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Prediction and optimization of heavy metal ions removal efficiency from the active sludge using intelligent systems

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ABSTRACT

In the current study, the capability of artificial neural network (ANN) and adaptive neuro-fuzzy inference system (ANFIS) techniques were investigated in prediction of heavy metal ions (lead and nickel) removal efficiency from the active sludge of an industrial zone's wastewater treatment plant. The effect of experimental parameters such as solution pH, contact time, initial ions concentration, and temperature were studied to find the optimal values of these parameters. During the modeling multilayer perceptron network and a Sugeno Fuzzy model were applied in the proposed ANN and ANFIS models, respectively. The results of comparison between the experimental and the predicted data were satisfactory and the achieved correlation coefficients (>98%) approved the high accuracy of these two models, although ANFIS performed slightly better than ANN. Moreover, the optimal operating conditions were achieved by minimizing the objective function using genetic algorithm. Additionally, a real sludge sample was then treated under the achieved optimal conditions which yielded acceptable adsorption efficiency values. Based on these observations, the proposed intelligent models were acceptable tools for the prediction of these pollutants' adsorption efficiencies.

Keywords: Modeling; Artificial neural networks; Adaptive neuro-fuzzy inference system; Nickel; Lead; Absorption; Optimization; Sludge

1. Introduction

Discharge of heavy metal pollutants into the surface waters and rivers has become a critical environmental issue in recent years. These chemicals can threat the human health by entering our food chain because of their toxic and non-biodegradable nature. Metallic ions are inorganic stable species with low level of biodegradability; as a result, they are more liable to accumulate in organisms. Additionally, they have drastic impacts on our health even at low traces [1]. United States Environmental Protection Agency (US EPA) has categorized plumbum (Pb), arsenic (As), nickel (Ni), chromium (Cr), copper (Cu), zinc (Zn), cadmium (Cd), and mercury (Hg) as the most serious water pollutants. The allowed limits of these metals in industrial wastewater which is suggested by EPA are 0.1, 0.01, 0.2, 0.1, 0.25, 1, 0.01, and 0.05 (mg/L), respectively [1,2]. Although the most important source of these metallic ions are industrial wastewaters, fertilizers and sludge are considered as the other important sources of such pollutants. The sludge which is produced in a wastewater treatment plant contains various types of heavy metals which pollute soil and

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water resources. As a consequence, having a comprehensive knowledge about wastewaters contain such heavy metal ions can reduce their drastic impacts on our health, soil, and water resources [2,3].

General chemical and physical treatment techniques such as membrane separation, filtration, ion exchange, coagulation, reverse osmosis, and adsorption have been assessed for the removal of heavy metals from water and wastewater sources. An important issue in selection of the appropriate method of treatment is the cost and efficiency of the process from the engineering aspects. Due to several important advantages such as low cost of operation, reusability of adsorbents, compatibility with environment, and ease of operation, adsorption is preferred over other techniques [4,5]. Various adsorbents including clay, zeolite, activated carbon, carbon nanotubes, nano-composites, graphene, chemical composites, and bio-adsorbents have been applied for the separation of heavy metals from polluted aqueous solutions [6]. Generally, the success of these techniques depends on composition, structure, and nature of the applied adsorbent. Moreover, the adsorption process efficiency is affected by different parameters such as adsorbent surface area, solution pH, contact time, temperature, presence of salts, surfactants and other species [7,8].

Modeling is an accepted and liable engineering approach which can help understanding the metallic ion removal processes. However, modeling of such processes through common mathematical models is too costly and time-consuming due to the high number of required experiments. Additionally, wastewater treatment processes are complex since they are affected by various operating parameters and removal processes [9,10]. Consequently, modeling and optimization of adsorption process using general mathematical equations seems difficult. Some empirical models such as machine learning, fuzzy network, response surface methodology (RSM), artificial neural network (ANN), and adaptive neuro-fuzzy inference system (ANFIS) have been used for modeling of such processes recently [11–13]. These predictive models usually present the results with excellent correlation coefficients and can be applied for nonlinear correlations with a wide range of input variables. The capability of these modeling tools has been previously approved in economics, robotic, material science, chemistry and chemical industry, environment, novel energy sources, and petroleum industry. Considering the reported data in the literature, ANN is one of the most excellent modeling tools for prediction of process results [14–16]. Additionally, ANFIS is a powerful modeling method because of its main fuzzy inference part. The most outstanding feature of ANFIS is its learning and data prediction ability, which makes it an efficient tool for the modeling of vague systems.

This work is set up to predict Pb and Ni ions removal from a sludge sample using ANN and ANFIS models and comparing their results to evaluate their output accuracy. Furthermore, the outputs of the models were used to find the optimal operating conditions by genetic algorithm (GA) through minimization of an objective function. Finally, the adsorption of metallic ions from a real sample of leaching solution of an industrial active sludge in the obtained optimal values was conducted.

2. Theories

2.1. Artificial neural network

Artificial neural networks, which are inspired by biological systems, are a series of computer algorithms. The main component of these algorithms is called neuron which is applied for information processing task. In fact, neurons are neurocomputer with parallel distributed processors [17]. These neurons are connected to each other by a set of connections with certain weights. The performance of a network is highly dependent on these weight values. The neurons are divided into input, output, and hidden layers. An artificial neural network performs the modeling process by receiving inputs, adding them to their weights, addition of a bias to the results, and then sending the final result to the transfer function. The final output of the model strongly depends on the inputs and the transfer function [17,18].

One of the most common types of artificial neural networks is multilayer perceptron (MLP) [17]. MLP network includes an input, an output, and usually a hidden layer where the number of inputs and outputs of the network variables depends on the type of the process [17]. Since using a single hidden layer usually yields satisfactory results, the number of hidden layers in this research is considered one.

Network training is carried out by assigning a pattern which is called input pattern, following which the results of calculations which reach the level of activation (threshold) are sent to the output layer. As it can be seen in Fig. 1, before obtaining the network output in the output layer, the calculation units in the hidden layer collect the inputs from the input layer and use a function to calculate the output. The hidden layer errors are determined and their weights and bias are manipulated to minimize the error by comparing the network outputs with the target values. Collectively, it can be argued that the overall error reduction due to weight and bias adjustment is done using the training algorithm [19,20].

Neuron k can be expressed using Eqs. (1) and (2):

$$u_k = \sum_{j=1}^{m} w_{kj} x_j \quad (1)$$

$$y_k = \varphi\left(u_k + b_k\right) \quad (2)$$

where x_j is the input signal, w_{kj} is the neuron's weight, u_k is the outline linear compiler due to input signal, b_k is the related bias, φ is the activation function, and y_k is the neuron output signal.

2.2. Adaptive neuro-fuzzy inference system

The main purpose of a computer software which works on the basis of artificial intelligence is to achieve a set of input–output relationships which define specific processes. The term specific refers to processes whose mathematical modeling have difficulties such as nonlinearity, adaptive learning, and real-time behaviour. In ANFIS model, which is in fact a fuzzy inference in the form of adaptive networks, the learning ability of neural network is combined with

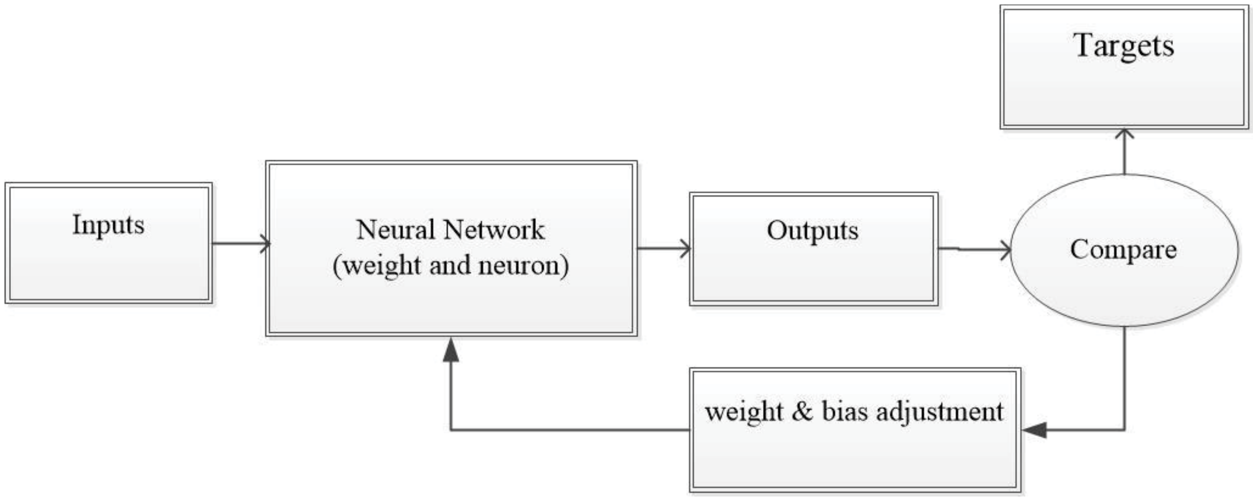


Fig. 1. Training of an ANN model.

fuzzy modelling [21]. It can be claimed that ANFIS is a combination of low-level ANN calculations with the high reasoning ability of a fuzzy logic system [22].

In ANFIS-based nonlinear system modeling, the input space is divided into a number of local areas. A simple area for each input is developed based on linear functions or adjustable coefficients, after which ANFIS uses membership functions (MFs) to divide the dimensions of each input. While the input space is covered by shared MFs, several local areas can be activated simultaneously. ANFIS layers and MFs play an important role in the ability of ANFIS model to estimate the output of the processes [17].

ANFIS consists of two sections: introduction and conclusion. It should be noted that fuzzy rules are applied to these two sections. For a first-order Sugeno Fuzzy model, a common fuzzy if-then rule is in the form of the following equation:

Rule 1:

- If x_1 is A_1 and x_2 is B_1 and…; then $f_1 = p_1x_1 + q_1x_2 + \ldots + r_1$
  Rule 2:
- If x_1 is A_2 and x_2 is B_2 and…; then $f_2 = p_2x_1 + q_2x_2 + \ldots + r_2$

where A_i and B_i are fuzzy sets and f_i is the system output. p_i, q_i, and r_i are design variables which are determined during training [23].

As it is illustrated in Fig. 2, each ANFIS model has a five-layer structure, which is designed for prediction of the target variable. The first layer's nodes are compatible with the following equation:

$$\mu_{\mathrm{Ai}}(x) = e^{-\left(\frac{x-x^*}{\sigma^2}\right)} \quad (3)$$

where x^* and σ are the assumed parameters adapted by a combined algorithm, and x is the input variable.

In the second layer, the firing power of each rule is determined by quantifying the input data of each rule. The output of a layer is the result of algebraic input signals:

$$O_{2,i}(x) = \omega_i = \mu_{\mathrm{Ai}}(x_1) \times \ldots \times \mu_{\mathrm{Ci}}(x_n) \quad (4)$$

Normalization is performed in the third layer, which is done by the calculation of the ratio of the ith firing power of the rule (propagation) to the sum of all the results of the firing power. It is calculated in each node by the following equation:

$$O_{3,i}(x) = \bar{\omega}_l = \frac{\omega_i}{(\omega_i + \cdots + \omega_n)} \quad (5)$$

The output of each node in the fourth layer is calculated as follows:

$$O_{4,i} = \sum \bar{\omega}_i f_i \quad (6)$$

The total output is obtained by summation of all the input signals in the fifth layer. The calculation of the oscillation height in the fifth layer is done using equation 7 [18,24,25]:

$$O_{5,i} = \frac{\sum_{i=1}^{n} \omega_i f_i}{\sum_{i=1}^{n} \omega_i} \quad (7)$$

It is clear that the first and fourth layers are adaptive and the fifth generates the total output in the form of the sum of all input signals.

3. Methods

3.1. Preparation of absorbent

Zinc chloride (Merck Co., Germany) was used for the preparation of adsorbents and Pb and Ni stock solutions were synthesized using $Pb(NO_3)_2$ and $Ni(NO_3)_2 \cdot 6H_2O$, respectively. In addition, HCl and NaOH were used for pH

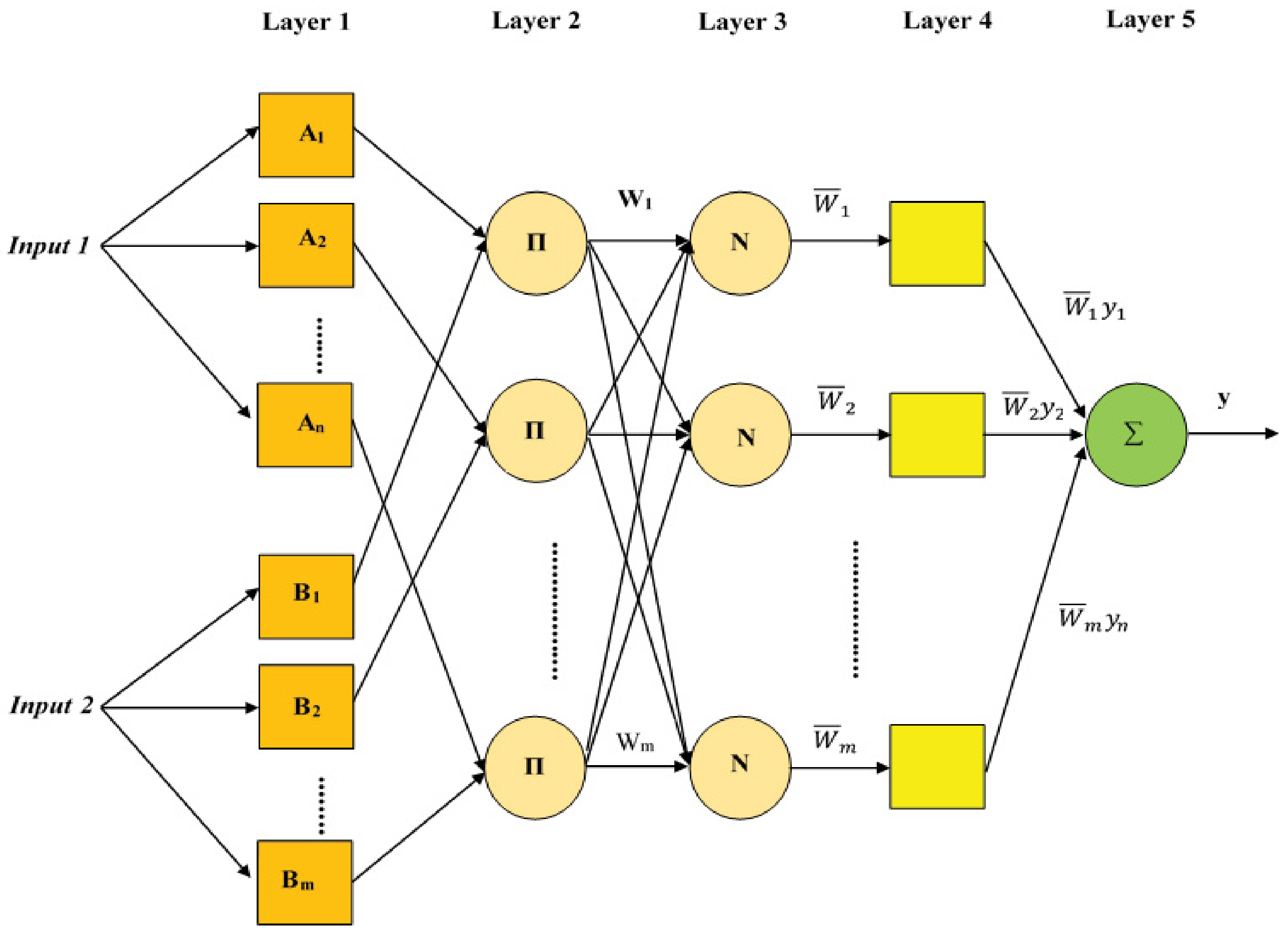


Fig. 2. Simple ANFIS architecture.

adjustment. It is worthy to note that all the chemicals were purchased from Merck Co., (Germany) and used without further purification [26].

Other apparatus which was used during the experiments were oven (EYELA-NDO-400) for drying the samples, JENWAY 2030 pH meter, vacuum pump (EYELA-ASPIRATOR A-35), shaker (IKA-KS 4000i), furnace (LENTON THERMAL DESIGNS), and mechanical stirrer (IKARW 20) [26].

Wet sludge which was collected from the filter press unit of Kaveh Industrial Wastewater Treatment Plant (Saveh-Iran) and used for the synthesis of activated carbon. The sludge samples were initially dried at 103°C for 24–36 h and then were grinded to 0.1–0.2 mm particles and screened. 5 g of the screened sample was mixed with 15 mL of $ZnCl_2$ 5 M for 24 h at room temperature using stirrer while the vessel was covered using aluminum sheets to prevent water vaporization. The achieved mixture was then placed in an oven at 103°C for 24 h. The pyrolysis of the activated samples was done in a horizontal furnace under nitrogen atmosphere at 600°C for 2 h. The nitrogen flowrate and pressure were 400 cm^3/min and 2 atm, respectively. After carbonization, the samples were cooled under pure nitrogen atmosphere and then were washed by HCl 3 M for 2 h to remove the inorganic particles and the activator agent from the pores created as the result of pyrolysis process. Afterwards, the samples were filtered and finally the synthesized adsorbents were neutralized by distilled water and dried in oven (103°C) [26].

Adsorption experiments were conducted in batch mode using a shaker. Ni and Pb solutions with the desired concentrations were achieved by dilution of the stock solution. After each test, the solution and used adsorbent were separated via filtration and the samples were prepared for further analysis [26].

In order to find the optimal values of operating conditions, pH should be initially optimized. During pH optimization tests, 0.2 g of the adsorbent was added to 100 mL of solutions with concentration of 50 ppm. The contact time for each test was 20 min and the solution pH was changed between 2–6 for lead and 3–8 for nickel, while temperature was fixed at 25°C.The next parameter which was optimized was the adsorbent dosage. To attain this goal, adsorbent dosage was varied between 0.1 and 0.5 g and pH was fixed at the achieved optimal value and the solution concentration and contact time were 50 ppm and 20 min, respectively. In order to optimize the contact time in the range of 5–200 min, the adsorbent 0.2 g of adsorbent was added to a 50 ppm solution and pH was adjusted at the optimal value. The effect of initial ion concentration on the adsorption efficiency was studied at the optimal values of pH, adsorbent dosage, and contact time while the concentration was varied between 5–300 ppm [26].

3.2. Modeling

In the current study, the adsorption efficiency of Pb and Ni ions from the active sludge was predicted using ANN and ANFIS models using MATLAB R2015b version. To attain this purpose 41 and 43 experimental datasets were used for the adsorption of Ni(II) and Pb(II), respectively. Additionally, 75% of the data was allocated for network training and the remained 25% were used for the final test. It should be noted that this classification was performed similarly for both ANN and ANFIS methods and the datasets were selected randomly.

For training of the applied neural network, the appropriate learning algorithm was selected among various algorithms including scaled conjugate gradient (SCG), variable learning rate gradient descent (GDX), gradient descent with adaptive learning rate backpropagation (GDA), gradient descent with momentum (GDM), gradient descent (GD), and Levenberg-Marquardt (LM). Based on the achieved data, among the tested algorithms LM performed better than the others. The criterion for this selection was a lower MSE and a regression curve (plot) with a higher R^2 value.

Since the sigmoid transfer function showed to have error values lower than linear transfer function, it was selected for the hidden layer while the purelin linear transfer function was applied for the output layer. The current training algorithm provided the lowest error value; as a result, the optimal number of hidden layer's neurons was obtained.

In this study, a first-order Sugeno Fuzzy ANFIS model with a subtractive clustering structure was used to predict the adsorption efficiency of lead and nickel ions from active sludge. Moreover, a hybrid algorithm which combines backpropagation gradient descent algorithm and least squares method was implemented for ANFIS training task, which produced lower error values in comparison with backpropagation method.

3.3. Adsorption experiments in the optimal conditions

Biological wastewater treatment produces high amount of excess sludge. Finding an appropriate method for the final sludge removal and reduction of its volume is an important issue of such processes. The conventional solution is using it as agriculture fertilizers. The principal drawback of using such fertilizers is their high content of heavy metals which is generally higher than the standard limits. There has been an increasing interest in chemical extraction method compared to other methods for removal of heavy metal ions from the produced sludge because of its easy operation, high efficiency, and rapidness. Previous studies have demonstrated that low pH, high temperature, and long contact time can promote the extraction of heavy metal ions. Inorganic acids such as sulfuric acid, nitric acid, hydrochloric acid, oxalic acid, citric acid and EDTA are considered as appropriate extractors [27]. By addition of these acids to the sludge, the metallic ions dissolve in the solution. Such a process is a proton exchange reaction which is shown as follows:

$$\text{Sludge} - \text{M} + \text{acid} \rightarrow \text{Sludge} + \text{M}^{2+} + \text{acid} \tag{8}$$

In this work, heavy metal ions were removed from a real sample of leaching solution of the industrial active sludge sample under the obtained optimal conditions by acid wash process. In order to do this, nitric acid was applied to adjust the solution pH. During acid extraction process, the dried sludge samples were grinded to 1 mm particles. Afterwards, five 100 mL samples of nitric acid in different concentrations (0.2, 0.04, 0.6, 0.8, and 1 M) and pHs (0.5, 1, 1.5, 2, and 2.5) were prepared. Then, 1g of the totally dried sewage sample was added to the acid container. It should be noted that acid wash process was carried out at room temperature and the stirring rate and contact time were adjusted at 150 rpm and 8 h, respectively. pH of solutions was measured each 2 h in order to control the unusual tolerances. It is worthy to note that the observed fluctuations were about 0.1 which could not have any significant impacts on the process. Finally, the samples were filtered and the concentrations of Pb and Ni ions were recorded.

4. Modeling results

4.1. ANN results

In the current research, the operating parameters were solution pH, contact time, initial ion concentration, adsorbent dosage, and process temperature; consequently, the number of neurons in the input layer is equal to five. The number of neurons in the output layer is one since the individual output parameter is the removal percentage for each ion.

Due to the fact that there is no comprehensive and accurate method for determining the optimal number of neurons in the hidden layer, the appropriate number is usually achieved by trial-and-error procedure. In this study, in order to determine the optimal number of neurons in the hidden layer, training of several networks with different numbers of neurons in their hidden layer was examined. After comparing the results of the predictions with the target data in terms of mean error squared (MSE), the optimal number of 24 neurons for the hidden layer was achieved.

As shown in Fig. 3, the ANN structure consists of three layers, namely input, hidden, and output layers. In the input layer, five neurons corresponding to the five input parameters are observed. The input values are directly sent from the first layer to the hidden layer, in which the main processing of the data is performed by summation of the weighted inputs. The initial values assigned to the weights are modified during the training process by comparing the experimental data and the output of the model. It should be noted that the error values are minimized using back propagation of the results.

The technical specifications of the proposed ANN model are presented in Table 1. These values are optimal and have been achieved through a trial-and-error procedure.

To assess the capability of the ANN model, the datasets were tested which were not used during the training process. Fig. 4 shows a comparison between the predicted

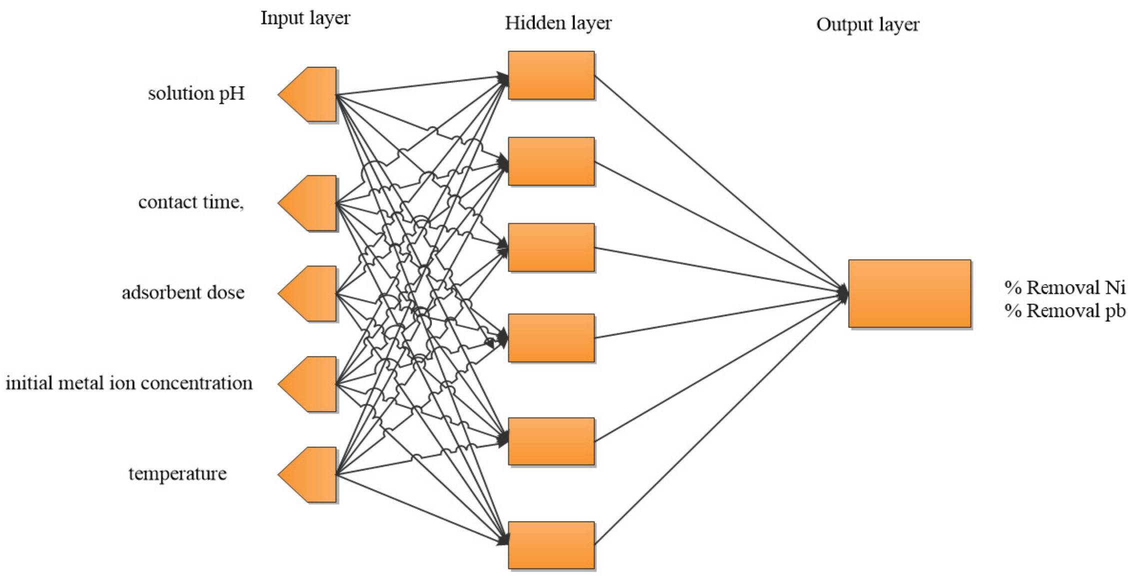


Fig. 3. ANN model structure for prediction of lead and nickel removal from the active sludge.

Table 1
Specifications of the neural network used for the model

Characteristic	Value
Number of input nodes	5
Optimum number of neurons in first layer	5
Number of output nodes	2
Learning rule	Trainlm (LM)
Number of epochs	50
Error goal	0
Mu	10e-5

values of heavy metal ions uptake and the real values for all data (including training and testing). As can be seen, R^2 values were higher than 0.98; these values indicate that the results obtained by the proposed ANN model were in a satisfactory agreement with the experimental data.

4.2. ANFIS results

The technical information of the proposed ANFIS model is reported in Table 2. It should be mentioned that during the network training, back propagation of errors method with the rate of 0.02 and replication of 1,000 was used.

Based on the achieved data, the current ANFIS model explained the correlation between the experimental and the predicted data successfully, and there was an acceptable agreement between ANFIS output and the experimental results; the correlation coefficient (R^2) reported in Fig. 5 ($R^2 > 0.99$) confirms such an excellent accuracy.

Table 3 presents the statistical parameters including R^2, SD, SSE, and Root Mean Square Error (RMSE) corresponded to the data predicted by ANN and ANFIS models.

It should be noted that all the train and test datasets were predicted by ANN and ANFIS models. Based on the data reported in Table 3, it is obvious that ANFIS simulation results were more accurate than ANN outputs.

5. Optimization using GA

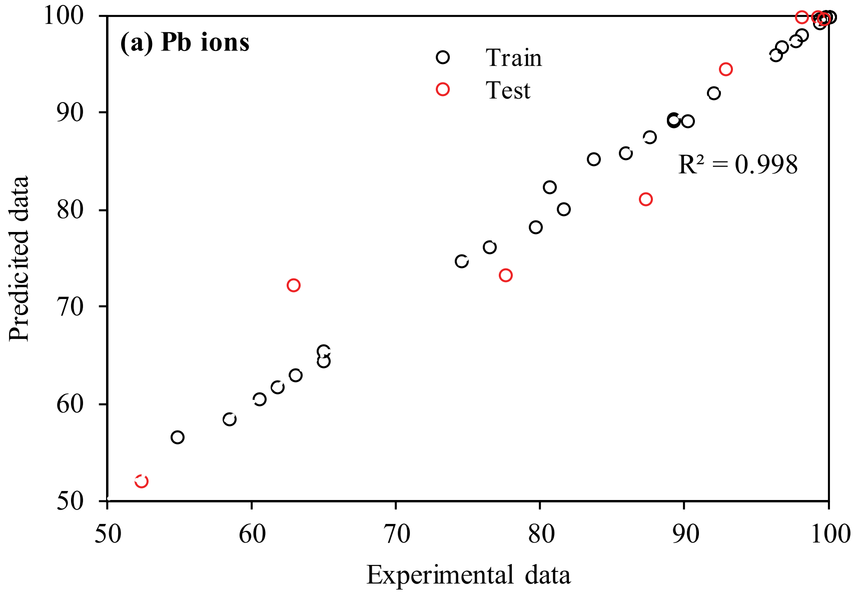
In previous sections ANN and ANFIS models were proposed to predict the removal efficiency of Pb and Ni ions from the active sludge. The model will be accurate if the predicted data are in accordance with the experimental results. In this work, GA was used to determine the optimal operating conditions of the adsorption process through minimization of the deviation between the experimental and the predicted data. GA is a powerful optimization technique which is inspired by nature and searches the problem space accidentally to find better answers for the problem [28,29]. This algorithm is one of the best optimization tools in numerical solution to engineering and scientific problems. The objective function to minimize the mean squared error is as follows:

$$\text{Minimize } F_{\text{obj}} = \sum_{i=1}^{n} \left(y_{\text{Model},i} - y_{\exp,i} \right)^2 \quad (9)$$

The range of decision variables (pH, contact time, adsorbent dosage, and temperature) are listed below:

$$\text{pH} \in \{5,6\}$$
$$\text{Contact time} \in \{5,10,20,150,200\}$$
$$\text{Adsorbent Dosage} \in \{0.001,0.002,0.003,0.004,0.005\}$$
$$\text{Temparature} \in \{298,308,318\}$$

The current optimization process is not too complex since the input parameters of the function have definite and discrete values. The results of optimization showed that the minimum error between the experimental and the predicted



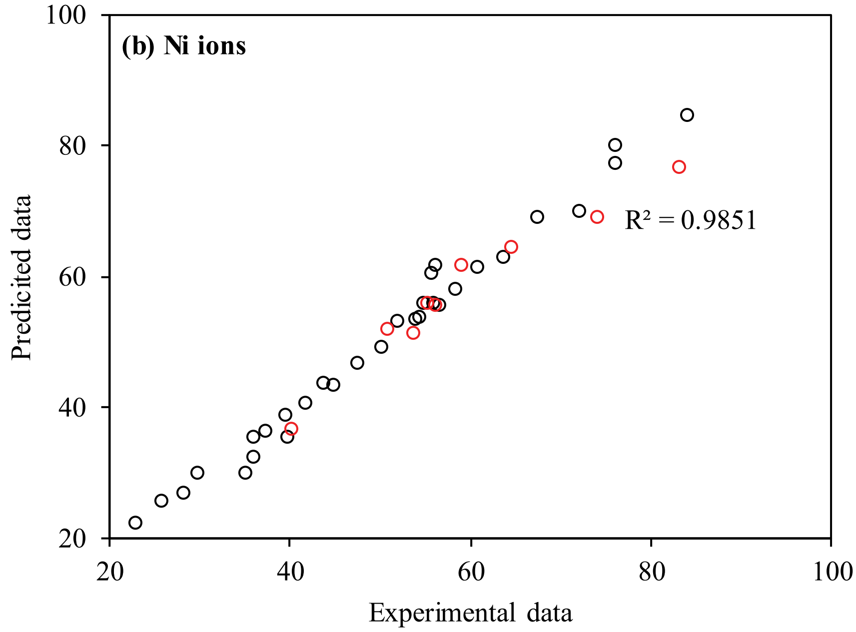


Fig. 4. Comparison between experimental data and ANN predicted values; (a) lead and (b) nickel ions.

Table 2
Technical information of ANFIS model

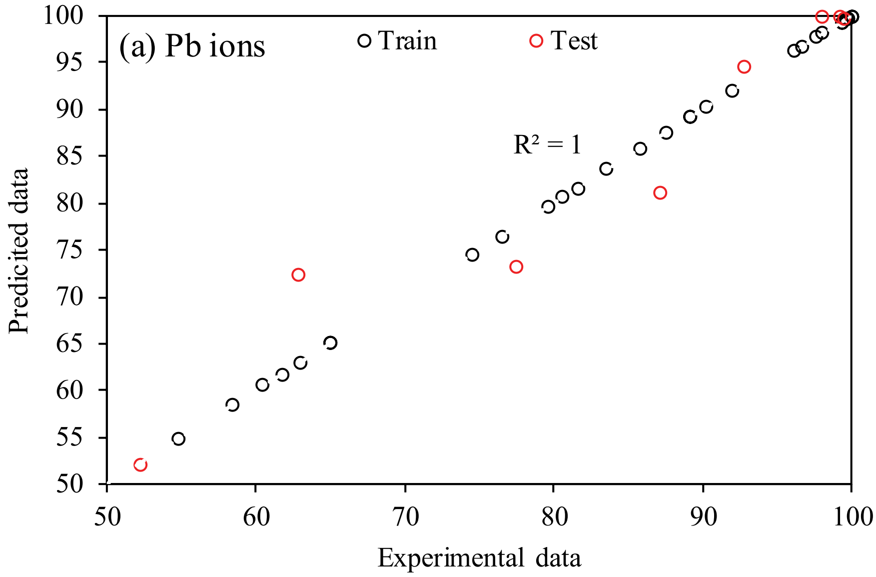
ANFIS information		
Characteristic	Pb removal	Ni removal
Number of nodes	272	272
Number of linear parameters	132	132
Number of nonlinear parameters	220	220
Total number of parameters	352	352
Number of training data pairs	32	31
Number of testing data pairs	11	20
Number of fuzzy rules	22	22

data was achieved in the following optimal conditions: pH = 5, 200 min, adsorbent dosage = 0.002 g/mL, and 298 K. Based on the optimization results, a real sample of leaching solution was then treated under the achieved optimal conditions to test these results experimentally.

6. Adsorption of ions in the optimal conditions

The result of acid wash process using HNO_3 is presented in Table 4. As it can be seen, the appropriate pH value for the extraction process was 2.

pH is a significant component in the extraction system, since it affects the solution of these metals in the acid. The metallic ions are removed from the leaching solution



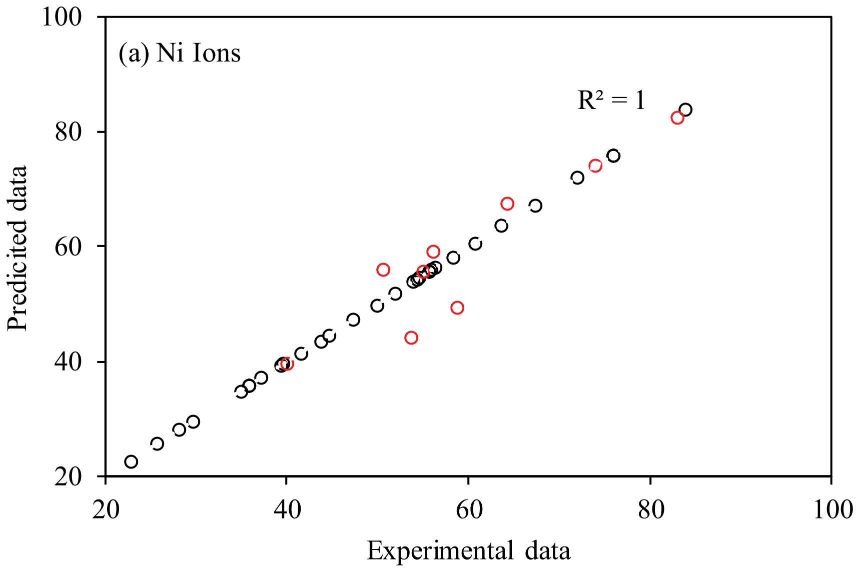


Fig. 5. Comparison between experimental data and ANFIS predicted values; (a) lead and (b) nickel ions.

Table 3
Statistical data of ANN-GMDH and ANFIS models

Removal	Variable	RMSE		HYBRID		ARE		R^2	
		ANFIS	ANN	ANFIS	ANN	ANFIS	ANN	ANFIS	ANN
	Train	0.0001	0.712	0	1.1433	0.0006	0.571	1	0.999
Pb removal	Test	3.8143	2.1186	0.3194	1.5845	3.3879	1.9688	0.9717	0.9895
	Total	0.9758	1.0718	0.0817	1.2562	0.8671	0.9286	0.9927	0.9964
	Train	0	4.8894	0	11.8546	0.0007	3.0875	1	0.9925
Ni removal	Test	1.5355	1.2415	6.3151	4.19	1.9815	1.3333	0.9572	0.9741
	Total	0.3745	3.9997	1.5403	9.9852	0.4838	2.6596	0.9896	0.988

Table 4
The result of extraction of some metallic ions by HNO_3 acid wash

Pb Conc. (ppm)	Ni Conc. (ppm)	Acid pH	Acid Conc.
750	110	2.5	0.2
850	110	2	0.4
770	100	1.5	0.6
740	100	1	0.8
760	120	0.5	1

Table 5
The results of heavy metal ions adsorption from the sewage extraction solution by the activated carbon prepared from the sludge sewage

Sample	Pb (ppm)	Ni (ppm)
Before adsorption	850	60
After adsorption	310	8

Table 6
Comparison between the current work and published earlier articles

Adsorbent	Ni	Pb	References
Activated carbon	–	51.81	[31]
Activated carbon	17	–	[32]
Activated carbon	14.025	–	[33]
Activated carbon	–	6	[34]
Natural zeolite	–	16.81	[35]
Brewed tea waste	82	97.97	[36]
PANI@APTS-Fe_3O_4/ATP	63	87	[37]
Activated carbon	83.33	100	This work

after the extraction. Such a removal is carried out by the adsorption process [27].

The adsorption of metallic ions from the acid wash solution is done by addition of 0.002 g/mL of activated carbon prepared from the sewage sludge to the extraction solution. The solution pH, contact time, and temperature were adjusted at 5, 200 min, and 298 K, respectively, and the results are presented in Table 5.

It is apparent from these data that metals concentration decreased to the limits reported by the study of Treybal [30], which denotes the efficiency of the applied adsorbent.

Table 6 summarizes a comparison between the present work and the other methods given in the earlier published articles for the evaluation of adsorption performance.

7. Conclusions

The main objective of the present study was to investigate the capability of ANN and ANFIS models in prediction of the percentage of Pb and Ni removal from the active sludge. According to the reported results, it was evident that these models were promising prediction techniques which can be used effectively with satisfactory accuracy for the prediction of such processes. In all the train and test datasets, the performances of both models were measured using the Average Relative Error (ARE), AARE, MSE, RMSE and R^2 values. The statistical analysis showed that these two models had almost similar efficiencies ($R^2 > 0.98$), although ANFIS presented more accurate results. Afterwards the optimization of the target function showed that the optimal conditions for the adsorption of Pb and Ni ions are pH = 5, 200 min, adsorbent dosage = 0.002 g/mL, and 298 K. Finally, a real leaching solution sample was treated based on these optimal conditions which yielded acceptable adsorption efficiencies.

Symbols

x_j	—	Input signal
w_{kj}	—	Neuron's weight
u_k	—	Outline linear compiler due to input signal
y_k	—	Neuron output signal
x^*	—	Assumed parameters adapted by a combined algorithm

Greek

ϕ	—	Activation function
σ	—	The assumed parameters adapted by a combined algorithm

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