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APPLICATION OF ARTIFICIAL NEURAL NETWORK (ANN) FOR PREDICTION DIAMETER OF SILVER NANOPARTICLES BIOSYNTHESIZED IN *CURCUMA LONGA* EXTRACT

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In this study silver nanoparticles (Ag-NPs) are biosynthesized from silver nitrate aqueous solution through a simple and eco-friendly route using *Curcuma longa* (*C. longa*) tuber powder extracts which acted as a reductant and stabilizer simultaneously. Characterizations of nanoparticles are done using X-ray diffraction (XRD) and transmission electron microscopy (TEM). We present an artificial neural network (ANN) approach is used to model the size of Ag-NPs as a function of the volume of *C. Longa* extraction, temperature of reaction, stirring time and volume of $AgNO_3$. The suitable ANN model is found to be a network with two layers that first layer has 10 neurons and second layer has 1 neuron. This model is capable for predicting the size of Ag-NPs synthesized by green method for a wide range of conditions with a mean absolute error of less than 0.01 and a regression of about 0.99. Based on the presented model it is possible to design an effective green method for obtain Ag-NPs, while minimum received materials are used and minimum size of Ag-NPs will be obtained. Also simulation of the process is performed using ANN media. According to the model's results, the volume of *C. Longa* extraction, temperature of reaction, and volume of $AgNO_3$ about 18 mL, 30 °C and 2 mL are chosen as the optimum size of Ag-NPs, respectively. Results obtained reveal the reliability and good predicatively of neural network model for the prediction of the size of Ag-NPs in green method.

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Keywords: Artificial neural network, curcuma longa, silver nanoparticles, levenberg–marquardt, Green method

1. Introduction

The "green synthesis" of metal nanoparticles receives greater attention due to their unusual optical, chemical, photo-chemical and electronic properties [1]. Metal nanoparticles especially the noble metals; have mainly been studied because of their strong optical absorption in the visible region caused by the collective excitation of the free electron gas [2].

Among noble metal nanoparticles, silver nanoparticles have wide area of interest as they have large number of applications such as in non-linear optics, spectrally selective coating for solar energy absorption, biolabelling, intercalation materials for electrical batteries as optical receptors, catalyst in chemical reactions and as antibacterial capacities [3]. Silver nanoparticles (Ag-NPs) have definite properties. This may perhaps have numerous applications in the fields of dentistry, clothing, catalysis, mirrors, optics, photography, electronics and food industries [4].

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Because of such broad variety of applications, wide ranges of different preparation methods have been developed. However, the developing methods of Ag-NPs preparation, must give preference to control size of Ag-NPs. Therefore, nanosilver with small particle size and devoid of aggregation between particles is favorable in this purpose.

There are several ways to reduce Ag^+ for instance, application of physical methods using of γ-rays, UV-irradiation, ultrasonic irradiation, microwave irradiation, heating and electrochemical reduction [5–7], application of reducing chemicals, such as hydrazine, sodium borohydride [8–12], polyethylene glycerol [13], N,N-dimethyl formamide [14], glucose [15], ethylene glycol [16], formaldehyde [17], sodium in liquid ammonia, etc [18].

However, there is still need for a more economic, commercially viable as well environmentally green synthesis route to synthesize Ag-NPs. The green synthesis of Ag-NPs involves three main steps, which must be evaluated based on green chemistry perspectives, including selection of solvent medium, reducing agent and nontoxic stabilizers for Ag-NPs [3]. The biosynthesis of nanoparticles, which represents a connection between biotechnology and nanotechnology, has received increasing consideration due to the growing need to develop environmentally friendly technologies for material syntheses. The search for appropriate biomaterials for the biosynthesis of nanoparticles continues through many different synthetic methods. The biosynthetic method using plant extracts has received more attention than chemical and physical methods and even than the use of microbes. The method is suitable for nanoscale metal synthesis due to the absence of any requirement to maintain an aseptic environment [19]. The possibility of using plant materials for the synthesis of nanoscale metals was reported initially by *Gardea-Torresdey* et al [20]. Later, the bioreduction of various metals to nanosize materials of various shapes, capable of meeting the requirements of diverse industrial applications, was extensively studied [21]. In continuation, we have demonstrated the prospect of using *Vitex Negundo L.* leaf and *Callicarpa manigayi* stem bark methanolic extracts and Curcuma longa tuber powder water extract for the synthesis of the Ag-NPs at ambient conditions, without any additive protecting nanoparticles from aggregating, template shaping nanoparticles or accelerants [22–24, 19].

Artificial neural network (ANN) is a powerful and efficient simulation tool for managing the engineering regression-based processes and also for classification of problems specially, when the involving parameters and their corresponding relationships are very complicated, non-linear and multi-dimensional [25]. The artificial neural networks (ANNs) allow one to estimate relationships between one or several input variables called independent variables or descriptors and one or several output variables called dependent variables or responses. Information in an ANN is distributed among multiple cells (nodes) and connections between the cells (weights) [26]. Also can say ANN is a computer program capable of learning from examples through iteration, without requiring a prior knowledge of the relationships between process parameters. Major benefits in using ANN are excellent management of uncertainties, noisy data and non-linear relationships [27]. Neural network modelling has generated increasing acceptance and is an interesting method in the estimation, prediction and control of bioprocesses [28].

Lately, ANNs have been used to study a wide variety of chemical problems such as spectral analysis [26], prediction of dielectric constants [29], and mass spectral search [30], and also in recent years, ANN have been introduced in nanotechnology applications as techniques to model data showing non-linear relationships and or estimation of particle size in variety nano particle samples [30–32]. The main aim of this investigation is using artificial neural network for prediction of size of Ag-NPs prepared by green method. In this study, an artificial neural network method using the back-propagation algorithm is proposed for the prediction of size of nano silver under different operational conditions.

2. Experimental Section

2.1 Materials

The *C. longa* tuber is purchased from a local market in Malaysia. $AgNO_3$ (99.98%) is used as a silver precursor, and is provided by Merck (Frankfurter, Germany). HNO_3 (70%) and HCl

(37%) are also obtained from Sigma-Aldrich (St Louis, MO, USA). All reagents in this effort are analytical grade and are used as received without further purification. All solutions are freshly prepared using double distilled water and kept in the dark to avoid any photochemical reactions. All glassware used in experimental procedures are cleaned in a fresh solution of HNO_3/HCl (3:1, v/v), washed thoroughly with double distilled water, and dried before use.

2.2 Extraction Preparation

The *C. longa* tubers are washed to remove the adhering mud particles and possible impurities. Later it is dried under sunlight for a week to completely remove the moisture. The tubers are cut into small pieces, powdered in a mixer and then sieved using a 20 mesh sieve to get uniform size range. The final sieved powder is used for all further studies. For the production of extract, 0.1 g of *C. longa* tuber powder is added to a 100 mL Erlenmeyer flask with 20 mL sterile distilled water and then mix for 4 h in room temperature.

2.3 Synthesis of Ag-NPs in *C. longa* Suspension

Briefly, water extract of *C. longa* tubers powder are added to the different volumes of Ag NO_3 (1×10^{-3} M) then mixed reactants at different stirring time and reaction temperature (Table 1). Ag-NPs are gradually obtained during the incubation period. Throughout the reduction process, solutions are kept in the dark to avoid any photochemical reactions. The solutions components are purged with nitrogen gas prior to use. Subsequently, reduction proceeded in the presence of nitrogen to eliminate oxygen. The obtained colloidal suspensions of Ag-NPs in *C. longa* are then centrifuged at 15000 rpm for 20 min and washed four times to remove silver ion residue. The precipitate nanoparticles are then dried overnight at 30 °C under vacuum overnight to obtain the Ag/*C. longa*.

2.4 Characterization Methods and Instruments

The synthesized Ag/*C. longa* suspensions are characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM). The structures of the Ag-NPs produced are examined by X-ray diffraction (XRD-6000, Shimadzu, Japan). The XRD patterns are recorded at a scan speed of 4°/min. TEM observations are carried out on a H-7100 electron microscope (Hitachi, Tokyo, Japan), and the particle size distributions are determined using the UTHSCSA Image Tool version 3.00 program.

2.5 Data Set

In Table 1 is presented the experimental data used for the training of the ANN model. The database is randomly divided into three sets: training, validation and testing data. Training category is specifically applied to adjust the network weights and errors in the each iteration. Validation category is required to modify and optimize the ANN architecture (activation function, training function and number of hidden layers), and adjust the number of neurons for hidden layer. A testing date sets is supposed to be the new data that evaluate the effectiveness and efficiency of the trained network [33]. Also the predicted particle size is compared to the observed particle size and the difference between the predicted and observed size is stated as particle size error based on the difference between these two values.

Table 1. Experimental values (training, validation and testing data set), actual and model predicated of size of Ag-NPs.

No.	Volume of *C. longa* Extract (mL)	Temperature (°C)	Stirring time (h)	Volume of $AgNO_3$ (mL)	Ag-NPs Particle Size (nm) (Actual)	Ag-NPs Particle Size (nm) (Predict)	Particle Size Error Actual-Predict
				Training Set			
1	20	40	48	5	5.52	5.5243	-0.0043
2	20	50	48	10	6.08	6.0824	-0.0024
3	20	70	24	20	7.35	7.3293	0.0207
4	10	25	24	1	8.18	8.4098	-0.2298
5	10	30	24	2	8.41	8.4803	-0.0703
6	10	60	12	15	9.35	9.3185	0.0315
7	10	70	12	20	9.78	9.7753	0.0047
8	5	25	24	1	10.46	10.5446	-0.0846
9	5	40	12	5	10.86	10.9758	-0.1158
10	5	70	6	20	11.82	11.8955	-0.0755
11	2	25	6	1	12.37	12.6080	-0.2380
12	2	30	6	2	12.49	12.6792	-0.1892
13	2	40	3	5	12.73	12.7863	-0.0563
14	2	50	3	10	12.96	12.9687	-0.0087
15	2	70	3	20	13.78	13.9291	-0.1491
16	1	25	3	1	14.36	14.5713	-0.2113
17	1	30	1	2	14.55	14.6982	-0.1482
18	1	40	1	5	14.65	14.6805	-0.0305
19	1	50	1	10	14.85	14.8393	0.0107
20	1	70	1	20	15.32	15.5211	-0.2011
				Validation set			
21	20	30	48	2	5.18	5.5761	-0.3961
22	10	50	24	10	9.11	9.2364	-0.1264
23	5	60	6	15	11.69	10.8051	0.8849
24	2	60	3	15	13.47	13.0482	0.4218
25	1	60	1	15	14.93	15.0683	-0.1383
				Test set			
26	20	25	24	1	4.90	5.3281	-0.4181
27	20	60	48	15	6.67	6.7278	-0.0578
28	10	40	24	5	8.85	9.1156	-0.2656
29	5	30	12	2	10.74	10.6969	0.0431
30	5	50	6	10	11.23	11.2346	-0.0046

2.6 ANN Description

In Fig. 1. is shown a diagram of a typical MLP neural network with one hidden layer structure of proposed ANN.

The input to the node l in the hidden layer is given by:

$$\partial_l = \sum_{i=1}^{q}(x_i w_{il}) + \theta_l \qquad l = 1,2,\dots,s \tag{1}$$

Each neuron consists of a transfer function expressing an internal activation level. The output from a neuron is determined by transforming its input using a suitable transfer function [34]. Generally, the transfer functions for function approximation (regression) are sigmoidal function, hyperbolic tangent and linear function [35]. The most popular transfer function for a

nonlinear relationship is the sigmoidal function [36,37]. The output from l th neuron of the hidden layer is given by:

$$O_l = f(\partial_l) \qquad l=1, 2,\ldots, s \tag{2}$$

In Equations (1) and (2), q is the number of neurons in the input layer, s is the number of neurons in the hidden layer, θ_l is the bias term, w is the weighting factor and f is the activation function of the hidden layer such as hyperbolic-tangent transfer function [38]. The output of the jth neuron in the output layer is given by:

$$Y_j = \sum_{i=1}^{s}(O_i w_{ij}) + b_j \qquad j = 1,2, \ldots, m \tag{3}$$

Where w is the weighting factor, b is the bias term and m is the number of neurons in the output layers.

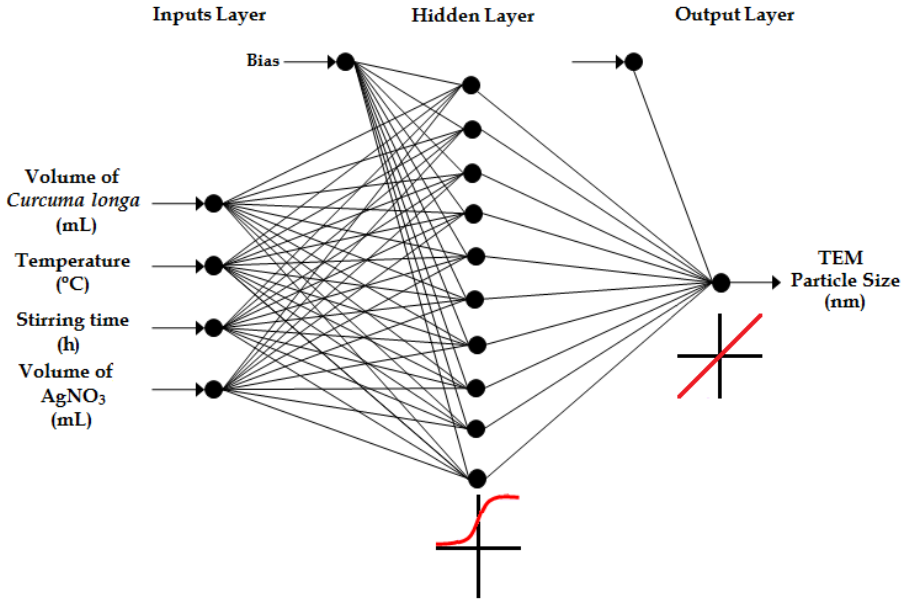

Fig. 1. Schematic diagram of neural network model.

The values of the interconnection weights are determined by the training or learning process using a set of data. The aim is to find the value of the weight that minimizes the error [35]. A general measure for evaluation of predicts ability ANN models is the Mean Square Error (MSE):

$$MSE = \frac{1}{r}\sum_{i=1}^{r}(x_{pi} - x_{ai})^2 \tag{4}$$

Where r is the number of points, x_{pi} is the predicted value obtained from the neural network model, x_{ai} is the actual value. The coefficient of determination, R, of the linear regression line between the predicted values from the neural network model and the desired output was also used as a measure of performance [27]. The closer the R value is to 1, the better the model fits to the actual data [39].

$$R = \left(1 - \frac{\sum_{i=1}^{r}(x_{pi}-x_{ai})^2}{\sum_{i=1}^{r}(x_{ai}-x_m)^2}\right)^{1/2} \tag{5}$$

Where r is the number of points, x_{pi} is the predicted value obtained from the neural network model, x_{ai} is the actual value, and x_m is the average of the actual values. The network having minimum MSE, maximum R is considered as the best neural network model [40].

3. Results and discussion

3.1 Powder X-ray Diffraction

The Fig. 2. shows the X-ray diffraction (XRD) patterns of vacuum-dried Ag-NPs synthesized using 20 mL of *C. longa* in 1 mL of $AgNO_3$ that mixed for 24 h at 25 °C, respectively. The XRD patterns of Ag/*C. longa* indicated that the structure of Ag-NPs is face-centered cubic (fcc). In addition, all the Ag-NPs had a similar diffraction profile and XRD peaks at 2θ of 38.18°, 44.25°, 64.72°, and 77.40° could be attributed to the 111, 200, 220 and 311 crystallographic planes of the face-centered cubic (fcc) silver crystals, respectively [32]. The XRD pattern thus clearly illustrated that the Ag-NPs formed in this study are crystalline in nature. The main crystalline phase is silver and there is no obvious other phases as impurities are found in the XRD patterns (Ag XRD Ref. No. 01-087-0719).

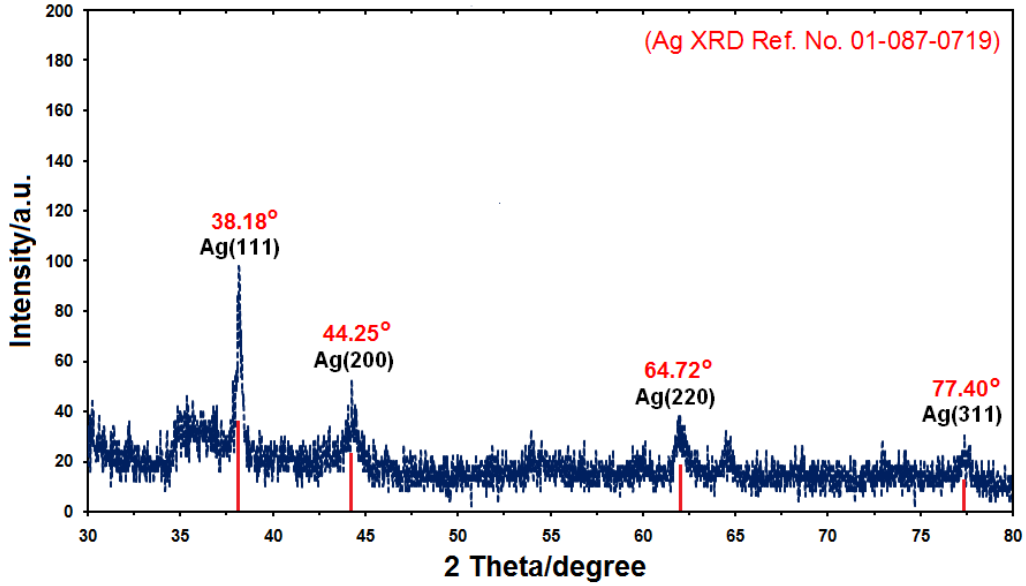

Fig. 2. XRD patterns of Ag-NPs synthesized in C. Longa for determination of Ag-NPs.

3.2 Morphologies Study

TEM images and their corresponding particle size distributions of Ag/*C. longa* suspensions with the different volumes of *C. longa* extract (5, 10 and 20 mL) are shown in Fig. 3. For the TEM study, a drop of the Ag-NPs solutions synthesized by treating $AgNO_3$ aqueous solution with different volumes of *C. longa* were deposited on to a TEM copper grid. After drying, the grids are imaged using TEM. The TEM images and their size distributions revealed that, the mean diameters and standard deviation of Ag-NPs are about 10.46±5.58, 8.18±3.53 and 4.90±1.42 nm for 5, 10 and 20 mL (a–c) of *C. longa* extract at 25 °C, and after 24 h of stirring reaction time.

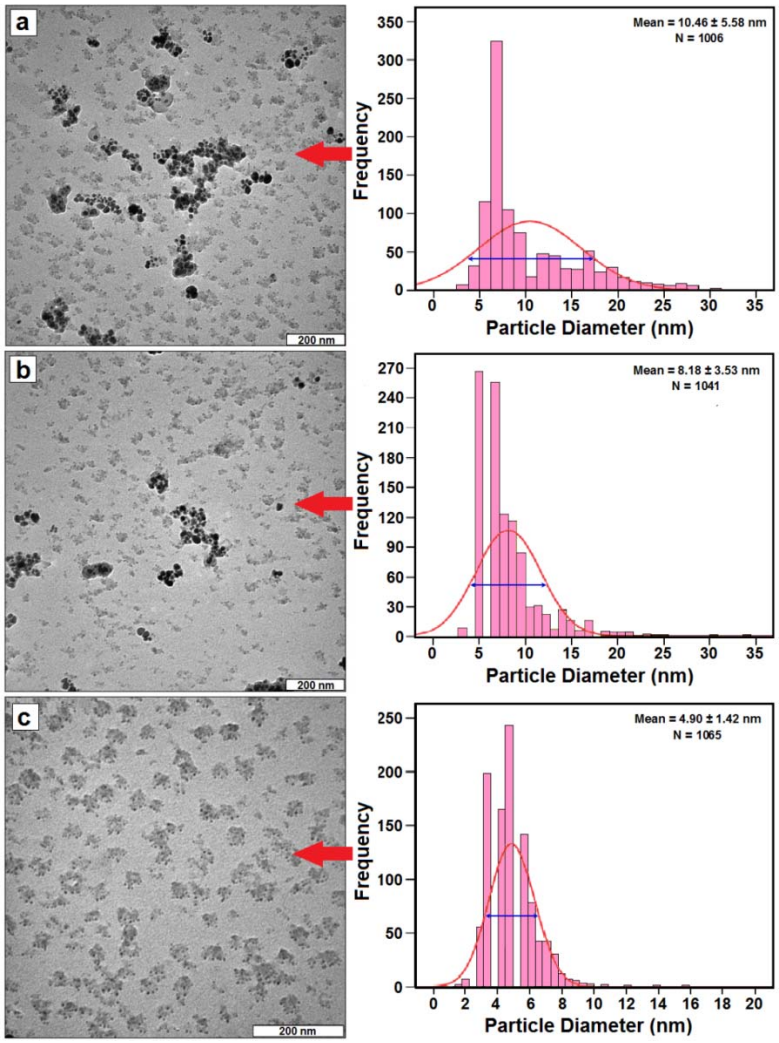

Fig. 3. TEM images and corresponding size distributions of Ag-NPs in C. longa (5, 10 and 20 mL) under 25 °C and after 24 h of stirring reaction time (a–c).

Fig. 3. (a-c) shows the Ag-NPs are surrounded by the extract of *C. longa*. The dark points in these figures represent the large scale distribution of Ag-NPs. The Ag-NPs surrounded by *C. longa* extract are shown by TEM. The numbers of Ag-NPs counted for TEM images are around 1006, 1041 and 1065 for 5, 10 and 20 mL for *C. longa* extract.

3.3 ANN Modeling

The volume of plant extract, temperature, stirring time, and volume of $AgNO_3$ are used as inputs to the network. The output of the model was particle size of nano particle. Numbers of hidden layers and number of nodes in each hidden layer plays a significant part in the network procedure. It does not have any specific situation or rule but is fundamentally depends upon the analyzer's experience and problem's nature [41]. Normalizing all the input data to values between 0 and 1 is the first step of the calculation before using the neural networks. The last step is the renormalization of outputs. Former job is for sensitivity increscent and accuracy of network, and the goal of the latter is to calculate the actual desired values and real simulation error [33]. Also optimization process of ANN contains selection of the parameters related to each function known as the bias and weight values of the transfer-function of each neuron in order to maximize the regression (R) and minimize the error of MSE of an observing dataset. The first analysis is the optimal number of hidden neurons for the model. Therefore the number of hidden neurons varied from 1 to 20 in accordance with trustworthy obtained results in training step. Also in hidden layer is picked and chosen a hyperbolic-tangent transfer function. The linear activation function is similarly applied as the output layer activation function. In this paper, best architecture for the

ANN model is selected a two layer feed forward network that input neurons are attached to a layer of 10 hidden neurons, which are connected to the output neurons.

Therefore using the ANN analysis, the optimal configuration of the ANN model is found to be 4-10-1. The suggested network is trained with Levenberg–Marquardt (LM) back-propagation algorithm. The back-propagation algorithm is utilized in model training. The back-propagation algorithm uses the supervised training technique where the network weights and biases are initialized randomly at the beginning of the training phase. For a given set of inputs to the network, the response to each neuron in the output layer is calculated and compared with the corresponding desired output response. The errors associated with desired output response are adjusted in the way that reduces these errors in each neuron from the output to the input layer. The error minimization process is achieved using gradient descent rule [42]. The LM algorithm [43], is an approximation to the Newton method used also for training artificial neural network. The Newton method approximates the error of the network with a second order expression, which contrasts to the back-propagation algorithm that does it with a first order expression. Therefore LM back propagation is a network training function that updates weight and bias values according to LM optimization [44]. Table 2 shows the values of connection weights (parameters of the model) for the complete ANN model trained on the whole datasets. This information allows other researchers to compare present ANN models with their own experimental data.

Table 2. Values of connection weights (parameters of the model) for the completed ANN model.

	Node 1	**Node 2**	**Node 3**	**Node 4**	**Node 5**	**Node 6**	**Node 7**	**Node 8**	**Node 9**	**Node 10**	**Bias 2**
Input 1	-1.6823	-2.6397	2.4590	-4.3611	2.4539	0.0525	-2.4357	1.0752	-0.0493	1.1877	
Input 2	0.0242	-0.3755	-1.0036	-0.5187	1.6785	-0.9912	-1.5892	-1.2649	-0.6639	1.0005	
Input 3	0.7343	-0.2823	0.0627	0.6445	1.3309	-1.5541	0.2129	0.0318	-0.7304	-1.5136	
Input 4	1.1828	0.5438	-2.3693	-0.7728	-0.9676	0.0793	-0.6098	2.0588	2.4077	-1.2244	
Bias 1	2.8607	-4.6558	-0.4913	-1.6012	0.0745	0.2393	-0.5033	2.3933	1.7281	2.5784	
Output	0.1594	0.8554	0.4302	-0.3419	-0.6868	-0.6584	-0.4632	-0.3914	0.0194	0.3367	0.3586

The performances of ANN model on training, validation and testing data sets are evaluated by MSE and R. These resulting values are reported in Table 3 and this results show that the predictive accuracy of the model is high.

Table 3. The performances of ANN model on training, validation and testing data sets.

	Train	**Validation**	**Test**
MSE	0.0155	0.2305	0.0501
R	0.9996	0.9920	0.9987

In Fig. 4. is also shown the scatter diagram of predicted values in comparison with actual values. It shows that the model prediction fits well with the experimental observations.

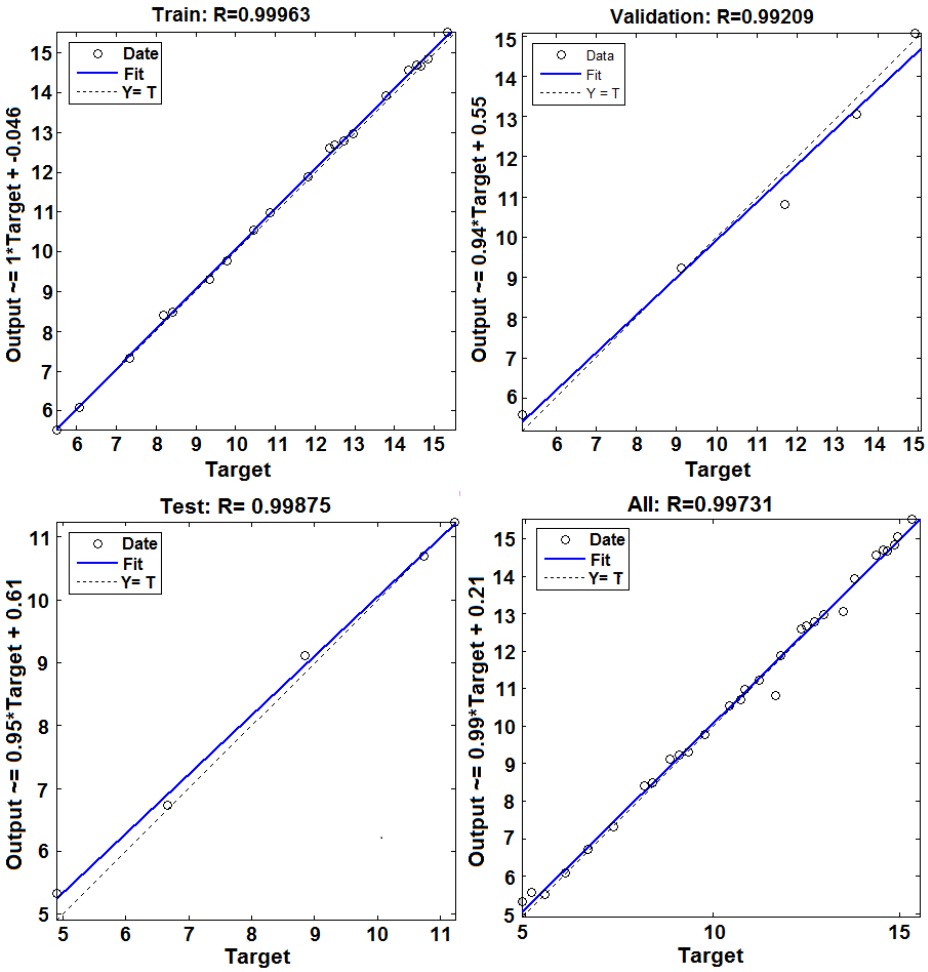

Fig. 4. The scatter plots of ANN model predicted values in comparison with actual values for training, validation, testing and all data sets.

In Fig. 5. is shown the errors histogram of train, validation and test sets. These results show errors three data sets is less than 0.86.

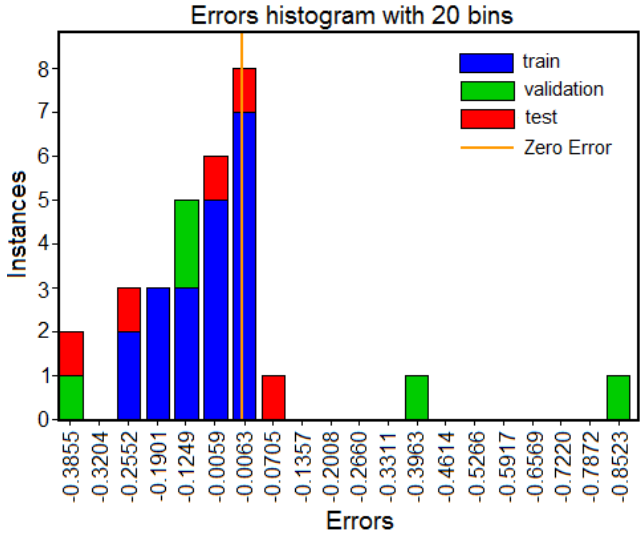

Fig. 5. Errors histogram of train, validation and test sets.

In Fig. 6. series 1 and 2 respectively show mean values real and simulated our experiments andall graphs show that our proposed model can well predict the results of experiment of the

biosynthesized method. Also it is indicated in Fig. 6. (a), that size of Ag-NPs in green method decreases significantly by increasing volume of *C. longa* tuber powder. Effects of the reaction temperature on the size of Ag-NPs is also investigated. It is indicated in Fig. 6. (b), that size of Ag-NPs increases significantly by increasing temperature. Based on the current experiments and same ANN results, the operative temperature in green method is suggested to be about less than 30 °C.

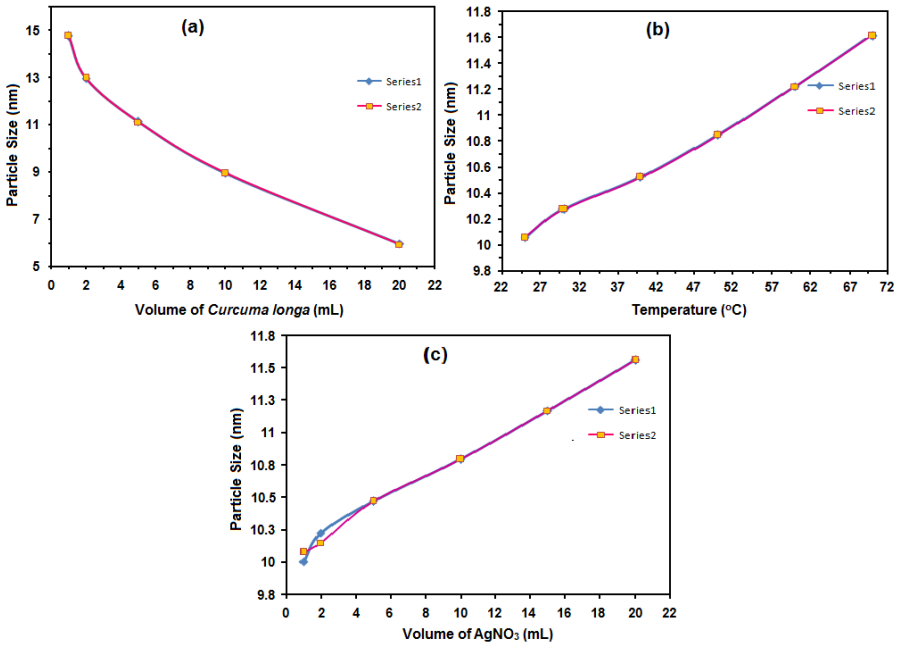

Fig. 6. Actual and ANN predicted plating rates for different levels of the parameters: volume of Curcuma longa, temperature and volume of $AgNO_3$ (a-c).

Fig. 6. (c) shows the influence of volume of $AgNO_3$ on the size of Ag-NPs, which by increasing volume of $AgNO_3$ increases the size of Ag-NPs. Therefore based on the presented experiments and the ANN results, the volume of $AgNO_3$ in green method is recommended to be about less than 2 mL. Therefore on the based simulated analysis, optimum parameters for minimum size of Ag-NPs are, more than 18 mL for volume of *C. longa*, less than 30 °C for the reaction temperature, and less than 2 mL for the volume of $AgNO_3$. The volume of *C. longa* powder and reaction temperature are the most effective parameters on the size of Ag-NPs. Implying the empirical model derived from the ANN can be used to sufficiently describe the relationship between the independent variables and response.

4. Conclusion

In this research, an ANN model for predicting the size of Ag-NPs is offered. The model accounts the effect of volume of *C. longa* tuber powder, temperature, stirring time, and volume of $AgNO_3$ on the size of nanoparticle. Based on the obtained results it can be concluded that the LM neural network model with 10 neurons in 1 hidden layer will be the very good training algorithm and can present a worthy performance for ANN modeling of nano composites behaviors. Data analysis showed that volume of *C. longa* tuber powder and reaction temperatures are two most sensitive parameters. Can say using ANN models is useful tools for saving time and cost by predicting the results of the reactions and also its analysis shows that they are powerful tool for analysis and modeling of the chemical reactions.

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