Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2021.26910

217 (2021) 243–252
March

Attaining water efficiency and reduction in chromium release through wastewater reuse in basic chromium sulfate production industry

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Received 10 August 2020; Accepted 4 December 2020

ABSTRACT

Current study was aimed to investigate potential water savings and pollution reduction through effluent reuse at basic chromium sulfate (BCS) plant located in Lahore, Pakistan. Specific water consumption and effluent discharge were calculated as m^3/ton product based on an average production in 2016 and 2017. Based on assessment criteria, the environmental performance was evaluated through best available options including improving washings, controlling leakages, enhancing efficiency of SO_2 scrubber. Further, reuse applications of chromium rich effluent were tested in process using standard methods at pilot and production scale without advance treatment techniques. As a result, process/non-process water consumption was reduced by 2.96–1.5 m^3/ton product reflecting overall 49.6% as well as effluent discharge by 1.63–0.73 m^3/ton product reflecting overall 55%, which resulted in 2,142.6 m^3/y of water savings led to achieving zero liquid discharge (ZLD). Economic benefits were gained as the production of sodium sulfite after scrubbing which gives $6,000 annually as well as the recovery of chromium resulted in excessive BCS yield which gives an annual saving of $8,420 to the industry. This study proved a sustainable model of increasing water use efficiency and achieved ZLD economically to the industry with reduced environmental impacts through chromium release.

Keywords: Tanning agent; Water-saving; Effluent management; Pollution reduction; Sustainable solutions

1. Introduction

The chemical industry is a growing sector in Pakistan which has an impact on the environment in terms of water usage and hazardous effluent discharge [1]. Due to extensive usage of chemicals in industrial processes of this technology era, water is incorporating in almost every process leading toward its scarcity and purity [2,3]. Globally, green technologies are introduced which highlighted the need for the protection of the environment from the peril of chemical pollution. [4]. Eversince, the water shortage crisis erupted in the world, reuse, and recycling of wastewater in industries have become a preferred approach to reduce freshwater intake as well as treatment costs [5].

In leather chemical industry, basic chromium sulfate (BCS) is one of the major tanning chemicals which is used in leather industries worldwide. In leather industries about 90% tanning process is carried out by using chromium (Cr) in powdered form [6]. In BCS processing, water is extensively used as process and non-process thus a large quantity of it is drained out in the form of wastewater in freshwater streams. This drainage of untreated wastewater in a high concentration of Cr ranging from 2,656 to 5,420 mg/L not only poses the risk of pollution and environmental hazards as well as leads toward the depletion of some resources which can be reused if recovered [7].

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As Cr is highly toxic, several sustainable methods are suggested to eliminate it from effluent to provide a cleaner and sustainable production environment in the industry [8]. Therefore, chromium manufacturing industries have been advised to modify their processing in order to reduce water consumption and to adopt new wastewater treatment technologies [9,8]. Recovery methods of chromium have been suggested in a literature [10], using absorption and thermal treatment respectively which are reused in the leather tanning industry. Leathers made from this tanning agent had properties comparable to conventionally processed chrome-tanned leathers. This utilization of dangerous liquid wastes has a positive impact on the environment and an approach toward sustainable development.

It was revealed in many studies that sustainable methodology for reduction in water consumption and effluent can be attained in different industrial units. A study examined process alterations and management practices to enhance water and chemical use efficiency in order to increase environmental and economic benefits of a metal processing plant. Overall effluent production was reduced by 3,255 m^3/y which was 50.9% of the total main drain discharge [5]. Another literature claimed that the total reduction in water consumption using best available techniques (BATs) of the woven fabric mills was 40.2% whereas wastewater production was minimized by 43.4% [11]. In a recent study, about 46% reduction in water consumption has been obtained at polyethylene terephthalate production by espousing smart management practices and process alterations that has resulted in both increased environmental performance and profitability [12]. Various other application of reduction in water usage can be achieved by applying good management practices and process modifications as well as technology changes that results in environmental performance and profitability. Therefore, sustainable production in the chemical industry has a significant evidence of the relationship between environmental expenditures and financial performance [13]. Ozturk and Cinperi [14] suggested nine minimization techniques in the textile industry in order to reduce water consumption by 41%–69% as well as effluent by 48%–75%. These results were achieved after detail on-site investigations in non-process and process waters.

Recently, due to the enforcement of much strict environmental regulations on various industrial effluents have preferred conservatory approaches to water usage [15]. However, industries in Pakistan do not pay much attention for adopting these water saving approaches as well as facing problems in effluent management [16]. Moreover, BCS manufacturing units are lacking in appropriate water saving and effluent management hence wasting a high amount of useful chemicals in their effluent which posing a serious threat to environment and public health [17]. In view of abovementioned problems, the current study was designed to (i) to reduce the water consumption in overall BCS plant using best available strategies and (ii) applying reuse techniques of effluent in order to reduce the release of chromium ultimately leading to achieve zero liquid discharge (ZLD). Further, this study also estimated the economic benefits achieved as a result of Cr recovery from wastewater reuse.

2. Materials and Methods

2.1. General information and process description

The current study was carried out at a BCS manufacturing plant located in Lahore, Pakistan. BCS is a greenish color powder mainly a mixture of BCS [$Cr(OH)SO_4$], sodium sulfate [$Na_2(SO_4)$], and an anti-oxidant product (Ecotan Crom VI) used for chrome tanning in the leather industries [18]. This plant is producing 120–150 tons/month BCS to meet the demands of leather tanning locally and internationally. Fig. 1 shows a process flow sheet of the conventional production of BCS (data shared by the company). Sulfur and sodium dichromate (SDC) are used as raw materials. Sulfur is burnt in a furnace at 300°C–350°C to produce sulfur dioxide (SO_2) gas. SDC containing chromium(VI) is reduced to chromium(III) by SO_2 in a packed tower and circulation tank. During the reaction, recirculating tanks and an absorption tower circulate the mixture in order to achieve the complete reduction of Cr(VI) to Cr(III). Then the mixture is transferred to aging tanks where SO_2 saturated environment and slow stirrer movements keep the mixture homogenize to facilitate the conversion of remaining traces of Cr(VI) into Cr(III). Further, an antioxidant product (Ecotan Crom VI) is also added as an additive in aging tanks to prevent the oxidation of Cr(III) back into Cr(VI). After aging, the mixture is sent with pressure toward the spray dryer. A big conical shaped, pre-heated spray drier is used to dry the mixture and turns it into a solid powdery greenish product that is finally packed through a conical-shaped

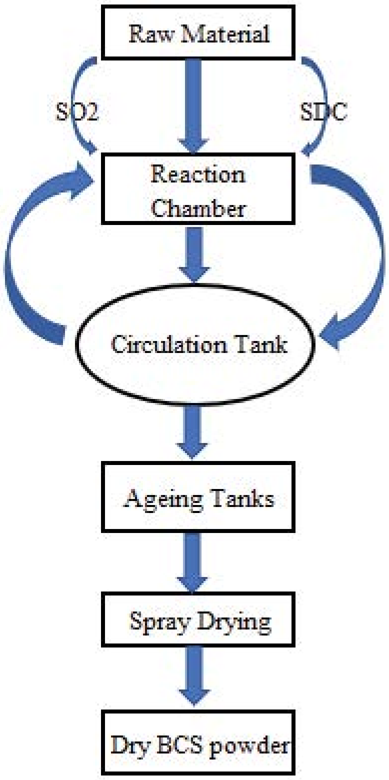


Fig. 1. Flowchart illustrating various processes of BCS manufacturing.

packing machine into sacks after sifting. It is sold in the market for further industrial use. The process reaction is given below:

$$Na_2Cr_2O_7 + 3SO_2 + H_2O \rightarrow 2\left[Cr(OH)SO_4\right] + Na_2SO_4 \quad (1)$$

2.2. Development of water consumption baseline data

To analyze the current scenario, a systematic baseline study of the plant was necessary to be carried out, as suggested by Ozturk and Cinperi [14], because the study organization did not have the sufficient data of total water consumption for process and non-process (washings and others). For this purpose, a total of nine water flow meters were installed on various locations to identify process/non-process water consumption values and subsequently to calculate the complete data of water consumption. It was also necessary to calculate non-process waters including floor washings, vessel washings, spray dryer washings, leached residues, and other leakages because these activities are also contributing a major chunk in total water consumption [19]. The company does not have any treatment facility to meet the legislative requirements but pays huge amount per meter cube of wastewater to outsource company for effluent treatment. Measuring and monitoring were performed in the year 2016 with respect to the average monthly production of 122 tons and compared with the year 2017 after introduction of additional technology. Specific water consumption and wastewater generation data was calculated as m^3/ton product.

2.3. Sampling method for the wastewater

It was observed during detailed surveys that there were two drains [(i) Cr containing effluent (BCS effluent) and (ii) SO_2 scrubber water] coming out from the plant and disposed of in the main drain. One day composite samples (a total of 24 composite samples) were collected in amber bottles of 500 mL from both drains. These samples were analyzed at the source quality assurance (QA) laboratory of the company and an environmental protection agency (EPA) certified laboratory.

2.4. Parametric analysis of effluent

The pH and electrical conductivity of wastewater were determined by using a multi-meter (model = HANNA HI 9811-5). Chemical oxygen demand (COD) was determined using the standard American Public Health Association (APHA) method [20]. The color/appearance of wastewater was visually tested. The temperature was measured by the digital thermometer model TP 3001. Total solids were quantified in wastewater and in product samples by gravimetric analysis following oven drying. Total chromium, chromium hexavalent, and sulfate was tested using standards, APHA-3500-Cr B [21], HACH-8027, and HACH-8051, respectively (HACH, Chemical Company, Loveland, CO).

2.5. Implementation of BATs

In order to reduce water consumption and wastewater generation, non-process water usage practices, and processes that are needed to improve were determined. A total of nine available options were selected in order to meet all objectives for improving environmental performance and production costs associated with determined practices/processes (Table 1). The assessment was carried out using environmental criteria including environmental benefits, technical applicability, economic viability, easiness of implementation, long-term sustainability, operational, and maintenance requirements for sustainable production options [12,22,23].

2.6. Reuse potential of effluent and chromium recovery

A pilot-scale BCS plant was designed to perform experiments to check the quality of the BCS finished product after reuse of effluent. After success in these experiments, a collection pit of 10 m^3 volume was constructed at a production scale to collect BCS effluent with a level maintained at 7 m^3

Table 1
Objectives of application and respective options to achieve

Objectives	Selected available options
Measure a total water consumption in the plant for process and non-process waters	1. Install water flow meters
	2. Develop a water flow balance in order to measure total water consumption
Reduction in water consumption of non-process activities	3. Minimize or completely eliminate floor washing by bucket mop/dry air blower.
	4. Improve vessel/container washing by efficient pressure nozzles
	5. Detect and control leaching and leakages from all locations
	6. Improve housekeeping and control other washing water in the plant area
Reduction in water consumption of process activities and effluent generation	7. Collection of effluent in a constructed effluent collection pit/sump
	8. Reuse of raw effluent into the process as feedwater after preliminary treatment in the collection pit
	9. Increase the number of cycles of SO_2 scrubber water to reduce feed water and eliminate liquid discharge by producing an intermediate product Na_2SO_3

and reuse it in the circulation tank of the process. A pump was installed to recycle it into the wet-process. A finished BCS product quality test with freshwater and a combination of freshwater and reuse of effluent was applied using standard testing methods, as given in Table 2.

All the above-mentioned action plans were implemented step-wise starting from December 2016 to March 2017. Monitoring was performed throughout the year 2017 until final results were successfully obtained. The above assessment was aimed to achieve the future vision and objectives of the water efficiency at BCS plant [14].

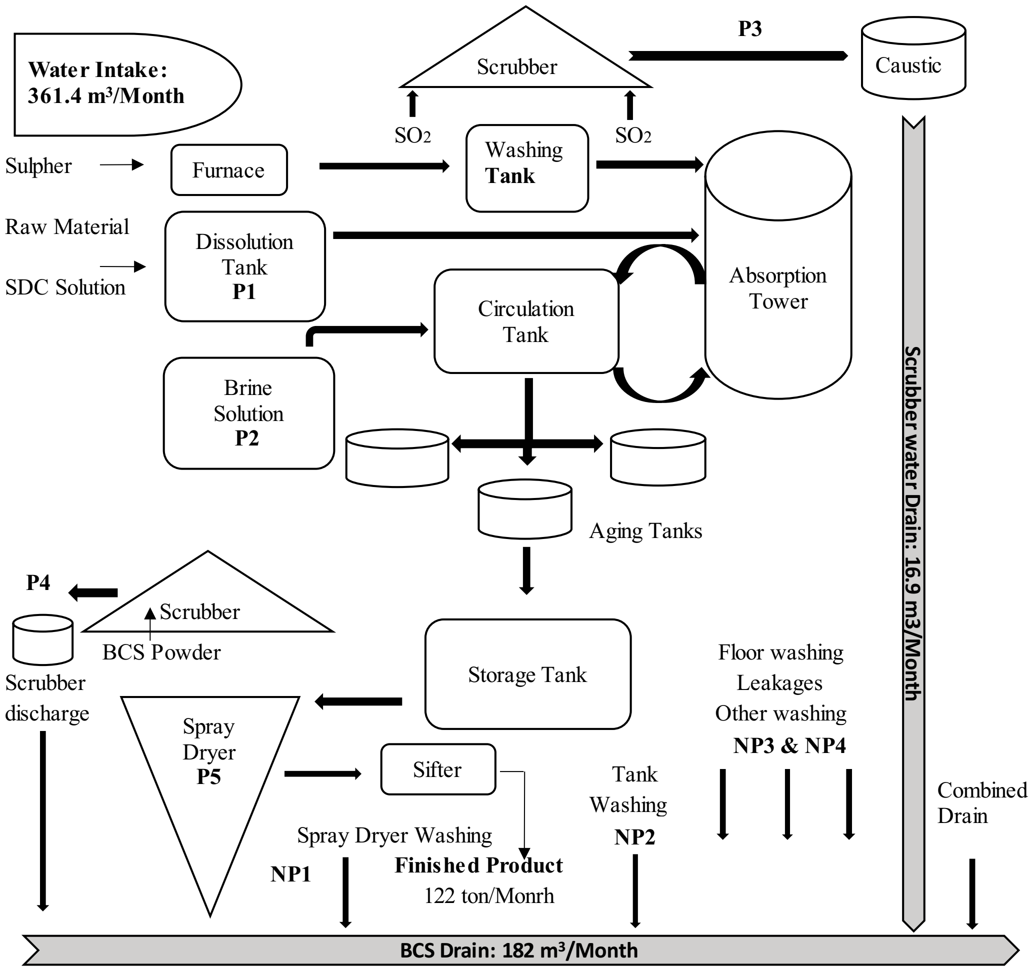
3. Results and discussion

3.1. Water consumption and liquid discharge evaluation of BCS plant

The process water consumption was measured by about 5 m³ for one batch/d of 4,500–5,000 kg BCS production in dry form. Initial measurements were performed for the last 4 months of 2016, afterward, calculations were made for the whole year for production during each month. It was observed that the total water consumption was 361.4 m³/month with an average production of 122 tons per month (Fig. 2). Groundwater was used for all processes and non-process activities in the BCS plant. According to Salma et al. [30], 2.5 m³ water is used for the production of 1 ton of BCS manufacturing whereas the total water

Table 2
Finished BCS product quality test methods

Test applied	Standard testing code
% Basicity	SLC 136 [24]
% Chrome contents	IUC 8-1 [25]
% age residue	ASTM D3042-17 [26]
Moisture	ASTM D2832-92 [27]
Presence of chromium(VI)	ASTM D5257-17 [28]
pH 10% solution	ASTM E70-07 [29]



P1: SDC, P2: Brine preparation, P3: SO2 Scrubber charging, P4: Spray dryer scrubber charging,
P5: Makeup water for spray drying, NP1: Spray dryer washing, NP2: Storage tank washing, NP3:
Floor washing, NP4: Leakages and others

Fig. 2. Baseline evaluation of water consumption and effluent discharge of BCS plant.

consumption was measured as 2.97 m^3 for 1 ton BCS production (Table 3) in the current study. The ratio between processes and non-process water usage was about 60% and 40%, respectively. Process activities include P1 (SDC solution preparation), P2 (brine preparation), P3 (SO_2 scrubber charging), P4 (spray dryer scrubber charging), and P5 (makeup water for spray drying), whereas non-process activities include; NP1 (spray dryer washing), NP2 (storage tank washing), NP3 (floor washing), and NP4 (leached residues, leakages, and others). Wastewater was generated majorly by all washing activities (NP1, 2, 3, and 4), whereas another contribution was from P3 and P4 process activities (Fig. 2).

Table 3 presents the mass flow balance of all consumptions and liquid generation baseline scenario of the industry. The consumption data was collected as per month activity for processes/non-processes based on an average of 122 ton/month of the finished product for the base year 2016. Major contribution in process water consumption was measured in SDC (P1) where 130 m^3/month water was used which is equal to the specific water consumption of 1.07 m^3/ton product. P3 and P4 contributed to water consumption at 16.9 and 32.5 m^3/month, respectively, which are the only sources of effluent discharge through process activities. However, in non-process activities, tank washing (NP2) presented the major input in non-process water consumption and discharge which contribute to 0.43 m^3/ton product. NP3 and NP4 contributed to 0.32 and 0.11 m^3/ton product, respectively, showing the excessive water consumption and liquid discharge in this activity. The evaporation rate was also calculated after spray drying and the end of the pipe of both effluent streams. The total effluent discharge was measured as 178.1 m^3/month (equal to 1.63 m^3/ton product) excluding the evaporation loss of 13 m^3/month from spray drying and 7.8 m^3/month naturally. The liquid discharge was drained out from the company without any treatment.

3.2. Analysis of raw effluent

Wastewater analyses were performed to evaluate the quality of effluent of both drains (BCS drain and SO_2 scrubber water drain) on the basis of selected parameters, as shown in Table 4. Results revealed that effluent of both

Table 3
Water consumption and liquid discharge flow in different process and non-process activities at BCS plant

Water utilization areas	Consumption m^3/month	Specific water consumption m^3/ton product	Discharges m^3/month	Specific liquid discharge m^3/ton product
		Processes		
P1	130	1.07	–	–
P2	19.5	0.16	–	–
P3	16.9	0.13	16.9	0.13
P4	32.5	0.27	32.5	0.27
P5	13	0.11	13[a]	0.11[a]
		Non-processes		
NP1	45.5	0.37	45.5	0.37
NP2	52	0.43	52	0.43
NP3	39	0.32	39	0.32
NP4	13	0.11	13	0.11
Others			7.8[a]	0.06[a]
Total	361.4	2.97	178.1	1.63

[a]Evaporation loss (measured after spray drying and output stream directly).

Table 4
Values of effluent parameters of both drains of BCS plant

	Test applied	Unit	BCS drain	SO_2 scrubber water drain	NEQS
1	pH		3–4	5.11	6–9
2	COD	mg/L	1,811	2,399	150
3	TDS	ppm	10,000	4,460	3,600
4	Conductivity	(µS/cm)	10.3	78	–
5	Temperature	°C	20	20	–
6	% Solids	–	2.5	1.8	–
7	Chromium	mg/L	2,830	–	1
8	Chromium hexavalent	mg/L	0.019	–	–
9	Sulfate	mg/L	4,900	89	600

drains was highly polluted comparing with national environmental quality standards (NEQS), as pH value measured 3–4 and 5.11, total dissolved solids (TDS) values were 10,000 and 4,460 mg/L, COD value was between 1,811 and 2,399 mg/L, conductivity was measured as 10.3 and 78.0 µS/cm, respectively. As shown in Table 4, BCS drain contained Cr content and sulfate of 2,830 and 4,900 mg/L, respectively, in dissolved condition as compared with NEQS value of 1 mg/L, showing the greater potential of chromium recovery [31]. The effluent containing Cr of this concentration can cause various environmental and health problems if directly discharged into the drains [7]. On the other hand, SO_2 scrubber drain contained 1.8% Na_2SO_3 salt, and this salt solution which has also the recovery potential, which was drained after every batch of BCS production as discussed in section 3.1 (Water consumption and liquid discharge evaluation of BCS plant).

3.3. Reusing of raw chromium rich effluent

3.3.1. Pilot scale test

Regardless of COD and TDS, the presence of total Cr and sulfate with suitable pH, the current study clearly indicates that BCS effluent has 100% potential of reuse/recycling comparing with Panda et al. [8], where the suggested method of BCS production releases effluent with 80% reuse/recycling potential. For this purpose, a trial was conducted for pilot scale BCS production by replacing 50% of freshwater with BCS effluent. Final quality parameters proved that there was no significant change found in basicity and chrome content of the final spray-dried BCS product as compared to the standard testing codes (Table 5). Many literatures also proved that the BCS final product with 33% basicity, 25.5% chrome content, and 2.2%–2.4% residues are suitable for use in the leather tanning industry [6,8,10].

3.3.2. Production scale

After the success of pilot production, arrangements were made for the collection of Cr contained effluent at the main source production of BCS as discussed in section 2.6 (Reuse potential of effluent and chromium recovery). In a standard batch of BCS powder (4,700 kg), 5,000 L water was used. As a result, 50% of the process water was replaced with BCS effluent (2.5 m^3). After the implementation of this exercise in the production scale, the resulting product was as per standard.

3.4. By-product recovery of SO_2 scrubber effluent

Another evaluated option was applied to increase the number of cycles of SO_2 scrubber water. Previously, 500 L wastewater from scrubber were drained out after each batch containing Na_2SO_3 with a total solid value of 1.8% (Table 4). According to Zhou et al. [32], the solubility factor of caustic soda (NaOH) and SO_2 to convert into sodium sulfite can be increased up to 25%. This application was achieved after 10–11 batches of wet BCS production. The resultant solution can be concentrated by evaporation to make is an intermediate powder or can be sold in liquid form to pulp and paper industry directly [33]. Another approach of sodium sulfite reutilization is the leather tanning process after mixing with sodium dichromate [10]. After achieving solubility of sodium sulfite and its utilization for other industrial purposes is also another attempt toward effective sustainable strategies to reduce water consumption as well as to eliminate wastewater discharge.

3.5. Evaluation of total water consumption before and after application

Fig. 3 illustrates 2 y (2016 and 2017) comparison of total water consumption with production. Implementation of all evaluated options was started in the month of December 2016 and became fully operational in March 2017 including reusing of Cr containing effluent. It is clearly indicated that overall water consumption declined in the year 2017, though the production was increased to the average from 122 ton/month (year 2016) to 144.6 ton/month (year 2017).

Specific reduction in consumption and effluent generation with all process/non-process activities using all nine evaluated options was calculated based on average production in 2016 (Table 6). Maximum reduction in consumption and liquid generation was achieved in NP3 as 92%, which corresponds to water-saving and effluent discharge of 429 m^3/y. While in SO_2 scrubber water charging (P3) 85% reduction in water consumption and 100% in a liquid generation were achieved corresponding to 171.6 m^3/y water-saving and no end of pipe effluent treatment was required. Other process and non-process activities like P1, P5, NP2,

Table 5
Quality tests of BCS finished product – a comparison of real sample and the sample with 50% effluent reuse

Test applied	Reference value		Real sample value	Sample value after reuse of BCS effluent
	Minimum	Maximum		
% Basicity	33.0	37.0	33.9	33.8
% Chrome contents	25.50	26.50	25.5%	25.5%
% Residue	2.0	4.00	2.2%	2.4%
Moisture	7.50	9.50	9.4%	9.4%
Presence of chromium VI	–	–	ND[a]	ND
pH 10% solution	2.50	3.50	3.4%	3.4%

[a] Not detected.

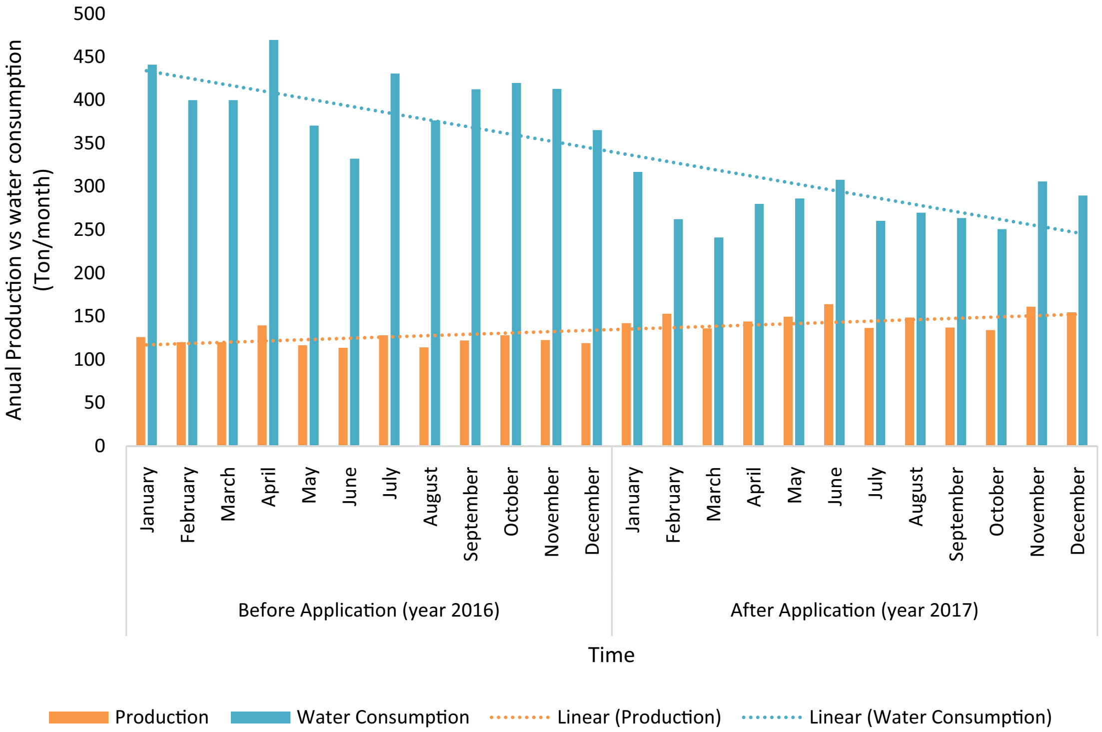


Fig. 3. Comparison of total water consumption and total production data at BCS plant during 2016 and 2017.

Table 6
Reduction of overall water consumption and the effluent generation before and after application

Water utilization areas	Specific water consumption m³/ton product			Specific liquid discharge m³/ton product		
	Before application	After application	Change %	Before application	After application	Change %
P1	1.07	0.53	–50	–	–	–
P2	0.16	0.16	0	–	–	–
P3	0.14	0.02	–85	0.14	0	–100
P4	0.27	0.27	0	0.27	0.27	0
P5	0.11	0.05	–50	–	–	–
NP1	0.37	0.17	–54	0.37	0.17	–54
NP2	0.43	0.21	–50	0.43	0.21	–50
NP3	0.32	0.03	–92	0.32	0.03	–92
NP4	0.11	0.05	–50	0.11	0.05	–50
Total	2.96	1.49	–49.6	1.63	0.73	–55

and NP4 had a 50% reduction in water consumption after all applications which corresponds to the annual saving of 1,248 m^3 for BCS plant, while NP1 showed a reduction in water consumption as 54%. However, NP1, NP2, and NP4 had the same reduction in effluent generation. Replacing freshwater consumption with reuse of almost half of raw BCS effluent in the process was the major cause of the reduction in P1. Some processes (P2 and P4) showed no change due to process requirements.

After all applications, the results proved that overall water consumption and liquid discharge in the BCS plant were reduced by 49.6% and 55%, respectively.

Fig. 4 shows the improved scenario of the BCS plant after the implementation of selected evaluated options. The monthly reduction in water required by the plant was reduced to 182.85 m^3/month with constant production of 122 ton/month. Reuse/recycling of BCS effluent in the circulation tank to fulfill the water needs of the process as well as a by-product of Na_2SO_3 resulted finally as ZLD from the industry [34].

3.6. Environmental and economical approach of the study

Apart from the sustainable management of water consumption in order to reduce resource depletion, almost

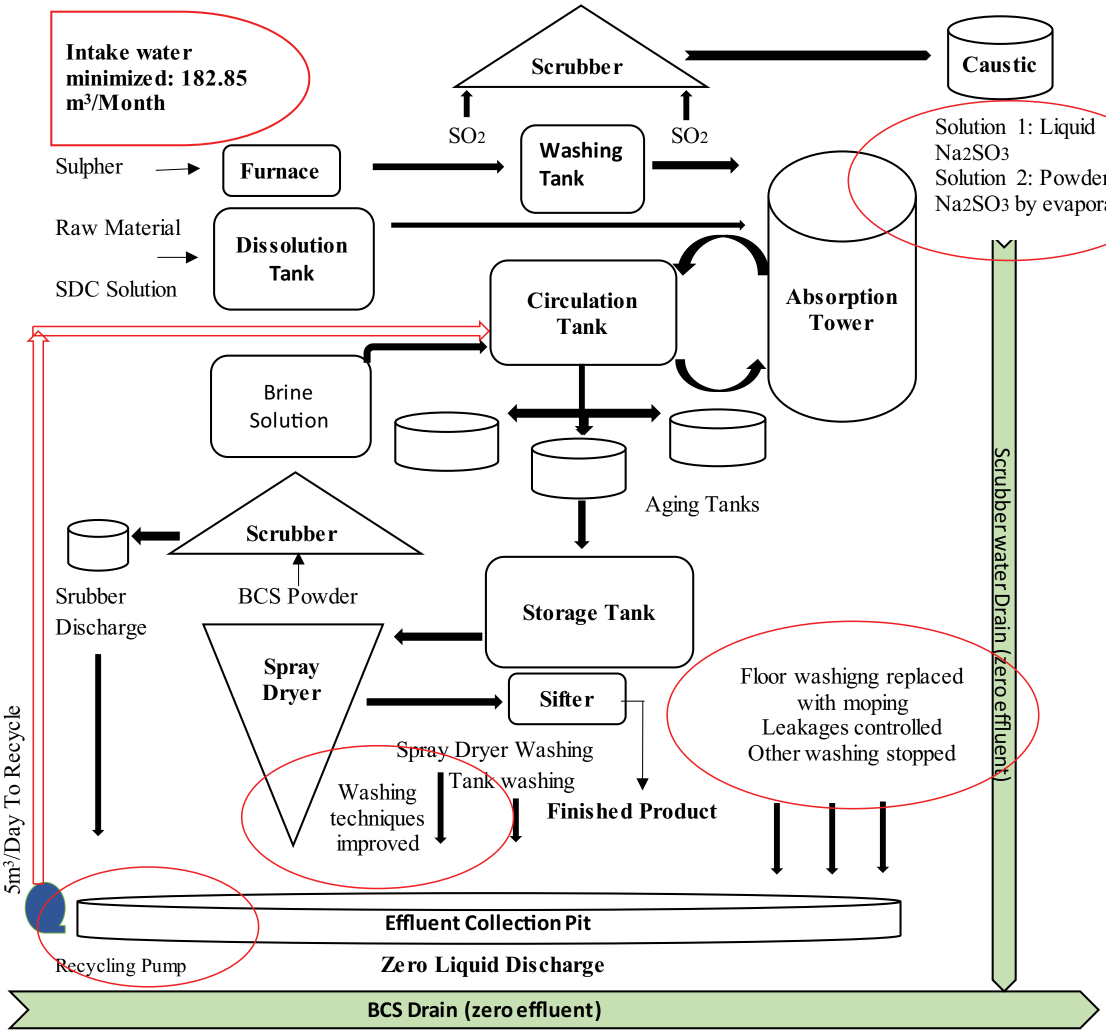


Fig. 4. Overall water efficiency and zero liquid discharge approach after application in BCS plant.

half of the SDC water (2.5 m³) per batch was replaced by Cr containing effluent which contains 2,830 mg/L which was the whole reduced effluent generated after plant operation (Table 4). Before application, this high concentration of chromium (a notorious environmental pollutant) was released into the environment thorough inappropriate effluent discharge management. Various studies have reported the high toxic effects of chromium release into the environment through aforementioned route. This release may cause severe threat to aquatic life, soil, plant, animals, and humans in varying concentrations [35]. Reutilization of the reduced effluent into the production of BCS has a remarkable influence on the environment in addition to economic benefits to the industry [31].

According to Eq. (1), the relation of BCS production with chromium quantity is given in Table 7.

Table 7 presents the total 472 kg BCS product is produced by 262 kg SDC so that the ratio between SDC and BCS becomes 1:1.8. Therefore, 2.5 m³ effluent contains 7 kg of Cr which is equal to 17.64 kg SDC leading to additional production of 31.75 kg of BCS per batch.

Table 7
Relation of BCS production with chromium quantity

	2 Cr	SDC ($Na_2Cr_2O_7$)	BCS [$2\ Cr(OH)SO_4 + Na_2SO_4$]
	kg	kg	kg
Equation quantities	104	262	472
Effluent (2.5 m³)	7[a]	17.64	31.75

[a]Cr concentration of 2,830 mg/L in effluent.

The monthly recovered value of BCS obtained 701.67 $ which corresponds to the annual saving of 8,420 $ to the industry (Table 8).

After the implementation of 9th selected options, the study provides a valuable gain economically as the production of secondary product, two tons concentrated solution (25% ± 1% solids) of sodium sulfite obtained in a month which is used as further industrial activities. Dry powder of 1 kg sodium sulfite costs 1 $ in Pakistan.

Table 8
Economical benefits of BCS effluent reutilization

	BCS recovered (kg)	Price ($) per kg	Total value ($)
Recovery per batch	31.75	0.85	26.97
Recovery per month	825.5	0.85	701.67
Recovery per annum	9,906	0.85	8,420

The economic benefit of 25% sodium sulfite solution costs 500 $/month was calculated for this study which corresponds to the annual saving of 6,000 $. It is revealed in literature that the capital and operation cost of 50–100 thousand dollars is normally required for the effluent treatment (100–200 m^3 flow) as well as the application of reuse and recovery [36,37].

4. Conclusions

BCS is the most versatile and common tanning agent in the leather industry causing chromium release as effluent discharge in the environment during its manufacturing. The reduction in water consumption at the BCS production unit in the selected industry makes an effective and sustainable approach to freshwater saving. The study focuses on the built-in process greener practices that along with saving of water (100% reuse) implements the management strategies without the requirement of any advance treatment in the industry. As a result of the applications, overall water consumption was reduced to 49.6%, which corresponded to a water-saving of 2,142.6 m^3/y if the plant works with the production of 122 tons/month. While effluent was reduced by 55% corresponding to makeup as chrome rich feedwater reutilization which resulted in elimination in the release of chromium as effluent discharge in the environment. The study provides the economical approach to the industry regarding recovery of chrome effluent with enhanced the total yield saving annual cost of 8,420 $, the lesser input of new water in the process, by-product of sodium sulfite solution (Na_2SO_3) saving of 6,000 $/y. This could be a better future outcome by adopting these strategies and thus can lead to the ZLD approach for many manufacturing industries in the near future.

Acknowledgments

The authors would like to thank the chemical industry to provide their facilities, data, and finances for the project. Special thanks to Mr. Muhammad Naveed Ashraf and Ms. Nabeela (industrial officials) for their valuable comments and co-operation.

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