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Coagulation/flocculation process for dye removal using water treatment residuals: modelling through artificial neural networks

Mahesh R. Gadekar, M. Mansoor Ahammed*

Civil Engineering Department, S V National Institute of Technology, Surat 395007, India, emails: maheshgadekar@ymail.com (M.R. Gadekar), Tel. +91 98258 75162; email: mansoorahammed@gmail.com (M.M. Ahammed)

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ABSTRACT

The potential of aluminum-based water treatment residuals (WTR) discharged from water treatment plants was evaluated as a coagulant for color removal from a disperse dye solution. The effects of WTR dose, initial dye concentration, and initial pH on color removal were studied. The results showed higher color removal at lower pH values. Maximum color removals of 88, 87, and 76% were obtained for initial dye concentrations of 25, 50, and 75 mg/L, respectively, at pH 3.0 with a WTR dose of 3,000 mg/L. Different artificial neural networks (ANN) were developed for predicting the color removal. The performance of the models was found to be very good, with correlation coefficient (R^2) values greater than 0.90. The results showed that simulation employing ANN incorporates non-linear behavior of the system, and the model-predicted and observed values of color removals were in close agreement with each other. The study thus indicates that reusing water treatment sludge as a coagulant for color removal would be an attractive option.

Keywords: Artificial neural networks; Coagulation; Color removal; Disperse dye; Water treatment residuals

1. Introduction

Rapid development of textile industries which use different synthetic dyes has resulted in severe water pollution [1]. Considerable amount of colored effluent is generated from these industries due to consumption of large quantities of water at its different steps of dyeing and finishing among other processes. Coloration of water bodies due to disposal of untreated effluent from these industries is a major environmental problem especially in developing countries. The presence of even very small amounts of dyes in water (less than 1 ppm for some dyes) is not only esthetically displeasing [2,3] but also hinders light penetration, disturbing the biological processes in water bodies [4]. Over 100,000 commercially available dyes exist and more than 7×10^5 tonnes per year are produced annually [5–9]. Further, some dyes cause allergy, dermatitis, skin irritation, and cancer to humans, in addition to being mutagenic [10,11].

The complex aromatic structure and synthetic origin of the dyes make them stable to heat, oxidizing agents, photodegradation, and biodegradation [2–4,12], thus complicating the selection of suitable methods for their removal. Since the removal of dyes from wastewater is considered as an environmental challenge and governmental legislation requires textile wastewater to be treated, there is a constant need to

*Corresponding author.

develop effective processes that can efficiently remove these dyes [5,13]. Different treatment techniques for the removal of dyes from water have been reported in the literature. These include precipitation, coagulation/flocculation, adsorption, membrane filtration, ion exchange, reverse osmosis, advanced oxidation processes, and aerobic and anaerobic biological processes. These processes have shown varying degrees of dye removal.

Adsorption techniques have much potential in the treatment of dye-containing waters when high performance and low-cost adsorbents are available. Generally, advanced oxidation processes are effective for the removal of most dyes [14], but a common problem is their relatively high cost in large-scale utilization. Moreover, the possible formation of some more toxic intermediates is of great concern. On the other hand, biological processes for dye removal have upper hand in concern with higher dye concentration and treatment time, though these processes are largely ineffective for color removal from wastewater due to poor biodegradability of some dyes [15]. Dye removal by coagulation is the most commonly used method in industries, and is not based on the partial decomposition of dye compounds and thus no potentially harmful and toxic intermediates are produced. However, there is the inherent sludge formation and the need for disposal of the sludge produced. While the process has been widely employed, the operating conditions need to be carefully selected in order to maximize color/organic matter removal and to reduce chemicals consumption.

Large amounts of water treatment residuals (WTR) or water treatment sludge are produced when chemical coagulants are used for water treatment. These WTR typically contain minerals and humic matters which are removed from the raw water, together with the residuals of any treatment chemicals used as coagulant (commonly aluminum or iron salts) and coagulant aids (mostly organic polymers) [16]. In many countries, due to regulatory changes in the recent past, WTR now have to be disposed of into landfills or through land application. In India, however, WTR is still disposed of into water bodies [17–19]. Adverse effects are seen in water bodies due to the disposal of WTR. The toxicity of aluminum species towards various aquatic life and benthic organisms is well documented [20]. Some researchers have linked aluminum's contributory influence to occurrence of Alzheimer's disease [17]. Consequently, reuse of WTR is getting more attention.

A number of research efforts have been made particularly in recent years to reuse WTR in many beneficial ways either by recycling or by direct application in building and construction materials, use in water and wastewater treatment, and land-based application for soil improvement [16–19,21–30]. A few studies have been reported on the reuse of aluminum-and iron-based WTR for treating acid textile dyes [21,26,27].

Artificial neural network (ANN) has been used for modeling different water and wastewater treatment processes. The ANN does not require the mathematical description of the involved process phenomenon, and hence can prove useful in simulating coagulation systems. The reliability and robustness of networks depend mainly on the choice of process variables as well as the available data-set and the domain used for training purposes [31,32]. No studies have been reported in the literature on the use of WTR for color removal from disperse dyes and also of ANN for modeling coagulation process using WTR for treating dye wastewater.

The present study focuses the potential and effectiveness aluminum-based WTR as an alternative coagulant for the removal of a disperse dye, Disperse Blue 79 from water, which is widely used in textile industries in India. The effect of three parameters, namely, initial pH, WTR dosage, and initial dye concentration on color removal efficiency was studied. The ANN was used to simulate the non-linear behavior of coagulation process to predict the color removal.

2. Materials and methods

2.1. Synthetic dye solution

Synthetic dye solution was prepared by dissolving Disperse Blue 79 (Disperse Navy Blue 3G) which is widely used in the textile industries. A stock dye solution of 1,000 mg/L was prepared in distilled water and it was diluted to the required concentrations with tap water (pH 8.1, total dissolved solids 190 mg/L, total hardness 160 mg/L as $CaCO_3$, alkalinity 76 mg/L

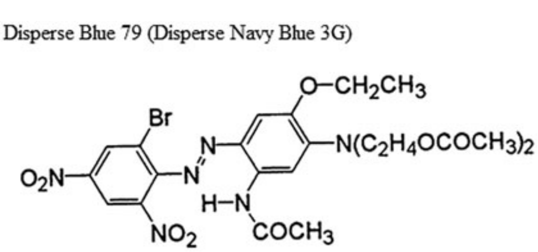

Fig. 1. Color index number, commercial name, and chemical structure of the dye used.

as $CaCO_3$, chlorides 16 mg/L). The color index (CI) number, commercial name and chemical structure of the dye are as shown in Fig. 1.

2.2. Water treatment residuals

The WTR was collected from the coagulation/flocculation unit of a water treatment plant in Bhandup, Mumbai, India, where polyaluminium chloride (PACl) is used as a coagulant. The collected sludge was stored at room temperature (27–30°C) in its original form and was used in its raw state. Elemental analyses of the dried PACl-based WTR were carried out by ICP (Element XR, Thermo Fisher Scientific, Germany).

2.3. Experimental procedure

A six-beaker jar-test apparatus (DBK Instruments, Mumbai) was used to simulate the coagulation/flocculation process. Each beaker contained 250 mL of the synthetic dye solution. WTR in its raw form was added as the coagulant. The WTR dosages were calculated based on dry weight. The coagulation/flocculation procedure consisted of 2-min rapid mixing at 150 rpm, followed by 30-min slow mixing at 25 rpm, and 20-min settling. Three important parameters affecting coagulation process, namely, initial pH, WTR dosage, and initial dye concentration were varied in different experiments in specified ranges. The ranges for these parameters were selected based on the values reported in the literature and on the preliminary tests. Initial pH of the samples was adjusted to a desired value by adding 0.1 N H_2SO_4 or NaOH using a pH meter (Hanna 209). The ranges of the three parameters used in the study are shown in Table 1. Centrifugation (at 5,000 rpm for 3 min) of the sample was performed before analysis. Color was measured using a UV–visible spectrophotometer (Varian Cary 50) at a wavelength corresponding to the maximum absorbance of 532 nm for the dye used. Percentage of dye removal was calculated by the following equation (Eq. (1)):

$$\text{Colour removal } (\%) = (C_r - C_t)/C_r \times 100 \quad (1)$$

Table 1
Ranges of different parameters used in the study

Sl. no.	Parameter	Range
1	Initial pH	3.0–7.5
2	WTR dosage (mg/L)	500–3,000
3	Dye concentration (mg/L)	25–75

where C_r and C_t are color concentration of raw and treated solutions, respectively.

Flocculation ability of WTR was determined to assess the ability of WTR to produce flocs. Flocculation ability (Q_t) of WTR was calculated by Eq. (2):

$$\text{Flocculation ability } (Q_t) = (C_r - C_t) \times V/m \quad (2)$$

where V is the volume of dye solution (L), and m is the mass of WTR (g).

2.4. Artificial neural network model

The neural network toolbox of MATLAB (Version R2010a) was used to predict the color removal efficiency of the coagulation process. A multi-layered perceptron neural net has an input layer of neurons (independent variables), a number of hidden layers, and the output layer (dependent variables). The hidden layers attribute to feature detectors in input and output parameters and there can be more than one hidden layer. In the present study input and output parameters used are presented in Table 2. A three-layered feed forward backpropagation algorithm with tan sigmoid (tansig) in hidden layer, and a linear transfer function (purelin) at output layer were designed as shown in Fig. 2. The Levenberg–Marquardt back propagation algorithm was used for ANN model training. A total of 108 data points were used in the present study. These data points were split randomly into training (60%), validation (20%), and test (20%) subsets. All inputs and target data were normalized into the (–1) –1 range, using "mapminmax" function of MATLAB. During the training process, small weights were assigned to the connection between neurons in a random way. The weights were modified until the error between the predicted and experimental values of color removal efficiency were minimized. It is desired that the difference between predicted and

Table 2
Ranges of different variables used for ANN modelling

Sl. no.	Parameter	Range
Input parameters		
1	Initial pH	3.0–7.5
2	Final pH	3.0–7.5
3	WTR dosage (mg/L)	500-3,000
4	Dye concentration (mg/L)	25–75
Output parameter		
1	Color removal (%)	0–100

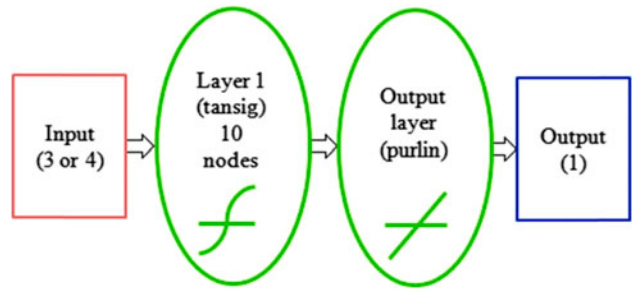

Fig. 2. Feed forward network for experimental design and modelling.

observed values should be as small as possible. During testing, the network was tested for its generalization ability with the observed value after the training process was completed. When the neural networks were tested successfully, they can be used for prediction. Furthermore, linear regression analysis between predicted and observed experimental values was carried out to investigate the network response.

For constructing neural network model, the influence of varying sets of inputs (initial dye concentration, initial pH, WTR dosage, and final pH that respond to the output (color removal efficiency (%)) was studied. Table 2 presents the range of parameters used in modeling. Two types of models were generated: (i) combined model for all initial dye concentration where initial dye concentration was included as an input parameter (M_4) and (ii) different models for each initial dye concentration (M_1, M_2, and M_3 for initial dye concentrations of 25, 50, and 75 mg/L, respectively). The structure of models is presented in Fig. 3.

3. Results and discussion

3.1. Characterization of WTR

Important physicochemical characteristics of WTR collected from the Bhandup water treatment plant are presented in Table 3. The WTR contained 54.04 mg Al/g of dry mass and 35.93 mg Fe/g of dry mass. Other elements such as calcium, magnesium, and potassium were also present (Table 3). WTR was slightly basic (pH 7.4) with a solid content of 5.4%. Typical aluminum content in the range of 27.09–171.77 mg/g dry mass has been reported in the literature [16,18,22,23]. However, most of the values reported are for alum-based WTR while PACl-based WTR was used in the present study. In addition to aluminum, iron present in WTR also contributes to the coagulation process [19,26]. The elemental composition of WTR will depend on the type of the coagulant used, raw water quality, and the dose of the coagulant [19].

3.2. Effect of water treatment residual dose on color removal

Fig. 4 shows the effect of WTR dose on color removal at different initial dye concentrations. These tests were conducted at an initial pH of 3.0. It can be seen that the color removal increased with the increase in WTR dose and the highest color removal was obtained at a dose of 3,000 mg/L. The maximum color removal of 88, 87, and 76% were obtained for initial dye concentrations 25, 50 and 75 mg/L, respectively. However, increasing WTR dose beyond 2,000 mg/L showed only marginal increase (up to 4%) in color removal. Similar trends of increase in color removal with the increase in coagulant dose were obtained using WTR as a coagulant on acid dyes [26,27], and a dye removal of 94.2% was reported at a dose of 4.55 g/L with PACl-based WTR [27]. Further, previous studies on disperse dyes employing alum as coagulant reported color removal in the range of 80–90% at doses of 40–200 mg/L [33,34], whereas with ferric salt as coagulant gave 75% color removal at a dose of 60 mg/L [34].

3.3. Effect of pH on color removal

The pH of the solution is one of the most important parameters affecting coagulation efficiency. Fig. 5 shows the color removal as a function of initial pH of the solution at a constant WTR dose of 500 mg/L. It is seen that there was a decrease in color removal with the increase in pH. Thus, maximum color removals of 68, 67, and 64% were obtained at pH 3 for initial dye concentrations of 25, 50, and 75 mg/L, respectively, which were decreased to 50, 43, and 54 at pH 7.5. Similar results have been reported in the literature for color removal of acid red dyes using PACl- and ferric chloride-based WTR as coagulants, and maximum removals of 94.2–96.5% were obtained at pH values 3.42–3.50, respectively, using PACl [27] and ferric chloride [26]-based WTR.

In order to further explore the influence of WTR dose on pH of system, initial pH and final pH after the addition of WTR were plotted for different WTR doses and is presented in Fig. 6. It is clear that the addition of WTR increased the solution pH. However, the increase was sharp at lower initial pH values of 3.0–6.0. At initial pH values ≥7.0 there were little increase in the final pH. It can also be noted from Fig. 6 that increase in pH was also influenced by WTR

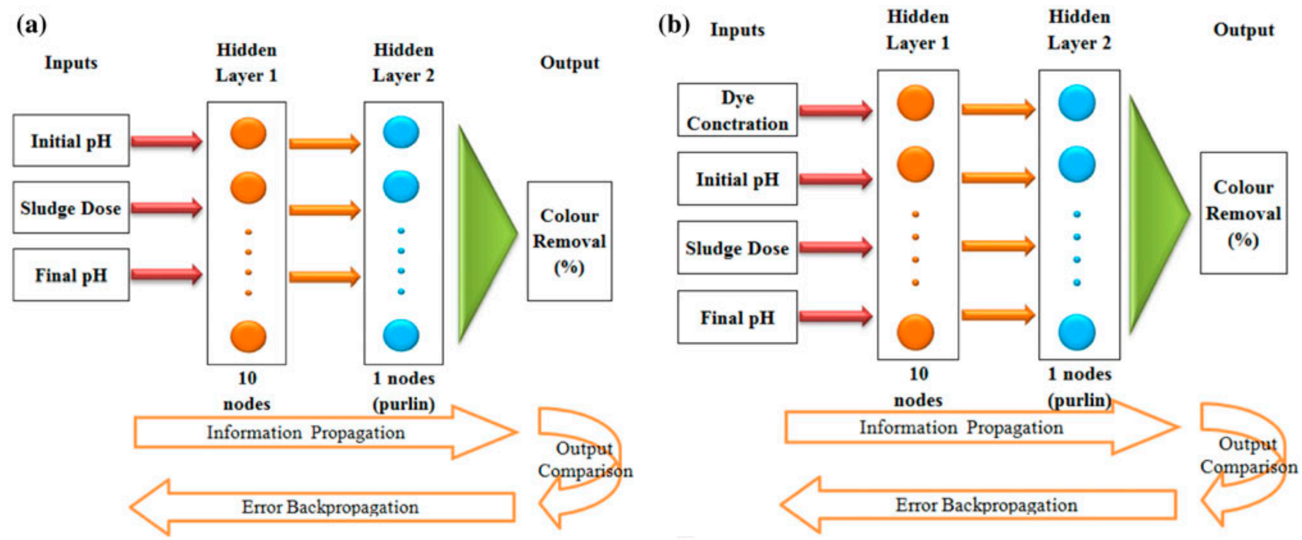

Fig. 3. Neural network structure to predict color removal: (a) for different initial dye concentrations (M_1, M_2, and M_3) and (b) with dye concentration as an input parameter (M_4).

Table 3
Physiochemical characteristics of WTR

Parameter	Value
pH	7.4
Solid Content (%)	5.4
Al (mg/g of dry WTR)	54.06
Fe (mg/g of dry WTR)	35.93
Ca (mg/g of dry WTR)	77.62
Mg (mg/g of dry WTR)	49.16
K (mg/g of dry WTR)	20.06

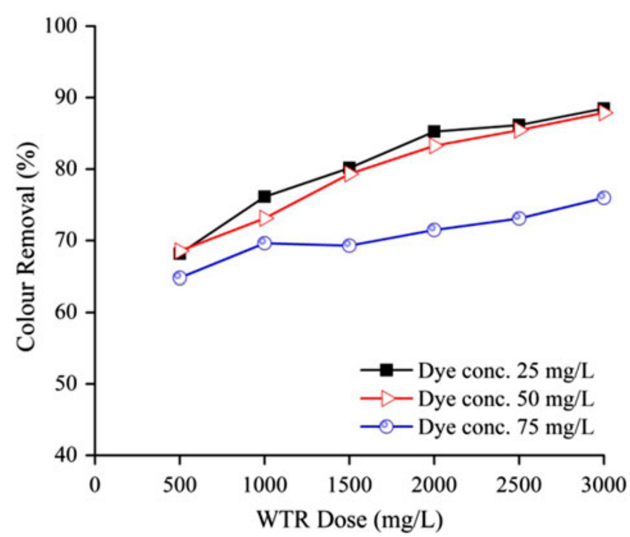

Fig. 4. Effect of WTR dose on color removal (initial pH 3.0).

doses with higher increase with larger doses. At an initial pH of 3.0, the final pH varied between 3.3 and 4.4 at different sludge dosages. This indicates that a final pH around 4.0 would be beneficial for enhanced color removal efficiency to which several reasons may be adduced. Shi et al. [35] reported behavior of different aluminum species on color removal which are pH dependent, and coagulation efficiencies of polyaluminum chloride and alum tended to increase with decrease in pH and approached complete removal when pH was sufficiently lower. Merzouk et al. [34] reported changes in pH during coagulation process due to the formation of $Al(OH)_3$, which precipitate when the final pH was about 4 for alum. However, color removals were reduced at lower alum doses as pH value of 4 could not be achieved at lower doses. Several reports on utilizing aluminum salts for color removal described maximum yield of disperse dye at lower pH range of 4–5.5.0, and 5.0–6.0 for polyaluminum chloride and alum, respectively [33–35]. The surface charge of the coagulants (as related to the zeta potential measurements) is pH dependent, and can affect removal efficiency to some extent [29]. In order to achieve color removal, the negative charges of dye molecules need to be sufficiently neutralized [26], thus at optimum pH dye particles retains net negative charges which facilitate the performance of cationic coagulants [33]. Thus, charge neutralization could be considered as prerequisite condition for color removal of the dye. Structural stability of dye molecules was also affected by pH, and thus the color reduction [5].

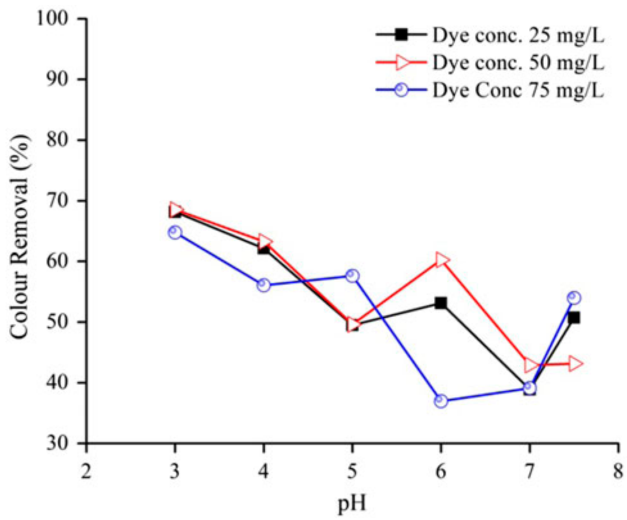

Fig. 5. Effect of pH on color removal at different dye concentration (WTR dose 500 mg/L).

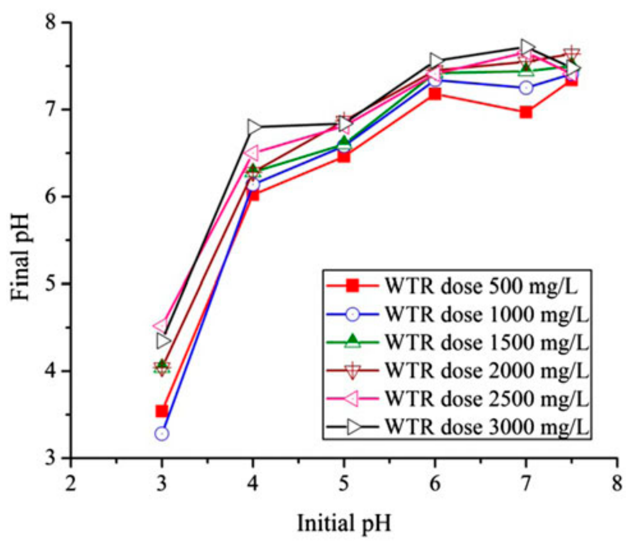

Fig. 6. Effect of WTR dose on final pH (dye conc. 50 mg/L).

3.4. Effect of initial dye concentration on color removal

Effect of dye concentration on color removal at two different initial pH values of 3.0–7.0 at a WTR dose of 2,000 mg/L is presented in Fig. 7. It is seen that at low pH (3.0) color removal was reduced with the increase in dye concentration, whereas at neutral pH (7.0) this trend was reversed, and increase in color removal was observed with the increase in dye concentration. As presented in Fig. 5 maximum color removal was obtained at a lower pH of 3.0 and at this pH reduced color removal was obtained at higher initial dye concentrations. Similar results of reduced color removal with the increase in initial dye concentration at optimum pH have been reported for acid dyes with PACl and alum and also with WTR as coagulants [26,27,36]. This phenomenon could be due to increased dye aggregation and/or depletion of hydrolysis product of the coagulant [27]. At higher pH contrary effects of dye concentration may be due to the bridging of dye particles on to WTR in the absence of ionization of dye or destabilized WTR.

3.5. Flocculation ability

Flocculation ability (Q_t) of WTR, which is defined as the amount of flocs per gram of WTR was monitored as a function of pH at different WTR doses and is presented in Fig. 8. It is seen that increase in pH resulted in reduced flocculation ability of WTR, and maximum flocculation ability of 41 mg/g observed at the lower pH of 3.0. Reduction in flocculation ability was also observed with the increase in WTR dose. The flocculation ability is greatly influenced by charge balance between cationic and anionic groups of amphiprotic flocculants [7]. This decrease in flocculation ability of the WTR at higher pH values could be due to reduced cationic charge on WTR at elevated pH as well as due to the electrostatic repulsion between the WTR and the dye molecules. In addition, as stated earlier, with the increase in WTR dose there was an increase in final pH, which could result in lower cationic charge on WTR. Thus, the present

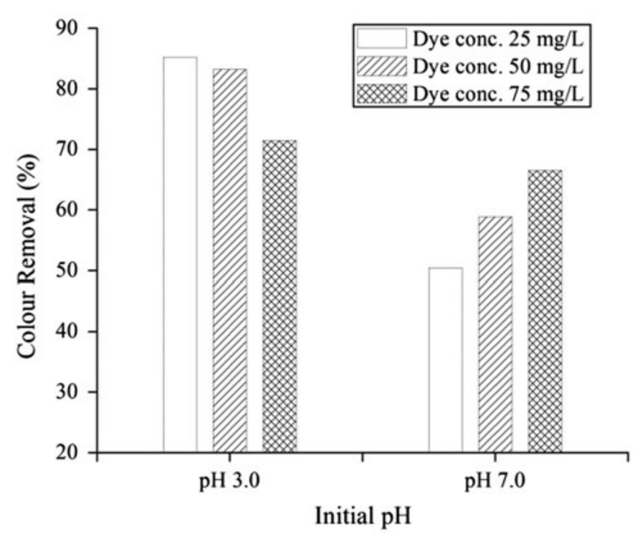

Fig. 7. Effect of initial dye concentration at acidic and neutral pH on color removal (WTR dose 2,000 mg/L).

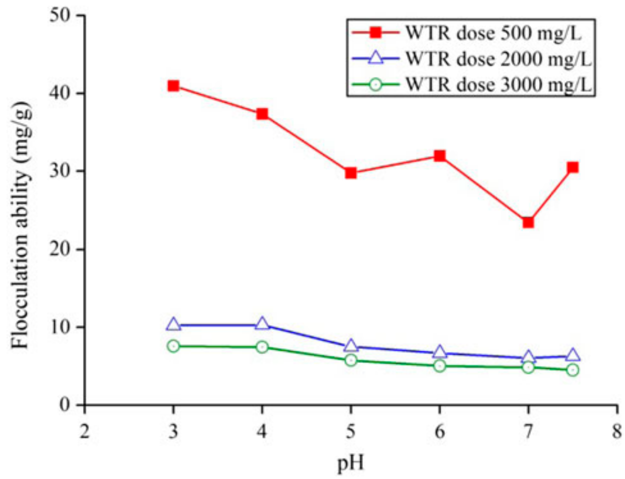

Fig. 8. Effect of pH on flocculation ability at different WTR dose (dye concentration 25 mg/L).

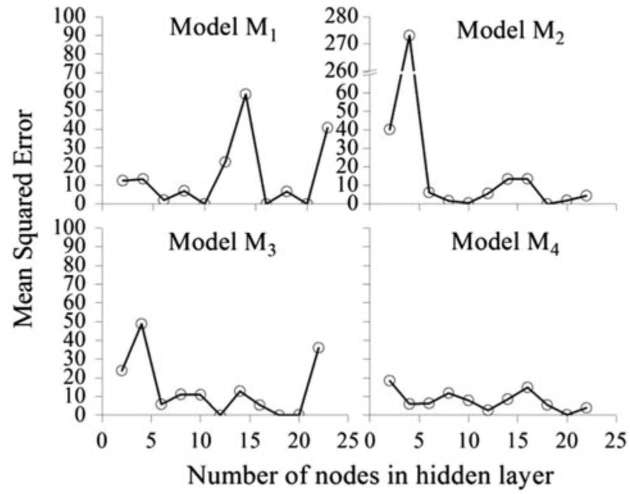

Fig. 9. Performance of ANN network with varying number of nodes different models.

results suggest that the dominant force that results in flocculation could be electrostatic attraction between anionic dye and cationic WTR, which in turn depends on pH of the system.

3.6. Simulation of color removal through ANN

As presented earlier, two types of models–M_1, M_2, and M_3 for color removal with initial dye concentrations of 25, 50, and 75 mg/L, respectively, and M_4 for color removal considering all three initial dye concentrations are employed. Modeling nonlinear systems such as coagulation process are difficult and ANN tenders the potential of generic approach to modeling of nonlinear systems [37]. In the present work, a feed forward Levenberg–Marquardt back propagation network with tan-sigmoid transfer function at hidden layer and a linear transfer function at output layer was utilized.

Number of nodes in hidden layer was selected based on the mean squared error (MSE). Fig. 9 presents MSE for different models at various nodes. For models M_1, M_2, and M_3 there were more variations in MSE with increasing nodes, whereas for model M_4, which incorporated initial dye concentration as an input parameter, MSE was low, irrespective of the number of nodes. The network with less number of nodes in the hidden layer cannot converge effectively for predictability [32]. Levenberg–Marquardt algorithm used in the study is node sensitive, and theoretically, the number of nodes in the hidden layer is directly proportional to the fitting performance of the network. However, adding extra nodes in the hidden layers leads to over-fitting, which could compromise the robustness and performance of the model [38,39]. Thus, number of nodes was selected based on the MSE and correlation coefficient (R^2) values. Topologies of the best-fitted models are presented in Table 4.

Table 4 also presents MSE, correlation coefficient (R^2) at training, validation, testing, and overall test phases, and absolute error between predicted and observed color removals. The topology selected was based on the performance of networks, which gave minimum MSE close to zero and R^2 close to one. The training R^2 in all cases of models were close to one because the same data were repeated several times for adjusting the weight of the network, whereas validation data show R^2 higher than 0.8. Test data was not supplied in training phase of network and gave R^2 greater than 0.8. Further, overall R^2 of the models were greater than 0.8 showing that the developed models are strong [40]. The absolute errors in networks were less than 3% suggesting good overall efficacy of the model for predicting color removal.

Fig. 10 presents the observed and ANN-predicted values of color removal efficiencies. The linear regression analysis between ANN-predicted and observed color removals showed linear regression coefficient (R^2) close to one, with slope approaching one and intercept value equal to 0 (Fig. 10). It is clear that the results obtained through ANN were satisfactory and thus validated the models.

A comparison of the different models show that the model that used initial dye concentration as an input parameter (M_4) performed equally well com-

Table 4
Performance of ANN network models

Model	Topology	MSE	Correlation coefficient (R^2)				Absolute error (%)
			Training	Validation	Testing	All	
M_1	3:16:1	0.0010	0.99	0.90	0.83	0.94	2.32
M_2	3:10:1	0.4600	0.99	0.90	0.95	0.97	1.73
M_3	3:20:1	0.0006	0.99	0.86	0.91	0.87	2.16
M_4	4:20:1	0.2350	0.99	0.94	0.81	0.96	2.50

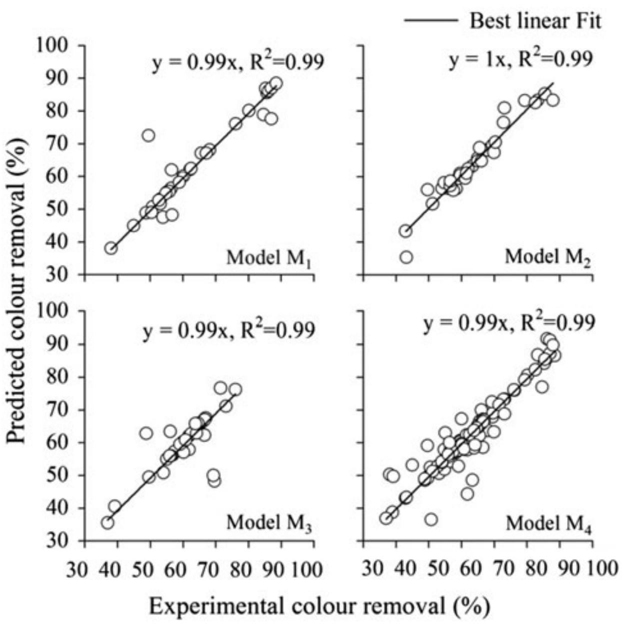

Fig. 10. Experimental and predicted color removal.

pared to the models that did not use dye concentration as an input parameter (M_1, M_2, and M_3). This suggests that the non-linear behavior of effect of dye concentration along with variation in pH was well integrated into the model for better predictability of color removal.

4. Conclusions

The usability of WTR as a coagulant for the color removal of a synthetic disperse dye (Disperse Blue 79) solution was assessed in laboratory studies. The results of this study indicated that up to 88% color removal could be obtained with WTR dosage of 3,000 mg/L and at initial pH 3. Color removal was greatly affected by the pH of the system, with lower pH showing higher removal. Different ANNs were developed to predict the color removal efficiency of WTR. The model-predicted and observed values of color removal were in close agreement with each other.

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