



## Adaptive Neuro-fuzzy Inference System Prediction of Zn Metal Ions Adsorption by $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/Polyrhodanine Nanocomposite in a Fixed Bed Column

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### ABSTRACT

This study investigates the potential of an intelligence model namely, Adaptive Neuro-Fuzzy Inference System (ANFIS) in prediction of the Zn metal ions adsorption in comparison with two well known empirical models included Thomas and Yoon methods. For this purpose, an organic-inorganic core/shell structure,  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/polyrhodanine nanocomposite with  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticle as core with average diameter of 15 nm and polyrhodanine as shell with thickness of 3 nm, was synthesized via chemical oxidation polymerization. The properties of adsorbent were characterized with transmission electron microscope (TEM) and Fourier transform infrared (FT-IR) spectroscopy. Sixty seven experimental data sets including the treatment time (t), the initial concentration of Zn (C<sub>0</sub>), column height (h) and flow rate (Q) were used as input data to predict the ratios of effluent-to-influent concentrations of Zn (C<sub>t</sub>/C<sub>0</sub>). The results showed that ANFIS model with the R coefficient of 0.99 can predict C<sub>t</sub>/C<sub>0</sub> more accurately than empirical models. Also it was found that the result of the Thomas and Yoon methods with R coefficient of 0.828 and 0.829, respectively were so close to each other. Finally, performance of our ANFIS model was compare to Thomas and Yoon methods in two different conditions, i.e. variable initial influent concentration and variable column height. High performance of ANFIS model was proved by the comparative results.

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## 1. INTRODUCTION

The heavy metals are the common pollutants found in the industrial effluents. Zinc is widely used in industries and the wastewater from these industries is polluted with zinc in large quantities [1]. Adsorption is a common practice for heavy metals removal from drinking water due to the technological and cost advantages [2, 3]. Recent advances in nanotechnology focuses on the fabrication of nano-sized adsorbents with enhanced adsorption capacity and rapid sorption rate for the removal of target contaminants [4]. This is due to the large surface area and highly active surface sites of the nanoadsorbents. Magnetic nanoparticles,

especially maghemite as an efficient adsorbent received continuous attention because of their high adsorption capacity due to their high surface area, low cost, environmental sustainability, easy separation and easy recovery via an external magnetic field [5]. Among the conductive polymers, polyrhodanine has attracted considerable attention in various application fields such as anticorrosion and antibacterial [6].

For the removal of heavy metals in large scale wastewater treatment, utilizing the column type continuous flow operations have distinct advantages over batch treatment. The mechanism of adsorption in continuous flow operations is highly complex and is difficult to model by means of conventional mathematical modeling [7]. Nonlinearity of adsorption mechanism is mainly due to the interaction of adsorption process variables such as pH, initial heavy

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metal concentration, adsorbent mass, agitation speed and temperature, etc. [8]. Artificial neural network (ANN) technique has emerged as a powerful tool which can be used for many scientific and/or engineering applications such as process control and system modeling [9]. ANNs are flexible modeling tools with capabilities of learning the mathematical mapping between input and output variables of nonlinear systems. One of the most powerful types of neural network system is adaptive neuro fuzzy inference system (ANFIS) [10]. ANFIS integrates the fuzzy inference system with a back-propagation learning algorithm of neural network. Although some researchers used neural network to model adsorption of heavy metals [11] and other species such as dyes [12], but to the best of our knowledge, nothing has been reported on the utilizing the ANFIS model to predict heavy metals ion adsorption in a batch or continuous system. In this study, ANNFIS model will be established to study fixed bed column adsorption behavior of Zn ions using  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/polyrhodanine core-shell structure nanocomposite.

**2. THEORY**

**2. 1. Thomas Model** Thomas model is mostly used to describe the column performance and predict the breakthrough curves. The model follows Langmuir kinetics of adsorption-desorption with assumption of negligible axial dispersion in the column adsorption as the rate driving force follows the second-order reversible reaction [13]. The linearized form of the model is given as follows:

$$\ln\left(\frac{C_0}{C_t} - 1\right) = \frac{K_{Th}q_0m}{Q} - K_{Th}C_0t \tag{1}$$

Where C<sub>t</sub> and C<sub>0</sub> are the Zn effluent and influent concentration (mg/L), respectively. The k<sub>Th</sub> is the Thomas rate constant (mL/min mg), q<sub>0</sub> is the adsorption capacity (mg/g), m is mass of adsorbent (g), and Q is the influent flow rate (mL/min).

**2. 2. Yoon-Nelson Model** Yoon and Nelson [14] developed a model to investigate the breakthrough behavior of adsorbate gases on activated carbon. The linearized Yoon-Nelson model is expressed as follows:

$$\ln\left(\frac{C_t}{C_0 - C_t}\right) = K_{YN}t - \tau K_{YN} \tag{2}$$

where, C<sub>t</sub> and C<sub>0</sub> are the Zn effluent and influent concentration (mg/L), respectively. The k<sub>YN</sub> (min<sup>-1</sup>) is the rate constant and τ (min) is the time required for 50% adsorbate breakthrough.

**2. 3. ANFIS Model** The ANFIS model which was proposed by Jang [15] is a combination of fuzzy

inference system and neural network and took the advantages of both techniques. This combination employs a fuzzy system to represent knowledge in an interpretable way and a neural network to capture the learning ability in order to adjust the membership functions parameters and linguistic rules directly from data which in such a way the system’s performance will enhance. As shown in Figure 1, the ANFIS architecture has generally five layers which are input nodes, rule nodes, average nodes, consequent nodes and output nodes respectively.

The description of each layer for this architecture is as follows:

**Layer 1.** This layer is called the fuzzification layer and every node i in it has a membership function (MF) which can be any continuous and piecewise differentiable function. Its role is to transform the input value x into a membership degree which is a value between 0 and 1. There are several membership functions but the most broadly used ones are the generalized bell (gbellmf) and the Gaussian function (gaussmf) which are represented by equations (3) and (4), respectively. Parameters a, b, and c are premise parameters set and are applied in order to describe the characteristics of each MF and determine its shape.

$$\mu_{Ai}(x) = \frac{1}{1 + \left[\frac{x - c_i}{a_i}\right]^{2b_i}} \tag{3}$$

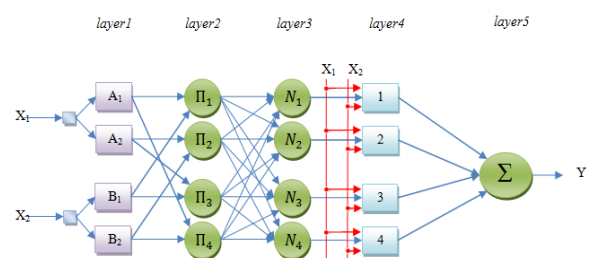
$$\mu_{Ai}(x) = \exp\left[-\left(\frac{x - c_i}{a_i}\right)^2\right] \tag{4}$$

**Layer 2.** The output of each node in this layer is the product of all the received signals and consequently the firing strength of each rule is found.

$$O_{2,i} = w_i(x) = \mu_{iA}(x) \times \mu_{iB}(x) \text{ for } i=1, 2 \tag{5}$$

**Layer 3.** This layer which is called the normalization layer uses the firing strength (w<sub>i</sub>) of layer 2 and normalizes them based on the following equation:

$$O_{3,i} = \bar{w}_i = \frac{w_i}{w_1 + w_2} \text{ for } i=1, 2 \tag{6}$$



**Figure 1.** General architecture of ANFIS model [15]

Layer 4. The nodes of this layer are adaptive nodes using the function bellow:

$$O_{4,i} = \overline{w_i f_i} = \overline{w_i (p_i x + q_i y + r_i)} \quad \text{for } i=1, 2 \quad (7)$$

$P_i$ ,  $q_i$  and  $r_i$  are the parameters of each node and are referred to as consequent parameters.

Layer 5. In the current layer the single node calculates the overall output as the summation of all received signals:

$$\text{Overall output} = O_{S,i} = \frac{\sum_i \overline{w_i f_i}}{\sum_i \overline{w_i}} \quad (8)$$

Generally in order to design an ANFIS model with high accuracy, the initial number of parameters, the number of inputs and rules of the system must be considered which are almost determined using trial and error method.

### 3. MATERIALS AND METHODS

#### 3. 1. Adsorbent Preparation

An organic-inorganic core/shell structure adsorbent,  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/polyrhodanine nanocomposite with  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticle as core and polyrhodanine as shell, was synthesized. According to previous work reported in literature [16] the reaction was carried out via chemical oxidation polymerization.

#### 3. 2. Data Description

Sixty seven experimental data sets were used to develop the ANFIS model. The input variables to the ANFIS model are as follows: the treatment time (t), the initial concentration of Zn (Co), Column height (h) and flow rate (Q). Also the ratio of effluent-to-influent concentrations of Zn (C<sub>i</sub>/C<sub>0</sub>) was chosen as the output variable. Data specifications of model variables are summarized in Table 1.

#### 3. 3. Methodology

In this study, in order to compare the results of different models and specially to evaluate performance of ANFIS model, statistical parameters (as shown in Table 2) were utilized. In statistics, the mean absolute error or MAE is a quantity used to measure how close forecasts or predictions are to the eventual outcomes. The smaller value of this index indicates higher accuracy of the model. The next parameter is RMSE which is a frequently used measure of the differences between values predicted by a model or an estimator and the values actually observed that must be minimum if a good model is expected. The Nash–Sutcliffe model efficiency coefficient is used to assess the predictive power of hydrological models. Nash–Sutcliffe efficiencies can range from  $-\infty$  to 1. An efficiency of 1 (N-S = 1) corresponds to a perfect match of modeled C<sub>i</sub>/C<sub>0</sub> to the observed data.

**TABLE 1.** Data statistics of model variables

Variable	Range
Time (min)	300- 6000
Co (ppm)	50, 100 and 150
Column height (cm)	10, 15 and 20
Flow rate (ml/min)	0.5
C <sub>i</sub> /C <sub>0</sub>	0.000002- 1

**TABLE 2.** Performance criteria used in this study

MAE	mean absolute error
RMSE	Root Mean Squared Error
N-S	Nash–Sutcliffe model efficiency coefficient
R	Pearson product-moment correlation coefficient

An efficiency of 0 (N-S = 0) indicates that the model predictions are as accurate as the mean of the observed data, whereas an efficiency less than zero (N-S < 0) occurs when the observed mean is a better predictor than the model or, in other words, when the residual variance (described by the numerator in the expression above), is larger than the data variance (described by the denominator). Essentially, the closer the model efficiency is to 1, the more accurate the model is. R or the Pearson product-moment correlation coefficient is a measure of the linear correlation (dependence) between two variables X and Y, giving a value between +1 and -1 inclusive, where 1 is total positive correlation, 0 is no correlation, and -1 is total negative correlation.

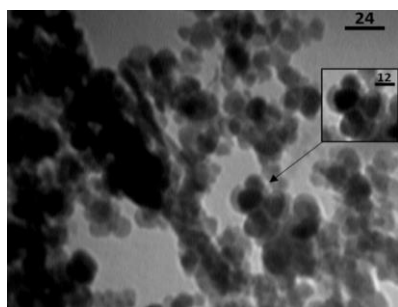
### 4. RESULTS AND DISCUSSION

#### 4. 1. Morphology

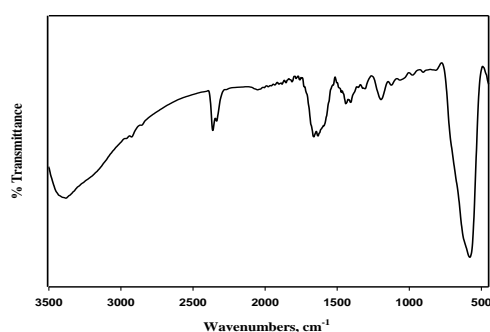
TEM image of nanocomposite is shown in Figure 2. From this figure, it can be obviously seen that the synthesized nanocomposite had a core–shell structure which surface of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> is coated with a thin layer of polyrhodanine. The dark color in the center is attributed to  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> and the light colored around the center can be attributed to polyrhodanine. The average size of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/polyrhodanine core/shell nanoparticles is about 18 nm. The maghemite core diameter is about 15 nm and thickness of the polymer shell is estimated to be 3 nm.

#### 4. 2. FT-IR Spectroscopy

Figure 3 nanocomposite particles. As displayed in this figure, the characteristic vibrations of Fe-O in  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> are clearly observed at 436 and 586 cm<sup>-1</sup>. In addition, the peak at 1601 cm<sup>-1</sup> is related to the OH bending of water, and the absorption peak at 3430 cm<sup>-1</sup> is for the hydroxyl group (-OH). There is a tiny dip in the spectra at 2362 cm<sup>-1</sup> due to the presence of atmospheric CO<sub>2</sub>.



**Figure 2.** TEM image of  $\gamma$ - $\text{Fe}_2\text{O}_3$ /polyrhodanine core-shell nanocomposite



**Figure 3.** FT-IR spectra of  $\gamma$ - $\text{Fe}_2\text{O}_3$ /polyrhodanine nanocomposite

The peak at  $1196\text{ cm}^{-1}$  is assigned to the  $\text{C}-\text{O}^-$  stretching vibration. A band at  $1441\text{ cm}^{-1}$  is assigned to the  $\text{C}=\text{N}^+$  stretching in the polymer chain. These peaks at  $1661\text{ cm}^{-1}$  are attributed to the stretching vibration of  $\text{C}=\text{C}$  groups in the polymer chain.

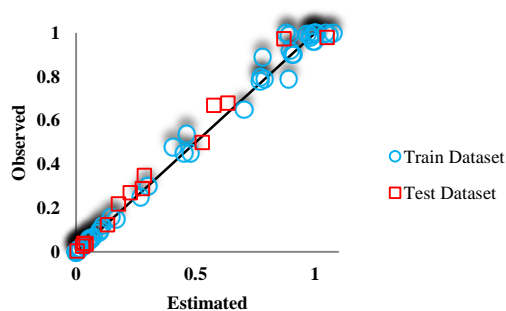
#### 4. 3. ANFIS Model

To achieve the best performance of the ANFIS model different architectures of it were constructed. Table 3 shows the effect of various membership functions. As it can be seen, the max value of R for the test data set is 0.994 when the MF is generalized bell-shaped built-in membership function (gbellmf). After it trapezoidal-shaped built-in membership function (trapmf) and gaussian curve built-in membership function (gaussmf) have the values of 0.993 and 0.991, respectively for R parameter and are considered as the best MFs in this research.

After training and evaluation of ANFIS, its ability to predict  $C_t/C_0$  has to be checked. In this regard performances of the proposed architecture are compared with both of the testing and training data in Figure 4. The lines with the slope of  $45^\circ$  in the middle of these figures are the locations of exact predictions and points in the figure show training and testing data.

**TABLE 3.** Results of modeling with ANFIS model with different transfer functions

Number	Function	Train			Test		
		RMSE (ppm/ppm)	MAE (ppm/ppm)	R	RMSE (ppm/ppm)	MAE (ppm/ppm)	R
1	gbellmf	0.0233	0.0156	0.9985	0.0379	0.0328	0.9941
2	dsigmf	0.0288	0.0191	0.9977	0.0599	0.0493	0.9848
3	gauss2mf	0.0314	0.0224	0.9972	0.0770	0.0485	0.9756
4	gaussmf	0.0160	0.0119	0.9993	0.0460	0.0370	0.9907
5	pimf	0.0473	0.0348	0.9937	0.0528	0.0449	0.9881
6	Psigmf	0.0248	0.0171	0.9983	0.0512	0.0445	0.9897
7	Trapmf	0.0384	0.0280	0.9959	0.0416	0.0330	0.9928
8	Trimf	0.0413	0.0280	0.9952	0.0977	0.0738	0.9649



**Figure 4.** Comparison of experimental and calculated values for training and testing data

The error of each prediction is relative to the distance between each point and the line, so disperse of points near this line may demonstrate the overall accuracy of the present model. As it shows the accuracy of ANFIS in prediction of  $C_t/C_0$  is remarkable.

#### 4. 4. Comparing ANFIS and Empirical Models

As it can be observed from Table 4, ANFIS model in comparison with empirical models has the lowest mean absolute error and root mean squared error. The value of Pearson product-moment correlation coefficient is equal to 0.994 where for empirical models this value is about 0.8. The Nash-Sutcliffe coefficient for ANFIS is

0.987 while it is 0.68 and 0.677 for Yoon and Thomas models, respectively. This reveals that the results from the proposed ANFIS model are in better agreement with experimental data.

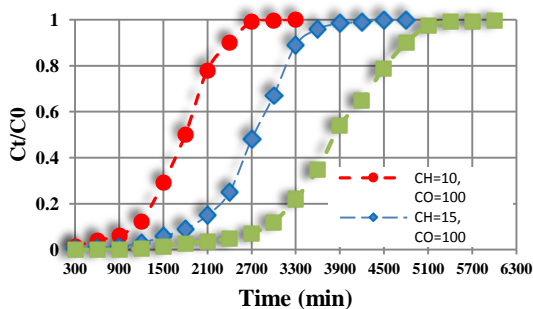
**4. 4. 1. Effect of the Column Height** After constructing the models some conditions of the system were considered and the models efficiency was checked. First for the constant value of influent concentration equals to 100 ppm, the absorption column height varied from 10 to 20 cm. The adsorption performance of the  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> /polyrhodanine nanocomposite was investigated at various bed heights of 10, 15 and 20 cm at a flow rate of 0.5 mL/min where influent concentration was kept constant at 100 ppm.

Figure 5 shows the breakthrough profile of Zn adsorption at different bed heights. For the different used bed depths, as the bed depth increases, the quantity of the removed Zn increases which is also illustrated by the service time change. Generally by increasing column height, contact time between adsorbent and contaminant will increase and also more adsorbent will be available in adsorption which effect total service time. As shown in Figure 5, in the interval of 1500 min, the value of  $C_t/C_o$  reached 0.29, 0.058 and 0.013 when bed depth was 10, 15 and 20 cm, respectively. At the column depth with 10 cm, the adsorbents become saturated very quickly because lower quantity of it is available in column.

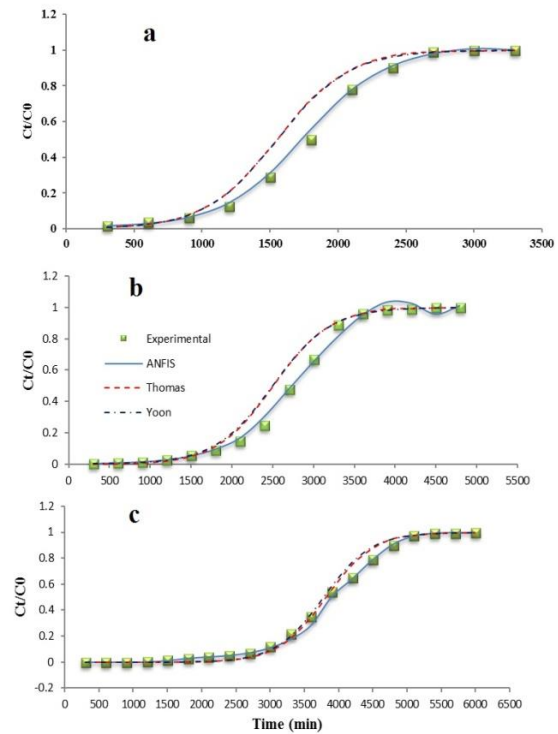
From Figure 6 the outputs of each model and experimental data were plotted versus time, such a way the usefulness of models is visualized. In this figure the squares show the experimental data, solid line shows the ANFIS model and each dashed line shows one of the empirical models.

**TABLE 4.** Statistical accuracy of ANFIS and empirical models

	MAE	RMSE	N-S	R
ANFIS Model	0.0376663	0.0506469	0.976	0.990418
Thomas Model	0.1213000	0.1866213	0.677	0.828102
Yoon Model	0.1207857	0.1859174	0.680	0.829063



**Figure 5.** The effect of bed height on the shape of the breakthrough curves



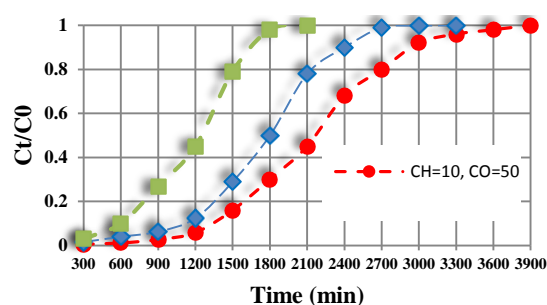
**Figure 6.** Comparison of ANFIS, Thomas and Yoon outputs and experimental data at  $C_o=100$  ppm and a) CH=10 cm, b) CH=15 cm, c) CH=20 cm.

It is confirmed that the results of ANFIS are in good agreement with experimental data considering all three absorption column heights and the MAE index has values of 0.013, 0.027 and 0.023, respectively for column heights equal to 10, 15 and 20 cm while empirical models were not as good as ANFIS and their MAE changes from 0.026 for CH= 20 cm to 0.062 for CH= 10 cm.

**4. 4. 2. Effect of Influent Concentration** To investigate effect of influent concentration on performance of fixed bed column, at the constant column height of 10 cm the influent concentration was varied from 50 ppm to 150 ppm.

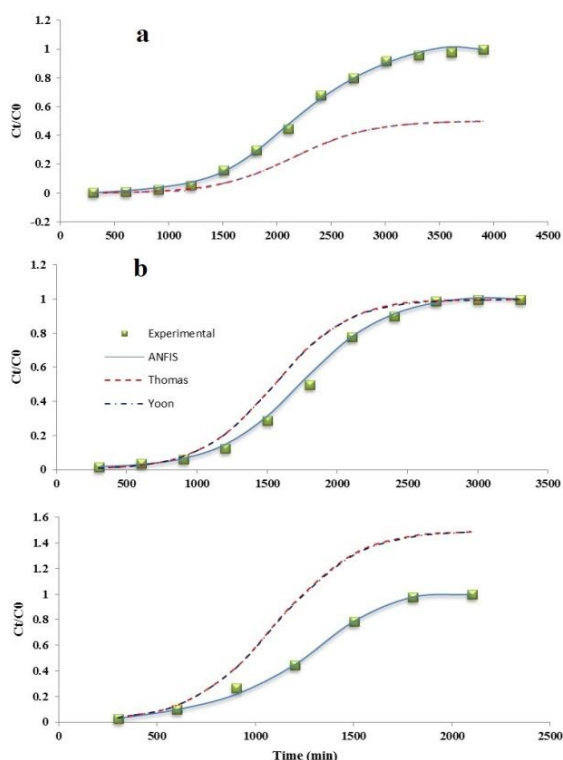
The effect of influent Zn concentration on the shape of the breakthrough curves is shown in Figure 7. As shown in this figure, in the interval of 1500 min, the value of  $C_t/C_o$  reached 0.16, 0.29 and 0.79 when influent concentration was 50, 100 and 150 mg/L, respectively.

It is illustrated in Figure 7 that the breakthrough time decreased with the increase of influent concentration. At lower influent concentration, breakthrough curve was dispersed and breakthrough occurred slower. As influent concentration increased, sharper breakthrough curves were observed. These results demonstrate that the change of concentration gradient affects the saturation rate and breakthrough time.



**Figure 7.** The effect of influent Zn concentration on the shape of the breakthrough curves.

These results also show that the change of concentration gradient affects the saturation rate of adsorbent and breakthrough time, in other words, the diffusion process is concentration dependent. Also as illustrated in Figure 8, it is obvious that there is a good agreement between ANFIS outputs (solid line) and experimental data (square points) and the value of MAE is less than 0.022 while it varies from 0.062 to 0.247 for empirical models. Although the results of Thomas and Yoon models are not bad for the condition of  $C_0=100$  ppm, but both of them underestimate and overestimate the results respectively for  $C_0$  equal to 50 and 150 ppm. Results indicate that non of empirical models have great performance in prediction of  $C_t/C_0$  specially when the value of  $C_0$  decrease from 100 ppm to 50 ppm or increases to 150 ppm.



**Figure 8.** Comparison of ANFIS, Thomas and Yoon outputs and experimental data at CH=10 cm and a)  $C_0 =50$  ppm, b)  $C_0 =100$  ppm, c)  $C_0 =150$  ppm

## 5. CONCLUSIONS

The  $\gamma$ - $\text{Fe}_2\text{O}_3$ /polyrhodanine nanoparticles with core-shell structure have successfully synthesized as adsorbent by chemical oxidation polymerization. The characterization of the obtained products shows that nanoparticles are spherical, monodisperse. Prepared adsorbent applied for adsorption of Zn ions from aqueous solution in a fixed bed column. The dynamic behavior of adsorption process has been investigated using ANFIS, Thomas and Yoon-Nelson models.

Results of testing subset clearly demonstrate that the ANFIS model has a maximum correlation coefficient of 0.99 and showed great performance while the R coefficient was less than 0.829 for empirical models.

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Polyrhodanine

Fixed Bed Column

این مطالعه به بررسی عملکرد مدل هوشمند سیستم استنتاج عصبی-فازی سازگار در پیشبینی جذب فلز روی و مقایسه آن با ۲ مدل تجربی مشهور توماس و یون می پردازد. بدین منظور یک ساختار هسته و پوسته آلی/غیرآلی پلی رودانین/ $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> که پوسته پلی رودانین ضخامت ۳ نانومتر و هسته  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> با متوسط قطر ۱۵ نانومتر به روش پلیمریزاسیون شیمیایی تهیه گردید. خواص جاذب تهیه شده با استفاده از آزمون های میکروسکوپ الکترونی عبوری و اسپکتروسکوپی تبدیل فوریه مورد بررسی قرار گرفت. ۶۷ مجموعه از داده های آزمایشگاهی شامل زمان آمایش، غلظت اولیه روی، ارتفاع ستون و دبی جریان به عنوان داده های ورودی برای پیش بینی نسبت غلظت روی در خروجی به غلظت اولیه مورد استفاده قرار گرفت. نتایج نشان داد که مدل سیستم استنتاج عصبی-فازی سازگار با ضریب R برابر با ۰٫۹۹ نسبت به دو مدل تجربی از دقت بالاتری برخوردار می باشد. همچنین مشخص گردید که نتایج مدل های توماس و یون به ترتیب با ضریب R برابر با ۰٫۸۲۸ و ۰٫۸۲۹ بسیار نزدیک به یکدیگر است. در نتایج نتایج مدل سیستم استنتاج عصبی-فازی سازگار با نتایج مدل های توماس و یون در دو حالت ارتفاع ستون جذب متغیر و غلظت اولیه متغیر مقایسه گردید. نتایج حاصل از مقایسه نشان دهنده عملکرد بهتر مدل سیستم استنتاج عصبی-فازی سازگار بوده است

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