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Recovery and Reuse of Chromium from Tannery Waste Chrome-liquor using Solar Evaporation Process

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ABSTRACT

Recovering and reusing chrome from chrome effluent is critical for improving the mass balance of the tanning process and implementing the zero liquid discharge concept in the tanning industry. Conventional chrome recovery methods require a large amount of chemicals or energy to recycle the chromium. Chrome recovery by solar evaporation has not been implemented yet in the tanning industry. It is mainly practised in seawater salt extraction. In this study, we demonstrate the construction of highly efficient solar evaporators, using photothermal materials. Our focus is on efficient solar-to-vapour conversion and chrome recovery. The average evaporation rate was calculated and found 38.69 mL/hour, which remained almost steady within 2.5 months of data collection. The highest temperature observed during the process was 108 °C and 62°C in the focal region and condenser respectively. After collecting the solid chromium, basicity was measured at 85.02%, and treated with sulphuric acid to achieve 33% basicity. Recovered chrome and fresh chrome at 0:8, 5:3, and 8:0 were used for chrome tanning and tested for various chemical and physical parameters of tanned leather. The shrinkage temperature of the produced leather was found to be more than 100 °C. Physical characteristics such as tensile strength and percentage of elongation test, stitch tear strength, ball bursting strength and flexing endurance showed better results than conventional methods. This new kind of research offers a promising solution that can reduce the environmental impact of the tanning industry while also providing a sustainable source of chromium for a low-cost strategy and high-performance evaporation approach.

KEYWORDS

chrome tanning, solar evaporation, tannery effluent, chrome recovery, sustainable tanning

INTRODUCTION

Chromium salts are used in leather manufacturing, electroplating, paint and pigment manufacturing, metal plating and other applications [1]. Chromium is not biodegradable and tends to accumulate in living organisms, causing serious diseases and disorders. It significantly contributes to environmental pollution through tannery waste liquor [2]. 80–90% of tanneries worldwide adopt chrome tanning [3]. Significant amounts of chrome salts are applied in tanning (~20000 mg dm^{-3} Cr) and approximately 60% of this amount is taken up by the leather in the tanning. The remaining chromium (~8000 mg dm^{-3} Cr) is spent in the tannery waste liquor [4]. Recovering and reusing chrome from chrome effluent is critical to improving the mass balance of tanning and implementing the zero liquid discharge concept. Although several techniques have been developed for chrome recovery and reuse, many of them are not cost-effective and have adverse effects. Chromium recovery and detoxification are practised in modern units, but still, many of the older and smaller units resort to conventional effluent treatment [5,6]. Precipitation, oxidation/reduction and lime neutralization have traditionally been the most commonly used [7]. Other possible methods include chemical reduction followed by precipitation, ion exchange, membrane technologies and adsorption by several types of adsorbents, such as activated carbon, bone charcoal, cork, Yohimbe bark wastes, waste-activated sludge, fly ash, natural zeolites and others [8-10]. Most of these methods and materials have drawbacks such as high capital or operational costs and are therefore uneconomical for small-scale plants. However, conventional methods require a large amount of chemicals or energy to recycle chromium. Common chrome recycling techniques used in chrome recovery operations require the installation of storage tanks, pumps, and filters, all of which can be costly. The principle of chrome recovery is based on extracting chrome from floats containing residual chrome through a series of steps involving precipitation, separation, and subsequent redissolution in acid for reuse. The precipitation is achieved with alkali substances such as sodium hydroxide, sodium carbonate, or magnesium oxide, which enhances coagulation when combined with polyelectrolyte. This is followed by thickening and dewatering the voluminous sludge through filtration or settling of the suspension, decantation of the supernatant (eliminating the need for a filter press), and the subsequent acidification of the relatively dense precipitate [11].

Solar energy is one of the sources that is most frequently used due to its reasonable installation costs and lengthy operational lifespan without maintenance [12]. Solar energy is so potent and plentiful that the world's yearly energy needs are met by only 30 minutes of solar radiation hitting the planet. Additionally, it is affordable, environmentally friendly, and almost endless. The majority of the planet is exposed to intense solar radiation and has a strong probability of making use of solar energy [13]. The annual average of diffuse solar radiation ranges from 1.8 to 2.2 kWh m^{-2} day^{-1}, which is comparable to global solar radiation [14,15].

Bangladesh, which has a surface area of 147,500 km^2, is ideally situated for the usage of solar energy. It receives an excellent amount of solar radiation on a daily average of 4.64 kWh/m^2, which can be used for a variety of purposes [16]. This solar thermal energy may thus be used to recover chromium from tannery effluent.

Reviewing the literature authors have not found research on the removal of chromium via solar energy from tannery waste effluent. All the mentioned processes are very costly. That is why a low-cost process is needed to tackle this problem. The process should have an ease of operation so that the feasibility of the process can be higher and used with the least technological knowledge. The goal of solar evaporation is to treat regenerative/non-biodegradable wastewater that is not effectively treated using conventional biological and chemical processes employed in the industry [17]. Higgins et al. demonstrated that the carbon fabric solar evaporator effectively removes inorganic contaminants, such as NaCl from saline water, and organic contaminants from industrial effluent through the evaporation-condensation mechanism [18]. Since solar thermal energy is abundant in Bangladesh, heavy metals like chromium can be removed and recycled to produce a traditional chrome liquor.

A low-cost solar evaporator was developed to recover and reuse chromium from tannery waste chrome liquor. The quality of the recovered chrome was evaluated by testing the percentage of Cr_2O_3. The recovered chromium was then used in the tanning process at different ratios to determine its effectiveness. Finally, the leather produced from the fresh and recovered chromium mixture was subjected to various physical, chemical, and characterizing tests.

Subsequently, the development of a low-cost methodology using easily available materials that can effectively recycle chrome is needed. In this research, a solar evaporator has been developed cost-effectively using natural solar energy except for chemicals or other energies to recover chromium from waste chrome liquor. To recover chromium from waste chrome liquor, a solar evaporator uses natural sun energy rather than chemical or other energies. The recovered chrome has been converted to 33% basicity as the industry standard for chrome tanning. A new environmentally friendly and sustainable approach to recovering chromium from tannery effluents and reusing it in leather manufacturing has been addressed and reported.

EXPERIMENTAL

Materials and Methods

A parabolic dish, steel stand, screw, glass, aluminium bowl, aluminium foil paper etc. were bought from the local market. The parabolic dish is made of 0.15 ″ thick stainless steel, grade 304, with a glass thickness of 0.12 ″ respectively. 10 L of waste chrome liquor have been collected from Tannery Industrial Estate Dhaka (TIED), Savar, Dhaka, Bangladesh. Immediately following the completion of the chrome tanning, the used chrome liquor was collected. The high-density plastic bottle was subsequently processed and used for storage. 0.5M HNO_3 was used to treat the bottle before storing the chrome liquor. The liquor was then aerated by a locally made aeration instrument. Since tannery waste chrome liquor contains different insoluble organic matter. The liquor was filtrated with glass wool and preserved in the refrigerator at 4 °C. Commercial basic chromium sulfate (BCS) supplied by Stahl, India and other tanning chemicals have been collected from the local market in Dhaka, Bangladesh. All other reagents used were of analytical grade.

Fabricated Solar Evaporator

The solar evaporator is developed with the required materials and conducts the evaporation process. The concave parabolic dish is mainly made of steel. To capture about 90% of the sun's rays by a concentrator, the reflecting plain mirror pieces (2″ × 2″) have been cut into forms, fixed with screws and adhered to the parabolic dish's reflective surface. Then, the focus of the evaporator was assessed by formula and fixes. The experimental receiver, constructed using aluminium and coated with a thin layer of black paint to minimize solar ray reflection, was positioned at the focal point of the parabolic dish. This arrangement aimed to investigate the impact of radiation and temperature on the receiver's aperture. Subsequently, the apparatus was installed on the rooftop for chromium recovery. Figure 1 illustrates the complete setup of the solar evaporator.

Determination of the Evaporation Rate and Basicity

Once the installation of the device was complete, the differences between the initial and final volumes concerning time were compared to determine the rate of evaporation. The tanning behaviour of a chrome tanning liquid is closely connected to its basicity. Basicity measurement was conducted to know about the percentage basicity of chromium which is important for leather tanning.

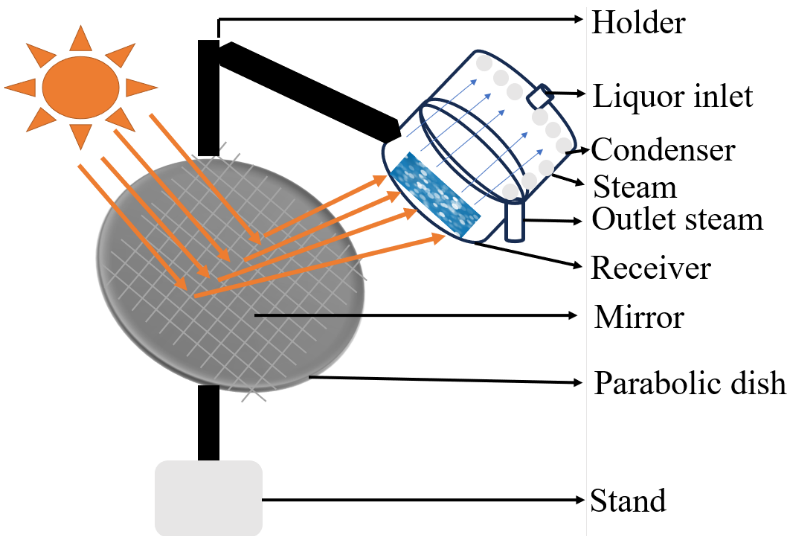

Figure 1. Schematic diagram of the chrome liquor heated system for chrome recovery

Adjustment of the Basicity

Once the basicity is known after titration, acid treatment is introduced to adjust the basicity to 33% for treating it in the chrome tanning process due to its requirement for proper tanning. Chromium penetrates and reacts very well with collagen fibre at a 33% basicity level.

Application of Recovered Chrome

Recovered chromium is applied to leather in the tanning process with different ratios such as 5% recovered chromium + 3% fresh commercial chromium (5:3), 8% recovered chromium and 8% fresh commercial chromium. 3 types of crust leather are made by adopting the above ratios.

Physical and Chemical Test of the Manufactured Leather

A variety of physical tests, including ball bursting and lastometer tests, stitch tear strength, tensile strength and percentage of elongation, flexing endurance, shrinkage temperature (SATRA, Ser. No.:032388), etc., are used to evaluate the leather made from various combinations of fresh chrome and recovered chrome followed by standard official methods of analysis named Society of Leather Technologists and Chemists (SLTC) [19]. All the tests were performed three times for both parallel and perpendicular to the backbone and reported the mean value with standard deviation in this study. Before the testing, the leather was conditioned for 48 h at a temperature of 23±2 °C, and relative humidity of 65±2% following the ISO-2419 standard.

RESULTS AND DISCUSSION

A solar collector is a device used to transform the sun's energy into heat. To concentrate solar energy and transform it into medium-high temperature thermal processes, solar thermal collectors have been widely used. Solar dish collectors are typically such types of devices that focus solar energy in a specific area known as the focal point. The parabolic dish bought from the market consists diameter of 0.59 meters. Its inside is coated with a reflecting layer, primarily made up of mirrors, which reflect solar rays onto the surface of a receiver located in the concentrator's focal position. The focal distance is given by the following expression [20]:

$$f = \frac{D^2}{16h} \quad (1)$$

where the focal distance of the parabola is f, the opening parabolic surface's diameter is D, and the depth of the parabolic dish is h.

Table 1. Characteristics of the Solar Concentrator

Diameter of the parabola (m)	0.59
Depth of parabola dish (m)	0.03
Area of collector (m^2)	0.27
Depth of the collector (m)	0.05
Focus distance f (m)	0.43

The receiver is fixed at the parabolic dish's focal point. Sunlight strikes this parabolic dish and concentrates at the focal point shown in Figure 1. The dish was positioned on the structure in such a way that it could be manually operated according to the direction of the sun's position.

The waste chrome liquor stored in the vessel is heated and concentrated. The receiver's exterior is coated in black paint to increase its heat absorption capacity. A covering bowl is used to collect the steam generated from evaporation and to condense it for further analysis. The solar evaporator device features a heat exchanger called a condenser, which facilitates the cooling and condensation of steam substances into liquid states. The receiver of the device gets an adequate amount of heat and with that, the liquor in the receiver bowl gets evaporated. As the waste liquor in the receiver heats up, it evaporates, and the condenser aids in the transformation of the evaporated substance into a pure liquid state. Stem produced due to evaporation was condensed in a condenser situated in the upper area of the receiver. The waste liquor gets evaporated and the solid chromium mainly Basic Chromium Sulphate remains as a residual.

Table 2. Characteristics of the receiver

Material	Aluminium
Diameter of the receiver (m)	0.18
Thickness (m)	0.005
Receiver area (m)	0.025

To reflect incoming sunlight and keep the condenser cool, an aluminium bowl wrapped in thin aluminium foil was utilized. This is because the aluminium foil is an excellent reflector of sunlight. Every hour checked the temperature and measured the evaporated water. Figure 2 illustrates the waste chrome liquor heated system, featuring a condenser and receiver.

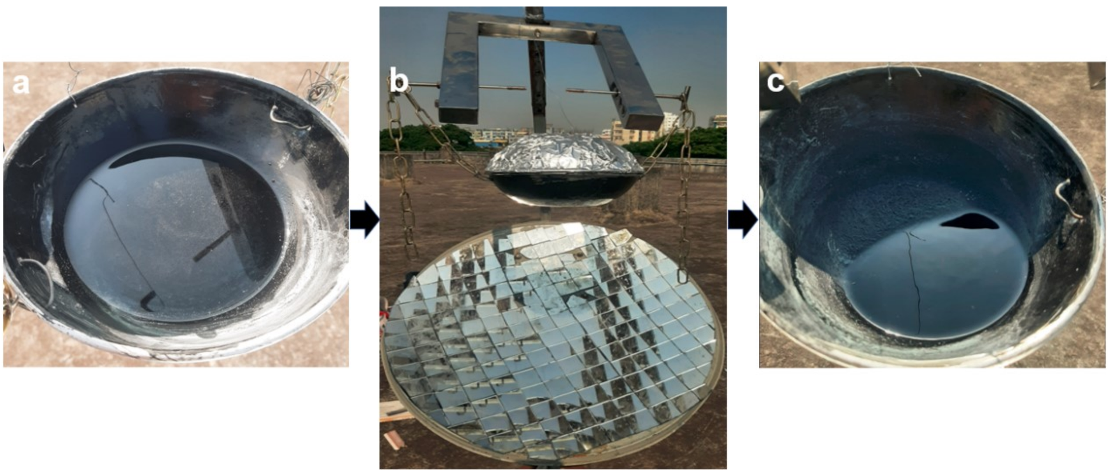

Figure 2. Flowchart of the waste chrome liquor heated system. (a) Waste chrome liquor, (b) Solar heated evaporator system, (c) Condensed chrome liquor.

The objective of this research is to determine the rate of solar evaporation. The device was installed in a convenient location where its performance could be observed. For this reason, tests were conducted at the Institute of Leather Engineering and Technology, University of Dhaka, Dhaka. The device was set up on the institute's rooftop and put into operation at the end of October. To determine the rate of evaporation, typical data were presented for one week and shown in Table 3, specifically from November 20th, 2022 to November 26th, 2022 from 9 am to 4 pm local time, on an average of seven hours of irradiation. The solar evaporator was employed for six months, during which it diligently accumulated the corresponding data. The study was originally designed to encompass a six-month evaporation investigation. However, due to adverse weather conditions, data collection was limited to approximately 2.5 months. Throughout this period, the evaporation rate exhibited a remarkable consistency across all batches, signifying a stable capacity. It is noteworthy that Table 3 represents an average of the results obtained during the 2.5-month data collection weekly.

Table 3. One week of temperature (°C) and evaporation data collection in the whole daytime

Time	9-10 am		11-12 am		12-1 pm		12-1 pm		1-2 pm		2-3 pm		3-4 pm		TE (mL)
Days	FT	CT	FT	CT	FT	CT	FT	CT	FT	CT	FT	CT	FT	CT	
20/11/22	78	40	87	46	90	51	96	54	80	43	79	41	68	39	232
21/11/22	80	50	87	52	92	55	105	60	95	56	78	48	70	40	249
22/11/22	86	52	91	55	98	58	108	62	98	58	83	50	78	45	296
23/11/22	91	50	87	50	97	55	99	57	92	56	81	49	76	40	285
24/11/22	88	47	92	50	101	56	97	53	92	53	71	50	62	43	279
25/11/22	86	51	90	52	97	54.5	102	58	91	54	78	48	60	43	280
26/11/22	81	50	86	52	90	54	98	60	96	56	80	52	61	42	275

*FT=Focal temperature, CT = Condenser temperature, TE= Total evaporation

The rate of evaporation of chrome liquor was calculated by the following formula:

$$\text{Rate of evaporation} = \frac{Condense\ liquor\ (ml)}{Time\ (h)} \quad (2)$$

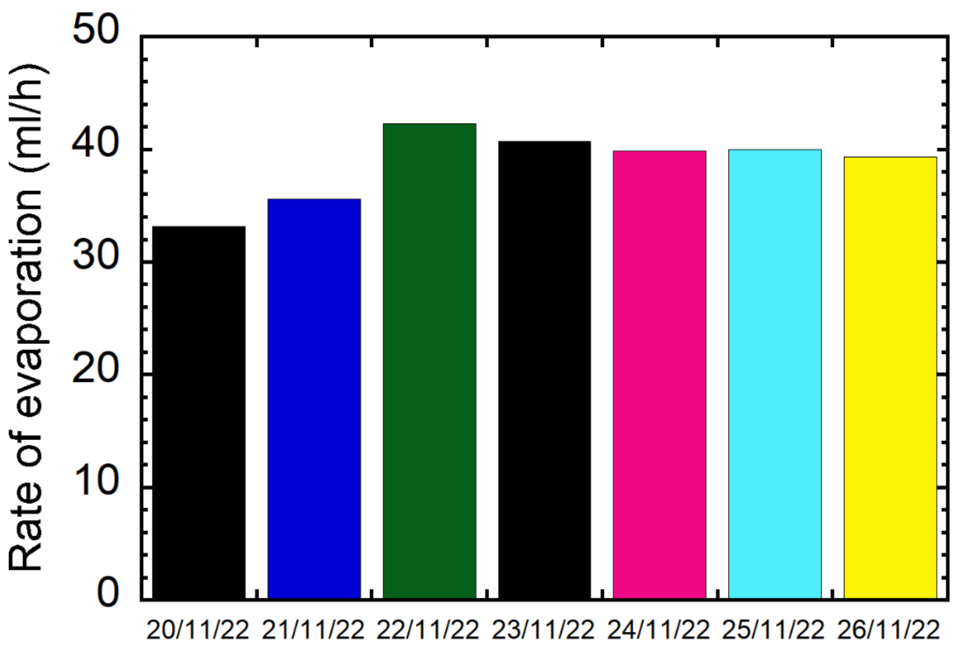

Figure 3. Graphical representation of evaporation rate for one week

The measured amount of chrome liquor was put in the evaporator and measured the time needed to evaporate the liquor and finally measured the amount of solid chromium. A different amount of chrome liquor (100, 200 mL) was given to the receiver to observe the variation in evaporation rate. The data was collected in different environmental conditions to observe the fluctuation of the rate of evaporation. The graphical rate of evaporation is shown in Figure 3 for one week. The average evaporation rate was found 38.69 ml/h for one week. Although this prototype device was constructed using readily available materials in the surrounding environment, it demonstrated an average evaporation rate when compared to the expensive and complex devices developed by other scholars for saltwater desalination [21-23].

The basicity of the recovered chrome was determined using the method described in the literature [24]. The Schorlemmer technique has been used to represent the basicity of the liquor. The basicity in this system is the percentage of total chromic oxide that is coupled with hydroxyl which is calculated below:

$$\text{Basicity (\%)} = \frac{A-B}{A}\ x100 \qquad (3)$$

where, A is the amount of thiosulphate, as ml of 0.1N $Na_2S_2O_3$ required to titrate 25 mL recovered chrome sample and B is the amount of NaOH, as mL of 0.1 N solution required to titrate 25 mL of the sample. The basicity of solid chromium was determined before treatment with sulphuric acid and after treatment with sulphuric acid.

The obtained solid chromium basicity is calculated by three times titration. The process of the average basicity calculation of solid chromium is given below.

Here: A = mL of 0.1 N $Na_2S_2O_3$ for Cr_2O_3 determination =12.17 mL

B = mL of 0.1N NaOH for acid titration =1.80 mL

$$\text{Basicity (\%)} = \frac{12.17-1.80}{12.17}\ x\ 100 = \frac{10.37}{12.17}\ x100 = 85.209$$

The obtained recovered chrome basicity has been found 85.209%. To introduce tanning it is needed to lower the basicity by 33%. The basicity was adjusted by treating the solid chrome with sulfuric acid. The amount of sulfuric acid that is needed to adjust the basicity of solid chrome to 33% is obtained from the following reaction.

$$Cr_2(OH)_6 + 3H_2SO_4 = Cr_2(SO_4)_3 + 6H_2O \quad (4)$$

With the help of this reaction, the amount of sulfuric acid that is needed to adjust the basicity of solid chrome to 33% is calculated and the procedure is given below:

104 parts of chromium or 152 parts Cr_2O_3 require 294 parts of H_2SO_4 to lower the basicity (100-0) = 100%.

For 104 g of Cr: 294 g of H_2SO_4 to lower basicity 100%

294/100 of H_2SO_4 to lower basicity 1%

(294×33)/100 of H_2SO_4 to lower basicity 33%

Therefore, for 1 g of Cr: (294 × 52)/ (100 × 152) = 1.006 g H_2SO_4 to lower basicity 33%.

It took 80 g of solid Cr for the experiment, therefore

(15,288 × 80)/ (100 × 152) = 80.46 g H_2SO_4 to lower basicity 33%.

It is known: 1.84 kg of H_2SO_4 with 95% concentration is equivalent to 1 Liter.

Therefore: 1.84 g of H_2SO_4 = 1 mL of H_2SO_4

80.46 g of H_2SO_4 = (80.46/1.84) mL of H_2SO_4 =43.72 mL of H_2SO_4.

According to the calculations, a total of 43.72 mL of H_2SO_4 is required to decrease the recovered chrome basicity from 85% to 33%. The basicity adjustment procedure is shown in Figure 4.

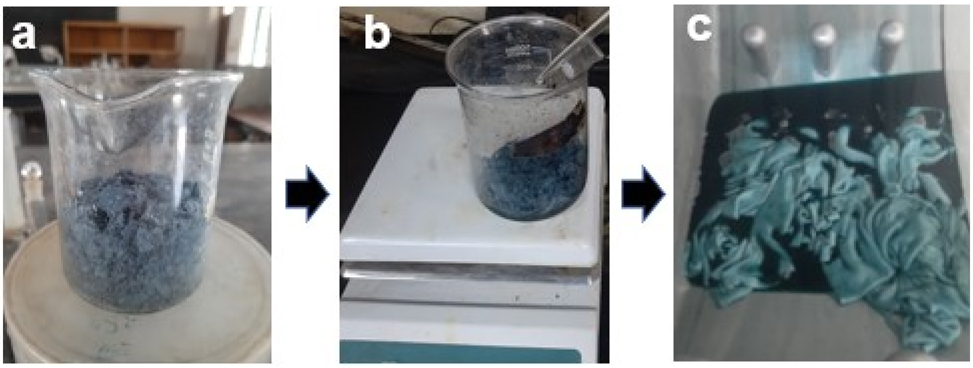

Figure. 4 Flowchart of basicity adjustment and application on the leather. (a) Recover chrome by solar evaporation, (b) Adjustment of recovering chrome basicity, (c) Recovered chrome application on leather tanning

The main concern of the research was to determine the evaporation rate, and a prototype device made from available local products was used, which showed an average evaporation rate compared to more expensive and complex devices built for saltwater desalination. The recovered chromium has been found 85% basicity, and H_2SO_4 was treated to decrease the 33% basicity because chromium reacts well at this basicity level with collagen fibre. After acid treatment, the basicity was found 37% in practice, which was close to the 33% basicity. The recovered chromium was then mixed with fresh conventional chrome in different ratios and used in the tanning process to manufacture leather. The physical and chemical properties of the resulting leather were comparable to those of leather made with fresh chrome.

The tanning viability was assessed through three different ratios during the manufacturing process. The ratios employed were as follows: number-A: fresh commercial chrome 8:0 (w/w; Fresh: Recovered), number-B: 5:3 (w/w; Fresh: Recovered), and number-C: recovered chrome 0:8 (w/w; Fresh: Recovered). The application of these mixtures in the chrome tanning process was carried out using various drums, leading to the creation of diverse leather samples following conventional leather manufacturing processes. It is crucial to emphasize that the manufacturing of leather encompasses three essential sub-processes: the preparation stage/beam house stage, tanning stage, and crusting stage, which were consistent throughout all experimental variations.

The shrinkage temperature was determined according to the IUP-16 method [25]. Each sample was divided into a size of 50 mm × 12 mm. The device was then bored with a 3 mm hole using the technique, and a sample was hooked to it. 350 cc of NaCl solution was used to submerge the sample. If the thermometer illustrates the temperature of the bath, then a stirrer disperses the heat evenly. The temperature was recorded when the sample shrank until the indicator moved away from the spot where the sample was fixed. The temperature of shrinkage and the boil test were examined on manufactured leathers and 3 samples met the standard requirements which are given in table 2.

Table 2. Summary of physical, chemical and mechanical properties of leather tanned by fresh chrome (Sample-A), recovered and fresh chrome (sample-B), Recovered chrome (sample-c) (mean±SD)

Test	Sample-A	Sample-B	Sample-C	Standard For Shoe upper Leather [27]
Shrinkage temperature (°C)	110±2.43	106±1.96	104±1.50	>100 °C
Tensile strength (N/mm^2) [26]	31.35±1.52	27.80+1.42	26.30+1.30	Minimum 24
Elongation at Break (%) [26]	26±1.35	25±1.12	22±1.12	Maximum 30
Stitch Tearing strength (Kg/cm)	100±1.21	83±2.31	100±1.15	Minimum 60
Instant lastoometer test (Kg/cm)	157±1.12	177±1.52	235±1.10	100-300
Flexing endurance test (30000 cycles)	Pipe labelling scale rating 3/4	Pipe labelling scale rating 3/4	Pipe labelling scale rating 3	Not below 3 (Max 50000 Cycles)

The shrinkage temperature of samples A (fresh chrome), B (recovered and fresh), and C (recovered chrome) tanned leather have been tested, and all the samples had a shrinkage temperature of more than 100 °C. Although the tensile strength and percentage of elongation meet the required standards, other tests, including stitch tearing strength, ball bursting strength, and flexing endurance, showed better results. The grain surface of the leather tanned using recovered chrome liquor had a smoother appearance than the leather tanned with new chrome liquor, and hair follicles were free of foreign materials in all samples.

Numerous techniques exist for the retrieval of chromium from tannery waste, although most are not practical for large-scale implementation. Nevertheless, the feasibility of utilizing solar energy for chromium recovery has been established, albeit with the need for enhancements to the solar device to augment the evaporation rate. In general, the utilization of solar energy as a means of chromium recovery holds significant promise owing to its widespread availability and abundance.

The waste chrome liquor treatment method using solar evaporation shows promise as an effective solution. The rate of evaporation can be enhanced by developing an efficient solar evaporator, and a well-designed effluent treatment plant can be constructed to improve overall results. However, it is essential to consider that this method relies solely on sunlight. Therefore, during periods of monsoon and snowfall when there is little to no sunlight, the solar evaporator may not be operational, and alternative waste treatment measures should be in place.

CONCLUSION

The hazardous nature of chromium extends its threat to both living organisms and the environment. Despite the extensive research on chromium recovery, most existing methods are neither feasible nor environmentally friendly. In this study, a solar evaporator was constructed to effectively retrieve chromium from tannery waste chrome liquor. The temperatures within the receiver were closely monitored, with the maximum recorded focal and water temperatures reaching 108 °C and 62 °C, respectively. On average, the solar evaporation rate was measured at 40 mL/hr.

Leather samples were tanned using varying ratios of fresh and recovered chrome, and their physical and chemical properties were rigorously examined, including boil test, shrinkage temperature, tensile strength, and stitch tear strength. Remarkably, the leather produced using recovered chrome exhibited superior properties compared to conventionally tanned leather, implying that the recovered chrome can be seamlessly incorporated into subsequent tanning processes without compromising the economic value of the resulting leather. It is strongly encouraged that tanneries in Bangladesh invest in the establishment of solar evaporator plants, enabling the cost-effective and sustainable recovery of chrome from spent chrome liquor. Such initiatives hold great potential for reducing environmental impact and promoting responsible practices in the tanning industry.

Author Contributions (Level III Heading)

Conceptualization – Abdulla-Al-Mamun M and Marma M; methodology – Abdulla-Al-Mamun M and Marma M; formal analysis – Abdulla-Al-Mamun M and Marma M; investigation Marma M; resources – Abdulla-Al-Mamun M, Marma M; writing-original draft preparation – Abdulla-Al-Mamun M, Marma M; writing-review and editing – Abdulla-Al-Mamun M; visualization – Marma M, Farhad Ali M, and Abdul Mottalib M; supervision – Abdulla-Al-Mamun M. All authors have read and agreed to the published of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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