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The Use of Artificial Neural Network for Modeling Coagulation of Reactive Dye Wastewater Using *Cassia fistula* Linn. gum

ABSTRACT

Natural seed gum extracted from Cassia fistula Linn. (CF) was experimentally evaluated to treat reactive dye (Red 195) in an aqueous solution, whose color and Chemical Oxygen Demand (COD) were to measure the treatment efficiency. To investigate five parameters i.e. pH, reaction time, agitation speeds, dye concentration and CF gum concentration were used to implement a one-factor-at-a-time experiment with Jar-test apparatus. Carried out under weak basic condition (pH 10) for 30 min, the COD and decolorization efficiency of the dye stuff wastewater was observed at 42.4% and 57.8%, respectively. A single-layer Artificial Neural Network (ANN) model was also developed to predict the removal efficiency of the dye by using the determination coefficient (R^2) and the root mean square error (RMSE). The observed and predicted outputs were found to be 0.924 and 3.759, respectively. Furthermore, the ANN model was analysed using Garson's algorithm, connection weight method, and neural interpretation diagram to understand the influence of each operation factor on the treatment process.

Key words: *Artificial neural network; dye removal; natural coagulant; reactive red 195*

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INTRODUCTION

Wastewater of textile industry has been considered as one of the most serious sources of pollution, not only because of its toxic organic compounds but also of its color, which is aesthetically displeasing. Among classified dyes, the reactive dye is the most noticeable due to its ability to resist biodegradation (*Gottlieb et al. 2003*).

In the last few years, various oxidation methods such as ozonation (*Constapel et al. 2009*), sonication (*Sivakumar and Pandit 2001*), Fenton (*Karthikeyan et al. 2011*), etc. have been successfully applied to eliminate dye-containing wastewater. Nonetheless, most of these techniques are limited by engineering practice, costs or difficulties in operation leading to limited application in real scenarios of dyeing wastewater treatment (*Asghar, Raman and Daud 2015*).

Coagulation and flocculation are traditional techniques using inorganic coagulant (alum, ferric chloride, polyaluminium chloride, etc.) or organic polymers (polyacrylamide,polydiallyl-dimethyl-ammonium chloride, polyacrylic-acid-coacrylamide, etc.) to alter the physical state of pollutant particles and facilitate their removal by sedimentation. These techniques have been successfully used for removing remaining dye from textile wastewater over many years (V*erma, Dash and Bhunia 2012*). However, these techniques usually produce a large volume of difficultly thickening sludge which increases operational costs. In addition, the use of metal-based coagulants has recently been shown to cause Alzheimer disease in human (*Kawahara and Kato-Negishi 2011*).

Recently, the use of locally available plant based coagulants such as *Moringa oleifera* (*Ndabigengesere and Subba Narasiah 1998*), *Plantago psyllium* (*Mishra, Srinivasan and Dubey 2002*), *Ipomoea dasysperma* (*Sanghi et al. 2006*), etc., have been gaining attention in developing countries because of their efficiency and economical factor. The polysaccharides extracted from plants have functional groups serving as biodegradable coagulants. They can replace traditional coagulants in order to reduce secondary pollution caused by toxic sludge. Among these polysaccharides, galactomannan gum extracted from the seeds of *Cassia fistula* Linn. (CF), a common solid waste in Vietnam, can be used as an alternative low-cost coagulant. (*Singh, Sethi and Tiwari 2009*).

Along with choosing removal techniques, modelling its process is also a significant task. The quality of textile wastewater often varies depending on the type of color and the phenomenological treatment of coagulation which is also influenced by several factors such as coagulation dosage, pH, reaction time, etc.. Thus, the treatment of textile wastewater by coagulation is quite complicated. Especially, it becomes more difficult to model or simulate using conventional mathematical method in poorly designed experiments (*Maier and Dandy 2001*). In the recent decades, the artificial neural network (ANN) has been used extensively for modelling the water and wastewater treatment processes (*Lamrini et al. 2005*). This method enables us to understand what is happening in a process without actually modelling the physical and chemical laws governing the system (*Daneshvar, Khataee and Djafarzadeh 2006*) Therefore, ANN promises an alternative technique for modelling the up-scaling complex coagulation of textile wastewater.

This work was accomplished with two groups of experiment. First, the dye removal capacity of CF gum was evaluated through Jar-test experiment based on five selected parameters including pH, agitation speed, initial dye concentration (IDC), gum dosage and reaction time. Second, the neural network model for the prediction of CF gum coagulation efficiencies was developed.

MATERIALS AND METHODS

Cassia fistula gum's characteristic and the stock preparation

The seeds of CF tree were collected in a park (located in Phu Nhuan district, Ho Chi Minh City, Vietnam) in January 2013. The extraction of polysaccharide (crude gum) from CF seeds was prepared according to *Singh, Sethi and Tiwari* (2009) and was used without any purification. The characteristic of the extracted gum could be found in the previous study of *Perng and Bui* (*2015*) and the analysed spectrums (Fourier transform infrared spectroscopy, high performance liquid chromatography, and gel permeation chromatography) confirmed that CF gum has the galactomannan polysaccharide structure with the average molar ratio of mannose and galactose about 3.5:1. Therefore, the gum structure has the potential for reducing the concentration of the reactive red 195 in aqueous solution.

The stock solution of CF gum was prepared as follows: 500±1 mg of gum powder was weighed and then poured into a 250-mL beaker; distilled water (5000 mg L^{-1}) was filled into the beaker up to the 100 ml mark; the mixture was stirred and heated for 5 min and was sonicated for an additional 60 min. Then, the solution was stored in a refrigerator at 5°C. This stock was diluted to the desired mass concentrations (from 100 mg L^{-1} to 350 mg L^{-1}) before use.

Reactive dye stock

The commercial reactive red 195 (Sunfix Red S3B 100%) was obtained from Oh-Young (a Korean company). Its molecular structure and absorption spectra are shown in **Figure 1**.

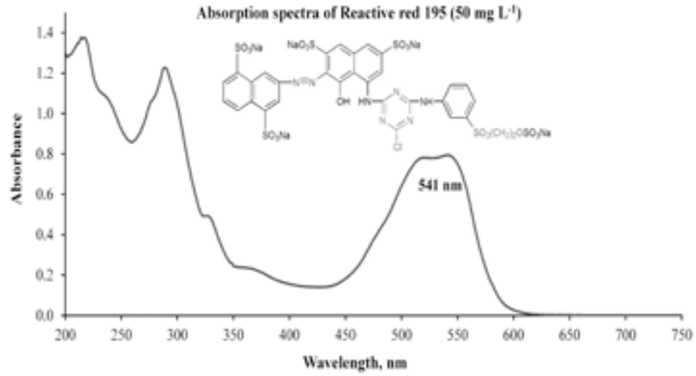

Figure 1. Chemical structure and spectral properties of reactive red 195 (*Perng and Bui 2014*).

The dye stock solution (1000 mg L^{-1}) was prepared by completely dissolving 1 g of dye powder into 1 L of distilled hot water at pH 11 for an hour to get the dye stock in the "hydrolyzed" form and the solution was diluted to the appropriate concentrations (10-140 mg L^{-1}) before use.

Decolorization experiments

Jar-test apparatus was used to conduct coagulation studies with traditional method described by *Ndabigengesere and Subba Narasiah* (*1998*). The initial conditions for the coagulation treatment are as follows: gum dosage 100 mg L^{-1}, initial dye concentrations (IDC) 50 mg L^{-1}, time (t) 30 min, and agitation speed (A) 60 rpm (**Table 1**). This study was designed based on one-factor-at-a-time experiment, i.e. in every series of experiments, only one independent factor varies while all other factors were kept constant.

Artificial neural network model

All ANN calculations were carried out using Matlab 7.6 software associated with ANN toolbox for Windows operating system and running on a personal desktop computer (Intel core i3, 3.7 GHz). The ANN model was constructed as a three-layer feed-forward back-propagation network with five inputs (**Table 1**). Accordingly, the two outputs were obtained as dependent variables which are: COD and color removal efficiencies. The experimental results were

Table 1. Experimental steps, evaluated factors and established ranges for the dye removing test.

Experimental Steps	Factor	Range of values
1	pH	3-12
2	Agitation speed (A), rpm	25*- 120
3	Reaction time (t), min	10-90
4	Gum dosage, mg L^{-1}	100-350
5	Initial dye concentration (IDC), mg L^{-1}	10-140

*The lowest speed of Stuart flocculator.

randomly divided into two groups (i.e. 80% in the training set and 20% in the test set), which were used as the data sets for the modelling. The Bayesian regulation algorithm (trainbr) was used as a training function to update the weight and the bias values. A sigmoid tangent was used as the transfer function in the hidden and output layers.

The performance of the ANN model was evaluated based on the maximum value of the coefficient of determination (R^2) and the minimum value of the root of mean square error (RMSE) of the whole set, as shown in following equations (1) and (2).

$$R^2 = 1 - \frac{\sum_{i=1}^{N}(d_i - y_i)^2}{\sum_{i=1}^{N}(y_i - \bar{y})^2} \quad (1)$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(y_i - d_i)^2}{N}} \quad (2)$$

Where N is the number of data points, d denote the desired target obtained from the ANN model, respectively; and $\bar{y}$ are the mean of the observed data.

The relative importance of the input variables was also determined according to the connection weight method (Eq. 3), Garson algorithm (Eq. 4) and illustrated by the neural interpretation diagram (*Elmolla, Chaudhuri and Eltoukhy 2010*).

$$V_{io} = \sum_{j=1}^{h}(W_{ij} \times W_{jo}) \quad (3)$$

$$Q_{io} = \frac{\sum_{j=1}^{h}\left(\frac{|W_{ij}|}{\sum_{i=1}^{n}|W_{ij}|}|W_{io}|\right)}{\sum_{i=1}^{n}\left(\sum_{j=1}^{h}\left(\frac{|W_{ij}|}{\sum_{i=1}^{n}|W_{ij}|}|W_{io}|\right)\right)} \quad (4)$$

Wherein, Q_{io} is the relative significance of the ith input variable upon the output variable; *n* and *h* denote the number of input and hidden neurons, respectively. W is the connection weight value. The subscripts "*i*" "*j*" and "*o*" represent the input, hidden, and output neurons, respectively.

Analytical procedures

The dye removal efficiencies were measured with the decreasing percentage of 541 nm absorbance and with decreasing percentage of chemical oxygen demand (COD) values between initial and final data (after a 30 min settling) of dye solutions using a UV-VIS Hach DR 5000 spectrophotometer. The COD values were determined by closed reflux colorimetric method according to the Standard Methods of the American Public Health Association (*Clesceri, Greenberg and Eaton 1998*) and the pH value was determined using a digital pH meter model 744, Metrohm Ltd., Swiss. All analyses were conducted twice and the results presented here are the average of both ± standard deviations.

RESULTS AND DISCUSSION

Coagulation Studies

The dye removal effects of CF gum were studied by using one-factor-at-a-time Jar-test experiment. Hereafter, the influences of each factor shall be discussed in the order as carried out in the actual experiments.

pH

It seems that pH is an important parameter influencing the performance of dye coagulation process (*Assadi, Amin, and Nateghi 2013; Sanghi et al. 2006*). To investigate the effect of pH, the sample was modified to the desired pH values (i.e. 3, 7, 10 and 12) by using 0.5 N NaOH or HCl solutions with other factors remained constant: gum dosage 100 mg L^{-1}, time contact 30 min, agitation speed 60 rpm, initial dye concentration (IDC) 50 mg L^{-1}.

The predicted values and experimental values of the dye removal efficiencies (COD and color removal) were plotted for comparison (**Figure 2**). This is considered as a function of the initial pH with 4 neurons in the hidden layer. From this plot it can be seen that obtained results from the proposed ANN model are in good agreement with the experimental data. The maximum removals of the dye were

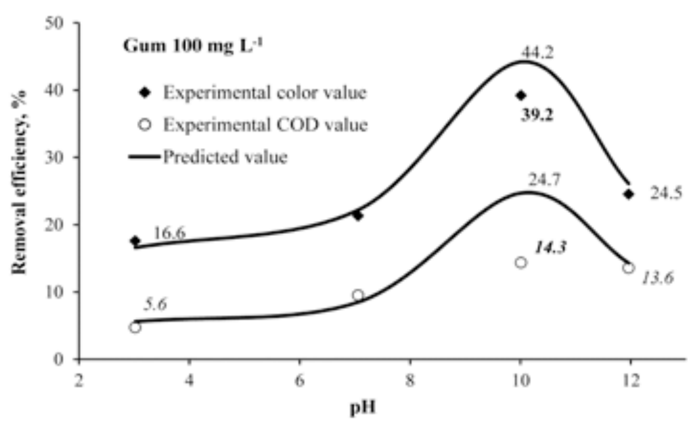

Figure 2. Influence of pH on removal efficiencies for the dye using *CF* gum (IDC 50 mg L^{-1}, 30 min and 60 rpm).

39.2% and 14.3% for color and COD removal efficiencies at pH 10, respectively. This pH value quite matches with the research of *Sanghi et al.* (*2006*) on *Ipomoea dasysperma* gum (pH 9.5). This observation may be explained by the easier formation of intermolecular force between the π electron system of the dyes and the cis-hydroxy groups in the galactomannan of the CF gum at pH 10 compared with other pH values (*Blackburn 2004*). The proposed mechanism of the dye and gum is illustrated in **Figure 3**.

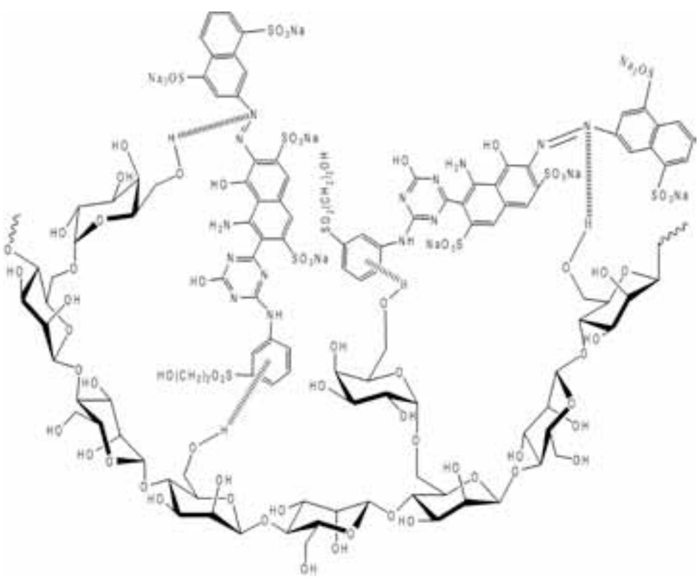

Figure 3. The hypothetic interaction of the *CF* gum and the reactive red 195 (*Blackburn 2004*).

Agitation speed

A suitable agitation speed would make the particles precipitated enough. Therefore, aggregates can be formed larger and larger, bringing optimal coagulation efficiency. Thus, it is critical to investigate the effect of agitation speed during coagulation process. The experimental and ANN calculated values of the removal percent (COD and color) for the dye solution with various agitation speeds are shown in **Figure 4**.

The best experimental results were obtained at 45 rpm (44.7% and 26.3% for color and COD removal efficiencies, respectively). The result is quite similar to the agitation speed recommended by *Tatsi et al.* (*2003*). Hence, this agitation speed was chosen for subsequent experiments.

Reaction time

Together with the agitation speed, reaction time at slow mixing phase between coagulant and dye solution also plays a relevant role. To investigate the effect of reaction time on the efficiencies of dye removal, the Jar-test experiments were carried out at various reaction times (15-90 min). It can be seen that the dye removal efficiencies of CF gum rapidly increase when reaction time increases from 10 min to 30 min, and then tend to be constant (even slightly decrease) when the reaction time incremented further (**Figure 5**). The mild drop in removal efficiencies above is likely due to the restabilization of the aggregated particles which reduces the interactions between the dye and the gum (*Sanghi, Bhattacharya and Singh 2002*). So the suitable reaction time for the next experiments is 30 min.

Coagulant dosage

In order to investigate the effect of *CF* gum amount on the dye removal, a series of experiments were carried out in a wide range of gum dosage of 100-350 mg L^{-1}.

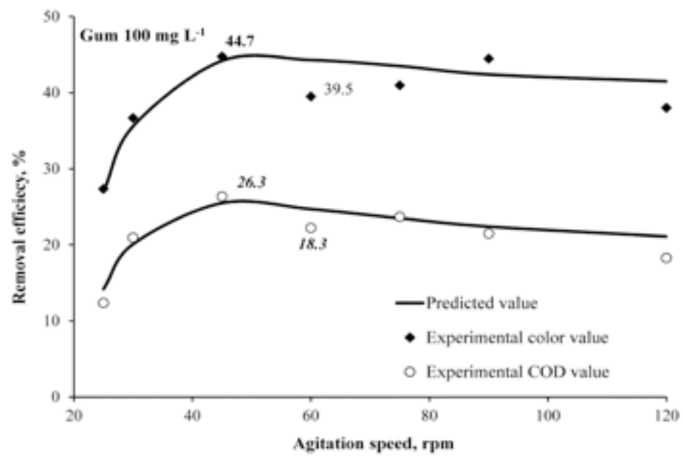

Figure 4. Influence of agitation speed on the removal efficiencies of the dye using *CF* gum (IDC 50 mg L^{-1},30 min and pH 10).

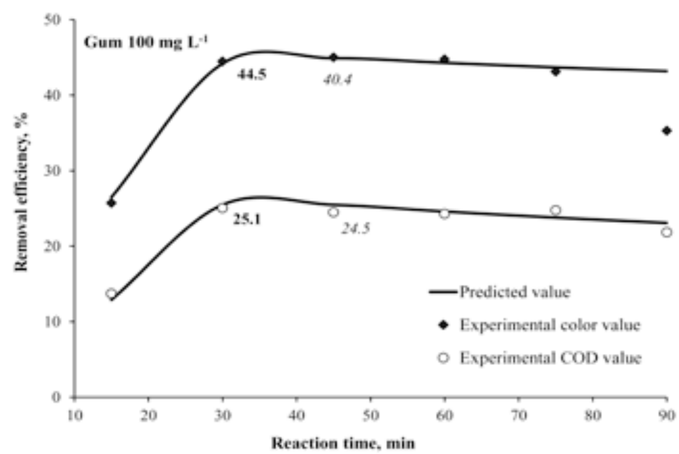

Figure 5. Effect of reaction time on the removal efficiencies of the dye using *CF* gum (IDC 50 mg L^{-1}, 45 rpm and pH 10).

It can be observed from **Figure 6** that the removal yields initially increase to 54.3% and 40.9% for color and COD removal efficiencies, respectively with CF gum dosage up to 200 mg L^{-1}. However, efficiencies seem to be decremented

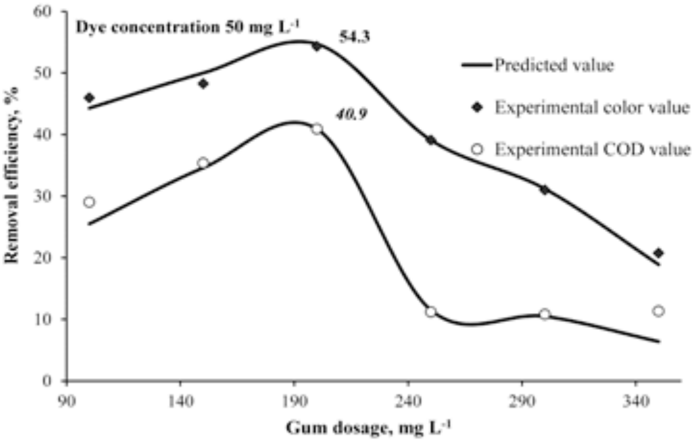

Figure 6. The effect of coagulant concentration on the removal efficiencies of the dye using *CF* gum (30 min, 45 rpm and pH 10).

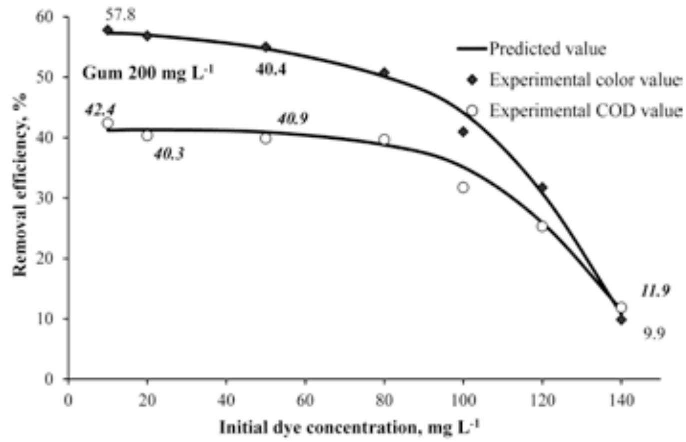

Figure 7. The effect of initial dye concentration on the removal efficiencies of the dye using *CF* gum (30 min, 45 rpm and pH 10).

under further increase of the gum dosages. A possible explanation is that the appropriate dosage of CF gum can cause the dye particles to aggregate (destabilization) and settle out, so that gum-dye bridging occurs (*Blackburn 2004; Sanghi et al. 2006*). Then, when the CF gum dosage in the solution exceeds an optimal threshold, there will be not enough bare dye particles with unoccupied surface available for the attachment of CF gum segments (*Sanghi, Bhattacharya and Singh 2006*). This results in a reduction of CF gum-dye bridging and the solution restabilizes. Hence, the optimal CF gum dosage of 200 mg L^{-1} was chosen for the next series of experiments.

Initial dye concentration (IDC)

Several experiments were conducted with different initial dye concentrations (IDC) in the range of 10–140 mg L^{-1} and other constant parameters: optimal pH (10), agitation speed (45 rpm), gum dosage (200 mg L^{-1}), and contact time (30 min). **Figure 7** presents the relationship between the experimental and ANN predicted values of the dye removal percentage versus the IDC. Accordingly, the maximum removal efficiencies reach 57.8% and 42.4% at the lowest IDC (10 mg L^{-1}), and then when IDC increases, they decreased and reached 9.9% and 11.9% at the highest IDC (140 mg L^{-1}) for color and COD removal efficiencies, respectively. Particularly, from 10 mg L^{-1} to 80 mg L^{-1} of IDC, the efficiencies seem to be decreased more steadily than at the later range of IDC.

The "flat then drop" variations in **Figure 7** can be explained by the relatively fixed ratio between coagulant particles and removed dye particles. This ratio is determined by the binding strength between the two kinds of particles (*Buthelezi, Olaniran and Pillay 2012*). As the IDC increases while the gum dosage being fixed, the amount of removed dye particles is relatively fixed and the amount of unremoved particles increases; Thereby, resulting to led to the decrease of removal efficiencies. As the IDC decreases, the removal yields increase but then becomes nearly unchanged after the dye concentration passed a threshold relative to the fixed gum dosage.

Evaluation of ANN model for the dye removal process

As mentioned in the experimental section, the basic topology for ANN consist five input parameters i.e. initial pH, reaction time, agitation speed, gum dosage and initial dye concentration (IDC) and two outputs, color and COD removal efficiencies. Four hidden neurons are chosen in order to obtain the best performance (data not shown due to space limitations). The performance of ANN model was estimated by R^2 and RMSE values.

In the comparison between the dye removal practical values and the predicted values using the ANN model, the figure consisted of two lines, one is the ideal fit Y = X (predicted data = experimental data) and the other is the best fit indicated by a solid line with the liner equation Y = 0.971X + 1.929, correlation coefficent R^2 0.924 and RMSE 3.759 (**Table 2** and **Figure 8**).

This indicates a high correlation coefficient between the measured and predicted output variables and this value agrees well with the modeling correlation coefficient of some dyes in aqueous solution reported in the literatures e.g. 0.950 for the prediction of vat green 1 dye decolorization by nano zero valent Iron (*Arabi and Sohrabi 2014*), 0.945 for prediction of acid red 119 decolorization by PAC (*Moghaddam, Moghaddam and Arami 2011*), and 0.945 for the prediction of acid red 119 decolorization by electrocoagulation method using alum electrode (*Taheri, Moghaddam and Arami 2012*). Therefore, the neural network modeling effectively simulated and predicted the influential factors in gum coagulation process.

Garson's algorithm and connection weights algorithm were used to determine the influence of each factor on outputs (**Table 3**). The neural interpretation diagram was also plotted to evaluate the accuracy of these algorithms (**Figure 9**).

For decolorization process, with Garson's algorithm the most influential factor is time (I_4). The increase in reaction time influenced positively the color removal efficiency and were confirmed by connection weight algorithm. So it is concluded that reaction time is a truly possitive factor for decolorization process. Similarly, the second influential factor, agitation speed (I_3) was approved by both of the two algorithms. However, in the case of C_{gum} (I_5) factor, it was the third influential factor based on Garson's algorithm, but it presented a minor or neutral effect to decolorization process according to connection weight method. This complication may come from the fact that the factor had both positive and negative effect to decolorization efficiency (**Figure 6** and **Figure 9**). The negative influences of I_5 to the decolorization process (O_1) was hidden neuron 1 (H_1) and 2 (H_2) while the possitive influence of the factor to the decolorization process hidden were neuron 3 (H_3) and 4 (H_4). Among them, the positive influence of I_1 to H_1 was predominated. Therefore, the total influence of I_5 to O_1 was weakly negative or neutral. Other remaining factors, the dye concentration (I_1) and pH (I_2) which followed this model do not have significant effect to decolorization yield.

For COD removal efficiency, I_3, I_4 and I_5 were still the key influence factors. However, these influences to COD removal were more complex than decolorization process. I_5 was the most influencing factor and had a negative trend to the COD removal efficiency (O_2). I_5 to O_2 by H_1 and H_2 has a negative effect, while I_5 to O_2 by H_3 and H_4 has a the positive effect. The trends were similar to the decolorization process, whereas the quantity was stronger (**Figure 9**), Especially, in the case of H_2 interaction. The following infuence factors were I_3 and I_4 that were "absolutely" positive. Therefore the connection weight indices of I_3

Table 2. The observed and predictive values by ANN of gum coagulation process for the dye removal.

Run No.	*IDC, mg L^{-1}	pH	A, rpm	T, min	C_{gum}, mg L^{-1}	% Decolorization		% COD removal	
						Observed	Predicted	Observed	predicted
1	50	3.03	60	30	100	17.6	16.6	4.7	5.6
2	50	6.97	60	30	100	21.3	22.2	9.5	8.5
3	50	9.97	60	30	100	39.2	44.2	14.3	24.7
4	50	12.07	60	30	100	24.5	44.1	13.6	24.3
5	50	10.0	25	30	100	27.4	27.0	12.4	14.2
6	50	10.0	30	30	100	36.7	35.6	20.9	20.1
7	50	10.0	45	30	100	44.7	44.2	26.3	25.5
8	50	10.0	60	30	100	39.5	44.3	22.2	24.7
9	50	10.2	75	30	100	41.0	43.5	23.7	23.5
10	50	10.0	90	30	100	44.5	42.4	21.5	22.4
11	50	10.3	120	30	100	38.0	41.5	18.3	21.1
12	50	10.07	45	15	100	25.7	26.5	13.7	12.9
13	50	10.10	45	30	100	44.5	44.2	25.1	25.5
14	50	10.05	45	45	100	45.0	44.9	24.5	25.5
15	50	10.01	45	60	100	44.7	44.3	24.3	24.6
16	50	10.03	45	75	100	43.1	43.7	24.8	23.8
17	50	9.99	45	90	100	35.3	43.2	21.8	23.1
18	50	10.20	45	30	100	46.0	44.3	29.0	25.5
19	50	10.03	45	30	150	48.2	50.0	35.4	34.6
20	50	10.01	45	30	200	54.3	54.7	40.9	40.9
21	50	10.05	45	30	250	39.1	39.1	11.2	11.5
22	50	10.03	45	30	300	31.0	31.2	10.8	10.5
23	50	10.01	45	30	350	20.8	18.9	11.3	6.4
24	10	10.0	45	30	200	57.8	57.3	42.4	41.2
25	20	10.0	45	30	200	56.8	57.0	40.3	41.3
26	50	9.97	45	30	200	55.0	54.7	39.9	40.9
27	80	10.0	45	30	200	50.8	50.1	39.6	38.8
28	100	10.0	45	30	200	40.9	44.1	31.8	35.1
29	120	10.0	45	30	200	31.7	30.8	25.3	25.9
30	140	10.0	45	30	200	9.9	10.8	11.9	11.5

*C_{gum}: gum dosage, IDC: initial dye concentration, T: reaction time and A: agitation speed

Table 3. Contribution of each input factor to output values on the coagulation process using *CF* gum.

Process	Output	Factor	Garson's algorithm, %	Rank	Connection weight	Rank	Influence
Gum coagulation	Decolorization (O_1)	Dye con. (I_1)	13.90	4	-2.94	3	negative
		pH (I_2)	12.65	5	2.81	4	positive
		Agitation (I_3)	27.78	2	5.14	2	positive
		Time (I_4)	29.32	1	6.06	1	positive
		Cgum (I_5)	16.35	3	-1.83	5	negative
	COD removal (O_2)	Dye con. (I_1)	9.94	4	-2.70	4	negative
		pH (I_2)	1.32	5	-0.22	5	negative
		Agitation (I_3)	22.50	2	4.23	3	positive
		Time (I_4)	20.36	3	5.48	1	positive
		Cgum (I_5)	45.88	1	-5.00	2	negative

*Rank of the absolute values

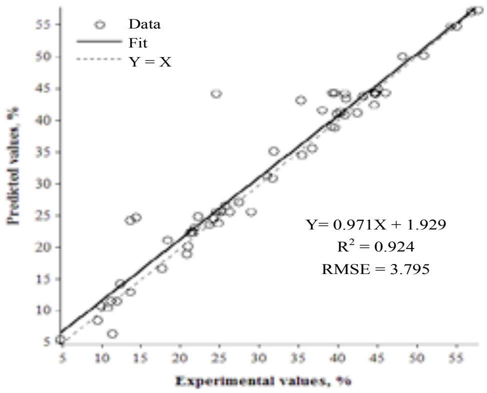

Figure 8. Comparison between predicted from ANN model and experimental values of the outputs.

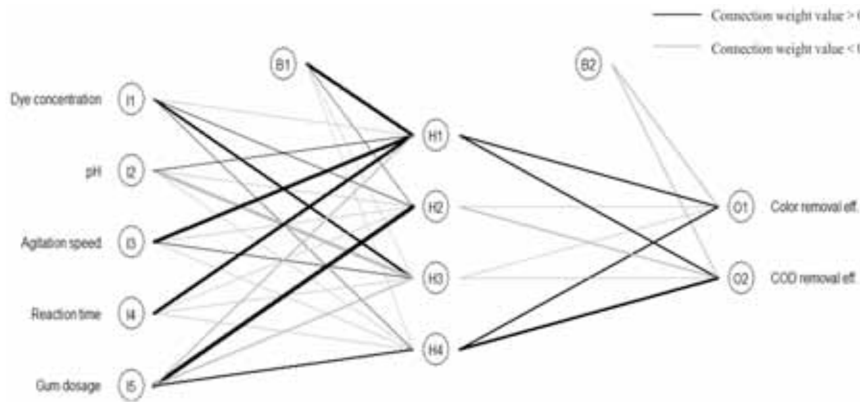

Figure 9. Neural interpretation plot of red 195 coagulation using CF gum.

and I_4 were more reliable than those under the Garson's algorithm's column. Corresponding with decolorization process, two factors pH and dye concentration have minor effect to COD removal efficiency.

CONCLUSIONS

Cassia fistula Linn. (CF) seed gum was investigated as a coagulant in the dye removal process of reactive vinyl sulfone dyes Red 195. The best treatment efficiencies for color and COD reached 57.8% and 42.4%, respectively using Jar-test experiment.

The newly proposed ANN model successfully predicted the coagulation process with determination coefficient (R^2) and the root mean square error (RMSE) found to be 0.924 and 3.759, respectively. Based on the model and sensitivity analysis methods (Garson algorithm, connection weight method and neural interpretation diagram), it can be concluded that the efficiency of coagulation process were highly dependent on agitation speed (I3), reaction time (I4) and concentration of gum C_{gum} (I5). This leads to a great potential of ANN as a robust tool for modeling coagulation process and for analyzing the effect of each factor on dye removal process results.

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