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Treatment of Vegetable Oil Effluent Using Factorial Design Experiment for Pleurotus Tuberregium Sclerotium Coagulant

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Abstract This research work presents results on the treatment of vegetable oil effluent using factorial design experiment for Pleurotus Tuberregium Sclerotium Coagulant (PTSC). Optimization studies on physical factors such rapid mix velocity gradient, contact time, pH and dosage of coagulant was also examined. The coagulation performance of vegetable oil effluent was investigated at room temperature using two coagulants, PTSC and alum. The experiments were carried out using the standard jar test method at varying pH and coagulant doses and the bio coagulant processing was based on the work reported by Adebowale and Adebowale (2007). More so, the coagulation reaction rate constant, K, the order of reaction ($-r_A$), coag-flocculation parameters and the distribution of particles were also determined. Turbidity measurement was employed using the nephelometric (Turbidiometric) standard method. PTSC as coagulant the optimum conditions of settling time of 8minutes, dosage of 0.2g and pH of 8.0 reduced the concentration of the particle from 56.4 to 18.5649mg/l and desirability of 0.727, whereas using alum reduced the concentration of the effluent to 16.0494mg/l. The results obtained confirmed that the theory of fast coagulation holds for the treatment of vegetable oil effluent using the coagulant investigated and the conditions of the experiment.

Keywords vegetable oil, Factorial Design, Response Surface Method, PTSC, Optimization, R^2

Introduction

Background of Study: Many coagulants are widely used in conventional water-treatment process for turbidity removal during potable water production. These coagulants may be classified as inorganic, synthetic organic polymer, and natural polymer. These coagulants are used for various purposes depending on the chemical characteristics of the water to be treated. Aluminium salts are by far the most widely used coagulant in water and wastewater treatment. However, recent studies have pointed out several serious drawbacks in using alum salts such as Alzheimer's disease and similar health related problems associated with residual alum in treated waters, beside production of large sludge volumes. There is also the problem of reaction of alum with natural alkalinity present in water leading to reduction of pH and a low efficiency in coagulation of cold waters [1]. Ongoing studies to produce more effective aluminium coagulants, such as polyaluminum chloride (PAC), have not corrected all the drawbacks mentioned above. Ferric salts and synthetic polymers have also been used as coagulants but with limited success because of similar disadvantages manifested in the use of aluminium salts. In addition to these problems, chemicals used for water treatment in developing countries constitute a high percentage of annual running expenditure of water treatment companies. The costs of these chemicals have also been increasing at an alarming rate because local manufacturing companies cannot cope with the demand for these chemicals in other industrial applications. Therefore, the shortfall has to be imported with scarce

convertible foreign currency. These problems force many water treatment companies to resort to under-dosing of chemicals so as to meet the increasing water demand. The result is the supply of poor quality water, especially during the rainy season, when suspended solids concentration and other pollutants in surface water are very high [2].

On the other hand, naturally occurring coagulants are usually presumed safe for human health. Some studies on natural coagulants have been carried out and various natural coagulants have been produced or extracted from microorganisms, animals, or plants [3]. In the course of this research, Pleurotus Tuberregium Sclerotium Coagulant (PTSC) was used as coagulant aid.

In recent time, the vegetable oil industry develop rapidly in Nigeria and other countries of the world with the cultivation of appropriate agricultural crops such as, especially, sunflower, cotton and maize. With this growth, effluent generation, treatment and subsequent disposal come into play.

Previously, effluents from vegetable oil industries were used to be discharged directly into the soil or ground water. But, due to the emergence of environmental consciousness, the Pollution Control Boards have become strict and imposed very stringent norms. The scarcity of water is also another incentive for recovering pure water from effluents.

The treatment of an effluent by the conventional methods like aerobic or anaerobic digestion, the ratio of BOD to COD should be >0.6. However, an effluent from the vegetable oil usually has its BOD/COD ratio around 0.2 which could cause destruction to micro organisms useful for the biodegradation. Methods such as multiple effect evaporation or incineration are highly energy intensive and hence, very expensive. These disadvantages emphasize the need for further research using novel separation methods like coagulation, flocculation, sedimentation, filtration and disinfection [5].

Most importantly, the aforementioned methods are often inappropriate in the developing countries because of the high cost and low availability of chemical coagulants. These factors led to the idea of using natural plants and animal materials as coagulant in clarifying vegetable oil effluent and turbid water. Among all the plant materials that have been tested over the years Pleurotus Tuberregium Sclerotium Coagulant (PTSC) has shown to be effective primary coagulant with potential usage on a large scale, in tropical developing countries and can be compared to those of alum (conventional chemical coagulant).

Jar tests are widely used to determine the optimum dosages for treatment. This laboratory test attempts to simulate the full-scale coagulation-flocculation process and can be conducted for a wide range of condition. The interpretation of result involves visual and chemical testing of the clarified water or effluent.

Objectives of the Study

In conducting a comparative coagulation study on the treatment of vegetable oil effluent using PTSC and alum in an experiment known as jar test.

Factorial Design Experiment for Pleurotus Tuberregium

For this design, 2 levels full factorial design was used. Since we have three factors, we have total of 2^3 which gave 8 experiments each. Factorial design studies the effect of process factors to obtain the significant ones.

Table 1: Design Matrix for Pleurotus Tuberregium

Run Order	Contact time (min)	Dosage (grams)	pH	Concentration (mg/ml)
1	4	0.2	4	28.2
2	8	0.4	4	39.95
3	8	0.4	8	37.6
4	8	0.2	4	23.5
5	4	0.2	8	28.2
6	4	0.4	8	51.7
7	4	0.4	4	51.7
8	8	0.2	8	18.8

The half normal plot was used to select the significant factors that were included in the model.

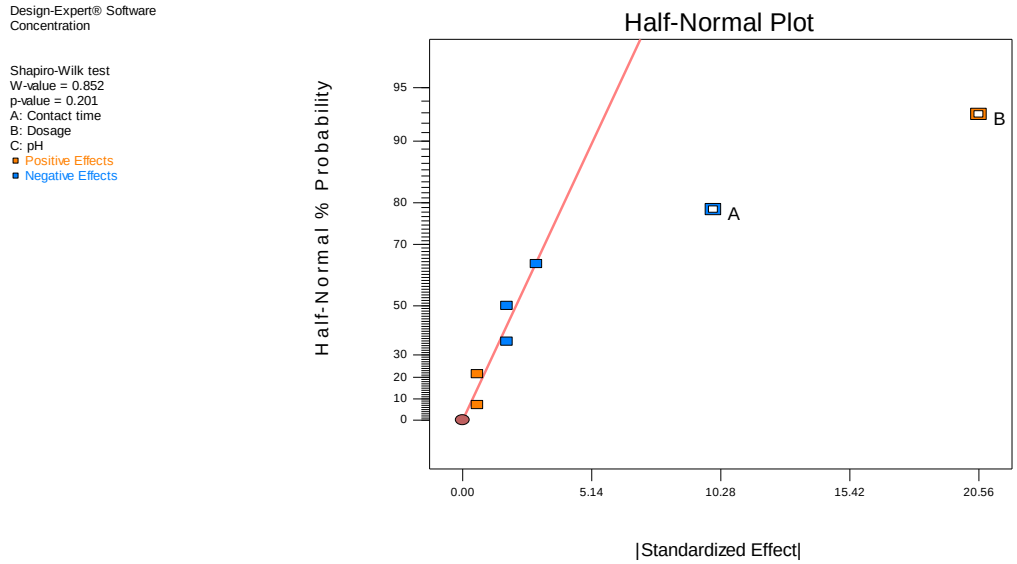

Figure 1: *Half Normal Plot for pleurotus tuberregium*

From the plot, it shows that effect of B which is dosage and effect of A which is contact time is significant.
Pareto chart was used to confirm the selection done using half normal plot. Any effect above t-value is significant, and any one below t-value is not significant. It also shows the magnitude of the effects: as can be seen, effect B has highest effect, followed by effect A

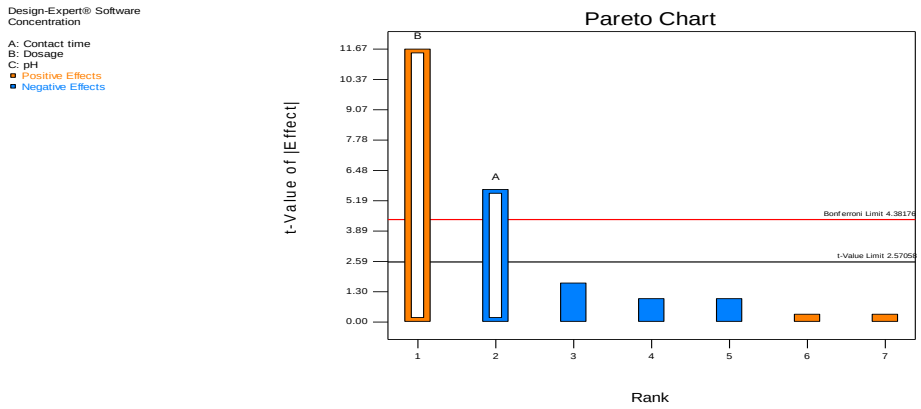

Figure 2: *Pareto Chart for pleurotus tuberregium*

The ANOVA partitions the effects separately and shows whether or not they are significant.

Table 2: Factorial design model using Anova for pleurotus tuberregium slerotium Sum of Mean F p-value

Source	***Sum of Squares***	***Df***	***Mean Square***	***F Value***	***p-value Prob > F***	
Model	1051.35	3	350.45	56.41	0.0010	significant
A-Contact time	199.50	1	199.50	32.11	0.0048	
B-Dosage	845.63	1	845.63	136.11	0.0003	
C-pH	6.21	1	6.21	1.00	0.3739	
Residual	24.85	4	6.21			
Cor Total	1076.20	7				

The Model F-value of 56.41 implies the model is significant. There is only a 0.10% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant.
In this case A, B are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. R-squared of 0.9769, Adj-R-Squared of 0.9596 and Pred-R-squared of 0.9076 The "Pred R-Squared" of 0.9076 is in reasonable agreement with the "Adj R-Squared" of 0.9596.

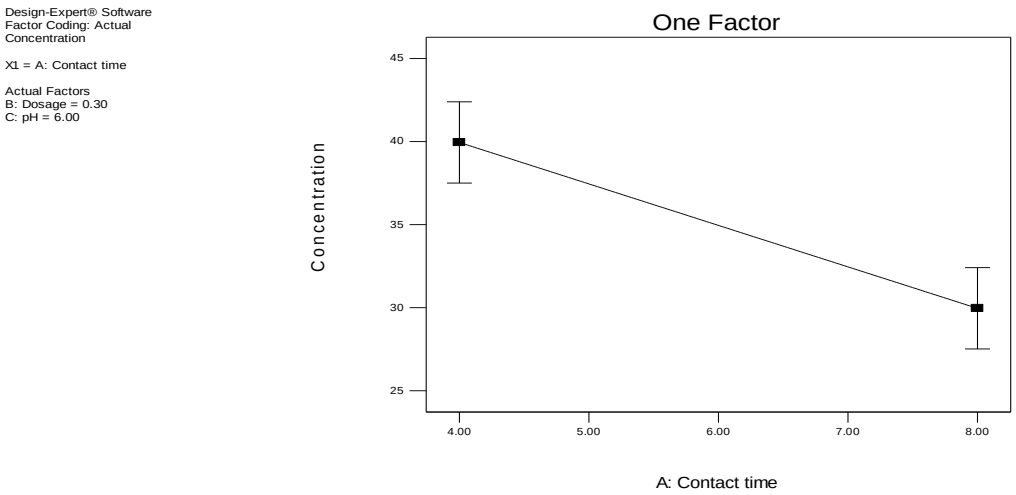

Figure 3: *Model graph of concentration Vs contact time for PTSC*

For the effect of Contact time, it has a negative slope, which showed that as the contact time was increased, the concentration decreased.

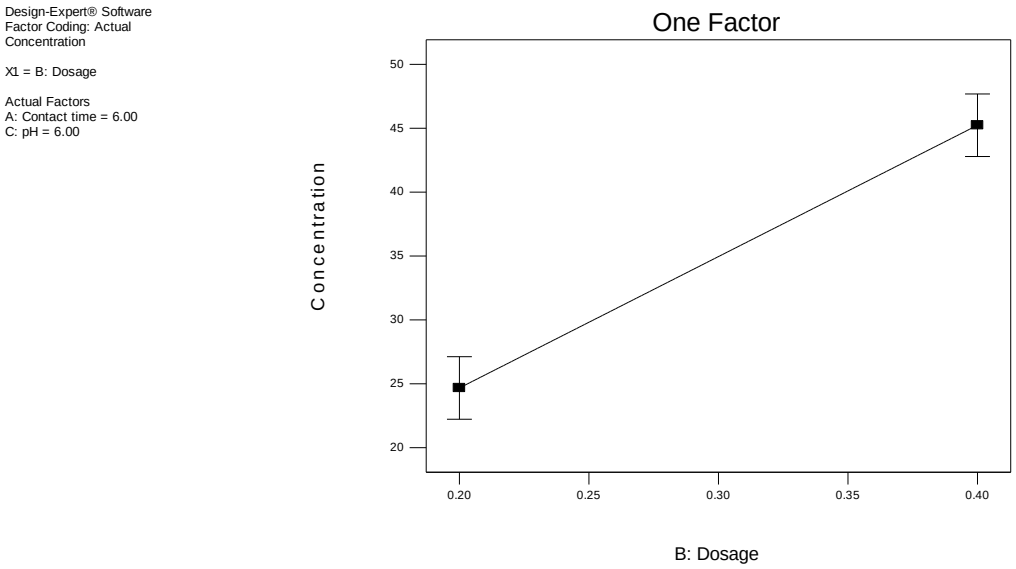

Figure 4: *Model graph of concentration Vs dosage for PTSC*

For the effect of dosage, it had a positive graph which showed that as the dosage was increased, the concentration equally increased.

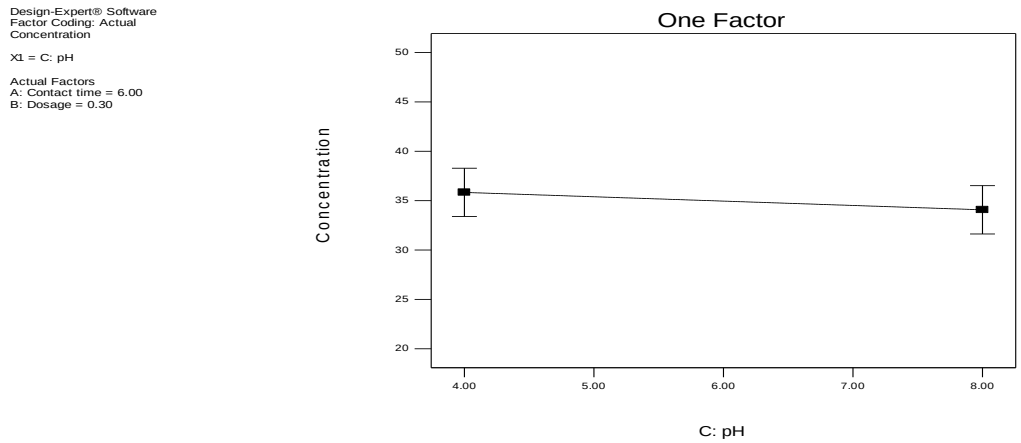

Figure 5: *Model graph of concentration Vs pH for PTSC*

For the effect of pH, it had no effect as can be seen from the graph, it is horizontal.

RESPONSE SURFACE METHODOLOGY(RSM)

The response surface method is used to model and optimize a process. The type of RSM used was Central composite design (CCD). This is made up of factorial points (2^n), axial points (2n) and center points which is six, where n stands for the number of factors involved. This gave a total of 20 experiments.

RSM FOR PLEUROTUS TUBERREGIUM

Table 3: RSM design matrix for PTSC

Run Order	Contact time (min)	Dosage (grams)	pH	Concentration (mg/ml)
1	4	0.4	8	51.7
2	6	0.3	6	37.6
3	4	0.2	4	28.2
4	8	0.2	8	21.15
5	4	0.4	4	51.7
6	6	0.3	2	42.3
7	6	0.3	10	28.2
8	10	0.3	6	28.2
9	6	0.1	6	18.8
10	8	0.4	8	37.6
11	8	0.2	4	23.5
12	8	0.4	4	39.96
13	6	0.3	6	37.6
14	6	0.3	6	37.6
15	6	0.3	6	37.6
16	6	0.5	6	65.8
17	6	0.3	6	37.6
18	8	0.3	6	37.6
19	6	0.3	6	56.4
20	8	0.2	8	28.2

ANOVA Response 1: Concentration (mg/l)

Table 4: ANOVA Response Surface Linear Model for PTSC

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
Model	2510.12	3	836.71	56.01	< 0.0001	significant
A-Contact time	552.13	1	552.13	*36.96*	*< 0.0001*	
B-Dosage	1890.29	1	1890.29	*126.53*	*< 0.0001*	
C-pH	67.69	1	67.69	*4.53*	*< 0.0001*	
Residual	239.04	16	14.94			
Lack of Fit	239.04	11	21.73			
Pure Error	0.000	5	0.000			
Cor Total	2749.15	19				

The Model F-value of 56.01 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. R-Squared of 0.9131, AdjR-Squared of 0.8967 and Pred R-Squared of 0.8341.

MATHEMATICAL MODEL for PTSC

The model equation is the mathematical representation of the process. It can be used to predict the results obtained from the experiments.

The coded equation can only be used for the prediction because the factors were assumed to be dimensionless while the equation in actual form cannot be used for prediction because the factors have been scaled to accommodate different units

Final Equation in Terms of Coded Factors:

Concentration (mg/l) = +37.37-5.87 * A+10.87 * B-2.06 * C

Final Equation in Terms of Actual Factors:

Concentration (mg/l) = +28.55113 - 2.93719 * Contact time + 108.69375 *Dosage -1.02844 * pH

Diagnostic Plots for PTSC

The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. Expect some moderate scatter even with normal data.

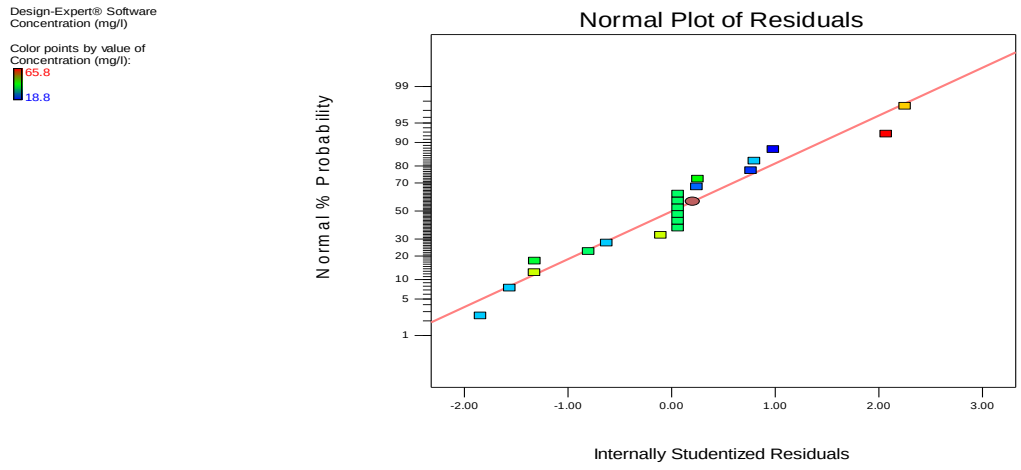

Figure 6: *Normal Plot of Residuals for PTSC*

The plot of the residuals versus the ascending predicted response values. It tests the assumption of constant variance. The plot should be a random scatter

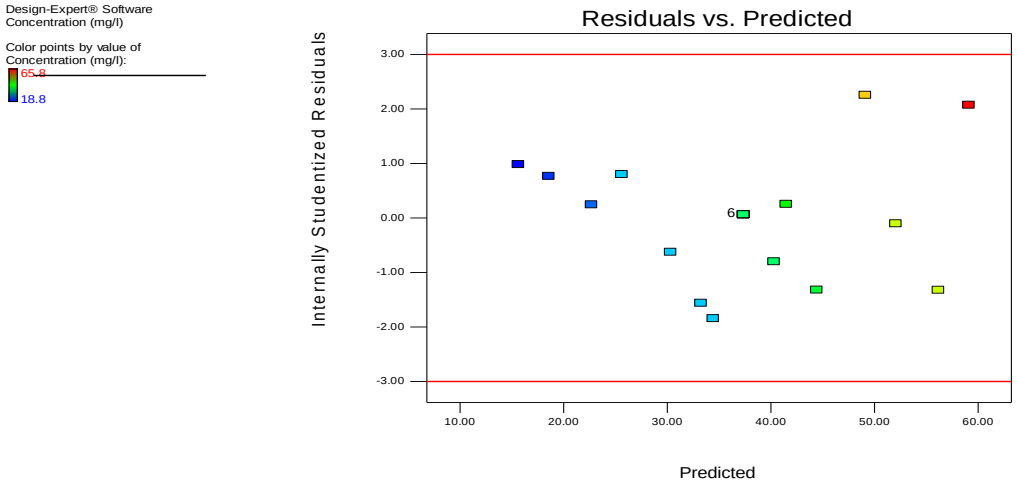

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Figure 7: *Residuals Vs Predicted Plot for PTSC*

The plot of the residuals versus the experimental run order allow you to check for lurking variables that may have influenced the response during the experiment. The plot shows a random scatter. Trends indicate a time-related variable lurking in the background.

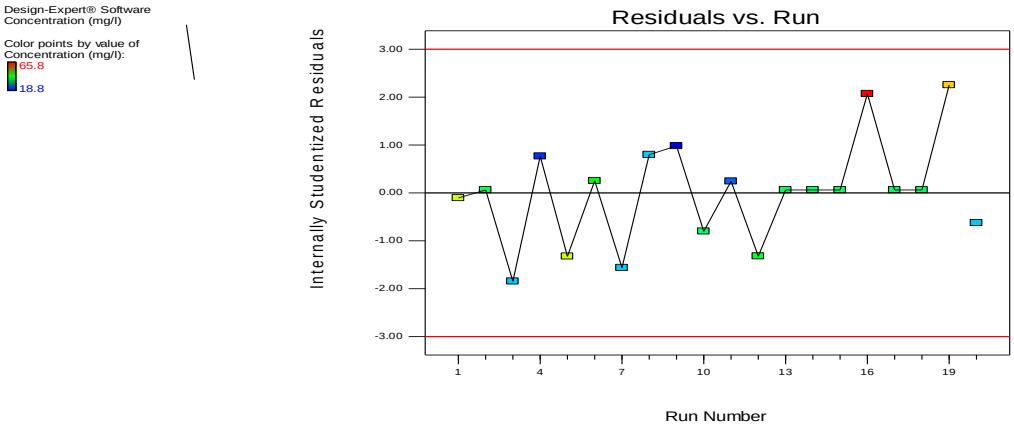

Figure 8: *Residuals vs Run for PTSC*

A graph of the actual response values versus the predicted response values. It helps you detect a value, or group of values, that are not easily predicted by the model. The data points show a split evenly by the 45 degree line.

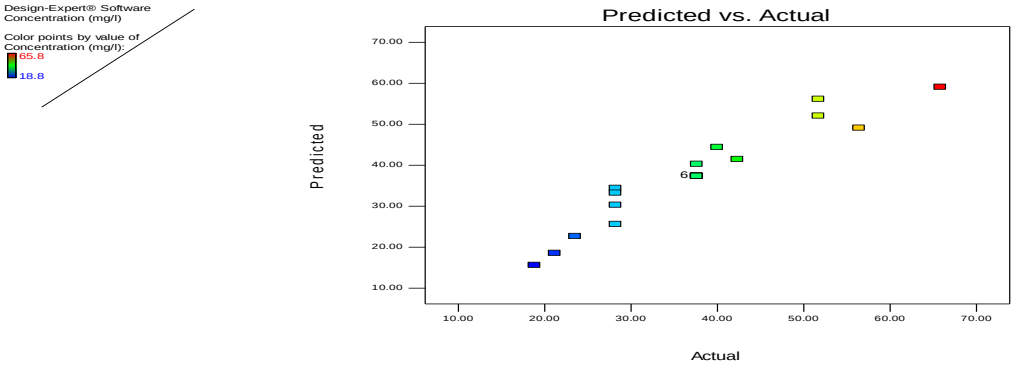

Figure 9: *Predicted Vs Actual for PTSC*

The diagnostic plots revealed no problems, meaning that the model equation can predict the process well.

Optimization for PTSC

Optimization was done to obtain the best conditions that will give the minimal concentration, therefore, our goal was to minimize. The optimal conditions are Contact time of 8minutes, dosage of 0.2 g and pH of 8.00 with concentration of 18.5649mg/l at a desirability of 0.727.

ANOVA Response Surface Quadratic Model for ALUM

Table 5: Analysis of variance table [Partial sum of squares - Type III] Sum of Mean F p-value

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
Model	169.47	9	18.83	1.78	0.1901	significant
A-Contact time	12.43	1	12.43	1.18	0.3034	
B-Dosage	22.09	1	22.09	2.09	0.1786	
C-pH	5.52	1	5.52	0.52	0.4861	
AB	8.527E-014	1	-8.527E-014	-8.08E-15	1.0000	
AC	-2.842E-014	1	-2.842E-014	-2.69E-15	1.0000	
BC	2.76	1	2.76	0.26	0.6201	
A^2	1.15	1	1.15	0.11	0.7484	
B^2	3.51	1	3.51	0.33	0.5767	
C^2	114.75	1	114.75	10.87	0.0081	
Residual	105.56	10	10.56			
Lack of Fit	105.56	5	21.11			
Pure Error	0.000	5	0.000			
Cor Total	275.02	19				

The "Model F-value" of 1.78 implies the model is not significant relative to the noise.There is a19.01 % chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant.

In this case C^2 are significant model terms.

R-Squared	0.6162
Adj R-Squared	0.2708
Pred R-Squared	-2.0653
Adeq Precision	5.370

Final Equation in Terms of Coded Factors:

Alum Concentration = +18.05-0.88 * A-1.18 * B+0.59 * C+0.000* A * B+0.000 * A * C+0.59* B * C+0.21* A^2-0.37* B^2-2.14 * C^2

Final Equation in Terms of Actual Factors:

Alum Concentration = +7.07670-1.08153* settling time-6.94318
* Dosage+5.82159* pH+1.09906E-013
* Contact time * Dosage+4.47476E-015
* Contact time * pH+2.93750* Dosage * pH
+0.05340* Contact time^2-37.38636* Dosage^2
-0.53409* pH^2

Diagnostic Plots for Alum

The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. Expect some moderate scatter even with normal data. Look only for definite patterns like an "S-shaped" curve,

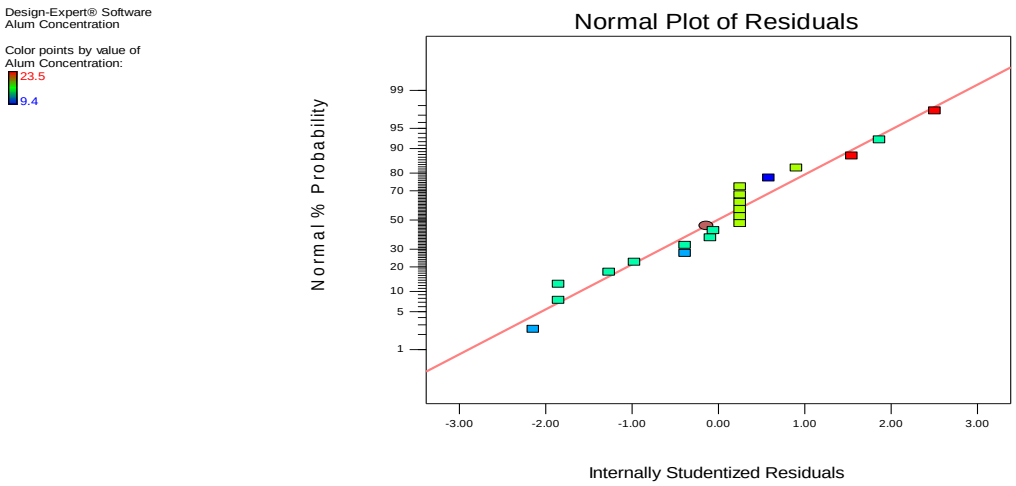

Figure 10: *Normal plot of residuals for alum*

This is a plot of the residuals versus the ascending predicted response values. It tests the assumption of constant variance. The plot is a random scatter.

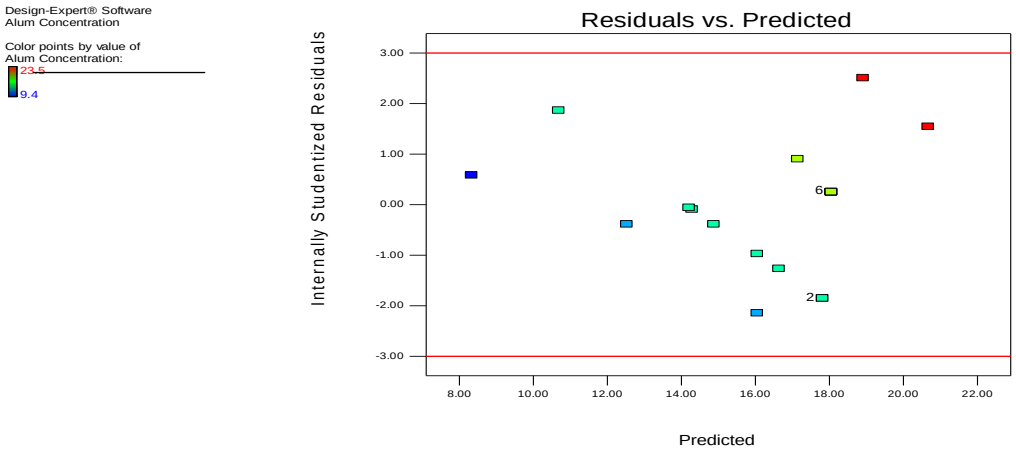

Figure 11: *Residuals vs predicted plot for alum*

Plot of the residuals verse experimental run order allow you to check for lurking variables that may have influenced the response during the experiment. The plot shows a random scatter.

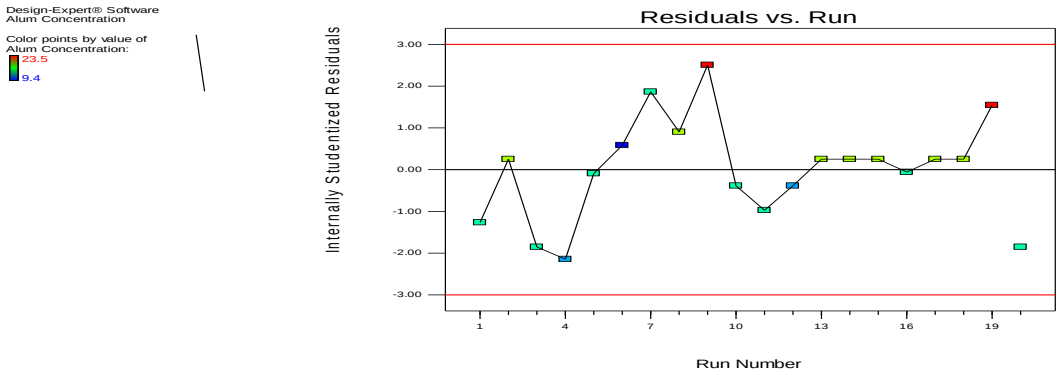

Figure 12: *Residuals vs Run for alum*

A graph of the actual response values versus the predicted response values. It helps you detect a value, or group of values, that are not easily predicted by the model. The data points show a split evenly by the 45 degree line.

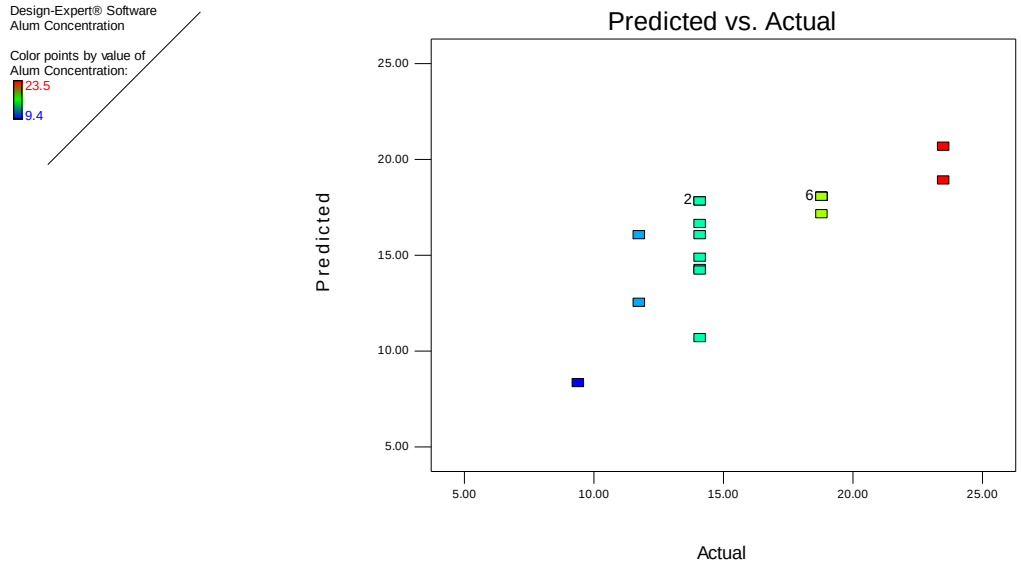

Figure 13: *Predicted vs Actual for alum*

The diagnostic plots revealed some abnormalities which showed that the model was not adequate in explaining the process well

Model Graph

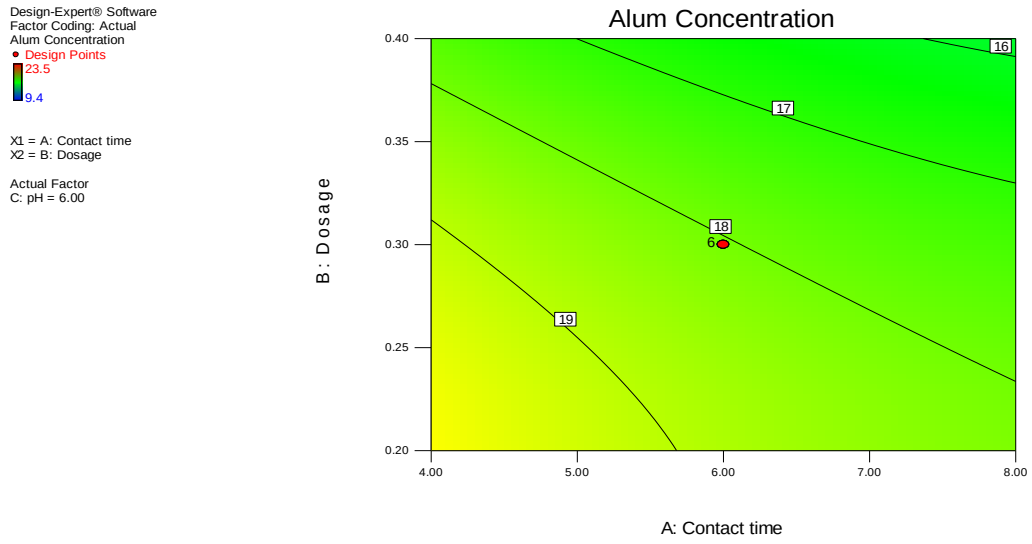

Figure 14: *Contour plot of interaction effect of dosage and contact time for alum*

The contour plot of interaction effect of dosage and contact time showed that increase in contact time with increase in dosage will decrease the concentration

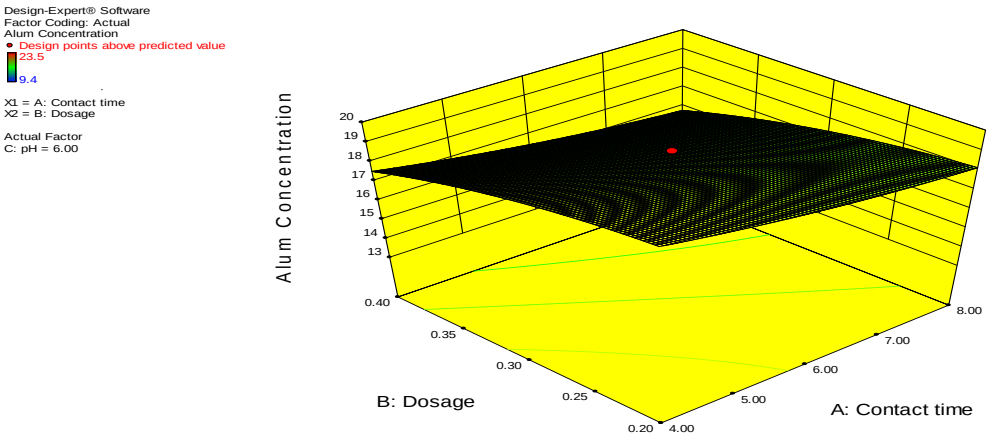

Figure 15: *3D surface plot for alum*

The 3D surface plot also gave the same interpretation with the contour plot, as well as the surface morphology. It showed that the surface is flat, meaning is a linear interaction effect.

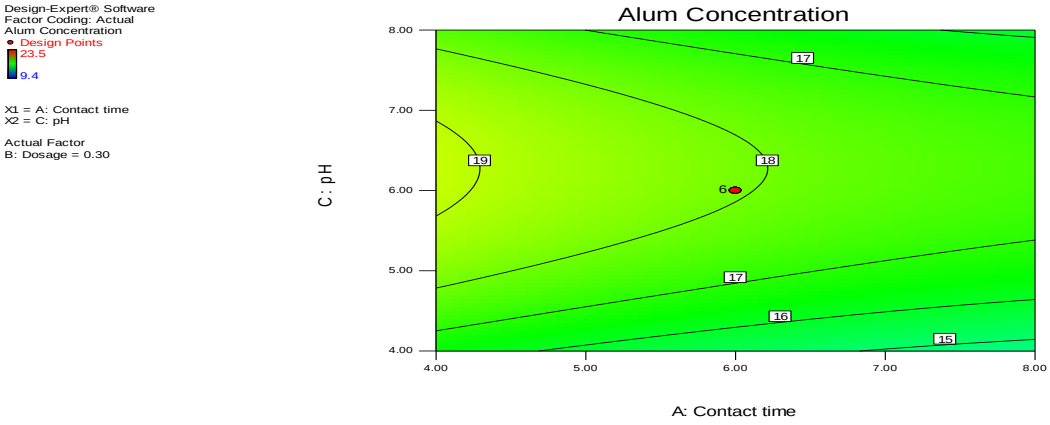

Figure 16: *Contour plot of the interaction effect of pH and contact time for alum*

The contour plot of the interaction effect of pH and contact time showed that as you increase the contact time, the concentration decrease at a particular region of pH. As you increase the pH, the concentration increases to a point that further increase in pH resulted to decrease in concentration at constant increase in contact time.

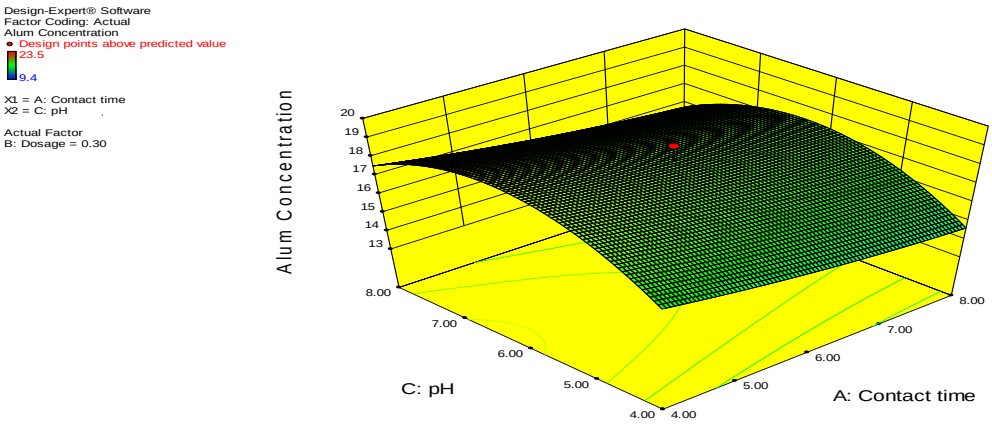

Figure 17: *3D surface plot for alum*

The 3D surface plot gave the same explanation with the contour plot and showed a quadratic surface.

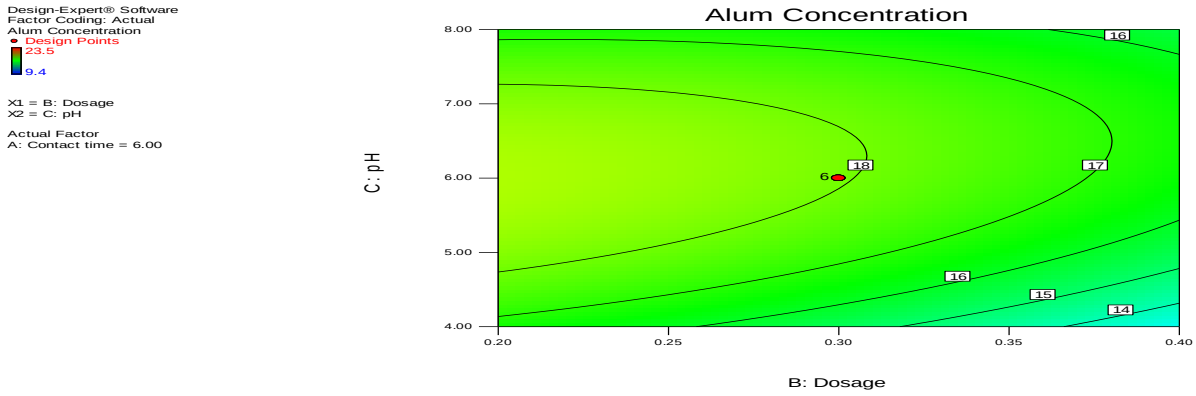

Figure 18: *Contour plot of the interaction effect of dosage with pH for alum*

The contour plot of the interaction effect of dosage with pH showed that concentration decreased as dosage was increased with increase in pH . Contact time of 8minutes, dosage of 0.2g and pH of 8 with concentration of 16.0494 at desirability of 0.727

Results and Discussion

The tables and figures below show results obtained from experiments conducted in order to achieve the desired result as proposed in the theoretical background and development.

Optimum Dosage

The column with the least turbidity was in each experiment for our kinetics analysis.

Table 6: The Characteristics of Wastewater from Vegetable Oil Factory

Parameter	Minimum	Maximum	Maximum
Temperature, ^{o}C	46.45	65.26	49.56
pH	7.43	9.56	8.52
Alkalinity, mg $CaCO_3$/L	800	1990	1680
Oil-grease, mg/l	540	7640	3030
COD, mg/l	5600	15300	9350
BOD5, mg/l	1050	10300	5260
Sulphate, mg/l	160	1080	1080
Total phosphorus, mg/l	216	556.20	378.56
TKN, mg/l	19.80	125.40	76.92
TSS, mg/l	3700	12800	7830
SS, mg/l	410	3240	1650
VSS, mg/l	4300	9680	5820
TDS, mg/l	4590	10200	6180
Flow rate, m^3 /day	308.60	321.80	315.88

TKN = total Kjeldahl nitrogen; TSS = total suspended solids;
SS = suspended solids; VSS = volatile suspended solids; TDS = total dissolved solids.

Table 7: Characteristics of Bio-Coagulant Precursor (Pleurotus Tuberregium Sclerotium)

Parameter	Value
Moisture content %	15
Ash content %	5
Fat content %	9
Crude protein content %	35.7
Crude fibre content %	15
Carbohydrate content %	20.3

Table 8: Coag-Flocculation Kinetic Parameter and Linear Regression Coefficient of PTSC at varying dosage and pH=2.0

Parameter	100 mg/l	200 mg/l	300 mg/I	400 mg/I	500 mg/I
α	2.0	2.0	2.0	2.0	2
R^2	0.957	0.978	0.916	0.985	0.947
K(l/mg.min)	0.002	0.002	0.001	0.001	0.001
No(mg/l)	43.5	76.9	55.6	125.0	100.0
$(\alpha_{cf})_{BR}$(l/mg.min)	0.004	0.004	0.002	0.002	0.002
$(-r_A)$	$0.002Nt^2$	$0.002Nt^2$	$0.001Nt^2$	$0.001Nt^2$	$0.001Nt^2$
$\tau_{1/2}$	11.49	7.50	17.98	8.0	10.0

Table 9: Coag-Flocculation Kinetic Parameter and Linear Regression Coefficient of PTSC at varying dosage and pH=4.0

Parameter	100 mg/l	200 mg/l	300 mg/I	400 mg/I	500 mg/I
α	2.0	2.0	2.0	2.0	2.0
R^2	0.800	0.598	0.923	0.835	0.996
K(l/mg.min)	0.001	0.004	0.003	0.004	0.003
N_o(mg/l)	27.02	27.02	62.50	52.63	83.33
$(\alpha_{cf})_{BR}$(l/mg.min)	0.002	0.008	0.006	0.008	0.006
$(-r_A)$	$0.001Nt^2$	$0.004Nt^2$	$0.003Nt^2$	$0.004Nt^2$	$0.003Nt^2$
$\tau_{1/2}$	37.0	92.52	53.33	47.0	40.0

Table 10: Coag-Flocculation Kinetic Parameter and Linear Regression Coefficient of PTSC at varying dosage and pH=6.0

Parameter	100 mg/l	200 mg/l	300 mg/I	400 mg/I	500 mg/I
α	2.0	2.0	2.0	2.0	2.0
R^2	0.281	0.785	0.897	0.923	0.814
K(l/mg.min)	0.0004	0.001	0.001	0.0003	0.0004
N_o(mg/l)	19.23	29.41	55.55	71.41	83.33
$(\alpha_{cf})_{BR}$(l/mg.min)	0.0008	0.002	0.002	0.0006	0.0008
$(-r_A)$	$0.004Nt^2$	$0.001Nt^2$	$0.001Nt^2$	$0.0003Nt^2$	$0.0004Nt^2$
$\tau_{1/2}$	130.0	34.0	18.0	46.67	30.0

Table 11: Coag-Flocculation Kinetic Parameter and Linear Regression Coefficient of PTSC at varying dosage and pH=8.0

Parameter	100 mg/l	200 mg/l	300 mg/I	400 mg/I	500 mg/I
α	2.0	2.0	2.0	2.0	2.0
R^2	0.835	0.805	0.835	0.531	0.837
K(l/mg.min)	0.004	0.004	0.004	0.002	0.004
N_o(mg/l)	15.62	12.82	15.62	11.23	18.86
$(\alpha_{cf})_{BR}$(l/mg.min)	0.008	0.008	0.008	0.004	0.008
$(-r_A)$	$0.004Nt^2$	$0.004Nt^2$	$0.004Nt^2$	$0.002Nt^2$	$0.004Nt^2$
$\tau_{1/2}$	16.0	19.5	16.0	44.52	13.25

Table 12: Coag-Flocculation Kinetic Parameter and Linear Regression Coefficient of PTSC at varying dosage and pH=10.0

Parameter	100 mg/l	200 mg/l	300 mg/I	400 mg/I	500 mg/I
α	2.0	2.0	2.0	2.0	2.0
R^2	0.822	0.368	0.930	0.940	0.675
K(l/mg.min)	0.002	0.0004	0.002	0.001	0.0003
N_o(mg/l)	23.80	23.25	47.61	66.66	58.82
$(\alpha_{cf})_{BR}$(l/mg.min)	0.004	0.0008	0.004	0.0002	0.0006
$(-r_A)$	$0.002Nt^2$	$0.0004Nt^2$	$0.002Nt^2$	$0.001Nt^2$	$0.0003Nt^2$
$\tau_{1/2}$	21.0	107.52	10.50	15.0	56.67

Conclusion

The experiment results obtained from the research showed that the use of Pleurotus Tuberregium Sclerotium as coagulant is suitable for physical treatment of vegetable oil effluent and thus contributes little or no pollution to the environment as compared to the use of chemical coagulant and as such makes this approach a good method for effluent treatment. Therefore physical method that uses coagulant should be recommended for treatment of vegetable oil effluent.

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