International Journal of Applied Science and Mathematics
Volume 1, Issue 1, ISSN (Online): 2394-2894

Utilization of Response Surface Methodology and Factor Design in Micro Organic Particles Removal from Brewery Wastewater by Coagulation/Flocculation Technique

Obiora-Okafo I. A.
Department of Chemical Engineering,
Madonna University, Elele, Nigeria
Email: ifyobioraokafo@yahoo.com;
Ph.: +234 8038238902

Menkiti M. C.
Department of Chemical Engineering,
Nnamdi Azikiwe University, Awka, Nigeria
Email: cmenkiti@yahoo.com;
Ph.: +234 8037441882

Onukwuli O. D.
Department of Chemical Engineering,
Nnamdi Azikiwe University, Awka, Nigeria
Email: onukwuliod@yahoo.com;
Ph.: +234 8063635864

Abstract **– In this article, the coagulation flocculation method is used for the removal of total suspended and dissolves particles in wastewater using bio – coagulant (*Brachystegia eurycoma).* The cationic coagulation process was optimized using general full factorial design (GFFD) and response surface methodology (RSM). The coagulation/ flocculation experiment was carried out using nephelometric jar test technique. The coagulation process can be influenced by different parameters. The combined effects of pH, dosage and settling time were investigated and optimized using response surface methodology. Optimum conditions for the coagulation process using the coagulant were the following: pH at 10, dosage at 300mg/l, settling time is 30minutes and TSDP concentration of 9514.184mg/l.**

Keywords **– Coagulation/Flocculation, Brewery Wastewater, *Brachystegia Eurycoma,* Response Surface Methodology.**

I. INTRODUCTION

Effluents form brewery represents harmful and noxious menaces in the present and should be treated before dumping into the environment. Brewery effluent can generally be characterized as high-strength organic waste with high Total Suspended and Dissolves Particles (TSDP) concentration, which occurs as a result of combination of waste from various stages of the beer production, melting, milling, wort boiling, stabilization, maturation, fermentation and clarification [1].

Coagulation is a known process of destabilizing colloids and other substance that usually appear dispersed in water, it is considered a chemical treatment as it involves the addition of a coagulant. Coagulation/flocculation is an essential process in water and in industrial wastewater treatment [2] – [4]. Several studies have been reported on the examination of this process for the treatment of industrial wastewater, especially with respect to performance optimization of coagulants, determination of experimental conditions, assessment of pH and investigation of flocculants addition [4]-[5]. Coagulation/flocculation process has been found to be cost effective, easy to operate and energy saving treatment alternatives [6]. Coagulant dosages vary in a wide range aiming at maximum removal efficiency of pollutants using minimum doses at optimum pH [7]-[8].

Brachystegia eurycoma coagulant (BREC) is a non-toxic, biodegradable organic polymer. It can also be referred to as cationic polyelectrolyte. It is a polymer-long chained, high- molecular weights (ranging between 500 and some thousand Daltons) organic coagulant. BREC water soluble coagulant is a highly efficient contributor of cationic charge in the effluent to neutralize the negative charges of the particles when applied in specific applications. [9].

The objectives of this study were to simulate coagulation/flocculation process efficiency for brewery industrial wastewater treatment with respect to removal of TDSP, also, to investigate optimum coagulant pH, dosage and settling time. This study's main objective was to develop an approach for better understanding of the relationships between the variables and the response (TSDP) in other to obtain the optimum conditions for the coagulation process using General Full Factorial methodology (GFFM) and Response Surface Methodology (RSM).

II. MATERIALS AND METHOD

A. Brewery Wastewater Sample Collection and Characterization.

The effluent was collected from a brewery in Enugu, Nigeria. The jerry can was coiled and remained sealed until the commencement of the analysis [10]. Characterization of the wastewater sample presented in table 1 was determined following the standard method of water and wastewater purification [11].

Table 1: Brewery Wastewater Characterization Data.

Parameters	Values
pH	6.9400
Turbidity (NTU)	4268.0000
Total hardness (mg/L)	69.0000
Ca hardness (mg/L)	51.0000
Mg hardness (mg/L)	18.0000
Alkalinity (mg/L)	1500.0000
Fe^{2+} (mg/L)	0.1800
SO_4^{2-} (mg/L)	48.1400
NO_3^{2-} (mg/L)	0.1500
Cl^- (mg/L)	18.9941

E.cond (µm/m^2)	480.0000
TDS (mg/L)	268.8000
TSS (mg/L)	27.2000
T.Coliform	13.0000
E-Coli	Nil
BOD_5	672.0020

B. Coagulation – Flocculation Experiment

Coagulation experiment was carried out using a jar - test apparatus (2L square jars, with six paddle stirrers, manufactured by Philips and Birds, VA USA). Adequate dosages of the coagulant ranging from 100- 500mg/l were added directly into 200ml of BW (Brewery Wastewater). The suspension were set to pH range of 2- 10 using 0.1M H_2S0_4 or 0.1M NaOH and subjected to 2minutes of rapid mixing (200rpm), 20 minutes of slow mixing (40rpm), following 30minutes of settling time. During settling, samples were with-drawn using pipette from 2cm depth and analyzed for TSDP. Factor experiment was done and the experimental result was obtained from the 3^3 – General Full Factorial Model (GFFM) and interpreted using Design Expert Software (DES v8.0.5) to determine the response of the dependent variable.

III. Results and Discussion

The statistical study of the coagulation cationization process drives to an optimum coagulant and conditions. The studies on this section show the various combinations of the independent variables to obtain the optimum condition of the dependent variable.

A. Design of Experiments (DOE).

Design of experiment is a statistical process that can reduce significantly the number of experiments, keeping however, the reliability of the conclusion at high standard. It can provide some unique and relevant information about how variables interact [12]. Some difficulties are encountered when observing optimum values of working parameters because there are no interactions. To proffer a solution in order of obtain optimum values DOE offers a better alternative to study the effect of variables and their response with minimum number of experiments [13]. Using DOE based on Factor Analysis (FA) and Response Surface Methodology (RSM), the equal mixture can be with a minimum number of experiments without the need for studying all possible combinations of the experiments [13].

The figures collected are analyzed in a statistical approach using multiple regression method. The coded values of the independent variables for the design of experiment for the process are given in Table 2. Where X_1 is the design matrix that represents the pH of the mixture, X_2 represents the coagulant dosage and X_3 the settling time. The behavior of the system discussed is described by a mathematical Eq. (1). The optimum values of selected variables were obtained by solving the multiple regressions Eq. (1):

$$Y = b_0 + \sum_{j=1}^{k} b_j X_j + \sum_{i<j}^{k} b_{ij} X_i X_j + \sum_{j=1}^{k} b_{jj} X_j^2 \quad (1)$$

Where Y is the predicted response; b_o, b_j, b_{ij} and b_{jj} are coefficients constant; X_i and X_j are the coded independent variables or factors.

We have selected a General full factorial method (GFFM) which is suitable for third level factorial design (3^3). The runs were carried out ones (r = 1) giving a total number of samples 27. The experimental range and levels of independent variables for the cationization process are given in Table 2. In the GFFM, the low, middle and the high level factor codes were -1, 0 & +1 respectively. The results of the GFFM done based on the multiple regressions Eq. (1) obtaining the response is tabulated in Table 4.

Table 2: Experimental Factor Ranges and Levels.

Independent Variable	Range and Level		
	Lower limit (-1)	Middle limit (0)	Upper Limit (+1)
pH (X_1)	2	6	10
Coagulant Dosage (mg/l) X_2	100	300	500
Stirring Time (min) X_3	10	20	30

Table 3: GFFM Design Matrix for Coagulation-Flocculation Process Using BREC.

Independent variables in Coded form				**Independent variables in Actual form**			
				pH	**Dosage**	**Settling Time**	**Conc. (mg/l)**
RUN	**X_1**	**X_2**	**X_3**	**X_1**	**X_2**	**X_3**	**RESPONSE**
1	0	0	0	6	300	20	940.13
2	-1	-1	-1	2	100	10	858.27
3	1	-1	-1	10	100	10	710.52
4	-1	1	-1	2	500	10	1609.93
5	1	1	-1	10	500	10	749.63
6	0	-1	1	6	100	30	750.34
7	-1	-1	1	2	100	30	134.97
8	1	-1	1	10	100	30	541.5
9	-1	1	1	2	500	30	1266.38
10	1	1	1	10	500	30	750.71
11	-1	-1	0	2	100	20	939.17

12	-1	-1	0	2	300	20	1101.22
13	1	0	0	10	300	20	562.66
14	0	-1	0	6	100	20	801
15	0	1	0	6	500	20	1350.66
16	0	0	-1	6	300	10	1141.75
17	0	0	1	6	300	30	678.85
18	-1	0	1	2	300	30	939.17
19	-1	1	0	2	500	20	951.83
20	-1	0	-1	2	300	10	938.21
21	-1	0	1	2	300	30	715.37
22	1	-1	0	10	100	20	936.39
23	0	-1	-1	6	100	10	855.34
24	0	1	1	6	500	30	750.31
25	1	0	-1	10	300	10	738.27
26	-1	1	0	2	500	20	964.15
27	0	1	-1	6	500	10	801.11

Table 4: Analysis of Variance Table for Selected Factorial Model

Source	Sum of Squares df	Mean Difference	Square	F-Value	P Value Prob > F
Model	1.26E+06	10	1.26E+05	2.58	0.0441 Significant
X_1 - pH	3.27E+05	2	*1.64E+05*	3.35	0.0609
X_2 - Dosages	3.35E+05	2	1.68E+05	3.43	5.75E-02
X_1 –Stirring Time	2.66E+05	2	1.33E+05	2.72	9.58E-02
X_1X_2	3.31E+05	4	82805.2	1.7	0.2001
Residual	7.81E+05	16	48837.7		
Cor. total	2.04E+06	26			

Std. Dev.	220.99	R-Squared	0.935
Mean	859.78	Adj. R-Squared	0.951

B. Regression Equation and ANOVA Report for the Coagulation Process.

The TSDP removal was the target variable that depends on the principle variables X_1, X_2 and X_3 in the optimization stages, which were the most important parameters affecting the efficiency of the particle removal. In order to study the interaction factors, experiments were performed varying the physical parameters, using experimental design. By the application of multiple regression analysis on the Table 3 data, the experimental results of the GFFM were fitted to the polynomial Eq. (1). The adjusted model obtained for particle removal, as a function of the more significant variables, as shown in Eq. (2).

$$Y_{conc.} = 859.78 + 112.61X_1 + 36.82X_1^2 - 134.50X_1 - 3.90X_2^2 + 73.89X_3 + 66.45X_3^2 - 193.75X_1X_2 + 40.12X_1^2X_2 + 24.34X_1X_2^2 + 27.53X_1^2X_2^2 \quad (2)$$

According to the ANOVA report Table 4, three factors and one interaction were considered significance (X_1, X_2, X_3 and X_1X_2). This fit of the model was checked with the coefficient of determination R^2, which was calculated to be 0.935, indicating that 93.5% of the response variability is obtainable. This study indicates that the model can be considered statistically significant according to the F-test value of 2.58 with 95% confidence level. There is only a 4.41% chance that a model "F-value" could occur due to noise. The probability P value is low (0.0050) and "Prob P > F" indicating the significance of the model as specified previously according to the ANOVA Table 4. The fact that ANOVA report gives high R^2 correlation factors allows us to present the GFFM and the DOE procedure as a consistent statistical model for analyzing the system.

C. Significant and Diagnostic Graphics

A graphical demonstration of ANOVA test may be the Half Normal Probability plot (Fig. 1). The Half normal probability plot is a graphical tool that uses these ordered estimated effects to help asses which factors are important and not important. Unimportant ones are those that have a near zero effects while important factors are those whose effects are considerably removed from zero like (X_1, X_2, X_3 and X_1X_2). The factors shown on Fig. 1 are the important factors, other interactions not shown are the unimportant factors.

Shapiro-Wilk normality testing the selected terms to determine if they follow a normal distribution. The Shapiro-Wilk P - Value is greater than 0.10 as shown in Fig. (1), meaning the factors shows statistically significant effects. The graph of the actual response values versus the predicted response values Fig. (2), helps to detect group of values not easily predicted by the model. The data point are splitted evenly by the 45 degree line showing no further transformation is needed to improve the fitness of the model.

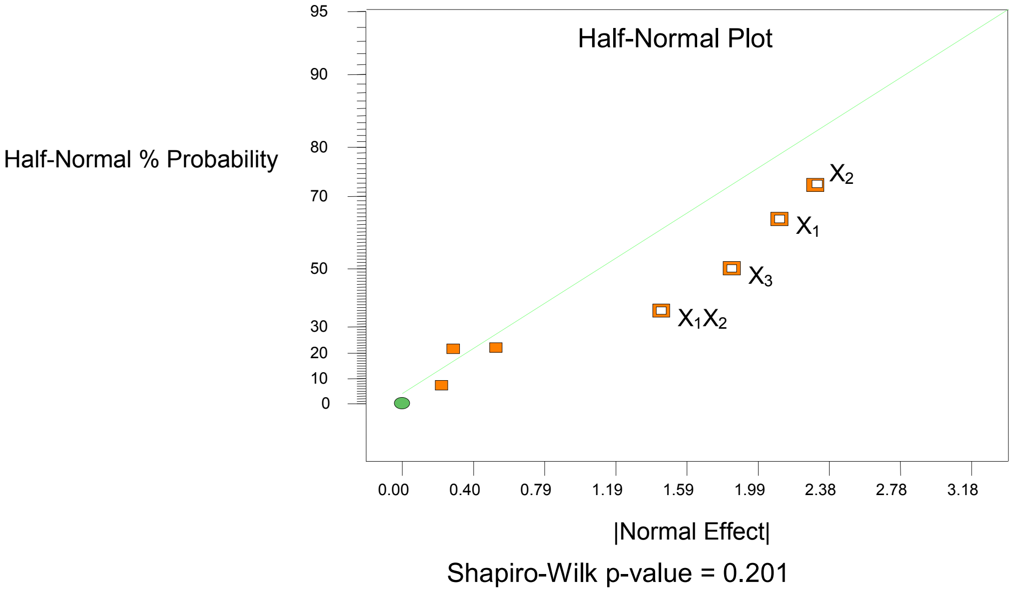


Fig.1. Half Normal Plot of important factors on coagulation process.

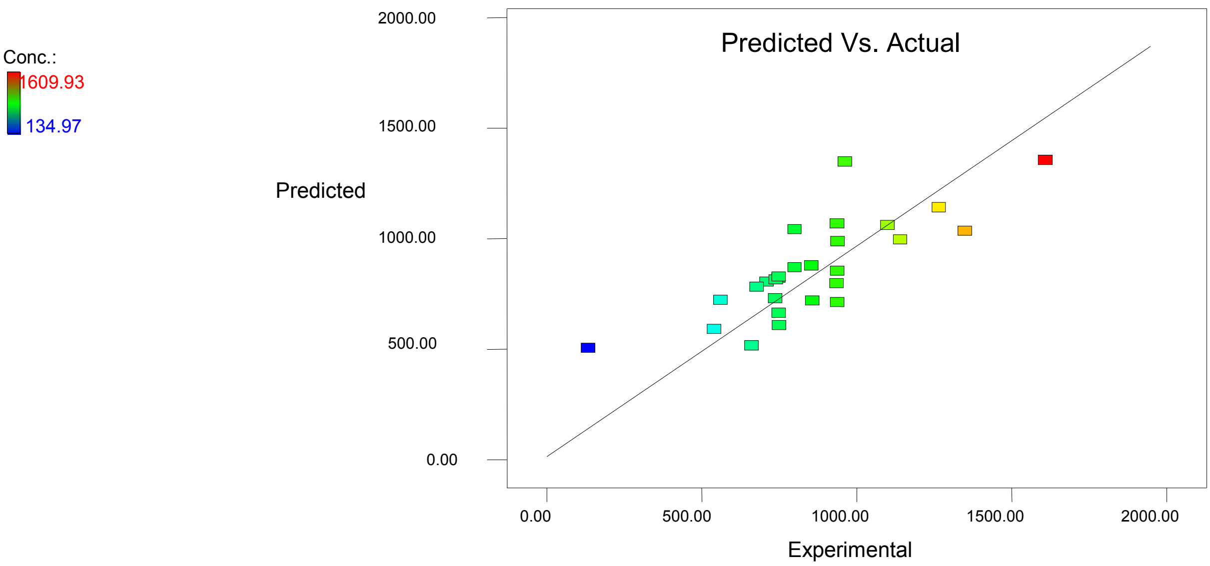


Fig.2. Predicted versus Experimental on coagulation process.

D. Model Graphics

Factor Graphics

The one factor graphics are used to look at main effects and the effects of significant terms [14]. The factor effect on Fig. 3, for the TSDP removal and pH signify the linear effect of changing the level of a single factor. The effect of pH on the plot shows that as the pH value changes from 2-10, the TSDP removal rate increases and the optimum conditions for the removal is at pH 10, Dosage 500mg/l and Settling time 30minutes. Fig. 4, Indicates a sharp decrease in TSDP in wastewater as dosage increases until equilibrium is set at 300-500mg/l dosage. The conditions for the plot are pH 10, Dosage 500mg/l and settling time 30minutes.

Response Surface and Contour Graphics.

Design–Expert Software provides various graphs to interpret the model selected. For Response Surface Methodology (RSM), the primary graph will be the contour plot and 3D-Surface plot. The contour plot, plots Eq. (1), and allows evaluating from qualitative point of view how the behavior of the whole studied system is and it showed how two factors affect the response [15]. The response surface, which are the graphical results of interactive effects are shown in Fig. 5, 6 and 7. Fig. 5 shows the response for the interactive factor pH and Dosage at different conditions. As expected, Fig. 5 shows that TSDP concentration in the effluent decreases as the pH tend to alkalinity. The response surface corresponding to the second-order model indicates that a high removal process is experienced with an increase in pH, and medium removal of TSDP is also experienced at increase in coagulation amount [16]. The flag on Fig. 5 was planted at TSDP of 514.184mg/L which shows the optimum value at pH 10, 300mg/L dosage and settling time of 30minutes. Fig. 6 indicates the flag planted at 720.984mg/L TSDP which is the optimum value for the conditions of pH 10, 300mg/l dosage and settling time of 20minutes. The graphics shows that increase in pH value, and the 300mg/l dosage provides the best removal for the process. Fig. 7 also indicates the flag planted at 728.422mg/L TSDP. Therefore the maximum TSDP removals are obtained for high pH values. Also to Fig 5, 6 and 7, TSDP removal in the effluent increases when dosage is kept between 300 - 500mg/L for a given settling time.

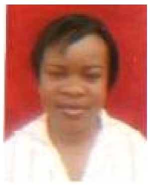
IV. Conclusion

This research studied the variables affecting the removal of TSDP from brewery wastewater and was carried out under laboratory conditions. Using response surface methodology and regression analysis, it was possible to study the effect of key factors on the coagulation process. Process optimization was accomplished by the application of factorial design and response surface methodology. The study shows clearly that response surface methodology was an adequate method to optimize the operating conditions in order to maximize particle removal from the effluent. A general full factorial design (27 assays) was employed for the the regression analysis. Satisfactory prediction equation was derived for the analysis. The optimum value for an effective removal of total suspended and dissolves solid particles was achieved at pH 10, 300mg/L dosage and settling time of 30minutes and the flag planted at the TSDP value of 514.184mg/L which indicates the lowest value for the analysis. Based on the study results, the conclusion is that maximum particle removals are obtained as pH values tend towards alkalinity. Also particle removals in the effluent increases when dosages are kept between 300-500mg/L for a given settling time. The purpose of this research to investigate the possible uses of low cost and easily available bio - coagulant material (*Brachystegia eurycoma*) for the removal of total suspended and dissolved solid particles in brewery wastewater was achieved.

References

[1] S. Sen, G. N Demirer, "Anaerobic treatment of real textile waste water with a fluidized bed reactor," Water research, vol. 37, 2003, pp. 1868-1878.
[2] M. A. Ismail, "Chemical coagulation," Proc. Florida State Hort. Soc. Vol. 91, 1978, pp 142–150.
[3] H. I Abdel-Shafy, O. Abo-El-Wafa, M. A. Azzam, "Chemical treatment of industrial effluent, in: International Conference on Heavy metals in the Environment," New Orleans, 1987, pp 452.
[4] A. A. Tatsi, A. I. Zouboulis, K. A. Matis, P. Samara, "Coagulation – flocculation pre – treatment of sanitary landfill leachates," Chemosphere, vol. 53, 2003, pp 737–744.
[5] H. I. Abdel-Shafy, S. E. Abdel-Basir, "Chemical treatment of industrial wastewater," Environ. Manage. Health, vol. 2, issue 3, 1991, pp 19–23.
[6] D. Bromley, M. Gamal El-Din, D. W. Smith, "A low cost treatment process to reduce phosphorus and suspended solids in liquid wastes from animal farm operations, in: Proceedings of the Fourth International Livestock Waste Management Symposioum and Technology Expo," Malaysia Society of Animal Production, Penang, Malaysia, 2002, pp 215.
[7] Y. Watanabe, Y. Kanemoto, K. Takeda, H. Ohno, "Removal of soluble and particulate organic material in municipal wastewater by chemical flocculation and bio film processes," Water Sci. Technol., vol. 27, 1993, pp 201 – 209.J. Szpak, D. Woods, K. Bouchard, "Critique jar testing for the design of coagulation–flocculation systems," Water Qual. Res. J. Can., vol. 31, 1996, pp 51–64.
[8] D. Gregory, K. Carlson, "The impacts of chemical sequencing on filtration performance," AWWA Ref, Denver, Co, 2003.
[9] M. C. Menkiti, O. D. Onukwuli, P. K. Igbokwe, F. X. O. Ugodulunwa, "Rapid Coagulation/flocculation kinetics of coal Effluent with high organic content using Blended and unblended Chitin Derived Coagulants (CSC)," Journal of Applied sciences, vol. 3, issue 4, 2008, pp 317 - 323.
[10] L. S. Clesceri, A. E. Greenberg, A. D. Eaton, "Standard methods for the examination of water and waste water," 20th edition. APHA. USA, 1999.
[11] C. E. P. Box, W. G. Hunter, J. S. Hunter, "Statistics for Experimenters; An introduction to Design, data analysis and model building," John Wiley, New York, 1978, pp 432.
[12] Beltran-Heredia, J. Sanchez-Martin, Davila – Acedo, "Optimization of the synthesis of a new coagulant from tannin extract," Journal of Hazard Materials, vol. 188, 2011, pp 1704-1712.
[13] P. C. C. Faria, J. J. M. Orfao, M. F. R. Pereira, Water Res, vol. 38, 2004, pp 2043.
[14] Y. H. Kim, "Coagulants and Flocculants. Theory and practice," Tall oak publishing, Littleton, 1995.
[15] J. P. Wang, Y. Z. Chen, X. W. Ge, H. Q. Yu, "Optimization of coagulation-flocculation process for a paper-recycling wastewater treatment using response surface methodology," colloids and surface A; physicochemical and Engineering Aspects, vol. 302, 2007, pp 204-210.

Author's Profile



Obiora-Okafo Ifeoma Amaoge was born in Anambra State, Nigeria, on the 28th day of March, 1983. A graduate of Nnamdi Azikiwe University Awka, Nigeria, from Department of Chemical Engineering with a B.Eng (second class upper honors) in July, 2006. Also, obtained Master's degree in Chemical Engineering (M. Eng.) from same school in July, 2011 and she is presently doing Doctorate degree in the above mentioned school.

She presently works as a Lecturer 11 in Chemical Engineering Department, Madonna University, Elele, Nigeria. She has acquired some experiences as a lecturer for close to 2years now in Madonna University. She has some journal articles published from some researches she did in some reputable journals.

Engr. Mrs Obiora-Okafo is a Corporate Member, Nigerian Society of Engineers (NSE 32436) and a Corporate Member, Nigerian Society of Chemical Engineers (NSCHE).

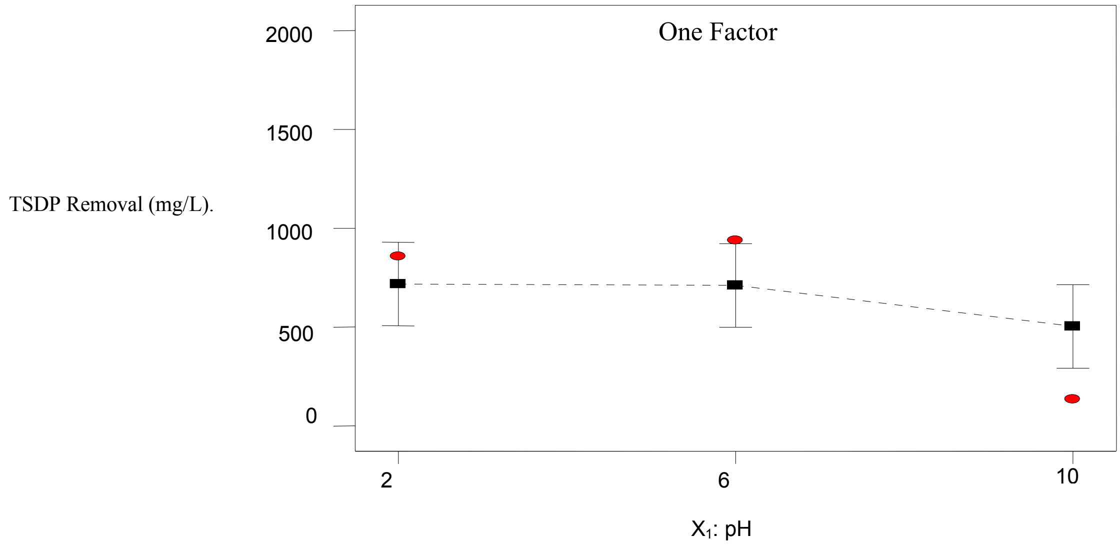


Fig.3. Factor Graphics for TSDP Removal and pH. Conditions: pH 10, Dosage 500mg/L and Settling time 30minutes.

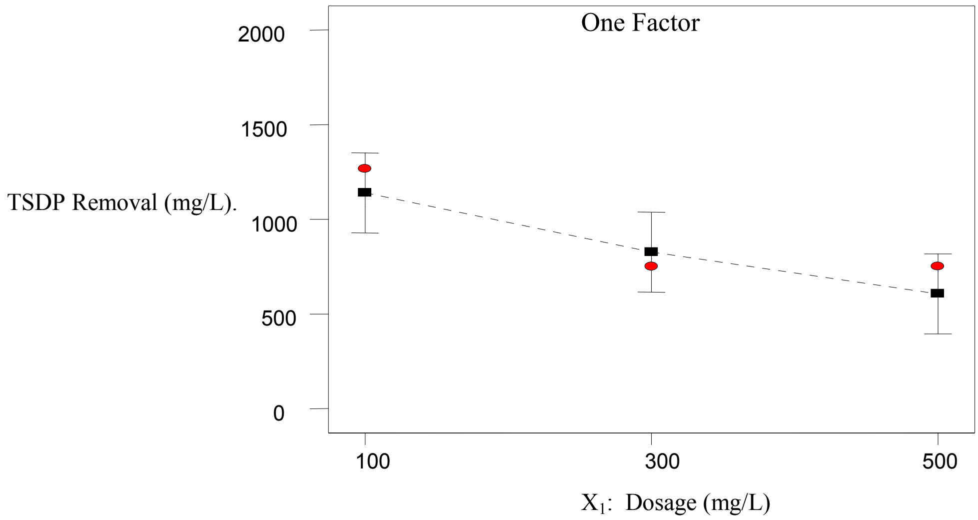


Fig.4. Factor Graphics for TSDP Removal and Dosage. Conditions: pH 10, Dosage 500mg/L and settling time 30minutes

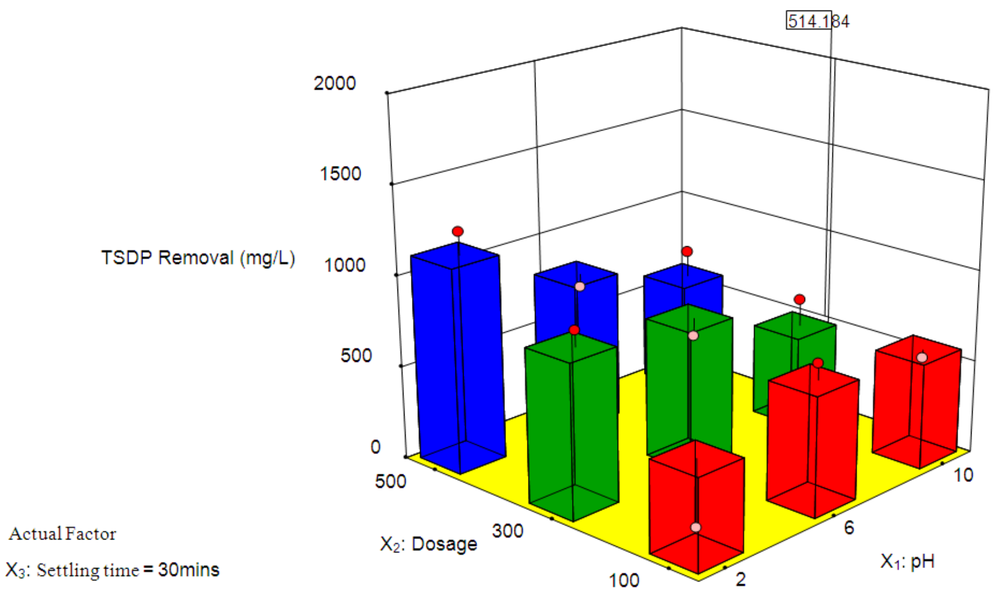
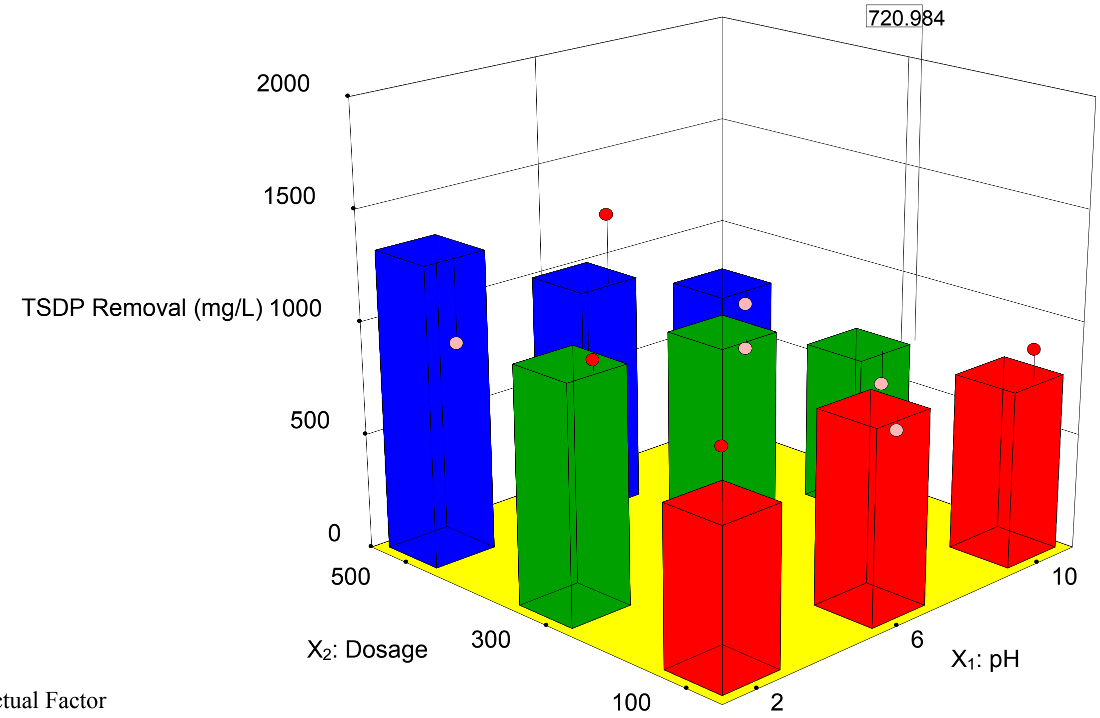


Fig.5. Response Surface contour Graphics for interaction on TSDP removal between pH and Dosage at settling time of 30minutes.



Actual Factor

X_3: Settling time = 20mins

Fig.6. Response Surface contour Graphics for interaction on TSDP removal between pH and Dosage at settling time of 20minutes.

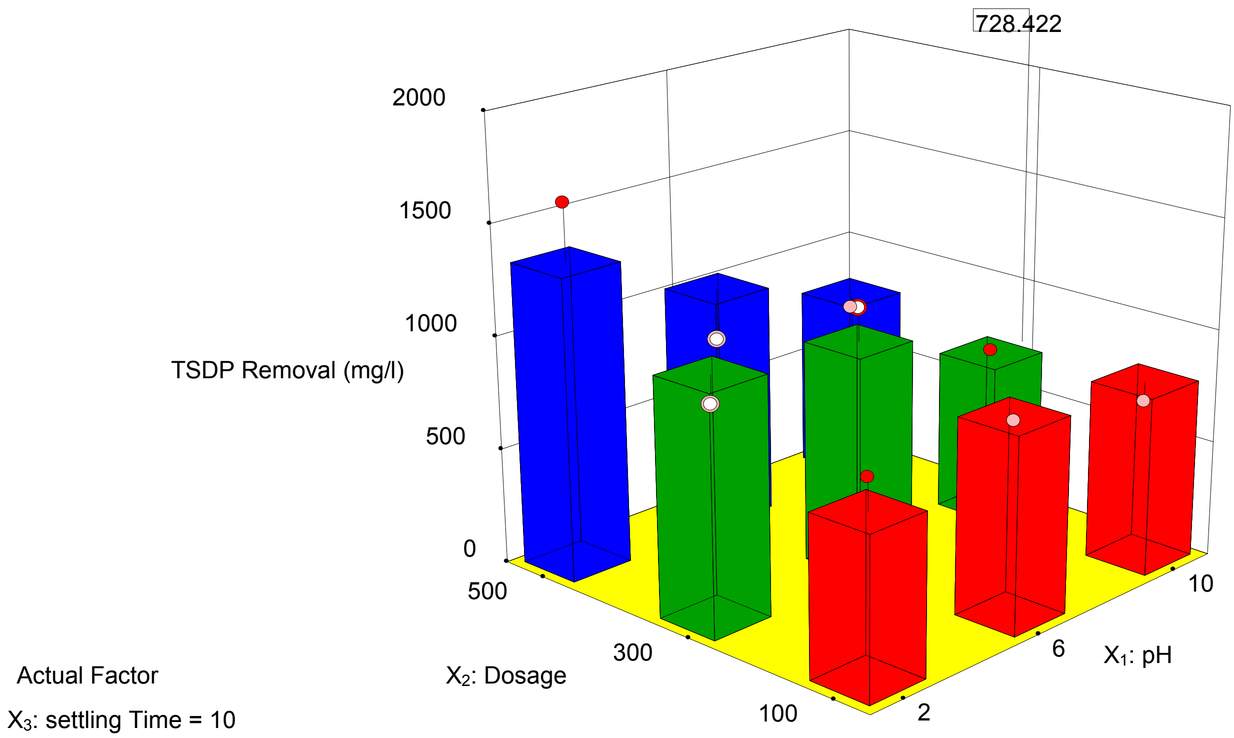


Fig.7. Response Surface contour Graphics for interaction on TSDP removal between pH and Dosage at settling time of 10minutes.