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Optimization Of Telfairia Occidentalis Seed, A Natural Coagulant For Turbidity Removal In Pharmaceutical Industry Wastewater: Response Surface Methodology Applied

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***Abstract*—Telfairia occidentalis seed, an eco-friendly plant seed a precursor to coagulant (TOSC) derivative was used in this study for pharmaceutical industry wastewater treatment. The effects of three independent variables; coagulation pH, coagulant dosage and settling time were ascertained. Response Surface Methodology (RSM) using central composite design (CCD) was used to optimize the three variables. Increase in the turbidity removal efficiency was achieved in higher alkaline coagulation pH (13) at longer settling time (40mins) and minimum coagulant dosage (0.1g/l). Analysis of variance (ANOVA) demonstrated that the contribution of the quadratic term of pH (X_1^2) had more significant effect on the response. There was close agreement between the experimental (89.76%) and predicted (85.24%) values. Hence Telfairia occidentalis seed derived coagulant (TOSC) could be a potential natural coagulant for pharmaceutical industry wastewater.**

Keywords—Pharmaceutical Industry wastewater, Turbidity removal, coag-flocculation, optimization, Telfairia Occidentalis Seed Coagulant (TOSC).

1.0 INTRODUCTION

Degradation of the environment owing to the discharge of wastewater from growing population, increased economic activities and industrialization has caused a lot of problems in the public health, aquatic lifes and vegetations [1];[2]. Although industrialization is imperative in any nation, it had led to increased generation of organic and inorganic pollutants of varying magnitude which eventually finds its way to the host environment and beyond [3];[4]. Industries producing wastewater with high content of coloring matters are pharmaceutical, cosmetics, textile, printing, food, rubber, paint among others. The associated industries made use of dyes, pigments in conjunction with other various materials along their production lines, hence generate wastewater dominantly high in organic, inorganic and color. These parameters are what prominently characterized a turbid waste water which has the potentials of inhibiting photosynthetic activities in the environment (including aquatic life) [5];[6];[7];[8];[9].

Turbidity in water is due to the presence of non settleable solids such as clay, silt, finely divided organic and inorganic matter, plankton and color producing organic substances resulting in the formation of microflocs. [1];[9]. Owing to the fact that environmental protection and sustainability has become a major global concern, wastewater produced must be adequately treated prior to discharge to ameliorate the level of damage to the environment [10]. Many techniques have been developed for turbidity removal from wastewater such as coag-flocculation, ion exchange, carbon adsorption, oxidation and biological treatments etc [9].

Among the processes stated, coag-flocculation is the most widely physicochemical treatment in use, due to its simplicity and effectiveness [10];[11]. Coagulation involves addition of ions having the opposite charge to that of the colloidal particles. In coagulation operation, a coagulant which is generally positively charged is added causing compression of the double layer and neutralization of the electrostatic surface potential of the particles. The resulting destabilized particles will aggregate [1].

Synthetic coagulants such as alum and iron salts are commonly used as chemical coagulants, but utilization of alum has raised a public health concern because of the large amount of sludge produced during the treatment and high level of alum that remains in the treated water. The intake of large quantity of alum salt may cause Alzheimer disease. Based on this back drop, attention is shifted towards natural coagulants for coagulation – flocculation treatment processes. The natural coagulants of interest is *Telfairia Occidentalis*. *Telfairia Occidentalis* seed, commonly known as fluted Gourd is a tropical vine grown in West Africa as a leaf vegetable and for its edible seeds. It is a member of the cucurbitaceae family and is indigenous to southern Nigeria. *Telifairia Occidentalis* seeds locally available, cost effective, biodegradable, eco-friendly and safe to human health. The proximate analysis result reported by [12], shows that, it has 0.01% moisture, 2.00% ash, 53.00%, lipid, 27.00%, crude protein, 15.0% carbohydrate and 3.00% crude fibre.

In this present study, central composite design (CCD) and RSM will be used to design the experiments, develop models and determine the optimum conditions. It monitored how Turbidity (in form of Total dissolved and suspended solids) removal (as response) were affected by changes in the levels of *Telifairia Occidentalis* seed derived coagulants (TOSC) dosages, coagulation pH and settling time (as independent factors). Also coag-flocculation efficiency was determined.

2. Materials and Method.

2.1 Material collection and Preparation.

2.1.1 Pharmaceutical Industry Effluent

The effluent used in this study was collected from a local pharmaceutical industry situated in Anambra State, Nigeria.

2.1.2 *Telfairia Occidentalis* Seed sample

The seeds of *Telfairia Occidentalis* was sourced from Nkwo, Enugwu Ukwu market dehusked and dried at room temperature for several days. The seed samples were processed to TOSC according to the procedure reported by [13].

Table 1: Characteristics of Pharmaceutical Effluent

Parameters	Values
pH	3.87
Turbidity(g/l)	11731.330
Total hardness (g/l)	6000
Ca hardness (g/l)	3344.00
Mg hardness (g/l)	2656.00
Fc^{2+} (g/l)	Nil
cl^{-}(g/l)	
E-cond ($\mu m/m^2$)	8.17
TDS (g/l)	57.25
TSS (g/l)	225.50
BOD5 (g/l)	620.00
COD (g/l)	660.00

The wastewater was characterized following standard method [14].

2.2. Experimental

Experiment was carried out at room temperature in a conventional jar-test apparatus equipped with a six-unit multiple stirrer system. Appropriate dose of TOSC in the range of (0.1 – 0.7)g/l was added to 250ml of pharmaceutical effluent. The suspension, was tuned to pH range of 1-13 by addition of 10M HCL/NaOH, and subjected to 2mins of rapid mixing (120rpm), 20mins of slow mixing (10rpm), followed by 40mins of settling. During settling, samples were withdrawn using pipette from 2cm depth and analysed and changes in Total dissolved and suspended solids measured in order to determine the optimal conditions (pH, dosage, settling time through 2^3 – CCD) and coag-flocculation rate related parameters. The rate related parameters and extent of aggregation were monitored at optimal conditions at 2, 4, 8, 10, 20, 30 and 40mins. The experimental data obtained were subsequently fitted in appropriate models for evaluation.

2.3 Analytical Methods

The turbidity determination was achieved following the standard procedure reported by [14]. The percentage removal efficiency (RE) of turbidity was calculated using the following relation:

$$RE = \left(\frac{C_o - C_1}{C_o}\right) \times 100 \qquad (1)$$

Where Co and Ci are the turbidity concentrations before and after the process respectively.

2.3 Experimental Design

In this study, the Box-Behnken response surface experimental design with three factors at three levels was used to generate the experimental matrix and the model equations for the optimization of the performance of TOSC for pharmaceutical Industry Effluent (PIE).

In experimental designs and RSM, the independent variables (pH(X_1), dosage (X_2), settling time X_3) are codified to normalize the variables before regression analysis and eliminate the effect of different units and ranges in the experimental domain and allows parameters of different magnitude to be investigated in a range between -1 and +1 [15]. The equation adopted for the coding is stated below:

$$B = \frac{b - \frac{(b^{11} + b^{1})}{2}}{\frac{(b^{11} + b^{1})}{2}} \qquad (2)$$

Where B is the code variable, b is the natural variable, b^{11} and b^{1} are the maximum and the minimum of the values of the natural variable respectively. Independent variables range and levels for the process optimization are presented in Table 1, while Table 2 shows the 2^3 – CCD factorial design matrix with output (response). The experimental results of the dependent, MATLAB 7.0 to estimate the response of the dependent variable. Furthermore, the dependence of the response (turbidity removal) on these manipulated variables was determined with first and second-order polynomials as shown below

$$Y = \beta_o + \sum_{i=1}^{n} \beta_i X_i + \Sigma \qquad (3)$$

Where β_o – the constant term and β_i – coefficient of the linear parameters respectively. X_i – the factor and ε is the residual from the treatments.

The critical points in the RSM plot are obtained from the quadratic polynomial equation.

$$Y = \beta_o + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} X_{ii}^{2} + \sum_{i<j}^{n} \beta_{ij} X_i X_j + \Sigma \qquad (4)$$

Where Xi and Xj represent the factors,

βii represents the coefficient of the quadratic parameters, and βij represents the coefficient for the interaction parameters [16].

Table 2: Optimization range and levels of manipulated operating variable.

Manipulated Variable	Lower Limit(-1)	Base Level (0)	Upper Limit (+1)
pH(X_1)	1	7	13
Dosage (X_2) (g/l)	0.1	0.4	0.7
Settling Time (mins)	2	21	40

Table 3: Full design matrix and response results for the experimental variables.

S/No	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3	X_1^2	X_2^2	X_3^2	Y_1	Y_2	$\bar{Y}_{ave}$
1	0	0	0	0	0	0	0	0	0	372	375	373.5
2.	-1	-1	-1	1	1	1	1	1	1	728	725	726.5
3.	1	-1	-1	-1	-1	1	1	1	1	1154	1156	1155
4.	-1	1	-1	-1	1	-1	1	1	1	1632	1630	1631
5.	1	1	-1	1	-1	-1	1	1	1	1676	1650	1678
6.	0	0	0	0	0	0	0	0	0	372	373	372.5
7.	-1	-1	1	1	-1	-1	1	1	1	528	531	529.5
8.	1	-1	1	-1	1	-1	1	1	1	212	214	213
9.	-1	1	1	1	-1	1	1	1	1	656	654	655
10	1	1	1	1	1	1	1	1	1	1192	1196	1194
11.	0	0	0	0	0	0	0	0	0	372	368	370
12.	-1	0	0	0	0	0	1	0	0	1188	1189	1188.5
13.	1	0	0	0	0	0	1	0	0	970	972	971
14.	0	-1	0	0	0	0	0	1	0	202	204	303
15.	0	1	0	0	0	0	0	1	0	398	401	399.5
16.	0	0	-1	0	0	0	0	0	1	724	721	722.5
17.	0	0	1	0	0	0	0	0	1	332	335	333.5

4.0 Results and Discussion

4.1 Effects of independent variable on coag-flocculation activity.

The independent variables (such as; coagulation pH, coagulant dosage and settling time) are the primary factors that influences the coagulation process. Experiments were performed to study the effect of these variables on the turbidity removal efficiency at varying ranges of; pH (1, 3, 5, 7, 10, 13), dosage (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7)g/l and settling time (2, 4, 8, 10, 20, 30, 40)min. General observation showed that the turbidity removal efficiency increases linearly with increasing pH, settling time at constant dosage. This phenomenon indicates that with increasing pH from 3 to 13, increases the solubility of TOSC, which increases the protonation of the TOSC adsorption site (surface) and enhances the removal efficiency of turbidity.

However, pH lower than 13 resulted in comparatively lower turbidity removal efficiency due to the decrease in the solubility of TOSC as can be seen in figure 1. Similarly, turbidity removal efficiency was increased with increasing settling time from 4 – 40mins. This could be that increasing settling time would ensure adequate time for interaction between the cationic and Anionic radicals (TOSC – Turbidity) leading to formation of more flocs (ie more adherence of turbidity particles on TOSC surface) through particle bridging mechanism, which apparently increases the turbidity removal efficiency. Conversely, increasing coagulant dosage of TOSC led to returbidization of the system (i.e making the system to become turbid) with the remote consequence of low turbidity removal efficiency value. Also the decrement may be due to saturation or equilibrium stage being achieved by the TOSC particles. Similar results was reported by [9].

Summary of the results obtained in figure 1, shows that optimum turbidity removal efficiency was achieved at 89.76% for pH 13, dosage 0.1g/l and settling time of 40mins.

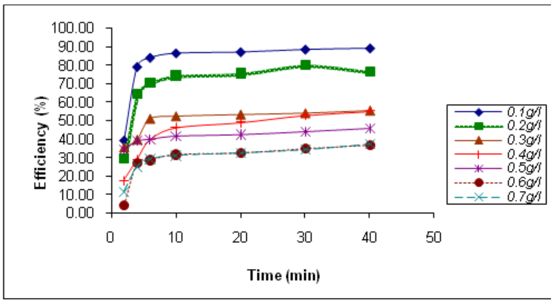

Fig.1:Coag-flocculation efficiency turbidity (TDSS) removal at optimum pH and settling time.

4.2 Process Optimization and Analysis

Optimization of coag-flocculatiom of pharmaceutical industry wastewater operational parameter using RSM. The performance of TOSC can be influenced by parameters (independent variable) such as, coagulation pH, (X_1) coagulant dosage (X_2) and settling time (X_3). Therefore it is imperative to ascertain the suitable process condition for TOSC to perform optimally (best operating conditions). The turbidity removal of the treated wastewater was chose as the dependent variable of interest. The results obtained from 17 experimental runs and response using CCD are presented in Table 4.

Table 4: Process Optimization results for Turbidity removal.

Sample	X_1 (pH)		X_2 (Dosage)		X_3 (Settling) time		$Y_{Turb.}$ removal g/l
	CV*	RV**g/l	CV*	RV** (g/l)	CV*	RV** (min)	
PIE	1.0000	13.0000	1.0000	0.1000	-1.0000	40.0000	1731.7

* Coded Value

** Real value

From Tables 1 and 4, it can be observed that at best operating conditions, turbidity was reduced from 11731.330g/l to 1731.7g/l. this translates to 85.24% turbidity removal from pharmaceutical wastewater at the condition of the experiment for $X_1 = 13$, $X_2 = 0.1$g/l and $X_3 = 40$mins. The experimental turbidity removal efficiency obtained under the same optimal condition was 89.76% which was in agreement with the predicted value by the developed mathematical model, hence giving credence to the optimized conditions and values obtained. ANOVA, was used to analyze the significance of the model by using ‘F’ test and P-values presented in Table 5

Table 5: Results of Analysis of Variance for turbidity removal model.

Variables	Coefficient	Se	tstat	Pval	Fstat
Constant	415.3239	98.22	4.2285	0.0038954	Sse = 3.6882×10^5
X_1	48.0500	72.587	0.66196	0.52918	
X_2	273.0000	72.587	3.7617	0.0070592	dfe = 7
X_3	-298.8000	72.587	-4.1164	0.0044795	
X_1X_2	59.2500	81.155	0.73009	0.48904	dfr = 9
X_1X_3	-31.6250	81.155	-0.38969	0.70835	
X_2X_3	-40.1250	81.155	-0.49443	0.63614	Ssr= 3.2423×10^6
X_1^2	631.9331	140.23	4.5063	0.0027773	
X_2^2	-146.5669	140.23	-1.0452	0.33069	F = 6.8373
X_3^2	80.1831	140.23	0.57178	0.58535	
	$R^2 = 0.8979$	Adj $R^2 = 0.7665$	MSE $= 5.2689 \times 10^4$		Pval = 0.0095103

The fisher’s ‘F’ test gives the linear, quadratic and interaction effects of the independent variables. If P-value is less than or equals 0.05, ($P \leq 0.05$), the effect of the variables are significant. In addition, the lower P-value indicates the more significant effect. Furthermore, the high R^2 values of the models validates their agreement with the experimental data. The results from Table 5, shows that, the F value of the model was higher then the P-value, hence supporting the fact that the developed model was significant. Equally the R^2 was high, very close to unit, indicating that the model was robust, thus the experimental data was in agreement with the predicted data confirming the reliability of the conducted experiment. From Table 5, the results shows that, the linear terms of X_2 (dosage) and X_3 (settling time); the quadratic term of pH (X_1^2) were significant, with all P-values < 0.05 and none combined terms were significant. Hence the mathematical model equation for turbidity removal was developed based on the significant terms.

$$Y_{Turb} = 415.3239 + 273.000X_2 - 298.8000X_3 + 631.9331X_1^2 \qquad (5)$$

Table 5, also shows that the quadratic pH variable was more significant than others with the corresponding lowest P-value. Therefore, the quadratic pH variable could be ascribed to be the major determining condition of turbidity removal for the system.

From the developed mathematical model equation, it can be observed that the coefficients for X_2 and X_1^2 were positive implying that the turbidity removal increases at low dosage of TOSC in high alkaline medium (pH). This phenomenon indicates that increasing the coagulant dosage can have negative effect on the turbidity removal.

4.3 Response Surface Methodology

The turbidity removal efficiency was investigated at the minimum of coagulant dosage and optimum values of pH and settling time. The importance of employing response surface methodology was to study the influence of the operating variables i.e coagulation pH, coagulant dosage and settling time on the removal of turbidity from the pharmaceutical industry wastewater. The graphical illustration of RSM is the three dimensional plots (3-D). This was undertaken by evaluation of equation 5 and data generated there from was employed for 3D surface plots as shown in figures 2 - 4.

Figures 2 - 4; presents the interactions of pH and dosage; pH and settling time; dosage and settling time as they relates to turbidity removal (response) respectively. In figure 2, the interaction of pH range from 7 to 13 and dosage 0.1g/l, more than 75% turbidity was removed of 89.76% was achieved at the pH of 13. It important to note that the response value obtained are dependent on the intensity of the colour of the plots [17]. Observation of figure 2, indicates that the concentration turbidity residue was zero similarly, figure 3, shows that interaction of pH and settling time has significant effect on turbidity removal. This implies that increasing pH from 3-13 and with corresponding increase in the settling time from 4-40 mins resulted in the optimum value of turbidity removal at, approximately 90%, for pH 13 and settling time 40mins.

Also, the concentration turbidity residue was recorded at 200g/l. for figure 4, the concentration turbidity residue was obtained at 100g/l. The general trend observable from the figures, shows that the interactive effects of pH and settling time was very predominant and significant to the turbidity removal. Whereas, dosage in its various combinations had negligible effect on turbidty removal but the linear term of dosage supported the increase in turbidity removal.

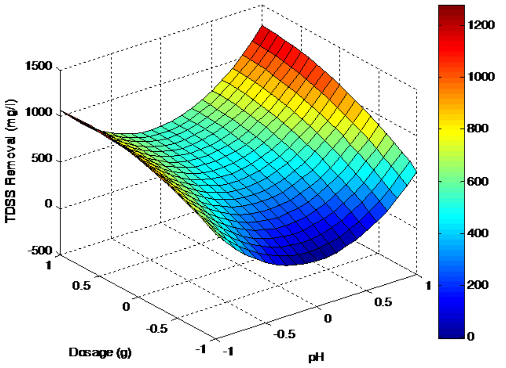

Fig.2:Surface response plot of pH interaction with dosage.

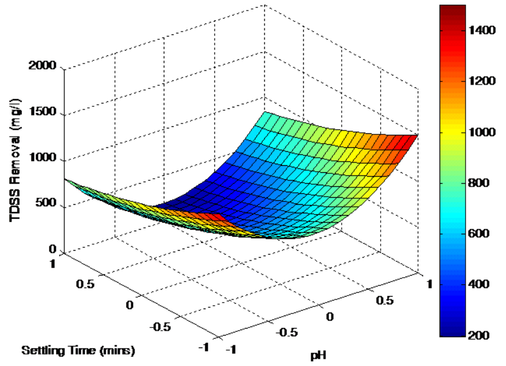

Fig.3:Surface response plot of pH interaction with settling time.

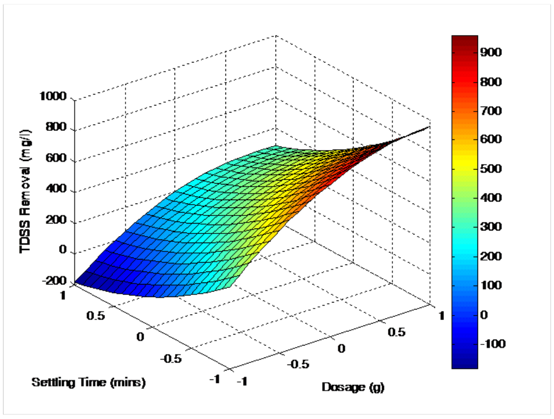

Fig.4: Surface response plot of dosage interaction with settling time.

4.4 Coag-Flocculation Rate at Optimal pH and settling time.

The implication of result presented in figure 5 was that the maximum rate of coagulation driven by optimum rate of coagulation, rate constant, K, was achieved at 0.1g/l dosage and pH of 13.

This is achievable if K is determined by fitting the experimental data on the plot of $1/C_t$ or 1/Turbidity against time as show in equation (6)

$$\frac{1}{C_t} = Kt + \frac{1}{C_o} \qquad (6)$$

Where C_o, C_t are turbidity concentration at time zero and t, respectively. K is coag-flocculation rate constant for Brownian coag-flocculation transport of destabilized particle at α^{th} order [18];[17]. β_{BR} is Brownian aggregation factor for flocculation transport mechanism, t is settling time.

The coag-flocculation rate related parameters at optimal pH and settling time generated from analysis of equation 6 that affects the ability of TOSC to remove turbidity are presented in Table 6. These parameters have relevant applications on the design, construction and practical implementation of this study.

In Table 6, 0.1g/l dosage has the highest, K value (0.000075615min) at 0.21min ($\tau_{1/2}$) coag-flocculation period, giving credence to the result obtained in figure 1 and Table 4. This gives account for the highest Turbidity removal efficiency obtained at 0.1g/l dosage in figure 1. K value determines the rate of coag-flocculation. High K value with the corresponding low $\tau_{½}$ ensures effective and efficient coag-flocculation.

The linear regression coefficient (R^2) was used to evaluate the level of accuracy of fit of experimental data on equation 6. Table 6, R^2 = 0.827 shows that only 17.3% of the total variation of the data could not be explained by equation 6, indicating that 82.7% of the experimental data were significantly described by equation 6.

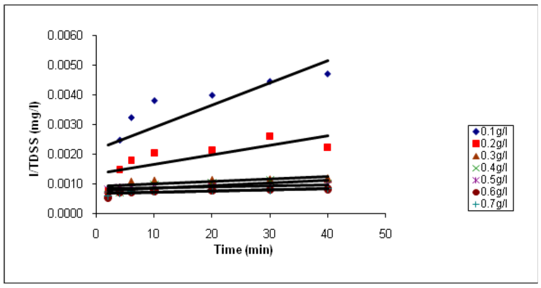

Fig.5: Kinetic rate plot of 1/TDSS against settling time.

Table 6: Coag-flocculation Rate related result at optimal pH and settling time.

Parameter	**0.1g/l**	**0.2g/l**	**0.3g/l**	**0.4g/l**	**0.5g/l**	**0.6g/l**	**0.7g/l**
α	2.000	2.000	2.000	2.000	2.000	2.000	2.000
R^2	0.827	0.591	0.599	0.752	0.660	0.555	0.674
K(l/g.min)	**7.5** E-05	3.21E-05	7.701E-06	1.012E-05	3.50E-06	4.764E-06	4.46E-06
B_{BR}(l/g.min)	1.5E-04	6.42E-05	1.54E-04	2.024E-05	7.0E-06	9.528E-06	8.92E-06
τ½ (min)	0.21	0.50	2.09	1.59	4.60	3.38	3.61

5.0 Conclusion

This study has demonstrated the applicability of Telfairia Occidentalis seed derived coagulant (TOSC) for the removal of turbidity in Pharmaceutical wastewater by optimizing the operational parameters. Linear terms X_2, X_3 and quadratic term of X_1^2 derived from RSM successfully predicted turbidity removal (response). ANOVA result showed that there was significant effect on turbidity removal with increase in coagulation pH and settling time. There was no significant difference between the experimental and predicted values of Turbidity removal. This has given credence to the fact that RSM was an effective tool for optimization of Turbidity removal from pharmaceutical wastewater.

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