



Application of response surface methodology to water/wastewater treatment using *Coccinia indica*

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ABSTRACT

This study investigates the optimization of coagulation–flocculation process for turbid water using *Coccinia indica* as a coagulant by the use of response surface methodology (RSM). To minimize residual turbidity, the experiments were carried out using jar test for low, medium, and high turbid water by varying pH and dosages of *C. indica*. A central composite design, which is the standard design of RSM, was used to evaluate the effects and interactions of three major factors: coagulant dosage, initial turbidity, and pH on the turbidity removal efficiency with 2³-factorial design having three star points, six center points, and two replications. The quadratic model developed for the response studied indicates the optimum conditions to be 0.6 mg/L, 6.5, and 300 NTU for coagulant dosage, coagulation pH, and initial turbidity, respectively. At optimum conditions, turbidity removal was found to be 92.60%. The experimental findings were in close agreement with the model predictions. The response model was developed in this study with the value of R^2 as 94.1 and R^2 adjusted as 92.6%. Thus, RSM has demonstrated to be an appropriate approach for the optimization of the coagulation–flocculation process by statistical evaluation.

Keywords: Coagulation; *Coccinia indica*; Water/waste treatment; Optimization; Response surface method

1. Introduction

Excessive turbidity, or cloudiness, in drinking water is esthetically unappealing, and may also represent a health concern. Turbidity can provide food and shelter for pathogens. If not removed, turbidity can promote regrowth of pathogens in the distribution system, leading to waterborne disease outbreaks. The particles of turbidity provide “shelter” for microbes

by reducing their exposure to attack by disinfectants. Microbial attachment to particulate material or inert substances in water systems has been documented by several investigators [1–3] and has been considered to aid in microbe survival [4]. Traditional water treatment processes have the ability to remove turbidity effectively when operated properly.

Coagulation–flocculation followed by sedimentation, filtration, and disinfection, often by chlorine, is used worldwide in the water treatment industry

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before the distribution of treated water to consumers. Aluminum salts are the most widely used coagulants in water and wastewater treatment. However, recent studies have pointed out several serious drawbacks of using Aluminum salts, such as Alzheimer's disease and similar health-related problems associated with residual aluminum in treated waters [5–7] besides production of large sludge volumes. There is also the problem of reaction of alum with natural alkalinity present in the water leading to a reduction of pH and low efficiency in coagulation of cold waters. Many developing countries can hardly afford the high costs of imported chemicals for water and wastewater treatment [8].

Natural macromolecular coagulants are promising and have attracted the attention of many researchers because of their abundant source, low price, multipurpose, and biodegradation. *Chitosan*, *Cactus*, *Okra*, *Plantago Ovata*, and *Moringa oleifera* are the natural compounds which can be used in turbidity removal [9–14].

For individual water, the performance of a coagulant depends on two valuable factors; the coagulant dose and coagulation pH. To search for the optimal conditions to these factors, jar tests using a conventional multifactor method was employed. In this, optimization is usually carried out by varying a single factor, while keeping all other factors fixed at a specific set of conditions. This method is not only time and energy consuming, but also usually incompetent to reach at the true optimal conditions because interaction among variables is not taken into consideration [15–18].

As a solution, the statistical method of response surface methodology (RSM) has been proposed to include the influence of individual factors as well as their interactive influences.

The RSM technique gives better product yields and provides closer confirmation of the output response toward the nominal and target requirements. The experiments designed by this statistic are extensively used in various fields; review of few is reported below,

Adinarayana et al. [19] applied the RSM tool for biochemistry to optimize the nutritional parameters for neomycin production by *Streptomyces marinensis* under solid-state fermentation. The experimental data and model predictions agreed well. Application of RSM was used for describing the performance of coated carbide tools when turning AISI 1045 steel by Noordine et al. [20]. They found that RSM is a very effective tool for the optimization of the performance of coated carbide tools. Zaman et al. [21] successfully applied RSM for the optimization of effects of process variables and their interactions on solubility of metal ions from crude kaolin particles. Speciation study of

Cr (VI)/Cr (III) in environmental waters by fluorimetric method was done by Massumi et al. [22] using central composite, full, and fractional factorial design. Bacaoui et al. [23] studied the preparation of activated carbon used in water treatment. The experimental design to optimize preparation of activated carbons was done by RSM. Wang et al. [24] used a coagulation–flocculation process to treat a paper-recycling wastewater with aluminum chloride as coagulant and a modified natural polymer, chitosan-g-PDMC (poly (2-methacryloyloxyethyl) trimethyl ammonium chloride), as flocculant. To minimize turbidity and sludge volume index, the experiments were carried out using jar tests and RSM was applied to optimize this process. The RSM was demonstrated as an appropriate approach for the optimization of the coagulation–flocculation process by confirmation experiments. Optimization of coagulation–flocculation process for pulp and paper mill effluent by response surface methodological analysis was studied by Ahmed et al. [25]. The experimental results and the predictions by RSM model agree with each other. Ghafari et al. [17] applied RSM to coagulation–flocculation of old and stabilized leachate. They studied the capability of polyaluminum chloride (PAC) in the treatment of stabilized leachate. Central composite design (CCD) and RSM were applied to optimize the operating variables. Quadratic models developed for the four responses (COD, turbidity, color, and TSS) studied indicated the optimum conditions to be PAC dosage of 2 g/L at pH 7.5 and alum dosage of 9.5 g/L at pH 7. The experimental data and model predictions agreed well. Moghaddam et al. [26] applied coagulation/flocculation process for dye removal using sludge from water treatment plant. Optimization study through RSM gives promising predictions with experimental results. Giani et al. [27] studied coagulation–flocculation process, based on aluminum sulfate, by using RSM, in order to establish the optimum parameters to achieve a maximum suspensions removal. They established RSM as a useful tool, which allows a treatment plant operator, to get optimum conditions with minimum trials to achieve maximum suspensions removal efficiency. Emulsion liquid membrane technique was used by Balasubramanian [28] for the extraction of phenol from synthetic and industrial effluents. Statistical experimental design was applied for the optimization of process parameters for the removal of phenol. The effects of process parameters, namely, surfactant concentration, membrane or organic to internal phase ratio (M/I), and emulsion to an external phase ratio (E/E) on the removal of phenol were optimized using a response surface method. The optimum conditions for the extraction of phenol using RSM were: surfac-

tant concentration –4.1802%, M/I ratio: 0.9987(v/v), and E/E ratio: 0.4718 (v/v). Under the optimized condition, the maximum phenol extraction was found to be 98.88%, respectively.

The objective of the present study is to investigate the suitability of *Coccinia indica* as a natural coagulant for turbidity removal and to verify the feasibility of RSM for optimization of coagulation–flocculation process.

2. Materials and methods

The entire chemicals used in this study were purchased from Vijay Chemicals, Shrirampur Dist., Ahmednagar (India). *C. indica* fruits were purchased from a local vegetable market.

Description of *C. indica*:

Scientific name: *C. cordifolia*; Family: Cucurbitaceae
English name: Ivy Gourd; Hindi name: Kundru

Coccinia is a climber herb cultivated throughout India. Fig. 1 shows the photograph/ image of *C. indica* fruit. It is commonly known as Kovai in Tamil. In folklore medicine, the fruit is used to treat leprosy, fever, asthma, infective hepatitis, jaundice, and sore throats. It is also used as expectorant and astringent. Every part of this plant is valuable in medicine for ring worm, psoriasis, small pox, and ulcers. The qualitative analysis of photochemicals in *C. indica* fruit is displayed in the Table 1.

2.1. Preparation of *C. indica* powder

C. indica ripped fruits purchased from the market are thoroughly washed with water, cut into small pieces, and soaked in distilled water overnight. The



Fig. 1. Image of *C. indica* fruit.

Table 1
Qualitative analysis of photochemical in *C. indica* fruit

Phytochemical	Methanolic extract
Alkaloids	+
Steroids	+
Tannins	+
Saponins	+
Ellagic acid	+
Phenols	+
Glycosides	+
Lignans	–
Triterpenoids	+
Flavonoids	+

Note: + indicates presence; – indicates absence.

Source: Shaheen et al. [29].

mucilaginous extract was filtered through muslin cloth. Alcohol was added to precipitate the extract. The precipitate was then washed with acetone 2–3 times and then dried by keeping in an oven at the temperature of 40°C for 24 h. The filtered extract was then used in the experiment [30].

2.2. Preparation of synthetic turbid water

Local clay was used to prepare Synthetic turbid water by soaking the clay for 24 h in tap water and then blending it for 10 min. The suspension was washed through a 75 μ sieve. This was kept as a stock suspension for preparation of different turbidities. A portion of the stock suspension was diluted with tap water after 30 min of settling in a container. The supernatant was carefully decanted and desired turbidities of 34.2 (low), 146.8 (medium), and 314.4 NTU (high) were obtained by diluting it.

During sedimentation analysis of the suspension, particle size distribution results were obtained as:

73% of particles were finer than 8.6 μ m, 26% of particles were finer than 1.4 μ m, 19% of particles were finer than 1.25 μ m, 13% of particles were finer than 0.72 μ m, and 9% of particles were finer than 0.62 μ m [31]. Chemical characteristics of the tap water used to prepare turbid water are shown in Table 2.

2.3. Experimental work

Jar tests were carried with *C. indica* as a coagulant; tests were conducted on 500 mL synthetic turbid water samples. The samples were subjected to a rapid mixing at 300 rpm for 1 min and slow mixing at 40 rpm for 30 min. The floc allowed settling

Table 2
Composition of the tap water used to simulate raw water

Parameter	Concentration
pH	7.20–8.10
Turbidity	0.2 NTU
Alkalinity (as CaCO ₃)	247 mg/L
Total hardness (as CaCO ₃)	188 mg/L
Potassium (K ⁺)	5 mg/L
Sodium (Na ⁺)	23 mg/L
Chlorides (Cl ⁻)	103 mg/L
Calcium (Ca ⁺⁺)	34.6 mg/L
Sulfates (SO ₄ ⁻⁻)	8.7 mg/L

undisturbed for 30 min and samples for residual turbidity measurement were withdrawn from each beaker. According to the data available in the literature, where low concentrations were documented to be effective for turbidity removal, [32,33] the behavior of *C. indica* was investigated in the range of 0.2–1.0 mg/L. The chemical analysis was carried out as per Standard Methods [34]. The jar test apparatus used was of Secor-141 make with four blades. The turbidity was measured with Systronic Turbidity meter type 131, Systronics, India. The pH value of the test sample was measured using digital pH meter, model 121 of Elico make. Depending upon the requisite, pH of the turbid water was adjusted to the desired value by adding either 0.1M HCl solution or 0.1M NaOH solution. The procedure conformed to that described in Standard Methods for the examination of water and wastewater.

2.4. Experimental design and data analysis

In this study, CCD and RSM were applied to optimize three important operating variables: pH, coagulant dosage, and initial turbidity. Experiments were initiated as a preliminary study for determining a narrower range of pH, coagulant dosage, and initial turbidity prior to designing the experimental runs. Accordingly, pH from 4–9 were tried and the increment continued until appreciable reductions were observed in the process response, turbidity removal. Likewise, a wide dosage range of 0.2–1.0 mg/L and initial turbidities of 34.2, 146.8, and 314.4 NTU, respectively, were examined.

A CCD, which is very competent design tool for fitting second-order models, was selected in this study. The design included three factors with six center points and 23 runs. This design is made rotatable by the choice of α value (axial distance), $\alpha = (2^k)^{1/4}$ assures rotation of the CCD. In this study, α (Nos. of

factors) was 3 (coagulant dose, pH and initial turbidity), hence α becomes 1.682. In order to define the experimental domain explored, preliminary experiments were carried out to determine more effective ranges of the factors prior to designing the experimental run as done by many researchers [16,17,24,27]. Once the desired ranges of the factors had been defined, they were coded to lie at ± 1 for the factorial points, 0 for the center point, and $\pm \alpha$ for the axial points. The codes were calculated as a function of the range of interest of each factor as shown in Table 3.

Relation exists between the factors and response (percentage turbidity) was investigated from the experimental data by regression analysis. A regression design is employed to model a response as a mathematical function of a few continuous factors and “good” model parameter estimates are desired [35]. Each response of Y can be represented by a mathematical equation so as to correlate the response surface. The response can be expressed as second-order polynomial equation, according to Eq. (1).

$$Y = f(x) = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \sum_{j=i+1}^k \beta_{ij} x_i x_j + \sum_{i=1}^k \beta_{ii} x_i^2 \quad (1)$$

where Y is the predicted response (percentage turbidity removal) used as a dependent variable; k the number of independent variables, x_i ($i=1, 2$) the input predictors or controlling variables; β_0 the constant coefficient; and β_i , β_{ij} , and β_{ii} the coefficients of linear, interaction, and quadratic term, respectively. The coefficient parameters were estimated using a multiple linear regression analysis employing the Mini Tab software, which can also be used to find the 3-D surface and 2-D contour plots of the response model.

Table 3
Relationship between the coded and actual values of a factor

Code	Actual value of factor
$-\alpha$	X_{\min}
-1	$(\alpha - 1) X_{\max} + (\alpha + 1) X_{\min}$
	2α
0	$X_{\max} + X_{\min}$
	2α
$+1$	$(\alpha - 1) X_{\min} + (\alpha + 1) X_{\max}$
	2α
$+\alpha$	X_{\max}

Note: X_{\max} and X_{\min} are maximum and minimum value of X .

3. Result and discussion

The variation of different parameters: *C. indica* dosage (x_1), initial turbidity (x_2), and pH (x_3) is presented in Table 4, and its influence on turbidity removal, which represents response variable (Y) has been investigated.

3.1. Model building

Forty-five observed responses were used to compute the model using the least square method. The response (% turbidity removal) was correlated with the three factors (coagulant dose, initial turbidity, and coagulation pH), using the second-order polynomial, as represented by Eq. (1) above. From the experimental data, quadratic regression model was built using coded variables x_1 , x_2 , and x_3 as represented by Eq. 2.

$$\begin{aligned}
 Y = & 91.69 - 0.51x_1 + 6.48x_2 - 0.02x_3 - 6.4x_1^2 \\
 & - 4.3x_2^2 - 10.18x_3^2 + 0.56x_1x_2 + 0.17x_1x_3 \\
 & + 0.33x_2x_3
 \end{aligned} \tag{2}$$

The positive signs of the term indicate a synergistic effect, while the negative sign indicates an antagonistic effect.

3.2. Validation of the model

It is frequently essential to check whether the fitted model ensures an adequate approximation to the real system. Graphical and numerical methods are the primary techniques to validate the models. The graphical method characterizes the nature of residuals of the models.

The first plot, residuals versus the fitted values, as shown in Fig. 2, was used to evaluate the sufficiency of the functional part of the model. The second plot, residual versus observation orders, as shown in Fig. 3, was used to check for any drift in the process. From these two plots, it was observed that the graphical residual analysis indicated no obvious pattern, implying the residuals of the models were randomly

Table 4
Variables ranges

Parameter	Minimum	Maximum
<i>C. indica</i> dose, mg/L (x_1)	0.2	1.0
Initial turbidity, NTU (x_2)	34.2	314.4
pH (x_3)	4.0	9.0

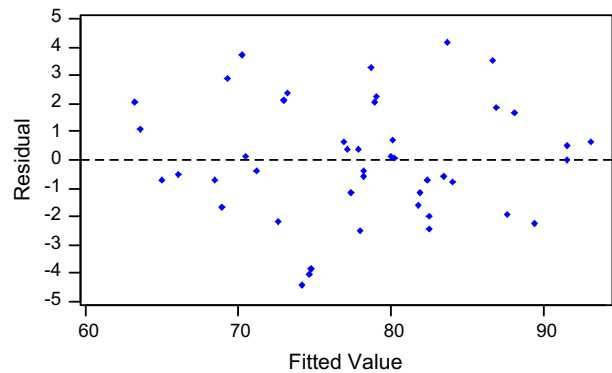


Fig. 2. Residual versus the fitted values for turbidity removal.

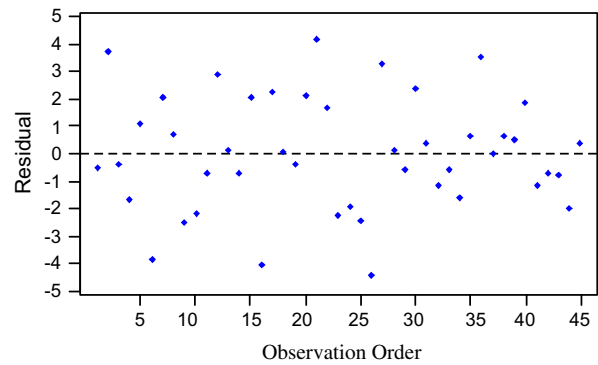


Fig. 3. Residual versus the observation orders of data for turbidity removal.

distributed. Fig. 4 is the normal probability plot for turbidity removal, which confirms that the assumptions of normality were satisfied for the coagulant data. Fig. 5 is the plot between experimental and predicted data by the model, which demonstrate a square value of correlation coefficient of 0.931, indicates a good fit between experimental data and model output.

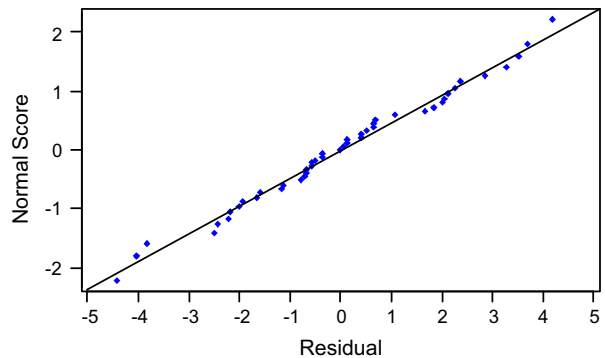


Fig. 4. Normal probability plot for turbidity removal.

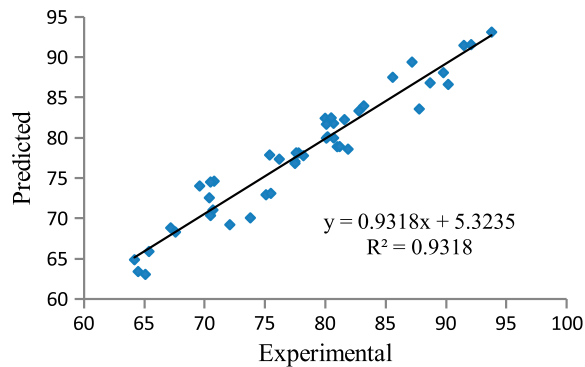


Fig. 5. Predicted versus experimental data plot for turbidity removal.

Model fitting was also checked by the determination coefficient (R^2). In this case, the value of the determination coefficient (R^2)=0.932 indicates that only 1.8% of the total variation is not explained by the model. The value of the adjusted determination coefficient (adjusted R^2)=0.914 is also high to advocate a high significance of the model. A higher value of the correlation coefficient justifies an excellent correlation between the independent variables. Therefore, the regression model explains the removal efficiency well. In addition, the ANOVA of the model, as shown in Table 5, reveals that the model is highly significant, as evident from the very low p value and F value of 53.15. Table 6 furnishes an insight into the linear, quadratic, and interaction effect of parameters. From Table 6, it was found that the linear effects of dose, pH, and quadratic effect of dose, initial turbidity, and pH are highly significant. On the other hand, linear effect of initial turbidity and interaction effect of dose \times initial turbidity, dose \times pH, and initial turbidity \times pH are all insignificant.

This trend was also being observed from Figs. 8 and 11 with both curves showing very prominent changes. The importance of the coagulant dose and pH in coagulation are also underlined in other studies by the various researchers [36–37].

Table 6

Estimated regression coefficients for turbidity removal (coded units)

Term	Coef	SE Coef	T	P
Constant	91.69	0.9405	97.491	0.000
Dose	-0.51	0.4929	-1.043	0.304
It	6.48	0.4255	15.220	0.000
pH	-0.02	0.4241	-0.036	0.972
Dose \times dose	-6.40	0.8253	-7.751	0.000
It \times it	-4.30	0.7666	-5.613	0.000
pH \times pH	-10.18	0.8050	-12.644	0.000
Dose \times it	0.56	0.5942	0.947	0.350
Dose \times pH	0.17	0.5903	0.283	0.779
It \times pH	0.33	0.5080	0.645	0.523

Note: It - initial turbidity.

The 3-D surface graphs and 2-D contour plots for turbidity removal are shown in Figs. 6–11, respectively. The saddle in the 3-D surface graphs in Figs. 7 and 8 indicate that the turbidity removal percentage increases at the center of the region, which involves the interaction between coagulation dosages with pH. The pattern of graphs indicates that the highest percentage is obtained generally at the intersection of zero levels of all of the factors. The mechanism of coagulation and flocculation occurs once sufficient coagulant has been dispersed to achieve optimum destabilization of the colloidal particles and the flocculent allowed particles to agglomerate into larger flocs, as reported by Peavy et al. and Divas [38–39]. The destabilization of particles by large polymeric molecules engrosses the bridging of particles by the polymer chain, hence forming larger structural units (floc) that are readily separated from the aqueous dispersing medium [38].

The optimum levels of the factors were obtained by analyzing the response surface contour and the derivatives of the equation of the above model. The optimum conditions were a set of X_1 (coagulant dose) and X_2 (pH), where the derivative becomes zero, i.e.

Table 5
Analysis of variance for turbidity removal

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	2566.07	2566.07	285.119	53.15	0.000
Linear	3	1209.88	1248.46	416.152	77.58	0.000
Square	3	1348.72	1348.72	449.573	83.81	0.000
Interaction	3	7.47	7.47	2.491	0.46	0.709
Residual error	35	187.74	187.74	5.364		
Total	44	2753.81				

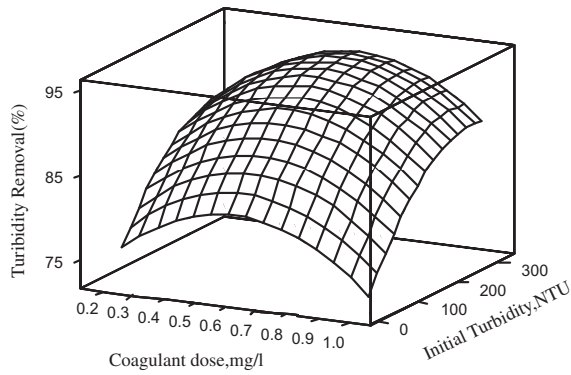


Fig. 6. 3-D surface plot for turbidity removal (pH hold at 6.5).

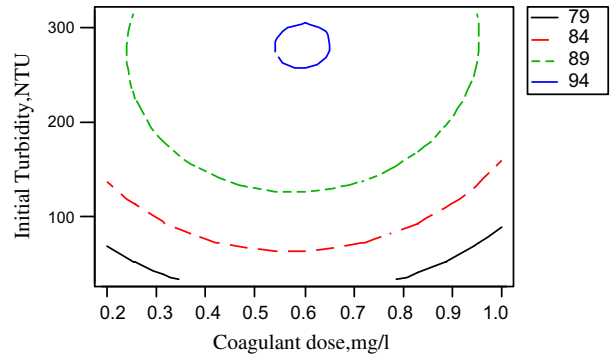


Fig. 9. Two-dimensional contour plot for turbidity removal (pH hold at 6.5).

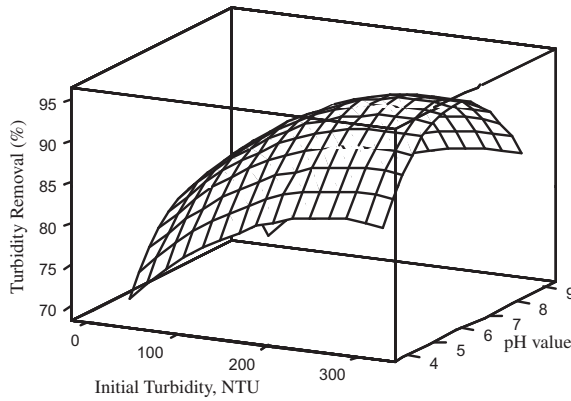


Fig. 7. 3-D surface plot for turbidity removal (C. indica dose hold at 0.6 mg/L).

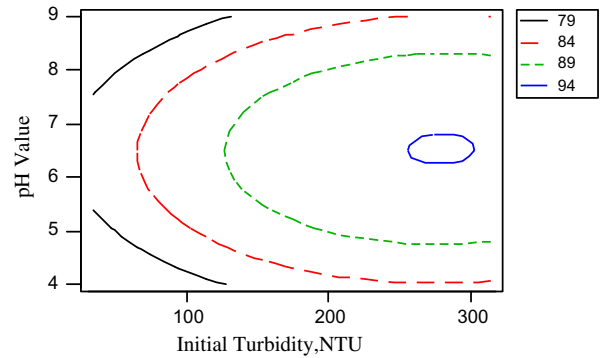


Fig. 10. Two-dimensional contour plot for turbidity removal (C. indica dose hold at 0.6 mg/L).

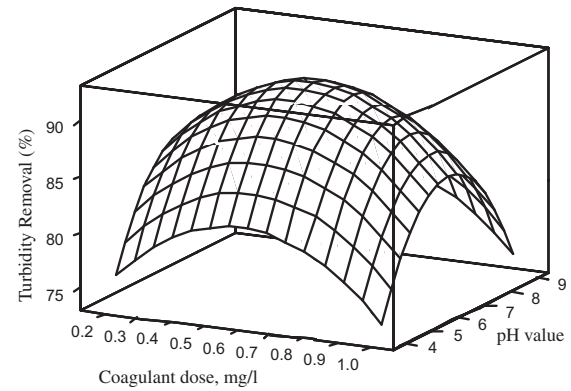


Fig. 8. 3-D surface plot for turbidity removal (initial turbidity hold at 174.3 NTU).

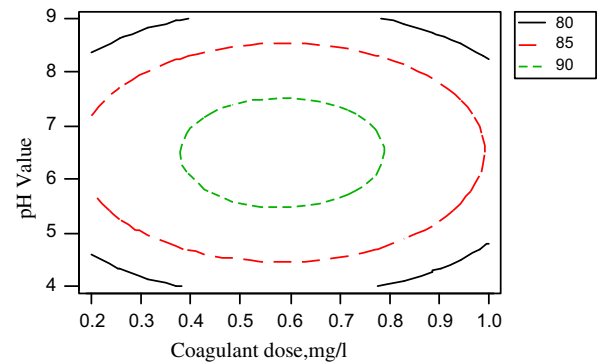


Fig. 11. Two-dimensional contour plot for turbidity removal (initial turbidity hold at 174.3 NTU).

$$\frac{\partial Y}{\partial X_1} = \frac{\partial Y}{\partial X_2} = \frac{\partial Y}{\partial X_3} = 0 \quad (3)$$

The maximum predicted value was indicated by the surface confined by the smallest ellipse in the

contour diagram (Fig. 9). From the contour plot, it was observed that the maximum % age of turbidity removal was 94% at coagulant dose 0.6 mg/l, pH 6.5, and for initial turbidity of 300 NTU. This conclusion was also supported by the canonical analysis using (MATLAB 7.1 Software) of the above model. The

Table 7

The results achieved from the model for *C. indica* can be conformed to the experimental data

Coagulant	Parameter	Dose mg/L	pH	Initial turbidity	Turbidity removal (%)
C. indica	Experimental	0.6	7.0	314.4	93.8
	Predicted	0.6	6.5	300.0	94.0

analysis resulted into the following set of Eigen values of λ_1 , λ_2 , and λ_3 for the model as: $\lambda_1 = -10.1862$, $\lambda_2 = -6.4356$, and $\lambda_3 = -4.2582$.

The model shows that all the Eigen values are negative; indicating the stationary points obtained by solving the equations is a maxima, which predict maximum response value.

As shown in Table 7, the percentage turbidity removal obtained from experiment is very close to those estimated using the model (estimated from Fig. 9). This implies that the RSM approach is an appropriate technique for optimizing the conditions of coagulation process.

4. Conclusions

This method allows establishing the optimum parameters for coagulation–flocculation process by using a small number of experiments. Nearly neutral pH, moderate coagulant doses, and a higher load in suspensions of raw water (high turbidity) presented a favorable influence on turbidity removal efficiency. Experimental results reveal that the optimal conditions for the minimum turbidity were coagulant dosage of 0.6 mg/L and pH 7.0. The verification experiments demonstrate that the RSM approach was appropriate for optimizing the coagulation–flocculation process. The optimal conditions obtained from the compromise regression equation were coagulant dosage of 0.6 mg/L and pH 6.5. At the optimized condition, the maximum turbidity removal was found to be 94.0% respectively. The results of a confirmation experiment were found to be in very good agreement with the values predicted by the model. This confirms that RSM and CCD can be successfully applied for modeling and optimizing the coagulation process.

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