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Evaluation of Regression model development of the experimental results for dye removal by electrocoagulation

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Abstract

This paper illustrates the treatment of dyes from textile wastewater using electrocoagulation with adding alum. The effects of various operational parameters such as rotational speed of the anode, current intensity, contact time, initial concentration of dyes and amount of added alum (20mg/l) on the removal efficiency of contaminants in terms of color and chemical oxygen demand (COD) were examined and it also aimed to make a comparison between results of electrocoagulation without adding alum and with adding alum. Regression analysis was employed to develop a prediction model for dye removal. The coefficient of determination of Excel model is (R^2=0.908) and The ANN model yielded a coefficient of determination (R^2=0.928). Current intensity is the most important factor that controls the interaction rate in the electrocoagulation cell and initial concentration of dye has no effect on COD and dye removal efficiencies.

keywords

Electro coagulation, removal efficiency, coefficient of determination.

1. Introduction

The textile sector is the second biggest job provider across the globe, a most important sector in a country's economy and offers huge job opportunities. The textile product industries have an important contribution to the Egyptian economy. Textile wastewater imposes serious environmental problems because of their high color, high chemical oxygen demand (COD), PH and high temperature (Kim et al. (2002, 2004); Wei et al. 2015). There are many problems of dyes such as: the greatest environmental concern with dyes is their reflection and absorption of sunlight entering the water, Light absorption minimize photosynthetic activity of algae and greatly influence on chain of the food, dyes can remain in the environment for an extended period, due to photo stability, and a lot of dyes and their breakdown products are carcinogenic and toxic to life (Paz et al. 2017).

EC has been successfully used to remove of contaminants such as dyes from various industrial wastewaters (Kobya et al. 2017). The EC technique is a clean technology. There are some merits of electrocoagulation such as Simple equipment , Easy operation, Shortened reaction period, decrease or absence of equipment for chemicals adding and decreased quantity of sludge which sediments rapidly (Kobya et al. 2016; Fajardo et al. 2015; Kim et al. 2002; Nandi& Patel 2017).

In the literature , Choudhary and Mathur studied Effect of rotation speed on efficiency of rotating anode in electrocoagulation cell and found A higher removal of COD was 96.40%, efficiency of color removal (99.88%) and low consumption of energy (0.028 J/mg) (Choudhary & Mathur 2017).

2. Theory of Electrocoagulation

In this operation, the anode resolves the coagulant (aluminum electrodes) with an immediate hydrogen gas production and hydroxyl ions at the cathode. The reactions at the anode, in the EC process involving the aluminum electrodes, are as follows:

Anode:

Al → Al^{+3}+ + 3 e^{-} (1)

The reaction at the cathode is:

$2H_2$O + $2e^{-}$→H_2 + 2OH^{-} (2)

When the anode potential is sufficiently high, the following secondary reaction can also happen at the anode:

$2H_2$O→ o_2+ $4H^{+}$+ $4e^{-}$ (3)

In the solution:

Al $(OH)^{+2}$+H_2O→ Al$(OH)_3$ + H^{+} (4)

The generated metallic ions go through further ordinary reactions to give insoluble corresponding hydroxides. These hydroxides-metallic compounds in eq (4) have a strong ability to adsorb the contaminants. Both hydrogen and oxygen gas are evolved near the anode and cathode as each gas bubble nucleates. These bubbles carried the contaminants (dye particle) to the liquid surface as shown in fig (1) (naje et al. 2016).

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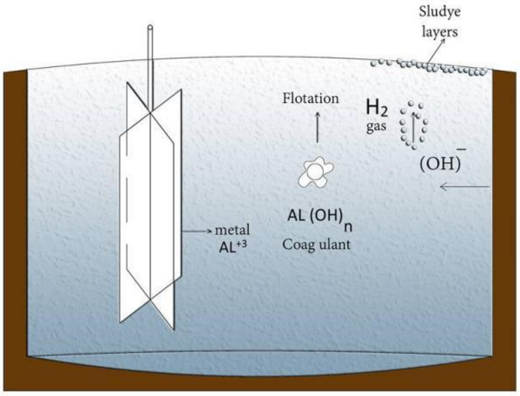

Fig (1) Electrocoagulation mechanism

When aluminum sulfate (alum) is added to water or wastewater, Al (OH) $_3$ precipitation occurs as described by the following equation (Huseyin Selcuk 2005) :

$$Al_2(so_4)_3 + 6\ HCo_3 \leftrightarrow 2\ Al(OH)_3 + 3\ sO_4 + 6\ co_2 \quad (5)$$

There are two types of electro oxidation (direct and indirect oxidation) .In indirect electrooxidation, Chloride salts of sodium or potassium are added to the wastewater .When the chlorides were presented in the samples the products from anodic discharge of chlorides were Cl_2 and OCl−. The OCl− is a strong oxidant, which capable to oxidize organic compounds presented in the wastewater (Tak et al. 2015).

When adding NaCl in solution there are three equations:

$2\ Cl^- - 2e \rightarrow Cl_2$ (6)

$Cl_2 + H_2O \rightarrow HOCl + Cl^- + H^+$ (7)

$HOCl \leftrightarrow OCl^- + H^+$ (8)

3. Materials and methods

3.1. Experimental set-up

Experiments were implemented in a batch electrochemical circular glass cell shown in Fig. 2 that had the following dimensions: 15 cm diameter and 20 cm height. The total volume of 2 liters of wastewater was treated in the electrochemical cell with 15 cm wetted depth and 5 cm free board. A rotating anode in the center of electrocoagulation cell and composed of rod with four shafts. Every shaft had the following dimensions: 2.5 cm width, 15cm height and .3 cm thickness. The rotating anode was attached to a motor with adjustable speed in order to maintain the anode rotations. The motor is DC electrical type and supplies various steady state speeds (20rpm, 40 rpm and 80 rpm). The cathode was a cylindrical roller with 11cm diameter and 17 cm height. The gap between cathode and reactor 1.5 cm whereas it was 3 cm between cathode and anode. These electrodes were made of aluminum and immersed height was 12 cm and there is clearance of 3 cm between electrodes and the bottom of reactor .The metal electrodes were dropped to the wastewater sample and connected to digital multi meter, KEW SNAP model-2012, for measurement the current and the potential between the electrodes. The D.C. power supply output had three different current conditions: .4 A, .7 A and 1 A with the volts of 13V, 17V and 21V, respectively. There is a stock of alum with concentration (20 mg/l) that it is used to add different amount of alum in reactor.

3.2. Industrial wastewater:

Synthetic wastewater was set by adding dosages of dye (reactive blue 19) and 1 gm/l of NACL to 1 liter of tap water and mixed for 3 min. The mixture showed a uniform blue color. The wastewater was prepared by initial dye concentrations of 100 mg/l, 200 mg/l and 300 mg/l. The initial conductivity ranged from 2295 µs to 2308µs.The initial pH ranged from 5.9 to 8.2.Textile wastewater sample was collected from El Nasr Co. For Spinning, Weaving & Pigment in mahalla with initial PH,conductivity , TDS and initial dye concentration are 6.68,2140 µs ,1427mg/l and 80 mg/l respectively.

3.3. Experimental method

First EC-cell (batch system) was filled with 2 litre of synthetic wastewater at room temperature (25° c). Electrodes were submerged and then the current was passed by the regulated DC power supply. The overall efficiency of the reactor was tested using three main variables; reaction time, current intensity and the anode's overall rotational speed. The electrolysis time (RT) was maintained in the range of 0 to 20 minutes each 5 minutes. Three main current intensities (CI); .4, .7, 1 A with various steady-state anode rotational speeds of 0, 20, 40 and 80 rpm were examined. The reaction was timed, beginning when the D.C. power supply and D.C. motor were switched on. Samples of 15 ml of wastewater were withdrawn from the depth of 5 cm below the free surface of wastewater at regular time intervals of 5 minutes. The effect of the electrochemical treatment was determined by measuring concentration of dye at the regular time intervals of 5 min. . COD was measured at the beginning and at the end of the run. After each run the electrodes were cleaned and rinsed with HCl

(10%concentration) to remove the oxides formed at the anode surface and then dried. The EC batch rounds were executed 18 times, for real textile wastewater were 4 times and for synthetic reactive blue 19 dyes were 14 times.

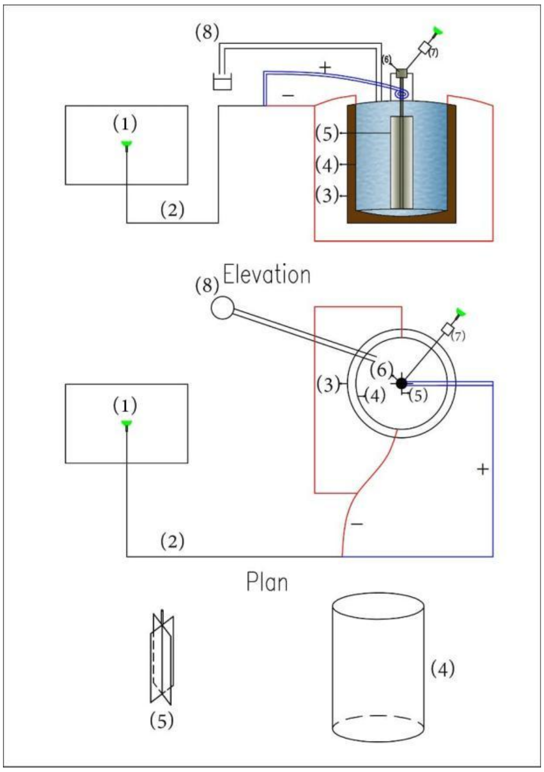

Fig. 2 schematic diagram: (1) DC power supply (2) Electrical wires (3) Electrocoagulation cell (4) Cathode (5) Rotating anode (6) DC motor (7) speed regulator (8) alum solution tank.

3.4. Mechanism of adding alum (chemical coagulation)

The alum solution (20 mg/l) was added with different amount (5,10,15 ml) after the reactor was set up. Rapid mixing was implemented with rotational speed (100 r.p.m) for the first 1 minute and slow mixing was implemented with (20 r.p.m) for 20 minute after the Dc power supply switched on.

3.5. Analytical measurement& Modeling of experimental results

The experimental parameters measured were initial concentration of dye, COD, conductivity, TDS and pH. Analysis was carried out by the standard method for the examination of water and wastewater (22nd edition,

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2012). the colour was also estimated using a 4802 uv/vis a double beam spectrophotometer (λ → 585) (18). The removal efficiency of dye was determined as (A0 -A)/A0. In order to accomplish the aims of these study three sets of runs were planned using concentration of dye 200 mg/l. the three sets of runs were done using 21 V, 17V and 13V, respectively. Each set of them contained four experiments with different rotating anodes (0, 20,40, 80, rpm.). The four set of runs were planned at 13 V and 20 r.p.m using three different concentration of dye (100,200,300 mg/l). The last set (five set) was done on textile wastewater samples with different amount of alum.The experimental work has been extended to cover different operational conditions of EC with adding alum for the dye removal efficiency. The results were combined in one database. The database was used to create a model for the prediction of the dye removal efficiency. Regression analysis by the least square method was used for model development. In addition, an artificial neural network (ANN) model was developed based on Tanch axon function to describe the experimental results.

4. Result and discussion

4.1. Comparison between electrocoagulation of wastewater samples without alum added and with alum added

4.1.1 Sludge production

The volume of sludge was 141 cm^3 in case of no chemical added and it was 176 cm^3 in case of 5 ml of alum for real textile wastewater samples at 0.4 ampere current intensity and 20 r.p.m rotational speeds as shown in table (1) .

It is well known that increasing amount of alum increases the volume of sludge due to the sludge resulting from adding chemicals but the removal efficiency of dye don't exceed 90 %. The volume of sludge was 212 cm^3 in case of 10ml of alum and it was 265 cm^3 in case of 15 ml of alum for real textile wastewater samples at 13 volt current voltage and 20 r.p.m rotational speeds as shown in table (1).

It is well known that adding chemicals increased volume of sludge and this is the main drawback of chemical coagulation. This explained by most of $AL(OH)_3$ particles formed in the reactor according to eq(5) adsorbed dye particles and some of them were exceeded and extracted with sludge . Firstly, additional sludge is formed due to enhanced removal of suspended and colloidal particles. Secondly, a portion of additional sludge volume is due to chemical sludge, which is formed especially in case of metal salts due to the formation of metal hydroxide (Yuksel et el. 2011).

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Table (1) Results of Sludge (thickness and volume) for textile wastewater samples (13 Volt-0.4 Ampere-20 r.p.m)

RUN NO	Amount of alum added (ml)	Thickness of Sludge (cm)	Volume of Sludge (cm^3)
1	0	0.8	141
2	5	1	176
3	10	1.2	212
4	15	1.5	265

4.1.2. PH and conductivity

Initial PH was 6.68. Final PH was 9.62 in case of no chemical added. At this cathode, water molecules dissociate into hydroxyl ions according to eq (2) that leads to increasing the pH medium. Final PH was 7.29 in case of 5 ml of alum for real textile wastewater samples at 0.4 ampere current intensity and 20 r.p.m rotational speeds as shown in fig.(3).At previous conditions, it was 7.25 and 7.31 in case of 10 ml and 15 ml of alum , respectively as shown in table (2). With the same initial pH, the final pH of chemical coagulation is more acidic than that of EC. This can be explained by the acidic Character of AL^{+3} in alum that reacted with OH ions of Wastewater precipitate in the form of $AL(OH)_3$ (Palahouane, el al. 2015) .
Initial conductivity and TDS were 2140 µs and 1427mg/l. Final conductivity and TDS were 2250 µs and 1500mg/l in case of no chemical added and they were 2269 µs and 1512mg/l in case of 5 ml of alum for real textile wastewater samples at 0.4 ampere current intensity and 20 r.p.m rotational speeds as shown in fig.(3). At previous conditions, they were 2270 µs and 1514mg/l in case of 10 ml of alum and they were 2277 µs and 1518 mg/l in case of 15 ml of alum, respectively as shown in table (2). This explained by adding chemical increased ions and impurities movement (song et al. 2003).

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Finally, it was noted that adding amount of alums helps agglomerates to form in short time because of formation more (AL$(OH)_3$) besides (AL$(OH)_3$) of EC and increased conductivity. For these reasons, the removal efficiency of COD and dye reached 83% and 90% after 5 minute.

Table (2) Results of conductivity and TDS for textile wastewater samples

(13 Volt-0.4 Ampere-20 r.p.m)

RUN NO	Amount of added alum (ml)	Conductivity (µs)		TDS (mg/L.)		Dye (mg/L.)	
		Initial	Final	Initial	Final	Initial	Final
1	0	2140	2250	1427	1500	80	9
2	5	2140	2269	1427	1512	80	8
3	10	2140	2270	1427	1514	80	8
4	15	2140	2277	1427	1518	80	8

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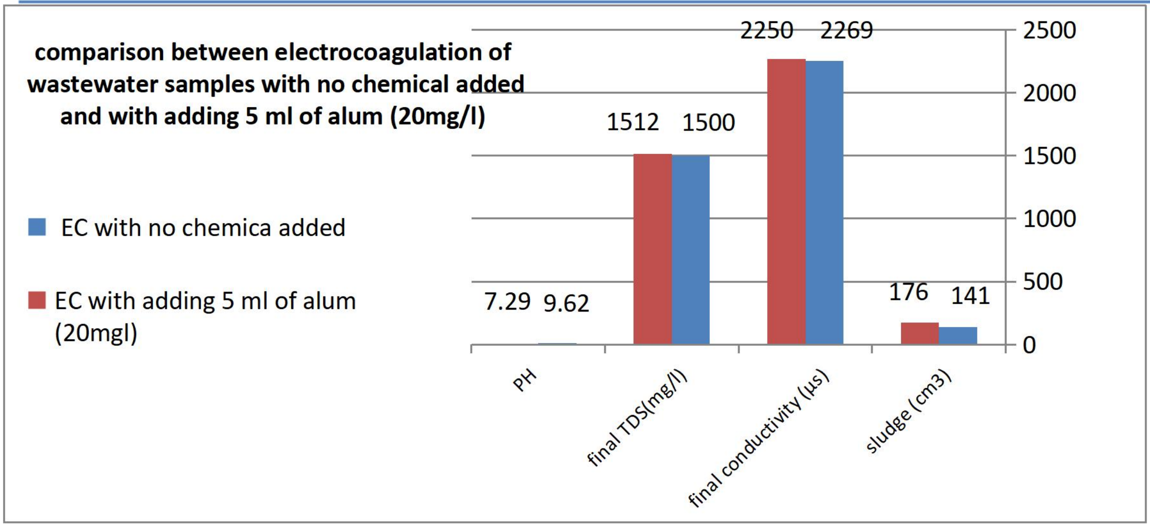

Fig (3)

4.2. Regression model development

In order to evaluate the accuracy of the developed model, the mathematical relation between the experimental results and the predicted values was done using goodness-of-fit statistics

. (Se/Sy) is the standard error divided by the standard deviation of measured values about the mean and it used to express on accuracy of relation which means asmaller value leads to higher accuracy. The R2 is known as the square of the correlation coefficient between the predicted values and the experimental results. Non-linear regression analysis was performed by Excel software program (solver function) for the model development. Many trials were implemented to develop a simple accurate prediction model for dye removal based on the measured experimental data. The optimal model was performed by taking into consideration the five parameters (reaction time (T), rotational speed (v), initial dye concentration (c), current intensity (I), and amount of alum dose (A)) to get higher accuracy. The final form of this model is shown in this equation no. (9)

$$\text{Dye removal (\%)} = 4.694849*T+2.779756*v-0.03465*C+31.7447*I+4.496219*A-0.18242*T^2+.000305*v^2+.000615*C^2-5.00791*I^2-0.09881*A^2+0.020803*T*v+.009834*T*C+0.293776*T*I-0.11399*T*A-.01753*v*C+0.2881*v*I-0.27289*v*A-0.07435*C*I+0.050623*C*A+0.999977*I*A \quad (9)$$

Where: T=reaction time (minute),v=rotational speed (r.p.m), C=Initial dye concentration (mg/l), I=current intensity (ampere) and A= Amont of alum with concentration 20 mg/l (ml).

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As shown in Fig. (4) , This model yielded a coefficient of determination (R^2=0.908) ,adjusted (R^2=0.897) and percent of (Se/Sy 0.32) . The developed model is appropriate for the prediction of the dye removal with an acceptable accuracy.

It is observed that the model has been implemented based on the experimental data. The recommended conditions to apply the model are: reaction time (0–20) minute, rotational speed (0-80) r.p.m, Initial dye concentration (80-300) mg/l,current intensity (0.4-1) ampere and Amont of alum with concentration 20 mg/l (0-15)ml.

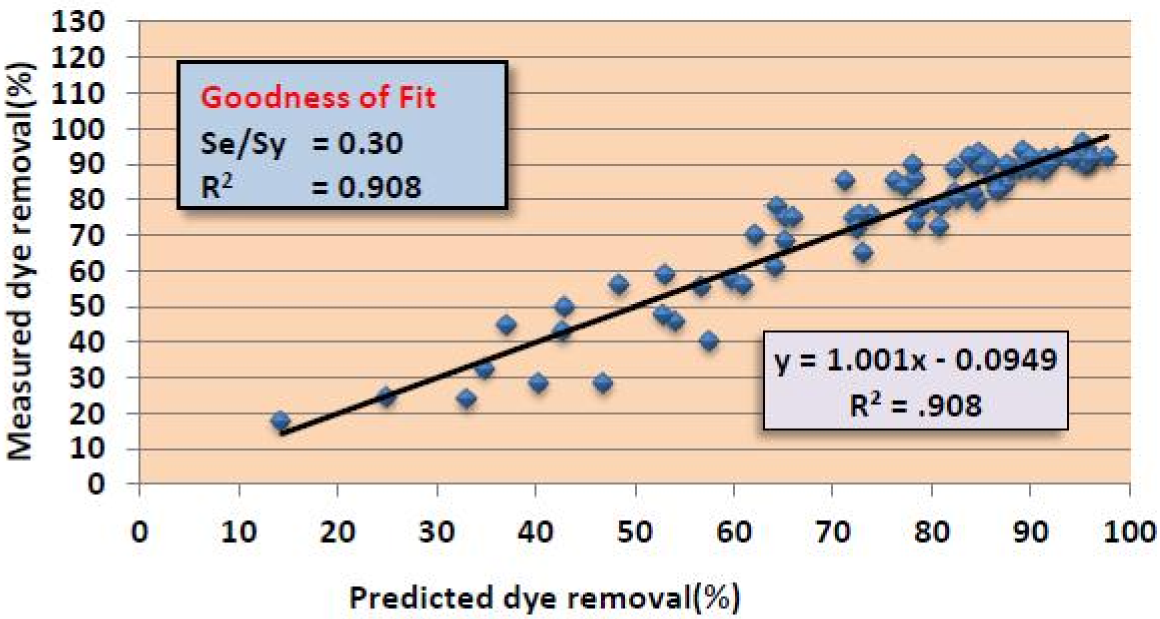

Fig (4) Regression model (Measured versus Predicated dye removal %.)

4.3. Model development by using artificial neural networks

An artificial neural network is defined as a computational tool stimulated by biological neural systems. It is a method to study the ability for modeling complicated relationships between outputs and inputs or obtain similar patterns in data. It consists of an interrelated group of artificial neurons (nodes), classified into numerous layers (one or more input, one or more hidden layer and one output layer) . These nodes are joined to each other through links [Yang et al 2015]. The construction of artificial neural network is shown in Fig. 5a.

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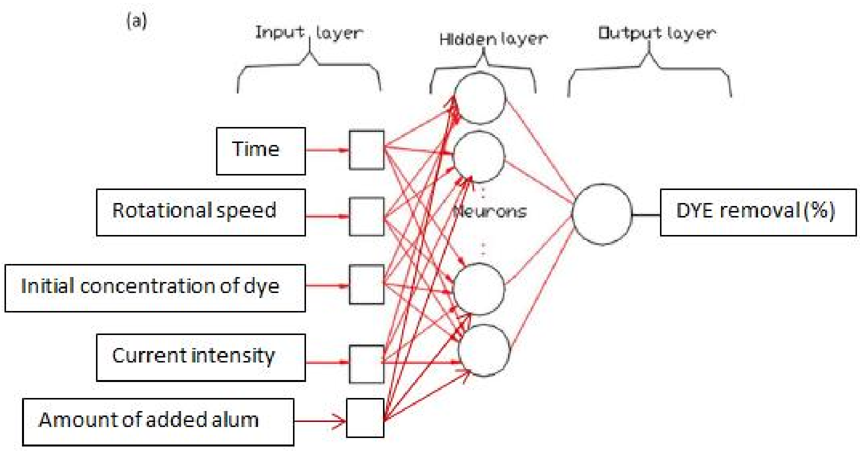
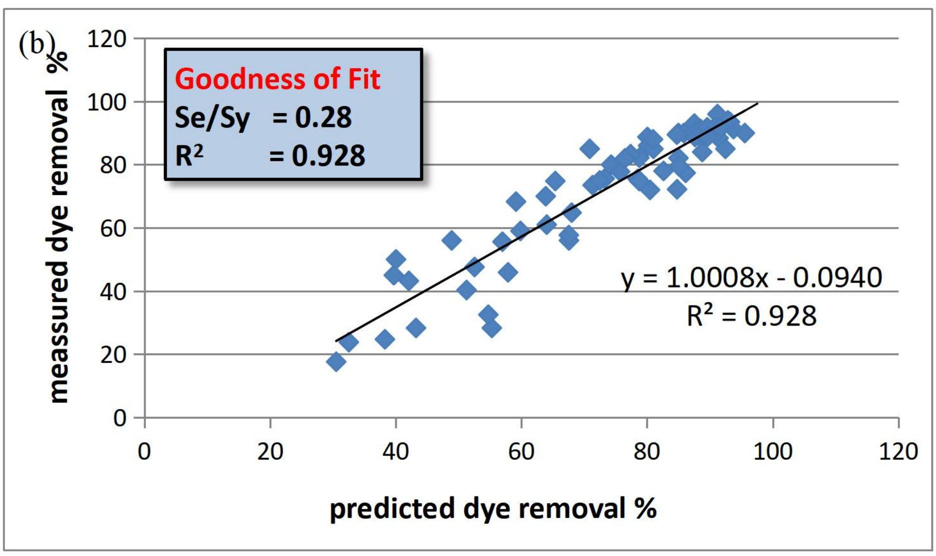
ANN model of three layers, as shown in Fig. 5a was developed for the prediction of removal efficiency of dye under various experimental conditions. The experimental conditions are reaction time, rotational speed of anode, initial concentration of dye, current intensity and amount of added alum were used as inputs to the ANN model.

72 experimental sets were selected to feed the ANN structure. The samples were divided into three sets training (37 samples), validation (12samples) and test (23 samples). Trail (5-20-1) (five inputs, one hidden layer consisting of one 20 neurons and one output), evaluated the highest determination coefficient (R2=0.928) and lowest percent of (Se/Sy=0.28). Fig. 5b showed the relationship between the predicted and measured dye removal .

The sensitivity results from the ANNs in Fig. 5 c were in accordance with results from the regression analysis that the time is the most significant parameter affects the predicated dye removal, then rotational speed , amount of added alum ,current intensity and initial concentration of dye ,respectively.





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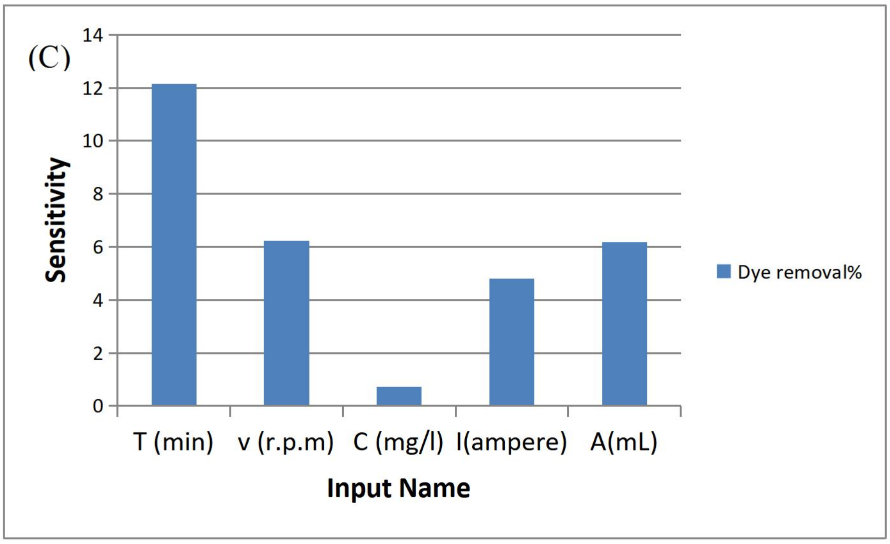

Fig. (5) Artineural network (ANN) modeling. (a) ANN structure. (b) Measured versus predicated dye removal %.(c) Sensitivity analysis for prediction of dye removal by ANN.

Conclusions

1. Current intensity is the most important factor that controls the interaction rate in the electrocoagulation cell and initial concentration of dye has no effect on COD and dye removal efficiencies.
2. Adding amount of alums helps agglomerates to form in short time because of formation more (AL $(OH)_3$) besides (AL $(OH)_3$) of EC and increased conductivity. For these reasons, the removal efficiency dye reached 90% after 5 min.
3. The coefficient of determination of Excel model is (R^2=0.908) and The ANN model yielded a coefficient of determination (R^2=0.928).

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