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Research Article

Modeling of copper removal from aqueous solutions by using carbon-based adsorbents derived from hazelnut and walnut shells by artificial neural network

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ABSTRACT

The aim of this study is to model the removal of copper from an aqueous solution using Artificial Neural Network (ANN). Two different carbon-based materials (biochar) obtained by pyrolysis of hazelnut and walnut shells were used as adsorbents in a batch adsorption system. The surface area of biochars with a porous structure obtained from hazelnut and walnut shells were 124.347 m^2/g and 256.931 m^2/g, respectively. In adsorption experiments, initial copper concentration, adsorbent amount, temperature, pH, contact time and mixing speed were the adsorption parameters considered for the system and defined as inputs in the modeling studies. Experimental results for removing copper ions were found by changing pH 2.5-5, initial heavy metal concentration 15-45 ppm, adsorbent amount 1-3 g/L, mixing speed 200-600 rpm and temperature between 25–45°C. The % copper removal, which is tried to be maximized during the modeling phase, was selected as the model estimation parameter and defined as output to the system. ANN training was done with the Levenberg–Marquardt (LM) feed-forward algorithm and the data was categorised into 50% training, 25% validation and 25% testing. The maximum epoch value was determined as 8 iterations. Correlation coefficient (R^2) values of the system were determined as 99% for education, validation and testing for the two different carbon-based adsorbents.

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INTRODUCTION

Water is one of the basic elements of life not only for humans but also for all living things that make up the ecosystem, and it is a natural resource with social and economic value. However, with the climate change resulting from global warming, drought, population growth, industrialization, agricultural activities and the increase in the amount of water consumed per person in daily life make it compulsory to protect limited water resources and prevent pollution [1]. Although serious measures have been taken in many countries in this regard, pollution in water resources is still shown as the most important cause of water shortage in the world. The main factors causing pollution in water resources are known as domestic and industrial waste water and the use of pesticides and fertilizers [2, 3]. There are many different types of pollutants that are mixed into water from these sources, and the environmental and health problems caused by these pollutants have reached increasingly important dimensions in the 21st century. In addition, economic losses caused by water pollution have caused the effect of this problem on societies to increase even more [4].

Due to rapid population growth, urbanization and industrial activities, large amounts of pollutants are released into water sources. Water is a natural resource with no alternative. Due to its importance for living life, water pollution is a serious problem that needs to be taken care of. Inorganic pollution is pollution caused by metals and its amount and diversity in the water sources increases every year. The difference of inorganic pollution from other pollution types is it causes toxic effects because of accumulation in living things [5-8].

Heavy metals, which have a significant share in water pollution, cause problems by being released to the environment in large amounts from various industrial sources. Lead, copper, cadmium, chrome, nickel, mercury are the most common heavy metals in wastewater [9, 10]. Copper has a wide range of usage areas due to good electrical and thermal conductivity, resistance to corrosion as well as shrinkable and malleable properties. Mining, printed circuits, metallurgical, fibre production, metal plating, paper and pulp, petroleum refining industries discharge big amounts of copper in effluent streams [11]. Removal of copper in wastewater treatment plants is quite important and it should be reduced to the allowable discharge limit before released to the environment [12]. If the copper limit is exceeded, it may cause serious toxicological effects such as vomiting, convulsions, cramps and even death [13]. Therefore, modeling studies are so important to predict copper concentrations in the effluent stream.

Adsorption technique, which is one of the wastewater treatment methods, has been widely used in the treatment of water in recent years due to its high efficiency and its ability to remove a wide variety of chemical contaminants, and it is a method with industrial application potential [3, 14]. In this purification technique, materials such as clay, activated alumina, silica gel, zeolite, activated carbon, carbon nanotubes, graphene, functionalized iron nanoparticles and many different nanocomposites are used as adsorbents [15-20]. Among these materials, activated carbon is most used in wastewater treatment [21]. However, its high cost limits its use. Due to this disadvantage, researchers have become increasingly important in recent years to use biochar, which can be an alternative to activated carbon and obtained from urban, industrial and agricultural wastes, to remove organic and inorganic pollution from soil and wastewater. Biochar is a different material from activated carbon. It is defined as a solid carbon-rich material obtained as a result of thermochemical transformations (pyrolysis) of biomass in an environment where oxygen is limited. The temperatures applied in activated carbon production are higher than the temperatures applied in biochar production. Therefore, in the production of activated carbon, biomass mostly loses its functional groups and becomes highly carbonized. In order to activate the carbonized surface and increase its porosity, it is also treated with steam or chemicals. In the production of biochar, biomass is carbonized at lower temperatures without the need for all these, and it is transformed into suitable material for different applications without destroying or even activating its surface functional groups [22].

Wastewater treatment plants (WWTPs) have great importance for the reduction and prevention of pollution in water resources. Many wastewater treatment facilities cannot operate at full efficiency due to design and operational errors. Operational control of the WWTPs quite difficult due to its complex structure. Failure of existing facilities to meet the discharge standards required by legislation or high operating costs brings the need for improvement and optimization. By modeling the process dynamics that occur during treatment, it is possible to monitor and control important parameters of the plant in a reliable and economical way [23]. On the other hand, traditional modeling methods do not provide efficient results due to the large number of variables and fixed parameters required. In recent years, adaptive and flexible modeling algorithms like ANN, fuzzy logic etc. have been used [24]. ANN is used for the prediction of responses in different disciplines due to its ability to employ learning algorithms and distinguish the relationships between the input and output for non-linear systems [25, 26]. It is suitable for many engineering problems [27]. The ANN is not programmed, it is trained through a learning process based on experimental data. Recently, it has been used as a tool to model adsorption processes. In particular, it is applied to model the adsorption data of various pollutants [28, 29].

WWTPs include highly complex operation which are depended on all process variables and removal mechanisms. Therefore, the ANN is powerful method to obtain correlation between input and output in complex nonlinear systems like WWTPs. Tümer and Edebali (2015) used ANN for modeling of Konya WWTP and ANN effectively predicted the performance of the process [30]. Nasr et al. (2012) used ANN for the prediction of El-Agamy WWTP performance. Plant performance was predicted with R^2 value reached up to 0.90 between the observed and predicted output variables [31]. Güçlü and Dursun (2010) used ANN to predict chemical oxygen demand (COD), suspended solids (SS) and mixed liquor suspended solids (MLSS) concentrations of the Ankara central WWTP [32]. Hassen and Asmare (2019) predicted wastewater quality for Habesha brewery WWTP by using ANN successfully with correlation coefficient between 0.9201 and 0.9692 [33]. Alsulaili and Refaie (2020) used the ANN prediction model to estimate biological oxygen demand (BOD). The developed prediction model for the influent BOD concentration attained a high accuracy with R^2 0.754 [34]. Viyajan and Mohan (2016) used ANN prediction for wastewater treatment plant performance in a dairy industry. Removal efficiencies of the plant for BOD, COD and total suspended solids (TSS) were 85%, 78% and 75%, respectively [35]. Jami et al. (2012) used ANN for Sewage Treatment Plant in Malaysia. Single input-single output results were compared with those of single input-multiple output for BOD, SS, and COD [36]. Bilgin Şimşek and Alkay (2020) used ANN for the prediction of effluent stream parameters of two different WWTPs in Kocaeli. R^2 values for training, validation and test were found as 0.94, 0.96 and 0.95, respectively [37].

Modeling studies are also very important to predict the copper concentration that in the effluent stream of WWTPs. The parameter prediction in the effluent stream brings chance to operate the WWTP economically and efficiently with less error to meet the requirement of discharge standards. There are few modeling studies in the literature to predict copper removal with artificial intellenge. Petroviç et al. (2014) used ANN and second order polynomial (SOP) regression model for copper removal prediction. Results showed that ANN performed with high prediction accuracy (0.980-0.986) in comparison to experimental results than SOP [38]. Bingöl et al. (2016) used ANN model for the evaluation of copper biosorption process using black cumin. A comparison between the model results and experimental data showed that the ANN model was able to predict the removal of copper using black cumin [39].

In this study, the effectiveness of two different carbon-based adsorbents produced from hazelnut shells and walnut shells by using pyrolysis technology, which is one of the thermochemical transformation processes, on the removal of copper ions from aqueous solution was tried to be modeled with ANN. This work is a continuation of the work done by Kaya et al. (2020) [40]. The % copper removals by two different adsorbents were predicted through ANN and the results compared with experimental values. In order to create the optimum network architecture, ANN parameter selections were made and the predictive ability of the network was evaluated by R^2 and mean square error (MSE) values.

MATERIALS AND METHOD

Preparation of Carbon-Based Adsorbents Derived from Hazelnut and Walnut Shells

Pyrolysis of hazelnut and walnut shells biomass was carried out at different temperatures in an electrically heated fixed bed pyrolysis reactor in 1 hour. During the process, the inert nitrogen gas flow rate was set to 100 mL/min and the heating rate to 20°C/min. The hazelnut shells was pyrolyzed at 500°C and the walnut shells at 700°C. Chemical bonds of biomasses were thermally degraded in an oxygen-free environment at high temperatures during the pyrolysis process, and carbon-based adsorbents that have high surface area were obtained [40].

Adsorption Studies

To determine the adsorption properties of carbon-based adsorbents produced by pyrolysis of hazelnut and walnut shells for removal of copper ions from aqueous solutions, batch adsorption experiments were carried out. Parameters affecting the adsorption process such as pH (2.5–5), adsorbent dosage (1–3 g/L), initial heavy metal concentration (15–45 ppm), 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 copper ion concentration. Based on the acquired values, the adsorption yield was calculated by using Eqn. 1.

$$Copper\,\mathrm{Re}moval\,(\%) = \frac{C_0 - C_e}{C_0} \times 100 \tag{1}$$

where C_0 and C_e are the initial and final copper concentrations in the solution phase (ppm), respectively. 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% [40].

ANN MODELING

Model Theory

Today, computers can both make decisions about events and can learn the relationships between events [41]. Mathematically formulation of unsolved and unsolvable problems can be solved by using computers and heuristic

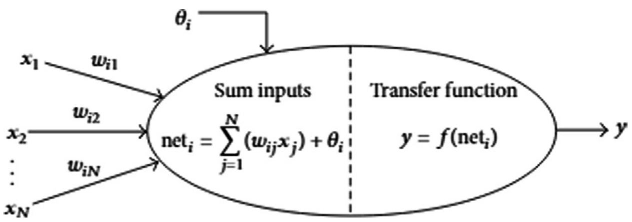

Figure 1. ANN working scheme.

methods [42]. ANN is parallel and distributed information processing structures that are inspired by the human brain and are connected to each other through weighted correlations, each consisting of processing elements (Fig. 1). Artificial nerve cells coming together in parallel connection to each other connected by their weight and ANN is trained by correcting the values of these connecting weights between nerve cells (Eqn. 2) [43].

$$y = F\left(\sum_{i=0}^{n}(u_i w_i) + w_0\right) \tag{2}$$

where y is output signal value, F is activation function, w is weight value of the connection number i, u is the value of input signal number i, n is the number of input signals and i is bias term [44].

ANN Parameters

ANN consists of three layers; input layer, hidden layer (s) and exit layer as shown in Fig. 2.

Inputs: Inputs layer is created by user and chosen from the data sets which are the independent variables and affects the output directly.

Output: Inputs that are collected by the summation function and evaluated in the activation function then produce output or response.

Summation Function: Generally, data come from more than one cell to an artificial nerve cell. This function is used to calculate the net input from the incoming data.

Activation Function: After input values are summed with the addition function it must process the information and generate a response. The function used for this calculation is called the activation function. Linear function, sigmoid function, hyperbolic tangent function, sine function, digit function are types of activation function.

Hidden Layer: It is the layer where the input units are processed. It can be a single layer or composed of more than one layer as well. The number of neurons in the hidden layer are determined by trial and error. Number is important for a good solution.

Data Division: Data are divided into 3 parts as training, testing, and validation subsets and there is no systematic way for optimal division.

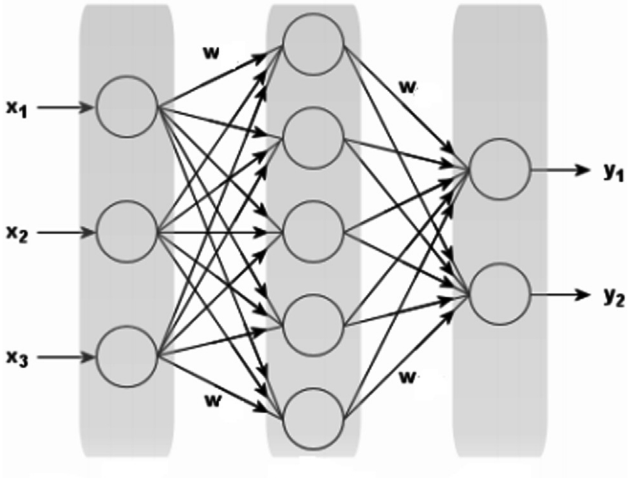

Figure 2. ANN Layer Structure [45].

Model Success Evaluation Criteria

R^2 and MSE were used for measuring the accuracy of prediction results for ANN. MSE is the mean square difference between outputs and targets (Eqn. 3). For MSE, lower values are better. R^2 measures the correlation between outputs and targets (Eqn. 4). If R^2 is 1 it means a close relationship, 0 is a random relationship [46].

$$MSE = \frac{1}{n}\sum_{i=1}^{n}(Y_{observed} - Y_{predicted})^2 \tag{3}$$

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(Y_{observed} - Y_{predicted})^2}{\sum_{i=1}^{n}(Y_{observed} - Y_{mean})^2} \tag{4}$$

RESULTS AND DISCUSSION

The carbon-based adsorbents (biochars) used in this study were obtained by pyrolysis process. Since the carbonization temperature changes the surface area of the biochar and consequently the its adsorption capacity, pyrolysis process was carried out separately at different carbonization temperatures in the range of 400–700°C for both hazelnut and walnut shells in our previous study. Experimental results have shown that the high temperature had an adverse effect on the surface area of the biochar obtained from hazelnut shells and was reached the highest Brunauer-Emmett-Teller (BET) surface area value at 500°C. On the contrary, the high temperature had an favorably effect on the surface area of the biochar obtained from walnut shells and the surface area increased considerably by increasing the pyrolysis temperature from 400°C to 700°C. Therefore, while the hazelnut shells were pyrolyzed at 500^{o}C in this study,

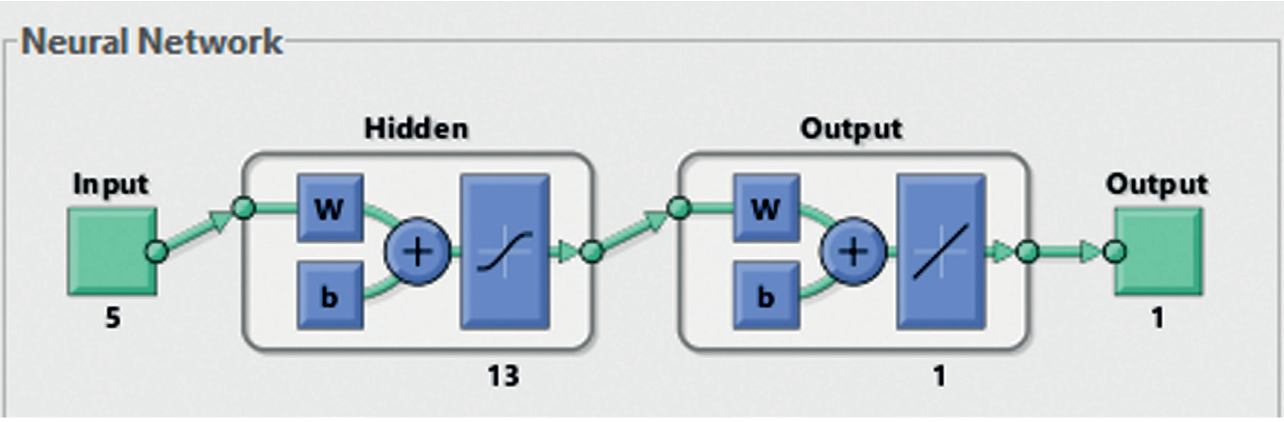

Figure 3. Structure of ANN for carbon-based adsorbent derived from walnut shells.

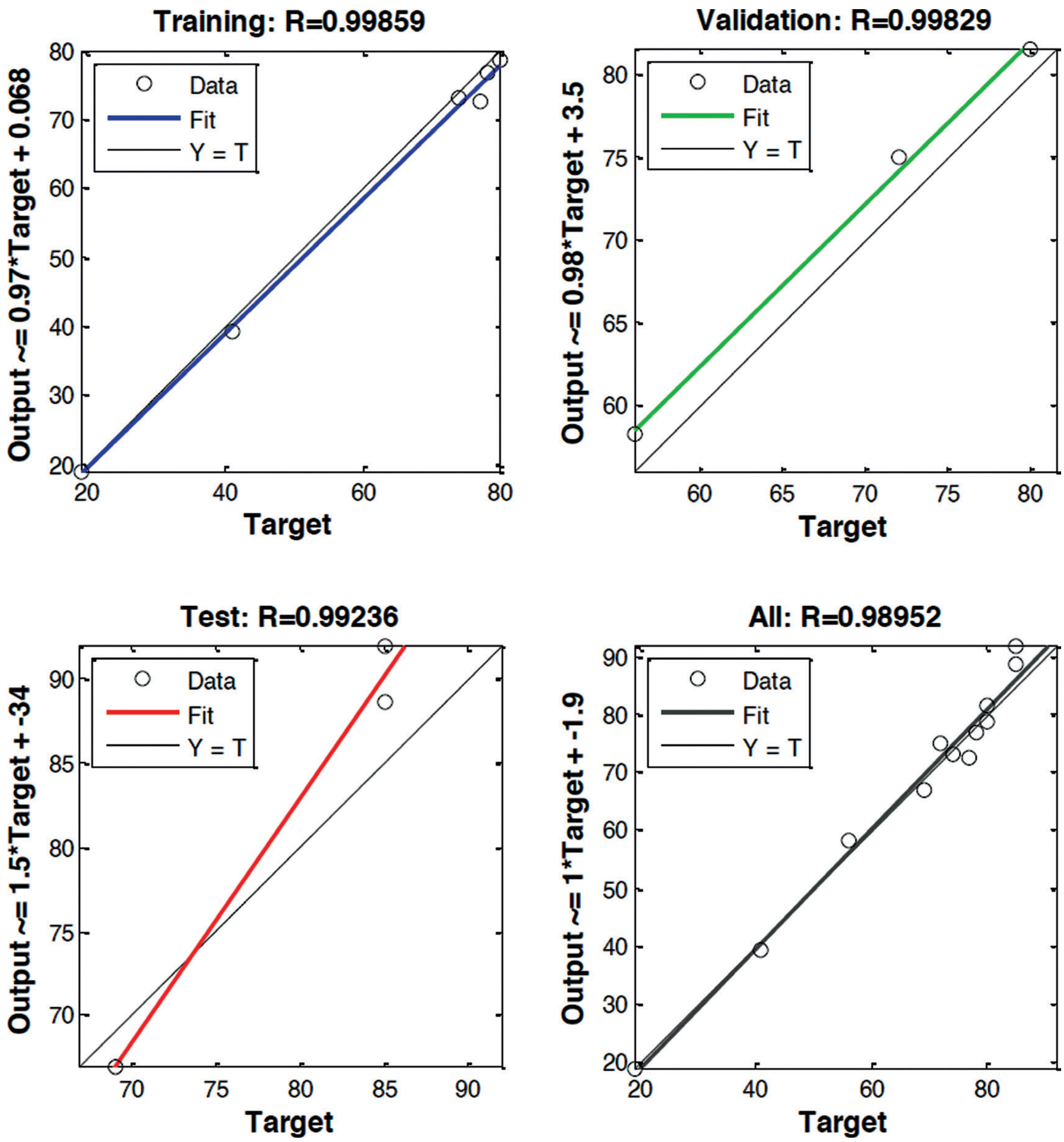

Figure 4. Network regression graph in Matlab for the effectiveness of the adsorbent derived from walnut shells.

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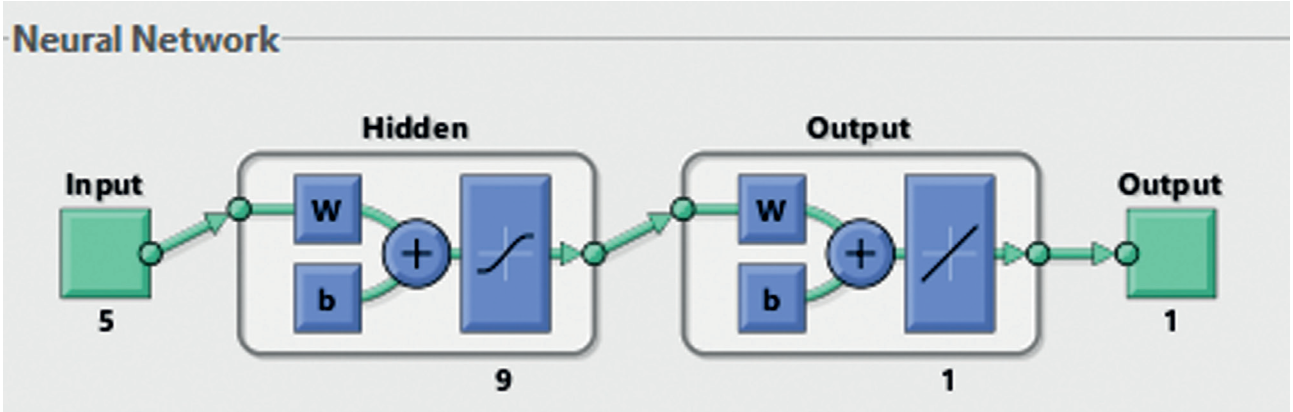

Figure 5. Structure of ANN for carbon-based adsorbent derived from hazelnut shells.

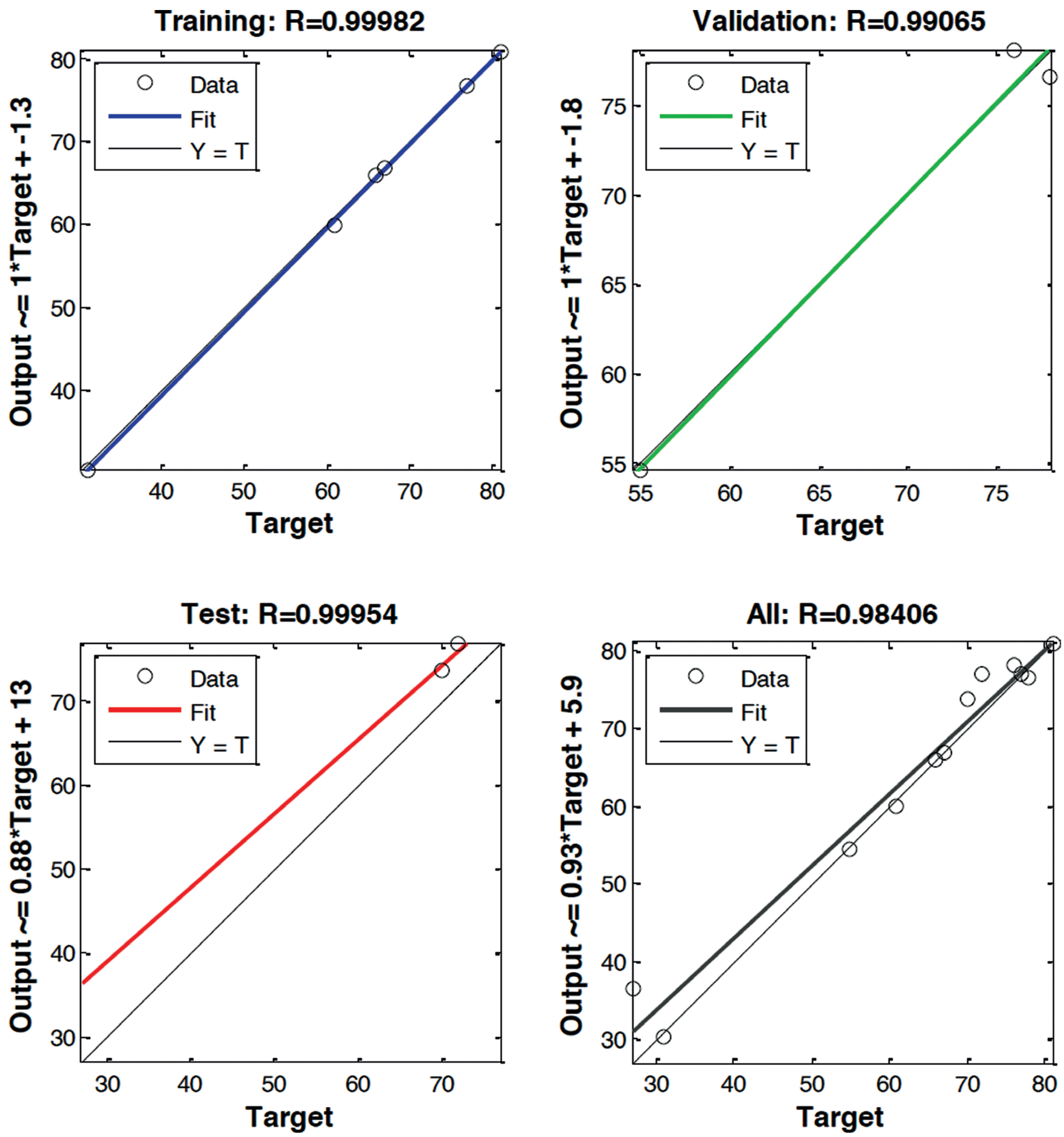

Figure 6. Network regression graph in Matlab for the effectiveness of the adsorbent derived from hazelnut shells.

walnut shells were pyrolyzed at 700°C. The BET surface area of the biochars which were having a porous structure, obtained from hazelnut and walnut shells were determined as 124.347 m^2/g and 256.931 m^2/g, respectively [40].

In this study, the % removal value estimation in copper removal was made with ANN with Neural Fitting toolbox in Matlab R2017a and the model estimation results obtained were compared with the experimental results. Different ANN architectures have been tried to find the most suitable ANN structure. The Levenberg-Marquardt algorithm with the feed-forward algorithm was used. Levenberg-Marquardt (LM) algorithm is a hybrid technique that uses both Gauss–Newton and steepest descent approaches together to converge to an optimal solution. LM algorithm is the fastest algorithm and ensures the best convergence towards a minimum error [47]. According to this algorithm, firstly inputs were introduced to the network then entered with weights by multiplying each cell in the hidden layer transmitted and then net input values are calculated.

In the ANN configuration, the input variables were selected as the adsorption parameters; initial heavy metal concentration, adsorbent amount, temperature, pH, contact time and mixing speed, while the % copper removal value was chosen as the output variable. Tangent sigmoid (tansig) was used in the hidden layer as a transfer function due to its nonlinear structure. Many attempts were made to determine the number of neurons in the hidden layer and the appropriate number of neurons were determined.

When the experimental data set was firstly divided into 70-15-15% and 60-20-20%, the R^2 value was consistently taken as 1 and it was seen that different data should be used during the training phase. When the data were divided into 50-25-25%, this problem was solved.

After the training, the ANN model was tested and the results were evaluated considering the R^2 and MSE values. The R^2 value being close to 1 at low MSE value shows the model performance is exceptionally good.

ANN for Adsorbent Derived from Walnut Shells

After the inputs and output were determined, the number of neurons in hidden layer was found 13 by trial and error (Fig. 3). The maximum epoch number was found 8 iterations.

The performance criteria R^2 value was found 0.99 for training, validation and test steps as seen from Fig. 4. MSE values were found 0.43, 0.55 and 0.21 for training, validation and test, respectively.

ANN for Adsorbent Derived from Hazelnut Shells

After the inputs and output were determined, the number of neurons in hidden layer was found 9 by trial and error (Fig. 5) . The maximum epoch number was found 8 iterations. The performance criteria R^2 value was found 0.99 for training, validation and test steps as seen from Fig. 6. MSE values were found 0.27, 0.22 and 0.43 for training, validation and test, respectively.

The plot of experimental results (test data), which were obtained from batch adsorption experiments, vs predicted ones showed that the points were well distributed around the X=Y line and showed that the ANN model had higher prediction accuracy between actual and predicted data for removing copper ions from aqueous solution. As a result, the ANN model showed good performance in prediction of removal of copper, which is one of the heavy metals from aqueous solution.

The results obtained from this study are consistent with the articles in which modeling results related to heavy metal removal are reported in the literature. When other heavy metal removal modeling studies are investigated; Yıldız (2017) used ANN to predict adsorption efficiency of peanut shells for the removal of Zn(II) ions from aqueous solutions. The experimental results and the predicted results by the model with the ANN were found to be highly compatible with each other [48]. Olajubu et al. (2017) compared predicted removal quantity of lead ions and cadmium ions by ANN with the result of the experimental work in the laboratory and ANN showed very high prediction accuracy [49]. Allahkarami et al. (2017) used ANN and nonlinear multi-variable regression (NLMR) models to predict Co(II) and Ni(II) ions removal. Results showed that ANN was better than NLMR with higher R^2 and lower MSE value [50]. Therefore it can be said that, ANN model is one of the powerful tools for predicting heavy metal removal.

CONCLUSIONS

Since the adsorption technique can remove a wide variety of inorganic and organic origin contaminants from water, it is accepted as a universal method today and is preferred more than traditional water treatment techniques in practice. Due to the high metal adsorption capacity of adsorbents prepared from different types of biomass, carbon-based materials derived from biomass especially agricultural wastes are generally used as adsorbents in recent years. The aim of this study is to predict the % copper removal by using the ANN model for carbon-based adsorbents derived from hazelnut and walnut shells which are abundant in many countries. The results obtained from this study are thought to be enlightening in terms of determining the theoretical adsorption efficiency in the removal of copper ions from aqueous solution in cases where purification studies cannot be performed in the laboratory.

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

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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