

Performance Evaluation of Batch Reactor for Vinasse Wastewater Treatment Using Chemical and Natural Coagulants

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Abstract

Vinasse, the liquid residue left in the distillation of ethanol from sugar cane derivatives, frequently poses serious disposal challenges, when it is discharged without/partial treatment. The aim of the present study was to investigate the efficiency of Chemical Coagulation (CC) and Natural Coagulation (NC) to treat the vinasse wastewater from the sugarcane industry. Aluminium sulfate ($Al_2(SO_4)_3 \cdot 6H_2O$) was used as a chemical coagulant and Tanfloc, a cationic organic polymer, was used as the natural coagulant. The effects of rotation speed, mass of coagulant and settling time on the removal of colour, turbidity and COD were investigated using Box-Behnken Design (BBD) of experiments. The results showed that NC could effectively reduce the colour, turbidity and COD at the optimum conditions compared to CC. The operating costs were 0.9 US \$/m³ and 1.2 US \$/m³ for CC and NC, respectively. These results showed that natural coagulation using Tanfloc is an effective technique compared to chemical coagulation for vinasse wastewater treatment.

Keywords: Vinasse wastewater; Chemical coagulation; Natural coagulation; Batch reactor design; Cost evaluation

Introduction

With the increased demand for sugarcane based products, the sugarcane industry and its wastewaters have been increasing proportionally, making it one of the main sources of severe pollution problems worldwide. Vinasse wastewater from the sugarcane industry with high colour, COD (Chemical Oxygen Demand), BOD (Biological Oxygen Demand), COD/BOD ratio, potassium, phosphate and sulfate poses serious environmental concern, because of its pollution problems. Typically, Brazilian sugarcane industries produce more than 1300 m³ of vinasse to produce 100 m³ of ethanol. The free disposal of this wastewater presents a serious challenge to natural ecosystems and can cause considerable environmental problems such as eutrophication or the reduction of sunlight penetration in natural water bodies. Consequently, sugarcane industries have been forced to seek effective treatment technologies that are not only beneficial to the environment but also cost effective in order to fulfill the strict quality standards regarding environmental protection that are currently being developed. Therefore, there is a critical need to find out the suitable treatment method to treat the sugarcane industry.

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Last few decades, various treatment methods have been proposed to treat wastewater include adsorption, biological process, ozonation, photocatalysis, electro-oxidation and coagulation/flocculation. Coagulation/flocculation is widely used for various wastewater treatment, as it is efficient and simple to operate compared to another methods. Aluminium and iron salts are widely used as coagulants in chemical coagulation wastewater treatment for removing a broad range of impurities from wastewater, including colloidal particles and dissolved organic substances. The mode of action is generally explained in terms of two distinct mechanisms; charge neutralization in negatively charged colloids by cationic hydrolysis products and incorporation of impurities in an amorphous hydroxide precipitate (sweep flocculation). But, nowadays plant based coagulants to treat wastewater called natural coagulation is also experienced as an effective method treat industrial wastewater. The main advantage of natural coagulation is non-toxic, easy to handle, higher removal efficiency and formation of biodegradable sludge [1-3].

In coagulation/flocculation process, many factors can influence its efficiency, such as the type and dosage of coagulant/flocculant, pH, mixing speed and settling time and temperature etc. Optimization of these process parameters considerably enhance the treatment efficiency. In conventional multi-parameter experiments, optimization of process is usually carried out by varying single parameter while keeping all other parameters constant at a specific set of location. This is not only time-consuming, but also usually lacking ability of understanding the accurate optimum set of parameters, due to ignoring the relations among process parameters. Response Surface Methodology (RSM) has been predictable to conclude the influences of individual process parameter and their interactive effects on the responses namely efficiency includes removal of organic matters. The RSM is a statistical technique for designing experiments, building models, evaluating the effects of several factors and searching optimum circumstances for desirable responses. RSM with Box-Behnken Design (BBD) can be used to study the interactions of process parameters on treatment efficiency with a limited number of planned experiments. The advantage of BBD is each factor or independent variable, is placed at one of three equally spaced values, usually coded as -1, 0, +1. BBD has been widely used for optimization of various wastewater treatment techniques such as adsorption, electro-Fenton and electrocoagulation and membrane separation.

Hence the purpose of this study was to investigate and optimize the individual and the interactive effect of process variables such as rotation speed, mass of coagulant and settling time on the removal of colour, turbidity and COD from vinasse wastewater from sugarcane industry using Box-Behnken response surface design coupled with Derringer's desired function methodology. The different type of coagulation/flocculation process namely Chemical Coagulation (CC) and Natural Coagulation (NC) were employed. Aluminium sulfate ($\text{Al}_2(\text{SO}_4)_3 \cdot 6\text{H}_2\text{O}$) was used as a chemical coagulant and Tanfloc (vegetable originated low molecular weight cationic organic polymer which may be act as coagulant, flocculant, auxiliary flocculation agent) was used as the natural coagulant. Also, in order to find out the economic viability of the coagulation/flocculation process, the operating cost of the each individual process was calculated to treat unit vinasse [4].

Materials and Methods

Vinasse was collected from sugarcane industry (Maringa, Parana, Brazil) and were stored at 4°C prior to the experiments. The characteristics of vinasse wastewater. Aluminium sulfate ($\text{Al}_2(\text{SO}_4)_3 \cdot 6\text{H}_2\text{O}$) with analytical grade was used in chemical coagulation. Tanfloc SG was used as a coagulant in natural coagulation (TABLE 1) [5].

TABLE 1. Characteristics of vinasse wastewater sugarcane industry.

Vinasse wastewater	
Characteristics	Value
pH	4.65
Colour (pt-co unit)	54600
Turbidity (FAU)	10400
Chemical Oxygen Demand (mg/L)	65000

Coagulation/flocculation process

Coagulation/flocculation studies for vinasse wastewater from sugarcane industry was carried out in batch reactor with chemical and natural coagulants. 200 ml of composite vinasse wastewater was taken in 250 ml beaker with varying coagulant dose. The experiments were carried out with various rotational speed and settling time. The samples were agitated 100 rpm for 1 min and 40 rpm for 30 min. Then samples were allowed to settle for various times. Then supernatant portion of vinasse wastewater in beaker was used for determination of colour, turbidity and COD. Replicates of experiments were performed to conform the reproducibility [6].

Analytical methods

Initial pH, colour, COD and turbidity of the vinasse is determined using American Public Health Association (APHA) standard methods. The removal efficiency (R, %) was calculated using as follows

$$R = \frac{Y_0 - Y}{Y_0} \times 100$$

Where, R is removal efficiency (%), Y_0 and Y were initial and final values of colour, turbidity and COD.

Box-Behnken experimental Design (BBD)

In this work, Response Surface Methodology (RSM) with Box-Behnken Response Surface Experimental Design (BBD) was used to optimize and investigate the influence of process variables such as rotational speed, coagulant dose and settling time on the treatment of vinasse wastewater for each chemical and natural coagulation. The process variables were designed with three factors at three levels. Then 17 experiments with five centre points (used to estimate the experimental error) were carried out for each coagulation process. The total number of experiments was calculated from the following equation

$$N = 2K(K - 1) + C_0$$

Where, K is number of factors and C_0 is the number of central point. Then results obtained were fitted in a second-order polynomial equation to compare the connection between process variables and removal efficiency of colour, turbidity and COD. Here, linear, quadratic and interactive effect of the process variables were examined. The general mathematical form of equation is given in following equation

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \sum_{i < j=2}^k \sum \beta_{ij} X_i X_j + e_i$$

where, Y is the response; X_i and X_j are variables (i and j range from 1 to k); β_0 is the model intercept coefficient; β_j , β_{ij} and β_{ij} are interaction coefficients of linear, quadratic and the second-order terms, respectively; k is the number of independent parameters ($k=3$ in this study); and e_i is the error. Then the developed mathematical models was analyzed using actual versus predicted plot, ANOVA and R^2 . Also, the models were used for the draw a contour plots to estimate the relationship between process variables and responses. Derringer's desired function methodology was used to find out the optimal conditions. All the statistical analyses were carried out with Statease design expert 8.0.7.1 statistical software package [7,8].

Results

Chemical coagulation and natural coagulation is carried out to treat vinasse wastewater from sugarcane industry using aluminium sulfate and Tanfloc, respectively. The treatment efficiency is determined using removal of colour, turbidity and COD with respect to coagulation process variables such as rotational speed, mass of coagulant and settling time [9].

Chemical coagulation

In chemical coagulation process, stirring speed is one of the most significant variable that affects the coagulation, strongly. So, the effect of stirring speed on the chemical coagulation process is investigated by varying stirring speed in the range of 10 rpm-50 rpm with pH of 4.5, Aluminium sulfate of 1 g/L and settling time of 20 min. The removal of efficiency of colour, turbidity and COD from vinasse is increased upto 30 rpm. Thereafter, removal of efficiency of colour, turbidity and COD from vinasse is significantly decreased. This can explained by the fact that elevated stirring speed breaks the bond between the chemical coagulant and organic matters present in the vinasse wastewater. Coagulant dose is also an key process variable to control the reaction rate in chemical coagulation to treat vinasse. In order to investigate the effect of coagulant dose on the chemical coagulation process, experiments were carried out by varying coagulant dose in the range of 0.8 g/L-3.4 g/L with pH of 4.5, stirring speed 10 rpm and settling time of 15 min (FIG. 1) [10].

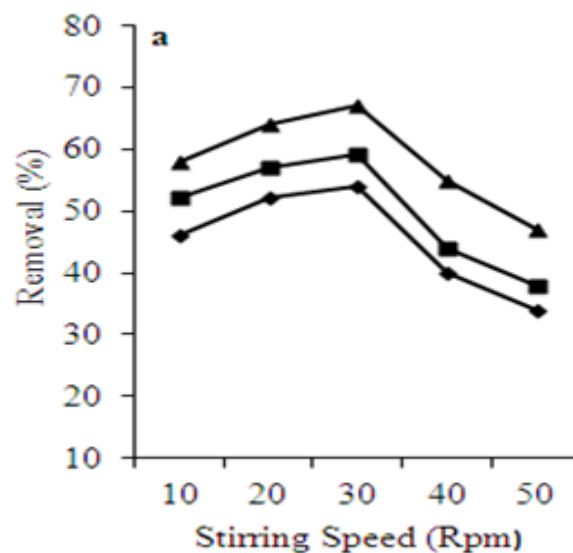


FIG. 1. Effect of stirring speed in pH of 4.5, Aluminium sulfate of 1 g/L and settling time of 20 min. Note: ◆ Turbidity removal, ■ Colour removal, ▲ COD removal

From the results, it is found that removal of colour, turbidity and COD from vinasse are increased with increasing coagulant dose due to the production of large amount of $Al(OH)_3(s)$. However it is noticed that increasing coagulant dose beyond 2.8 g/L shows significant reduction on the percentage of colour, turbidity and COD from vinasse. In coagulation process, the settling time is an important issue which can influences the overall removal efficiencies of colour, turbidity and COD from vinasse. To conclude the effective settling time, experiments are carried out at different settling time of (5 min-25 min) with pH of 4.5, Aluminium sulfate of 1 g/L and stirring speed of 10 rpm (FIG. 2) [11].

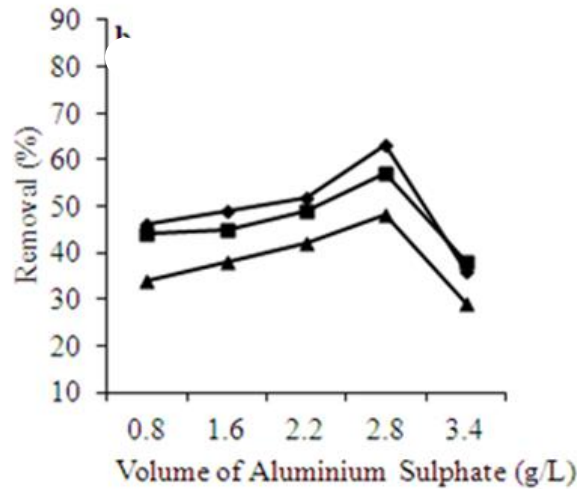


FIG. 2. Effect of coagulant dose in pH of 4.5, stirring speed 10 rpm of 1 g/L and settling time of 15 min. Note: ◆ Turbidity removal, ■ Colour removal, ▲ COD removal

The results indicated that, the percentage removal of colour, turbidity and COD from vinasse are increased with increasing settling time upto 20 min and then did not show any significant change in removal efficiencies. The aggregation of flocs increases up to 20 min, in which most of the flocs are settle down, thus removal efficiencies are increased during coagulation process (FIG. 3) [12].

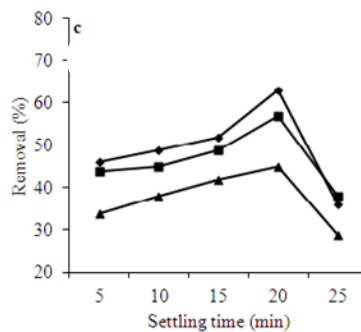


FIG. 3. Effect of settling time pH of 4.5, Aluminium sulfate of 1 g/L and stirring speed of 10 rpm. Note: ◆ Turbidity removal, ■ Colour removal, ▲ COD removal

Natural coagulation

Stirring speed is an important process variable influencing the performance of coagulation process, considerably. Therefore, it is necessary to study about the role of stirring speed on natural coagulation to treat vinasse. The experiments are carried out at various

stirring speed (10 rpm-50 rpm) while other parameters such as pH (4.5), coagulant dose (60 mL) and settling time (60 min) were kept as constant and the results are shown graphically in (FIG. 4) [13].

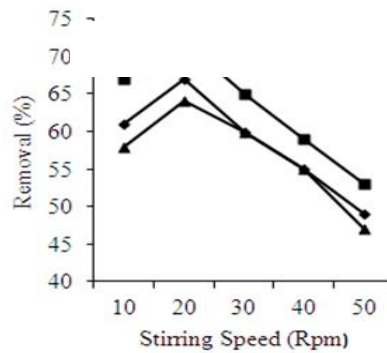


FIG. 4. Effect of stirring speed in pH (4.5), coagulant dose (60 mL) and settling time (60 min). Note: ◆ Turbidity removal, ■ Colour removal, ▲ COD removal

From the results, it is found that the percentage of colour, turbidity and COD removal from vinasse is increased with increasing stirring speed and reaches to a maximum level when stirring speed is equal to 20 rpm. Thereafter, the percentage of colour, turbidity and COD removal from vinasse is decreased due to the negative effect of higher stirring speed on the bond between coagulant and organic matters present in the vinasse. Coagulant dose is a crucial variable to determine the percentage removal of colour, turbidity and COD in vinasse wastewater. In order to evaluate coagulant dose effect, experiments were performed at various ranges of Tanfloc dose (20 mL/L-100 mL/L), while other parameters such as pH (4.5), stirring speed (20 rpm) and settling time (60 min) were kept as constant [14].

The percentage colour, turbidity and COD in vinasse wastewater are increased with increasing tanfloc dose upto 70 mL/L, further increase in Tanfloc dose did not show any significant effect on coagulation. During earlier stage, Tanfloc can effectively react with the organic and inorganic pollutants present in the vinasse wastewater, due to the availability of more number reaction sites, which can increase the removal efficiency. Beyond this dosage, the coagulation process attains an equilibrium stage and did not improve the efficiency of coagulation. Settling time is also an important parameter in coagulation process to treat vinasse. In order to find out the effective settling time on coagulation process, settling time is varied in the range of 15 min-75 min while other parameters such as pH (4.5), stirring speed (20 rpm) and Tanfloc dose (60 mL/L) were kept as constant (FIG. 5).

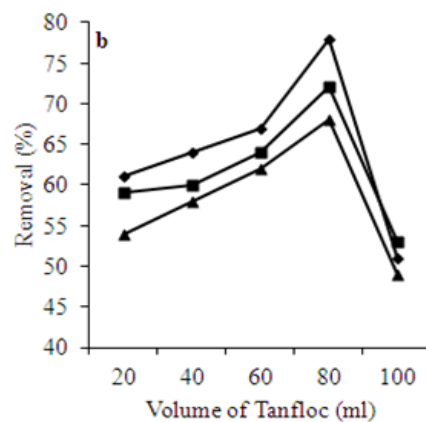


FIG. 5. Effect of coagulant dose in pH (4.5), stirring speed (20 rpm) and settling time (60 min). Note: ◆ Turbidity removal, ■ Colour removal, ▲ COD removal

The results indicates that, the pertantage removal of colour, turbidity and COD in vinasse wastewater are increased with increasing settling time upto 50 min. After 50 min of settling time did not show any significant effect on coagulation (FIG. 6).

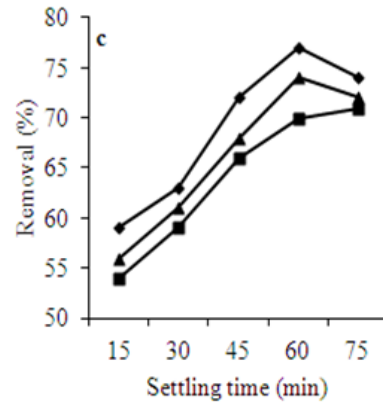


FIG. 6. Effect of settling time in pH (4.5), stirring speed (20 rpm) and Tanfloc dose (60 mL/L). Note: ◆ Turbidity removal, ■ Colour removal, ▲ COD removal

Mathematical model development

Based on the above results, the coagulation process variables ranges are selected and shown (TABLE 2).

TABLE 2. Ranges of coagulation process variables and their levels.

Variable	Symbol	Chemical coagulation			Symbol	Natural coagulation		
		-1	0	1		-1	0	1
Stirring speed	A	10	30	50	D	10	30	50
Coagulant dose	B	0.8	2.1	3.4	E	20	60	100
Settling time	C	5	15	25	F	15	45	75

Then BBD design of experiments are carried out. The results obtained from BBD experiments are evaluated by multiple regression analysis method and empirical relationship between the response and independent variables has been expressed by a second-order polynomial equation with interaction terms. Six second-order polynomial equations are developed to understand the interactive correlation between the responses and process variables (TABLE 3).

TABLE 3. BBD experimental results.

Chemical coagulation			Natural coagulation		
Y ₁	Y ₂	Y ₃	Y ₄	Y ₅	Y ₆
25.33	38.04	13.54	67.75	70.91	44.97
29.12	32.54	19.58	67.75	70.91	44.97

29.44	30.24	18.54	67.75	70.91	44.97
42.38	38.65	32.38	30.95	67.23	30.89
39.48	32.48	29.48	14.35	65.57	41.83
32.48	19.58	21.86	53.55	69.49	32.97
36.05	30.24	26.05	46.55	68.79	37.89
38.74	30.54	28.74	23.95	66.53	47.95
32.2	34.58	22.58	43.55	66.5	27.89
42.26	38.45	32.26	67.75	70.91	44.97
37.45	29.95	26.84	23.95	66.53	32.89
36.78	20.26	26.78	67.75	71.49	44.97
42.78	38.65	33.58	63.55	70.49	37.65
42.86	30.28	32.86	53.35	69.47	20.89
32.87	27.54	21.68	20.95	66.23	37.99
42.46	38.65	32.46	53.55	69.49	27.89
42.44	38.65	33.84	50.35	69.17	32.89

The final model obtained in terms of coded factors are given below

$$Y_1=42.46+0.12A+0.54B+4.93C+2.61AB+3.49AC+0.59BC-3.50A^2-3.81B^2-4.78C^2$$

$$Y_2=38.61+4.48A+1.01B+0.32C-2.83AB-4.61AC+0.057BC -6.07A^2-4.31B^2-3.00C^2$$

$$Y_3=32.90+0.17A+0.59B+5.21C+2.82AB+3.78AC+0.40BC-4.00A^2-3.96B^2-5.33C^2$$

$$Y_4=67.75-3.62A-0.42B+4.85C+17.90AB-9.15AC-5.95BC-25.05A^2-7.15B^2-9.60C^2$$

$$Y_5=71.03-0.36A+0.21B+0.73C+1.79AB-0.91AC-1.09BC-2.31A^2-1.02B^2-1.27C^2$$

$$Y_6=44.97+2.98A+1.05B+3.99C-7.76AB+4.25AC-1.19BC-5.36A^2-7.46B^2-3.43C^2$$

Where, Y_1 , Y_2 and Y_3 are colour removal, turbidity removal and COD removal in chemical coagulation. Y_4 , Y_5 and Y_6 are colour removal, turbidity removal and COD removal in natural coagulation.

Pareto Analysis of Variance (ANOVA) are used to analyze the BBD experimental data using F and p-values. The higher F values and lower p-values ($p < 0.0001$) of the developed mathematical models indicated that, the developed model is highly significant. The goodness of fit of the model is evaluated using Co-efficient of Variance (CV), PRESS and Adequate Precision (AP) (TABLE 4).

TABLE 4. ANOVA results of responses.

Source	Y1		Y2		Y3		Y4		Y5		Y6	
	F value	P value	F value	P value	F value	P value	F value	P value	F value	P value	F value	P value
Model	260	<0.0001	247.43	<0.0001	51.22	<0.0001	58.26	<0.0001	59.24	<0.0001	415.23	<0.0001
A	0.52	0.4942	609.37	<0.0001	0.18	0.6862	9.94	0.0161	8.81	0.0209	272.59	<0.0001
B	10.85	0.0132	30.93	0.0008	2.19	0.1827	0.14	0.7226	2.85	0.1352	33.9	0.0006
C	897.61	<0.0001	3.09	0.1223	169.34	<0.0001	17.79	0.0039	36.09	0.0005	488.89	<0.0001
AB	125.84	<0.0001	121.72	<0.0001	24.68	0.0016	121.15	<0.0001	107.4	<0.0001	925.76	<0.0001

AC	224.57	<0.0001	322.28	< .0001	44.57	0.0003	31.66	0.0008	28.06	0.0011	277.03	<0.0001
BC	6.36	0.0397	0.05	0.829	0.5	0.503	13.39	0.0081	40	0.0004	21.77	0.0023
A2	238.36	<0.0001	590.16	<0.0001	52.35	0.0002	249.76	<0.0001	188.9	<0.0001	465.36	<0.0001
B2	282.02	<0.0001	296.66	<0.0001	51.5	0.0002	20.35	0.0028	36.83	0.0005	899.99	<0.0001
C2	443.35	<0.0001	144.1	<0.0001	93.17	0.0001	36.68	0.0005	56.61	0.0001	190.67	<0.0001
C.V.%	1.27		1.59		4.25		6.76		0.5		1.37	
PRESS	22.15		29.02		111.78		1184.8		9.48		29.14	
AP	47.16		47.51		20.88		21.36		21.49		69.07	

The adequacy of developed mathematical models is evaluated by constructing diagnostic plots namely predicted versus actual plot. In diagnostic plots such as predicted versus actual the data points on this plot lie very close to the diagonal line which indicates a good adequate (R²) agreement between experimental data and the data predicted by the developed models ((FIG. 7-FIG. 12).

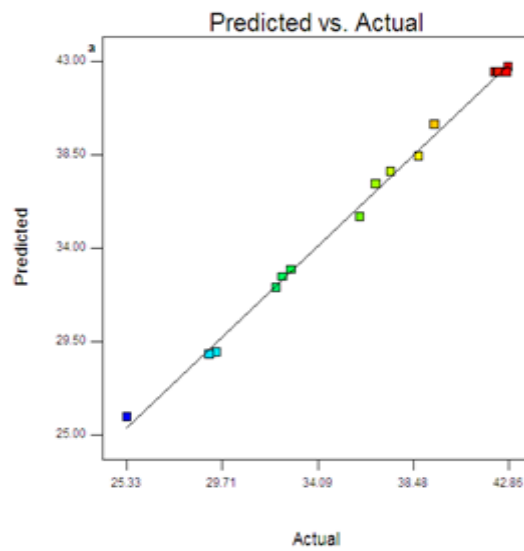


FIG. 7. Predicted versus actual for responses for chemical coagulation-colour removal.

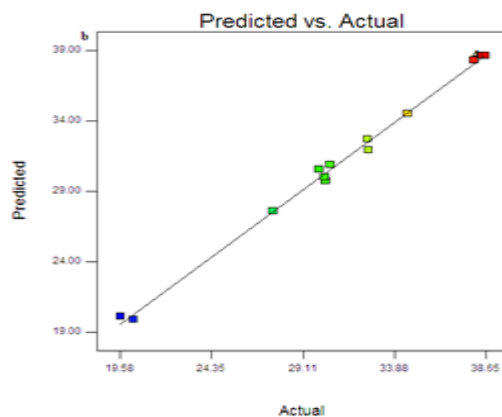


FIG. 8. Predicted versus actual for responses for chemical coagulation-Turbidity removal.

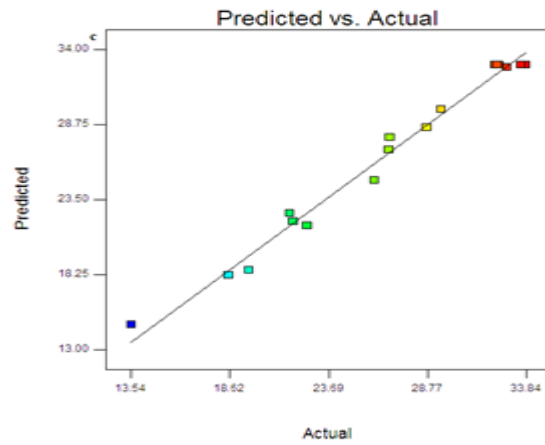


FIG. 9. Predicted versus actual for responses for chemical coagulation-COD removal.

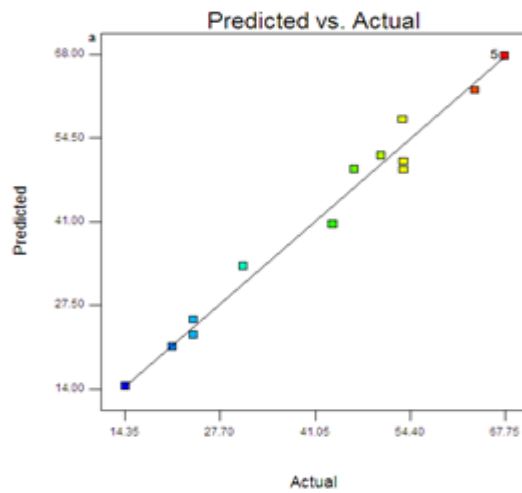


FIG. 10. Predicted versus actual for responses for natural coagulation-colour removal.

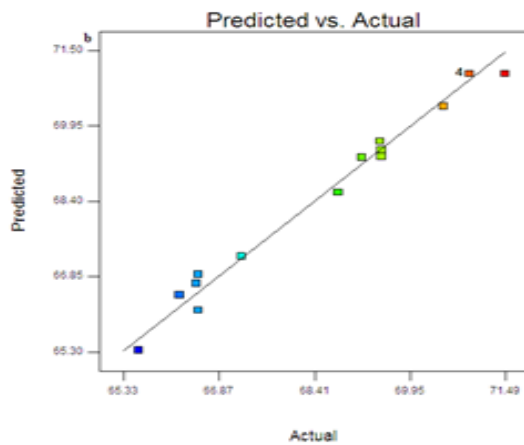


FIG. 11. Predicted versus actual for responses for natural coagulation-turbidity removal.

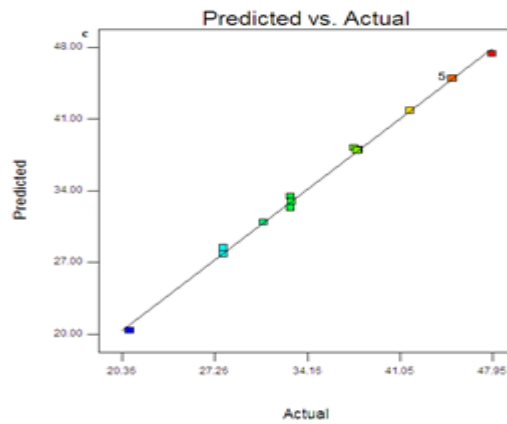


FIG. 12. Predicted versus actual for responses for natural coagulation-COD removal.

Optimization and cost evaluation

In order to find out the optimum process variables, simultaneous optimization of the multiple responses is carried out using Derringer’s desired function methodology. This numerical optimization technique evaluates a point that maximizes the desirability function. The optimal conditions to obtain the maximum removal of colour, turbidity and COD are determined for each coagulation and its cost to treat unit vinasse also calculated. The results indicate that natural coagulation is the effective and economically suitable technique to treat vinasse compared to chemical coagulation. The picture of raw vinasse wastewater, after chemical coagulation and after natural coagulation is shown in (FIG.13) (TABLE 5).

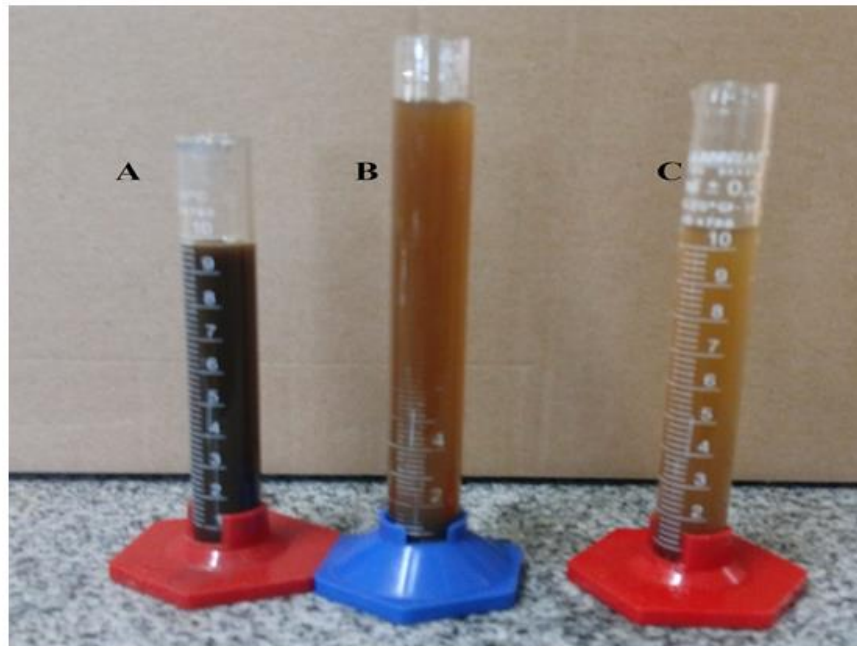


FIG. 13. Picture of vinasse wastewater; a) Raw, b) After chemical coagulation, c) After natural coagulation.

TABLE 5. Removal efficiency and cost evaluation of coagulation process.

Responses	Chemical coagulation	Natural coagulation
Colour removal (%)	43	67

Turbidity removal (%)	38	71
COD removal (%)	33	46
Cost evaluation (US \$/m ³)	0.9	1.2

Note: **CC:** Stirring speed of 40 rpm, coagulant dose of 2 g/L and settling time of 20 min; **NC:** Stirring speed of 30 rpm, coagulant dose 60 ml of and settling time of 55 min; 1kg of aluminium sulfate=0.4 US \$ and 1kg of tanfloc=1.1 US \$.

Discussion

In this study, treatment of vinasse wastewater from sugarcane industry is investigated using Chemical Coagulation (CC) and Natural Coagulation (NC). Aluminium sulfate ($\text{Al}_2(\text{SO}_4)_3 \cdot 6\text{H}_2\text{O}$) is used as a chemical coagulant and Tanfloc is used as the natural coagulant. The effects of rotation speed, mass of coagulant dose and settling time on the removal of colour, turbidity and COD in vinasse are investigated using Box-Behnken Design (BBD) of experiments [15].

Conclusion

The results showed that NC could be effectively reduced the colour, turbidity and COD at the optimum conditions compared to CC. Quadratic models are developed for predicting the responses such as colour, turbidity and COD removal. Optimum set of the independent variables is obtained by Derringer's desired function methodology. These results indicated that natural coagulation process can be applied as a primary treatment method for the vinasse wastewater from sugarcane industry.

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