

Performance evaluation of chemical coagulation process to treat bagasse wastewater: modeling and optimization

K. Thirugnanasambandham¹, V. Sivakumar^{2*}, K. Shine³

¹Excel College of Engineering and Technology, Department of Chemistry, Komarapalayam, Namakkal-637303, TN, India

²Anna University, Department of Chemical Engineering, AC Tech Campus, Chennai-600025, TN, India

³King Saud University, Department of Botany and Microbiology, College of Science, Post box 2455, Riyadh 11451, Saudi Arabia

*Corresponding author: e-mail: drvsivakumar@yahoo.com

In this present study, chemical coagulation process (CC) treatment process was investigated under different conditions such as pH, ferric chloride dose, agitation time and settling time to treat bagasse wastewater using response surface methodology (RSM). The outcomes were evaluated using Pareto analysis of variance (ANOVA) and second order polynomial models were created with the aim of being able to predict the responses. Ideal conditions were observed to be as per the following: agitation time of 25 min, pH of 7, ferric chloride dose of 6 g/L and settling time of 60 min. Under these conditions, turbidity removal of 62%, COD removal of 67%, TDS removal 53% and sludge production of 32 mL/L were obtained with operating cost of 3.50 Rupee/L. The mechanism of CC was analyzed using XRD spectrum and founds to be adsorption.

Keywords: bagasse wastewater, chemical coagulation, ferric chloride, Box-Behnken design, sludge, operation cost, optimization.

INTRODUCTION

Nowadays, water is the crucial part in any and all paper industries. It is used almost in every step of paper making procedure, which certainly picks up the unwanted and sometimes harmful wastes¹. Therefore, paper industries are considered to be the main pollution threats in India with concern to wastewater regulation, since these produce huge quantity of waste streams that are dangerous to the ecological system². The United States Environmental Protection Agency (USEPA) and the Central Pollution Control Board of India (CPCB) made a stringent regulations to discharge the paper industry wastewater³. However, there is still a greater need to decrease the amount of pollutants from paper wastewaters by using the newer methods for clean and safe industrial processes⁴. Most paper industries in India use wood as the main raw material and bagasse as an alternative potential raw material for the paper manufacture, due to the limited availability of wood. Bagasse is the fibrous residue remaining after sugarcane crushed to extract their juice and is a “waste” from the sugar industries. In addition of some advantages in uses of bagasse for producing pulp, there are few practical complications in the form of collection and storing of bagasse due to its seasonal availability. Bagasse stored under wet condition by spraying water to preserve its quality, which uses large quantity of water and produce an effluent called bagasse wastewater⁵. Discharge of this wastewater causes negative impact on receiving water bodies. Therefore, there is a crucial need to design the environment friendly and economically viable method to treat bagasse wastewater.

Last few decades, treatment operations include adsorption, oxidation, ozonation, photocatalysis, biological and chemical coagulation were in various wastewater treatment plant (WTP). Each technique has its own merits and demerits in application⁶. As of late, Chemical coagulation treatment procedure has warranted a significant amount of consideration for providing high removal proficiency in the treatment of wastewater; this

procedure can be specifically practiced on waste waters to eliminate organics as well as suspended solids⁷. In essence, chemical coagulation (CC) is one of the emerging technique for eliminating the colloidal types of pollution from waste water and for decreasing chemical oxygen demand (COD). This method comprises of destabilizing colloids, amassing and binding them to each other into flocculates; the flocs that occur can at long last be isolated either by settling or by flotation⁸. The destabilization process entails initially a raise in ionic strength which encourages dual-layered pressure, and/or the balance of the molecule surface charge by adsorbing counter anions, making use of the inclusion of chemicals known as coagulants. During the process of wastewater treatment, the normally utilized coagulants are hydrolysable metal salts; these are efficient for the process of destabilizing colloidal particles, as almost all colloids are charged negatively in ordinary waters and industrial effluents: the coagulant for the most part was ferric chloride (FeCl₃) that are readily soluble in water. Meanwhile, many factors can influence CC treatment efficiency, such as the agitation time, coagulant dose, pH and settling time. Optimization of these process variables significantly increase the process efficiency with reasonable operating cost⁹.

In conventional multi-factor tests, optimization is generally performed by changing single variant whereas keeping all other variants constant¹⁰. It is time-taking and usually incompetent of realization the true optimum, because of ignoring the interactive effect among process parameters. Response surface methodology (RSM) is a statistical strategy for experimental design, structuring models, assessing the impacts of different variants, and looking for optimal conditions for attractive responses¹¹. However, to the best of our knowledge, chemical coagulation treatment technique to treat bagasse wastewater using RSM has not yet reported in literature. Therefore, in this research work the efficiency of CC process to treat bagasse wastewater was investigated under different conditions such as agitation time, pH, ferric chloride

dose and settling time using Box-Behnken design (BBD). Finally, the quality of produced sludge and operating cost was examined for its practical implementation.

MATERIAL AND METHODS

Wastewater was collected from a local paper industry TamilNadu, India and it was stored in 4°C in order to avoid the changes in composite wastewater. Analytical reagent grade chemicals such as Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used, which was supplied by local suppliers, Erode.

A conventional batch studies were performed under various operating conditions include agitation time, coagulant dose, initial pH and settling time in 2.5L of beaker containing 1.6L of composite wastewater. Magnetic stirrer was used to agitate the sample. After the treatment, sample was centrifuged at 6500 rpm for 15 min (Remi R-24 Centrifuge, India) and the supernatant was filtered through a 0.45 µm filter papers and the filtrate was collected. Then, it was used to analyze the reduction in turbidity, chemical oxygen demand (COD) and total dissolved solids (TDS). Each trials was carried out in triplicates and the outcomes were exhibited as the mean value of the triplicates

Physico-chemical properties of the wastewater such as pH, turbidity, COD and TDS were find out according to the APHA standard procedure explained in elsewhere¹². The removal efficiency was calculated as follows¹³

$$RE = \left(\frac{c_0 - c_e}{c_0} \right) \times 100 \quad (1)$$

where, c_0 and c_e is the initial and final concentrations of turbidity, COD and TDS, respectively. Operating cost of CC process was calculated using unit price of $FeCl_3 \cdot 6H_2O$ price (0.58 Rupee/g) according to Merck chemicals, Chennai.

In this recent research, Box-Behnken response surface design (BBD) with four components at three different levels was utilized for optimization and examine the impact of procedure variables, for example, agitation time (A), pH (B), ferric chloride dose (C) and settling time (D) on chemical coagulation (CC) treatment process to obtain a maximum treatment efficiency from bagasse wastewater. Process factors and their levels (Table 2) were find out based on the preliminary studies¹⁴. Experimental runs were prepared based on a BBD and the design consists of 29 trials with five center points were designed. Then the Design-Expert 8.0.7.1 (State-Ease Inc., Minneapolis, MN, USA) statistical package was used for the statistical

Table 2. Process variables and their ranges

Characteristics	Bagasse wastewater
pH	7.5
Turbidity	1768 NTU
Chemical oxygen demand [COD]	6500 mg/L
Total dissolved solids [TDS]	4525 mg/L

Table 1. Characteristics of bagasse wastewater

Process variables	Level		
	-1	0	1
Agitation time [A, min]	15	25	35
Initial pH [B]	6	8	10
Ferric chloride dose [C, g/L]	2	4	6
Settling time [D, min]	20	40	60

calculations. The correlation between the responses and four independent factors were assessed by building up the second order polynomial scientific models and the summed up type of mathematical equation was given as follows¹⁵.

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

Sufficiency of created model was studied by the pareto analysis of variance (ANOVA). Regression coefficients of these second order polynomial models and their effects were analyzed with the help of the F-values at probability levels ($p \leq 0.05$). The experimental data were analysed with various statistical analysis such as determination coefficient (R^2), adequate precision (AP) and coefficient of variation (CV) to emulate the statistical significance of the developed polynomial equations¹⁶. After that, the individual and interactive impacts of treatment parameters on reaction were concentrated on 3D response surface graph from created numerical models. Optimization of treatment factors for the most effective treatment productivity was completed by Derringer's desired function strategy utilizing numerical optimizing technique¹⁷.

RESULTS AND DISCUSSION

In this work, evaluation of chemical coagulation (CC) process to treat bagasse wastewater was investigated under various operating conditions using statistical method RSM coupled with BBD for the maximum efficiency of the bagasse wastewater treatment. BBD experimental design consists of 29 experiments were carried out and the results were shown in Table 3. The responses presented in Table 3 were correlated with the four independent factors utilizing a polynomial mathematical statement, Eq. (2). Least squares regression was employed to correlate with the acquired result to Eq. (2). The best fit models in the coded factors as follows:

$$Y_1 = 67.84 - 2.32A + 2.80B + 0.38C + 21.56D + 2.25AB - 1.64AC - 6.51AD - 8.24BC + 1.85BD - 1.83CD - 19.86A^2 - 19.50B^2 - 5.93C^2 - 19.85D^2 \quad (3)$$

$$Y_2 = 72.26 - 2.26A + 2.80B + 0.40C + 21.60D + 2.22AB - 1.65AC - 6.51AD - 8.25BC + 1.95BD - 1.83CD - 19.82A^2 - 19.41B^2 - 6.00C^2 - 19.85D^2 \quad (4)$$

$$Y_3 = 51.42 - 1.24A - 2.23B - 4.96C - 17.28D - 1.26AB - 1.45AC - 1.49AD - 10.42BC + 2.18BD + 6.33CD - 17.67A^2 - 17.46B^2 - 11.87C^2 - 15.00D^2 \quad (5)$$

$$Y_4 = 43.00 - 0.83A + 0.33B - 0.92C + 3.25D - 0.25AB - 0.25AC - 2.00AD - 1.25BC + 0.50BD - 2.75CD - 6.50A^2 - 7.00B^2 - 4.13C^2 - 5.88D^2 \quad (6)$$

Where Y_1 , Y_2 , Y_3 and Y_4 are turbidity removal (%), COD removal (%), TDS removal (%) and sludge production (mL/L) respectively. ANOVA evaluations of these models, imply that the developed models can describe the chemical coagulation process significantly. Adequacy of the developed model fit the experimental values, the parameters F-value, R^2 , p-value, CV and AP values were used¹⁸. As can be seen in results, F-values of four responses such as Y_1 , Y_2 , Y_3 and Y_4 implied that the developed quadratic model was significant. Also, each term in the models are also examined for significance¹⁹. A p-value smaller than 0.05 implies that

Table 3. BBD experimental design with results

Run	A	B	C	D	Y ₁	Y ₂	Y ₃	Y ₄
1	25	6	6	40	48.51	52.95	32.12	32
2	25	6	4	20	8.54	12.94	4.56	28
3	15	6	4	40	31.53	35.98	15.12	30
4	15	10	4	40	32.54	36.96	16.12	31
5	15	8	4	20	2.52	6.92	3.71	27
6	35	10	4	40	27.64	32.32	11.22	29
7	25	10	6	40	37.58	41.93	21.12	30
8	25	8	2	20	16.32	20.68	2.65	28
9	35	8	6	40	42.54	46.95	26.12	32
10	25	8	4	40	67.84	72.26	51.42	43
11	25	10	2	40	52.59	56.94	36.12	34
12	25	6	4	60	45.24	49.67	28.82	31
13	25	8	6	20	17.54	21.92	1.12	29
14	25	8	4	40	67.84	72.26	51.42	43
15	35	8	4	60	40.52	44.96	34	30
16	25	8	4	40	67.84	72.26	51.42	43
17	35	8	2	40	42.54	46.93	16.24	32
18	25	8	6	60	61.87	66.28	55.42	33
19	25	8	4	40	67.84	72.26	51.43	43
20	25	10	4	20	10.54	14.95	5.24	28
21	15	8	2	40	40.78	45.14	15.25	32
22	35	6	4	40	17.64	22.45	15.25	29
23	35	8	4	20	12.55	16.96	3.56	28
24	25	6	2	40	30.54	34.98	5.42	31
25	25	10	4	60	54.64	59.48	38.22	33
26	25	8	2	60	67.98	72.34	31.61	43
27	15	8	4	60	56.54	60.94	40.12	37
28	25	8	4	40	67.86	72.26	51.42	43
29	15	8	6	40	47.34	51.76	30.92	33

the corresponding model term is highly significant. From ANOVA results, it is clear that the linear terms for A, B, C and D have large effects on responses, due to high F-values. The p-values <0.0001 show that there is only a 0.01% chance that a model F-value this large is the product of noise in the conducted experiments²⁰. In addition, the linear term for B and D is also significant but with a smaller effect of A and C on the response Y₄, due to its smaller F-value. However, the linear term for the independent variables except A has a large F-value and a p-value <0.05 for Y₁–Y₃. The quadratic terms for all the responses are not significant but with F-values smaller than their corresponding linear terms. But, the interactive term of BD for all the responses is significant and other terms are not significant²¹. These results indicate that the suggested model is an appropriate description of the chemical coagulation (CC) process. The obtained regression equations and coefficient of determination were evaluated to test the fit of developed models. Actual values are data for each specific run from Table 3, and predicted values are created by the model, Eq. (3–6). In experimental design, R² is a calculation of amount of variation around the mean explained by the developed models. On the other hand, a large value of R² can be misleading if the model contains extraneous terms²². By adding factors to the models, correlation coefficient (R²) always increases whether the added factor is significant or not. The data in Figure 2 clearly shows that the adjusted determination coefficient is very high to prove the high significance of the developed mathematical models. The goodness of fit of the model was also evaluated by the adequate precision (AP) and coefficient of variance (CV), which clearly stated that, the differences between experimental and predicted values are low and confirms the reliability of the conducted experiment²³. The obtained results indicate that the developed mathematical model has the ability to explain the chemical coagulation process very robustly.

3D response surface graphs were created to evaluate the interactive and individual effects of factors for the maximize the bagasse wastewater treatment efficiency²⁴, which were shown in Figures 1–2.

Mixing time is one of the major factor for the treatment of bagasse wastewater using CC process, which is associated with treatment efficiency. In order to investigate the effect of agitation time on CC process, experiments were carried out various agitation time (15, 25 and 35 min) and results are shown in Figure 1. The result showed that turbidity removal, COD removal, TDS removal and sludge production are increased with agitation time upto 30 min. This phenomenon could be described by fact that, at higher agitation time increases the collisions between coagulant and the colloidal particle present in the wastewater, thus treatment efficiency is decreased²⁵. Beyond the agitation time of 30 min shows the negligible effect on the treatment efficiency of CC process for the bagasse wastewater.

pH is one of the crucial factor, which affects the CC process to treat bagasse wastewater. So that, experiments were done to investigate the effect of pH (6, 8 and 10) over the CC treatment process and the results was presented in Figure 1. The result showed that, the turbidity removal, COD removal, TDS removal and sludge production are increased linearly with increasing pH from 6–8. This because of the increase in pH would increase the Fe(OH)₃ species, which has the strong affinity towards colloidal particle in the bagasse wastewater to be treated, which leads to the increase in treatment efficiency²⁶. Thereafter, there is a formation of Fe(OH)₄[–] which is insoluble in wastewater, thus treatment efficiency is decreased beyond pH of 7.

Ferric chloride dose is also an important factor influences the CC process to treat bagasse wastewater. Therefore, in this study influence of ferric chloride on the CC treatment process was examined by varying its range (2, 4 and 6 g/L) and the results are illustrated in

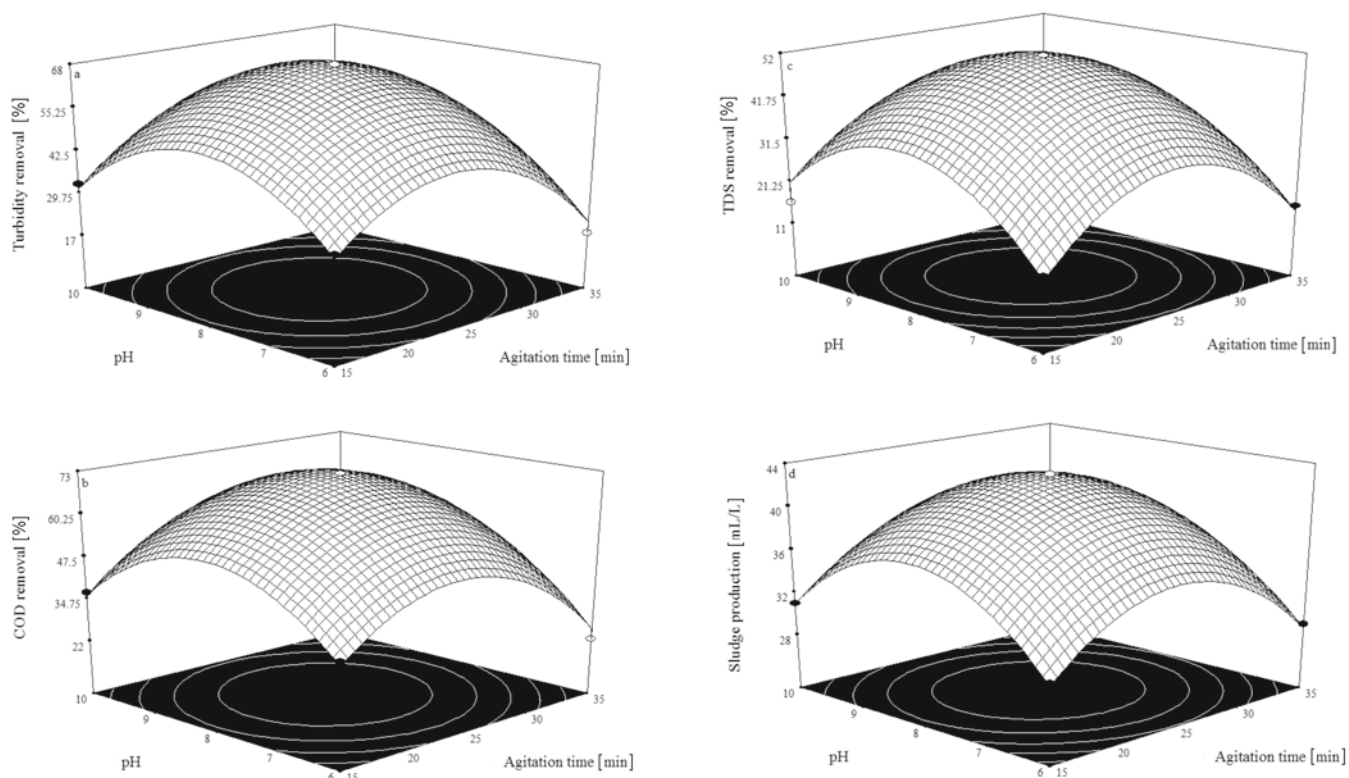


Figure 1. Response surface plots representing the effect of process variables (A & B) on CC process

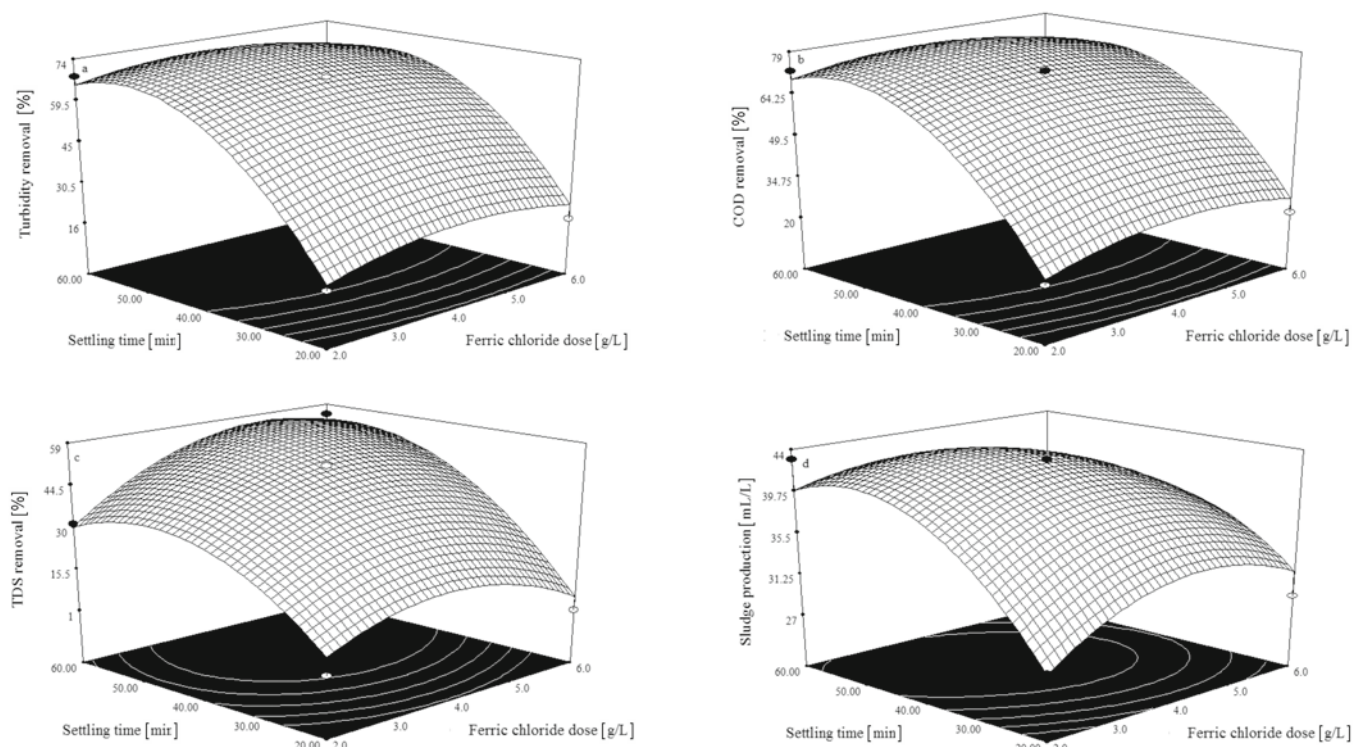


Figure 2. Response surface plots representing the effect of process variables (C & D) on CC process

Figure 2. The result showed that turbidity removal, COD removal, TDS removal and sludge production are increased with the increasing ferric chloride dose throughout the experiment. It is because of the increase in ferric chloride dose increases the formation of larger active $\text{Fe}(\text{OH})_3$ species which increase the treatment efficiency²⁷.

Efficiency of CC process to treat bagasse wastewater is extremely affected by settling time and its influence are investigated by varying settling time (20, 40 and 60 min) and the results are depicted in Figure 2. The

result showed that, turbidity removal, COD removal, TDS removal and sludge production are increased with the increasing settling time upto 50 min and it can be described by the fact that increase in settling time increases the formation of larger flocs, thus efficiency of CC process is increased. Beyond the settling time of 50 min shows the decreased treatment efficiency due to the negative impact of formation or aggregation of flocs²⁸.

Concurrent optimization of the various responses was done making use of Derringer's desired function proce-

ture so as to investigate the ideal operating conditions for the most efficient treatment of CC method to treat bagasse wastewater^{29, 30}. This will enhance any mix of one or more objectives; these could be either process factors or responses^{31, 32}. The conceivable objectives are: minimize, maximize, within range, target, none (for just responses alone) and tuned to a specific value (only variables). In this current research, objectives of the process parameters were chosen as in an extent and the response objective was chosen as maximize; with the exception of sludge generation. This numerical optimization method assesses a point that boosts the attractive capabilities and ideal conditions were observed to be as per the following: agitation time of 25 min, pH of 7, ferric chloride dose of 6 g/L and settling time of 60 min. Under these conditions, turbidity removal of 62%, COD removal of 67%, TDS removal 53% and sludge production of 32 mL/L were obtained with operating cost of 3.50 Rupee/L with a desirability value of 0.999. Subsequently, the correctness of ideal conditions for anticipating ideal responses values are tested taking into account the aforementioned conditions. Triplicate analyses were done under the optimized conditions and the mean values (< 3% error) gotten from real experiment, exhibited the approval of the optimized parameters³³.

To confirm the mechanism of CC process, the composition of the sludge produced by chemical coagulation was analyzed using X-ray diffraction (XRD) spectrum at the optimum process conditions. As seen in Figure 3, the strongest peaks appeared were identified to be adsorption of organic matters on to the $\text{Fe}(\text{OH})_3$. At pH 7 amorphous $\text{Fe}(\text{OH})_3$ formed adsorb negatively charged ions and this is beneficial for a rapid adsorption of soluble compounds and colloids particles. This is the main reason for the chemical coagulation process.

CONCLUSIONS

In this study, BBD response surface design (BBD) was used to investigate and optimize the chemical coagulation (CC) process parameters includes agitation time, pH, ferric chloride dose and settling time on the treatment of bagasse wastewater. Second order polynomial models were developed for predicting the responses with high correlation coefficient values (R^2). Optimum set of the independent variables was attained by derringer's desired

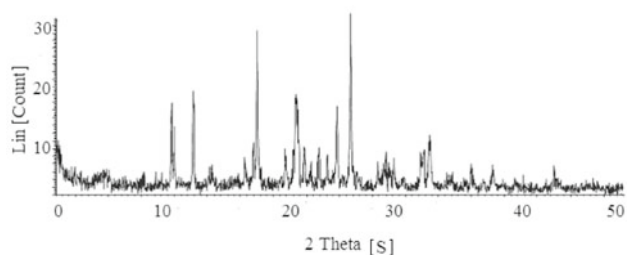


Figure 3. Experimental conversion-time profiles at different temperatures and CO_2 concentrations for CaO from calcined CaCO_3 and original CaO

function methods and it was found to be: agitation time of 25 min, pH of 7, ferric chloride dose of 6 g/L and settling time of 60 min. Under these conditions, turbidity removal of 62%, COD removal of 67%, TDS removal

53% and sludge production of 32 mL/L were obtained with operating cost of 3.50 Rupee/L. The properties of sludge was analyzed using EDAX spectrum and it shows the higher content of trace metals.

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