Response Surface Methodology Approach to Optimization of Process Parameters for Coagulation-Flocculation Process of Paint Wastewater Using *Telfairia Occidentalis* Seed

Ifeoma H. Anyaene, Okechukwu D. Onukwuli, Ifeoma A. Obiora-okafo., Babayemi, A. K.

Abstract— Coagulation-flocculation process was used to treat paint effluent stream with Telfairia occidentalis seed powder (FPSC) as coagulant. The crude effluent was characterized by a high level of BOD5 with initial concentration of 2205 mg/l. Proximate analysis of the coagulant revealed that the Percentage moisture content was 9.06 %, ash content 6.81%, fat content 35.28 %, crude protein 25.47 %, crude fiber 9.33 % and carbohydrate content 14.05 %. Fourier Transform Infrared Spectroscopy (FTIR) showed that the coagulant contained -NH2, -OH, and -C-H, which are some functional groups that enhance coagulation process. Jar test experiments employed for the coagulation-flocculation process involved 4 min of rapid mixing at 100 rpm and 20 min of slow mixing at 40 rpm. A central composite design (CCD), which is a standard design of response surface methodology (RSM) was used to evaluate the effects and interactions of three major factors (coagulant dosage, coagulation pH, and settling time) on the biochemical oxygen demand (BOD5) removal efficiency. The results indicate that at the optimum conditions of 4.0 g/l for the coagulant dosage, 4.0 for pH, and 40 min settling time, the maximum BOD5 removal efficiency achieved was 80.29 %. The quadratic model developed for the response variable successfully describes the experimental data (R2 = 96.83 %).

Index Terms— Coagulation, flocculation, efficiency, colloids.

I. INTRODUCTION

Industrial effluents from various sectors have become a matter of major environmental concern. The paint industry is a huge water consumer and discharger of volumes of colourful wastewater with great chemical oxygen demand (COD) and nonorganic loading, establishing it as one of the major supplies of serious contamination around the world [1]. Paints generally consist of organic and inorganic pigments and dyestuffs, extenders, cellulosic and non-cellulosic thickeners, latexes, emulsifying agents, anti-foaming agents, preservatives, solvents and coalescing agents [1]. Its wastewater is generated primarily due to cleaning operations of mixers, reactors, blenders, packing machines and floors. Thus, due to the varying degree of chemicals used, the

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wastewater contains appreciable concentrations of biological oxygen demand (BOD) or chemical oxygen demand (COD)), suspended solids, toxic compounds and colour [2]. The discharge of such wastewater into the environment impedes light penetration, damages the quality of the receiving streams and may be toxic to treatment processes, to food chain organisms and to aquatic life [3]. The treatment of wastewater plays a significant role in order to remove pollutants and safeguard the water resource [4]. A number of methods such as coagulation, precipitation, ozonation, adsorption, ion exchange, reverse osmosis and advanced oxidation processes have been used for removal of organic pollutants from water and wastewater [5]. These methods have been found to be limited, since they involve high capital and operational costs [6]. Among the possible technique for water and wastewater treatment, coagulation-flocculation shows potential as one of the most efficient methods for the treatment and removal of organic contaminants from wastewater [7]. Coagulation-flocculation is one of the physio-chemical treatment processes commonly used for water and wastewater treatment. Coagulation process is described as the destabilization of colloids initially present in water supply [8]. The destabilized colloids overcome their repulsive forces leading to aggregation of the particles to form flocs. It has a wide application in water and wastewater facilities because it is efficient and simple to operate. Many factors can influence the efficiency of coagulation-flocculation process, such as the type and dosage of coagulant/flocculant [9], pH [9], etc. Optimization of these factors may significantly increase the process efficiency.

Aluminium salts, iron salts, and synthetic polymers are widely used coagulants in conventional coagulation-flocculation processes [10]. However, their use results in the production of large quantities of metal-contaminated sludge, and in addition, aluminum and iron residues in treated water has been linked to the development of certain diseases, as Alzheimer's and some cancers [11], [10]. To overcome this issue, some plants extracts like Moringa oleifera, Hibiscus esculentus (Okra), Strychnos potatorum and Bridelia ferruginea have shown great potentials to serve as coagulation/flocculation agents [12], [13], [14]. Plants extracts used as natural coagulants/flocculants are macromolecules which could be extracted from different parts of plants (barks, leaves, roots, among others) [15], [16], [17]. The emerging technologies nowadays lead to the introducing of more green conventional



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methods of waste water treatment. As the populations grow, there is always an exciting need to keep developing the remedial action according to present and future problems.

Fluted pumpkin (*Telfairia occidentalis*) is a tropical creeping vegetable vine that spread on the ground with large lobbed leaves and long twisting tendrils, cultivated in some parts of Nigeria and Africa. It belongs to the family Oliffieace and the sub-family cucurbitaceae. It is called ubong, ugu, ewekoro and ekumarkuEjashains in Nigeria and Cameroon [18]. Studies have shown that the fluted pumpkin seed as shown in Fig. 1 is rich in oil, protein and other nutritional materials which make them generally acceptable as food substances [19]. In this way, fluted pumpkin seeds are potential sources of oil and protein which if well exploited can be used in many industrial preparations such as food supplements and body cream.



Fig. 1 Fluted pumpkin pod and seeds

In this research, Coagulation-flocculation process was carried out on paint wastewater using fluted pumpkin seed powder to evaluate the efficiency of the coagulant.

II. MATERIALS AND METHODS

A. Collection of Paint Wastewater Sample and its Analysis

The paint wastewater used in this research was collected from the wastewater channel of a paint factory located in Emene Industrial Layout, Enugu Nigeria. The sample was collected in an air tight 20 L polyethylene bottle. The pH, electrical conductivity and turbidity were determined using Mettler Toledo Delta 320 pH Meter, EI Digital Conductivity Meter (model number 161) and EI Digital Turbidity Meter (model no. 337), respectively. Determination of dissolved oxygen, biochemical oxygen demand (BOD₅), total dissolved solid (TDS), total suspended solid (TSS), chemical oxygen demand and conductivity were carried out according to the standard method for the examination of water and wastewater [20]. The characteristics of the wastewater collected from paint industry are given in **Table 1**.

B. Preparation of Coagulant Stock Solution

Matured pods containing *Telfairia occidentalis* seeds were purchased from local market in Enugu city. The seeds were removed from the pod, dried under sun for 3 days, and the external shells were removed. Matured seeds showing no signs of discolouration, softening or extreme desiccation were used. The seeds kernels were ground to fine powder, using an ordinary food processor and were classified using 600 μ m sieve. The seed powder was then used in the experiment.

C. Extraction of Active Component

The active component from the powder was extracted by adding distilled water to the fine powder to make 2 % suspension (2g of fine powder samples in 100 ml water). The suspension was stirred using a magnetic stirrer for 20 min at room temperature to accomplish extraction and then filtered through a rugged filter paper (Macherey Nagel, MN 651/120). The resultant filtrate solution was used as coagulants. Fresh solutions were prepared daily and kept refrigerated to prevent any ageing effects (such as change in pH, viscosity and coagulation activity). Also solutions were shaken vigorously before use [21], [9].

D. Characterization of the Seed

Proximate analysis of seed powder sample was carried out to determine the Moisture, protein, crude fat, crude fibre and ash contents of the seed powder by the standard official methods of analysis of the A.O.A.C [22], while carbohydrate content was calculated by difference. Coagulants powder were analyzed using FTIR (Fourier Transformed Infra-Red) spectrophotometer supplied by IR Affinity-1, Shimadzu Kyoto, Japan. The spectrum was measured

in the range of $4000 - 400 \text{ cm}^{-1}$. Infrared spectroscopy was used to obtain information on the chemical structure and functional groups present in the samples.

E. Coagulation-Flocculation Experiment

Jar test was performed to evaluate the performance of the coagulants agent extracted from the process described above based on standard methods [9], [23], [24]. A known amount of extracted coagulant (1-5 g/100 ml), 20 ml of wastewater, and 20 ml of buffer solution pH 4 were added in 250 ml beaker. The coagulation-flocculation procedure involved 4 min of rapid mixing at 100 rpm. The mixing speed was reduced to 40 rpm for another 20 min. All the suspensions were left to settle for 30 min. After settling, a supernatant sample was withdrawn for further analysis. Coagulation experiment was performed on BOD₅, before each analysis. Removal efficiencies (%) were calculated according to Eq. 1:

$$BOD_5 \ removal \ (\%) = \left(\frac{C_0 - C_f}{C_0}\right) x \ 100$$
 (1)

where C_0 and C_f are the initial and final BOD₅ concentrations in the wastewater before and after coagulation-flocculation treatment, respectively.

2.6 Optimization Studies of Coagulation Process of Paint Wastewater

Optimization of the process variables affecting the coagulation process was done using Response surface methodology (RSM). Three independent factors studied were coagulant dosage, pH, and settling time and their effect on the BOD5 of the paint wastewater was investigated. 20 experimental runs were generated using Box Behnken Design (BBD). The fitness of model was evaluated using test of significance and analysis of variance (ANOVA). The chosen variables were fluted pumpkin seed coagulant (FPSC) dosage, pH, and settling time, represented by X_1 (A), X_2 (B), and X_3 (C), respectively. The method of coding of the variables had been reported by Myers [25], [26] as shown in (Table 1). Multiple regressions were used to fit the



coefficient of the polynomial model of the response shown in Eq. (2).

III. RESULTS AND DISCUSSION

$$\begin{array}{l}Y + b_{o} + \sum_{i=1}^{k} b_{i} X_{i} + \sum_{i=1}^{k} b_{ii} X_{i}^{2} + \sum_{i=1}^{k} b_{ij} X_{i} X_{j} + e \\ (2) \quad (2)\end{array}$$

where, Y is the BOD_5 , bo is the intercept, bij is the interaction effect, and bii represents the quadratic coefficients of Xi, and e is the random error [26]. Design Expert 11 software was used to design and analyze the experimental data. Design Expert is a statistical software package that performs design of experiments, comparative tests and optimization of process variables. It also contains the graphical tool that help to determine the impact of each considered parameter on the yield of a process [27].

A. Characterization results

3.1.1 Characterization of the wastewater

The outcome of the physicochemical analysis of the raw paint effluent used in this study was shown in Table 1. The results were compared with WHO and EPA standards for effluents discharge [28]. The Biochemical Oxygen Demand carried out in five days (BOD5) was observed to be higher than WHO and EPA maximum limits of 90 and 100 mg/l, respectively. The value of BOD5 obtained necessitates the need for treatment of the wastewater before discharging into land or water bodies. Biodegradability index (BI) value of 0.333 obtained for the wastewater indicates the need for the wastewater quality to be improved on to obtain a standard acceptable BI close to one [29]. BI is a reliable parameter to evaluate whether wastewater is harmful to be discharged to the environment or not.

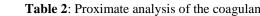
Parameters	PWW			
	Before coagulation- flocculation	*WHO STD	*EPA limit	
Conductivity, µs/cm	370.00	2000	750	
TSS, mg/l	384.17	60	50	
TDS, mg/l	1510.72	1000	1500	
TS, mg/l	1906.72		1500	
BOD ₅ , mg/l	2208.00	30	50	
COD, mg/l	6640.00	90	100	
Turbidity, NTU	126.20	250	300	

*WHO STD = World Health Organization Standards (2001), EPA = Environmental Protection Agency (Source: [28]).

3.1.2 Proximate analysis of the coagulant

The proximate analysis of coagulant precursors was presented in Table 2. It was suggested that protein was an active component in plant extracts responsible for coagulation processes [30]. This is essential for neutralization and consequent adsorption of an oppositely charged coagulant on the colloidal surface of the colloidal particles in the waste water; this in turn induces the process of coagulation through bridging, which stimulates the flocs development [31], [32], [33]. The protein content of 25.47 % for FPSC validates its potential to actually act as a coagulant in water treatment. The cationic protein of the coagulant precursors neutralizes and adsorbs particles present in the wastewaters [21]. Moisture content values of the coagulants show the water absorption ability of the coagulants [34]. The presence of fibre proved that the precursors are organic polymers with monomers that could extend as tails and loops when dispersed in water [9].Okuda et al. [23] suggests that polysaccharides such as carbohydrate are active coagulating agents. The high carbohydrate content 14.05 % for FPSC also suggests that it is a potential coagulant for wastewater treatment.

Table 2: Proximate analysis of the coagulant				
Composition	<i>Telfairia occidentalis/</i> Fluted pumpkin seed (FPSC)			
Ash (%)	6.81			
Crude fat (%)	35.28			
Crude fibre (%)	9.33			
Moisture content (%)	9.06			
Protein (%)	25.47			
Carbohydrate (%)	14.05			





3.1.3. FTIR of the coagulant (FPSC)

The FTIR spectrum of fluted pumpkin was shown in **Fig. 2a & b**. The spectra were characterized by 19 visible bands. The bands at 3429.2 cm⁻¹ represents amide, N-H and O-H stretching; bands at 3242.8 cm⁻¹ and 1558.0 cm⁻¹ represents O-H stretching and bending, respectively [35]. Also band at

2855.1 cm⁻¹ were due to the present of N-H and O-H stretching. The band at 1558.0 cm⁻¹ and 1360.5 cm⁻¹ were due to O-H bending. The result showed that fluted pumpkin has inorganic and organic components. Other functional group identified in fluted pumpkin were C=O stretching, C=O stretching, C-H deformation, C-H bend, etc.

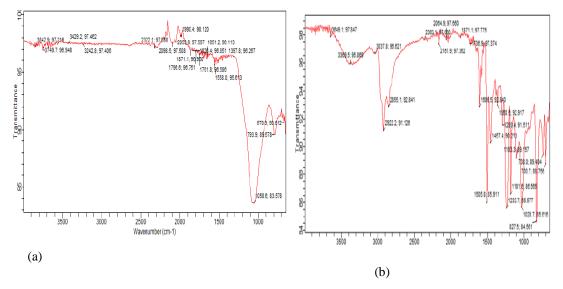


Fig. 2: (a) FTIR of FPSC before Coagulation-Flocculation (b) FTIR of FPSC after Coagulation-Flocculation

B. Effect of coagulant dosage and pH on BOD₅ removal efficiency

Dosage is an important parameter that has been considered in determining the optimum conditions for the performance of natural coagulants in wastewater treatment. Basically, insufficient dosage or overdosing would result in poor performance in flocculation. Fig. 3a showed that as the dosing rate increased from 1 to 4 g/l the contaminants removal efficiency increased. At dosage rate beyond 4 g/l, there was no remarkable increase in removal efficiency for BOD₅ using the coagulant. This is mainly because higher dosages of coagulants increase the number of exchangeable reaction sites available for an effective reduction process of contaminants in the effluent which could increase the reduction percentage [36]. The use of cationic polymer coagulants for coagulation-flocculation of negatively charged colloidal particles in wastewater is required. This results in the neutralization and destabilization of the particles that are keeping the colloidal particles stable and thus resulting in consequent adsorption and agglomeration of the particles. Charge neutralization and adsorption is the predominant mechanism. Also, the high charge density (CD) of the polymer coagulant is an important factor in using polymer coagulant. As a result of the high CD of the polymer coagulants, the optimum density was reduced tremendously [37]. Sweep- flocculation also takes place in the coagulation-flocculation process. The reduction of the removal efficiency of the contaminants after the optimum dosage of 4 g/l can be attributed to overdosing. This results in the restabilization of the particles and reversal of the charge in opposite direction. The excess dose results in the water being turbid. Ezemagu et al. [38] got similar result in the adsorptive study of paint effluent coagulation using Tympanotonos fuscatus extract as coagulant. Igwegbe & Onukwuli [33] also got similar result in their work on treatment of aquaculture wastewater using Sesamum indicum extract.

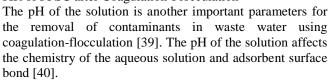


Fig. 3b shows the effect of pH on BOD₅ removal efficiency for paint waste water. As shown in the figure, BOD₅ removal by the coagulant decreases with increasing pH. The efficiency of BOD₅ removal at pH higher than 4 did not significantly increased. In acidic pH most natural coagulants form $-NH_2^+$ groups which attracts the negatively charged contaminants in wastewater which increased the removal percentage of the contaminants present. Above pH 4 the positive charge on the coagulant surface decreased and it became insoluble and this negatively affected the treatment process and decreased the removal efficiency of the contaminants. As a result, pH 4 was determined to be the optimal pH and the removal efficiencies of BOD₅ was 80.29 %. Charge on the hydrolysis product and precipitation of polymeric hydroxides are both controlled by pH variation [41]. At positive pH, the anionic functional groups of the bio coagulants can neutralize the negative charges of the wastewater, followed by polymer adsorption. Charge neutralization, sweep coagulation-flocculation and polymer adsorption played a vital role in the coagulation-flocculation process due to the pH value. As the concentration of H^+ increased with lowering pH, the neutralization of the negatively charged colloidal particle were enhanced and this increases the diffusion of BOD₅ onto the bulk of the polymer. To study the effect of time on the treatment process, studies were performed at different settling time in the range of 0 to 50 min at the optimum pH of 4 and optimum dosage of 4 g/l. The bridging flocculation mechanism of biocoagulants enhances the compact nature and strength of flocs [36] which could affect the treatment process significantly. In colloidal suspension, particles will settle very slowly or not at all due to the surface electrical charges that mutually repel each other. To induce coagulation, a coagulant with the opposite



charge is added to the water to overcome the repulsive charge and destabilize the suspension. Once the repulsive charges have been neutralized, Vander Waal's force will cause particles to cling together and form microflocs [42]. Cationic polyelectrolytes which has high molecular weight and long chain branching adsorbs colloids and thus neutralizing the charge. This leads to the growth of the floc particles which will be affected by the gravitational force and the flocs will settle at higher settling velocity [43]. The removal percentage of BOD₅ increased with settling time up to 40 min, as can be seen in **Fig. 3c**. After 40 min, no significant changes were observed in the removal percentages of the contaminants. This is due to the settling behaviour of suspended particles during their movement in the bulk of the solution forming settled particles [44]. The initial BOD₅ of 2208 mg/l was reduced to 435.3mg/l resulting to the removal efficiency 80.29%.

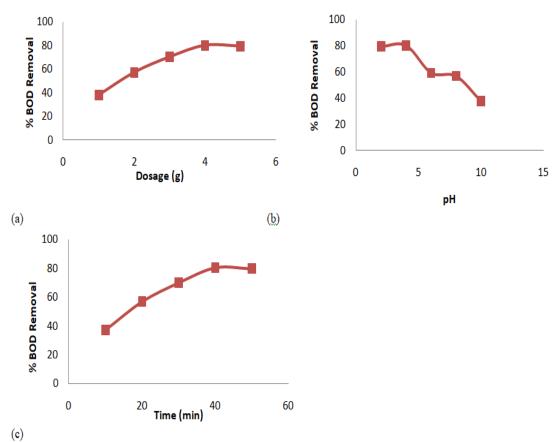


Fig. 3: Effect of (a) coagulant dosage (b) pH and (c) settling time on BOD₅ removal efficiency.

C. RSM result

3.4.1 Final equation in terms of coded factors

The central composite design used in this work allowed the development of mathematical equation where predicted results, Y, were assessed as a function of coagulant dosage (A), pH (B), and settling time (C) and calculated as the sum of constant; three main effects (in terms of A, B, and C), three interactive effects (AB, AC, BC) and three second-order effects (A^2 , B^2 , C^2) according to **Eq. 2**. The model equation was used to make prediction about the BOD₅ removal **Table 3:** RSM result showing experimental and predicted results

efficiency as shown in Table 3. The results obtained were then analyzed by means of ANOVA to access goodness of fit. The positive sign in front of the model terms indicates synergistic effect of the factor, whereas the negative sign indicates antagonistic factor effect [45].

$$Y_{\% BOD removal} = +80.12 + 4.53 A - 5.18 B + 2.94 C + 1.84 AB + 2.74 AC + 0.4150 BC - 4.83 A^2 10.46 B^2 - 5.11 C^2$$
(2)

Std	Run	F 1 A: Dosage (g)	F 2 B: pH	F 3 C: Time (min)	R: BOD removal (%)		Residual (%)
					Exp.	Pred.	
6	1	5	2	50	71.54	72.85	-1.31
1	2	3	2	30	62.11	62.43	-0.32
11	3	4	2	40	79.56	74.84	4.72
19	4	4	4	40	80.29	80.12	0.17
10	5	5	4	40	79.64	79.81	-0.17



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20	6	4	4	40	80.29	80.12	0.17
7	7	3	6	50	50.00	48.78	1.22
16	8	4	4	40	80.29	80.12	0.17
9	9	3	4	40	70.41	70.76	-0.35
3	10	3	6	30	49.00	47.56	1.44
12	11	4	6	40	59.23	64.47	-4.55
4	12	5	6	30	56.92	54.80	2.12
17	13	4	4	40	80.29	80.12	0.17
5	14	3	2	50	60.00	61.99	-1.99
18	15	4	4	40	80.29	80.12	0.17
2	16	5	2	30	61.23	62.32	-1.09
13	17	4	4	30	69.92	72.06	-2.14
15	18	4	4	40	80.29	80.12	0.17
8	19	5	6	50	67.44	66.99	0.45
14	20	4	4	50	79.57	77.94	1.63

3.3.2 ANOVA result

Table 4 showed the ANOVA results for BOD₅ removal efficiency for paint wastewater. The analysis was done by means of F-test. The P-value was used as a tool to check the significance of each factor and interaction between factors. The result of Anova for BOD₅ indicates that the P-value for the model is significant at 95 % confidence level by the Fisher's test. The P-value of the model for the BOD₅ removal efficiency was less than 0.05, indicating that the model was statistically significant. The F-values for BOD₅ was 33.89. This value implied that the model is good. The overall measure of model's performance, R² (correlation coefficient) and Adj-R² (Adjusted determination coefficient) was considered in order to ascertain the adequacy of the model **Table 4:**Anova for % BOD₅ removal from PWW using FPSC

[4]. A high value of \mathbb{R}^2 close to 1 is desirable and ensures adequacy of the model [3]. The \mathbb{R}^2 of 96.83 % for BOD₅ denotes that the model could not explain 3.17 % of the total variations for BOD₅. The Adj- \mathbb{R}^2 of 93.97% was in close agreement with the values \mathbb{R}^2 . The adequate precision of 17.0879 for BOD₅ was more than 4, showing model accuracy. The C.V. of 3.85 % for BOD₅ was less than 10 %; also the standard deviation of 2.69 for the contaminant removal was low, implying significant of model. In **Table 4**, the variable with largest effect on BOD₅ removal efficiency was the coagulant dosage (quadratic term) and pH (linear term) with P-value < 0.0001. The P-values of all the factors were significant, except the P-value of the interactive effects of dosage and pH (AB) which is 0.0826.

Source	Sum of Squares		df	Mean Square	F-value	p-value	
Model	2214.81		9	246.09	33.89	< 0.0001	Significant
A-Dosage	204.76			204.76	28.20	0.0003	
B-pH		268.84	1	268.84	37.03	0.0001	
C-Time		86.26	1	86.26	11.88	0.0063	
AB		27.01	1	27.01	3.72	0.0826	
AC	60.17		1	60.17	8.29	0.0164	
BC	1.38		1	1.38	0.1898	0.6724	
A ²	64.17		1	64.17	8.84	0.0140	
B ²	300.91		1	300.91	41.44	< 0.0001	
C ²	71.82		1	71.82	9.89	0.0104	
Residual	72.61		10	7.26			
Lack of Fit	72.61		5	14.52			
Pure Error		0.0000	5	0.0000			
Cor Total		2287.41	19				
Std. Dev.	2.69		R ²				0.9683
Mean	69.92		Adjusted R ²				0.9397
C.V. %	3.85		Predicted R ²				0.7474
			Adeq Precision				17.0879

3.3.3 Predicted versus actual for percentage BOD₅ removal

For a model to be reliable, the response should be predicted with a reasonable accuracy when compared with the experimental data. Fig. 4 compares experimental BOD_5 removal efficiency with the corresponding predicted values using FPSC. It was observed that there was a good agreement between the experimental and predicted values of the variables. The observed points on the plots revealed that the actual values were distributed relatively close to the straight lines, showing that the quadratic regression model is able to predict the removal efficiency of the contaminant.



%tage BOD removal

Color points by value of %tage BOD removal:

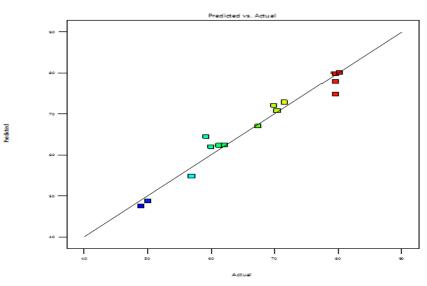
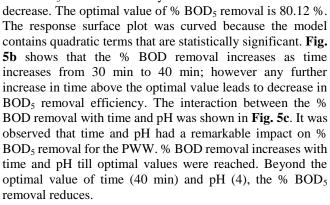


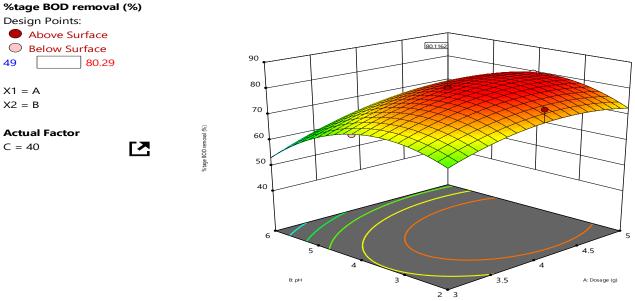
Fig. 4: Predicted versus Actual plot of BOD₅ removal efficiency

3.3.4 3D -surface plot for BOD₅ removal from PWW using FPSC

Three dimensional response surface plots were plotted to investigate the relationship between the different variables and their responses in order to obtain the optimal coagulation conditions that would maximize the contaminant removal efficiency. **Fig. 5** shows the effect of the process variables on the percentage BOD₅ removal. **Fig. 5a** shows the response surface plots as a function of pH and dosage and their mutual interaction on BOD₅ removal efficiency. This process was held at a fixed settling time of 40 min. It was observed that the % BOD₅ removal increases as pH increases from 2 to 4 and dosage increases from 3 to 4. Thereafter it starts to Factor Coding: Actual



3D Surface



(a)



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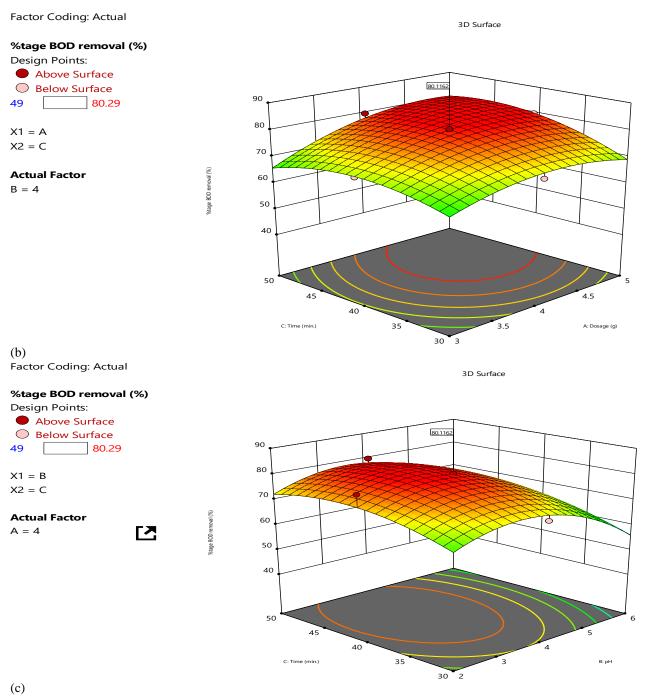


Fig. 5: 3D-surface plot for BOD5 removal efficiency versus (a) pH/dosage (b) time/dosage (c) Time/pH for PWW using FPSC

IV. CONCLUSIONS

Characterization and optimization of coagulation process of paint wastewater was carried out in this study. The effects of coagulation dosage, pH, and settling time, on the BOD₅ removal efficiency of the wastewater was studied using Response Surface Methodology (RSM). It was established that coagulant dosage, pH, and settling time, are significant factors on BOD₅ reduction of paint wastewater. A second-order mathematical model fitted well to the experimental data obtained. Optimum conditions established were coagulant dosage 4 g/l, pH 4, and settling time of 40 min. The optimum condition obtained from this study could be scaled up and applied to industrial treatment of paint wastewater.

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