Journal of
Applied Research in Water and Wastewater

Journal homepage: www.arww.razi.ac.ir

Original paper

Combination of ozonation with aerobic sequencing batch reactor for soft drink wastewater treatment: experiments and neural network modeling

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ARTICLE INFO

Article history:
Received 12 October 2015
Received in revised form 14 December 2015
Accepted 27 December 2015

Keywords:
Soft drink
Wastewater
SBR
Ozone
Artificial neural network

ABSTRACT

In this study, ozone combination with a sequencing batch reactor was tested in laboratory scale for treating a soft drink wastewater characterized by high concentrations of chemical oxygen demand (COD). A bench scale aerobic sequencing batch reactor (SBR) is carried out by two stages. The system was operated under three different mixed liquid suspended solids (MLSS) concentrations (3000, 4500, 6000 mg/l). The results show that the integrated ozonation with biological process was able to achieve high removal efficiencies for chemical oxygen demand (COD), with residual concentrations much lower than the current discharge limits. Also, the process was characterized by a very low MLSS concentration. Hence, the ratio between ozone dose and the COD removal was 0.72, indicating that the removed COD was higher than the dosed ozone. Artificial neural networks (ANN) was also employed to model the COD data obtained. A network consisting of two layers of five neurons in the hidden layer was considered. Regression coefficient between experimental data and data predicted by neural networks and root mean square error (R^2, RMSE) obtained 0.991, 80.36, respectively. Very low error in the network estimation confirmed validity of the obtained networks for further analysis and optimization.

1. Introduction

Wastewater, a liquid waste that is discharged from, industries, agricultural, commercial, hospital properties potentially release significant amounts of toxic and pathogenic contaminants into local treatment plants for processing. The activated sludge process uses a mass of microorganisms to aerobically treat wastewater. The microorganisms use organic pollutions in the wastewater as a food source, converting a portion of the carbon in it into new biomass and the remainder into carbon dioxide. Biological treatment is often a cheaper and more environmental salubrious, offer for the treatment of the organic pollutants from industrial wastewater (Hai et al. 2007; Jadhav et al. 2010). Unfortunately, biological treatment alone is not profitable to eliminate hardly biodegradable such as toxic, refractory compounds (Ledakowicz et al. 2001), hence, additional special treatment step such as advanced oxidation processes (AOPs) are necessary for optimization of treating wastewater (Di Iaconi. 2012). The AOPs, are characterized by the generation of highly reactive free radicals in aqueous, such as hydroxyl radicals ($OH^{\bullet}$). With a high oxidation potential of 2.80 V, $OH^{\bullet}$ radical has a singular demolition power (Kurniawan et al. 2006):

$$OH^{\bullet} + H + +e^{-} \rightarrow H_2O \qquad E_0 = 2.80\,V \qquad (1)$$

AOPs are effective in destruction organic chemicals; because they react rapidly and have no selectively with nearly all organic compound (Stasinakis. 2008). Through the $^{\bullet}OH$ radicals, the ozone (O_3) oxidation is a powerful oxidizing method with a high oxidation–reduction potential of 2.07 V. Ozone reacts with great number of with organic and inorganic compounds directly by molecular ozone or via hydroxyl radicals generated by ozone decomposition in water (Somensi et al. 2010). But, advanced oxidation processes (AOPs) cannot meet the environmental discharge standard by itself alone (Somensi et al. 2010). Therefore, the combination of ozonation with biological treatment would be capable to remove refractory organic compounds, including color, coliform and virus (Sangave et al. 2007). Furthermore, no solid residues are produced during ozonation (Baig and Liechti. 2001; Arslan-Alaton. 2007). In pre-ozonation, ozone reacts with many biorefractory compounds, converting them into simpler and biodegradable molecules (Yasa et al. 2007; Cortez et al. 2011). Combination of ozone used as pre-treatment stage with activated-sludge aerobic process was efficacy to remove organic carbon, and nitrogen from the winery wastewater and increased ability of biological treatment. Also the use of ozone has improved settling property of the sludge (Oller et al. 2011). Cortez et al. discovered that ozone pretreatment could improve the biodegradability of recalcitrant organic compounds for subsequent biological treatment (Cheng et al. 2011; Cortez et al. 2011). On the other hand, post-ozonation also improved the quality of a secondary effluent by increasing the dissolved oxygen (Paraskeva and Graham. 2005). Variety research works have been conducted to evaluate the performance of ozonation in combination with biological treatment to remove chemical oxygen demand (COD) from wastewater. Treatment of paper mill wastewater with combination of ozone and algal treatment was examined by Balcıoğlu et al. (2007). The results showed that the BOD_5/COD ratio increased from 0.11 to 0.28 (Akmehmet Balcıoğlu et al. 2006; Balcıoğlu et al. 2007).

Treguer et al. (2010) studied effect of ozonation on natural organic matter in drinking water treatment in membrane bioreactor containing activated carbon (Treguer et al. 2010). The soft drink industry generates large volumes of wastewaters, characterized by high COD concentrations reflecting their high organic content. Typical wastewaters from soft drink industries are mainly composed of washing

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waters from production and packing of syrup, bottling production runs, purification of process water, and equipment and conduit cleaning.

The use of artificial intelligence techniques such as fuzzy logic and artificial neural networks has been recommended in environmental pollution issues due to system complexity, validity and reliability (Mjalli et al. 2007; Ongen et al. 2013; Kundu et al. 2014). Also application of artificial intelligent model (MLP) and Adaptive Neuro fuzzy inference system (ANFIS) to predict the side weir discharge coefficient has been studied by Parsaie et al. (2014).

Prediction of dissolved oxygen (DO) in water reservoirs in Serbia by Rankovi et al. (2010) indicate that using ANN with training algorithm Levenberg–Marquardt (LM) was suitable. Which good agreement between experimental and predicted data was established (Ranković et al. 2010). Similarly, the ability of the neural network model to modeling urban wastewater quality characteristic was examined by Güçlü and Dursun (2010). Their findings showed that root mean square error (RMSE) for predicting SS, COD and MLSS were 5, 17.1 and 3.8 %, respectively, which indicated the ability of ANN to model the phenomenon.

Kundu et al. (2014) carried out a research work on ANN application for modeling in biological removal of COD and total Kjeldahl nitrogen (TKN) for the treatment of slaughterhouse wastewater. Thus, this study aimed to develop and evaluate the efficiency of the neural network model to investigate the performance of the hybrid system SBR and ozonation for COD removal of wastewater produced from the soft drink factory.

2. Materials and methods
2.1. Wastewater composition

Wastewater samples, with a pink color, were collected from a soft drink production plant located in Kermanshah-Iran. A sample was reaped in plastic containers from the effluent channel and transferred to the laboratory. The characterization of wastewater is presented in Table 1.

Table 1. Characterization of soft drink wastewater used.

Parameter	Value
COD	2500 mg/l
Biodegradability (BOD_5/COD)	0.43
pH	6.5-8

2.2. Biological process

Biological unit was based on sequencing batch reactor (SBR) technology. The SBR unit consisted of a cylindrical reactor (working volume: 1L). Oxygen was supplied by infusion at a flow rate of 2 L.h^{-1} using porous stones set at the bottom of the reactor in the liquid phase. All treatment process is performed at environment temperature which is about 25 ^{0}C. Activated sludge from a soft drink wastewater treatment plant was used as inoculums and nutrients (such as nitrogen and phosphorous) to obtain a COD: N: P ratio of about 100: 7: 1 in order to give a suitable growth of microorganisms. During the first 9 days of operation, the biomass was permitted to acclimatize to the influent substrate. The operation of a sequencing batch reactor (SBR) is based on five steps: fill, react, settle, decant, and idle (Fig. 1), which all steps are accomplished in a single tank.

2.3. Ozonation process

The ozone experiments were carried out in a bubble column reactor (working volume: 1L). Ozone generator consists of a stainless steel cylindrical vessel with a mercury vapor lamp inside the running length of the tube (Fig. 2). The lamp power was 300 W. The ozone stream was fed into the wastewater through a bubble gas.

2.4. Analytical methods

The ozone was bubbled into 2 % KI solution, where the potassium iodide solution reacted with the excess ozone resulting the following equation:

$$O_3 + KI + H_2O \rightarrow I_2 + 2KOH + O_2 \quad (2)$$

The produced iodine was titrated using standard sodium thiosulfate, in the presence of starch as indicator. The quantities of unused ozone were determined, accordingly (Turhan and Turgut. 2009). Analytical procedures followed in this study for COD and MLSS determinations were those outlined in standard methods for the examination of water and wastewater (Awwa. 1998). For the MLSS measurements, 50 ml of sample was filtered and placed in oven at 103-105 ^{0}C for 1h and the biomass weight was found out by drying filter paper and its weight. The pH, DO and temperature in the reactor were measured by on-line monitors.

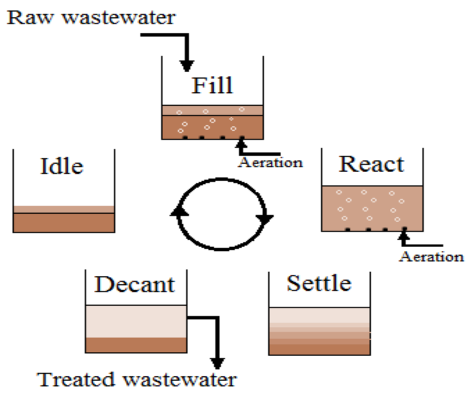

Fig. 1. Schematic of the SBR process.

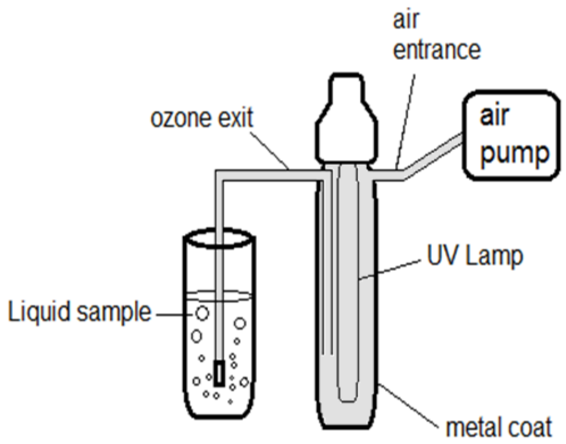

Fig. 2. Schematic representation of the ozone generator.

2.5. Artificial neural network (ANN) modeling

In this work, a feed forward neural network multilayer model was applied to predict the COD removal of refinery wastewater. The MATLAB software was employed for the ANN modelling. Fig. 3 shows the layout of the ANN architecture used in this work. In the ANN architecture, the numbers of input and output neurons were determined according to the problem definition. In this work, the input and output layers have four and one neurons, respectively. Theoretical methods for determining the appropriate number of hidden layers are not available. Moreover, the number of neurons in each hidden layer prior to training cannot be obtained theoretically. Therefore, the trial-and-error method is commonly used to design the ANN.

In the present study, the trial-and-error method was employed to attain the number of layers and neurons. For this purpose, a MATLAB script was written that creates and screens different structure several times, such as: one and two hidden layers as well as various numbers of neurons in each hidden layer. Because the initial weights may have a large effect on the convergence, runs were repeated 100 times with different randomly generated initial values. The results presented in this work are the best ones obtained from this procedure.

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An increase in the number of hidden neurons may cause over fitting that usually occurs when a model is complicated. The complicated neural network means that there are a large number of neurons (with a large number of weights and biases) relative to the number of training data. In these networks, the optimal number of neurons should be determined.

In the each network, there are many types of transfer functions. In this work, the "tansig" transfer function was used for the hidden layers and "purelin" function was considered for the output layer. The training process of the ANN was carried out using the Levenberg–Marquardt back propagation algorithm. In this training method, connection weights w_{ij} and biases b_j are iteratively adjusted to minimize the output deviation (predicted by ANN) from the target values according to Levenberg–Marquardt (Levenberg. 1944; Marquardt. 1963; Eslamloueyan et al. 2011) optimization method. The early stopping method was used to avoid memorizing the training data set. In this method, in case of a decrease in deviation of the predicted values without decrease in predicted values of the validation data set, the training will be stopped. Moreover, the error values should be calculated for different network structures with different numbers of neurons in hidden layers with one and two hidden layers. Table 2 gives the overall range of data points employed in this work for developing the ANN model.

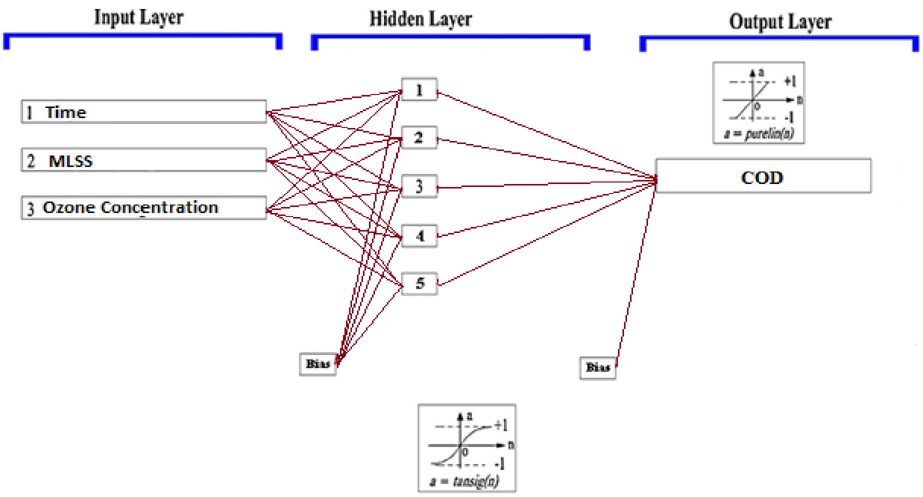

Fig. 3. The ANN architecture.

Table 2. The range of variables as employed data.

Variable	Range
Time (h)	0-24
Ozone concentration (mg/h)	0-500
MLSS (mg/L)	3000-6000

3. Results and discussion

3.1. One-stage SBR

The effect of aeration time on effluent chemical oxygen demand (CODe) during one stage SBR was examined at different MLSS concentrations. Therefore, the reactor was aeration for a period of 24 h. The system was operated at different MLSS concentrations (3000, 4500, 6000 mg/l). The COD removal was increasing with the increase in MLSS concentration. This clearly shows that the mass of substrate and bacterial can have an important influence on the COD removal. At the MLSS concentration of 6000 mg, COD removal was 94±1.4 %. When the MLSS value is equal to 3000 and 4500 mg/l, COD removal was 75±1.8 and 85.68±1.5 %, respectively. The optimum aeration time was 12 h. COD removal efficiency increased with rising aeration time up to 12 h (Fig. 4). No significant COD removal efficiency was observed when aeration was applied more than 12 h.

3.2. Two-stage SBR

Basically, the two-stage process is a combination of two independent SBR plants which work in series. The two-stage SBR was operated with selected HRT of 24 h. The effluent from the first-stage SBR after 12h is feeding the second SBR (Fig. 5). MLSS concentrations in two consecutive reactors are equal and changed to 3000, 4500 and 6000 mg/l.

Percentage of COD removal at this stage for the amounts of MLSS, 3000, 4500, 6000 mg/l, were 85.64±1.1, 94.28±2.12 and 96.67±0.79, respectively, which were higher than the single stage process used. Over time, aerobic biological treatment increased the reproduction of bacteria and the bacteria consumed organic matter in the wastewater. By providing sufficient oxygen to the wastewater, Microbial growth is somewhat accelerated by the time when the organic material in the wastewater is not enough. So, in the next stage, COD removal efficiency is severely less. The two-stage SBR process is a very good approach to resolve this problem. Due to the high ability of young microorganisms in consumption of the organic material; COD removal rate will be higher. The purpose of this two-stage process is to achieve the desired removal efficiency of COD with MLSS values less than that of a single-step process.

The soft drink wastewater treatment was studied by Kalyuzhnyi et al. (1997). The COD of wastewater was between 1.1 to 30.7 g/L. The soft drink wastewater was treated by using a UASB reactor and a hybrid reactor, an anaerobic filter combined with a sludge bed reactor. Results showed that the efficiency of removal COD for hybrid reactor and UASB reactor was 80 and 73 %, respectively (Kalyuzhnyi et al. 1997).

3.3. Two-stage SBR integrated with ozonation

Fig. 6 shows the sketch and operation of the plant based on SBR system integrated with ozonation used for treating the soft drink wastewater. The examination was accomplished at different MLSS concentrations in each process. The operation was based on the succession of 24.5 h treatment cycles each consisting of three consecutive stage: a preliminary biological degradation (for 12 h), ozonation degradation (for 0.5 h) and finally the ozonated wastewater came back to the SBR reactor for the final biological treatment. The effluent from the one-stage process is used as feed of the next stage. The samples are taken from the effluent of every stage in order to module the residual COD concentration.

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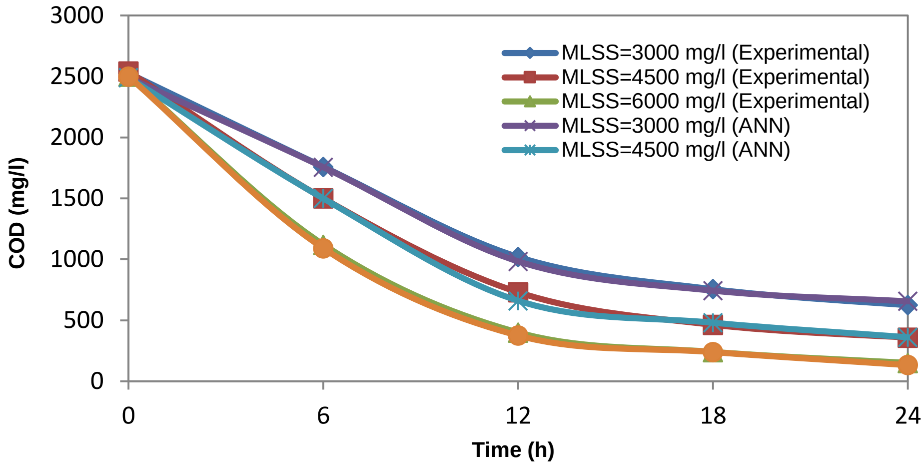

Fig. 4. Profile of effluent chemical oxygen demand (COD_e) during one-stage SBR.

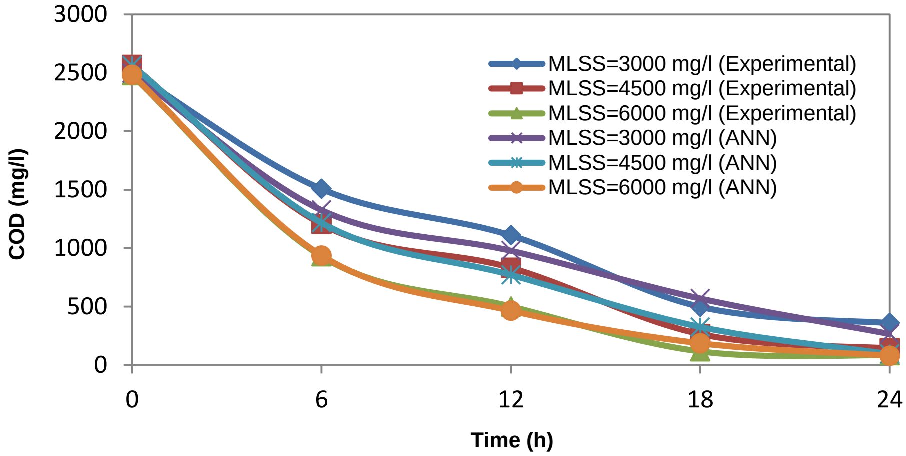

Fig. 5. Profile of effluent chemical oxygen demand (CODe) during two-stage SBR.

COD concentration in the effluent was about 125±33, 65±55, 34±15 mg/l for MLSS concentrations of 3000, 4500 and 6000 mg/l, respectively. Hence, the ratio between ozone dose and the COD removal was 0.72. This ratio showed that the removed COD was higher than the dosed ozone. Combination of ozonation and biological treatment is considered as a new integrated treatment system. The results shows chemical oxidation can change the molecular structure of the slowly biodegradable compounds and breakdown them into smaller molecules (Giannis et al. 2007). These results indicated that the multi-stage ozonation-biological treatment gives higher COD removal efficiency than the conventional single-stage biological treatment. Therefore, the treatment time and cost are substantially minimized. The COD concentration in effluent and removal efficiencies of the SBR and integrated treatments of soft drink wastewater reported in Table 3. The data reported in Table 2 show that biological treatment by one-stage SBR was able to reduce concentrations in the effluent as high as 625, 358, 150 mg/L for MLSS 3000, 4500, 6000 mg/l, respectively. The data obtained for the two-stage SBR (Table 3) show high removal efficiencies for COD, with residual concentrations about the discharge limits.

Table 3. Experimental values of final COD and % COD removal for SBR and combined treatment processes.

MLSS(mg/l)	Treatment	COD effluent (mg/L)	% COD removal
3000	One stage SBR	625±46	75.00±1.8
	Two stage SBR	359± 29	85.64 ± 1.1
	O_3+ biodegration	125 ± 33	95.00 ± 1.3
4500	One stage SBR	358±38	85.68 ± 1.5
	Two stage SBR	143±53	94.28 ± 2.12
	O_3+ biodegration	65 ± 55	97.4± 2.2
6000	One stage SBR	150± 35	94± 1.4
	Two stage SBR	84± 19	96.67± 0.79
	O_3+ biodegration	34± 15	98.64 ± 0.6

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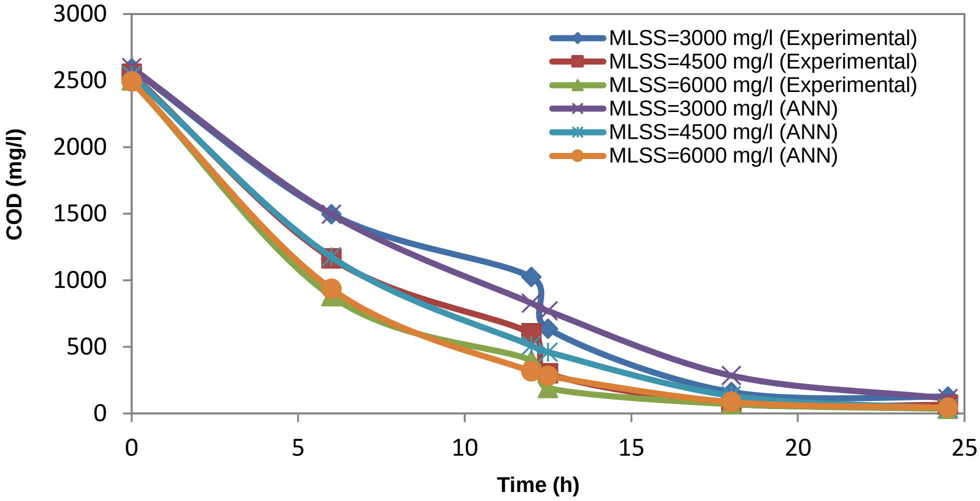

Fig. 6. Profile of effluent chemical oxygen demand (CODe) during two-stage SBR/O_3.

In the latter study, olive mill wastewater (OMW) treatment is perused experimentally in several combined processes of photo degradation by UV radiation, advanced oxidation with ozone (O_3), and an aerobic biodegradation by stirred tank reactor. For both single stage treatment of O_3 and two-stage treatment of O_3/UV, COD remains quite high. But, an integrated system of biological and UV/O_3 process for the olive mill wastewater treatment seems to be an efficient alternative in the reduction of the COD. In particular, biodegradation of UV/O_3 pretreated OMW found to have the highest removal levels; the percent of COD removal reached about 91 % (Lafi et al. 2009; Shannak et al. 2009).

3.4. Modeling results with ANN

In the present study, the ANN was developed to COD removal of wastewater emanated from the soft drink factory using some variables as ANN input data. These data are accessible by experimental studies. In this research, the input variables were selected according to the experimental studies.

In this work, the application of ANN models with one hidden layer was investigated. The ANN with different architectures lead to different outcomes in each run, so there is more possibility to reach the best answer with more runs. Fig. 7 shows the lowest obtained RMSE after several runs in terms of increase in the number of neurons in one hidden layer for COD concentration. Here, the ANN with a structure of 3-5-1 (five neurons in hidden layer) was chosen as optimum topologies.

Table 4 depicts the ANN network performances for COD using different number of neurons in the hidden layer and the Levenberg-Marquardt (LM) algorithm. The optimum number of neurons in the hidden layer was obtained 5 neurons.

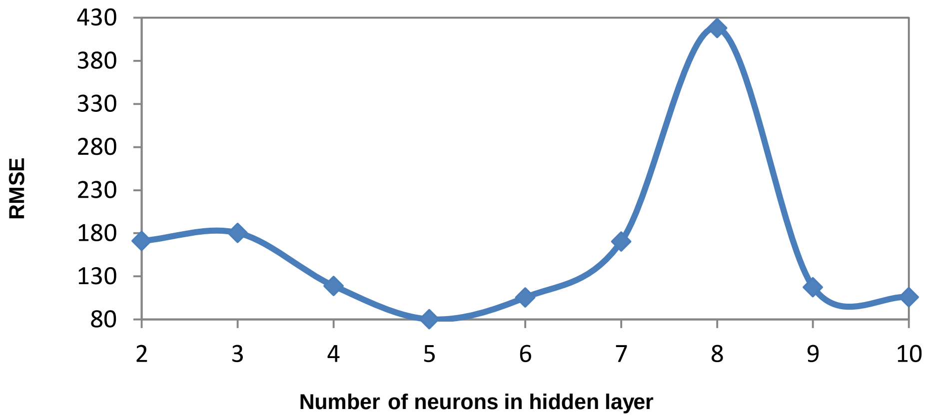

Fig. 7. Effect of number of neurons in hidden layer on ANN performance.

Table 4. Effect of number of neurons in the hidden layer on the performance of neural networks for COD removal.

Number of neurons in the hidden layer	RSME	R^2	Eq.
2	171.20	0.960	Y=0.939X+74.08
3	180.50	0.958	Y=0.974X+27.81
4	119.10	0.981	Y=0.968X+12.90
5	80.36	0.991	Y=0.982X+74.08
6	105.70	0.985	Y=0.974X+22.88
7	170.60	0.960	Y=0.944X+83.22
8	418.00	0.816	Y= X+10.23
9	117.70	0.983	Y=0.991X-2.50
10	106.00	0.986	Y=0.990X+5.07

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In addition to the Levenberg–Marquardt algorithm (trainlm), other algorithms such as traincgb (conjugate gradient back propagation with Powell–Beale restarts), trainb (batch training with weight and bias learning rules), trainbr (bayesian regularization back-propagation), traingdm (gradient descent with momentum back-propagation), and traingda (gradient descent with adaptive learning rate back-propagation), were used to train the network. Table 5 shows the mean square error (RMSE) and correlation coefficient (R^2) obtained for each algorithm.

The reported errors in the table illustrate that using the Levenberg–Marquardt algorithm leads to the lowest error. Fig. 8 indicates a comparison between the predicted results from the ANN and the experimental values.

Table 5. Best RMSE and R^2 values of different training algorithm for ANN with 3-5-1 configuration.

Neuron No.	algorithm	Transfer fun.		RMSE	R^2
		Hidden lay.	Output lay.		
1	BFG	Tansig	Purelin	158.60	0.967
2	CGB	Tansig	Purelin	364.60	0.838
3	CGF	Tansig	Purelin	208.60	0.945
4	CGP	Tansig	Purelin	321.90	0.853
5	GDA	Tansig	Purelin	186.40	0.950
6	LM	Tansig	Purelin	80.36	0.991
7	OSS	Tansig	Purelin	155.80	0.967
8	RP	Tansig	Purelin	230.20	0.919
9	SCG	Tansig	Purelin	200.90	0.944

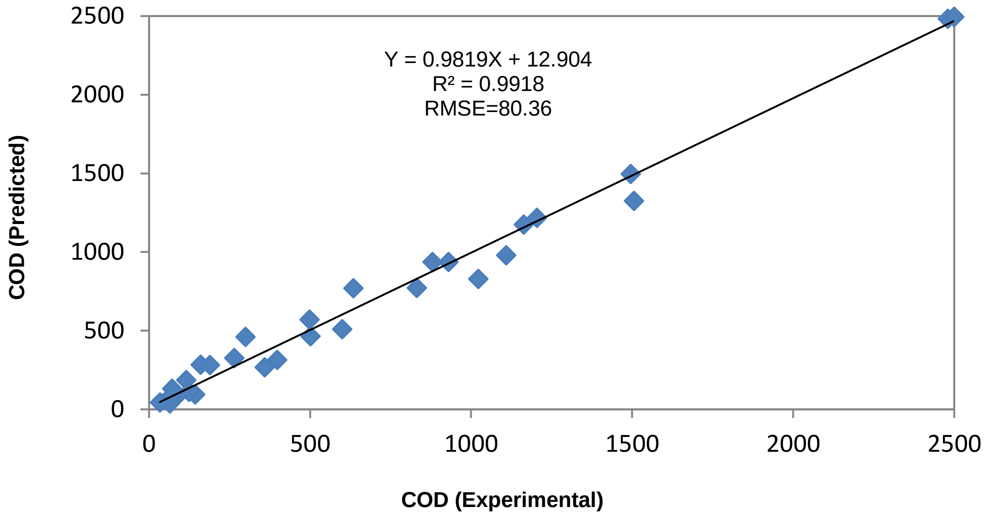

Fig. 8. Cross-correlation between measurement values of COD and predicted values of COD by ANN.

This figure shows that the ANN predicted values for all data points are quite close to the experimental data values. The developed two-layered ANN (3-5-1) in this work provides the weights, which are listed in Table 6. Using the parameters (W, b) presented in Table 6, the COD can be calculated from the following equation:

$$D = F_p \left\{ \sum_{j=1}^{7} W_{kj} \left[F_t \left(\sum_{i=1}^{4} W_{ji} X_i + b_j \right) \right] + b_k \right\} \quad (1)$$

where, X is the input value of the network, W is the weight, b is the bias, 'i', 'j' and 'k' refer to the input, hidden, and output layer, respectively. F is the transfer function that is used to get the normalized output values from the neurons. In this study, the "tansig" transfer function was considered for hidden layers and the "purelin" transfer function was used for the output layer.

The presented ANN in this work introduces a technique to avoid curve fitting of a large number of polynomials, any of which can be useful just for one system. Here, the developed ANN works similar to a box that contains many polynomial equations. One can enter input and find the COD value with a high precision.

Table 6. Connection weights and biases.

Neuron No.	W_1			b_1	b_2= 586.12
	Time	MLSS	O_3 Concentration		W_2
1	-1.4501	3.3237	0.5166	1.4686	0.0177
2	-2.0461	-0.3550	1.1135	-1.4180	0.4253
3	2.4355	6.2937	1.7096	1.0733	-0.0399
4	1.3671	0.3792	0.7346	0.7078	- 0.4830
5	4.4350	-0.2905	0.0715	4.3544	-0.6541

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4. Conclusion

This work represents treatment of soft drink industrial wastewater by a two-stage system, combination of SBR and ozonation. The effect of several parameters on the efficiency of COD removal e.g. time, MLSS, and O_3 concentration were examined experimentally, and the following results are obtained:
1. In one-stage SBR, notable COD removal efficiency was observed when aeration was applied more than 12 h.
2. In two-stage SBR, COD removal efficiency could be improved in comparison to the one-stage treatment.
3. The role of ozonation was to breakdown the big molecule to small molecules that is easily biodegraded in biological treatment.

The presented ANN in this work introduces a technique to predict COD of the wastewater with a high precision by introducing input data component instead of sophisticated polynomial fitting.

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Please cite this article as: N. Amiri, M. Ahmadi, M. Pirsaheb, Y. Vasseghian, P. Amiri, Combination of ozonation with aerobic sequencing batch reactor oxidation for soft drink wastewaters treatment: experiments and neural network modeling, Journal of Applied Research in Water and Wastewater, 2(2), 2015, 156-163.

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