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# Spectrophotometric Study of Cr(VI) in Shaving Dust, Waste Liquor, and Wet Blue Trimming Samples

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**Abstract**

Humans are exposed to chromium ions from leather materials owing to everyday contact with different need based activities. The concern is that Cr (VI) is extremely toxic and may cause contact allergic dermatitis on the skin and may also be a trigger for a plethora of diseases, hence it is essential to monitor the presence of chromium as Cr (VI). For the determination of levels of chromium (VI) the shaving dust samples from four different tanneries and chrome liquor, and wet blue leather trimmings from three different tanneries were collected. The fat content was determined in collected shaving dust and wet blue trimmings with the aid of Soxhlet extraction method using petroleum ether- ethanol solvent. Then the extraction of Cr (VI) from the samples was done following the standard procedure of society for Leather Chemistry with some modification. Afterwards, UV- visible spectrophotometric determination of Cr (VI) was carried out through the complex formation with 1, 5-diphenylcarbazide (DPC). The weight percentages of fat content were found to be in the range of 2.98-3.71. Analytical grade potassium-dichromate reagent was used as a standard source of Cr (VI) in calibration process. Among the collected four shaving dust samples in three samples were found to contain Cr (VI) in the range of 1.96-2.19 mg/kg. In three analyzed wet blue trimmings samples the Cr (VI) was found to be from 1.59 to 1.86 mg/L. The amount of hexavalent chromium was found 1.27 mg/L in one chrome liquor sample out of three samples.

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## 1. Introduction

Chrome tanning is currently the most popular tanning process in the leather industry due to the excellent qualities of chrome-tanned leather like high hydrothermal stability, good dyeing characteristics and softness. There is an average estimation that approximately 90% of the raw hides in the world are tanned by chrome-tanning method but it is controversial due to high Cr (III) content in the wastewater released from tannery (Supamas & Aree, 2009). Around 65% of the chrome added is absorbed by leather in the conventional chrome tanning process, and the rest is discharged in to spent tan liquor (about 2-4 $gL^{-1}$), resulting in serious environmental pollution and a great waste of chrome resource. However, chrome may be released from leather in post tanning processes like washing, re-tanning, dyeing and fat-liquoring because of the existence of free and unstably combined chrome in leather (Adam et al, 2018; Jian et al. 2012). Hexavalent chromium is known to be toxic to animals and humans, so it needs to be carried out under extremely high safety precautions from professional chemical companies only in in-house systems akin to many other hazardous chemical intermediates. Therefore, the need for a scientific risk assessment of the use of chromium for leather tanning is required, in order to exclude these oxidations during normal conditions of usages. This has been done in many scientific studies and the studies are the base for legal requirements to protect consumers (Tegtmeyer, D. and Kleban, M., 2013). Moreover, chromium is a major water pollutant, usually as a result of some industrial pollution including tanning factories, steel works, dyeing or chromium plating, wood preservation, and artificial fertilizers. For this reason, determination of chromium in environmental samples like freshwater, wastewater samples and soil samples has become inevitable (Narin et al, 2006). Due to the different toxicity of chromium species, total chromium measurement cannot be used to determine the actual environmental impact. Thus, chemical speciation of chromium in environmental samples is necessary to accurately assess pollution levels (El-Shahawi et al, 2005). Recently there is an upsurge of interest for rapid and sensitive analytical methods for the determination and speciation of chemical forms of toxic metal ions in environmental samples. Trivalent chromium has lower human toxicity and is also less environmentally mobile due to its low solubility and its tendency to be less absorbed or complexed by organic molecules making its disposal an acceptable environmental risk (Pillay et al., 2003; Rinehart et al., 1997; Bartlett and James, 1979). With its large positive charge, Cr (III) is attracted to negatively charged soil particles, and has been found to associate with the smallest size fractions in soils. Therefore, Cr (III) is less mobile in soils than Cr (VI).

Hexavalent chromium is an oxyanion commonly occurring as relatively water soluble chromate ($CrO_4^{2-}$) or dichromate ($Cr_2O_7^{2-}$). In temperate region soils, where negatively charged clay minerals predominate, these Cr (VI) anions are repelled by the negative charge on the soil particles (Pettine et al., 1998; Rinehart et al., 1997; Zachara et al., 1987). The Cr (VI) form is a strong oxidizer which makes it the more toxic form to biological systems. Determination of Cr (VI) especially in solid samples is regarded as one of the most challenging speciation tasks due to inter-conversions between Cr (VI) and Cr (III) in the course of the analytical process. (El-Shahawi et al, 2005; Yamaguchi et al., 2006; Borai et al., 2002; Mori et al, 2001; Kristiansen et al., 1997; Frias and Sanchez de Rojas, 1995; Bartlett and James, 1979). In acidic solutions, especially in the presence of dissolved organic compounds and sulfides, there exists a risk of Cr (VI) reduction to Cr (III). In an alkaline medium on the contrary, Cr (III) oxidation may occur (Pettine et al., 1998). Hence, the selection of an appropriate analytical method should take into account: the type of matrix (natural water, effluents, leather, sediments, biological materials, etc.), the concentration level of the chromium forms and the presence of substances, which may disturb the chemical equilibrium between the chromium oxidation states (Bobrowski et al., 2004; Yamaguchi et al., 2006). The usual acid digestion for preparation of solids is inappropriate because Cr (VI) is reduced in acidic media if any reductants, such as organics or oxidizable inorganics, are present, and this is typically true for environmental samples (Borai et al, 2002; Yamaguchi et al, 2006; Kristiansen et al, 1997).

In order to determine the oxidation states of chromium in solid samples by most analytical techniques, it is required that prior to the analytical determination, the chromium must be extracted from the solid, as has been recently described by several authors (Korolczuk and Grabarczyk, 2005; Borai et al, 2002; Yamaguchi et al., 2005). In the study of the effect of Cr (VI) on a solid sediments and its leachability, (Morales et al., 2004) where extraction was followed by analysis using UV-vis spectroscopy with DPC. This work is focused especially on commonly available spectrophotometric method based on the reaction with 1, 5-diphenylcarbazide, because not every laboratory is equipped with modern devices for this pollutant measurement. Special attention was focused on the variability possibilities of the basic spectrophotometric method with practical demonstration of individual variants, some critical points and moments in laboratory conditions using real samples containing hexavalent chromium. Among others, there are mentioned other spectrophotometric methods with which we do not meet often.

## 2. Materials and Methods

**2.1 Sample Collection and Preparation**: Shaving dust samples were collected from four different tanneries, chrome liquor and wet blue leather trimmings were collected from the three different tanneries of “Tannery Industrial Estate” located at Savar, Hemayetpur, Dhaka, Bangladesh. The collected samples were stored with labeling in laboratory until analysis was carried out. During the preparation of samples, the dry wet blue trimmings were reduced into small pieces by cutting. Sparing the mill and yielding a more uniform ground sample. In case of wet samples, the sample was dried at a temperature not exceeding 50 $^{o}$C. Conditioning was done in an atmosphere of temperature 20 ± 2 $^{o}$C and relative humidity 65 ± 2 % for 24 hours before grinding. The whole sample were ground and mixed the leather powder and stored in a clean, dry, and staunch container.

**2.2 Chemicals, Reagents and Solvents:** The certified standards; potassium di-chromate, 1, 5- Diphenylcarbazide (DPC) (99% purity) were purchased from Dr. Ehrenstorfer, Germany. Analytical grade Di-potassium hydrogen-phosphate, aluminum sulfate, sodium bisulfate, sodium hydroxide were purchased from Scharlau, Spain. Analytical grade solvent such as acetic acid and acetone were purchased from Sigma Aldrich. Sulfuric acid (98%), phosphoric acid and sodium chloride were purchase from Merck, Germany.

**2.3 Apparatus and Equipment**: Calibrated balance, volumetric flasks and pipettes were used for the analysis. UV-visible spectrophotometer (UV- 1800 PC, Shimadzu, Japan) was calibrated by local agent of the company (AQC). All the glass apparatus was rinsed by petroleum ether and dried it in the oven at 102°C and after removing, they were kept in the desiccator until using.

### 2.4 Preparation of different reagent's solutions

**2.4.1** Potassium di-chromate [$K_2Cr_2O_7$] solution: 0.071 g analytical grade $K_2Cr_2O_7$ was weighed by analytical balance and transferred into 100 mL volumetric flask. Then 100 mL distilled water was added to prepare the standard 25 ppm solution of chromium (VI).

**2.4.2** 1, 5- Diphenylcarbazide (DPC) [$C_{13}H_{14}N_4O$] solution: DPC was prepared in 100 mL volumetric flask. For this purpose, 1.0 g of DPC was weighed by electric balance and it was transferred into 100 mL volumetric flask and it was dissolved in acetone and finally 1 drop acetic acid was added to it.

**2.4.3** Di-potassium hydrogen phosphate [$K_2HPO_4$. $3H_2O$] solution: 4.56 g di-potassium hydrogen phosphate was measured and transferred into 100 mL volumetric flask and it was up to the marked using distilled water.

**2.4.4** Ortho-phosphoric [$H_3PO_4$] acid: 50 mL concentrated O-phosphoric acid (d = 1.71 g/mL) was diluted with 30 mL water in 100 mL volumetric flask.

**2.4.5** Sodium hydroxide [NaOH] solution: 10.0 g sodium hydroxide was dissolved in 50 mL distilled water in a volumetric flask.

**2.4.6** Aluminum sulphate [$Al_2(SO_4)_3$] solution**:** 4.67 g aluminum sulphate was dissolved in 25.0 mL distilled water in a 100 mL beaker.

**2.2.7** Sodium sulphite [$Na_2SO_3$] solution: 2.95 g sodium sulphite was measured by using analytical balance and it was transferred into a 100 mL beaker. Then, it was dissolved in 25.0 mL of distilled water.

### 2.5. Determination of fat content

10.0 ± 0.01 g of the prepared sample was weighed and placed evenly into the glass bell. The trimmings and shaving dust were covered with a thin layer of cotton wool. The extraction flask was dried with two glass beads in it by heating for half an hour at 102 ± 1 $^oC$. Weighed was taken after cooling in a desiccator. The continuous extraction was started with Petroleum ether. The extract in the oven was dried for four hours at 102 ± 1 $^oC$ (added 1-2.0 mL of ethanol, when drops of water were visible before drying). After cooling for 30 minutes weighed was taken in the desiccator. The drying, cooling and weighing was repeated at least twice more, but with drying period of 1 hour until either the further loss in mass did not exceed 10 mg, or the total drying time equals 12 hours.

Extractable fat/lipid, percentage by mass = $(100 \times M)/M_o$, where M is the mass of extract and $M_o$ is the mass of sample used.

### 2.6 Standard potassium dichromate solutions preparation for calibration process

The calibration process for determination of Cr (VI) was done by using potassium dichromate ($K_2Cr_2O_7$) as a source of hexavalent chromium as it is primary standard substance. Working standard Cr (VI) concentrations of 0.125 ppm, 0.250 ppm, 0.500 ppm, 0.750 ppm and 1.000 ppm were prepared in five different 100 mL volumetric flask through dilution of primary standard 25 ppm stock solution (2.4.1). To carry out this, 0.5, 1.0, 2.0, 3.0 and 4.0 mL of 25 ppm $K_2Cr_2O_7$primary standard solutions was diluted to 100 mL in respective volumetric flasks. Then 2.0 mL 1, 5- diphenylcarbazide solution (2.4.2) was added and followed by the addition of 2.0 mL ortho- phosphoric acid solution (2.4.4). Afterwards, these were made up to 100 mL solution by adding distilled water and kept them 15 min to be stabled. Finally, the absorbance of these solutions was taken by using the UV- visible spectrophotometer and the wavelength was fitted at 540 nm.

### 2.7 Extraction and estimation of Cr (VI) in Shaving dust and Wet blue Trimming samples

Shaving dust samples were blended using blender machine and they were dried in an oven at 105 $^oC$. Triplicate of 10.0 ± 0.01 g of dried and blended shaving dust sample was placed into a 250 mL conical flask. Then, 2.0 mL di-potassium hydrogen phosphate ($K_2HPO_4$. $3H_2O$) buffer solution (2.4.3) was added. After that, 98 mL of distilled water was added. The solution of conical flask was shaken in shaking machine for 2.5 hours. After completing of shaking the shaving dust samples were filtered through Whatmann filter paper and 25.0 mL extracted solution was taken as a sample solution and it was transferred into 100 mL volumetric flask. Then 2.0 mL O-phosphoric acid solution (2.4.4) was added into it and left for 30 minutes to be stabled. Furthermore, 2.0 mL 1,5-Diphenylcarbazide (DPC) solution (2.4.2) was added and distilled water was added up to 100 mL. This solution mixture was kept 5 to 10 minutes to be stabled. Finally, absorbance of Chromium (VI)-DPC complex was measured using UV-visible spectrophotometer at the 540 nm wavelength. The extraction and estimation of Cr (VI) from wet blue trimming samples were done following same procedure like shaving dust sample (Adam, 2018; SLC, 1996).

### 2.8 Extraction and estimation of Cr (VI) from chrome liquor samples

A triplicate of 100 mL of chrome liquor was placed into a 250 mL conical flask. Then added 1.0 mL di-potassium hydrogen phosphate ($K_2HPO_4$. $3H_2O$) solution. Then it was shaken for 15 minutes and then required amount sodium hydroxide (NaOH) (2.4.5) added to get pH 7.5-8.0. Afterwards, 1.0 mL aluminium sulphate ($Al_2(SO_4)_3$) solution (2.4.6) was added and it was shaken again. pH was measured again and kept at 7.5-8.0. Then 1.0 mL of sodium sulphite ($Na_2SO_3$) solution (2.4.7) was added. After completing extraction the filtration was done by using filter paper and 25.0 mL filtered was taken as a sample solution into a 100 mL volumetric flask. After that, 1.0 mL of sodium hypochlorite (NaOCl) solution was added and left 1 minute to stand. Then 2.0 mL O-phosphoric acid solution (2.4.4) was added into it and left for 30 minutes. 2.0 mL of 1,5- diphenylcarbazide (DPC) solution (2.4.2) was added. Furthermore, the solution was made up to the 100 mL with distilled water and kept 5-10 minutes for stable (Adam, 2018; SLC, 1996). Finally, absorbance of Chromium (VI)-DPC complex solution was measured using UV-visible spectrophotometer at the 540 nm wavelength.

## 3. Results and Discussions

### 3.1 Percentages of fat

Saturated and unsaturated fat may interfere the formation of Cr (VI)–DPC complex as fat can form emulsion during extraction process. For this reason fat content was determined (i.e fat was removed from leather sample through extraction). Varying amounts of fat/lipid were found in analyzed shaving dust and wet blue leather trimming samples and the overall fat content was found to be in the range 2.98 – 3.71 %. The percentage of fat content was found maximum 3.71% in wet blue trimmings sample in Tannery 1 and minimum 2.98 in shaving dust sample in Tannery 3 which were below in average range of 3-6% found in literature. However, the rest of the samples contained less than the acceptable range. So determination of Cr (VI) would not be hindered and extraction as well as absorbance of Cr (VI) would not be prejudiced.

**Table 1:** Percentages of fat content in shaving dust and wet blue leather trimming samples

| Sample type | Sample from different tanneries | Initial mass (Mo) g | Extracted mass (M) g | % of fat content |
|---|---|---|---|---|
| **Shaving dust** | Tannery 1 | 10.0 | 0.325 | 3.25 |
| | Tannery 2 | 10.0 | 0.313 | 3.13 |
| | Tannery 3 | 10.0 | 0.298 | 2.98 |
| | Tannery 4 | 10.0 | 0.302 | 3.02 |
| **Wet blue leather trimmings** | Tannery 1 | 10.0 | 0.371 | 3.71 |
| | Tannery 2 | 10.0 | 0.342 | 3.42 |
| | Tannery 3 | 10.0 | 0.309 | 3.09 |

### 3.2 Calibration Curve

The calibration curve was made according to the absorbance of the known concentrations 0.125 mg/L, 0.250 mg/L, 0.500 mg/L, 0.750 mg/L and 1.000 mg/L of the standard Cr ( VI) solutions. The regression coefficient ($R^2$) was 0.9995 and it showed a very good linearity of the calibration curve which was almost 1.0.

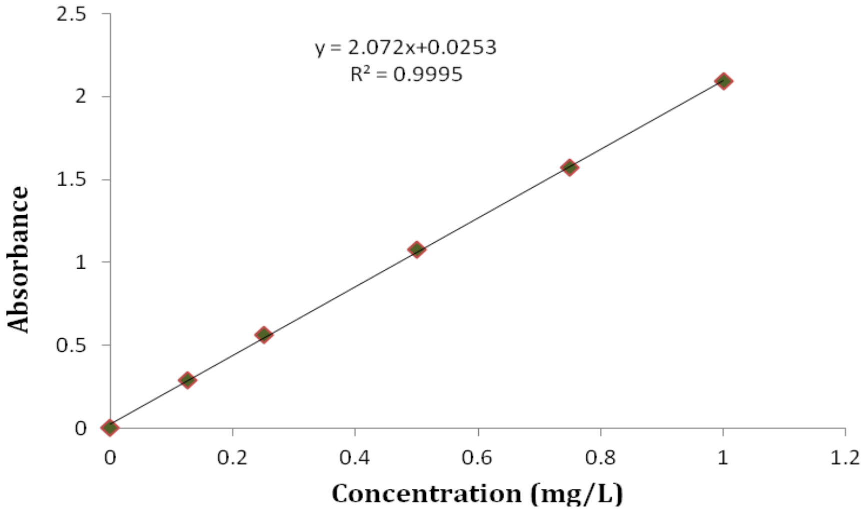

**Fig. 3.1 Calibration curve of chromium (VI)-1,5-DPC complex**

### 3.3 Levels of chromium (VI)

Extraction and determination of hexavalent chromium were successfully carried out by UV-visible spectrophotometric method with some modifications. To measure the concentration of the toxic metal Cr (VI) the fat content of the samples were determined as fat content may accelerate the transition of Cr (III) to Cr (VI). It is critical to monitor the toxic Cr (VI) with the most suitable analytical procedure. The UV–visible 1,5-diphenyl-carbazide spectrophotometry method has a low detection limit of 15 µg/L for chromium determination, which was an advantage of this analytical method. Various shaving dust samples from different tanneries were analyzed. Hexavalent chromium was detected in three samples out of four shaving dust samples within the range of 1.96-2.19 mg/kg (Table 2). The highest amount of Cr (VI) was found in Tannery -1. From the analyzed data it can be said that the amount of Cr (VI) in shaving dust was in acceptable range as the maximum value for Cr (VI) is 3 mg/kg according to REACH Regulation (Hess, 2016; Iva & Michaela, 2008). There was no Cr (VI) detected in Tannery-4 and the lowest amount of Cr (VI) found in Tannery-2 which was 1.96 mg/kg. The amount of hexavalent chromium was found 1.27 mg/L in one chrome liquor sample out of three samples, which was detected in the sample of Tannery-1. However, No Cr (VI) was detected in the chrome liquor sample of Tannery-2 & 3. In addition, Varying amounts of Cr (VI) were found in the wet blue leather trimming samples of three different tanneries. It can be predicted from the analyzed data that the amount of wet blue leather samples were in minimum range and the found amount was in acceptable level against toxicity limit of Cr (VI). From the experimental result, the highest concentration of Cr (VI) was found 1.8565 mg/kg and the lowest value of the concentration of Cr (VI) was found 1.5965 mg/kg. How much and what types of environment requires for the conversion of trivalent chromium to hexavalent form depend on range of factors, including the quality of used reagents, temperature control, and the circumstances within the tanning process. What is more, Hexavalent chromium in leather samples may come about due to mistakes in local production or can emerge later in the manufactured leather. Chinese researchers have shown that leather containing high proportions of unsaturated fatty acids results in the formation of chromium (VI). Irregularities in the tanning process and certain additives, combined with the presence of chromium (III) compounds, can result in the formation of chromium (VI) compounds. Meanwhile, scientists are targeting chrome tanning agents, which are provided with additives that counteract the emergence of chromium (VI). However, these materials are expensive, and the consumer should not assume that all imported goods are safe. In such cases, the customer can only rely on responsible manufacturers who undergo voluntary inspections of their batches and can prove it by having their goods tested by reputable independent testing institutes. A study in 2011 found that the use of heat and adhesives in shoe production promotes the formation of chromium VI. Leathers with a higher proportion of tannin chromium (III) are more prone to the formation of chromium (VI) than leather with lower chromium (III) content (Leather D., 2009).

From table 2, it is evident that, overall, the chrome liquor samples contained the lowest level of Cr (VI), in shaving dust samples were contained higher amount, somehow, these values are below toxicity limit. The relative standard deviation (RSD) was calculated as triplicates analysis were carried out. The highest RSD value was found around 8.51 % in chrome liquor which is far less than 20% (maximum RSD allowable limit) and the lowest value calculated in shaving dust which was 1.49 %. The RSD values found in this study eventually show the very good precision of this analytical analysis. Moreover, among the three tanneries, Tannery -1 discharged the highest amount of Cr (VI) in tem of shaving dust, chrome liquor and wet blue trimmings samples and obviously the Tannery-3 released the lowest in environment.

**Table 2:** Levels of Cr (VI) in different samples from different tanneries

| Types of sample | Sample from different tanneries | Concentration (mg/Kg) | RSD |
|---|---|---|---|
| **Shaving dust** | Tannery 1 | 2.1934 ± 0.0234 | 1.1829 |
| | Tannery 2 | 1.9640 ± 0.0287 | 1.4591 |
| | Tannery 3 | 1.9659 ± 0.0166 | 3.8466 |
| | Tannery 4 | ND | ---- |
| **Chrome Liquor** | Tannery 1 | 1.2692± 0.2152 | 8.509 |
| | Tannery 2 | ND | -- |
| | Tannery 3 | ND | ---- |
| **Wet blue leather trimming** | Tannery 1 | 1.8327 ± 0.1162 | 6.3398 |
| | Tannery 2 | 1.8565 ± 0.1390 | 7.488 |
| | Tannery 3 | 1.5965 ± 0.0849 | 5.3204 |

**N.B:** ND means Not Detected

## 4. Conclusion

From the present study of hexavalent chromium in the analyzed samples it can be evident that the level of Cr (VI) was below the maximum residual limit for leather sample 3.0 mg/kg. Therefore, the level of Cr (VI) found in analyzed samples is out of risk with respect to maximum residual limit (MRL) as analyzed data was below MRL, but continuous and prolong exposure of Cr (VI) may cause a great threat to human health as a result of bioaccumulation and bio magnification. Thus, it is advisable to avoid prolonged contact with leather materials containing a high amount of chromium. Moreover, the current study was area-specific, giving a holistic picture of the heavy metals especially chromium of our leather industrial environment. Further work is required to determine the overall picture of the pollutants in the aquatic and atmospheric environments around the industrial area.

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