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Chitosan based grey wastewater treatment—A statistical design approach

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ABSTRACT

In this present study, grey wastewater was treated under different operating conditions such as agitation time (1–3 min), pH (2.5–5.5), chitosan dose (0.3–0.6 g/l) and settling time (10–20 min) using response surface methodology (RSM). Four factors with three levels Box–Behnken response surface design (BBD) were employed to optimize and investigate the effect of process variables on the responses such as turbidity, BOD and COD removal. The results were analyzed by Pareto analysis of variance (ANOVA) and second order polynomial models were developed in order to predict the responses. Under the optimum conditions, experimental values such as turbidity (96%), BOD (91%) and COD (73%) removals are closely agreed with predicted values.

1. Introduction

Grey wastewater is recognized as one of the wastewater, which corresponds to wastewater produced in bathtubs, showers, hand basins, laundry machines and kitchen sinks in households, office buildings and schools. Moreover, grey wastewater is estimated to account for about 75% of the combined residential sewage (Eriksson, Auffarth, Eilersen, Henze, & Ledin, 2003). Discharge of untreated grey wastewater to ecosystem causes harmful effect to living organisms and human beings. Therefore, there is an increasing interest in the treatment of grey wastewater in many parts of the world due to its high amount of production compared to industrial activities. Many new grey wastewater treatment technologies such as physico-chemical methods (Diaper et al., 2010; Friedler, Kovalio, & Galil, 2005; Gross, Shmueli, Oron, Ronen, & Raveh, 2007) and biological treatment technology options include membrane bioreactor (Jefferson, Laine, Stephenson, & Judd, 2001; Lesjean & Gnirss, 2006), rotating biological contactor (Friedler et al., 2005; Nolde, 1999) and constructed wetland (Dallas & Ho, 2005) were developed. But, these technologies were not sufficient to treat grey wastewater due to large variation in grey wastewaters composition.

Chitosan, a nitrogenous polysaccharide composed mainly of poly(β-1-4)-2-amino-2-deoxy-d-glucopyranose, is produced through the deacetylation of chitin, which is widely spread among marine and terrestrial invertebrates (Ngah & Fatinathan, 2006). Chitosan is well established as an excellent natural adsorbent/coagulant for wastewater treatment due to the presences of the amino ($-NH_2$) and hydroxyl ($-OH$) groups. These groups serve as the coordination and reaction sites for effective treatment process (Chang & Juang, 2004). Apart from that, chitosan has also other useful features such as being polycationic, non-toxic, biodegradable, good adsorption capability and anti-bacterial properties (Ngah, Ghani, & Kamari, 2005). Chitosan has been used to treat various industrial wastewaters such as textile wastewater, rice mill wastewater and egg processing industry wastewater (Thirugnanasambandham, Sivakumar, & Prakash Maran, 2013a). An extensive literature analysis shows that no research report is available for the treatment of grey wastewater using chitosan.

In recent years, wastewater treatment studies have been carried out to determine the feasibility and to optimize the operating parameters of technologies with response surface methodology (RSM), because it decreases the usage of raw material and reduces the time. RSM is an important branch of experimental design and a critical technology in developing new processes, optimizing their performance, and improving design and formulation of new products. The most popular class of RSM is Box–Behnken design (Sridhar, Sivakumar, Prakash Maran, & Thirugnanasambandham, 2013). BBD is an effective design that is ideal for sequential experimentation and allows a reasonable amount of information for testing lack of fit while not involving an unusually large number

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of design points. Last few decades, RSM has been used to optimize the wastewater treatment processes such as deproteinated whey wastewater (Guven, Perendeci, & Tanyolac, 2008), industrial paint wastewater (Korbahti, Aktas, & Tanyolac, 2007) textile dye wastewater (Korbahti, 2007), electrochemical removal of mercury ions from wastewater (Madaria, Mohan, Rajagopal, & Garg, 2004), sodium from fermented food composts (Baek, Shin, Lee, Jun, & Yang, 2002) and chromium from industrial wastewater (Rana, Mohan, & Rajagopal, 2004).

However, to our best knowledge, no publications are available on the treatment of grey wastewater using chitosan through RSM. Hence, the main objective of the present study was to investigate and optimize the individual and the interactive effect of process variables such as agitation time, pH, chitosan dose and settling time on the maximum removal of turbidity, biological oxygen demand (BOD) and chemical oxygen demand (COD) from grey wastewater using Box–Behnken response surface design (BBD) coupled with Derringer's desired function methodology.

2. Materials and methods

2.1. Raw materials

Grey wastewater was collected from Erode region, Tamil Nadu, India and used as raw material. The characteristics of grey wastewater were shown in Table 1. All the chemicals used in this were analytical grade. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) having the purity of 95% were supplied by Merck chemicals, Chennai. Distilled water was used to prepare a 0.1 N solutions of HCl and NaOH. Chitosan (powder) of AR grade was purchased from Sigma chemicals, Mumbai.

2.2. Methods

Experiments were carried out by method described by Thirugnanasambandham, Sivakumar, and Prakash Maran (2013) under the various operating conditions such as agitation speed (1–3 min), pH (2.5–5.5), chitosan dose (0.3–0.6 g/l) and settling time (10–20 min). The measurement of turbidity, BOD and COD was done according to APHA standard methods (Sridhar, Sivakumar, Prince Immanuel, & Prakash Maran, 2011).

2.3. Experimental design

In this study, Box–Behnken response surface experimental design (BBD) with four factors at three levels was used to optimize and investigate the influence of process variables such as agitation time (1–3 min), pH (2.5–5.5), chitosan dose (0.3–0.6 g/l) and settling time (10–20 min on the removal of turbidity, BOD and COD from grey wastewater. After selection of process (independent) variables, experiments were established based on a BBD and the complete design consists of 29 experiments with five centre points (used to estimate the experimental error). For predicting the optimal point after performing experiments, a second-order polynomial equation was fitted to correlate the relationship between independent variables and responses, which accounts for variations caused by linear, quadratic and interactive effect of the process variables. Regression coefficients of linear, quadratic, and interaction involved in the model and their effects were analyzed by Pareto analysis of variance (ANOVA) and all the terms in the model were tested by student's *F*-test and significance of the *F*-values at probability levels ($p \leq 0.05$) were analyzed (Prakash Maran, Manikandan, Thirugnanasambandham, Vigna Nivetha, & Dinesh, 2013a).

The experimental data were analyzed with various statistical analyses, such as sum of squares (SS), determination coefficient (R^2), adjusted determination of coefficient (R_a^2), predicted determination of coefficient (R_p^2), coefficient of variation (CV) and adequate precision (AP). All the statistical analyses were done with the help of Stat ease Design Expert 8.0.7.1 statistical software package (Stat-Ease Inc., Minneapolis, USA) and the developed mathematical models were used for the construction of three dimensional (3D) response surface plots in order to predict the relationships between independent and dependent variables. Meanwhile, the optimization process variables were carried out by derringer's desired function methodology (Prakash Maran, Sivakumar, Sridhar, & Prince Immanuel, 2013b).

This numerical optimization technique will optimize any combination of one or more goals; these may be either process variables or responses and in this study, goals of the process variables were selected as in a range and the response goals were selected as maximum. After optimization, adequacy of the model equation for predicting the optimum response values was validated under the optimal conditions. Verification experiments were performed under the optimal conditions and the value of the experiments was compared with the predicted values of the developed model equations. The detailed methodology used in this experimental design was described in elsewhere (Sridhar, Sivakumar, Prince Immanuel, & Prakash Maran, 2012).

Table 1
Characteristics of grey wastewater.

Composition of grey wastewater	
pH	5.78
Turbidity (NTU)	35
BOD (mg/l)	328
COD (mg/l)	646

3. Results and discussion

In this study, four factors with three levels BBD were used to evaluate the effect and optimize the process variables on the responses such as turbidity, BOD and COD. A total number of 29 batch experiments including five centre points were carried out in triplicates using statistically deigned experiments (BBD) and the results are shown in Table 2.

3.1. Experimental design analysis

In this study, BBD experimental data were analyzed by two different tests namely the sequential model sum of squares and model summary statistics in order to obtain regression models and decide about the adequacy of various models (linear, interactive, quadratic and cubic) to represent treatment process effectively and the results are listed in Table 3. From the results, it was found that, linear and interactive (2FI) models are exhibited lower – R^2, adjusted – R^2, predicted – R^2 and also having high *p*-values, when compared with quadratic model. Cubic model is found to be aliased (Maran, Sivakumar, Sridhar, & Thirugnanasambandham, 2013). Therefore the quadratic model is chosen to describe the effects of process variables on the grey wastewater treatment process using chitosan.

3.2. Mathematical model fitting

An empirical relationship between the response and independent variables has been developed by a second-order polynomial equation with interaction terms was fitted between the experimental results obtained on the basis of BBD which will help to predict the removal efficiency of different sets of combinations of four process variables on the responses. Three empirical models were developed to understand the interactive correlation between

Table 2
BBD and their experimental results.

S.No	Agitation time (A)	Initial pH (B)	Chitosan dose (C)	Settling time (D)	Turbidity removal (%)	BOD removal (%)	COD removal (%)
1	2.00	2.5	0.60	15	75.46	77.16	51.84
2	2.00	2.5	0.45	10	40.39	37.06	11.74
3	1.00	2.5	0.45	15	63.39	56.87	35.08
4	1.00	5.5	0.45	15	64.39	61.4	36.08
5	1.00	4.0	0.45	10	27.58	31.12	5.80
6	3.00	5.5	0.45	15	59.49	55.80	30.48
7	2.00	5.5	0.60	15	69.39	66.34	41.02
8	2.00	4.0	0.30	10	48.17	45.12	19.80
9	3.00	4.0	0.60	15	74.39	71.26	45.94
10	2.00	4.0	0.45	15	88.54	80.24	71.32
11	2.00	5.5	0.30	15	84.39	81.34	56.02
12	2.00	2.5	0.45	20	77.09	74.04	48.72
13	2.00	4.0	0.60	10	49.39	46.06	20.74
14	2.00	4.0	0.45	15	88.54	80.24	71.32
15	3.00	4.0	0.45	20	72.39	69.32	44.00
16	2.00	4.0	0.45	15	88.54	80.24	71.32
17	3.00	4.0	0.30	15	74.39	71.12	45.80
18	2.00	4.0	0.60	20	93.69	90.64	65.32
19	2.00	4.0	0.45	15	88.54	80.24	71.32
20	2.00	5.5	0.45	10	42.39	39.26	13.94
21	1.00	4.0	0.30	15	72.63	69.16	43.84
22	3.00	2.5	0.45	15	49.49	46.06	20.74
23	3.00	4.0	0.45	10	44.39	41.34	16.02
24	2.00	2.5	0.30	15	62.39	59.16	33.84
25	2.00	5.5	0.45	20	86.49	83.44	58.12
26	2.00	4.0	0.30	20	95.48	90.54	71.74
27	1.00	4.0	0.45	20	88.39	85.32	60.00
28	2.00	4.0	0.45	15	88.54	80.24	71.32
29	1.00	4.0	0.60	15	79.19	76.02	50.70

Table 3
Multi regression analysis of responses.

Source	Sum of Squares	Df	Mean Square	*F*-Value	Prob > *F*	Remarks
Sequential model sum of squares for turbidity removal						
Mean	143,156.16	1.00	143,156.16			
Linear	5847.20	4.00	1461.80	9.30	0.0001	
2FI	513.02	6.00	85.50	0.47	0.8201	
Quadratic	3131.97	4.00	782.99	85.55	<0.0001	Suggested
Cubic	83.93	8.00	10.49	1.42	0.3429	Aliased
Sequential model sum of squares for BOD removal						
Mean	127,932.89	1.00	127,932.89			
Linear	5526.35	4.00	1381.59	11.70	<0.0001	
2FI	475.35	6.00	79.23	0.60	0.7232	
Quadratic	2260.84	4.00	565.21	81.80	<0.0001	Suggested
Cubic	43.23	8.00	5.40	0.61	0.7498	Aliased
Sequential model sum of squares for COD removal						
Mean	56,843.12	1.00	56,843.12			
Linear	5791.77	4.00	1447.94	5.62	0.0025	
2FI	501.04	6.00	83.51	0.26	0.9465	
Quadratic	5566.43	4.00	1391.61	160.50	<0.0001	Suggested
Cubic	99.22	8.00	12.40	3.36	0.0787	Aliased

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	Remarks
Model summary statistics for turbidity removal						
Linear	12.5385	0.6078	0.5424	0.4713	5086.67	
2FI	13.4580	0.6611	0.4729	0.2652	7068.80	
Quadratic	3.0253	0.9867	0.9734	0.9233	738.26	Suggested
Cubic	2.7144	0.9954	0.9786	0.3383	6365.97	Aliased
Model summary statistics for BOD removal						
Linear	10.8645	0.6611	0.6046	0.5254	3967.20	
2FI	11.4445	0.7180	0.5613	0.3204	5681.29	
Quadratic	2.6286	0.9884	0.9769	0.9333	557.56	Suggested
Cubic	2.9861	0.9936	0.9701	0.0783	7704.32	Aliased
Model summary statistics for COD removal						
Linear	16.0583	0.4834	0.3973	0.3414	7890.16	
2FI	17.7761	0.5252	0.2615	0.1099	10,664.40	
Quadratic	2.9445	0.9899	0.9797	0.9416	699.47	Suggested
Cubic	1.9220	0.9981	0.9914	0.7336	3191.71	Aliased

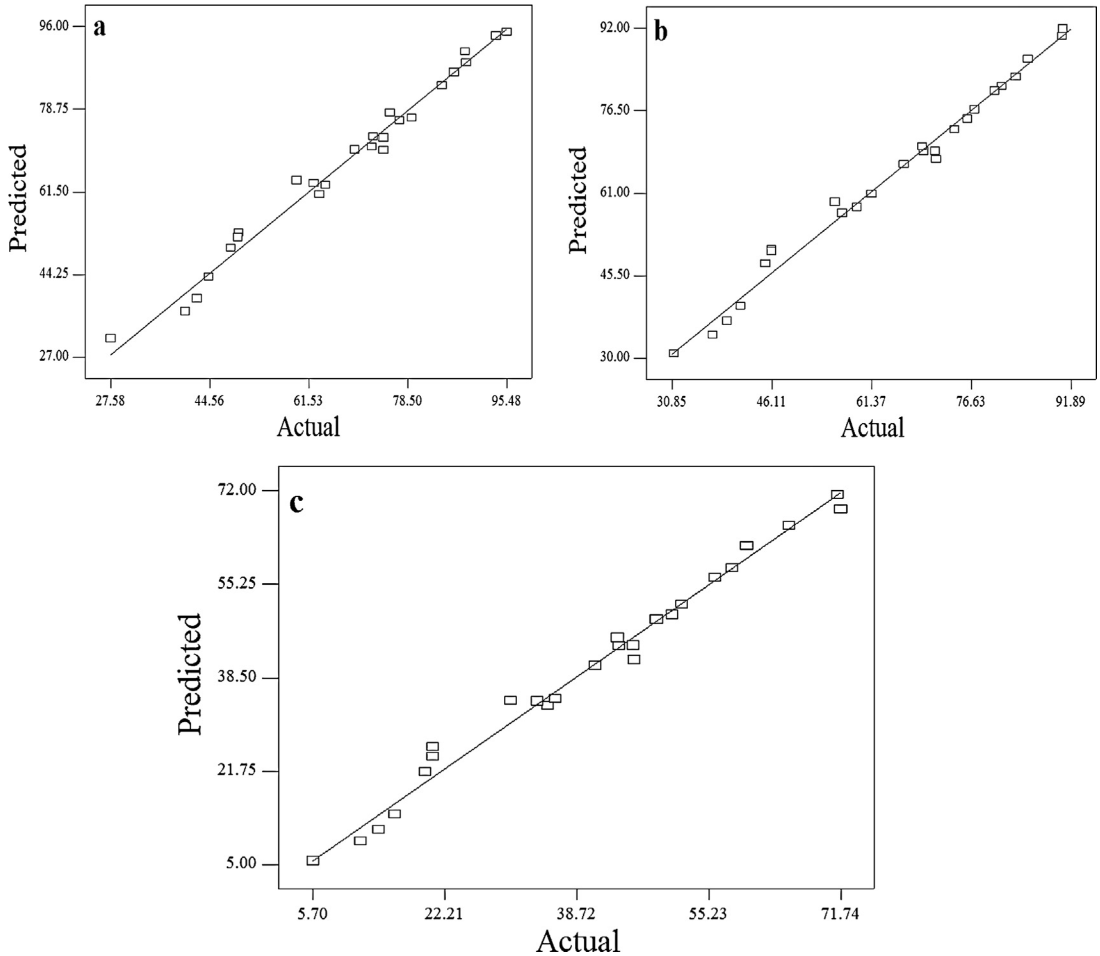

Fig. 1. Diagnostic plots for the model adequacy. (a) Turbidity removal; (b) BOD removal; and (c) COD removal.

the responses and process variables. The final model obtained in terms of coded factors are given below

$$\text{Turbidity removal}\ (\%) = 88.54 - 1.75A + 3.20B + 0.34C + 21.76D + 2.25AB - 1.64AC - 8.20AD - 7.02BC + 1.85BD - 0.75CD - 14.46A^2 - 13.88B^2 - 0.85C^2 - 14.99D^2 \quad (1)$$

$$\text{BOD removal}\ (\%) = 80.24 - 2.08A + 3.10B + 0.92C + 21.11D + 1.30AB - 1.68AC - 6.56AD - 8.25BC + 1.80BD - 0.21CD - 11.81A^2 - 11.42B^2 - 1.83C^2 - 12.00D^2 \quad (2)$$

$$\text{COD removal}\ (\%) = 71.32 - 2.38A + 2.81B + 0.38C + 21.65D + 2.18AB - 1.68AC - 6.56AD - 8.25BC + 1.80BD - 1.84CD - 19.99A^2 - 19.60B^2 - 5.97C^2 - 19.81D^2 \quad (3)$$

where A, B, C and D are agitation time, pH, chitosan dose and settling time respectively.

3.3. Adequacy of developed mathematical models

The adequacy of developed mathematical models was evaluated by constructing diagnostic plots such as predicted versus actual and normal probability plots for the experimental data obtained from this study and it is shown in Figs. 1 and 2. Diagnostic plots such as predicted versus actual (Fig. 1a–c) help us to find out the relationship between predicted and experimental values and evaluate the model suitability. From the Fig. 1a–c, it was observed that, the data points on this plot lie very close to the diagonal line indicating a fair agreement between experimental data and the values predicted by the developed models. Normal probability plot is a suitable graphical method for judging residuals normality. The observed residuals are plotted against the expected values, as they lie reasonably close on a straight line and show no deviation of variance (Fig. 2a–c). This can confirm the normal distribution of the observed data and adequacy of the developed models (Prakash Maran, Sivakumar, Thirugnanasambandham, & Sridhar, 2013).

3.4. Statistical analysis

Pareto analysis of variance (ANOVA) was used to analyze the experimental data statistically. Significance of the developed model equations was evaluated by their corresponding F and p-values and it is shown in Table 4. The higher model F values and lower p-values ($p < 0.0001$) of developed models demonstrated that, the developed models were highly significant. The goodness of fit of the models was evaluated by the determination co-efficient (R^2), adjusted determination co-efficient (R_a^2), predicted determination

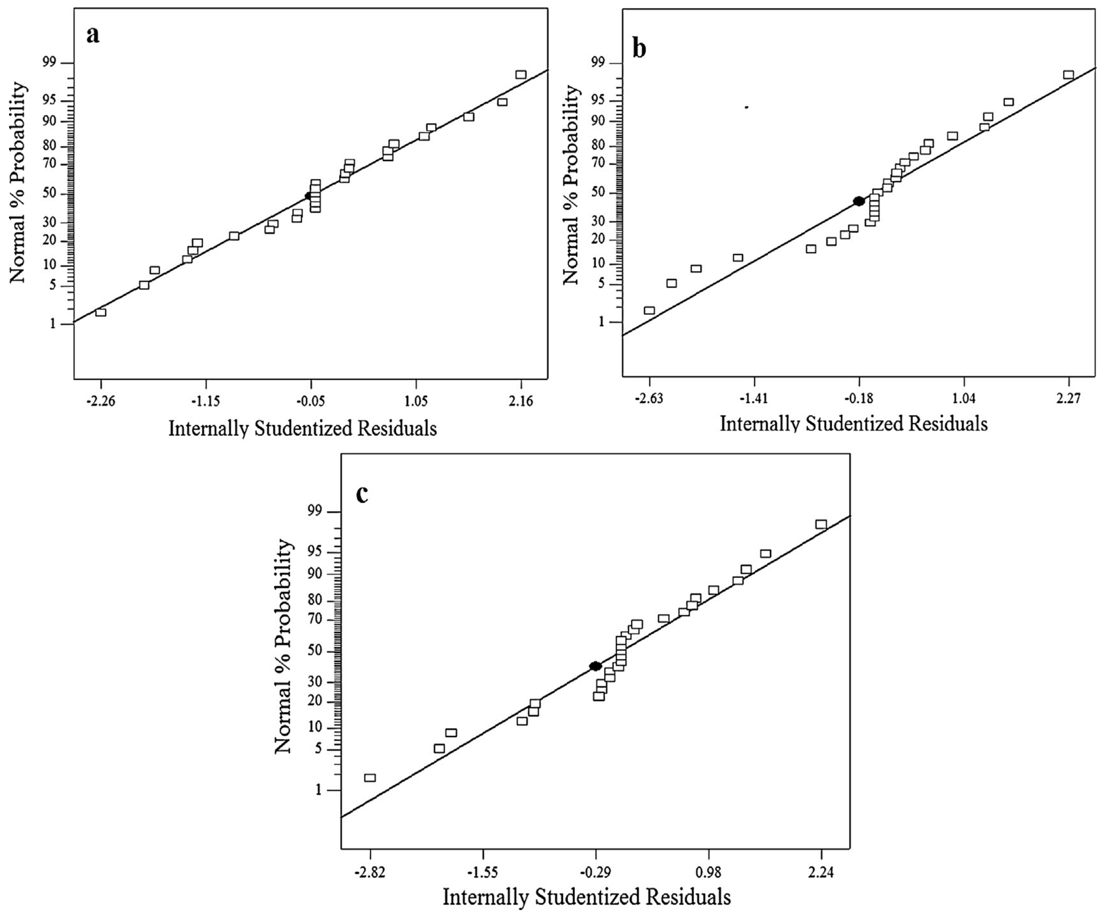

Fig. 2. Normal probability plot for responses. (a) Turbidity removal; (b) BOD removal; and (c) COD removal.

co-efficient (R_p^2), co-efficient of variance (CV) and adequate precision (AP). The high R^2, R_a^2 and R_p^2 values revealed that, the models are statistically significant and only small variations are not explained by the models. Lower CV values of models clearly stated that, the deviations between experimental and predicted values are low and AP values showed a high degree of precision and reliability of the conducted experiments (Beg, Sahai, & Gupta, 2003). These results indicate the best fitness of the developed mathematical models.

3.5. *Effect of process variables*

Three dimensional (3D) response surface plots were plotted from the developed mathematical models in order to study the individual and interactive effects among the process variables on the responses and also used to determine the optimal condition of each factor for higher removal of turbidity, BOD and COD from grey wastewater using chitosan.

Table 4
ANOVA table for responses.

Responses	Turbidity removal		BOD removal		COD removal	
	F-Value	*p*-Value	*F*-Value	*p*-Value	*F*-Value	*p*-Value
Model	74.08	<0.0001	85.42	<0.0001	97.70	<0.0001
A	4.03	0.0645	7.53	0.0158	7.82	0.0143
B	13.39	0.0026	16.73	0.0011	10.92	0.0052
C	0.15	0.7050	1.47	0.2459	0.20	0.6652
D	621.02	<0.0001	773.82	<0.0001	648.81	<0.0001
AB	2.21	0.1591	0.98	0.3384	2.20	0.1600
AC	1.17	0.2969	1.63	0.2221	1.30	0.2733
AD	29.40	<0.0001	24.88	0.0002	19.83	0.0005
BC	21.52	0.0004	39.40	<0.0001	31.40	<0.0001
BD	1.50	0.2415	1.88	0.1924	1.49	0.2417
CD	0.25	0.6266	0.03	0.8753	1.56	0.2319
A^2	148.10	<0.0001	130.92	<0.0001	298.84	<0.0001
B^2	136.49	<0.0001	122.40	<0.0001	287.28	<0.0001
C^2	0.51	0.4854	3.15	0.0979	26.69	0.0001
D^2	159.27	<0.0001	135.23	<0.0001	293.47	<0.0001

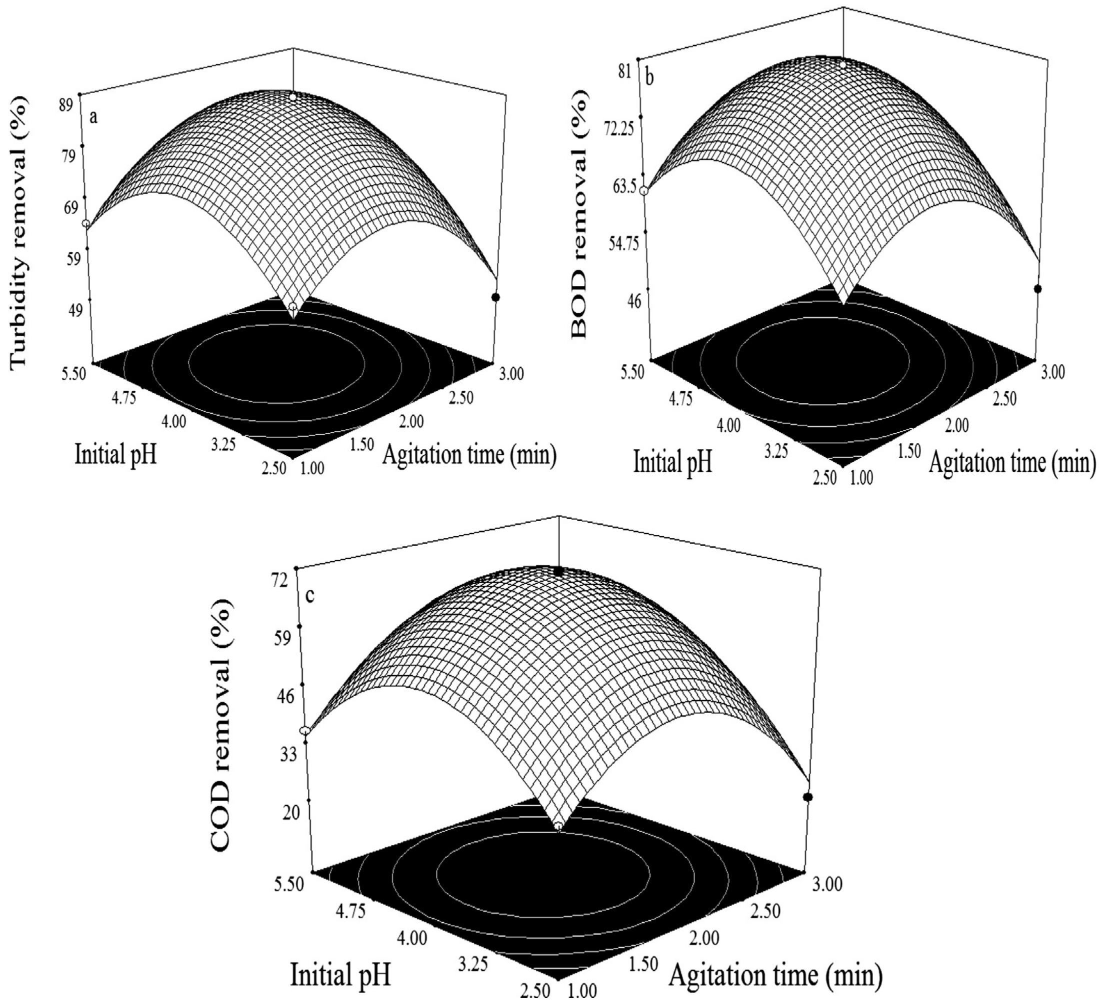

Fig. 3. Effect of process variables (A and B) on responses.

3.5.1. Effect of agitation time

In order to investigate the effect of agitation time on turbidity, BOD and COD removals, the experiments were carried out on different agitation time and the results were shown in Fig. 3. From Fig. 3a–c, it was found that the removal of turbidity, BOD and COD were increased with increasing agitation time from 1 to 2 min. This is explained by the fact that the mechanical agitation increased the specific surface area of chitosan for effective removal efficiency. Further increase in the agitation time, decreases removal efficiencies of turbidity, BOD and COD due to the creation of disturbance for the bond formation between chitosan and organic matters present in the wastewater (Thirugnanasambandham, Sivakumar, & Prakash Maran, 2013b).

3.5.2. Effect of pH

To examine the effect of pH on treatment process, experiments were carried out at various pH (2.5–5.5) and the results are given in Fig. 3. From Fig. 3a–c, it was found that the removal efficiencies of turbidity, BOD and COD were increased with increasing pH up to 4. This can be explained by the fact that in the pH range of 4, there is a formation of $-NH_3^+$ ions which effectively adsorbs the negatively charged organic matters in the grey wastewater; thus the removal efficiencies of turbidity, BOD and COD were increased (Thirugnanasambandham et al., 2013b). Thereafter, there is a drastic decrease in removal efficiencies, during the treatment process due to the solubility of chitosan decreases beyond pH 4 and shows the lower removal efficiencies (Baisali, Chakrabarti, Vijaykumar, & Vijay, 2006). This result indicates that pH is an important parameter and it significantly influences the performance of treatment efficiency.

3.5.3. Effect of chitosan dose

To understand the effect of chitosan dose on the removal efficiencies of turbidity, BOD and COD, a series of experiments were performed by varying a chitosan dose in the range of 0.4–0.6 g/l and the results are shown graphically (3D) in Fig. 4. From Fig. 4a–c, it was found that the removal efficiencies of turbidity, BOD and COD were increased with increasing chitosan dose (Gregorio & Pierre-Marie, 2008) throughout the experiment. This is explained by the fact that more amount of chitosan creates the prominent reaction sites for effective treatment process; thus the removal efficiencies of turbidity, BOD and COD were increased.

3.5.4. Effect of settling time

Settling time is an important factor because it influences the removal efficiencies of turbidity, BOD and COD in grey wastewater treatment. To investigate the effective settling time, serious tests were recorded and the results are shown graphically (3D) in Fig. 4. From Fig. 4a–c, it was found that the removal efficiencies of turbidity, BOD and COD were increased with increasing settling time up to 15 min, further increase in settling time shows the negligible effect. This is due to the fact that the longer settling time creates the more compact, high strength and larger flocs by bridging mechanism; thus removal efficiencies of turbidity, BOD and COD were

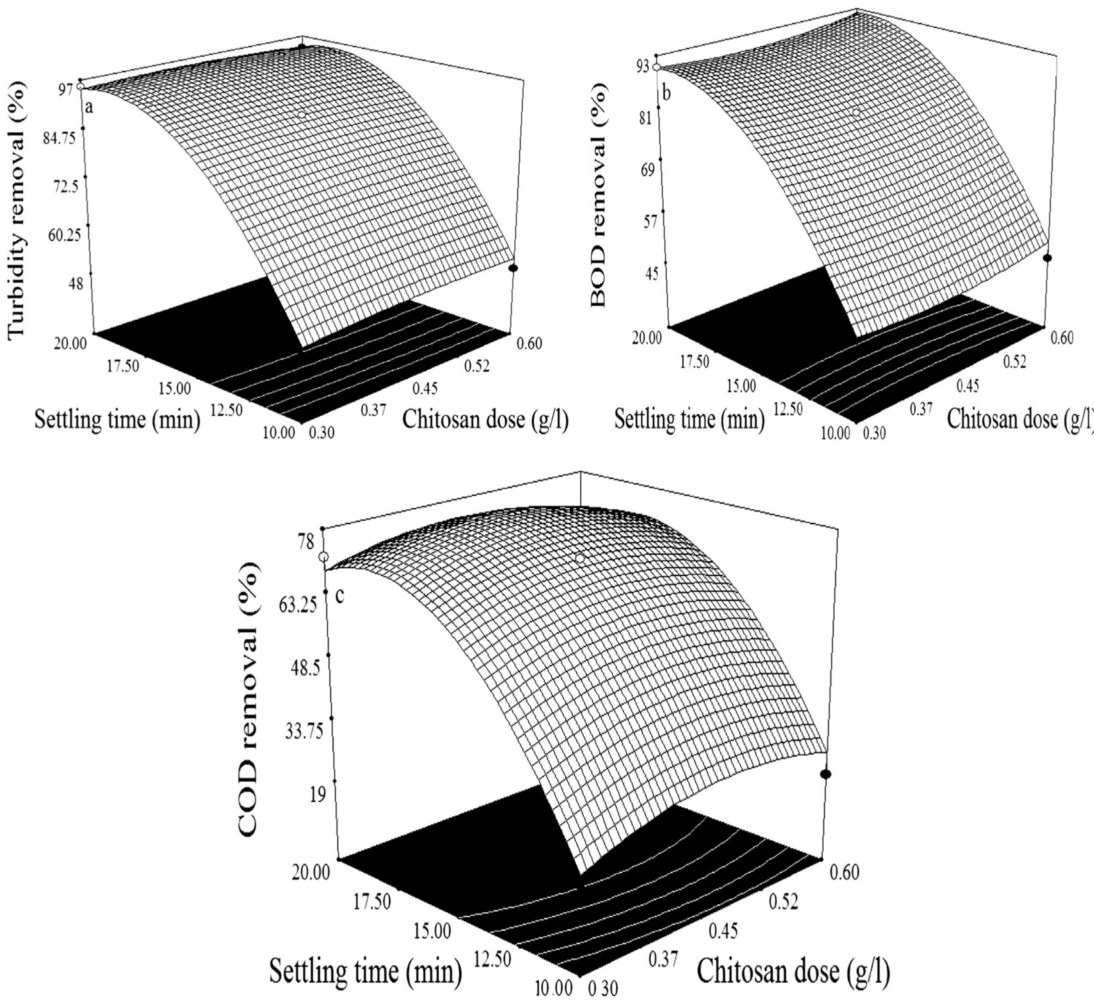

Fig. 4. Effect of process variables (C and D) on responses.

increased (Amit & Mika, 2009). This result clearly stated that settling time is a key parameter which affects the treatment efficiency significantly.

3.6. Optimization

Simultaneous optimization of the multiple responses was carried out using derringer's desired function methodology. According to BBD results, optimal operating conditions to obtain the maximum removal efficiency of turbidity, BOD and COD from grey wastewater as follows: agitation time of 2 min, initial pH of 4, chitosan dose of 0.6 g/L and settling time of 20 min. Under these conditions, predicted removal efficiency of turbidity, BOD and COD was found to be 96%, 91% and 73%, respectively, with a desirability value of 0.9989.

3.7. Validation

The suitability of optimum conditions for predicting optimum response values was tested under the above mentioned optimized conditions. Experiments were performed under the optimized conditions and the results are shown in Table 5. From Table 5, it was found that experimentally obtained results are in close agreement with predicted values by developed mathematical models. These results clearly indicate that RSM is an effective mathematical tool to optimize and investigate the effect of operating variables in grey wastewater treatment process.

Table 5
Validation of optimized conditions.

Responses	Predicted values	Experimental values
Turbidity removal (%)	96.73	96.22
BOD removal (%)	91.90	91.24
COD removal (%)	73.07	73.18

3.8. Comparison with chemical coagulation treatment technique

In this present study, the treatment efficiency of chitosan to treat grey wastewater is compared with chemical coagulation treatment process reported in elsewhere (Pidou et al., 2008), which clearly indicates that treatment of grey wastewater using chitosan is the best option to treat grey wastewater in terms of removal efficiency as well as eco-friendly sludge disposal.

4. Conclusion

In this study, BBD was employed to study and optimize the process variables such as agitation time, pH, chitosan dose and settling time on the removal of turbidity, BOD and COD from grey wastewater. From the results, it was observed that all the process variables have significant effects on the removal efficiency. ANOVA showed that the effects of all process variables on responses were significant and quadratic models were developed for predicting the responses. Optimum process variables were obtained by derringer's desired function methodology and they were found to be agitation time of 2 min, initial pH of 4, chitosan dose of 0.6 g/L and settling time of 20 min. Under these conditions, predicted removal efficiency of turbidity, BOD and COD was found to be 96%, 91% and 73%, respectively, with a desirability value of 0.9989, which was experimentally validated.

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