JEAS

JOURNAL OF ENGINEERING AND APPLIED SCIENCES

Journal of Engineering and Applied Sciences, Volume 15, Number 1, December 2019, 99-108

Optimal Route for Turbidity removal from Aquaculture Wastewater by Electrocoagulation-flocculation process

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Abstract
The purpose of this study is to investigate and verify the applicability of the electrocoagulation technology using iron-aluminum (Fe-Al) electrodes in the effective treatment of wastewater generated from aquaculture ponds. The wastewater was characterized before and after treatment to know the effectiveness of this treatment method. A laboratory electrocoagulator was used to investigate the effects of different parameters such as initial pH, charge time, and settling time on turbidity removal from aquaculture wastewater (AW) at initial pH of 8 and temperature of 30 ^{0}C. The electrocoagulation process was optimized using the Response Surface Methodology (RSM) through the Box-Behnken Design (BBD). A quadratic model was also generated to represent the treatment process. The optimum conditions were found to be charge time of 11.970 min, settling time of 29.994 min and current density of 2.389A which gave turbidity removal of 91.84 % at 30 ^{0}C for 500 mL of the wastewater. The correlation between the predicted value (91.84 %) and measured value (91.67 %) confirms the validity of the results predicted by the regression model. A Predictive model describing the turbidity removal efficiency in terms of process variables was derived from multiple regression analysis. The coefficient of regression (R^2) value of 0.9943 and the adjusted R^2 of 0.9858 indicate the validity of the model equation. The RSM via BBD can be used to optimize and maximize the electrocoagulation-flocculation treatment of aquaculture wastewater.

Keywords: Electrocoagulation-flocculation, Iron- aluminium electrodes, Aquaculture wastewater, Response surface methodology, Analysis of variance

1.0 Introduction

The water after its contamination especially due to the anthropogenic activities can contain a wide range of soluble and insoluble pollutants including colloidal particles, microorganisms, dissolved organic and inorganic compounds. The colloidal pollutants have a negatively charged surface that prevents the particles to approach their separation, thus remaining in an environment, which can favour their stability (Zaleschi et al. 2012).

Fisheries and its resources comprises a wide source of food and feed a large part of the world's population besides a huge employment sector (FAO 2016). This sector also generates large amounts of wastewater which the treatment is particularly challenging due to the high content of organic matter, salts, and significant amount of oil and grease (Gonzalez 1995). This wastewater is commonly treated using chemical, biological, and electrocoagulation treatment processes. Chemical and biological treatment of wastewater is usually linked with the production of greenhouse gases and activated sludge along with some other limitations regarding required area and removal of residual chemicals, respectively (Ali & Yaako 2012). On the other hand, electrocoagulation is an extremely effective technique.

The application of electricity to treat water was first proposed in 1889 in England (Wang et al. 2007). Electrocoagulation is a complex method, with many synergistic mechanisms working to remove water pollutants

(Zaleschi et al. 2012). Electrocoagulation (EC) is a process in which the sacrificial anode material undergoes oxidation or corrode with the formation of various monomeric and polymeric metallic hydrolyzed species which is the active coagulant (Nwabanne et al. 2018). This technology is a treatment process which applies an electrical current to treat and flocculate contaminants without having to add coagulants. In the electrocoagulation process, it is important to use soluble anodes made of aluminum, iron or other material, and cathodes made of the same material, or steel (Zaleschi et al. 2012). Electrocoagulation is effective wastewater for the removal of pollutants (Naje et al. 2016), and at the same time produces hydrogen gas as revenue to recompense the cost of operation (Ezechi et al. 2010; Ali & Yaako 2012). Electrocoagulation has been documented effectively to treat many types of wastewater, provided that the pollutants can react to the electric field in a redox reaction (Razali et al. 2016; Tak et al. 2015). Other advantages of electrocoagulation method include the management of broad wastewater types, sludge reduction, ease of operation with less complex control and low sludge production (Razali et al. 2016).

The main goal of this research is to investigate and verify the applicability of the electrocoagulation technology using iron-aluminum electrodes in the effective treatment of wastewater generated from aquaculture ponds using the Box-Behnken Design (BBD) method. An experiment may require testing so many factors to know their effects which can be done using the one-factor-at-a-time (OFAT) method (Mark 1997). Most of the research on the optimization of wastewater treatment processes have centered on the traditional OFAT approach (Tak et al. 2015). Due to too many experimental runs are needed to get sufficient information about the set of conditions contributing to the problem, this consumes time and money with the high risk of error. Another limitation is that when factors change they generally change together, so it is impossible to understand the best solution by pointing to a single, isolated factor. This is to say that the traditional OFAT method of experimentation is a just trial and error method and reliance on common sense (Mark 1997). Therefore, the study of the aquaculture wastewater treatment using the Box-Behnken Design (BBD) method is necessary towards the development of more effective treatment of this wastewater.

Response surface methodology (RSM) is a collection of mathematical and statistical techniques for empirical model building, improving and optimizing processes parameter and it can also be used to find the interaction of several affecting factors (Beyki et al. 2017; Montgomery 2014). RSM can be used to determine the regression model and optimize a response which is influenced by several independent variables (Behera et al. 2018).

2.0 Material and methods

2.1 Effluent sample collection

The aquaculture effluent was collected from the Faculty of Agriculture, Nnamdi Azikiwe University at the point of flushing the fish pond using three 20 L jerry-cans in July 2015. The jerry-cans were filled to the brim in order to expel entrapped air within the jerry-can. The jerry-can was then corked sealed and refrigerated until the commencement of the experiment. The characteristics of the effluent were determined according to the standard methods for the examination of water and wastewater (APHA, AWWA, WEF 2012). All experiments were conducted at the laboratory of the Chemical Engineering Department of Nnamdi Azikiwe University in Awka, Anambra state, Nigeria.

2.2 Electrocoagulation-flocculation experiment

The Electrocoagulation unit is made of Perspex sheet with dimensions of 36 cm × 15 cm × 23 cm with an in-built current regulator. The electrodes used in the electrocoagulation process were aluminum and iron electrodes of size 13 cm × 0.7 cm with an immersion depth of 0.72 cm, the number of electrodes used were two: the anode (iron) and the cathode (aluminium) and distance of 3.5 cm between them with a direct current (DC) power supply. The electrocoagulation cell has a working volume of 500 ml. The current was regulated at a constant voltage of 220 V. The appropriate current was passed into 500 mL of the wastewater. Before each experiment, the pH of the wastewater was adjusted with 0.1 M HCl or NaOH solution. A constant pH of 8 was used in the study. A drop of NaCl was added to the volume to introduce charge to the wastewater for a known charge time. The contents were then stirred for a known time (that is, flocculation time) and allowed to settle. After the elapsed settling time, the samples were withdrawn from a depth of 2cm using syringe and were measured using the turbidity meter. Before each run, the electrodes were cleaned thoroughly to remove any surface grease or solid residues. The percentage turbidity removal (%R) was calculated using Eq. (1):

$$\%R = \frac{TUR_i - TUR_f}{TUR_i} \times 100 \qquad (1)$$

Where TUR_i is the initial turbidity and TUR_f is the final turbidity.

2.3 Design of Experiment (DOE)

The optimization of the removal of turbidity from AW using Fe-Al electrodes was done using the Box-Benhken Design (BBD). Three important factors such as current, charge time (electrolysis time) and settling time were used as the independent variables where their combined effects were examined while the percentage removal of the turbidity was the dependent variables. This was done to determine the best conditions for the optimum removal of turbidity from the wastewater. The BBD involves varying the independent variable at three different levels (-1, 0, +1). The experimental range and levels of the independent variables are presented in table 1. In this work, a set of 16 experiments were performed at random to avoid systematic error using table 2. The centre point's replicates verify the changes in the middle of the plan and measure the degree of precision while the other points verify the nonlinear suspected curvature (Menkiti et al. 2011). The data acquired were fitted to the empirical second-order polynomial regression model given as Eq. (2) (Tek et al. 2015):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^{\,2} + \sum_{i=1}^{k-1} \sum_{j}^{k} \beta_{ij} x_i x_j + \Sigma \qquad (2)$$

where Y is response (% turbidity removal); β_O,β_i (i = 1, 2,…..k) and β_{ij} (i = 1, 2,…k; j = 1, 2,…k) are the model coefficients; X_i and X_j are the coded independent variables.

Also, the adequacy of the model was checked using the coefficient of determination (R^2), adjusted R^2 and predicted R^2 values. The interactive effects of the independent (process) variables on the dependent variable (response) were examined using the analysis of variance (ANOVA).

Table 1: Experimental range and levels of independent variables used in this study.

Independent factors	Low Level (-)	Medium Level (0)	High Level (+)
Charge time (min)	8	11.5	15
Settling time (min)	10	20	30
Current (A)	1	1.75	2.5

Table 2: Box-Behnken Design of the experiment in terms of coded and real values.

Run	Charge time (min)		Settling time (min)		Current (A)	
	Coded	Real	Coded	Real	Coded	Real
1	-1	8	-1	10	0	1.75
2	+1	15	-1	10	0	1.75
3	-1	8	+1	30	0	1.75
4	+1	15	+1	30	0	1.75
5	-1	8	0	20	-1	1
6	+1	15	0	20	-1	1
7	-1	8	0	20	+1	2.5
8	+1	15	0	20	+1	2.5
9	0	11.5	-1	10	-1	1
10	0	11.5	+1	30	-1	1
11	0	11.5	-1	10	+1	2.5
12	0	11.5	+1	30	+1	2.5
13	0	11.5	0	20	0	1.75
14	0	11.5	0	20	0	1.75
15	0	11.5	0	20	0	1.75
16	0	11.5	0	20	0	1.75

3.0 Results and Discussions

3.1 Characterization results of aquaculture wastewater

The physicochemical results obtained for aquaculture wastewater (AW) characterization before and after treatment is presented in table 3. The decrease in the turbidity of the effluent after treatment indicated in table 3 confirms the efficiency of the electrocoagulation-flocculation (ECF) process in the treatment of AW because the primary purpose of this treatment is the removal of turbidity. Since turbidity is a cloudy appearance of water caused by small particles suspended hence a corresponding decrease in the total solids (TS) and total suspended solids (TSS) present in the effluent also occurred. The effluent possessed very high biochemical oxygen demand (BOD), chemical oxygen demand (COD), and total oxygen carbon (TOC) before treatment. The reduction in these parameters confirmed the efficiency of the ECF process in achieving organic load reduction (Nur & Zawawi 2012). Other parameters such as nitrate, phosphate, iron, and conductivity were also reduced. After the ECF treatment, the wastewater parameters met the standard set by WHO (2002) before discharge (table 3).

Table 3: Characterization of aquaculture wastewater before and after electrocoagulation-flocculation.

Parameter	Unit	Before Electrocoagulation	After Electrocoagulation	WHO standard, 2002
pH		6.54	8.72	6.5-9.0
Conductivity	µS/cm	365	306	1000
Turbidity	NTU	328	45	50
TS	mg/l	596	42	500
TSS	mg/l	216	17	-
Color	-	Lemon green	Colorless	-
BOD	mg/l	340	0.03	3.0
COD	mg/l	700	40	250
TOC	mg/l	477	24	-
Nitrate	mg/l	167	18	50
Phosphate	mg/l	62	33	-
Iron	mg/l	0.425	0.19	0.1-1.0
Odour	-	Positive	Negative	-

3.2 Model fitting and statistical analysis for the treatment of aquaculture wastewater using Fe-Al electrodes

The actual experimental response and the predicted response from the mathematical equation is given in table 4 and figure 1 where it is seen that there is a close correlation between the actual experimental response and the predicted response. The predicted versus the actual plots in figure 1 was used to check whether the points will follow a straight line which can then be concluded that the residuals follow a normal distribution. From figure 2, it can be seen that the points were closely distributed to the straight line of the plot, this confirms a good relationship between the experimental values and the predicted values of the response though some small scatter like an “S” shape is always expected. The normal probability plot is used to examine the normality distribution of the residuals (Ghanim 2014). Figure 3 shows that great deviance from normality was not seen in the normal probability plots of the residuals. These plots also confirm that the selected model was adequate in predicting the response variables in the experimental values and that treatment of the AW using the electrocoagulation process was very effective.

The test for adequacy of the regression models, the significance of individual model coefficients and the lack of fit test were performed using the same statistical package. The statistical analysis of variance (ANOVA) was carried out to determine the significance of the fitness of the selected quadratic model as well as the significance of the individual terms and their interaction on the chosen response. From the ANOVA in table 5. The model Fisher’s F-value of 117.02 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of “Prob > F” less than 0.0500 implies that the model terms are significant at a 95% confidence level. The p-value (probability of error value) is used to check the significance of each regression coefficient and the interactions between the test variables (Ajemba & Onukwuli 2012; Shrivastava et al. 2008). The larger the magnitude of the F-test value, the smaller the magnitude of p-values and the higher the significance of corresponding coefficient (Silva et al. 2011). Considering the F-values, the settling time was observed to have the greatest effect on the process with F-value of 339.73. The greatest interactive effect is between charge time and current (F-value = 15.12). P-values less than 0.05 shows the model terms are significant; values greater than 0.100

indicate the model terms are not significant. The lack of fit F-values of 377.57 implies the lack of fit is significant relative to the pure error.

Table 4: Experimental and predicted responses for turbidity removal (%) from aquaculture wastewater using Fe-Al electrodes.

Run	Charge time (min)	Settling time (min)	Current (A)	Predicted value (%)	Experimental value (%)
1	15	20	2.5	85.12	84.74
2	15	30	1.75	88.1	87.96
3	11.5	20	1.75	85.83	85.85
4	15	20	1	81.51	84.40
5	8	20	2.5	76.46	80.57
6	8	20	1	75.57	75.95
7	11.5	10	2.5	84.8	84.55
8	11.5	30	2.5	91.52	92.04
9	8	30	1.75	78.39	80.76
10	8	10	1.75	74.34	74.48
11	11.5	20	1.75	85.83	85.85
12	15	10	1.75	79.27	79.90
13	11.5	10	1	82.92	82.40
14	11.5	30	1	89.01	89.26
15	11.5	20	1.75	85.83	85.85
16	11.5	20	1.75	85.91	85.85

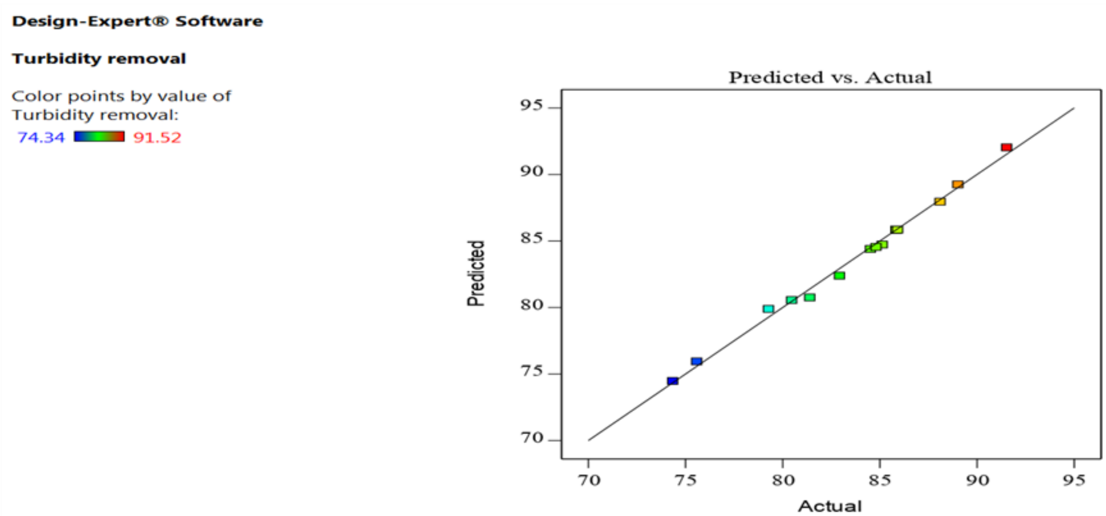

Figure 1: The plot of the predicted values versus the observed values for turbidity removal (%) using Fe-Al electrodes.

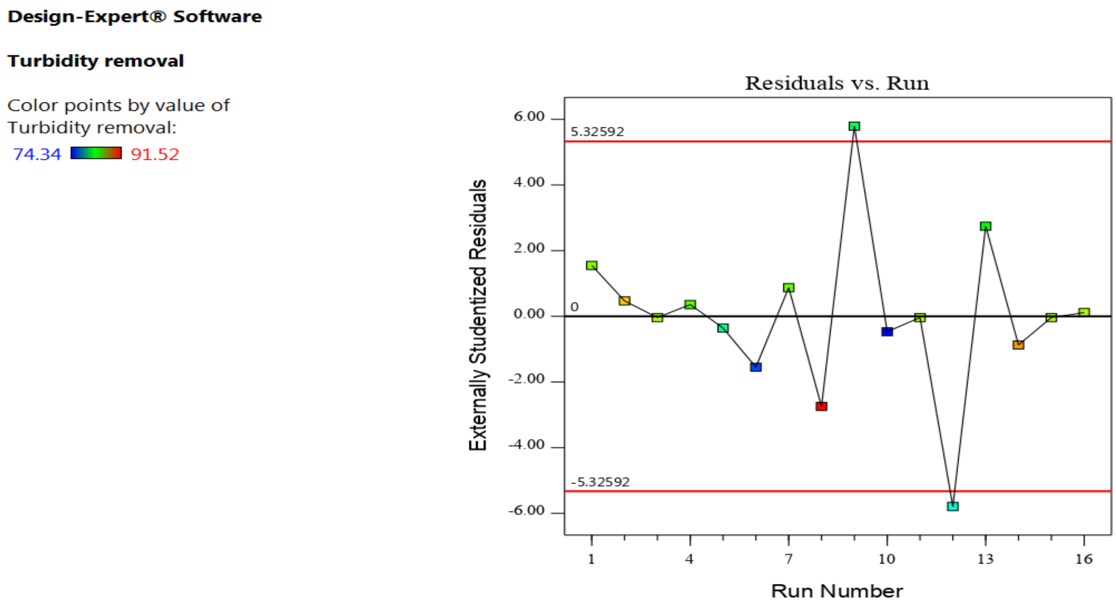

Figure 2: Residual versus run plot for turbidity removal (%) using Fe-Al electrodes.

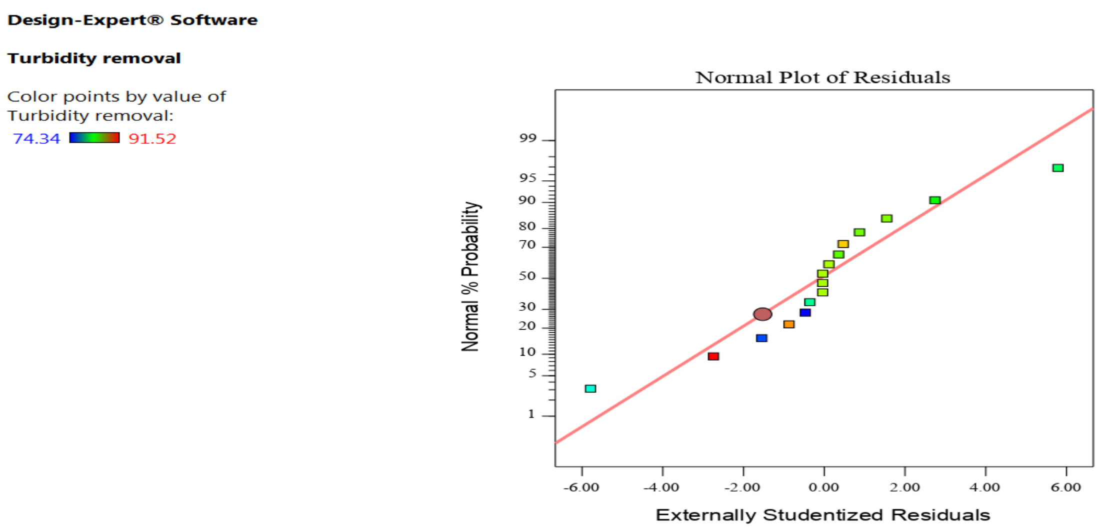

Figure 3: The studentized residuals and normal % of probability residuals for turbidity removal (%) using Fe-Al electrodes.

There is only a 0.02% chance that a Lack of Fit F-value this large could occur due to noise. The coefficient of regression (R^2) being a degree of the goodness of fit to the model was used to validate the fitness of the model equation. From the model summary statistics, R^2 has a high value of 0.9943 showing that 99.43% of the variability in the response can be explained by the model. This implies that the prediction of experimental data is quite satisfactory. The predicted R^2 of 0.9096 is in reasonable agreement with the adjusted R^2 of 0.9858. Adequate precision measures the signal to noise ratio and a ratio greater than 4 is desirable (Ahmadi et al. 2018; Igwegbe et al. 2019). A ratio of 40.370 indicates an adequate signal. This model can be used to navigate the design space.

The quadratic model was applied to reveal the mathematical correlation between the independent variables (charge time, settling time and current) and dependent variable (turbidity removal). The quadratic model equation obtained for turbidity removal from AW using Fe-Al electrodes is given as Eq. (3):

$$Y = 85.85 + 3.16A + 3.59B + 1.24C + 0.4450AB - 1.07AC$$
$$+0.1575BC - 5.36A^2 + 0.2862B^2 + 0.9263C^2 \quad (3)$$

Where A is the charge time, B is the settling time, C is the current and Y is the % turbidity removal.
According to Srim & Sudha (2012) in a regression equation when an independent variable has a positive sign, it means that an increase in the variables, charge time, settling time and current will cause an increase in the turbidity removal efficiency. A, B, C, AC, A^2, and C^2 are the significant model terms. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. Therefore eliminating the insignificant, the final model equation becomes:

$$Y = 85.85 + 3.16A + 3.59B + 1.24C - 1.07AC - 5.36A^2 + 0.9263C^2 \qquad (4)$$

Table 5: Analysis of variance (ANOVA) for response surface quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	318.95	9	35.44	117.02	< 0.0001	significant
A-Charge time	79.63	1	79.63	262.94	< 0.0001	
B-Settling time	102.89	1	102.89	339.73	< 0.0001	
C-Current	12.23	1	12.23	40.37	0.0007	
AB	0.7921	1	0.7921	2.62	0.1570	
AC	4.58	1	4.58	15.12	0.0081	
BC	0.0992	1	0.0992	0.3276	0.5878	
A^2	114.97	1	114.97	379.63	< 0.0001	
B^2	0.3278	1	0.3278	1.08	0.3383	
C^2	3.43	1	3.43	11.33	0.0151	
Residual	1.82	6	0.3029			
Lack of Fit	1.81	3	0.6041	377.57	0.0002	significant
Pure Error	0.0048	3	0.0016			
Cor Total	320.77	15				

Standard deviation = 0.5503, R^2 = 0.9943, Adj. R^2 = 0.9858, Mean = 83.78, C.V. % = 0.6569, Pred. R^2 = 0.9096, PRESS = 29.01, Adequate precision = 40.3702

3.3 Three Dimensional (3D) plots for the electrocoagulation process

The interactive effects of the independent variables on turbidity removal were studied by plotting the two-dimensional (2D) contour plots and three-dimensional (3D) surface curves against any two independent variables while keeping the others at their central (0) level. These plots are the graphical representations of the interactive effects of any two variables. The response surface curves were plotted to understand the interaction of the variables and to determine the optimum level of each variable for maximum response. The 2D contour and 3D surface plots of the response (turbidity removal) from the interactions between the variables are shown in figures 4 - 6. The elliptical shape of the curve indicates good interaction between the variables and the circular shape indicates no interaction between the variables. Figure 4 gives the interactive effect of charge time and settling time on the turbidity removal. The impact of the interaction is influenced mainly by the charge time as seen in figure 5. Figure 5 shows that the current applied increased the turbidity removal. This can be ascribed to the fact that at high current, the amount of iron oxidised was increased, resulting in a greater amount of precipitate for the removal of pollutants (Nwabanne et al. 2018; Holt et al. 2002). This could be to the fact that an increase in charge time results to an increased supply of positive metal ions into the solution which neutralizes the colloidal particles and leads to increase in hydroxide flocs and consequently results in the reduction of turbidity (Uzoh & Nwabanne 2014). The interaction between the current and the settling time also increased the efficiency of removal simultaneously.

3.4 Numerical optimization for the electrocoagulation process

Numerical optimization was implemented via the Design expert software (Stat-Ease, 11 version) to define the optimum conditions for adsorption. Table 6 shows the constraints for the optimization of aquaculture wastewater treatment using electrocoagulation. The highest turbidity removal obtained in this process is 91.84 % at charge time of 11.970 min, settling time of 29.994 min and current density of 2.389 A. The validity of the results predicted by the regression model was confirmed by carrying out experiments using these optimal conditions. The excellent correlation between the predicted (91.84 %) and measured value (91.67 %) from these experiments indicates the validity of the response model.

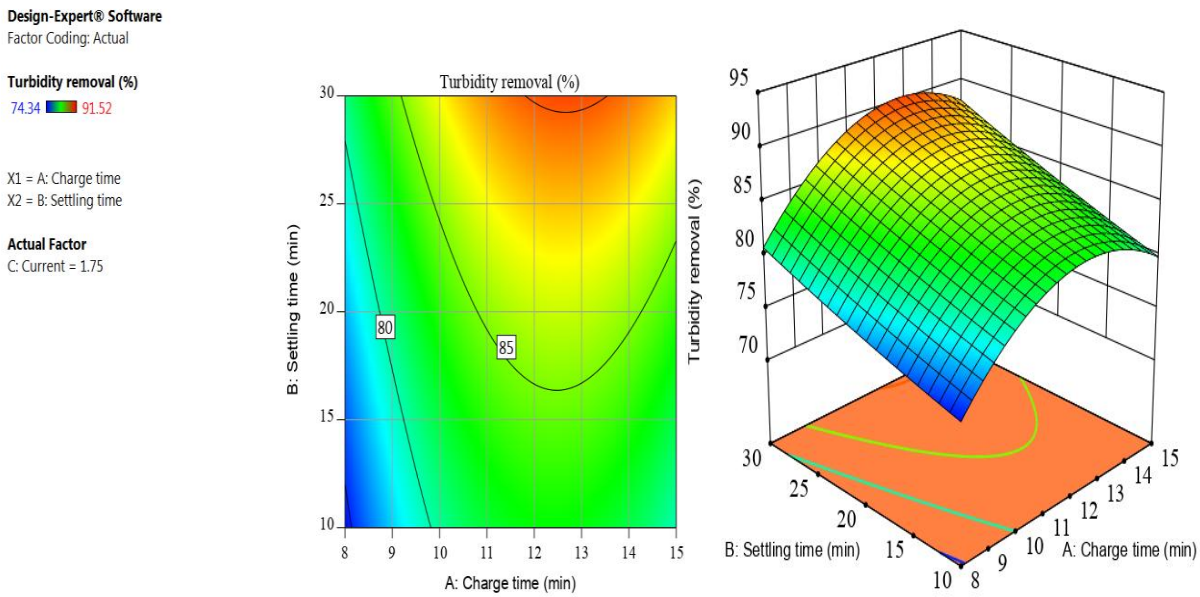

Figure 4: 2D contour and 3D surface plots of the interactive effect between the charge time and the settling time at current of 1.75 A.

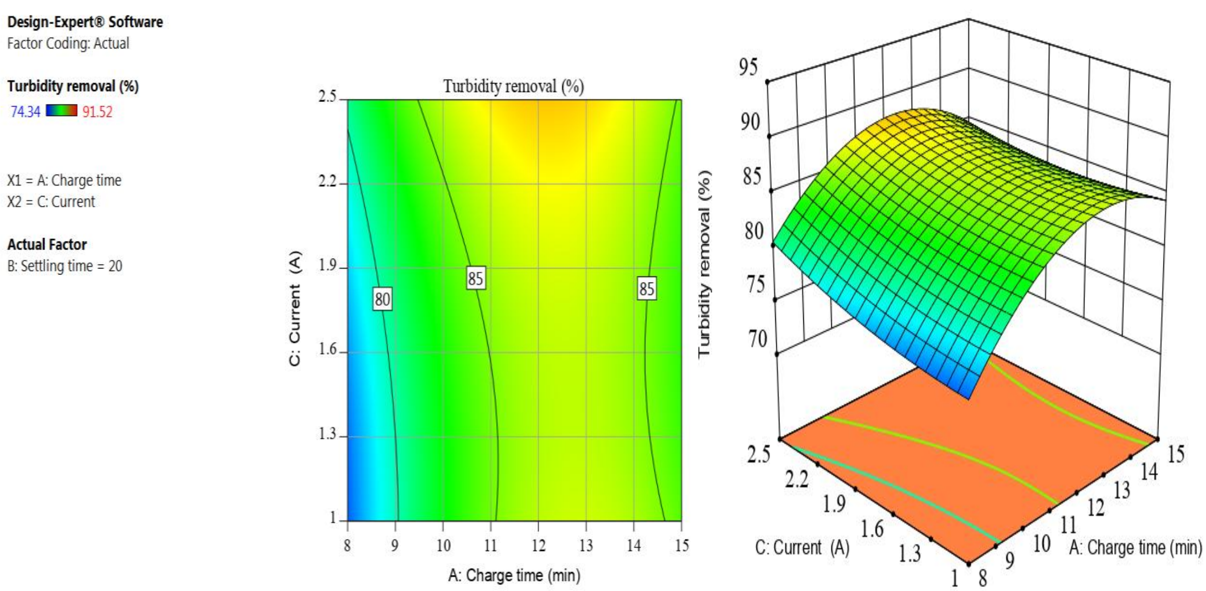

Figure 5: 2D contour and 3D surface plots of the interactive effect between the charge time and current at settling time of 20 min.

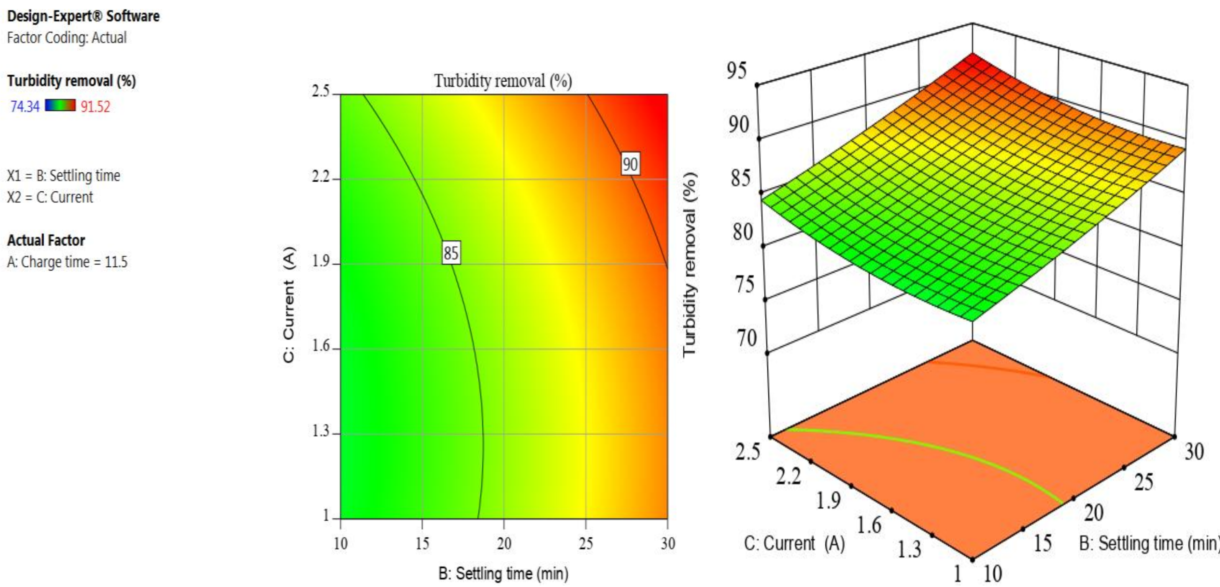

Figure 6: 2D contour and 3D surface plots of the interactive effect between settling time and current at charge time of 11.5 min.

Table 6: Constraints for the optimization of aquaculture wastewater treatment using Fe-Al.

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A:charge time (min)	is in range	8	15	1	1	3
B:settling time (min)	minimize	10	30	1	1	3
C:current (A)	is in range	1	2.5	1	1	3
Turbidity removal (%)	maximize	74.34	91.52	1	1	5

4.0 Conclusion

In this study, the treatment of aquaculture wastewater by electrocoagulation-flocculation technique using iron-aluminium electrodes was investigated using the response surface methodology (RSM) via Box-Behnken design (BBD). Optimal conditions of charge time of 11.970 min, settling time of 29.994 min and current density of 2.389 A were obtained at constant pH of 8 and temperature of 30 0C using the response surface methodology (RSM) via Box-Behnken design (BBD), which gave turbidity removal of 91.84 %. The correlation between the predicted value (91.84 %) and measured value (91.67 %) confirms the validity of the results predicted by the regression model. The results have demonstrated that the electrocoagulation technique using iron-aluminium electrodes is an effective method in the treatment of aquaculture wastewater by reducing the level of turbidity. Also, the developed model for the process was statistically significant which implies that the RSM can be employed to model the process.

5.0 Recommendation

It is recommended that the electrocoagulation-flocculation treatment process be utilized for the treatment of aquaculture wastewater. The mathematical model obtained from the statistical analysis can be employed to establish better conditions for the treatment of aquaculture wastewater using iron-aluminum electrodes.

Acknowledgements

The authors are grateful to the Department of Chemical Engineering, Nnamdi Azikiwe University, Awka, Nigeria for their support of this study. Also, we wish to thank the editors and reviewers for their helpful comments and careful review of this manuscript.

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