (III) salts are stable in the range pH 2-4, where the basicity changes, but at higher values they will precipitate. This can be modeled in the following way, using empirical formulae:



The chemical effect of sodium hydroxide on the recovery ratio (97.87%) was more effective than sodium carbonate (91.92%) at the same concentration 10%. That means the greatest effect of caustic soda in alkaline hydrolysis due to the change in pH value (Mohamed et al., 2013 and Sutyasmi and Supratiningsih, 2014).

In incineration method because of the high operating temperatures, the residue from incineration is mainly ash. The ash contains chromium trioxide (Cr_2O_3), which is a very stable compound. Therefore, the extraction method by incineration was the highest (99.37%) and best extraction method compared with other alkaline hydrolysis methods.

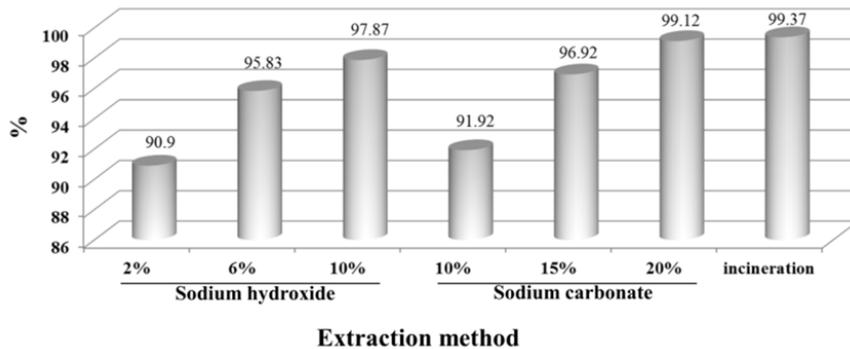


Fig. (4). Extraction yields of different extracted chrome shaving waste.

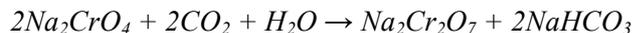
3. Maximization Chrome Extraction Yield of Incineration Method

Maximization of extraction yield was done in the second stage of this work by applying incineration under different levels of pH. Fig. (5) shows extraction yield and chrome basicity values of recovered chromium by incineration. The highest extraction yield was found when using sugar as reducing agent (99.65%), while using sodium sulphite at three levels of pH gave close values of yields.

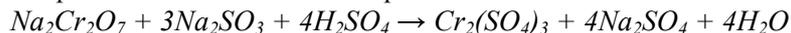
Covington (2011) explained that chrometrioide (Cr_2O_3) produces after incineration and converts into sodium dichromate ($Na_2Cr_2O_7$), which becomes a chrome tanning liquor, when treats with sugar or sodium sulphite. Sodium dichromate is fused with a base, typically sodium carbonate, at around $1000^\circ C$ in the presence of air:



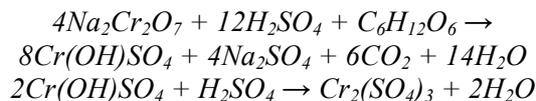
This step solubilizes the chromium and allows it to be extracted into hot water. Acidification of the resulting aqueous extract with sulfuric acid or carbon dioxide affords the dichromate:



The treatment with sodium sulphite is producing chromium sulphate in one step as described in the next equation:



While using sugar as a reduction agent is producing chromium sulphate in two consecutive chemical reactions as follows:



Therefore, when using sodium sulphite as a reducing agent, the basicity of produced chromium sulphate affected by the change in pH level. However, that is not done when using sugar, which produced chromium sulphate with 33% basicity. It is more common to be used in tanneries since it is more suitable to be cross linked with collagen fibers (Covington, 2011).

From the results of basicity and extraction yield, producing tanning liquor from CSW by incineration and acidification using sulphuric acid and sugar is considered the optimum method.

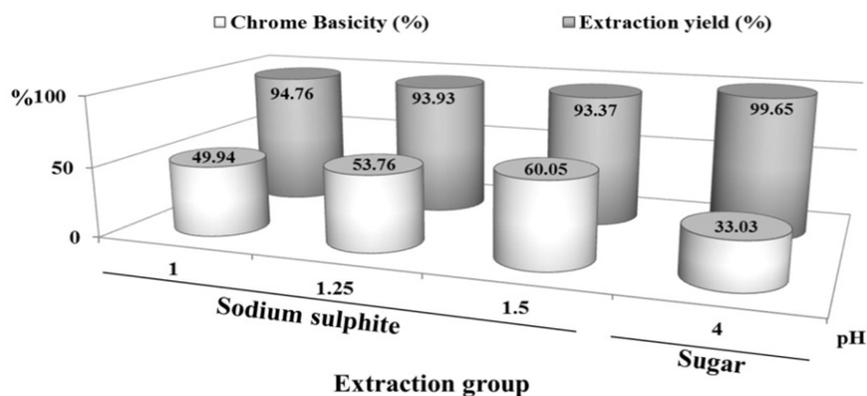


Fig. (5). Extraction yield and chrome basicity of recovered chromium by incineration.

4. Properties of Chrome Tanned Leathers

The physical and chemical properties of tanned leathers with different levels of prepared chromium sulphate were presented in tables (3) and (4), respectively. All values of physical and chemical properties were in acceptable range of commonly chrome tanned leathers, which means the similarity among all tanned groups and possibility of using them in different manufacturing purposes (BASF, 2007). Although the values of physical and chemical properties are in narrow ranges, tensile strength, ash content and fat content values showed significant differences. Tensile strength had significantly ($P<0.01$) the highest value (203.62 kg/cm^2) in tanned leathers with 50% of commercial chrome and 50% of recovered chrome. The values of ash and fat contents tended to increase by increasing addition of recovered chrome. Thus, tanned leathers with 100% of recovered chrome were the highest in ash content (6.60%) and fat content (10.13%). Dutta (2008) and Covington (2011) illustrated that in fat liquoring of chrome tanned leathers, the negatively charged ($-\text{OSO}_3^-$) ions penetrate into the chrome complex and therefore act as masking agents, which improve the crosslink among collagen fibers and fat liquor. It appeared that addition of recovered chrome may increase collagen fibers activity to be more covered with fat liquor, which increased fat content and thus ash content.

Table (3). Physical properties of tanned leathers with different additions of recovered chromium salts.

Property	Thickness	Strengths		Elongation	Water absorption		PWV	
		Tensile	Tear		2 h	24 h		
Unit	mm	kg/cm^2	kg/cm	%	%	%	$\text{mg/mm}^2/\text{h}$	
ASTM	D1813	D2209	D4704	D2211	D6015	D6015	D5052	
Recovered chrome addition	0%	0.91	158.82 ^b	36.41	59.01	214.12	228.05	5.48
	25%	0.91	201.91 ^a	40.80	55.43	194.46	209.73	5.80
	50%	0.89	203.62 ^a	38.51	57.82	192.43	199.96	5.40
	75%	0.91	156.76 ^b	34.46	58.10	185.35	197.11	5.56
	100%	0.90	184.99 ^{ab}	35.02	63.35	212.51	226.30	5.74
SEM	0.01	6.09	0.95	1.39	6.19	6.46	0.08	
Significance	NS	**	NS	NS	NS	NS	NS	

Means in the same column having different superscripts are significantly different ($P<0.05$).

ASTM: determination method according to American Society for Testing and Materials.

PWV: permeability to water vapor.

SEM: standard error of the mean.

NS: not significant, ** $P<0.01$

Table (4). Chemical properties of tanned leathers with different additions of recovered chromium salts.

Property	pH	Moisture	Fat	Ash	Cr	
Unit	ml mol/L	%	%	%	%	
ASTM	D2810	D6403	D3495	D2617	D6714	
Recovered chrome addition	0%	3.73	15.66	7.33 ^b	5.77 ^c	3.02
	25%	3.67	15.19	8.95 ^{ab}	5.45 ^c	3.31
	50%	3.85	15.53	10.39 ^a	6.15 ^b	3.91
	75%	3.68	15.45	9.96 ^a	6.24 ^{ab}	2.51
	100%	3.71	14.74	10.13 ^a	6.60 ^a	2.77
SEM	0.02	0.12	0.36	0.10	0.17	
Significance	NS	NS	*	**	NS	

Means in the same column having different superscripts are significantly different ($P<0.05$).

ASTM: determination method according to American Society for Testing and Materials

SEM: standard error of the mean

NS: not significant, * $P<0.05$, ** $P<0.01$

CONCLUSION

The results of the current study pointed out the efficiency of extracting chromium salts from chrome shaving wastes and converting it into tanning liquor in order to achieve some environmental and economic benefits. Incineration of chrome shaving wastes is considered as an easy method to obtain high yield of chromium salts and convert it into chrome sulphate (33% basicity) by acidification with sulphuric acid, then add sugar as a reducing agent. Furthermore, it generates good thermal energy, which can be utilized in tannery to save energy cost. However, the rising fumes from incineration need further studies to avoid their negative impact on the environmental.

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معالجة مخلف سلالة الكروم لتقليل أثره البيئي

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يعد مخلف سلالة الكروم هو أحد أكبر مخلفات دباغة الجلود، والذي يسبب خسارة بيئية وإقتصادية إذا لم يتم استخدامه بطريقة سليمة. تهدف هذه الدراسة لإستخلاص الكروم من مخلف سلالة الكروم بإستخدام التحلل القلوي أو الترميد، وذلك لإعادة إستخدامه في دباغة الجلود. تم استخدام هيدروكسيد وكربونات الصوديوم في التحلل الكيميائي ومقارنتها بطريقة الترميد في نسبة إستخلاص الكروم. تم دباغة خمسة وعشرين جلد من جلود الأغنام بإستخدام الكروم المستخلص والتجاري قاعدية 33% لمقارنة تأثيراتهم في الدباغة. أظهرت النتائج من بين المعاملات القلوية أن الصودا الكاوية كانت أكثر فاعلية من كربونات الصوديوم في الإستخلاص، ولكن طريقة الترميد كانت أفضل طرق الإستخلاص، وذلك بسبب إرتفاع نسبة استخلاصها (37.99%). إستخدام السكر كمادة مختزلة كان أفضل من كبريتيت الصوديوم، وذلك لتحضير سائل الدباغة، حيث أدت إلى زيادة نسبة الكروم وأنتجت كبريتات كروم 33% قاعدية، كذلك فهي مناسبة أكثر لدباغة الجلود. أوضحت النتائج أن معظم الخصائص الطبيعية والكيميائية، ماعدا محتوى الدهن والرماد، كانت متشابهة في الجلود المدبوغة بالكروم المستخلص وتلك المدبوغة بالكروم التجاري. وعلى هذا فإن كل الجلود المدبوغة في هذه الدراسة كانت مناسبة للإستخدام في تصنيع الجلود.