



## Note from the field

## Oxidation-coagulation-filtration processes for the reduction of sulfide from the hair burning liming wastewater in tannery

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## ABSTRACT

The liming wastewater in tanneries contain huge amounts of soluble sulfide; upon acidification, this produces toxic hydrogen sulfide, which is a grave environmental concern. In the present study, an approach was made to treat liming wastewater with oxidation-coagulation-filtration techniques; wastewater was treated with hydrogen peroxide to eliminate the emission of hydrogen sulfide and aluminum sulfate was used as coagulant. The physicochemical parameters of untreated liming wastewater were: sulfide  $7285.2 \pm 11$  mg/L; pH  $12.5 \pm 0.2$ ; total dissolved solids (TDS)  $23.8 \pm 0.3$  g/L; total suspended solids (TSS)  $17.8 \pm 0.2$  g/L; electric conductivity (EC)  $40.5 \pm 0.2$  mS; salinity  $23.4 \pm 0.1$  ppt; and dissolved oxygen (DO)  $8.3 \pm 0.1$  mg/L. The physicochemical parameters of the treated liming wastewater at optimum conditions were: sulfide  $81.9 \pm 0.5$  mg/L; pH  $6.9 \pm 0.2$ ; TDS  $4.5 \pm 0.1$  g/L; TSS  $0.8 \pm 0.02$  g/L; EC  $5.8 \pm 0.1$  mS; salinity  $3.1 \pm 0.04$  ppt; and DO  $7.3 \pm 0.03$  mg/L. The removal efficiency of sulfide, TDS, TSS, EC and salinity were 99%, 81%, 96%, 86% and 87%, respectively. The most promising outcome of this technique was the efficiency in the removal of sulfide. This simple technique for treating liming wastewater would be very effective in reducing environmental pollutants being emitted by the tanneries.

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## 1. Introduction

Throughout the world, tanneries are recognized as high-level waste-generating industries. The leather processing that takes place in the tanneries involves a series of chemical treatments and mechanical operations for attaining the prescribed characteristics. During leather processing, at least 300 kg of chemicals are added to a ton of hides (Verheijen et al., 1996), whereby only 15% of the chemicals are retained by the finished leather, while the remaining 85% enter into waste streams (Buljan et al., 1997). Suthanthararajan et al. (2004) reported that about 30–40 m<sup>3</sup> wastewater is generated for every ton of hide/skin processed. Wastewater production depends on many factors and as such the amount generated fluctuates between 10–100 m<sup>3</sup> for each ton of hide/skin processed (Tunay et al., 1995). This wastewater is highly polluted and is responsible for upsetting the delicate environmental balance (Rouf et al., 2013).

For instance, in beamhouse, unhairing and liming (commonly liming) is carried, which makes up 60–70% of the total pollution load of the leather manufacturing industries (Xu et al., 2009),

making it the stage of leather processing with the highest yield of pollution. Liming is carried out to dissolve keratins (epidermis, hair, wool), non-structural proteins (albumins, globulins), and subcutaneous adipose layer (Lofrano et al., 2013); to swell up the hide/skin; to separate the collagen fiber; to partially saponify natural fat/grease (Gutterres et al., 2011). The conventional liming process involves treatment of hide/skin with sodium sulfide (Na<sub>2</sub>S), and lime (CaO). Sulfide dissolves the keratin; hydrated lime makes the aqueous buffer alkaline pH (12.8–13.0) (UNEP/IE, 1996).

Wastewater expelled by the liming process contains high sulfide, chemical oxygen demand, biological oxygen demand, total suspended solids and total dissolved solids loads in the effluent (Jian et al., 2011). Moreover, it generates huge solid waste containing lime sludge, fleshings and hair (Saravanabhavan et al., 2003). Lime does not pose a serious environmental threat as it has low solubility; however, its main disadvantage is the high level of lime sludge produced (Saravanabhavan et al., 2003). Conversely, using Na<sub>2</sub>S in liming during leather processing produces one of the most dangerous substances in the entire process and the direst threat to the environment because upon acidification ( $8.0 < \text{pH}$ ) (Dixit et al., 2015) wastewater containing sulfide releases toxic hydrogen sulfide (H<sub>2</sub>S) gas to the atmosphere (Nazer et al., 2006). The wide use of sulfide-rich elements leads, not only to serious

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concerns for the environment, but also retards the efficiency of effluent treatment plants (Bailey et al., 1982).

In the past few decades, many researches have been carried out on the treatment of tannery wastewater (Song et al., 2004; Haydar and Aziz, 2009; Mandal et al., 2010), but most of the techniques approached the issue by treating the mixed wastewater. The main drawback to these processes is the mixing of liming wastewater with other processed wastewater, which together can emit  $H_2S$  gas at lower pH, which, in turn, has an adverse effect not only on the atmosphere, but also on human health, even at very low concentrations (Muruganathan et al., 2004; Dettmer et al., 2012). Thus, the liming wastewater needs to be treated separately before it is mixed with other processed wastewater, or transferred to a central effluent treatment plant. So far, a group of researchers worked on treating the liming wastewater by the process of electrocoagulation (Şengil et al., 2009).

In this study, an approach was made to investigate the efficacy of treating liming wastewater by a simple oxidation-coagulation-filtration technique to bring it within permissible levels. Prior to applying the coagulant, the liming wastewater was treated with hydrogen peroxide ( $H_2O_2$ ), so that both un-ionized  $H_2S$  and ionized  $HS^-$  were removed as colloidal sulfur (Kohl and Nielsen, 1997). Finally, the supernatant was filtered through three layers of sand and charcoal-activated beds to minimize the pollution load.

## 2. Materials and methods

### 2.1. Sample collection

The sample liming wastewater was collected from the SAF Leather Industries Ltd., Jessore, Bangladesh. After the liming operation, liming wastewater was collected in polyethylene container, pre-washed with diluted nitric acid, and immediately transported to the laboratory for experimentation.

### 2.2. Materials

The reagents  $H_2O_2$  (50%, TPL, Thailand), aluminum sulfate (Sanfeng, China), sulfuric acid (Qingdao Lasheng, China), potassium ferricyanide and barium chloride (UNI-CHEM, China); ammonium chloride, dimethyl glyoxime (Loba Chemie, India); ferrous sulfate, ammonia, sulfuric acid, ethanol and charcoal activated (Merck, India) were purchased from a local scientific store. The fine and coarse sand were procured from the university water treatment plant.

### 2.3. Treatment of the liming wastewater

Liming wastewater was examined batch-wise through oxidation-coagulation-filtration techniques. The scheme for the treatment of liming wastewater is shown in Fig. 1. Firstly, physicochemical parameters of the untreated wastewater were determined. Secondly, untreated wastewater was poured into a beaker, to which  $H_2O_2$  was added; the mixture was stirred for 5 min on a magnetic stirrer to eliminate the evolved  $H_2S$  gas. Previously prepared aluminum sulfate ( $Al_2(SO_4)_3$ ) solution was added to the  $H_2O_2$  treated wastewater and stirred for another 5 min. The final pH of the mixture was adjusted with the help of diluted sulfuric acid and the mixture was kept on the magnetic stirrer for an additional 5 min for final mixing, and then the mixture was allowed to settle. After settling, physicochemical parameters of the supernatant were determined. Then, the supernatant was filtered through three-layer filter beds, which had a coarse sand layer at the top, carbon activated layer in the middle and fine sand layer at the bottom. The width of the filter bed layers were 1 cm for each of the two sand

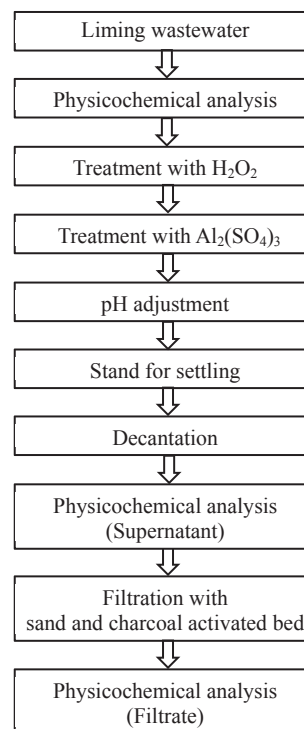


Fig. 1. Scheme for the liming wastewater treatment process.

layers and 0.5 cm for the carbon activated one. Finally, the physicochemical parameters of the filtrate were determined.

### 2.4. Analysis of physicochemical parameters

Total dissolved solids (TDS) and total suspended solids (TSS) were determined gravimetrically following the standard methods of APHA (APHA, 2012). Sulfide level was determined by following the official methods (SLC 202) of analysis (Society of Leather Technologist and Chemists (1996)). The sample was filtered by the glass fiber filter and the filtrate was diluted when necessary. 10 mL diluted filtrate, 15 mL buffer; 1 mL indicator and 1.5 mL barium chloride were pipetted into a conical flask and the flask was stoppered. The flask was left standing for 2–3 min to precipitate the sulfite. The solution was then titrated with 0.1 N potassium ferricyanide until the pink color disappeared. pH meter (UPH-314, USA) was used to measure the pH of the wastewater. Electrical conductivity (EC) and salinity were measured by using the conductivity meter (CT-676, Germany). The dissolved oxygen (DO) was measured with the DO meter (DO-580, Germany). Before measuring the parameters, all the meters were calibrated with standard solutions.

### 2.5. Process optimization

Assays were carried out to optimize the treatment parameters: pH,  $H_2O_2$  dose, coagulant dose and settling time. The optimized conditions were established by investigating the various physicochemical parameters, e.g. sulfide, TDS and TSS of the supernatant and filtrate. To optimize  $H_2O_2$  dose, varying doses were used for each batch 0.3, 0.6, 0.9, 1.2 and 1.5 mL, where other parameters were left unchanged, such as pH (7) and  $Al_2(SO_4)_3$  (2 g). To optimize pH, mild acidic to mild alkaline pH (5, 6, 7, 8 and 9) was preselected;  $H_2O_2$  (1.2 mL) and  $Al_2(SO_4)_3$  (2 g) doses were left unchanged. To optimize the coagulant dose, varying  $Al_2(SO_4)_3$  doses were used for

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