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Optimization of Electrocoagulation and integration of Microbial Fuel Cells for Brewery Wastewater Treatment

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Abstract:

Brewery industries hold a significant position in the economy of any country, but it also poses serious challenges including high water consumption and release of high strength wastewater into the environment. This study reports the sequential treatment of the brewery effluent by two electrochemical methods - Electrocoagulation (EC) and Microbial Fuel Cells (MFC). Initially, the brewery wastewater was pre-treated by EC process using aluminum electrodes. The primary factors involved in the process were optimized for maximizing COD removal and minimizing energy consumption using the Central Composite Design (CCD) of Response Surface Methodology. A mean COD removal of 44.2% and an energy consumption of 11.14 kWh/m^3 was achieved by the EC process at optimized conditions of pH - 6.6; current density - 13.2 mA/cm^2; electrolysis time - 39.3 minutes. The limited COD removal by EC process is attributed to the presence of most of the COD in the soluble form. EC process at the optimized conditions achieved a TSS removal of 88%, nitrate removal of 71.6%, phosphate removal of 93.17% and soluble COD removal of 11.5%. When the EC treated wastewater was used as substrate in a dual-chambered MFC with abiotic cathode, it attained a COD removal of 91.9% and highest power density of 11.81mW/m^2, thus releasing a treated effluent with discharge standards. The results show that the EC and MFC are thus complementary processes that can efficiently remove the suspended organic matter and soluble organic matter respectively when sequentially employed.

Keywords: Brewery wastewater, Electrocoagulation, Microbial Fuel Cells, Response Surface Methodology, COD removal

1. Introduction

Sustainability was defined as "meeting the needs of the present without compromising the ability of future generations to meet their own needs", according to the UN Brundtland Commission in 1987. The rapid industrialization and urbanization will not slacken in the current scenario for any developing country. Hence, it is highly important to incorporate sustainability into every aspect of the growth process for the sake of future generations. In this regard, currently, industrial wastewater treatment is an energy intensive process that lacks sustainability(Alazaiza et al. 2025).

Breweries are one of the rapid growing industries that generate large quantities of wastewater into the environment. Apart from the commercial large scale brewery industries, a large number of craft and microbreweries are evolving, driven by increasing consumer preference for unique flavors(Goldammer 2022).Reports project that, the global craft beer market will expand further at a Compound Annual Growth Rate (CAGR) of 8.74% from 2025-2033. With the advent of many such breweries all over the world, there is a huge demand for development of sustainable technologies that can treat the brewery wastewater effectively(Fillaudeau et al. 2006).

Electrochemical treatment of wastewater has become one of the established and efficient treatment technologies for any wastewater, among which combined technologies(Asaithambi et al. 2022) and Microbial Electrochemical Technologies (MET)(Butti et al. 2016) has been gaining importance in the recent days. Various processes including electrocoagulation, electro-oxidation and electro-fenton process had been employed for the treatment of brewery industry wastewater(Eyvaz 2016). Similarly, given its organic nature and high biodegradability index, MET have also been extensively studied for the brewery effluent treatment(Niju and Priyadharshini 2023).

Electrocoagulation (EC) is an electrochemical process that is used for the treatment of wastewater and industrial effluents. It involves the use of an electrical current to coagulate and precipitate suspended and dissolved particles in the water, such as metals, organic matter, and other contaminants(Tahreen et al. 2020). During this process, metal ions are generated at the anode, which undergo hydrolysis to form metal hydroxides. These metal hydroxides acts as coagulants, which can destabilize and react with the contaminants in the wastewater to form insoluble precipitates. The precipitates can then be removed by settling, flotation, and filtration. EC is a versatile and effective method for the treatment of wastewater and has several advantages over traditional chemical coagulation methods including less sludge formation, easier to dewater, does not increase Total Dissolved Solids (TDS) and more sustainable. It is a simple and cost-effective process that can be used to treat a wide range of contaminants, including heavy metals, oils, and fats. Additionally, the process can be easily scaled up or automated for continuous operation(Abdulhadi et al. 2021). EC is thus an attractive option for the removal of suspended solids and colour from the brewery wastewater. It also assists in the nutrient removal process of wastewater. However, EC is not very effective in removing soluble organic matter within the wastewater(Papadopoulos et al. 2020). Brewery wastewater indeed contains most of its organic content in the soluble form and hence requires a post treatment technique, that can complement the EC process to remove the sCOD (Soluble Chemical Oxygen Demand) effectively.

Microbial Fuel Cell (MFC) technology is one of the most commonly used METs, that has the scope of complementing EC as it can generate bioelectricity and efficiently remove the sCOD of the wastewater(Safwat 2019). MFC employs a specific class of microbes called exoelectrogens as catalysts to convert the biochemical energy within the wastewater into electrical energy(Kothari et al. 2024). Fundamentally, a MFC system is composed of an anodic chamber and a cathodic

chamber separated by a proton exchange membrane (PEM). The basic principle behind MFC technology is the ability of exoelectrogenic bacteria to transfer the electrons outside their cells, which is exploited by using a solid electrode within an anaerobic anode chamber(Logan 2009). Any organic substrate such as wastewater is provided within this anodic chamber(Sharma and Vijay 2025). The organic matter of the wastewater is oxidized by the microbes and produces electrons, protons and CO_2. The electrons are then transferred to the anode, which flow through the external circuit to the cathode. The protons from the oxidation process gets transferred to the cathode chamber through the proton exchange membrane between the two chambers. In the cathode chamber, the electrons and protons undergo reduction reaction, in the presence of a terminal electron acceptor (generally O_2). Many physico-chemical, biological and operating parameters influence the performance of MFCs(Venkata Mohan et al. 2014). Even though, MFC is a sustainable wastewater treatment option, it has significant limitations including scaling up and reliable power generation. EC, on the other hand, enables rapid removal of suspended solids, colloids and toxic compounds but has a high energy demand. Hence integrating such MFCs with conventional treatment technologies like EC will be beneficial, as EC can improve wastewater biodegradability and operational stability for MFCs, while electricity generated in MFCs can partially offset the energy requirement of EC, resulting in a more efficient and sustainable hybrid treatment system(Li et al. 2013).

The study of integration of electrocoagulation with a bioelectrochemical system for brewery wastewater treatment is limited in the literature. This study explores the performance of a sequential treatment system with electrocoagulation as a pre-treatment step, followed by Microbial Fuel Cells for the treatment of a high strength brewery wastewater. Initially, a Central Composite Design (CCD) of Response Surface Methodology (RSM) was utilized for optimization of primary

process conditions of EC including pH, current density and electrolysis time for maximizing the COD removal efficiency and minimizing the energy consumption. Then the pre treated brewery wastewater was fed as substrate to an acclimatized MFC, which resulted in further COD removal and bioelectricity generation. This sequential treatment of brewery effluent was evaluated in terms of treatment efficiency and energy efficiency.

2. Methodology

2.1. Sample collection and characterization

The brewery effluent used in this study was collected from a craft brewery at Bengaluru, India. The sample was collected in sterile plastic containers and transported to the laboratory, following standard protocols. Then the sample was stored in laboratory refrigerator at 4 °C until further use. All samples were used in the experiments after allowing for the primary settling process. The pH, conductivity and TDS of the wastewater was measured using a multiparameter analyzer (Lovibond Sensodirect 150). Other characteristics of the wastewater were analyzed following the APHA standard methods. All the chemicals used in the experiments were of analytical grade. Table 1 represents the physico-chemical characteristics of the brewery wastewater. Every experiment was carried out in triplicates and the average values along with the standard deviation were represented in the table.

Table 1 Physico-chemical Characteristics of collected Brewery Wastewater

Characteristics	**Units**	**Value**
Colour		Brownish
pH		3.81 ± 0.35

COD	mg/L	14920 ± 450
sCOD (soluble COD)	mg/L	8840 ± 280
Conductivity	mS/cm	0.915 ± 0.05
TDS (Total Dissolved Solids)	ppm	585 ± 15
TSS (Total Suspended Solids)	mg/L	2320 ± 50
Nitrate	mg/L	17.75 ± 0.7
Phosphate	mg/L	51.65 ± 2.5

2.2. Experimental set up and procedure of Electrocoagulation

The EC process was carried out in a simple set-up (Figure 1) comprising of a 1L beaker with 500 mL working volume, placed on a magnetic stirrer. Two aluminium electrodes of dimension 20 cm x 7 cm x 0.2 cm, were vertically immersed in the wastewater solution, 1 cm above the bottom of the beaker with an inter electrode distance of 1.5 cm. The total effective anode surface area immersed in the 500 mL working electrolyte was 70 cm^2. The electrodes were pre-treated by immersing them in distilled water overnight and soaking in 1M HCl for 15 minutes before each EC run to prevent passivation of electrodes(Ingelsson et al. 2020). The wastewater was stirred at a constant stirring speed of 300 rpm throughout the process. Supporting electrolytes were not added in the process to avoid the secondary pollutant risk. A variable DC power supply (0-50V; 0-5A) was used to pass electrical current through the electrodes.A multimeter was used to monitor the voltage at any particular current density. During each EC run, the pH of the raw wastewater was adjusted using 0.1 N sodium hydroxide solution according to the operating conditions of the run. After each EC run, a uniform settling time of 60 minutes is allowed for all runs(Zodi et al. 2009). The characteristics including COD, nitrate, phosphate and TSS of the wastewater

wereexamined before and after each experimental run. The scum and the sludge collected after the EC process was analyzed using FTIR and XRD.

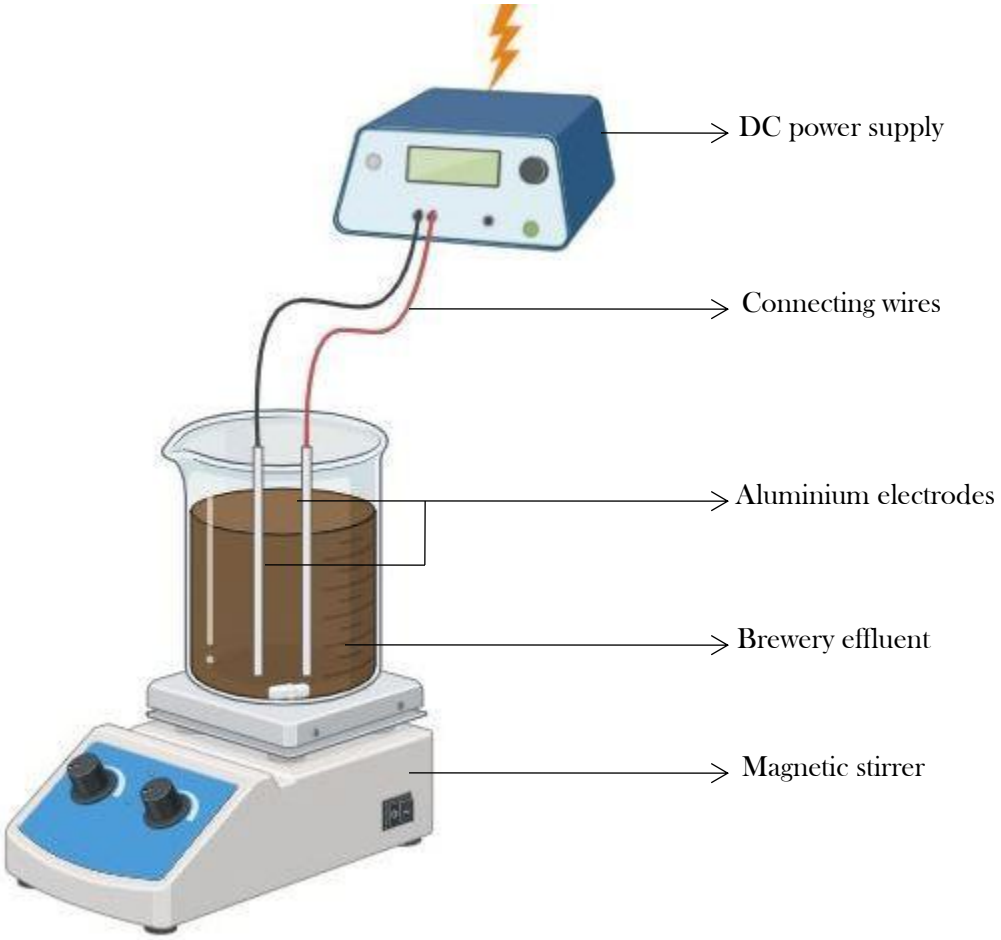


Fig1. Schematic representation of EC set up

2.3. Statistical Experimental Design for Optimization of Electrocoagulation process

CCD of RSM is one of the preferred experimental designs for any optimization study as it usesaxial points and center points apart from the factorial points, which explores the relationship between the dependent and independent variables more effectively. Here, the CCD experimental design was employed for optimizing the three variable factors- pH (A), current density (B) and

electrolysis time (C) of EC process in order to maximize the COD removal efficiency (R1) and minimize the energy consumption (R2) for the treatment of brewery wastewater, using the Design Expert 11 software (STAT-EASE Inc., Minneapolis, USA). The variables were coded in five levels as -α, -1, 0, +1 and +α according to the ranges provided for each variable (Table 2). The CCD experimental design matrix consisted of 17 experiments with 3 centre point repetitions as shown in the Table 2. The influence of the independent variables was examined using multiple regression analysis of the experimental data. Model suitability was evaluated through analysis of variance (ANOVA), while the quality of the regression models was determined using the coefficient of determination (R^2), adjusted R^2, and predicted R^2, confirming that the model effectively represented the actual relationship among the variables studied. The statistical significance of each parameter was assessed based on the p-values and F-values. Response surface plots were generated from the fitted model's equation derived from the regression analysis.

Table 2 Range and levels of independent variables for EC process

Variables	Symbols	Units	Variable levels				
			-α	**-1**	**0**	**+1**	**+α**
pH	A		2.64	4	6	8	9.36
Current Density	B	mA/cm^2	6.59	10	15	20	23.41
Electrolysis time	C	Minutes	13.07	25	42.5	60	71.93

2.4. Experimental set up and procedure of Microbial Fuel Cell

The brewery effluent treated at optimized conditions of EC, was next used as the substrate in a dual-chambered MFC operated in batch mode. The dual-chambered MFC was constructed using

plexiglass material, containing anode and cathode chambers with a working volume of 300 mL each. Graphite electrodes with surface area of 50 cm^2, were used as electrodes after soaking them in deionised water for 24 hours. A 15 cm^2Nafion membrane was used as proton exchange membrane between the two chambers. The schematic representation of the dual-chambered MFC used in this study was represented in the Figure 2. The EC treated brewery effluent was buffered using 0.1M phosphate buffer and used as anolyte for the MFC. It was continuously stirred to maintain uniform substrate concentration under anaerobic conditions. The catholyte consisted of 50 mM potassium ferricyanide in 50 mM phosphate buffer. The distance between electrodes was maintained at 3 cm apart. Copper wires were used for electrical connections. The indigenous microbes present in the wastewater was only used for acclimation and no external inoculum was added. MFC was run with raw effluent (without any treatment) for a few cycles under closed circuit condition with a resistance of 1000Ω, for the system to get acclimated with exoelecrogens and thus form a stable anodic biofilm(Elakkiya and Niju 2021). Each run lasted till the OCV dropped below 200 mV(Elakkiya and Matheswaran 2013). The collected effluent samples were filtered through a 0.25µm membrane filter and used for further chemical analyses.

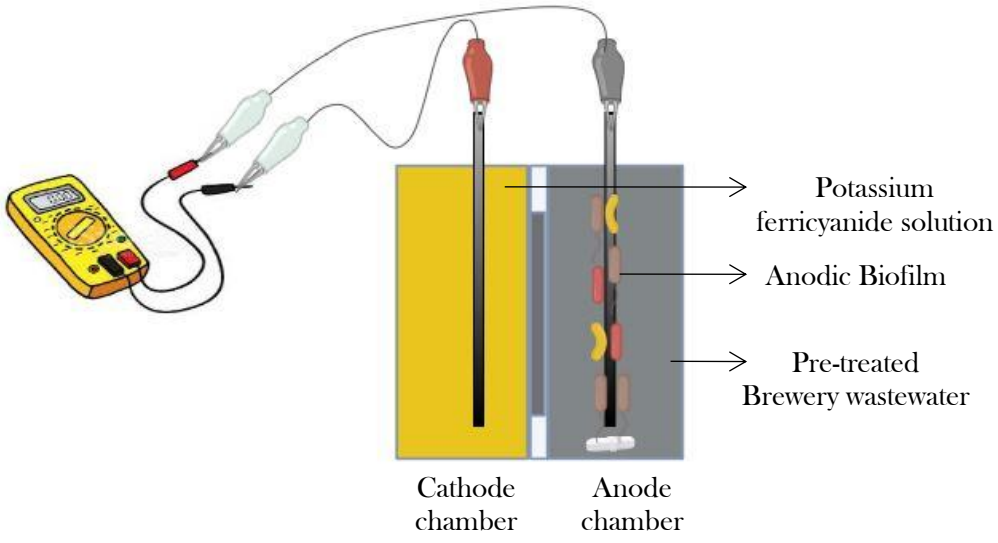


Fig2. Schematic representation of MFC set up

2.5. Analysis and Calculations

The COD of the samples were determined using closed reflux titrimetric method (5220C) with sample digestion performed in a HACH DRB 200 reactor. The COD removal efficiency was calculated as follows.

$$\text{COD removal \%} = (\text{COD}_{\text{initial}} - \text{COD}_{\text{final}})/\text{COD}_{\text{initial}} * 100\ \% \qquad \textbf{Equation 1}$$

where $\text{COD}_{\text{initial}}$ and $\text{COD}_{\text{final}}$ represent the initial and final concentrations respectively.

Power consumption during electrocoagulation process was calculated based on applied voltage, current, and electrolysis time using the following formula:

$$\text{Power consumption (kWh/m}^3) = V * I * t / \text{Volume of reactor} \qquad \textbf{Equation 2}$$

where V is the applied voltage (V), I is the current (A), t is the electrolysis time (hour) and the reactor volume is in m^3.

The nitrate and phosphate analysis was carried out by 4500-NO_3^- C (Second derivative ultraviolet spectrophotometric method) and 4500-P C (Vandomolybdophosphoric acid colorimetric method) respectively. TSS was determined using 2540D method and sCOD was determined by initially separating the solid fraction of the sample by filtration followed by 5220C method.

The current generated in such a lab scale MFC is relatively low and hence could not be measured directly. Therefore, voltage (V) was recorded using a multimeter and the current (I) and power (P) were subsequently calculated using Ohm's law, expressed as

$$I = V/R \qquad \textbf{Equation 3}$$

$$P = V \times I \qquad \textbf{Equation 4}$$

Current density (mA/m^2) and power density (mW/m^2) was determined by normalizing the calculated current and power to the surface area of the electrode. Polarization data were obtained

by varying the external resistance from 33kΩ to 150Ω. The voltage corresponding to each resistance was recorded after 20 minutes of stabilization. Subsequent resistors were applied only after the OCV (Open Circuit Voltage) had returned to its peak value. The polarization and power curves were constructed by plotting voltage against current density and power density against current density respectively(Pandit et al. 2017).

3. Results and discussion

3.1. Optimization of Electrocoagulation process using RSM

3.1.1. Statistical Analysis and Model Validation

The responses - COD removal and energy consumption, for the 17 runs, as given by the CCD of RSM was found through the experiments and it was all listed in Table 3. These actual (observed) responses are tabulated alongside the predicted responses in the same table. RSM was used to analyze the effect of each of the three variables (pH, current density and electrolysis time) on each of the response (COD removal and energy consumption) statistically. Figure 3 (a) and (b) depicts the predicted versus actual COD removal and predicted versus actual energy consumption data respectively. They both show that the data points are close to the diagonal line, demonstrating the high correlation. The R^2co-efficient value determines how well the chosen model explains the variability in the response data(Bezerra et al. 2008). A high R^2 value of 0.9972 and 0.9993 for COD removal and energy consumption respectively, thus asserts the adequacy of the quadratic model for both cases. The ANOVA results in Table 4 and Table 5, indicate that the selected quadratic model is statistically significant at the 95% confidence interval, as shown by its low p-value (<0.0001 for both responses) and high F-value (272.37 for COD removal and 1141.91 for

energy consumption). Thus, the quadratic model can be reliably employed to interpret and navigate the design space.

Table 3 Central Composite Design Matrix for EC process

Run	pH	Current Density (mA/cm^2)	Electrolysis time (minutes)	COD Removal (%)		Energy Consumption (kWh/m^3)	
				Actual response	Predicted response	Actual response	Predicted response
1	6	23.409	42.5	17.92	18.65	54.42	53.93
2	4	10	25	12.22	11.81	9.908	9.45
3	6	15	13.0686	15.38	16.23	4.51	3.99
4	4	10	60	15	14.33	21.4	20.84
5	4	20	60	20	19.69	68.88	68.39
6	6	6.59104	42.5	20.2	21.00	5.963	6.50
7	6	15	42.5	47.22	46.89	17.165	17.15
8	8	10	25	25.18	24.41	6.5	6.95
9	8	10	60	20	19.63	8.1	7.73
10	4	20	25	8.92	8.21	27.833	28.16
11	6	15	71.9314	21.18	21.86	37.91	38.48
12	8	20	60	21.1	20.43	44.996	45.42
13	6	15	42.5	46.25	46.89	17.35	17.15
14	9.36359	15	42.5	21.63	22.43	13.1	12.47
15	2.63641	15	42.5	10.5	11.22	33.2	33.88

16	6	15	42.5	47.45	46.89	16.958	17.15
17	8	20	25	16.66	16.25	15.28	15.80

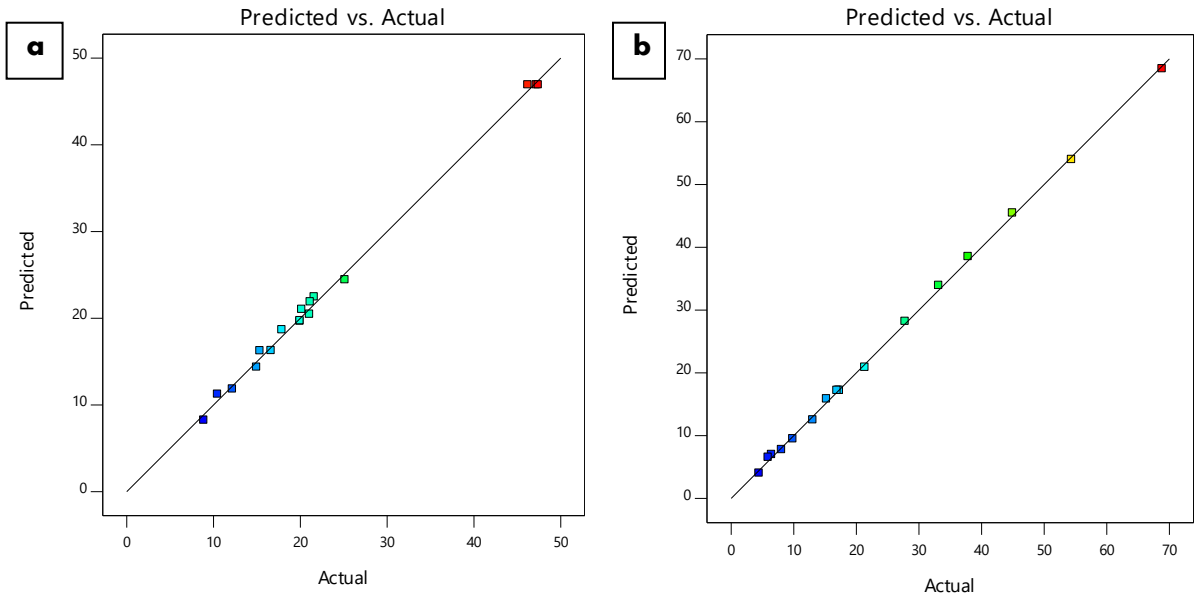


Fig3. Predicted versus actual plot of a) COD removal b) energy consumption

Table 4 ANOVA table for COD removal of EC process

Source	**Sum of Squares**	**Degrees of freedom**	**Mean Square**	**F-value**	**p-value**	
Model	2415.21	9	268.36	272.37	< 0.0001	Significant
A-pH	151.71	1	151.71	153.98	< 0.0001	
B-Current density	6.68	1	6.68	6.78	0.0352	
C-Electrolysis time	38.31	1	38.31	38.89	0.0004	

AB	10.40	1	10.40	10.55	0.0141	
AC	26.64	1	26.64	27.04	0.0013	
BC	40.14	1	40.14	40.74	0.0004	
A^2	1273.24	1	1273.24	1292.27	< 0.0001	
B^2	1032.15	1	1032.15	1047.58	< 0.0001	
C^2	1092.50	1	1092.50	1108.83	< 0.0001	
Residual	6.90	7	0.9853			
Lack of Fit	6.09	5	1.22	3.00	0.2686	not significant
Pure Error	0.8113	2	0.4056			
Cor Total	2422.10	16				

R^2 = 0.9972; adjusted R^2 = 0.9935; predicted R^2 = 0.9801

Table 5 ANOVA table for energy consumption of EC process

Source	**Sum of Squares**	**Degrees of freedom**	**Mean Square**	**F-value**	**p-value**	
Model	5473.29	9	608.14	1141.91	< 0.0001	Significant
A-pH	553.58	1	553.58	1039.45	< 0.0001	
B-Current density	2715.51	1	2715.51	5098.92	< 0.0001	
C-Electrolysis time	1435.73	1	1435.73	2695.86	< 0.0001	

AB	48.65	1	48.65	91.36	< 0.0001	
AC	56.30	1	56.30	105.72	< 0.0001	
BC	415.74	1	415.74	780.64	< 0.0001	
A^2	51.08	1	51.08	95.91	< 0.0001	
B^2	240.43	1	240.43	451.46	< 0.0001	
C^2	23.46	1	23.46	44.06	0.0003	
Residual	3.73	7	0.5326			
Lack of Fit	3.65	5	0.7302	18.99	0.0508	not significant
Pure Error	0.0769	2	0.0385			
Cor Total	5477.01	16				

$R^2 = 0.9993$; adjusted $R^2 = 0.9984$; predicted $R^2 = 0.9948$

The second-order polynomial equation is given in equation 5 and 6 and they both represent the mathematical relationship between the responses and the independent variables.

COD removal (%) = +46.89+3.33A - 0.6996B + 1.67C - 1.14AB - 1.83AC + 2.24BC -10.63A^2 - 9.57B^2 - 9.84C^2 **Equation 5**

Power consumption (kWh/m^3)=+17.15 - 6.37A+ 14.10B + 10.25C - 2.47AB - 2.65 AC + 7.21 BC + 2.13A^2 + 4.62B^2 + 1.44C^2 **Equation 6**

The three selected independent variables (A, B and C) are found to have significant impact on the responses COD removal and energy consumption, as shown by the lower p values on both the ANOVA tables. The 3D surface plots (figure 5 and 6) are generated using the developed

mathematical models (Equation 5 & 6), which help in illustrating the individual and interactive effects of the different variables on the EC process responses.

3.1.2. Effect of interaction between variables on COD removal

As can be observed from the ANOVA table (Table 4), factor B(current density) have the least F value when compared to A(pH) and C(electrolysis time). Current density, while important, only controls the rate of coagulant generation. Thus, its relative influence on pollutant removal is weaker in the tested conditions. In contrast pH plays a critical role in determining the speciation and stability of coagulant species(Bayar et al. 2014) and hence the highest F value of 153.98. While electrolysis time dictates the extent of pollutant-floc interaction, once sufficient coagulants are available, extending the time doesn't drastically improve the performance(Morales-Figueroa et al. 2022). Therefore, it comparatively has a weaker influence than pH. The markedly higher contribution of pH reflects its influence on aluminium hydrolysis reactions, floc formation and surface charge interactions, which directly influence the treatment efficiency of the EC process.

When the variable A(pH) was varied from 4 to 8 and variable B(current density) was varied from 10 mA/cm^2 to 20 mA/cm^2, with a constant electrolysis time of 42.5 minutes, a maximum CODremoval of 47.22% was achieved at pH 6 and current density 15 mA/cm^2. Thus as shown in the figure4(a), pH and current density, both have the same effect on COD removal in the selected range. COD removal increases as pH increases, but only till a certain value, after which the removal starts decreasing. This is mainly attributed to the high number of coagulant species formation in the acidic and neutral pH, rather than alkaline pH(İrdemez et al. 2006). The same effect is observed for current density also. This can be associated with increased heat and coagulant disintegration at a high current density. But together, their interaction on COD removal has least

significance as shown by lowest F value, as compared to the other two pairs of factors. This suggests that, while both factors individually affect pollutant removal, their interaction does not produce a substantial synergistic effect.

When the variable A was varied from 4 to 8 and variable C was varied from 25 minutes to 60 minutes, with a constant current density of 15 mA/cm^2, a maximum COD removal of 47.45% was achieved at pH 6 and electrolysis time of 42.5 minutes, as shown in the figure 4(b). Extending the time beyond the optimum level causes decrease in COD removal because excess coagulant ions may cause restabilization of colloids and increase the solution turbidity. Electrode passivation, a common phenomenon observed at high current density and excess electrolysis time may also be the reason for reduced COD removal(Bajpai et al. 2025). But together, pH and electrolysis time, have a stronger interaction than AB, as AC have a F value of 27.04.

The variables B and C together, exhibited the strongest influence on COD removal, highlighting their combined effect in determining the effective coagulant dosage available in the EC system. Because current density controls the rate of coagulant generation and electrolysis time controls the duration of action of coagulant leading to the floc and sludge formation(Zhang et al. 2021). At a low current density of 10 mA/cm^2, even a long electrolysis time of 60 minutes resulted in a COD removal of around 30% and at a high current density of 20 mA/cm^2 with short electrolysis time of 25 minutes resulted in a COD removal of 25% as shown in the figure 4(c). Only the right balance between them gives highest COD removal and hence the highest significant interaction with a F value of 40.74.

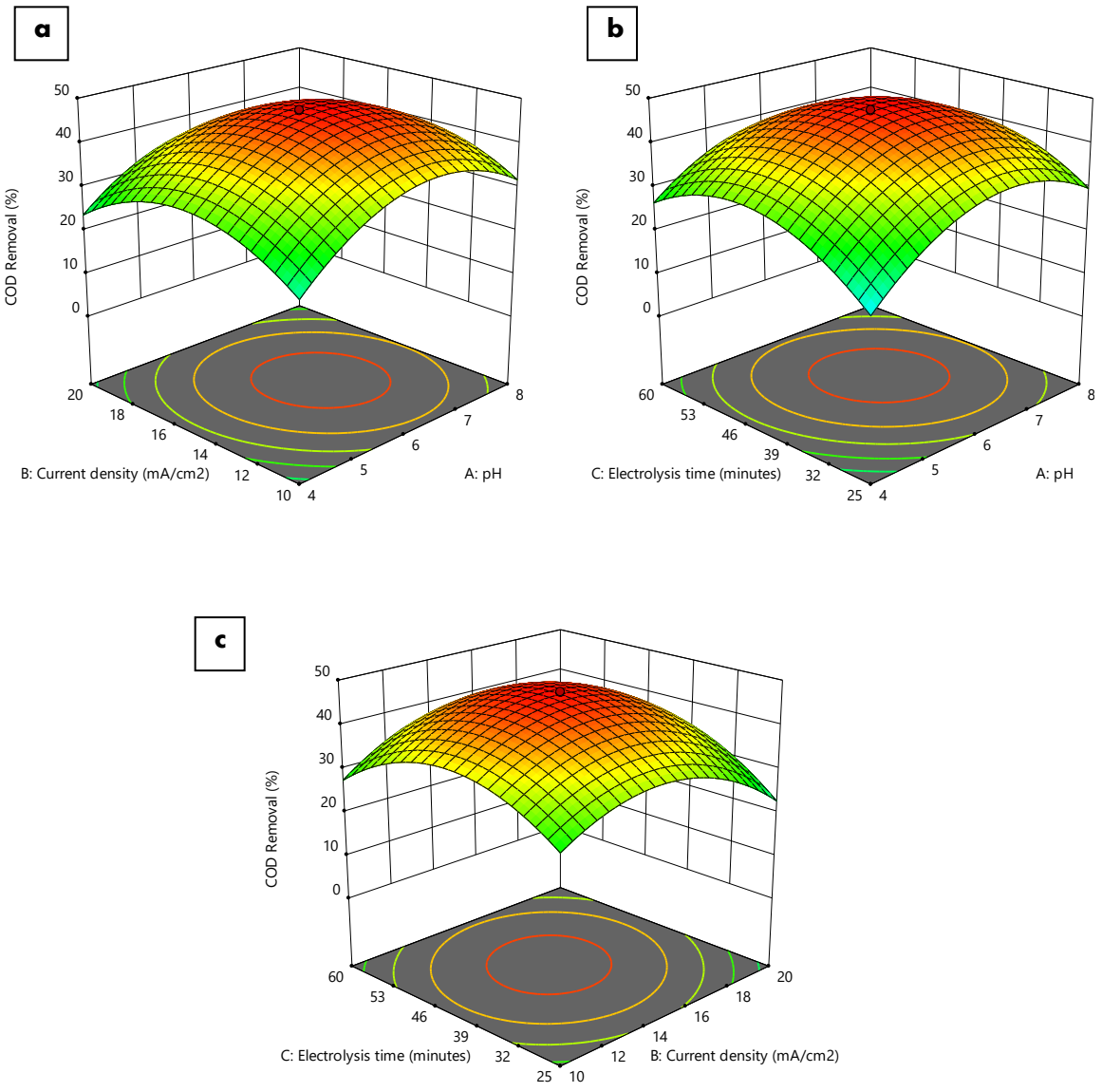


Fig4. 3D response surface plots showing interaction between parameters for COD removal a) pH versus current density (at electrolysis time=42.5 mins) b) pH versus electrolysis time (at current density=15mA/cm^2) c) current density versus electrolysis time (at pH=6)

3.1.3. Effect of interaction between variables on Energy Consumption

The statistical analysis reveals that energy consumption in EC process is predominantly governed by electrical operating parameters rather than solution chemistry. The ANOVA table (Table 5)

shows that all three variables have high F-value and very low p-values, thus demonstrating their significant effect on the energy consumed during the EC process. Among the three variables, factor B(current density) has the highest F-value of 5098.92 and thus the one which has the highest influence on energy consumption, followed by electrolysis time with a F-value of 1435.73 and then pH with a F-value of 1039.45. Current density and electrolysis time directly influences the energy consumption, reflecting their direct proportionality as shown in the Equation 2. In contrast,pH is exerting a weaker effect on energy consumption as it has an indirect influence by affecting the conductivity of the electrolysis solution(Ebba et al. 2022). A highly conductive solution consumes less energy compared to a less conductive solution, for the same applied voltage. A high energy consumption of 54.42 kWh/m^3 was obtained for the run 1 with operating conditions of pH 6, current density of 23.409 mA/cm^2 and electrolysis time of 42.5 minutes. This implies that, even with optimum values of pH and electrolysis time, a high current density causes a high energy consumption. It should be noted that unlike COD removal, a low energy consumption is preferred out of the EC process.

As shown in the figure 5(a) and (b), pH doesn't have much influence on the energy consumption. The EC processes conducted in acidic pH consumed more power than the alkaline pH. But current density and electrolysis time, both were found to have a positive linear relationship on the energy consumption, as shown in the figure 5(c)(Tanyol et al. 2018). Among the interacting variables (AB, AC and BC), BC exhibited the strongest significance with a F-value of 780.64. This is mainly due to the synergistic effect of the factors current density and electrolysis time on energy consumption. It is followed by AC and AB with a F-value of 105.72 and 91.36 respectively. This signifies the role of pH in prolonged operation of EC process. Although pH can influence

conductivity, its interaction with current density is weaker than time, so their combined effect on energy consumption is relatively small.

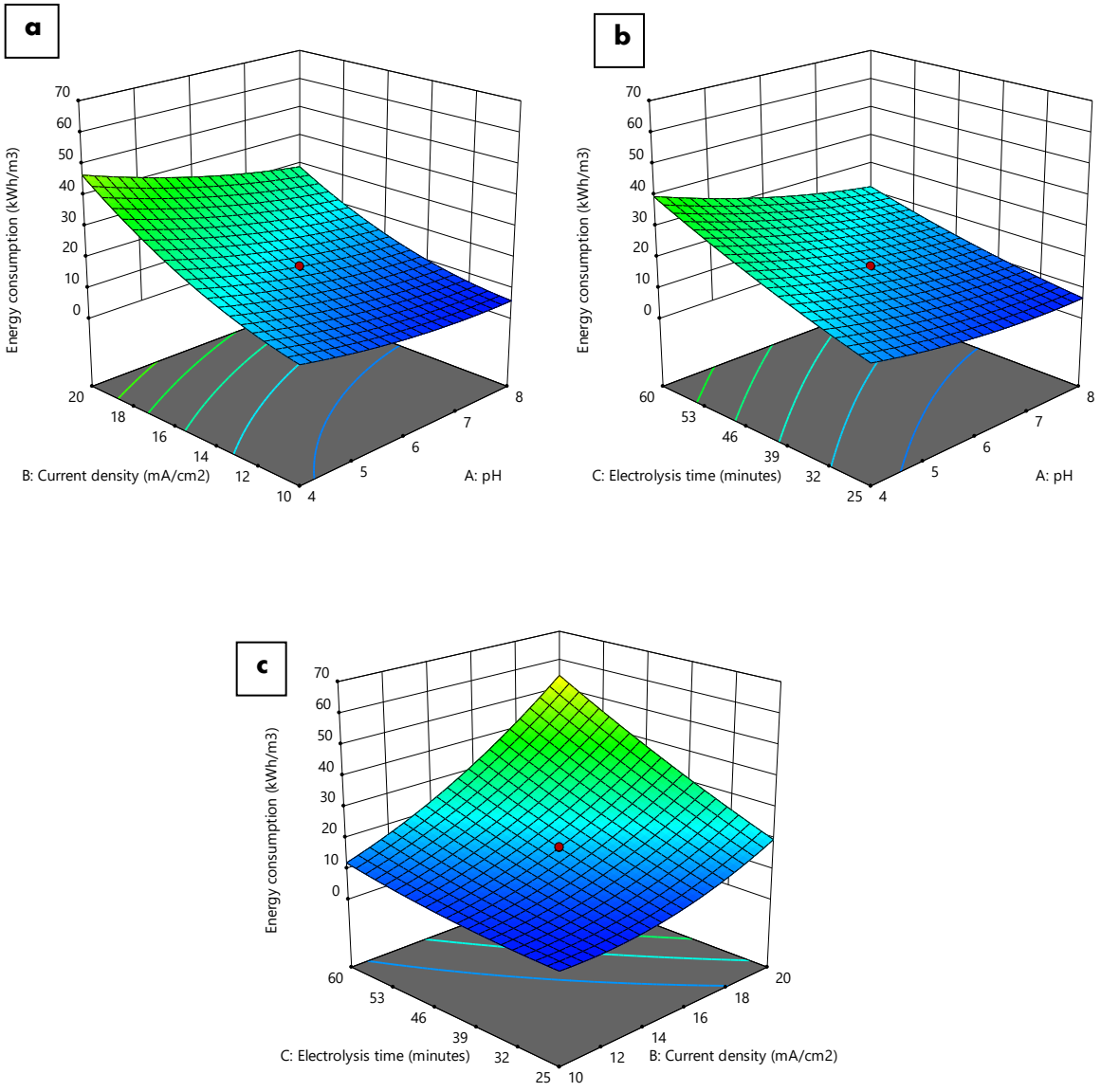


Fig5. 3D response surface plots showing interaction between parameters for energy consumption a) pH versus current density (at electrolysis time=42.5 mins) b) pH versus electrolysis time (at current density=15mA/cm^2) c) current density versus electrolysis time (at pH=6)

3.1.3. Optimization and validation

Using the CCD of RSM, the optimal conditions for maximum COD removal and minimum energy consumption were identified through the numerical optimization tool in the Design Expert software. All the three input variables were designated to be in range, whereas the response 1 COD removal and response 2 energy consumption were maximized and minimized respectively(Karabacakoğlu and Tezakıl 2023). As given in the figure 6, the optimum conditions suggested by the software at a desirability of 93.4% are as follows: pH - 6.6; current density - 13.2 mA/cm^2; electrolysis time - 39.3 minutes, with the expected COD removal of 45.67% COD removal and energy consumption of 10.01kWh/m^3. Experiments were conducted at these optimized conditions to validate the prediction of the software. A mean COD removal of 44.2% and energy consumption of 11.14 kWh/m^3 was attained, which is close to the predicted result.

At the optimized condition, the treatment efficiency of the system was also assessed for TSS, nitrate and phosphate removal. A TSS removal of 88% was achieved. Moreover, the EC process has attained a 71.6% of nitrate removal and 93.17% of phosphate removal, resulting in a nitrate concentration of 5.04 mg/L and a phosphate concentration of 3.53 mg/L. This is well below the standard discharge limits of nitrate (50 mg/L) and phosphate (5mg/L) set by CPCB (Central Pollution Control Board), India. Thus, the EC process proves to be efficient in removing the nutrients from the brewery effluent at these optimized conditions(Swain et al. 2020).

While EC process at the optimized conditions has achieved a pCOD (particulate COD) removal of 91.7%, sCOD removal of only 11.5% was attained. This demonstrates the fact that EC with aluminum electrodes is more effective in removing the particulate COD but not the soluble COD(Bani-Melhem et al. 2017). This aligns with the primary mechanism of EC which comprises

of charge neutralization, adsorption and flocculation. The dissolved organic compounds are too small to be trapped or destabilized by the coagulant flocs. Consequently, EC primarily acts as an effective pretreatment step, reducing the particulate load and thus enhancing the subsequent biological treatment of the remaining sCOD of the wastewater.

EC process thus facilitates the removal of suspended solids, colloidal particles, nutrients and a significant portion of particulate COD through in situ generation of coagulants. However, under the optimized conditions, the removal of soluble COD remains limited. A fraction predominantly composed of dissolved and biodegradable organic compounds remains behind EC pretreatment. This residual soluble COD fraction is well suited for subsequent biological treatment. MFC can efficiently oxidize these low molecular weight organics while simultaneously generating bioelectricity. Thus, the sequential EC-MFC configuration will enable complementary removal mechanisms, with EC acting as a physico-chemical pretreatment step and MFC providing biological polishing of the remaining soluble organics. Furthermore, EC is preferred over other conventional pretreatment methods due to its operational simplicity, rapid treatment ability, reduced chemical requirement, reduced sludge production and effectiveness in handling high strength industrial wastewaters.

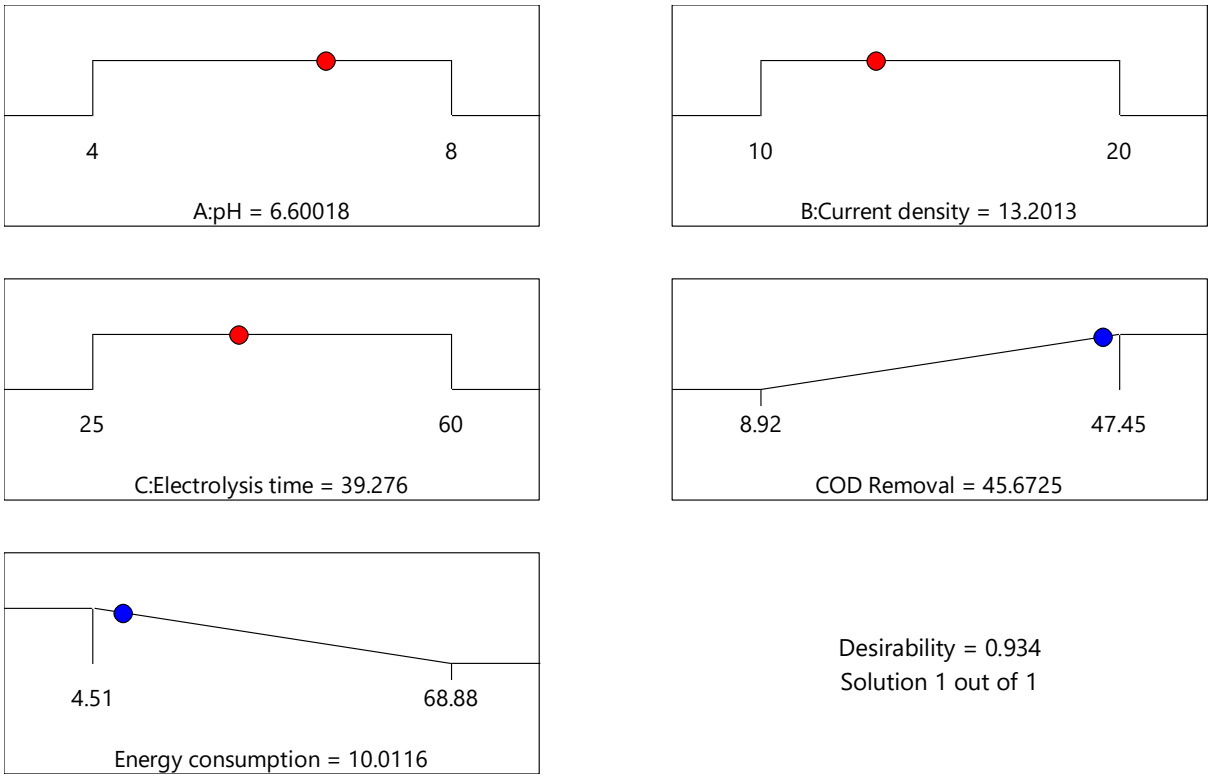
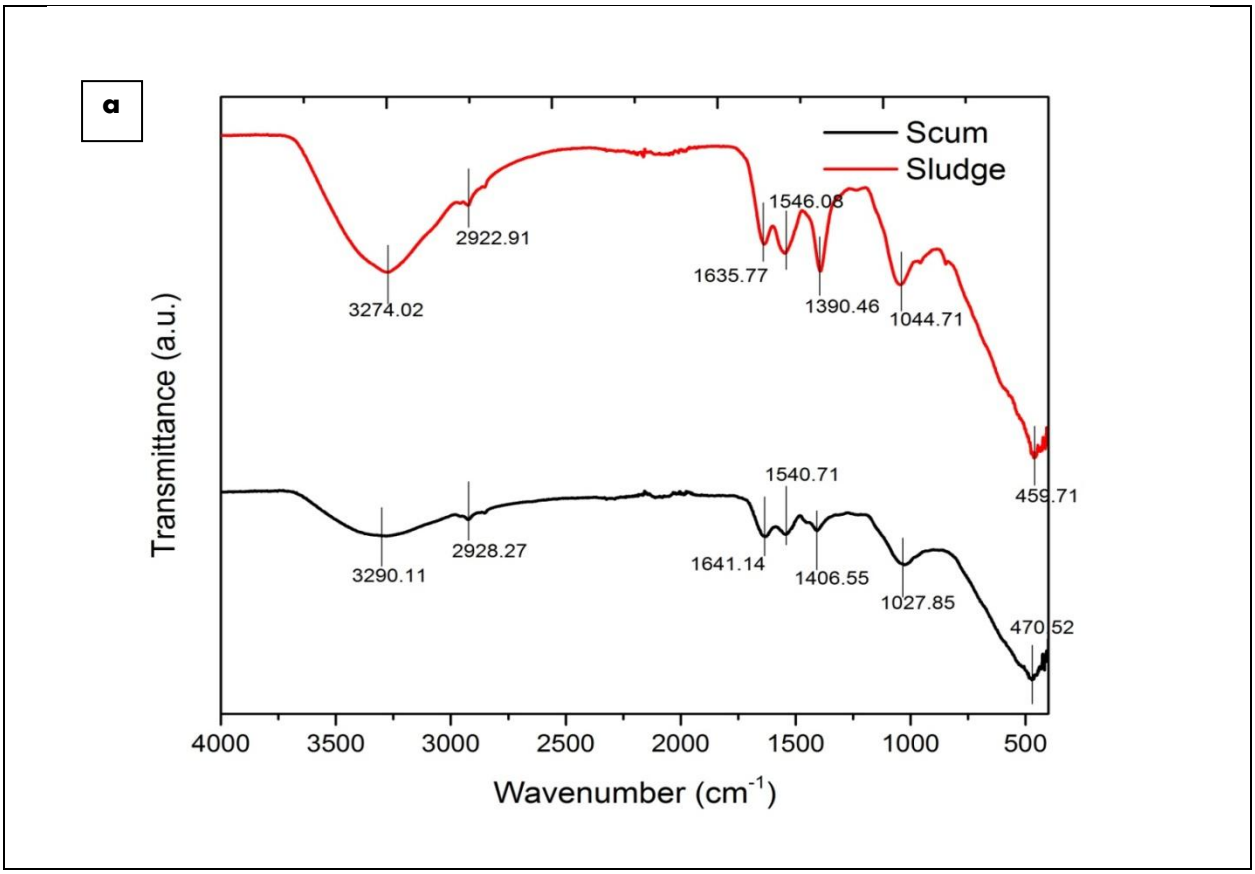


Fig6. Predicted optimal conditions for maximizing COD removal and minimizing energy consumption by RSM

3.2 Scum and Sludge analysis by FTIR and XRD

The scum and sludge collected from the optimized electrocoagulation process, was allowed to dry until all moisture is lost, and were analyzed using FTIR and XRD. Figure 7(a)shows the FTIR spectra of scum and sludge obtained from the EC process used for treating brewery wastewater. Both the spectrum exhibited negligible differences in their patterns, suggesting that both scum and sludge contains comparable functional groups. However, the FTIR spectrum of sludge demonstrated higher peak intensities indicating a higher concentration of the respective functional groups(Shankar et al. 2019).

The major peak at wavelength 3274.02 cm^{-1} of the sludge and the broad peak at wavelength 3290.11 cm^{-1} of the scum represents the O-H stretching vibration. This is attributed to the presence of hydroxyl groups from carbohydrates in the wastewater. The minor peak around 2900 cm^{-1} in the spectra of both samples represent the N-H stretching vibration, due to the presence of nitrogenous organic substances in the wastewater. It has a low intensity as its concentration is low in the wastewater, as compared to carbonaceous compounds. The peak at 1635.77 cm^{-1} wavelength in the sludge spectrum and peak at 1641.14 cm^{-1} wavelength is due to the C=C stretching of the aromatic ring, primarily the phenolic compounds derived from the malt and hops. The medium peaks around wavenumber1540 cm^{-1} represent C=C stretching vibrations of the alkene groups. The medium peaks around wavenumber 1400 cm^{-1} represent the C-H bending vibration. The peaks around 1000 to 1100 cm^{-1}wavenumber in the scum and sludge probably denote the C-O stretching group. The strong peak at wavenumber 459 cm^{-1} of the sludge and 470.52 cm^{-1} of the scum clearly indicates the Al-O stretching vibration, which represents the inorganic part of the sample. Thus, FTIR spectra affirms the presence of both organic and inorganic fractions in the scum and sludge obtained from the EC process.


a
Scum
Sludge
3274.02
2922.91
1635.77
1546.08
1390.46
1044.71
459.71
3290.11
2928.27
1641.14
1540.71
1406.55
1027.85
470.52
Transmittance (a.u.)
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)

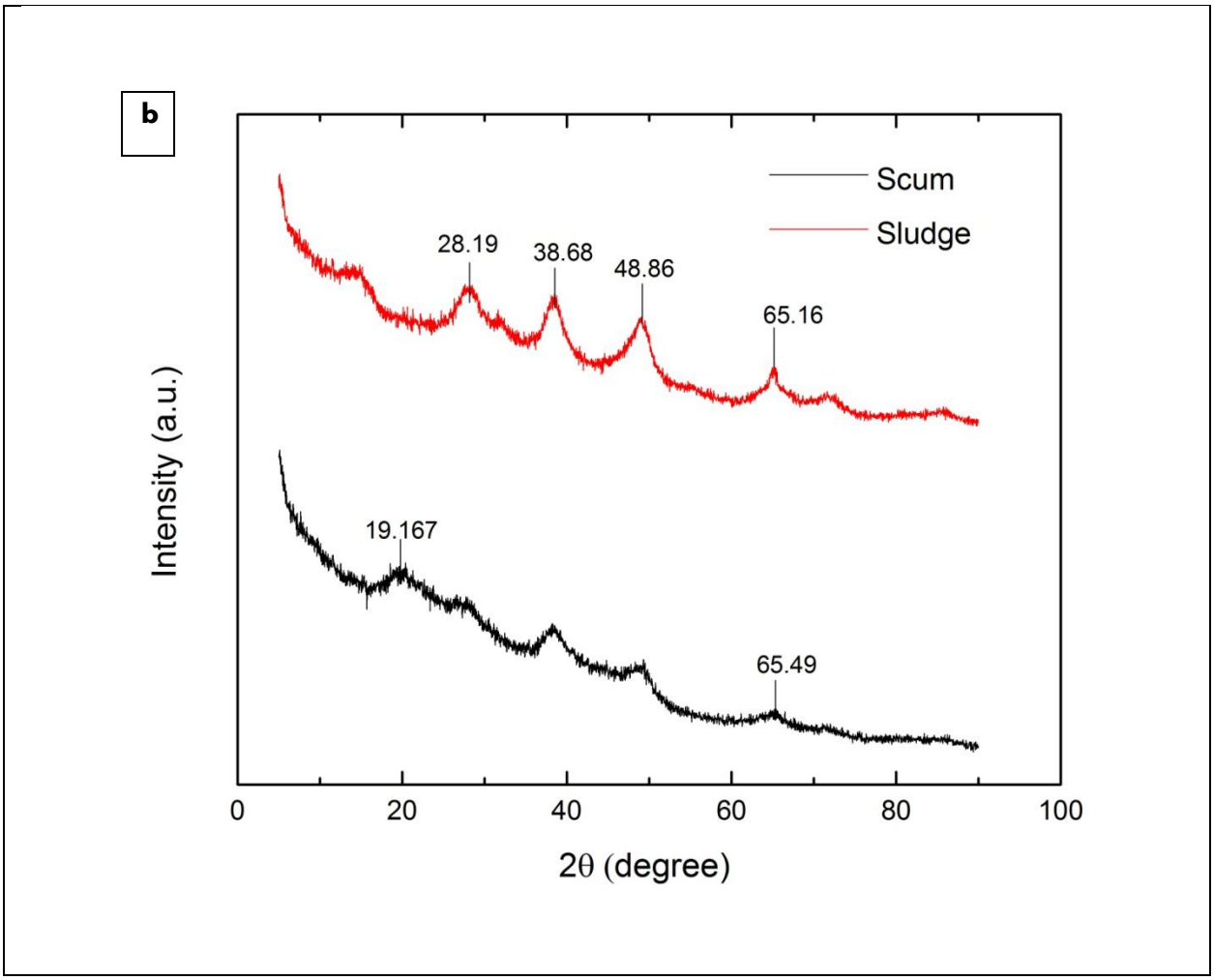


Fig7. FTIR (a) and XRD (b) plot of scum and sludge generated from Electrocoagulation process of brewery effluent

The XRD spectra of scum and sludge (Figure 8 (b)) exhibited broad and shallow diffraction peaks, indicating that the coagulants formed by aluminium electrode is largely amorphous. pH greatly influences the mechanism of the EC process and hence the nature of scum and sludge. As the EC process was carried out at near neutral pH, monomeric and polymeric aluminium hydroxide species will be the predominant species involved in the coagulation process. This is confirmed by the XRD spectra of scum and sludge. The sludge diffraction pattern shows three uniform shallow

peaks at 2Θ values of 24-30°, 36-41° and 45-51°, indicating the presence of poorly crystalline aluminium oxyhydroxide (AlOOH) and aluminium hydroxide ($Al(OH)_3$). These moderate peaks suggest partial crystallization due to gradual structural rearrangement of precipitated flocs during EC process. This observation matches with previous studies reporting that sludge generated from aluminium based EC process exhibit amorphous nature with minor crystalline phases, resulting from rapid hydroxide precipitation that restricts the development of long range crystalline order(Han et al. 2023). The XRD pattern of scum reveals a higher amorphous character, with broad and shallow peaks at 2Θ values of 15-23°, indicating the presence of amorphous aluminium hydroxide and orgnaic matter. The other peaks in the similar 2Θ values as sludge indicate the same AlOOH and $Al(OH)_3$. A small peak at 2Θ value of 65° in both the scum and sludge XRD spectra could represent traces of metallic Al or minor crystalline phases of Al_2O_3, possibly from electrode dissolution or from secondary reactions. The dominance of amorphous structure in scum along with the absence of sharp crystalline peaks is attributed to the differential structural configuration of aluminium species and higher organic content when compared to the sludge. Such similar XRD patterns have been observed in the earlier literature as adsorption by aluminium hydroxide is the predominant mechanism in EC process(Aswathy et al. 2016).

3.3. Electricity generation and treatment efficiency of MFC

Brewery wastewater, due to its high organic load and correspondingly elevated COD, requires a hybrid treatment system to achieve effective COD removal and meet discharge standards. As seen in the section 3.1.3, stand-alone optimized EC process was able to remove the nutrients and suspended solids of the brewery effluent effectively. But the important characteristic of the effluent - COD was only partially removed. This necessitates the integration with a favorable biological treatment technology in order to meet the discharge standards(Syaichurrozi et al. 2023).The energy

requirement of EC process could be reduced by integrating it with MFC, which generates energy and thus makes the treatment process more sustainable. Moreover, such a high strength wastewater cannot be treated in MFC without pre-treatment as the complex chemistry of the raw effluent would be toxic to the microbial biofilm(Kannaiah Goud and Venkata Mohan 2011).

During acclimation phase, untreated diluted brewery effluent was employed as substrate under closed circuit conditions (CCV) using an external resistance of 1000 Ω, in order to facilitate the development of biofilm(Zhuang et al. 2009).Connecting the external resistance creates a controlled environment allowing the exoelectrogenic microbes to respire efficiently, shaping their community composition, metabolism and electron transfer mechanisms. Phosphate buffer was added in every run to avoid pH fluctuations during the cycle. After 4 cycles (around 60 days), the current density achieved by the system became stable and hence marked the completion of acclimation phase.

Once the acclimation phase is over, the MFC was loaded with 1/3rd strength EC treated brewery effluent with a COD concentration of 2775 mg/L. The strength of the wastewater was determined based on the observations from preliminary trail experiments. The EC pre-treated brewery effluent contains most of its COD in soluble form and the anaerobic anodic environment, in general will facilitate several oxidation based processes such as fermentation, acidogenesis, acetogenesis, microbial electrogenesis and methanogenesis(Kokko et al. 2018). However, the MFC system had been previously acclimatized, leading to the development of a stable electrogenic biofilm, which is expected to enhance electrogenic activity. Each batch of substrate lasted for about 10 days, after which there was a notable voltage drop due to substrate depletion. During this batch MFC process, a high COD removal of 91.9% was achieved by the MFC system, resulting in a COD of 225 mg/L (which is below the 250 mg/L discharge limit set by CPCB). This is higher than a similar dual-

chamber MFC system which achieved 79-83% COD removal with real brewery effluent(Negassa et al. 2021).

Polarization experiment was performed by connecting a series of resistors and finding their respective current densities. A polarization curve plots the cell voltage against the current density and it helps us to understand the electrochemical losses that limit the performance of a MFC. As seen in the figure 8, a typical polarization curve was obtained for this MFC system with initial drop in voltage caused by activation losses and final drop in voltage caused by concentration losses(Logan et al. 2006). The mid linear region has a high slope value indicating the high ohmic loss and internal resistance of the MFC system. Based on the power curve derived from the experiment, a maximum power density of 11.81 mW/m^2 and a corresponding current density of 48.6 mA/m^2 was found to be achieved by this dual-chambered MFC at an internal resistance of 1000 Ω. While the generated power density is comparable to a similar dual-chambered MFC with abiotic cathode employing ghee industrial wastewater as substrate(Elakkiya and Niju 2025), it is low compared to other air-cathode MFCs with brewery wastewater as substrate(Negassa et al. 2021).The high internal resistance of the system here is mainly attributed to the dual-chamber system design. While the dual-chamber design offers control and separation, it inevitably causes an increase in the ohmic resistance. Moreover, in this study the diluted pre-treated brewery effluent is used as substrate in the MFC, which has low ionic strength. Using advanced system designs with better electrode materials will help in reducing the high internal resistance of such MFC systems(Gajda et al. 2018).

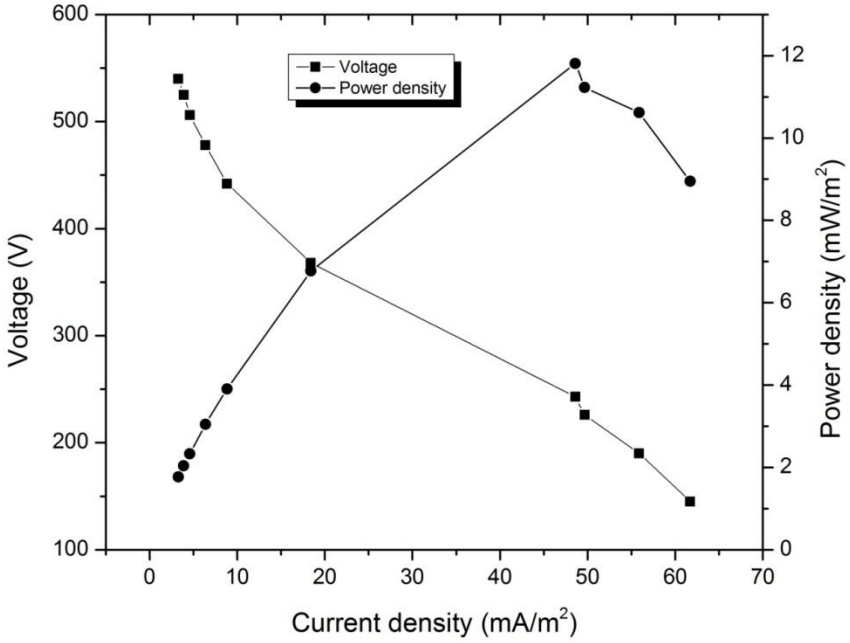


Fig8. Polarization and power curve of MFC with EC pre-treated brewery effluent as substrate

3.4. Combined effect of EC and MFC systems for treating brewery effluent

Figure 9 illustrates the performance of the sequential treatment system for treatment of brewery wastewater, comprising the optimized electrocoagulation process as a pre-treatment step followed by Microbial Fuel Cell technology as a post-treatment, evaluated in terms of COD removal efficiency. The EC and MFC processes exhibit complementary functionalities, with electrocoagulation facilitating nutrient and particulate COD removal, and the Microbial Fuel Cell enhancing the degradation of soluble organic constituents(Tejedor-Sanz et al. 2017). An overall COD removal efficiency of 98.5% has been achieved by this sequential treatment system. Under batch operation, the MFC produced approximately 0.014 Wh of electricity over a 10-day cycle while treating 300 mL of wastewater. When normalized to unit volume, this corresponds to an

energy recovery was 0.047 kWh/m³. Compared to the optimized EC energy demand of 11.14 kWh/m³, the recovered bioelectricity could offset approximately 0.42% of the EC energy requirement. This offset is indeed low in the energy perspective. However, future research aimed at improving the power generation efficiency of MFCs could enhance the overall energy balance and sustainability of the sequential treatment system.

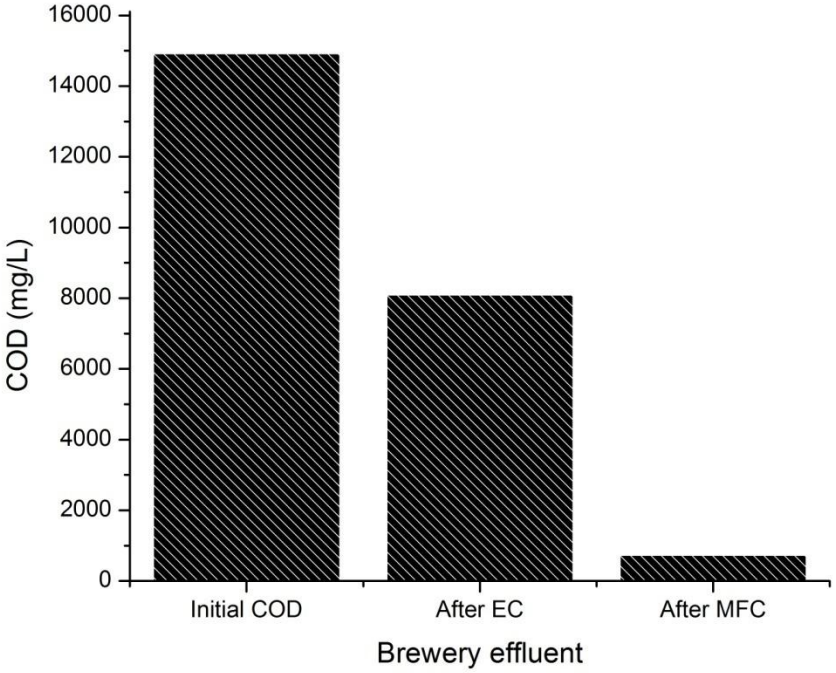


Fig9. Comparison of COD values throughout the sequential treatment process

4.Conclusion

The electrochemical methods, including electrocoagulation and MFC have a high potential in the treatment of brewery effluent. Their integration is advantageous because of the complementary nature of the two processes. The electrocoagulation process is a simple and effective process, that doesn't use any chemicals compared to other conventional techniques. It was employed here as

pre-treatment technique and it had efficiently removed the suspended organic matter along with nutrients such as nitrate and phosphate. The operating conditions of this process was optimized using CCD of RSM for higher treatment efficiency and lower energy consumption. The subsequent treatment involves biological treatment of the pretreated brewery effluent with the help of electroactive bacteria in the MFC. This step removes the soluble organic content of the MFC efficiently. Overall, this sequential treatment process of EC followed by MFC achieved a COD removal of 98.5% from the industrial brewery effluent. Further modifications in the design of MFC and its optimization would result in a much better energy production, that will contribute for a more sustainable wastewater treatment. In addition, the scum and sludge generated in the electrocoagulation process could be valorized for compensating the energy needs. Thus, this work is an exploratory step towards circular bioeconomy in wastewater treatment. EC and MFC technologies, both operating on electrochemical principles, are inherently complementary process that can synergistically enhance overall wastewater treatment efficiency.

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Statements and Declarations

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Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contributions

KP: Conceptualization; writing –original draft; methodology; writing – review and editing; data curation; validation

SN: Methodology; writing– review and editing; project administration; resources; formal analysis

Data Availability

The data supporting the findings of this study are available within this article.