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Research Article

Artificial neural network approach for lead removal from aqueous solution by agricultural waste derived biochars

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ABSTRACT

Lead (Pb^{2+}) which is one of the most important heavy metals found in water sources, has a toxic effect on living things in aquatic environment. Therefore, the removal of Pb^{2+} ions from wastewater is very important if its concentration is above the determined discharge limits. Due to its advantage such as high efficiency, the adsorption process is a widely used successful technique for heavy metals removal from an aqueous solutions. On the other hand, determining the adsorption efficiencies of different adsorbents experimentally is both costly, and time-consuming considering that there are large number of process variables. Therefore, ANN can be used to make theoretical predictions with high efficiency in this treatment process. In this study, modeling of Pb^{2+} removal from an aqueous solution by using biochars that produced by pyrolyzing of hazelnut, and walnut shell was studied at different adsorption conditions; pH (2.5-5), temperature (25-45°C), initial Pb^{2+} concentration (15-45 mg/L), adsorbent amount (1-3 g/L), and mixing speed (200-600 rpm). The purpose of modeling studies with ANN approach was to estimate lead ions removal (%) as an output. Inputs for ANN modeling approach were selected as pH, initial Pb^{2+} concentration, temperature, adsorbent dosage, and mixing speed. Experimental data were categorized 50:25:25 for two adsorption systems. Levenberg-Marquardt (LM) was preferred as a training function, and tansig was used as an activation function. The number of hidden neurons in the hidden layer was found by trial, and error. Values of correlation coefficient (R^2), and Mean Square Error (MSE) were taken to be performance criteria of the ANN modeling. R^2 values were found to be 97%, and 98% for biochars derived from walnut, and hazelnut shells, respectively. Results showed that ANN is an effective tool for modeling of adsorption system for the removal of lead ions from an aqueous solution. Additionally, different training algorithms such as Bayesian Regularization (BR), and Scaled Conjugate Gradient (SCG) were used to compare the prediction capability.

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INTRODUCTION

Lead is one of the oldest known heavy metals and is still used in various industries like battery production, metal plating, mining, pigments manufacturing, fuels, ceramic, and glass production due to its high density, low melting temperature, and corrosion resistance. On the other hand, due to its high solubility in aquatic environments, lead is a well-known major pollutant [1]. Lead is a toxic element to living organisms, and not necessary for physiological function. It can cause many diseases, and deaths even in low concentrations in water. Carcinogenic, and neurological damage, mental, and behavioral changes are associated with lead toxicity. It has negative effects on immune system, and reproductive system. Stomach, lung, and gallbladder cancers as well as increased incidence of all cancers were considered to be are associated with lead exposure [2]. According to the World Health Organization (WHO), the maximum permissible limit (MPL) of lead in wastewater treatment effluents is 10 μg/L. On the other hand, the permissible limit of Pb(II) which is given by the United States Environmental Protection Agency (USEPA) is 6 μg/L [3].

In the removal of lead from wastewater, some traditional methods such as precipitation, coagulation, ion exchange, reverse osmosis, electro-dialysis, chemical reduction, oxidation can be used [4]. With these traditional methods, heavy metals may not be completely removed. Apart from that, these techniques have some disadvantages; expensive equipment, and tracking systems requirement, excessive chemical, and energy needs, formation of toxic sludge, and other wastes. As an alternative to these methods, adsorption is a technique that has been successful in heavy metal removal with low cost, and high efficiency in many studies. In recent years, natural substances have been increasingly used as adsorbents in this process because of their low cost, and easy modification. Even though the availability of various commercial adsorbents, their widespread use is often restricted by the high cost, and limited availability. Therefore, researchers have been trying to develop alternative low-cost but efficient adsorbents using agricultural, and industrial wastes especially [5, 6].

Biochar, a carbon-rich byproduct synthesized through the carbonization of biomass in an oxygen-limited environment, has received more attention from researchers in recent years, and has been recognized as a multifunctional, and low-cost material for environmental applications due to its high absorptive ability. Many organic wastes such as agricultural, industrial, urban, and domestic solid waste can be used as raw materials to produce biochar. Carbonaceous materials are the best-known adsorbents that can be used to remove both organic, and inorganic contaminants from the aquatic environment. Therefore, biochar has also attracted worldwide attention as a useful, low-cost, environmentally friendly adsorbent for various pollutant reclamation due to its large surface area, high adsorption capacity, microporosity, and ion exchange capacity [7].

Adsorption is a process that contains non-linear relations involved in complex chemical processes due to physicochemical characteristics, and the particular structure of adsorbent. The effect of adsorption parameters on the adsorption process is estimated by modeling studies, parameter values can be estimated to ensure high efficiency of heavy metal removal from existing wastewater [8]. Artificial neural network (ANN) is widely used for prediction in many industrial areas for the last 10 years and is an artificial intelligence method that attracts attention in wastewater treatment [9-12]. ANN approach is not dependent only on data, it can evaluate biased data, and even learn complex relationships, generalize, and answer questions it has never encountered before with an acceptable error [13].

There are modeling studies to remove lead ions from an aqueous solutions by using different adsorbents in the literature. Yetilmezsoy, and Demirel used ANN approach for modeling the % Pb^{2+} removal by Antep pistachio (Pistacia Vera L.) shells with R^2 0.936 [14]. Khan et al. used ANN for the adsorption of Pb^{2+} ions by using rice husk carbon (RHC). Mean square error (MSE), and R^2 values were obtained as 6.005, and 0.989, respectively with Levenberg–Marquardt algorithm (LMA) [15]. Sadrzadeh et al. used ANN approach to predict separation percent (SP) of lead ions from wastewater by using electrodialysis process. The modeling results showed that there is an excellent agreement between the experimental data, and the predicted values [16]. Ma et al. used ANN for Pb^{2+} removal from water by using Scenedesmus obliquus which achieved the highest Pb^{2+} removal efficiency at pH 6.0, and algal biomass 2 g/L. Testing model results showed that the correlation between the predicted data, and the experimental data was 0.997, with an accuracy rate of 95.4% [17]. Kardam et al. used ANN approach for the abatement of lead ions from an aqueous solution by using nanocellulose fibers (NCFs) derived from rice straw. Comparison between the network results, and experimental data gave a high degree of correlation coefficient ($R^2 = 0.995$) indicating that the model is able to predict the sorption efficiency successfully [18]. Afroozah et al. used ANN, Fuzzy Inference System (FIS), and Adaptive Neuro-Fuzzy Inference System (ANFIS) for the prediction of Pb^{2+} removal from an aqueous solution using the magnetic nano adsorbent (Graphene/Nylon 6). ANFIS showed better performance than the other models [19]. Amiri et al. used ANN, and MNLR (Multivariate Non-Linear Regression) approach for Pb^{2+} removal from industrial wastewaters by ostrich bone char. ANN model showed good predicting performance with MSE=0.0002, and R^2=0.98. Additionally, this study indicated characterisation of flexibility, and extensibility of ANN model [20].

In this study, ANN modeling of Pb^{2+} removal from an aqueous solutions has been carried out by using two

different biochars produced from hazelnut shell, and walnut shell as an adsorbent. This work is a continuation of the work done by Kaya et al. (2020) [21]. % lead removal was predicted through ANN, and the results compared with experimental values. To create the optimum network architecture, ANN parameter selections were made, and predictive ability of the network was evaluated by R^2, and MSE value.

MATERIALS AND METHOD

Preparation of Biochars Derived from Hazelnut and Walnut Shells

In this study, hazelnut, and walnut shells which are widely available lignocellulosic agricultural wastes were carbonized in an electrically heated fixed bed pyrolysis reactor. Pyrolysis temperature was set to 500°C, and 700°C for hazelnut, and walnut shells, respectively. During the process, the inert nitrogen gas flow rate was set to 100 mL/min, and the heating rate to 20°C/min. During the pyrolysis process, chemical bonds of biomasses were thermally degraded in an oxygen-free environment at high temperatures, and the end of 1 h, biochars with high surface area were obtained [21].

Adsorption Studies

To determine the adsorption properties of biochars produced by pyrolysis of hazelnut, and walnut shells for the removal of lead ions (Pb^{2+}) from an aqueous solutions, batch adsorption experiments were carried out. Commercially available stock lead nitrate solution ($Pb(NO_3)_2$) with a concentration of 1,000 mg/L for lead metal were diluted with distilled water to the desired concentration to prepare the test solutions to be used in the adsorption experiments. Parameters affecting the adsorption process such as pH (2.5-5), adsorbent dosage (1-3 g/L), initial heavy metal concentration (15-45 mg/L), contact time (up to 300 min), temperature (25-45°C), and mixing speed (200-600 rpm) were studied in a batch system. The samples were collected at predetermined time intervals, and adsorbent separated from the samples by filtering. The filtrate was analyzed by ICP-OES (Perkin Elmer, Optima 5300 DV) to determine the residual lead ion concentration. Based on the acquired values, the adsorption yield was calculated by using Equation (1).

$$\text{Lead Removal (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \qquad (1)$$

where C_o, and C_e are the initial, and final lead concentration in solution phase (mg/L), respectively. In order to ensure the reproducibility of the results, all the adsorption experiments were performed in triplicate, and the average values were used in data analysis. Relative standard deviations were found to be within ± 1% [21].

ANN MODELING

Model theory

The first artificial neuron was produced in 1943 by neuropsychiatrist Warren McCulloch, and scientist Walter Pits. However, due to the limited possibilities of the period, there was not much progress in this area. After that, Minsky, and Papert published a book in 1969, eliminating ethical concerns in the field of ANN, and paving the way to this new technology. Hence, the first visible developments in this area was date back to the 1990s [22].

ANN performs operations with cells called neurons and is an improved network modeled on the way the human brain works. ANN can provide linear, and nonlinear modeling between input, and output variables without any prior knowledge. Although there are many studies by many researchers about ANN there is no certainty that what are the key factors affecting the ANN's performance [23]. ANNs are data-based systems created by connecting artificial nerve cells in layers. ANN aims to use the skills of the human brain, such as learning, and making very fast decisions under different conditions, in solving complex problems with the help of simplified models. Basically, all ANNs have a similar structure as shown in Figure 1 [24, 25].

where x (x_i , i = 1, 2, …, n) are inputs to the neuron, and Y is output. Each input is multiplied by its weight w_i, a bias b is associated with each neuron, and their sum goes through a transfer function f. As a result, the relationship between input, and output can be described as follows from Equation (2) [26]:

$$Y = f(\sum_{i=1}^{n} w_i x_i + b) \qquad (2)$$

In this structure, some neurons are connected to the outer space to receive inputs, and to transmit outputs. All the remaining neurons are in hidden layers, meaning they only have connections within the network. While the input, and output layers consist of a single layer, there may be more than one hidden layer between these two layers. These hidden layers contain a large number of neurons, and these neurons are completely connected with other neurons in the network. Network architecture, learning

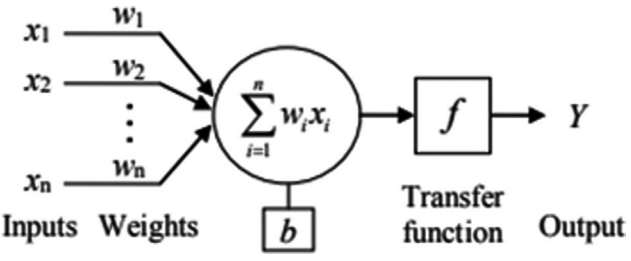

Figure 1. ANN algorithm.

function, and training function are the three main component of ANN. There are no rules or mathematical algorithms for determining the hidden layer, and the number of hidden neurons in this layer. The decision is made by applying the method of trial, and error. Independent variables affecting the system is chosen as an input, and the variable that is tried to be predicted is selected as the output variable [27].

Data division

ANN architecture contains data division step, and data set divided into 3 subsets: a training set, a testing set, and a validation set [28]. The data set used for training is called "training set". Throughout the training, the same training set weights are given to the network over, and over to bring it to the most appropriate level. The word epoch is expressed as showing the entire training set to the network. After completion of training step, validation set how much the network has learned in each iteration, and they use the error values of the validation set. If the desired success is achieved with the data in the training set, the network is tested with data. The data set in which the test is performed is called the "test set" [29].

Training function

Learning in a neural network means finding optimum values of all weights in ANN that will enable the network to produce correct outputs between input, and output data for a particular problem. The operation done to reach optimal weights is called "training the network". Accordingly, in order for a network to be trainable, the weight values must be dynamically changeable within a certain rule. Typically, a training algorithm is used for the learning process, and it determines how the weights are arranged according to a learning sample. Levenberg-Marquardt (LM) method, Bayesian Regulation (BR) method, and Scaled Conjugate Gradient (SCG) algorithm are type of learning algorithms. The Levenberg-Marquardt (LM) algorithm is an approach to the Newton method and is the combination of the speed of the Newton method, and the robustness of the step-down method [30-32].

Activation function

The activation function establishes the link between input, and output. It creates output information by processing the information coming from the summation function. Like the summation function, this function has various functions depending on the ANN model to be applied [33]. Different activation functions are as follows: linear function, sigmoid function, hyperbolic tangent function, sine function, digit function. Depending on the activation function used, the output value is usually [-1,1] or [0,1]. This is usually a nonlinear function. The use of non-linear activation functions has enabled ANNs to be applied to complex, and very different problems [34]. The choice of activation function depends largely on the data of the ANN, and depends on what the network is asked to learn. Commonly used activation functions are sigmoid, and hyperbolic tangent functions. For example, if the network desired to learn the behavior of a model, the sigmoid function can be used or if it desired to learn the deviation, the hyperbolic tangent function can be used [35].

Performance evaluation criteria

Mean square error (MSE) as a performance function, and coefficient of correlation (R^2) are used to evaluate models predicted results. They are shown in Equation 3, and 4 how MSE, and R^2 are calculated. The best model performance is the structure in which R^2 value approaches 1, and MSE approaches 0.

$$MSE = \frac{1}{n}\sum_{i=1}^{n}(X_t - X_t')^2 \quad (3)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(X_t - X_t')^2}{\sum_{i=1}^{n}(X_t')^2} \quad (4)$$

where, X_t, and X_t' are the actual, and predicted values, and n is the total number of observations [36].

RESULTS AND DISCUSSION

In this study, experimental results for the Pb^{2+} removal from an aqueous solution by biochars that produced by pyrolyzing of hazelnut, and walnut shell were modeled with ANN approach. Levenberg–Marquardt feed-forward algorithm was used. The ANN were developed in the Neural Fitting toolbox, Matlab R2017a. ANN structure of the adsorption process was given in Figure 2. pH, temperature, initial Pb^{2+} concentration, adsorbent amount, and mixing speed were used as input for the

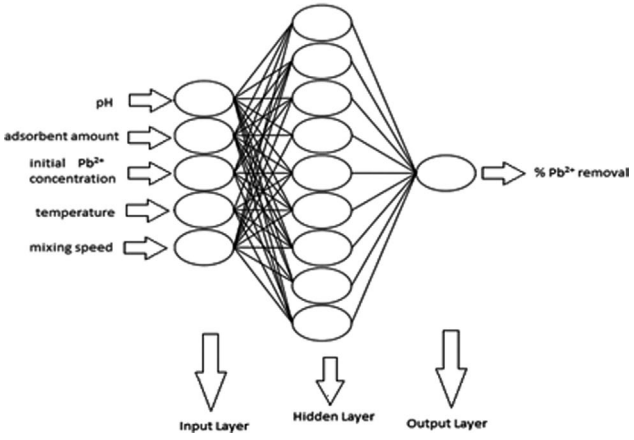

Figure 2. Figure 2.ANN architecture.

system to estimate the removal efficiency of lead ions. One hidden layer was used, and different numbers of neuron combinations were in this layer between 1-25. Optimum value was selected according to maximize of R^2, and reduction of MSE model performance criteria. Levenberg-Marquardt (LM) training function which is known that it is a fast algorithm was used in training the network. This algorithm frequently used in feed-forward networks [37]. Also, ANN models were developed and trained with two different training algorithms including BR and SCG, and have been evaluated on the basis of their predictive capability.

Tanjant sigmoid (tansig) function was preferred as an activation function in this study. The most common problem encountered during ANN training is the overfitting problem. Once the network has been trained with a sufficient number of cycles, the error can be reduced to reasonable levels but after the network is fed with test data, very high error values are encountered. This indicates that the network memorizes the training data, and weights for the values appropriate to it that it has updated but has not reached the ability to generalize. Therefore, data division ratio is important to solve the overfitting problem. In this study when data divided into 70:15:15,

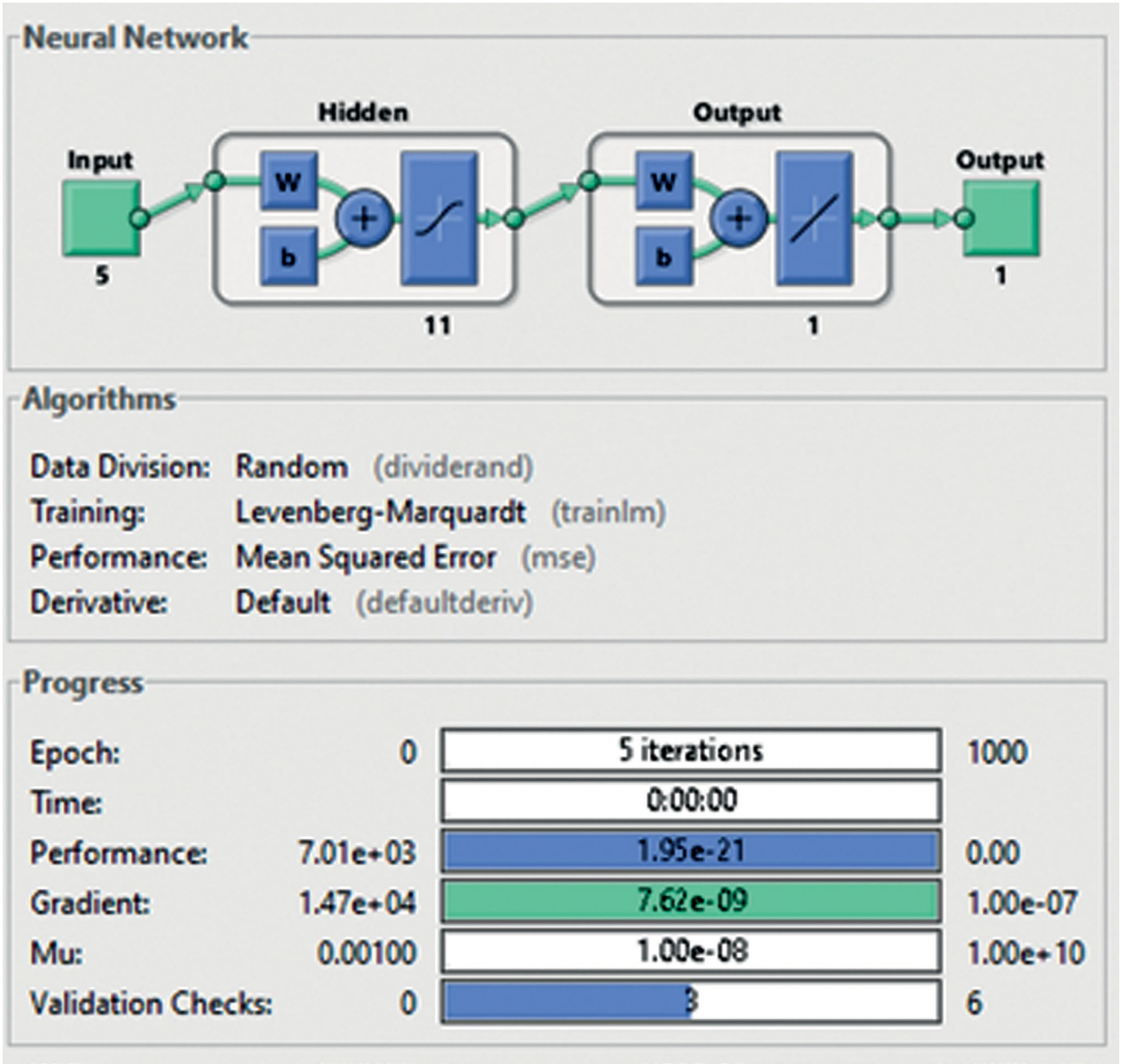

Figure 3. ANN toolbox for biochar derived from walnut shell.

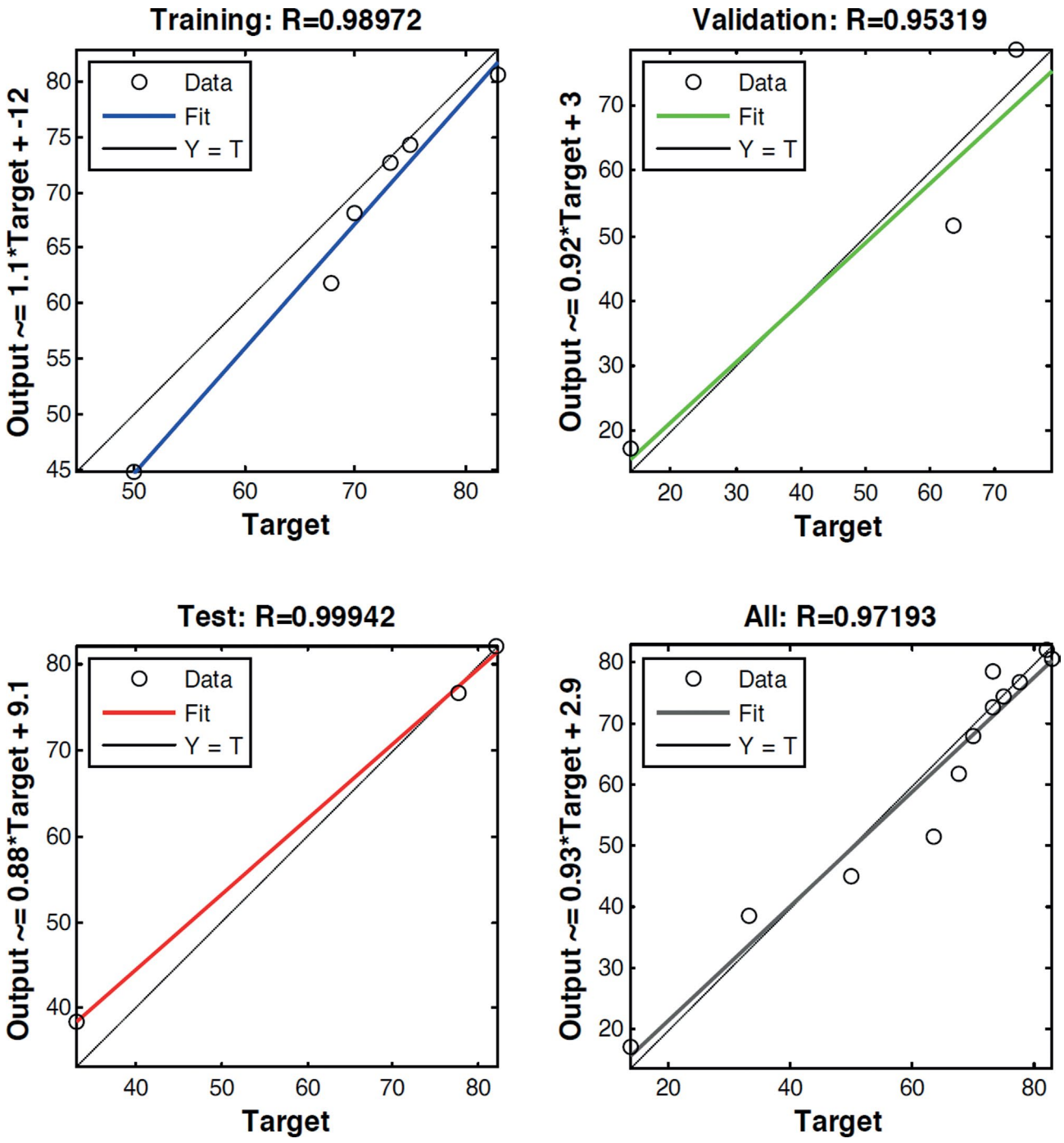

Figure 4. ANN regression plots for biochar derived from walnut shell.

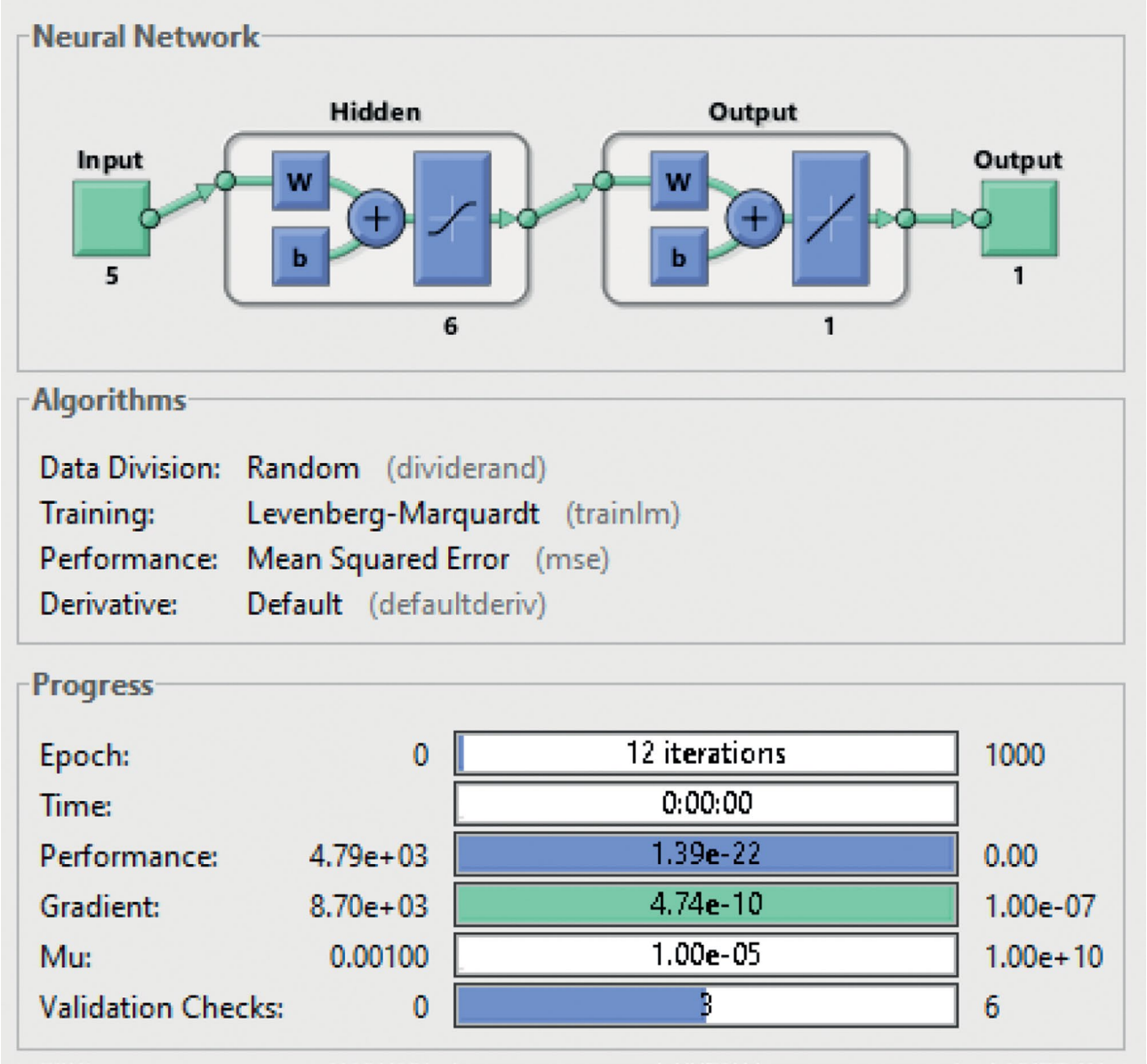

Figure 5. ANN toolbox for biochar derived from hazelnut shell.

and 60:20:20, R^2 value was 1 but MSE values were too high. That's why this data division ratio was changed to 50:25:25. The number of experimental measurement results used for training, validation, and testing were 6:4:4.

ANN Modeling of Pb^{2+} Removal by Using Biochar Derived from Walnut Shell

5 inputs (pH, temperature, initial Pb^{2+} concentration, adsorbent amount, and mixing speed) were defined in the system, and number of neurons in hidden layer was found to be 11 by trial, and error as seen from Figure 3. No significant change occurred on the method's performance after 5 epochs. Best validation performance was MSE 0.011 for trainig, 0.0063 for validation, and 0.0095 for testing steps; R^2 was 0.97 for all of the system (Figure 4).

ANN Modeling of Pb^{2+} Removal by Using Biochar Derived from Hazelnut Shell

5 inputs (pH, temperature, initial Pb^{2+} concentration, adsorbent amount, and mixing speed) were defined in the system, and number of neurons in hidden layer was found to be 6 by trial, and error as seen from Figure 5. Maksimum epoch number was found to be 12. Best validation

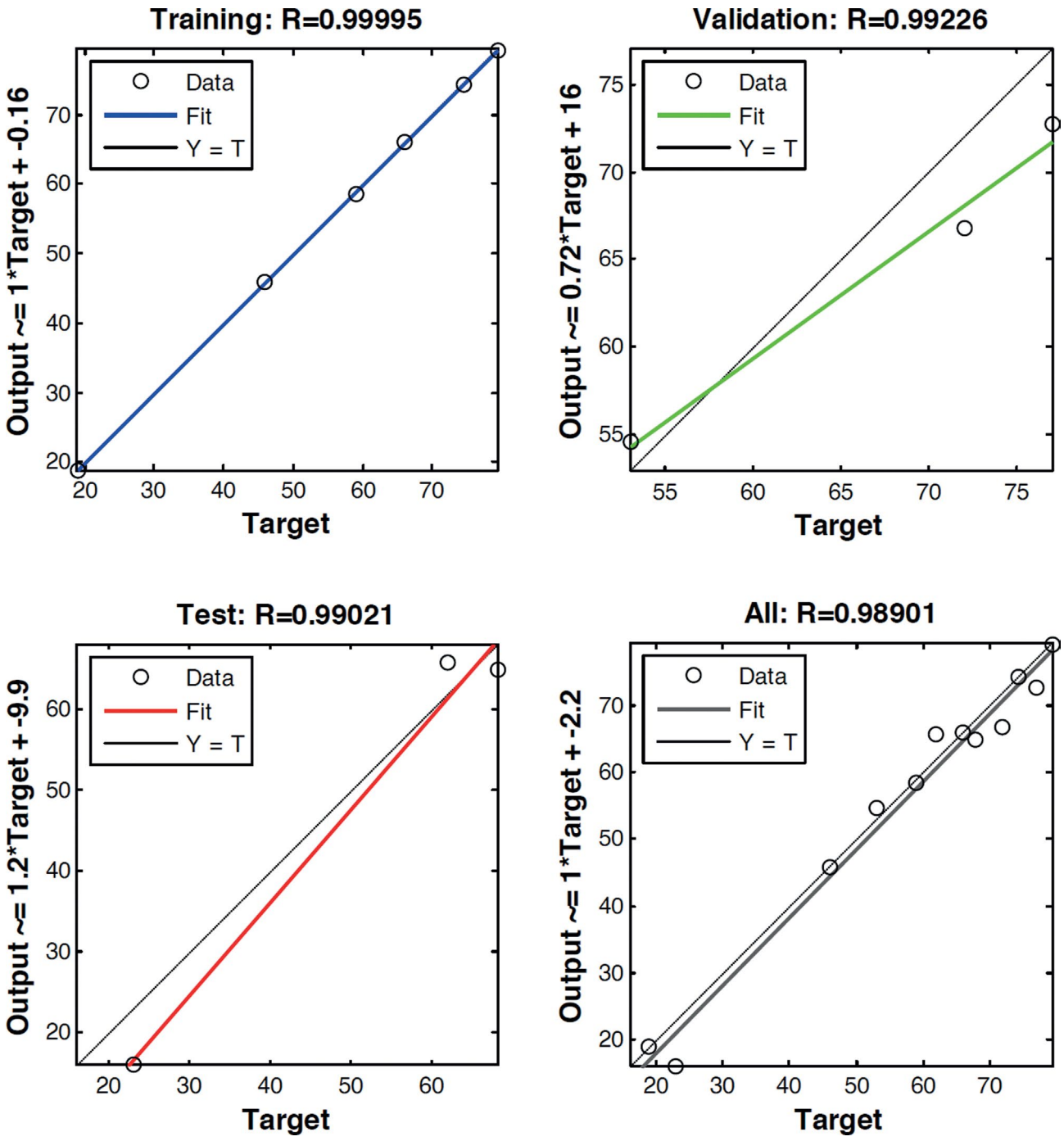

Figure 6. ANN regression plots for biochar derived from hazelnut shell.

performance was MSE 0.051 for trainig, 0.0015 for validation, and 0.0024 for testing steps; R^2 was 0.98 for all of the system (Figure 6).

ANN models were developed and trained with BR and SCG training algorithms, additionally. From this computation, LM has been recognized as the best training algorithm for Pb^{2+} removal efficiency predictions as seen from Figure 7.

As a result, LM algorithm provided better generalisation performance compared to the other algorithms due to its high R^2 value and showed closer results to the experimentally measured. Also it has been observed that an increase in the number of maximum epoch required for training step. When precision of predictive ability was measured for each training algorithm, their performances

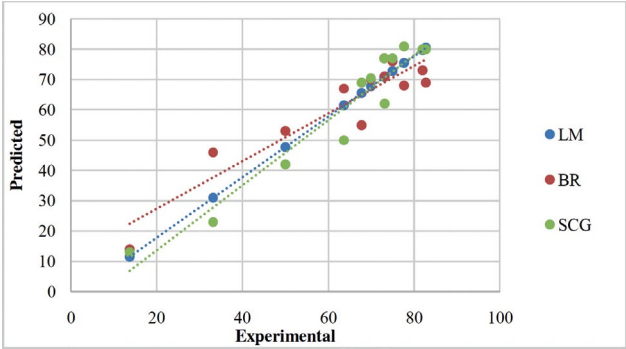

Figure 7. Comparative study results of LM, BR and SCG methods.

were determined as LM > BR > SCG for Pb^{2+} removal prediction.

CONCLUSIONS

In this study, removal efficiency of Pb^{2+} ions from an aqueous solution was predicted using ANN model which developed with the help of experimental adsorption data. ANN was successfully predicted the % removal by three-layered neural network. In experimental part pH, temperature, initial Pb^{2+} concentration, adsorbent amount, and mixing speed were selected as adsorption parameters, and used as inputs for ANN system. Three different training algorithm were used, and LM showed fast and better accuracy than two other algorithms. As a result of the ANN modeling, R^2 were found to be 97%, and 98% for biochars derived from walnut, and hazelnut shells, respectively. The relationship between the prediction results of the designed ANN model, and experimental adsorption data were organized, and ANN modeling tool has been shown that it can be used effectively to estimate the percentage removal value of Pb^{2+} from an aqueous solution.

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CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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