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Optimization and Cost Analysis of Electrocoagulation for Phenolic Syntan Removal from Synthetic Tannery Wastewater

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Abstract

Syntans are phenol-based synthetically produced tanning agents used in tanneries for improving the filling, grain properties and dyeing property of the hide. A significant portion of syntans emanating in the tannery effluents along with other heterocyclic aromatic compounds have high pollution load and imposes severe environmental impact. Hence, the present study adopted an Electrocoagulation (EC) method with perforated aluminum electrodes for the removal of phenolic syntan (PS) from PS bearing synthetic tannery wastewater. The one-variable-at-a-time (OVAT) experimental analysis was performed to optimize the PS removal efficiency. The OVAT experiments were studied to understand the impact of operating parameters, i.e. initial pH (3 - 7), current density (5.8 - 23.6 mA/cm²), and initial phenolic syntan (PS) concentration (0.25 - 1.5 g/L). The mixing speed was maintained at 200 rpm in each experiment. The results demonstrated that the EC process could efficiently remove PS (83.43%) from synthetic wastewater at the following operating condition: initial pH= 4, current density= 17.64 mA/cm² and initial PS concentration= 0.25 g/L, and electrolysis time = 90 minutes. The cost analysis showed the economic feasibility of EC techniques with a total cost of $3.46/m³ and demonstrated the need for a balance between the EC unit effectiveness and operating costs for economically viable wastewater treatment in the tannery sector. The study provided valuable information for improved sustainable and economical effluents treatment techniques in tanneries.

Keywords

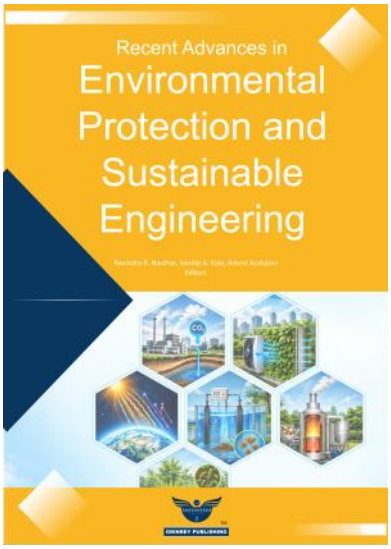
Carbonation Electrocoagulation, Syntan, One-Variable-at-a-Time (OVAT) Optimization, Operational cost.

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1. Introduction

The tanning process has been identified as among of the most water-polluting manufacturing processes producing huge quantities of wastewater with a highly intricate composition due to the presence of organic and inorganic contaminants. Among the organic contaminants, synthetic tanning agents (syntans), which are a type of phenol-based substance have been commonly utilized to enhance filling, grain, and color properties of leather. However, owing to their complex aromatic structure, they are recalcitrant in nature and highly toxic, leading to considerable environmental implications [1, 2]. These phenolic derivatives, obtained from phenols, cresols, naphthalene, melamine, and acrylic derivatives by formaldehyde-sulfuric acid treatment reactions, tend to remain unabsorbed by hide/skin in most tanning operations and are deposited into tannery wastewater streams [3, 4]. Due to their persistence, and resistance to biodegradation, these compounds render traditional biological treatment plants ineffective [3, 5]. Therefore, untreated or partially treated syntan-containing tannery wastewaters can have drastic effects on aquatic environments due to their bioaccumulation and toxicity to biotic species.

Various physico-chemical and advanced oxidation processes i.e., Fenton oxidation, ozonation, UV irradiation, and photo-catalysis have been explored for syntan degradation [6, 7]. Although such strategies can be efficient in degrading phenolic compounds, their use in real-scale tannery effluent treatment plants is limited due to the high operating costs, energy demands, and need for intricate operation control [1]. Alternatively, adsorption and coagulation processes, although cost-effective, are usually linked with very high hazardous sludge generation and restrictions in its disposal. Therefore, there is an urgent need for alternative treatments options that strike a balance between efficiency, sustainability, and economic viability.

Electrocoagulation (EC) has proven to be a viable solution for treating industrial effluents from various source, such as tannery, textile, food processing, and electroplating industries [8, 9]. The process involves in-situ generation of coagulants via electrochemical disintegration of the sacrificial anode (usually aluminum or iron), which destabilize contaminants, resulting in their aggregation and removal. the major benefits of this process are minimal chemical usage, easy operation, lower sludge yield, and suitability for on-site treatment [10, 11]. In recent research, EC was seen to attain significant elimination of chemical oxygen demand, total organic carbon and phenolic syntans at engineered conditions [7, 12]. However, limited systematic studies have been performed by optimizing the operational parameters like initial pH, current density, and initial syntan concentration in the influent for enhanced syntan removals, for reducing the overall cost of operation and electrical energy requirements.

The current research adds to the prevalent body of work by systematically examining a lab-scale EC process using stationary perforated Al electrodes for removal of phenolic syntans from simulated tannery wastewater. In contrast to the previous studies that mainly considered pollutant removal efficiencies, the current work prioritizes a holistic assessment by coupling treatment performance with operating cost and energy consumption. Optimization was performed using the one-variable-at-a-time (OVAT) method to investigate the significant role of input operating variables like pH, current density, and initial syntan concentration on the performance of the lab-scale EC treatment unit.

The main objective of the present investigation is to maximize the electrocoagulation (EC) process for efficient elimination of phenolic syntans from model tannery wastewater with a view to ensuring cost-effectiveness. In the above context, the study aimed at assessing the impact of the most important operating factors (viz., initial pH, current density, and pollutant concentration) on the efficacy of syntan removal. Special focus was given to the detection of the best operating conditions which ensured a proper balance between high removal efficiency, smaller electrolysis time, lower specific energy consumption (SEC), and

lowest operational costs. Additionally, the cost–benefit analysis was carried out for the EC process in order to determine its economic viability, so as to position the treatment process as sustainable and scalable for tannery wastewater treatment. The general direction of this investigation is toward building deeper understanding of EC process for efficient treatment of phenolic compound-contaminated effluents, and ultimately achieving environmentally friendly and industry-specific wastewater treatment approach.

2. Materials and Methods

2.1. Chemicals

In this investigation, commercial Phenolic Syntan (Basyntan DI) was acquired from M/s BASF India Ltd., Kanpur, India. Various other chemicals were utilized in this study i.e. hydrochloric acid (35 – 37%, Molychem, Thane, India), sodium hydroxide (AG,98%, SRL, Maharashtra, India), sodium chloride (AG, 99%, Merk Life Science Pvt. Ltd., Mumbai, India).

2.2. Electrocoagulation setup

The lab-scale batch EC unit was constructed of plexi-glass and had a working capacity of 1000 mL. EC reduction was carried out using Al anode-cathode electrodes. The Al electrodes were of dimensions 8.5 cm × 5 cm × 0.2 cm, with an effective surface area of 85.63 cm^2. Each electrode had a total of 10 perforations, with each opening having a diameter of 0.2 cm. Perforated aluminum electrodes were used to enhance electrolyte circulation and mass transfer within the EC reactor. The perforations can help release gas bubbles, decrease the concentration polarization, and enhance efficient utilization of the electrode surface, leading to better EC performance than the traditional solid plate electrodes [11, 13]. The two aluminium electrodes had a fixed spacing of about 2 cm., A magnetic stirrer was employed to maintain a normalized solution throughout the treatment process at a constant rate of 200 RPM. Anode-cathode were linked to a direct current (DC) power source (0 - 5 A, 0 - 30 V; Testronix) in a monopolar arrangement (Figure 1). The typical EC reactions expected in the reactor are given below in Table 1 [13].

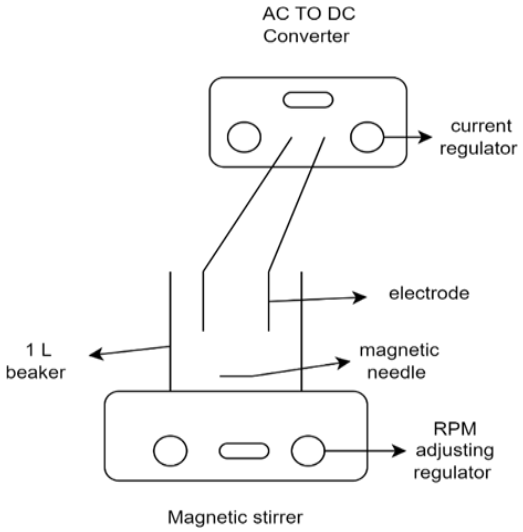


Fig. 1. EC Experimental Setup

Table 1. Anode and Cathode Reactions in EC Process.

At Anode	$Al\ (s) \rightarrow Al^{3+}\ (aq) + 3e^-$ $2H_2O\ (l) \rightarrow O_2\ (g) + 4H^+(aq) + 4e^-$
At Cathode	$2H^+\ (aq) + 2e^- \rightarrow H_2(g)$ $2H_2O\ (l) + 2e^- \rightarrow H_2(g) + 2OH^-\ (aq)$
Overall reaction	$Al^{3+}\ (aq) + 3OH^-\ (aq) \rightarrow Al(OH)_3\ (s)$

2.3. Experimental Procedure

To prevent variations in wastewater properties, simulated tannery solution was freshly created at the beginning of each experimental run. The desired syntan concentrations (i.e., 0.25 - 1.5 g/L) were maintained in the simulated tannery solution by adding required quantities of syntan powder to tap water. The characteristics of the prepared simulated wastewater are shown in Table 2.

Table 2. Characteristics of Synthetic Wastewater

Concentration of Phenolic Syntan (g/L)	pH	UV_{280} Absorbance	Conductivity (µS/cm)
0.25	7±0.3	1.32	760 ± 50
0.50	7±0.3	2.65	760 ± 50
1.0	7±0.3	5.3	760 ± 50
1.50	7±0.3	7.95	760 ± 50

0.1M HCl or 0.1M NaOH were added in solution to maintain the initial pH (3-7). The conductivity was maintained at 1.50 mS/cm during electrolysis by adding 1.5 g/L of NaCl to the synthetic wastewater. During each experimental run, samples were collected from the EC batch reactor for data analysis at an interval of 10 minutes upto 90 minutes. Later, the treated effluent samples were filtered by Whatman filter paper (0.45-micron pore size) and analyzed immediately to avoid estimation errors. Samples of raw and treated syntan were analyzed for UV280 to determine the degree of degradation of syntan. At the start of an experimental run, the electrodes were rinsed with diluted HCl and wiped with distilled water, to eliminate passivation from the electrode surface. The process optimization was performed in a systematic OVAT mode, where one parameter was varied within a specific range and the remaining three input operating variables were kept constant. The parameters were sequentially optimized based on successive measurements in experiments to get the optimal working conditions for maximum PS removal (Table 3).

Table 3. Experimental analysis Layout

Operating parameters their value	Experimental Phases		
	Phase -I	Phase -II	Phase -III
Initial pH = 3-7	Variable	Fixed (optimal)	Fixed (optimal)
Current intensity (mA/cm^2) = 5.8 – 23.3	Fixed	Variable	Fixed (optimal)
Initial PS concentration (g/L) = 0.25 – 1.5	Fixed	Fixed	Variable

In this study, the OVAT method was chosen due to its simplicity and systematic approach to assess the distinct impact of each operational variable on the EC efficiency while fixing the other variables constant. Therefore, the OVAT was considered suitable since the main objective of this study was to determine the individual effect of initial pH, current density and phenolic syntan concentration and determine suitable operational conditions for a laboratory-scale EC system. But this approach has inherent constraints. In contrast to statistical optimization methods like Response Surface Methodology (RSM), OVAT has the disadvantage of not being able to assess interactions between variables, may require a higher number of experiments to find the optimal conditions [14]. Hence, while OVAT is useful for early process optimization and mechanistic comprehension, future investigations should use multivariate optimization methods like RSM for investigating variable interactions as well as improving process efficiency.

2.4. Operational Cost Estimation

The purpose of treatment cost analysis for EC unit was to understand the economic benefit of the process without having to compromise on the system performance. The unit energy consumption and electrode dissolution were determined for the EC process. Electrode dissolution and energy consumption were determined for the optimum working condition obtained for the treatment approach. The operating cost was calculated by the equation as follows [13]:

$$Operating\ Cost\ \left(\frac{\$}{m^3}\right) = (a \times EMC) + (b \times ELC) \quad (1)$$

Where,

'a' refers to the unit costs of electrode material (0.73 USD kg^{-1}) and 'b' refers to unit cost of electric energy (0.078 USD kWh^{-1}). EMC refers to electrode material consumption (kg m^{-3}) and ELC refers to energy consumption (kWh m^{-3}).

2.4.1. Electrode material consumption

In EC process, the mass of electrode dissolution was determined using Faraday law [10].

$$EMC = \frac{(M \times I \times t)}{(Z \times F \times V)} \quad (2)$$

Where, M: Molecular weight material used in EC process i.e. Al, I: Current intensity (amp), t: Time (sec), z: valency of metal ion (e^-/mol), F: Faraday constant, (96500 coulomb/mol), V: volume of solution (m^3).

In conventional chemical precipitation procedures, the coagulant dose is determined from the quantity of disintegrated anode material. In another way, the amount of coagulant needed for traditional chemical precipitation processes is directly proportional to the quantity of material which emerges from the anode in EC. Therefore, the cost related to the consumption of the anode is identical for EC procedure and for conventional chemical coagulation methods. The comparison of chemical coagulation and the EC process does not take into account this cost element.

2.4.2. Energy consumption

Energy consumption for treatment unit at optimum condition is calculated using following formula [10].

$$SEC = \frac{V \times I \times T}{Q \times 1000} \quad (3)$$

Where, SEC: Specific energy consumption (kWh/m^3) V: Voltage, I: Current (A), T: Operating time (hr), Q: Volume of treated wastewater (m^3)

3. Results and Discussion

3.1. Impacts of influent pH

The influent pH of simulated solution is a critical parameter that can directly influence the efficacy of the EC process. This input variable primarily governs the solubility and precipitation of metal hydroxides in the

EC process [15]. pH also determines the elimination mechanism of contaminants in the EC reactor [15]. The impact of various initial pH (3-7) on the PS elimination in synthetic solution was investigated in EC method. Whereas, other operating conditions i.e. CD and initial PS concentration to be 11.6 mA/cm^2 and 0.5 g/L, respectively (shown in figure 2).

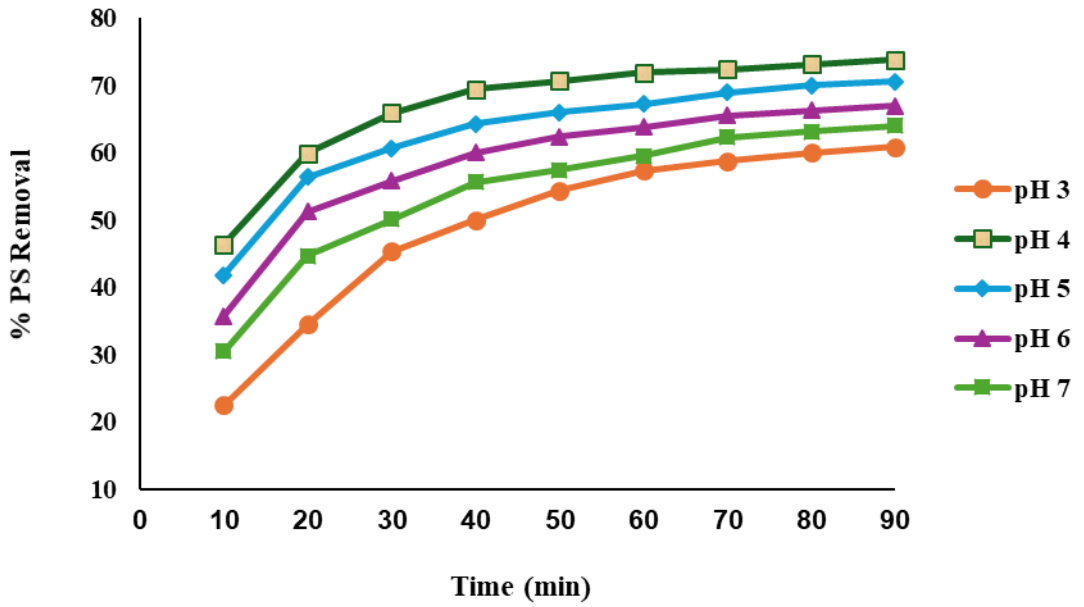


Fig. 2. Impacts of varying influent pH on the PS removal in EC process

After 90 minutes of electrolysis time (ET), 60.88% of the PS was removed at influent pH 3. As the influent pH increased from 3 to 4, PS elimination increased by 13% after 90 minutes of ET. At 90 minutes of ET, PS elimination reduces by 7.2% to 3.6% and 3% when the influent pH increases from 4 to 5, 5 to 6, and 6 to 5, respectively. The highest PS removal (73.8%) was obtained at an influent pH of 4, which is considered the optimal pH for PS removal. Therefore, influent pH 4 was maintained constant in subsequent trials when other parameters were one at a time changed. Fig. 2 indicates that PS removal was found to be more efficient under weakly acidic conditions, while strongly acidic and alkaline conditions were found to be less efficient. During experimental analysis, pH of treated solution was also observed to change in alkaline direction during treatment and ranges from 5 to 7 at influent pH of 4. This was due to the production of OH^- ions in EC process as a result of electrolysis of wastewater (as clear from Table 1), which further increased the pH of treated solution. Kim et.al, explains this behavior expected and asserts $Al(OH)_4^-$ to be the Al species in very alkaline conditions, which have poor coagulation properties [16]. Anode-formed Al cations can produce polymeric species like $Al_{13}O_4(OH)_{24}^{7+}$ and precipitated $Al(OH)_3$, and thereby make coagulation more effective when carried out under a weakly acidic condition (pH 6-7) [17]. These $Al(OH)_3$ species can adsorb the PS compounds and eliminate them from solution [18].

3.2. Impact of Current Density

Current density (CD) is a critical factor in regulating the rate of EC reactions [19]. This parameter plays an important role in electrode disintegration, power consumption, pH of treated sample and the yield of $Al(OH)_3$ flocs [20]. Thus, this parameter may have substantial effects on pollutant removal efficiencies. Figure 3 shows the impact of CD on PS elimination efficiency. Here, sets of experiments were conducted at various CD (5.8 – 23.3 mA/cm^2). Remaining factors i.e. influent pH and initial PS concentration were kept constant as 4 and 0.50 g/L respectively.

Figure 3 (a) indicates the PS removal 69% was found at minimum CD of 5.8 mA/cm^2 after 90 minutes of ET. An improvement in CD from 5.8 to 11.6 mA/cm^2 and from 11.6 to 17.6 mA/cm^2 resulted in a rise in the

PS elimination from 69% to 73.8% and 73.8% to 83.43%, respectively after 90 minutes ET. However, the main consequences of increasing CD were the elevated rate of anode dissolution. This should raise the amount of $Al(OH)_3$ flocs in the solution, and subsequently improve the rates of pollutants removal [21]. Table 4 displays that a rise in CD from 5.8 to 11.6 mA/cm^2 and from 11.6 to 17.6 mA/cm^2 enhanced higher anode dissolution (from 0.252 to 0.503 kg Al/m^3 and from 0.503 to 0.755 kg Al/m^3) after 90 min ET.

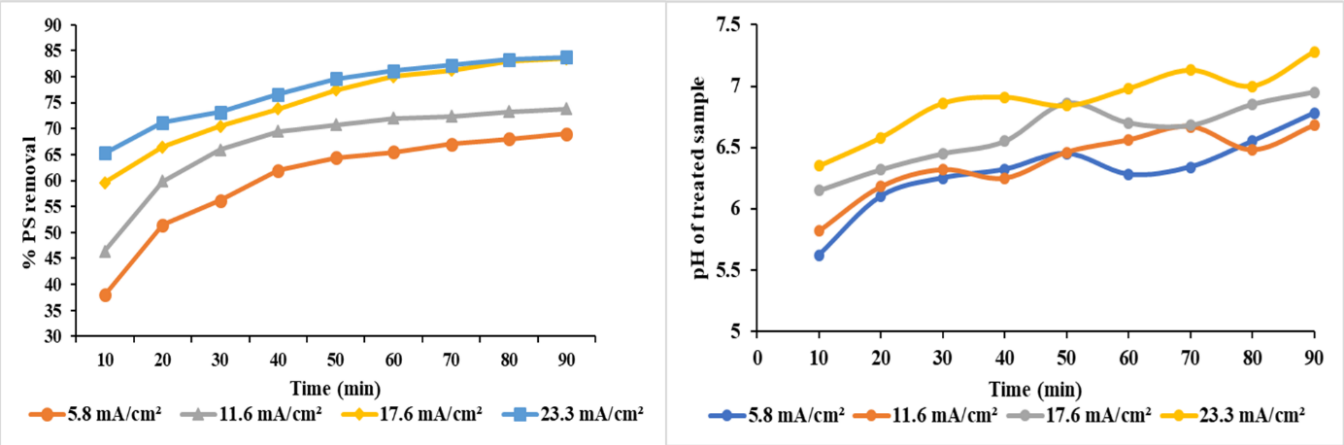


Fig. 3. (a) Impacts of varying CD on the PS removal in EC process. (b) Variation of treated solution pH with electrolysis time at various CD

Figure 3(b) shows the shift of pH of treated solution with electrolysis time at various CD. pH progressively increased over the entire EC procedure regardless of CD. It raised from 5.62 to 6.78, 5.82 to 6.68, 6.15 to 6.95 and 6.35 to 7.28 at 5.8, 11.6, 17.6 and 23.3 mA cm^{-2}, respectively. The rise in pH is primarily due to the production of hydroxide ions (OH^-) at the cathode throughout the reduction of water, which favors the production of amorphous $Al(OH)_3$ flocs that facilitate the elimination of pollutants [21]. At higher current densities, relatively higher value of final pH was found due to greater rate of electrochemical reactions and formation (OH^-). However, the treated solution still remained in the near-neutral pH range (~6.7–7.3), which is suitable for the generation of $Al(OH)_3$ species and efficient EC performance [22].

Table 4. Energy consumption, electrode dissolution and operating cost for various CD in EC process

Time	CD = 5.8 mA/cm^2			CD = 11.6 mA/cm^2			CD = 17.6 mA/cm^2			CD = 23.6 mA/cm^2		
(min)	SEC	EMC	Cost	SEC	EMC	Cost	SEC	EMC	Cost	SEC	EMC	Cost
10	0.27	0.028	0.10	0.917	0.056	0.24	2.03	0.084	0.41	3.53	0.112	0.62
20	0.50	0.056	0.20	1.767	0.112	0.46	4.00	0.168	0.81	7.00	0.224	1.22
30	0.70	0.084	0.29	2.550	0.168	0.69	5.85	0.252	1.21	10.30	0.336	1.82
40	0.90	0.112	0.39	3.333	0.224	0.91	7.60	0.336	1.59	13.47	0.447	2.40
50	1.04	0.140	0.48	4.000	0.280	1.13	9.38	0.419	1.98	16.50	0.559	2.98
60	1.20	0.168	0.57	4.700	0.336	1.34	10.95	0.503	2.35	19.60	0.671	3.56
70	1.34	0.196	0.66	5.250	0.391	1.55	12.60	0.587	2.72	22.63	0.783	4.13
80	1.40	0.224	0.747	6.000	0.447	1.77	14.20	0.671	3.09	25.33	0.895	4.67
90	1.50	0.252	0.83	6.600	0.503	1.97	15.75	0.755	3.46	28.20	1.007	5.23

Where, SEC in kWh/m^3; EMC in Kg Al/m^3; Cost in $/m^3.

According to Faraday's law (Equation 2), electrolysis time (ET) and CD are directly proportional to the amount of Al^{3+} ions produced during EC practice. Hence, the production of $Al(OH)_3$ flocs rises with rising CD for higher pollutant removal. Further, higher current densities have been reported to improve generation of tinny bubbles at the cathode, hence leading to better flocculated contaminant flotation in the EC unit [22]. As shown in Figure 3, an increase of CD from 17.6 to 23.3 mA/cm² resulted in merely a negligible rise in PS removal (0.28%), whereas SEC and OC rose by 44.15% and 33.84%, respectively (Table 4). This is because the efficiency of removal was not proportional to the increase in current density due to the fact that a greater portion of the current might be used for the oxygen evolution reaction (OER), which is competing with the anodic dissolution of aluminum. Therefore, the current efficiency for the production of Al^{3+} ions becomes lower and the energy consumption becomes greater. This phenomenon has been well known in the case of EC processes using Al electrodes, due to the increased rate of oxygen evolution with higher anodic potential (Mollah et al., 2004; Chen, 2004; Hakizimana et al., 2017). The best CD was thus determined to be 17.6 mA/cm² from these observations.

3.3. Impact of Initial PS Concentration

In the instance of industrial effluents the aqueous concentration of the pollutant emitted from various manufacturing processes varies widely. Therefore, the impact of different initial PS amounts on the efficacy of the EC procedures should be explored. The effect of different initial phenolic syntan concentration (0.25-1.5 g/L) in the EC procedures was studied while the remaining parameters were maintained as CD 11.6 mA/cm2 and pH 4. The results (Figure 4) demonstrate a noticeable reduction in PS removal efficacy with rise in initial PS concentration.

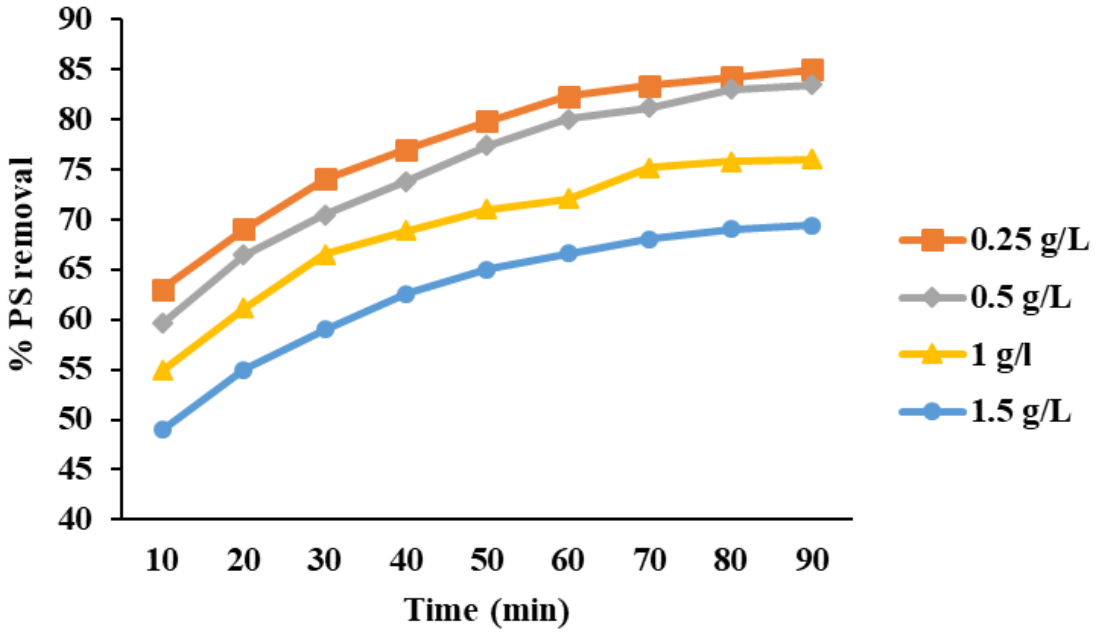


Fig. 4. Impacts of influent PS concentration on the PS elimination by EC process

The PS removal efficiencies were 85 %, 83.43 %, 76.03 % and 69.4 % at various PS concentrations of 0.25 g/L, 0.50 g/L, 1.0 g/L and 1.5 g/L respectively, after 90 minutes ET. Lower PS removal with higher influent PS concentrations may have been caused due to congestion of PS compounds onto the surface of $Al(OH)_3$ flocs. The PS concentration was substantially higher than the overall floc quanity. The influent PS concentrations doesn't have any impact on the amount of anode dissolution based on Faraday's law. For the fixed CD and ET, the rate of generation of $Al(OH)_3$ flocs produced would be very close to being constant [11, 17].

3.4. Comparison of various treatment approaches for syntan elimination

Table 5. Comparison of various treatment techniques for syntan removal.

Treatment method	Operating conditions	Removal efficiency	Main limitations	Reference
Ozonation	pH = 7, O_3 dose = 1/h	COD = 91%, tannin = 98%, UV absorbance = 99%	High ozone demand and energy consumption; partial mineralization	[26]
Ozonation	Initial syntan = 500 mg/L, pH = 7	COD removal = 84.2% UV_{280} = 92%	Requires ozone generation and specialized equipment	[27]
Biological degradation (*Trametes versicolor*)	Initial syntan = 500 mg/L; Treatment time = 8 days	COD = 76.66%, TOC 72.94%; higher with co-substrate	Long treatment time	[28]
Biological degradation (*Trichoderma aureoviridae*)	Syntan = 400 mg/L; incubation = 8 days	COD = 82.3%, TOC = 51.6%; BOD = 41.6%	Slow kinetics; suitable mainly as biological polishing	[29]
EC process (Present study)	Al electrodes, CD = **17.6 mA cm^{-2}**, pH = 5, ET = 90 min	**Syntan removal = 83.43%**,	Electrode consumption and sludge generation	Present study

Table 4 illustrates the comparison between the efficacy of the EC process carried out in the current research and the performance of other processes that have been used in the treatment of phenolic syntan. The use of ozonation in the treatment of phenolic syntans has been shown to result in up to 98% removal of tannins and 91% removal of COD, as well as improvement of wastewater biodegradability; however, the process is associated with continuous ozone production and high energy consumption. The use of Trametes versicolor and Trichoderma aureoviridae in biological wastewater treatment processes is environmentally friendly, but it takes days to degrade phenolic syntans because they are very difficult to degrade. On the contrary, the EC process conducted in the present study resulted in 83.43% removal of phenolic syntan within 90 min using optimal operation conditions, without any addition of external oxidants.

4. Conclusion

In this study, the batch scale electrocoagulation (EC) process with aluminum anode was successfully used for removal of phenolic syntan (PS) from simulated tannery wastewater. OVAT analysis revealed an optimum pH of 4, which not only increased PS removal efficiency but also decreased electrolysis time drastically, consequently reducing specific energy consumption (SEC) and cost of treatment. Current density was also important, and 17.6 mA/cm² was the optimum operating point, providing a 14.43% higher PS removal than that at 5.8 mA/cm² without incurring the steep SEC (44.15%) and cost (33.84%) rise experienced at a higher current density (23.3 mA/cm²). More importantly, the highest PS removal occurred at the lowest initial PS concentration (0.25 g/L). The best elimination efficiency (83.43%) was found at optimal conditions of CD 17.6 mA/cm², initial pH 4, and PS concentration 0.25 g/L, with the corresponding SEC of 15.75 kWh·m^{-3} and operational cost of 3.46 \$·m^{-3}.

In general, the results showed that EC treatment using aluminum electrodes is a highly efficient and economically feasible treatment method for phenolic syntan-bearing tannery wastewater. This process has

great potential for industrial applications for achieving high pollutant removals coupled with low energy requirements and operational costs, and thereby contributing to sustainable tannery wastewater treatment.

The results obtained from this research study are very important regarding the EC of phenolic syntan in the laboratory setup. However, tannery effluent is a complex system that contains high concentrations of organic and inorganic contaminants which can affect the efficiency of the electrocoagulation process. Therefore, wastewater containing higher concentration of contaminants requires more electrolysis time, higher current density, or multiple stages in order to obtain similar removal efficiencies. Despite being very important in designing the process, the optimum operating parameters found in this study must be investigated further using actual tannery effluent.

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