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Original scientific paper

Punched zinc-aluminium electrodes for efficient treatment of soap wastewater

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Abstract

This study investigates the efficiency of the electrocoagulation process using punched zinc and aluminium electrodes for the treatment of soap and detergent wastewater. Although the industry generates relatively small volumes of effluent during production, significant environmental concerns arise from its widespread domestic and industrial discharge. The objective was to optimize key operational parameters for maximum chemical oxygen demand (COD) and turbidity removal. Experimental design was carried out using response surface methodology with a Box-Behnken design to evaluate the effects of voltage, electrolysis time, and electrode distance. Optimal conditions were identified at 30 V, 60 min electrolysis time, and 2 cm electrode spacing, achieving removal efficiencies of 87.22 % for COD and 85.7 % for turbidity. Statistical analysis using ANOVA confirmed the reliability of the quadratic model, showing strong agreement between predicted and experimental results. The findings demonstrate that punched electrodes significantly enhance EC performance, and further development of advanced electrode materials could improve treatment efficiency.

Keywords

Pollutant removal; electrocoagulation; response surface methodology; Box-Behnken design; chemical oxygen demand; turbidity

Introduction

The increasing demand for freshwater, coupled with growing environmental concerns, has emphasized the importance of wastewater treatment, recycling, and reuse. Industrial activities are major contributors to wastewater generation, and the soap and detergent manufacturing industry is particularly significant because it consumes large volumes of water during processing, formulation, equipment cleaning, and other associated operations [1-3]. Wastewater generated from these activities may contain substantial concentrations of organic matter, surfactants, suspended solids, colour, and other dissolved and colloidal constituents [3]. Consequently, parameters such as biochemical oxygen demand (BOD), chemical oxygen demand (COD), turbidity, colour, and surfactant concentration can vary considerably depending on the manufacturing process and operating conditions [4-6]. The presence of fine colloidal particles and surface-active compounds can further complicate treatment because these contaminants are often stable in aqueous media and are not readily removed by conventional treatment methods [7,8].

Various treatment approaches, including physical, biological, chemical and physicochemical processes, have been employed for the removal of contaminants from industrial wastewater. Conventional chemical coagulation, precipitation, adsorption, membrane filtration, and biological treatment can be effective under appropriate operating conditions; however, their application may involve chemical consumption, generation of considerable quantities of sludge, complex operation, or inadequate removal of certain persistent contaminants [9-12]. In this context, electrocoagulation (EC) has emerged as an attractive alternative for the treatment of diverse industrial wastewaters because the coagulant is generated *in situ* through the electrochemical dissolution of a sacrificial electrode [13-15]. Recent studies have reported that perforated or punched electrode configurations can improve electrocoagulation performance by enhancing hydrodynamic conditions, reducing electrode passivation, improving mass transfer, and promoting more effective pollutant removal [16-18]. In addition, zinc-based electrode systems have shown promising results for the treatment of industrial wastewater due to their efficient coagulant generation and operational stability [17,19].

An EC system generally consists of an electrolytic reactor containing the wastewater as the conducting medium and two or more electrodes connected to an external power source. When an electric current is passed through the system, oxidation occurs at the sacrificial anode, resulting in the release of metal ions into the wastewater [20,21]. The released metal ions undergo hydrolysis and subsequently form various hydroxide species, which function as coagulants and adsorbents [22,23]. These species interact with dissolved contaminants and destabilize suspended and colloidal particles, thereby promoting their aggregation into larger flocs. The hydroxide ions contribute to the formation of metal hydroxide precipitates, while the hydrogen bubbles generated at the cathode can attach to the flocs and promote their separation through flotation [24,25]. Depending on the wastewater characteristics and operating conditions, the resulting flocs can subsequently be separated through sedimentation, flotation, or a combination of both processes.

For a zinc anode, the anodic dissolution can be expressed as $Zn \rightarrow Zn^{2+} + 2e^-$, while at the cathode, water reduction generates hydrogen gas and hydroxide ions according to the reaction $2H_2O + 2e^- \rightarrow$ $\rightarrow H_2(g) + 2OH^-$.

The present study investigates the treatment of soap and detergent wastewater through electrocoagulation using punched zinc as the sacrificial anode and punched aluminium as the cathode. The punched electrode configuration was expected to enhance electrocoagulation performance by increasing the effective electrode surface area available for wastewater interaction, improving electrolyte circulation, and facilitating ion transport within the reactor, thereby

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promoting superior mass transfer conditions [17,26,27]. In addition, the perforations in the electrodes may support the efficient release and movement of gases generated during electrolysis, reducing electrode passivation and ensuring continuous coagulant generation. Although a direct comparison with conventional flat electrodes was not conducted, the enhanced treatment efficiency observed in this study may be attributed to these advantages of the punched electrode design. Furthermore, response surface methodology (RSM) was employed to optimize the electrocoagulation process by evaluating the individual and interactive effects of applied voltage, electrolysis time, and electrode spacing [28,29]. A full quadratic model was developed to establish the relationship between operating parameters and pollutant removal efficiency, and its predictive capability was assessed by comparing model-generated responses with experimental results. The RSM approach enabled efficient process optimization with a reduced number of experimental runs while providing valuable insights into the significance and interactions of the selected process variables [30-32].

Experimental

Response surface methodology

A glass reactor measuring 14×9×15 cm was used for the batch electrocoagulation process. The reactor's working volume was 1 L. The EC unit consists of two punched zinc and aluminium electrodes connected in parallel in the reactor with a DC power supply. The electrodes were 150×34×1 mm, with four punches having a diameter of 0.5 mm and a 1 cm centre to centre distance. These electrodes were immersed in a beaker containing a detergent wastewater sample, with the anode and cathode at a specific distance apart. The whole setup was kept on the magnetic stirrer to maintain homogeneous mixing of the solution in the reactor during the process. The schematic representation of the experimental setup is shown in Figure 1.

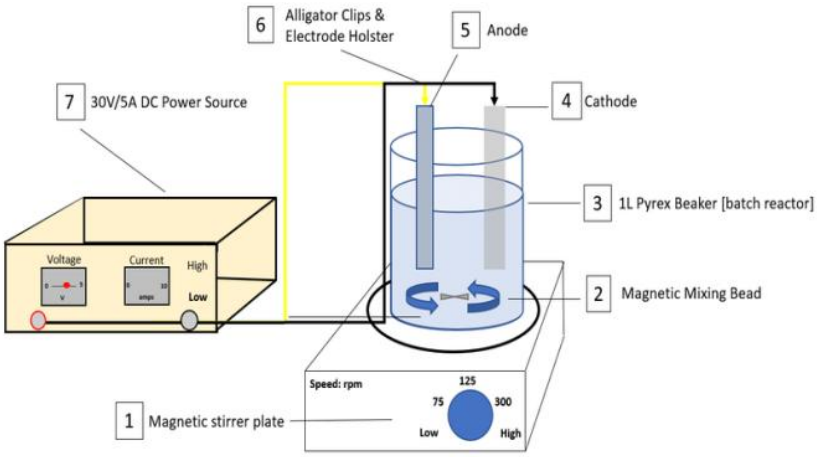


Figure 1. *Typical electrocoagulation setup*

The wastewater sample was collected from Maharaj Soaps Industry Private Limited, which is located near KIADB Industrial area Lokikere main road, Davangere, Karnataka-577005 and the sample contained impurities generated in the industry from different manufacturing steps. Some of the initial characteristics such as pH, total suspended solids, turbidity, BOD, COD and alkalinity were tested for raw wastewater by means of laboratory analysis. The initial physicochemical characteristics of the wastewater, including pH, conductivity, total dissolved solids (TDS), temperature, COD, BOD, turbidity and alkalinity, were determined prior to the electrocoagulation experiments to establish the baseline quality of the wastewater. Before each experimental run, the zinc and aluminium electrodes were

cleaned with abrasive paper, washed thoroughly with distilled water, and dried at room temperature to remove any impurities present on the surface. Throughout the electrocoagulation process, the wastewater was continuously mixed using a magnetic stirrer operating at 600 rpm to ensure uniform mixing and stable operating conditions. After analysing the initial characteristics of the wastewater, batch experimental studies were conducted to optimize the various parameters such as voltage, electrolysis time, and electrode distance using RSM. RSM uses an experimental design such as the Box-Behnken design (BBD) to fit a model by the least-squares technique.

Box-Behnken design has specific positioning of design points and always has three levels for each factor coded as −1, 0 and +1. It has been created for estimating a quadratic model fitting and the use of the second-order polynomial model can return higher-quality results in terms of precisely describing the linear relationships and quadratic impacts. Equation (1) is used to calculate the total number of experiments (N) required:

$$N = 2k(k-1) + C_0 \quad (1)$$

where k stands for the number of factors and C_0 signifies the number of central points.

In this study, the Box-Behnken statistical experimental design with RSM was used to investigate the effect of three independent variables, namely voltage (X_a: 10 to 30 V), electrolysis time (X_b: 30 to 90 min), and electrode distance (X_c: 2 to 6 cm) on coagulation. Table 1 lists the independent variables and their levels for EC in the Box-Behnken design. In this design, 15 experiments were assigned using three independent variables with three levels in MINITAB software [33].

***Table 1**. Independent variables and their levels for electrocoagulation treatment*

Independent variables	Factor	Level		
		−1	0	+1
Voltage, V	X_a	10	20	30
Electrolysis time, min	X_b	30	60	90
Electrode distance, cm	X_c	2	4	6

To perform regression analysis, experimental data were fitted to a full quadratic model, providing regression coefficients. The statistical analysis of the model was performed and the adequacy of RSM is justified using analysis of variance (ANOVA). ANOVA was applied to evaluate the statistical significance of the model. The model summary expressed the quality of the quadratic model equation by the regression coefficient R^2 and adjusted R^2 [34]. The adequacy of the model was evaluated by constructing diagnostic plots such as predicted versus actual plots for the experimental data obtained from this study. The surface plots were developed using the software, and these plots provide a three-dimensional view of the COD and turbidity removals with different combinations of independent variables [35]. The full quadratic model used in the response (Y_i) is described by Equation (2).

$$Y_i = \beta_0 + \beta_a X_a + \beta_b X_b + \beta_c X_c + \beta_{aa} X_a^2 + \beta_{bb} X_b^2 + \beta_{cc} X_c^2 + \beta_{ab} X_a X_b + \beta_{ac} X_a X_c + \beta_{bc} X_b X_c \quad (2)$$

where β_0, (β_a, β_b, β_c), (β_{aa}, β_{bb}, β_{cc}) and (β_{ab}, β_{ac}, β_{bc}) are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively; and X_a, X_b and X_c are the independent variables. Y_i indicates the removal efficiency, %.

Results and discussion

Figure 2 represents the comparison between experimental and predicted values shown in Table 2. From Figure 2, it is observed that the model predicted values matched the experimental (actual) data, with all the points close to the diagonal line. Actual values are the measured response data for a particular run (experiment) and the predicted values are evaluated from the model. The

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diagonal line indicates that the developed model adequately predicts each response. To develop a response surface regression model, a full quadratic model (Equation (2)) was applied to experimental observations of the removal efficiencies of COD (Y_1) and turbidity (Y_2) and the quadratic regression models are given by Equations (3) and (4) for Y_1 and Y_2, respectively:

Regression equations in uncoded units are:

$$Y_1 = 147.54 - 5.651\,X_a - 0.349\,X_b - 5.86\,X_c + 0.14592\,X_a^2 + 0.003169\,X_b^2 + 0.576\,X_c^2 - - 0.00098\,X_aX_b - 0.0240\,X_aX_c + 0.0090\,X_bX_c \quad (3)$$

$$Y_2 = 76.37 + 2.517\,X_a - 0.1477\,X_b - 9.00\,X_c - 0.08557\,X_a^2 - 0.000182\,X_b^2 - 0.360\,X_c^2 + + 0.00099\,X_aX_b + 0.3319\,X_aX_c + 0.06042\,X_bX_c \quad (4)$$

***Table 2**. Box-Behnken design with three factors and its measured and predicted values*

Trial	Voltage, V	Electrolysis time, min	Electrode distance, cm	X_a	X_b	X_c	COD removal, %		Turbidity removal, %	
							Experimental	Predicted	Experimental	Predicted
1	30	30	4	+1	−1	0	85.87	84.81	76.57	76.48
2	20	60	4	0	0	0	68.03	68.18	83.69	83.44
3	10	60	6	−1	0	+1	83.90	82.86	58.13	58.75
4	30	60	2	+1	0	−1	87.22	88.25	75.49	74.86
5	30	90	4	+1	+1	0	86.73	87.07	82.77	82.59
6	10	60	2	−1	0	−1	86.35	86.67	79.40	78.51
7	20	30	6	0	−1	+1	67.63	69.00	73.02	72.22
8	20	90	2	0	+1	−1	77.97	76.59	83.41	84.21
9	20	60	4	0	0	0	68.03	68.18	83.69	83.44
10	10	30	4	−1	−1	0	83.94	83.60	67.27	67.45
11	10	90	4	−1	+1	0	85.97	87.02	72.28	72.37
12	20	30	2	0	−1	−1	74.82	74.84	85.23	85.94
13	20	60	4	0	0	0	68.03	68.18	83.69	83.44
14	20	90	6	0	+1	+1	72.94	72.92	85.70	84.98
15	30	60	6	+1	0	+1	82.85	82.53	80.77	81.66

Coded factor levels

Factor	−1 (low)	0 (central)	+1 (high)
X_a: Voltage, V	10	20	30
X_b: Electrolysis time, min	30	60	90
X_c: Electrode distance, cm	2	4	6

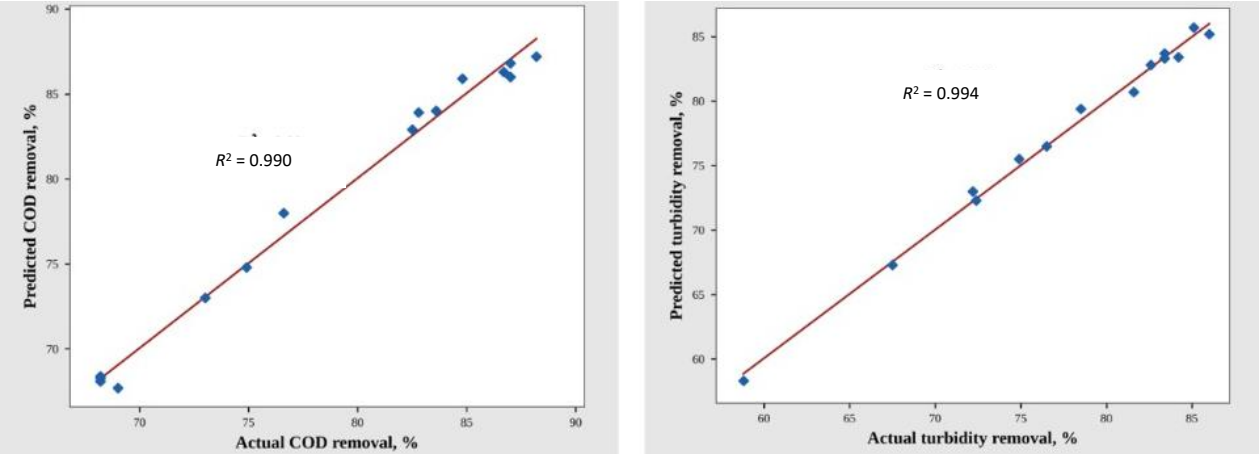


***Figure 2.** Predicted vs. actual (observed) results of (a) COD and (b) turbidity removal efficiencies*

The adequacy of the RSM is justified through analysis of variance (ANOVA). The ANOVA results for response parameters (COD and turbidity) using punched zinc and aluminium are shown in Table 3, which lists the Fisher distribution (*F*-value) and null hypothesis test (*P*-value) that are used to evaluate the significance of the regression model. The *F* value predicts the model quality by considering all independent variables simultaneously. The value of *F* should be large, and *P* should be small (<0.05). P and F are related, and P is used to estimate *F*. In the present case, the overall

value of *P* is 0.001, which is less than 0.05 and the value of *F* is 55.90, which is sufficiently high to show that the model is statistically significant. Furthermore, the ANOVA results indicate that the linear effect of voltage (X_a) on COD removal was not statistically significant (P = 0.529). However, electrolysis time (X_b), electrode distance (X_c), and several quadratic terms exhibited significant effects on the response. This suggests that COD removal was influenced more strongly by the combined and quadratic effects of the operating variables than by the linear effect of voltage alone.

Table 3. *Analysis of variance of the quadratic model for COD and turbidity removal*

Parameter	Model term	Sum of squares	Degrees of freedom	Mean square	*F*-value	*P*-value
COD	X_a	0.788	1	0.788	0.46	0.529
	X_b	16.103	1	16.103	9.35	0.028
	X_c	45.315	1	45.315	26.32	0.004
	X_aX_a	786.199	1	786.199	456.67	0.000
	X_aX_b	0.342	1	0.342	0.20	0.674
	X_aX_c	0.922	1	0.922	0.54	0.497
	X_bX_b	30.035	1	30.035	17.45	0.009
	X_bX_c	1.166	1	1.166	0.68	0.448
	X_cX_c	19.610	1	19.610	11.39	0.020
	Total error	8.608	5	1.722		
	Total (correlation)	874.738	14			
Turbidity	X_a	185.474	1	185.474	190.47	0.000
	X_b	60.886	1	60.886	62.53	0.001
	X_c	83.916	1	83.916	86.18	0.000
	X_aX_a	270.338	1	270.338	277.62	0.000
	X_aX_b	0.354	1	0.354	0.36	0.573
	X_aX_c	176.226	1	176.226	180.97	0.000
	X_bX_b	0.100	1	0.100	0.10	0.762
	X_bX_c	52.563	1	52.563	53.98	0.001
	X_cX_c	7.648	1	7.648	7.85	0.038
	Total error	4.869	5	0.974		
	Total (correlation)	838.018	14			

In the present study, the summary of the quadratic model is shown in Table 4. The value of R^2 for COD and turbidity removals is 0.990 and 0.994, respectively and the value of adjusted R^2 ($R^2_{adjusted}$) for COD and turbidity removals is 0.972 and 0.983, respectively. This high R^2 value (close to 1) is desirable and reasonably consistent with the adjusted R^2 (which is generally lower than R^2). A high regression coefficient (R^2) ensures a satisfactory adjustment of the quadratic model to the experimental data. Although the R^2 and adjusted R^2 values were very high for both responses, the predicted R^2 values were comparatively lower. This difference suggests that the developed models fit the experimental data well but showed slightly lower predictive capability for observations outside the experimental domain. Nevertheless, the predicted R^2 values remained sufficiently high to confirm the adequacy of the developed quadratic models.

Table 4. *ANOVA model summary*

Parameter	R^2	$R^2_{adjusted}$	$R^2_{predicted}$
COD	99.02	97.24	84.34
Turbidity	99.42	98.37	90.88

Figures 3 and 4 show the surface plots for the variations of COD and turbidity removal efficiencies according to voltage, electrolysis time and electrode distance. In each plot, two factors are varied, while the third is kept constant.

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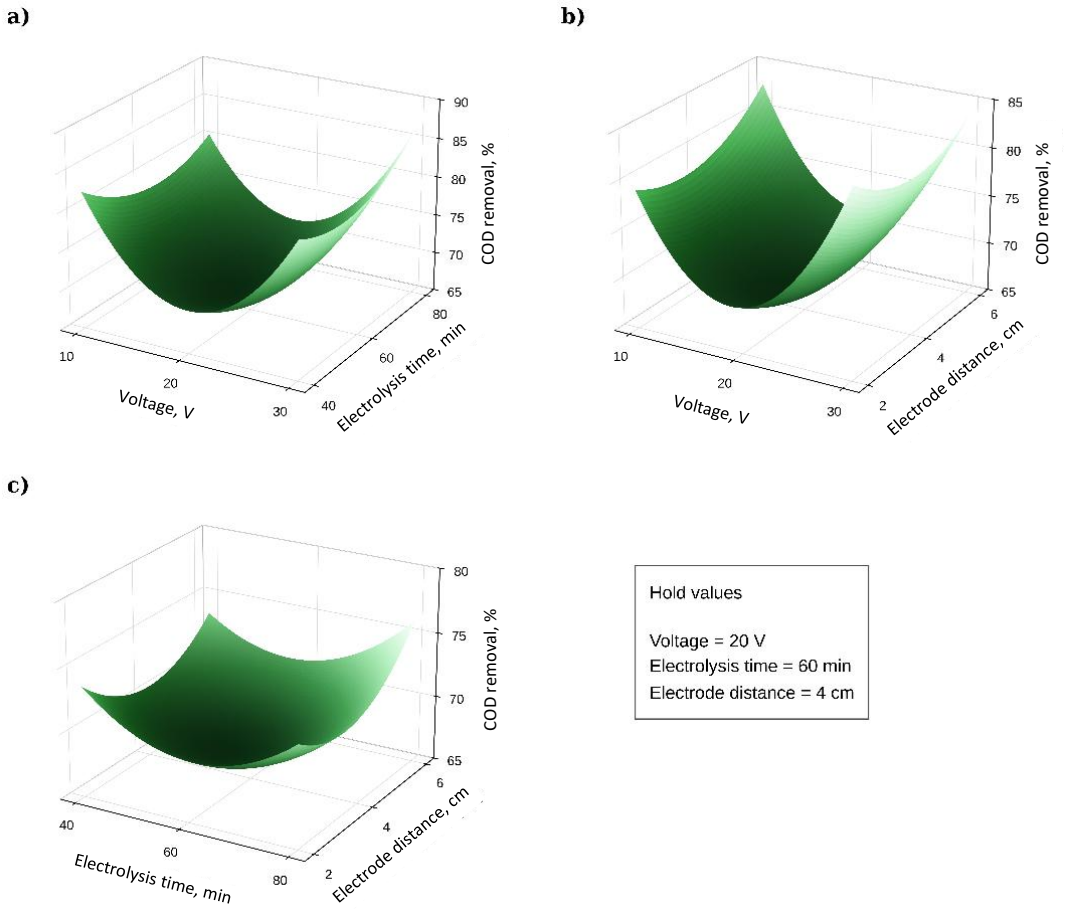


Figure 3. *Surface plots of COD removal efficiency*

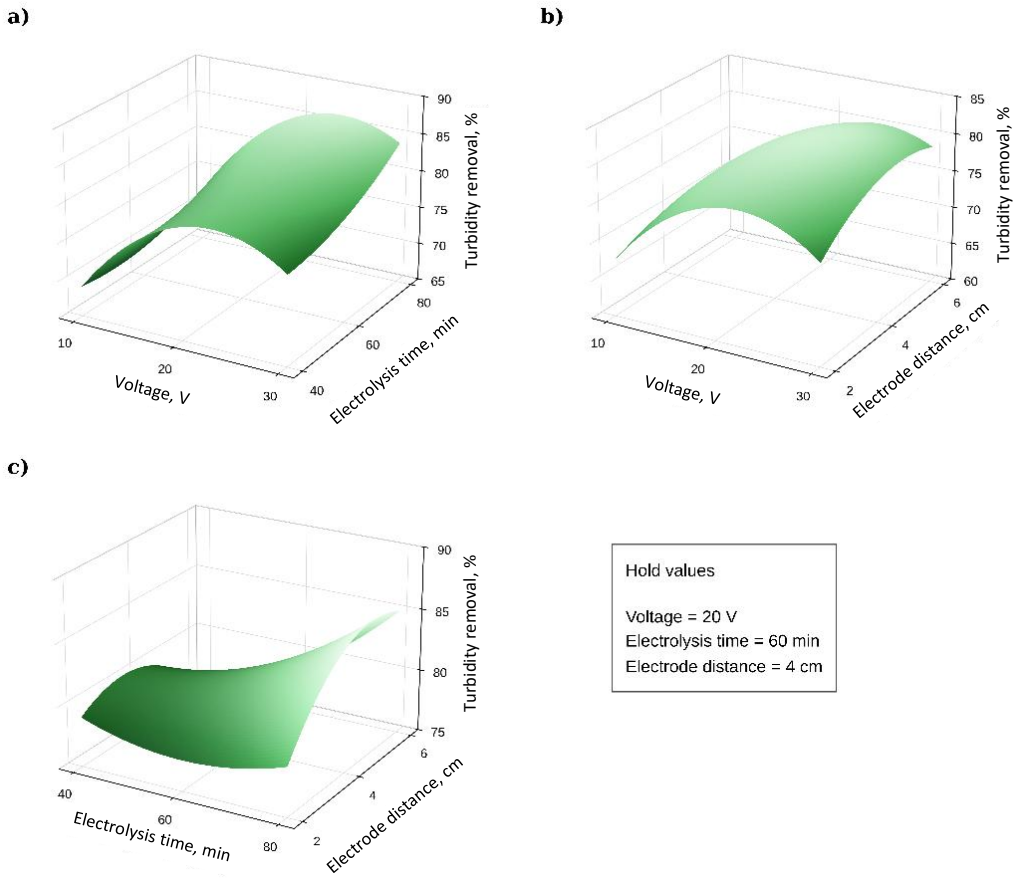


Figure 4. *Surface plots of turbidity removal efficiency*

The plots are derived from the quadratic models of Equations (3) and (4). These surface plots were generated using software and provide a three-dimensional view of the COD and turbidity removal surfaces across different combinations of independent variables. As shown in Figures 3 and 4, all surface plots have clear peaks. These peaks can be explained as the optimum conditions for maximum values of the responses attributed to the independent variables. Moving away from the peaks in the surface plots reduces removal efficiency.

Combined effect of independent variables

The combined effects of independent variables such as voltage (X_a), electrolysis time (X_b), and electrode distance (X_c) on COD and turbidity removal efficiency are discussed below using surface plots generated with Minitab 19 software.

Combined effect of voltage and electrode distance

The combined effect of voltage (X_a) and electrode distance (X_c) on COD and turbidity removal was analysed by varying X_a from 10 to 30 V and X_c from 2 to 6 cm and the outputs are presented in Table 2. Surface plots are shown in Figure 3(a-c) for COD and Figure 4(a-c) for turbidity. As shown in these plots, COD and turbidity removal efficiency increase with increasing voltage up to the optimum voltage. The increment in voltage resulted in a large amount of Zn^{2+} ions from anodic metal dissolution and more H_2 bubbles were formed at the cathode, which are beneficial for the separation or flotation process [36,37]. COD and turbidity removal efficiency decreases with increasing electrode distance at the voltage range of 10 to 30 V. This is due to the increasing voltage drop with increasing electrode distance [38]. In addition, Faraday's law states that the quantity of oxidized metal decreases with increasing distance between the electrodes [39].

Combined effect of voltage and electrolysis time

The interactive effect of voltage (X_a) and electrolysis time (X_b) on COD and turbidity removal was analysed by varying X_a from 10 to 30 V and X_b from 30 to 90 min and the outputs are presented in Table 2 and also surface plots are plotted as shown in Figure 3 for COD and Figure 4 for turbidity. As seen in these plots, the COD and turbidity removal efficiency increases with increasing electrolysis time. This is because, at minimum electrolysis time, there is insufficient time for both Zn^{2+} ion release by metal dissolution at the anode and H_2 bubble release at the cathode in the solution. Increasing electrolysis time increases metal dissolution and H2 bubble production at the anode and cathode, respectively, enhancing coagulation in solution. Hence, the removal efficiency also increased.

Optimization of process variables

The primary objective of this study was to determine the optimal operating conditions for maximizing COD and turbidity removal from soap and detergent wastewater using the electrocoagulation process. Response surface methodology (RSM) based on the Box-Behnken Design (BBD) was used to optimize the process variables: voltage (X_a), electrolysis time (X_b), and electrode distance (X_c). For COD removal, the optimum conditions were identified as 30 V, 60 min electrolysis time and 2 cm electrode distance, yielding a predicted removal efficiency of 88.25 % and an experimental removal efficiency of 87.22 %. For turbidity removal, the optimum conditions were 20 V, 90 min electrolysis time, and 6 cm electrode distance, resulting in a predicted removal efficiency of 84.98 % and an experimental removal efficiency of 85.70 %. The close agreement between the predicted and experimental values demonstrates the reliability and predictive capability of the

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developed RSM model. These results confirm that the BBD approach is an effective tool for optimizing electrocoagulation process parameters.

Conclusion

This study tested the removal efficiency of COD and turbidity from soap and detergent wastewater by EC process using punched zinc and aluminium electrodes. Analysis of the initial characteristics indicated that the concentrations of the wastewater quality parameters exceeded the standard effluent limits. Among those parameters, the initial concentrations of COD and turbidity were about 770 mg L^{-1} and 26 NTU, respectively. This indicates that the sample was unsuitable for discharge. Hence, the process was conducted with three independent variables, such as voltage (X_a: 10 to 30 V), electrolysis time (X_b: 30 to 90 min) and electrode distance (X_c: 2 to 6 cm) for the COD and turbidity removal. Design of experiments was conducted using BBD of response surface methodology, and the optimum conditions for each independent variable were determined to achieve maximum removal efficiency of COD and turbidity. The predicted COD and turbidity removal efficiencies under various operating conditions, and the comparison of actual results with the design of experiments, were analysed using a quadratic model.

The quadratic model obtained in this research shows a higher correlation between experimental and predicted values. The ANOVA showed that the coefficient of determination was very high (R2 for COD = 0.990 and R2 for turbidity = 0.994), indicating a satisfactory fit for the second-order regression model. Experimental results show that, under the best operating conditions, *i.e.* voltage (X_a) - 30 V, electrolysis time (X_b) - 60 min and electrode distance (X_c) - 2 cm for COD removal of 87.22 %, and voltage (X_a) - 20 V, electrolysis time (X_b) - 90 min and electrode distance (X_c) - 6 cm for turbidity removal of 85.70 %. Based on the results, it was recommended that the development of advanced materials and the use of different types of electrodes bring a new dimension to the electrochemical process. Hence, punched zinc and aluminium electrode in the EC process were successfully utilized for the treatment of soap and detergent wastewater.

***Authors' contributions**: Vijayakumar Hanumantha Naik contributed to conceptualization, original draft and methodology, and handled visualisation. Mahesh Kumar Chitrahalli Lingaraju carried out software analysis, provided resources, prepared the original draft, conducted review and editing, and handled visualisation. Ravindranath Chandrasekhar performed software analysis, formal analysis and data curation. Praveen Naik contributed to review and editing and handled visualisation. All authors have read and agreed to the published version of the manuscript.*

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