Enhancing textile wastewater treatment efficiency through double stage electrocoagulation optimization

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ABSTRACT:

Water has been a crucial element for human survival due to our reliance on it for various purposes. The textile industry, among other water-intensive sectors, releases wastewater containing high levels of BOD, COD, toxic colored compounds, variable pH, etc., into the environment.

Numerous technologies have been attempted to treat wastewater from the textile industry with limited success. One of the additional technologies used is Electrochemical-based treatment for removing organic dyes from wastewater. Electrocoagulation is one such technology in which consumable electrodes supply ions to the wastewater, causing the destabilization and aggregation of pollutant particles. This process involves the oxidation of the electrode, followed by the generation of gas bubbles, and subsequent flotation and/or sedimentation of the formed flocs.

The present study focuses on optimizing various operating parameters for treating textile wastewater using a double-stage Electrocoagulation system. To facilitate the study, various parameters influencing the Electrocoagulation operation were discussed, along with reactor design issues.

Experiments were conducted in a continuous mode double-stage reactor system followed by a lamella settler. It was observed that all response functions, including COD removal, color removal, electrode consumption, and energy consumption, varied directly with the current density of both reactors. Furthermore, it was noted that increasing the flow rate reduced the efficiency of removal, and the maximum removal was achieved with a lower combination when increasing the flow rate.

The Design of Experiments was used to develop a model in Design Expert software for optimizing various parameters. Validation of the model was done through an analysis of variance, which included analyses of R2 and adjusted R2 values and a normal probability plot of residuals. A confirmatory test was also performed using the results obtained from the model, which reaffirmed the applicability of the developed model.

Keywords: Textile Wastewater, Electrocoagulation, B.O.D, C.O.D, Current Density, Electrode Consumption.

1. INTRODUCTION

Various materials are discharged into water bodies, originating from land erosion, mineral dissolution, vegetation decay, and domestic and industrial waste. These materials include both organic and inorganic substances, along with various biological forms such as bacteria, algae, and viruses (Bratby, 2006).
However, there are many challenges in utilizing EC as a treatment technology. There is no systematic approach to EC reactor design and operation available in the literature. This highlights the need for thorough research in this area to develop better and more promising solutions.

1.1 Objectives of the Study

The study will examine the effects of the following two parameters:

i. Flow rate
ii. Current Density: Since the study utilizes a double-stage EC reactor system, the current densities of both reactors will be varied separately.

The effects of these parameters will be separately and collectively studied on the following output responses:

i. COD removal
ii. Color removal
iii. Electrode consumption
iv. Energy consumption

A continuous flow technique will be used to enhance the efficiency of the EC process. Additionally, new reactor designs have been proposed, and the combination of two reactors and a lamella settler in series will be used to conduct the study.

2. MATERIALS AND METHODS

2.1.Experimental Setup

The experimental setup used in the study is illustrated in the figure below. It primarily consisted of two reactors made of Perspex sheet, each with an effective volume of 3 liters, while their total volume was approximately 5.6 liters each. An electrode holding cassette was prepared to keep the electrodes in their respective positions during the experimental run. Aluminum electrodes, used as anodes and cathodes, measured 9.7 x 8.0 x 0.2 cm each, making their effective surface area 160 cm^2 each. Two anodes and three cathodes were arranged in a monopolar configuration in both reactors. The inter-electrode spacing was set at 2 cm, and they were connected to an external DC Power Supply unit in series mode. The DC power supply unit was equipped with an ammeter and voltmeter for current and voltage readings, respectively. The lamella settler, following the two reactors, was made of Perspex sheet and had a working volume of 7.7 liters. The lamella settler comprised 22 parallel plates made of polyvinyl chloride (PVC) material to enhance the settling characteristics of suspended solids.

Fig. 1: Experimental setup used in the study (1= Peristaltic Pump; 2= Overhead Tank; 3=DC Power Supply Unit; 4= Reactor 1; 5= reactor 2; 6= Lamella Settler)

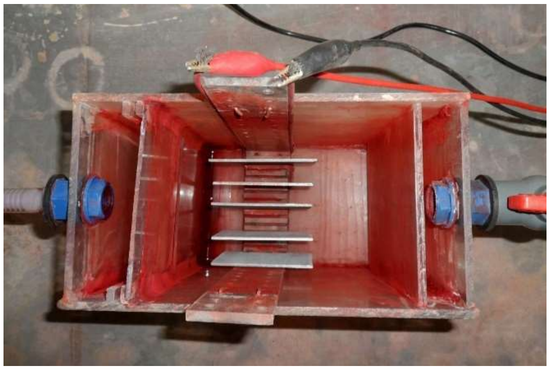

Fig. 2: Arrangement of electrodes in the cassette

2.2.Instruments Used.

i. DC power supply unit of KUSAM- MECO make, model number KM-PS-305D-IIwith 0-30V/0~5A dual output
ii. Closed Reflux apparatus of HACH (DRB 200)
iii. UV/VIS Spectrophotometer of SHIMADZU (UV-1800)
iv. Conductivity meter of Lutron (CD-4302) with range of 0-20Ms
v. Peristaltic pump of EnerTech, model number ENPD 100 Victor
vi. Digital weighing balance of CAS series, model number CAUW 220 D with range of a mg to 220 gm Glass ware - pipettes, test tubes, COD digestion vials, measuring cylinders, conicalflasks, etc.
vii. EC cells and lamella settler – Perspex sheet, PVC material.

2.3.Experimental Work

Synthetic textile wastewater was prepared to serve as a sample for the experiment. Different concentrations of the dye result in varying COD levels in the wastewater. For our experimental work, the dye concentration used was 300 mg/l, resulting in a COD level in the range of 500-600 mg/l.

The dye was then combined with the following chemicals to accurately simulate the actual dye waste released from textile industries. This mixture was added to tap water and heated to 100°C for 45 minutes. The resulting solution was then cooled to room temperature.

S.No.	Chemical required	Amount	For 100 l
1.	Dye	0.3 g/l	30 g
2.	Sodium chloride	3 g/l	300 g
3.	Hydrolyzed starch	0.00556 g/l	0.56 g
4.	Ammonium sulfate	0.0174 g/l	1.174 g
5.	Disodium hydrogen phosphate	0.0174 g/l	1.174 g
6.	Liquid detergent	Few drops	Approx. 50 ml

Table 1 Composition of dye waste water used (Alinsafi A, 2005)

2.4.Analytical methods used

For all the experiments performed, the physico chemical parameters were analyzed by the methods described in standard methods for the examination of waste water, 20th edition **(APHA.AWWA. WEF, 1999)**.

2.5.Statistical methods used

Statistical design of experiments (DOE) and statistical models were used to study the effectof different parameters (factors) and their interaction terms on pollutant removal from textile waste water. 2^k full factorial design containing all the possible variations was used.

2.6.Evaluation of removal efficiencies

Each sample collected from the EC cells and the lamella settler was analyzed for color and COD using spectrophotometer at 505 and 600 nm respectively.

The removal efficiency of COD in water sample after treatment using EC was calculated as follows:

$Y\% = ((C_{\circ} - C)/ C_{\circ}) \times 100$

Where,

$Y\%$ = COD removal efficiency

$C_{\circ}$ = initial COD concentration (mg/l)

C = COD concentration at any time t (mg/l)

Color removal efficiency of the treated synthetic waste water sample was also calculated as: $Z\% = ((D_{\circ} - D)/ D_{\circ}) \times 100$

Where,

$Z\%$ = color removal efficiency

$D_{\circ}$ = initial color in the sample (mg/l) D = color at any time t (mg/l)

3. RESULTS AND DISCUSSION

3.1.Design of Experiments

It is an experimental strategy in which the effects of multiple factors are studied simultaneously by running tests at various levels of factors. These factors are the variables that have a direct influence on the performance of the product or processes under investigation (Taguchi). Design of Experiments (DOE) helps to minimize the number of experiments to be conducted.

DOE techniques enable to determine simultaneously the individual and interactive effects of many factors that could affect the output results in any design. It also provides a full insight of interaction between design elements thereby helping to turn any standard design into a robust one (Technologies).

3.2.Full Factorial Design

DOE is a method used to identify important factors in a process, diagnose and rectify problems within a process, and assess the potential for estimating interactions. In practice, multiple factors typically influence a process, and it is crucial to consider them together if they interact, a task accomplished through full factorial DOE (Young).

If there are 'k' factors to be evaluated in a process, the experiment needs to be run 2k times, with each factor having two levels: a 'high' and a 'low' level (Young). These high and low values must be sufficiently distant to produce a noticeable effect, yet close enough to assume a linear change in the test outcome as each factor varies between high and low values (Coppola). Extrapolating beyond the extreme levels or ranges used in the test (coded as -1 and +1) assumes the factor's impact is linear beyond the tested region, which can be risky, so it is generally avoided.

The effect of a factor is defined as the average response when the factor changes from one level to another (Young).

3.3.The 2^k Factorial Design

Usually, in the early stages of experimental work, there are numerous factors to be investigated, and the 2k design is particularly useful here as it provides the smallest number of runs with which 'k' factors could be studied in a complete factorial design. Here, we assume that the response is approximately linear over the range of factor levels chosen since there are only two levels of each factor, which is often a reasonable assumption (Montgomery, 2001).

3.3.1. Inclusion of centre points.

It is a useful extension to the two-level factorial design, which allows a user to check the goodness-of-fit of the planar two-level factorial models. The inclusion of center points also provides extra error degrees of freedom to the model (Expert, 2009).

3.3.2. Replicates

Number of replicates means how many times an experiment is run for each design point. If two replicates are asked for, it means each experimental condition is repeated once. A true replicate of a design point is the result of physically re- creating all the conditions for that experiment thus giving a more accurate estimate of the overall process error (Expert, 2009).

3.4.Design Expert

The software is created to aid in the design and interpretation of multi-factor experiments. It provides a diverse range of designs, including factorials, fractional factorials, and composite designs. It has the capability to handle both process variables and mixture variables.

3.4.1. Three main Steps for Design Expert

1. Constructing the design
2. Evaluating the design
3. Modeling and interpreting the experimental data.

3.4.2. Methods used by Design Expert for analysis of a design

1. Effects :- Half Normal and Normal plots for highlighting active factors, Pareto Chartfor giving a picture of relative sizes of different effects
2. ANOVA :- includes Analysis Of Variance, Signal to Noise ratio (Adeq Precision), coefficients of fitted model
3. Diagnostics: - residual plots, Box-Cox plot for power transformations, and plots of leverage and influence statistics are used.
4. Model Graphs: - utilizes one factor, Interaction, Contours, 3-D surfaces and Cubesfor representation of results.

3.5.The procedure adopted for Design of an Experiment

i. A cause and effect analysis of all the process inputs (variables) and outputs (responses) was prepared.
ii. Documentation of the process to be studied which includes review of process flowcharts, written procedures, etc. was done.
iii. A detailed problem statement was written down which included identification of responses to be studied, identification of design variables and possible interaction between them, identification of assumptions, statement of goals and limitations, estimation of time and materials required, etc.
iv. Preliminary Experimentation was done which included refining of experimental procedure, confirming that process was in control and all equipment was operating correctly.
v. Design of Experiment applied which included selection of an appropriate design with

considering opportunities to add a variable if required.

vi. Replicates, Randomization and Blocking:- this included determination of number of replicates which means the number of times each experiment is repeated and helps in estimating errors and increases the precision of estimates of effects, randomization of study variables which reduces the risk of unanticipated sources of variation affecting the estimates of effects and helps to meet the assumptions of statistical methods used in analyzing experimental data, and blocking which splits up the design in two or more blocks so that some unavoidable sources of variation could be corrected.

vii. Experiments were conducted as per the decided strategy.

viii. Analysis of Data was done to confirm the accuracy of the data through graphing of the data, running ANOVA/ regression, determination of model standard error and r^2.

ix. Results were interpreted which included developing a predictive model for response, selecting the optimum variable levels without extrapolating outside the range of experimentation.

x. Optimization results were confirmed by running another test at the optimized settings to see if expected results do indeed result. It is always possible that some factor not tested has a significant impact on the outcome.

xi. Documentation of the results.

3.6.Desirability Functions

The provided cube illustrates the variation of the desirability function as we move across the ranges of the three considered factors. The maximum desirability is observed when the flow rate approaches 200 ml/min, with current densities in the first and second reactors reaching their upper limits, i.e., 60 and 45 A/m². From the cube, it is evident that optimized solutions could be obtained when the flow rate is near 200 ml/min, the current density of the first reactor is approximately 60 A/m², and that of the second reactor is varied over its range, resulting in desirability values between 0.881 and 0.993.

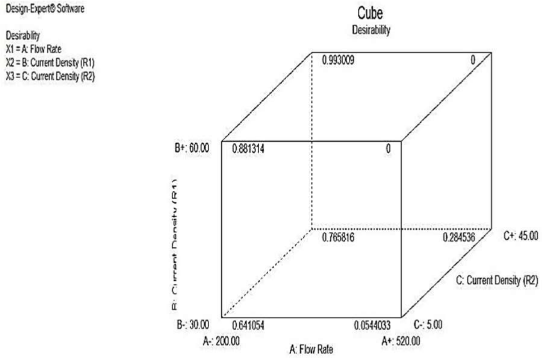

Fig. 3: Desirability Function

3.7.Result from Design Expert

A 2-level full factorial design was employed, allowing each factor to vary over two levels (high or low, with values +1 and -1, respectively). This design is beneficial for estimating main effects and interactions. Three input variables were chosen: Flow rate (ml/min), Current density in the first reactor, and Current Density in the second reactor (A/m²). Their effects and interactions were examined for four output responses: Color Removal Efficiency (%), COD Removal Efficiency (%), Electrode Consumption (mg/mg COD), and Energy Consumption (KWh/Kg COD).

3.7.1. Coded Design

Std.	Run	Block	actor 1: (A) Flow rate	Factor2: (B) Current Density R1	Factor3: (C) Current Density R2	Response1: Color Removal	Response 2: COD Removal	Response 3: Electrode Consumption	Response 4:Energy Consumption
16	1	{ 1 }	1	1	1	83.8	26.54	0.46	2.27
20	2	{ 1 }	0	0	0	73.73	50.52	0.31	1.5
18	3	{ 1 }	0	0	0	71.43	51.13	0.33	1.4
2	4	{ 1 }	-1	-1	-1	86.3	53.68	0.26	0.98

9	5	{ 1 }	-1	-1	1	93.7	74.36	0.21	1.33
14	6	{ 1 }	-1	1	1	98.9	93.2	0.35	2.28
17	7	{ 1 }	0	0	0	74.05	50.89	0.32	1.5
15	8	{ 1 }	1	1	1	84.74	25.67	0.45	2.28
3	9	{ 1 }	1	-1	-1	19.2	26.87	0.3	0.52
19	10	{ 1 }	0	0	0	73.02	52.5	0.33	1.4
11	11	{ 1 }	1	-1	1	53.4	31.49	0.24	1.16
7	12	{ 1 }	1	1	-1	58.1	17.12	0.5	2.06
6	13	{ 1 }	-1	1	-1	96.5	80.92	0.21	1.41
4	14	{ 1 }	1	-1	-1	20.4	25.67	0.31	0.54
12	15	{ 1 }	1	-1	1	53.09	30.98	0.24	1.17
8	16	{ 1 }	1	1	-1	57.35	16.79	0.48	2.03
1	17	{ 1 }	-1	-1	-1	87.1	54.6	0.27	0.97
13	18	{ 1 }	-1	1	1	99.12	94.06	0.36	2.3
5	19	{ 1 }	-1	1	-1	97.6	81.24	0.23	1.43
10	20	{ 1 }	-1	-1	1	93.1	74.07	0.2	1.32

Table 2: Randomized Coded design

Constraints						**Importance**
Name	**Goal**	**Lower Limit**	**Upper Limit**	**Lower Weight**	**Upper Weight**	
Flow Rate	is in range	200	520	1	1	3
Current Density (R1)	is in range	30	60	1	1	3
Current Density (R2)	is in range	5	45	1	1	3
Color Removal	maximize	19.2	99.12	1	1	3
COD Removal	maximize	16.79	94.06	1	1	5
Electrode Consumption	is in range	0.2	0.5	1	1	3
Energy Consumption	is in range	0.52	2.3	1	1	4

Table 3: Factors and Responses with their set criteria

In the desirability function, each response can be assigned an importance relative to other responses, with importance ranging from least important with a value of 1 to most important with a value of 5 (Expert, 2009).

Moreover, the goal of COD and color removal has been set to maximize to achieve a favorable outcome concerning both responses, while all other factors and responses have been set to fall within a range. This maximization, however, limits the model from providing all possible sets of numerically optimized solutions, and only those solutions that maximize COD and color removal are reported by the model.

3.7.2. Numerically optimized solutions from Design Expert

The solution table obtained from the software had 48 solutions varied over different values of various factors to obtain various responses. Most favorable results have been compiled here.

S.No.	**Flow rate (m^3/s)**	**Current Density 1 (A/m^2)**	**Current Density 2 (A/m^2)**	**Color Removal (%)**	**COD Removal (%)**	**Electrode Consumption (mg/mg COD)**	**Energy Consumption (KWh/Kg COD)**	**esirability function**
1.	200	60	45	100	93.197	0.355	2.116	0.993

2.	200	60	38.94	99.539	91.427	0.334	2.037	0.979
3.	200	60	38.85	99.313	90.817	0.327	2.009	0.974
4.	200	60	34.05	99.01	89.999	0.318	1.973	0.966
5.	200	60	30.33	98.607	88.912	0.305	1.924	0.956
6.	200	53.63	45	98.502	88.279	0.323	1.966	0.950
7.	200	51.39	45	97.906	86.549	0.312	1.913	0.933
8.	237.43	60	45	98.534	85.498	0.367	2.151	0.927
9.	200	46.73	45	96.67	82.945	0.289	1.803	0.897
10	200	31.62	45	92.646	71.274	0.213	1.449	0.729
11	200	36.66	5	89.71	63.636	0.254	1.05	0.698
12	200	30	18.27	89.32	62.21	0.245	1.062	0.683

Table 4: Numerically Optimized solutions from Design Expert

3.8.Confirmatory Test Run

A confirmatory test was performed to validate the results obtained after numerical optimization using Design Expert. The results from the test run confirmed the applicability of the model, as they closely aligned with those obtained from Design Expert.

S.No	Flow rate (m^3/s)	Current Density 1 (A/m^2)	Current Density 2 (A/m^2)	Color Removal (%)	COD Removal (%)	Electrode Consumption (mg/mg COD)	Energy Consumption (KWh/Kg COD)	Desirability function
Results from Design Expert								
1.	200	60	34.05	99.01	89.999	0.318	1.973	0.966
Result from test run								
2.	200	60	34.05	98.69	87.63	0.324	1.968	n.a.

Table 5: Comparison between results from Design Expert and Test run.

3.9.Model Graphs

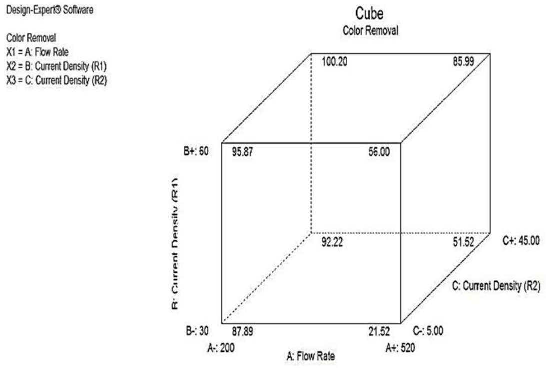

Figure 4: Model Graph for Color Removal

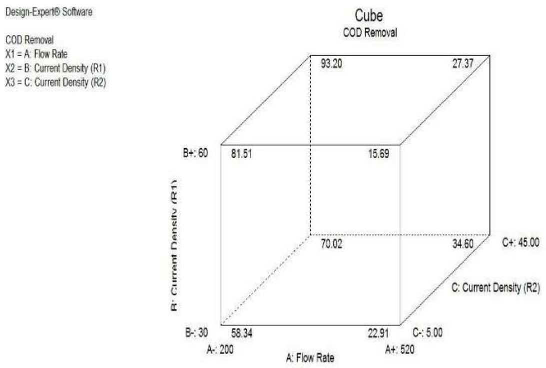

Figure 5: Model graph for COD Removal

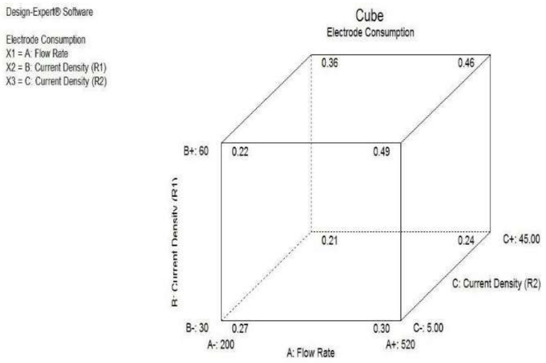

Figure 6: Model graph for Electrode Consumption

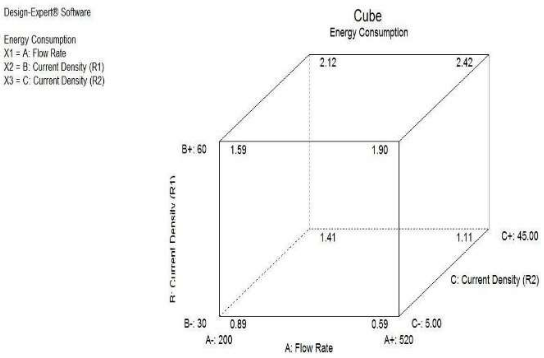

Figure 7: Model graph for Energy Consumption

3.10. Actual v/s Predicted Plots

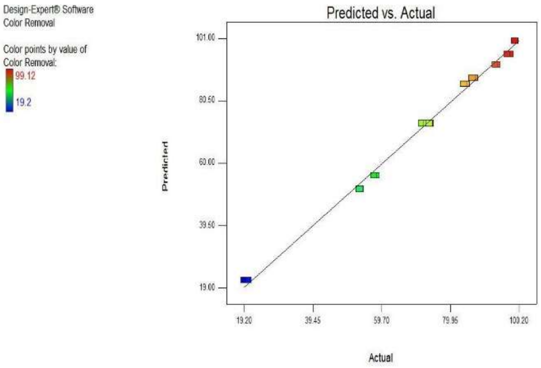

Figure 8: Actual vs. predicted plot for Color Removal

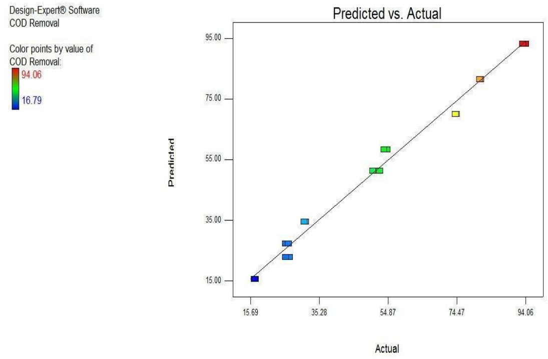

Figure 9: Actual vs. predicted plot for COD Removal

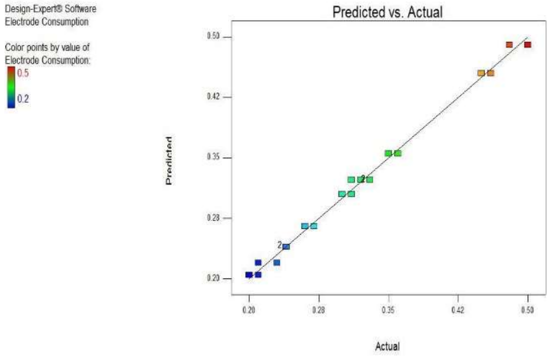

Figure 10: Actual vs. predicted plot for Electrode Consumption

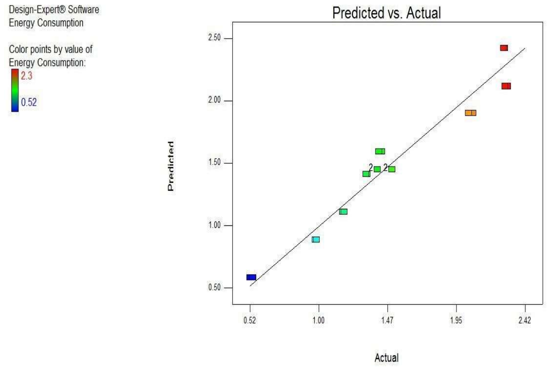

Figure 11: Actual vs. predicted plot for Energy Consumption

3.11. R- Squared and Adjusted R- Squared values

S.No.	Response Function	R squared	Adjusted R squared
1.	Color Removal	0.9962	0.9947
2.	COD Removal	0.9895	0.9865
3.	Electrode Consumption	0.9943	0.9907
4.	Energy Consumption	0.9551	0.9422

Table 6: R squared and Adjusted R squared values for different responses

3.12. Validation of the Model

A residual is defined for observation j in treatment I as: - eij = yij – ŷij
Where, ŷij is equal to the estimate of corresponding observation yij.

An extremely useful procedure is to construct a normal probability plot of the residuals. If the underlying error distribution is normal, this plot will resemble a straight line with more emphasis on the central values of the plot than the extremes. An error distribution that has considerably thicker or thinner tails than the normal is of more concern than a skewed distribution (Montgomery, 2001).

About 68 percent of standardized residuals should all within the limits ±1, about 95 percent of them should fall within ±2 and virtually all of them should fall under ±3. Values outside this range are potential outliers (Expert, 2009).

3.13. Normal Probability Plot of Residuals

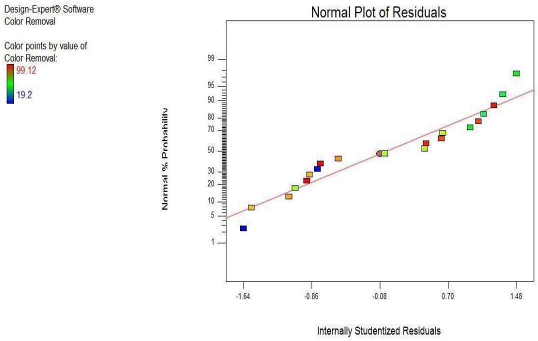

Figure 12: Normal probability plot of residuals for Color Removal

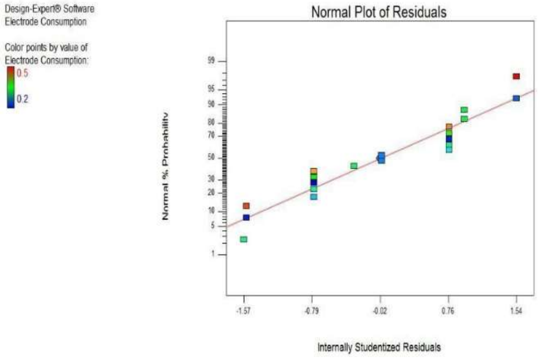

Figure 13: Normal probability plot of residuals for COD Removal

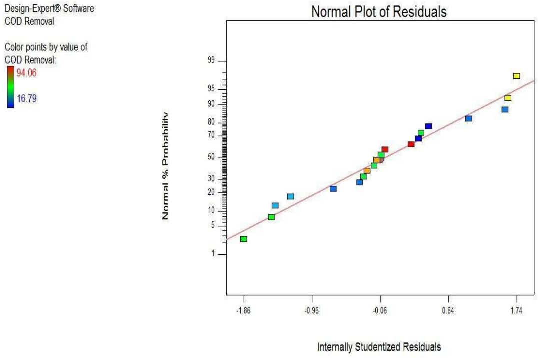

Figure 14: Normal probability plot of residuals for Electrode Consumption

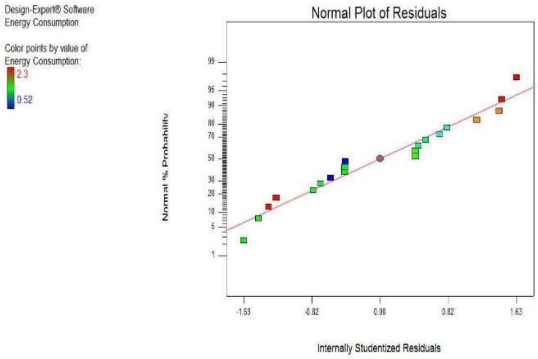

Figure 15: Normal probability plot of residuals for Energy Consumption

4. CONCLUSION

In the present study, experiments were conducted to determine the best operating conditions for achieving acceptable COD and color removal with low energy and electrode consumption.

i. Both COD removal and color removal increase with the current density, but the rate of increase decreases with increasing current density above a certain value.
ii. An increase in flow rate works against COD and color removal, as the removal rate drastically decreases with an increase in flow rate.
iii. Both electrode consumption and energy consumption vary in accordance with current density, resulting in a higher level of treatment being more cost-intensive at the same time.
iv. For a flow rate of 200 ml/min, the maximum efficiency of COD and color removal (93.20% and 99.60%, respectively) was obtained for the 60-45 A/m² combination of current densities, which is also the highest combination used. Whereas, for a flow rate of 520 ml/min, the maximum efficiency of COD and color removal (63.20% and 93%, respectively) was achieved for the 45-45 A/m² combination, after which the efficiency again reduced, suggesting that the maximum is achieved with a lower combination as we increase the flow rate.
v. The most energy-efficient combinations were found to be 60-15 A/m² for 200 ml/min and 45-05 A/m² for 520 ml/min, suggesting that these combinations removed more kilograms of COD for one kWh of energy consumed than others.
vi. A model was developed using the basics of Design of Experiments (DoE) with the help of Design Expert software. The model was validated using analysis of variance. The R² and adjusted R² values and normal probability plots of residuals suggested that the model used is adequate for the study.
vii. Numerically optimized solutions were obtained from Design Expert software, out of which an optimal solution was selected and tested for confirmation of the results. The results obtained from the test run reaffirmed the applicability of the model, as both results were close enough to be related.

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