



# Electrochemical treatment of evaporated residue of soak liquor generated from leather industry



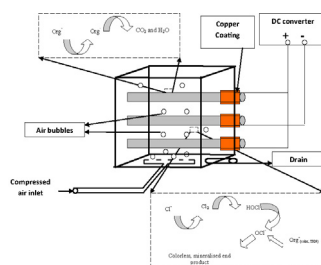
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## HIGHLIGHTS

- Electrochemical treatment of evaporated residue of soak liquor (ERSL) generated in Tannery.
- Copper coating on electrode surface and horizontal mounting of electrodes for ERSL treatment.
- Electrochemical oxidation of organic pollutants under high saline condition.
- The treated solution may be evaporated to dryness to get NaCl salt for hide/skin preservation.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The organic and suspended solids present in soak liquor, generated from leather industry, demands treatment. The soak liquor is being segregated and evaporated in solar evaporation pans/multiple effect evaporator due to non availability of viable technology for its treatment. The residue left behind in the pans/evaporator does not carry any reuse value and also faces disposal threat due to the presence of high concentration of sodium chloride, organic and bacterial impurities. In the present investigation, the aqueous evaporated residue of soak liquor (ERSL) was treated by electrochemical oxidation. Graphite/graphite and SS304/graphite systems were used in electrochemical oxidation of organics in ERSL. Among these, graphite/graphite system was found to be effective over SS304/graphite system. Hence, the optimised conditions for the electrochemical oxidation of organics in ERSL using graphite/graphite system was evaluated by response surface methodology (RSM). The mass transport coefficient ( $k_m$ ) was calculated based on pseudo-first order rate kinetics for both the electrode systems (graphite/graphite and SS304/graphite). The thermodynamic properties illustrated the electrochemical oxidation was exothermic and non-spontaneous in nature. The calculated specific energy consumption at the optimum current density of  $50 \text{ mA cm}^{-2}$  was  $0.41 \text{ kWh m}^{-3}$  for the removal of COD and  $2.57 \text{ kWh m}^{-3}$  for the removal of TKN.

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**Abbreviations:** ERSL, evaporated residue of soak liquor; RSM, response surface methodology; CCD, central composite design; VDS, volatile dissolved solids; APHA, American public health association; SS, stainless steel; C, concentration of ERSL % (w/v);  $j$  and  $j_L$ , current density and limiting current density ( $\text{mA cm}^{-2}$ );  $t_{\text{eco}}$ , electrochemical oxidation time (min); COD, chemical oxygen demand; TKN, total Kjeldahl nitrogen; ANOVA, analysis of variance;  $F$ , Faraday constant,  $96,487 \text{ (C mol}^{-1}\text{)}$ ;  $k_{\text{mCOD}}/k_{\text{m(TKN)}}$ , average mass transfer coefficient ( $\text{m s}^{-1}$ );  $A$ , electrode area ( $\text{m}^2$ );  $V$ , average cell potential (V);  $I$ , current (A);  $S_V$ , sample volume (L);  $K_e$ , equilibrium constant;  $R^2$ , regression coefficient;  $\Delta G$ , change in Gibbs free energy ( $\text{J mol}^{-1}$ );  $\Delta H$ , change in enthalpy ( $\text{J mol}^{-1}$ );  $\Delta S$ , change in entropy ( $\text{J mol}^{-1} \text{ K}$ );  $T$ , temperature (K);  $W$ , specific energy consumption ( $\text{kWh kg}^{-1}$  of COD/TKN);  $\text{COD}_0$  and  $\text{COD}_t$ , are the chemical oxygen demand at initial and at time 't' min;  $\text{TKN}_0$  and  $\text{TKN}_t$ , are the total Kjeldahl nitrogen at initial and at time 't' min;  $[\text{COD}]_{\text{opt}}$ , concentration of COD at optimum time (120 min).

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## 1. Introduction

Electrochemical treatment may be considered as an alternative process to conventional treatment technologies for the elimination of pollutants in liquid wastes [1]. Generally electrochemical processes are performed at ambient conditions and without addition of chemicals than those of other non-electrochemical treatments. Electrochemical treatment processes require compact reactors and simple equipments to control and operate the process. The process is relatively non-specific and applicable to a variety of contaminants but capable of preventing the production of unwanted side-products [2]. Hence, in recent years there has been an increasing interest in the treatment of industrial effluents by electrochemical methods as an alternative to conventional biological treatment systems [3]. There are reports on electrochemical oxidation of wastewater containing phenol [4–6], cyanides [7], nuclear wastes [8], human wastes [9], cigarette industry wastewater [10], textile wastewater [11], and tannery wastewater [12]. However, no report claims the treatment of ERSL, generated from leather industry, by electrochemical oxidation.

The salt laden soak liquor is being segregated, collected and evaporated in solar evaporation pans/thermal evaporator as no viable treatment technologies exist. The current practice is to dispose ERSL onto secure landfill site, but surface and ground water contamination is a serious environmental concern due to leaching of its constituents during runoff season. Hence, it has been advised by pollution control agencies to handle/treat ERSL residue with a suitable technology to modify the current disposal mechanism. Generally, destruction of organic pollutants in the electrochemical process was achieved by direct and indirect oxidations. In direct anodic oxidation, the organic pollutants are adsorbed onto the anodic surface, and destroyed by anodic electron transfer reactions, whereas in indirect oxidation the secondary oxidants such as chlorine, hypochlorite, hydroxyl radicals, ozone and hydrogen peroxide oxidise the organic pollutants in wastewater [13]. The generation of secondary oxidants such as chlorine and hypochlorite for the removal of organics and ammonia becomes more probable and thus the electrochemical oxidation was preferred over other technologies for the management of ERSL in leather sector. The resultant electrochemical oxidised solution can be considered for recovery of sodium chloride of high purity through selective precipitation technique [14].

The central composite design (CCD) is an effective design for sequential experimentation and allows a reasonable amount of information for testing interaction of selected parameters by response surface methodology (RSM) [15–17]. In the optimisation process, the responses can be simply related to the chosen factors by linear or quadratic models. A quadratic model, which also includes the linear and interaction terms, is shown in Eq. (1),

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{j < i=2}^k \sum_{i=1}^k \beta_{ij} x_i x_j + e_i \quad (1)$$

where  $Y$  is the response,  $x_i$  and  $x_j$  are the variables ( $i$  and  $j = 1$  to  $k$ ),  $\beta_0$  is the constant coefficient,  $\beta_j$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are interaction coefficients of linear, quadratic (second order) and interaction terms, respectively,  $k$  is the number of independent parameters and  $e_i$  is the error. The physical significance of the second order term is to determine the real function of the selected independent variable and the interaction terms are used to evaluate the interaction between the two selected independent variables. In this study, concentration of ERSL solution (% w/v), current density ( $\text{mA cm}^{-2}$ ) and electrochemical oxidation time (min) were selected as the operational (independent) variables for the removal of organic content;  $\text{COD}_t/\text{COD}_0$  and  $\text{TKN}_t/\text{TKN}_0$  were the response (dependent variables) from

the electrochemical treatment process for graphite/graphite system.

## 2. Materials and methods

### 2.1. Characterisation of evaporated residue of soak liquor (ERSL)

The ERSL was collected from a commercial export oriented tannery processing raw skins/hides into finished leather in Ranipet, Tamil Nadu, India. The industry had the provision to evaporate the soak liquor to dryness with moderate moisture. The ERSL was transported to the laboratory and stored in the dry atmosphere until for further use. The ERSL was characterised for moisture content, Chemical Oxygen Demand (COD), Volatile Dissolved Solids (VDS), Oils & fats,  $\text{NH}_4^+$ -N and sodium chloride by following the methods of APHA, 1999 [18] (Table 1).

### 2.2. Experimental set-up and electro chemical oxidation of ERSL

A glass jacketed electrochemical cell (length, 15; width, 3.5 and height, 15 cm) with a working volume of 500 mL was fabricated using poly acrylate material and used for the electrochemical oxidation ERSL solution by batch experiments. Three numbers of graphite electrodes (length, 15 cm and diameter, 2.5 cm) were positioned parallel to each other with an equidistance of 1 cm between the successive electrodes in the electrochemical cell. The electrodes were mounted onto the reactor walls horizontally and extended outside the reactor for the electrical connection as shown in Fig. 1. In order to reduce the electrical contact resistance and to ensure better connectivity, the graphite electrodes across their length were coated with metallic copper for a distance of 2 cm in one end to act as terminal for electrical connection. Similarly, another electrochemical cell with the same dimensions was fabricated with SS304 as anode and graphite as cathode materials. The AC to DC rectifier (0–60 V and 0–5 A) was used to supply predetermined current density and voltage for the experiments. The required bulk solution temperature of the electrochemical cell was maintained through water circulation in external jacket provided to the reactor/electrochemical cell using cryostat (Labnet, India). The oxygen required for the oxidation of organics in ERSL solution was supplied in the form of air using compressor at pressure  $2 \text{ kg cm}^{-2}$ . The air was distributed at the bottom of the electrochemical cell through fine bubble spargers.

The solution of ERSL was prepared by dissolving a known quantity of the dried ERSL in one litre of deionised water. The concentration of 60% (w/v) of ERSL means, 60 g of dried ERSL dissolved in 100 mL of distilled water. The suspended solids were filtered using glass fibre filter paper. Then, the ERSL solution (500 mL) was used in the electrochemical cell to optimise the process parameters. Aliquots of samples of volume 10 mL were withdrawn from the electrochemical cell at regular time intervals for the characterisation of pH, COD and TKN.

**Table 1**

Characteristics of evaporated residue of soak liquor (ERSL) generated from leather industry.

Sl. no.	Parameters	Values
1	Moisture content (%)	$12.3 \pm 1.5$
2	Chemical oxygen demand ( $\text{mg g}^{-1}$ )	$5.57 \pm 0.04$
3	Volatile dissolved solids ( $\text{mg g}^{-1}$ )	$18.8 \pm 2.5$
4	Oil and fats ( $\text{mg g}^{-1}$ )	$0.06 \pm 0.02$
5	Ammonium-nitrogen ( $\text{mg g}^{-1}$ )	$0.25 \pm 0.1$
6	Total Kjeldahl nitrogen ( $\text{mg g}^{-1}$ )	$0.68 \pm 0.3$
7	Soluble salt (%) (w/v)	$59.2 \pm 2.0$
8	Sodium chloride ( $\text{mg g}^{-1}$ )	$540 \pm 12.8$

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