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Neural Network Modeling of Placid ZNO Nanoparticle as a Sensible Adsorbent for Removal of as(III) Ions Ingrained on Activated Silica Using Acalypha Indica (ZNO-NPS-AS-AI)

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Abstract: A three-layered Artificial Neural Network (ANN) model was developed to envisage the efficacy of As(III) ions amputation from aqueous solution by Zinc oxide nanoparticle ingrained on activated silica using Acalypha Indica (ZnO-NPs-AS-AI) based on 95 experimental sets obtained in a laboratory batch study. On the basis of batch test results, most favorable operating initial As (III) concentration of 0.05N, at pH 6, an adsorbent dosage of 3.0 g and an agitation speed of 350 rpm for maximum removal of 98% As(III) ions. Experimental results showed that a contact time of 70 min was generally sufficient to accomplish equilibrium. ANN model was able to predict adsorption efficiency using neuro (R2011a) solution with a tangent sigmoid transfer function (tansig) at hidden layer with 20 neurons and a linear transfer function (purelin) at output layer via Back Propagation (BP) training combined with Principal Component Analysis (PCA). The Levenberg-Marquardt algorithm (LMA) was found as the best algorithms with a minimum mean squared error (MSE) of 0.227. The linear regression between the network outputs and the resultant targets were established to be reasonable with a correlation coefficient of about 0.992 for five variables with accuracy
Keywords : Neural Network; Back Propagation; Zinc oxide nanoparticle; Acalypha Indica; Kinetics; Isotherm

Introduction

Arsenic is widely distributed throughout the environment in the air, water and land which is naturally present at high levels in the groundwater. It is highly toxic in its inorganic form. Long-term exposure to arsenic from drinking-water and food can cause cancer and skin lesions. It has also been associated with developmental effects, cardiovascular disease, neurotoxicity and diabetes. Skin lesions and skin cancer are the most characteristic effects. The greatest threat to public health from arsenic originates from contaminated groundwater. Inorganic arsenic is naturally present at high levels in the groundwater of a number of countries, including Argentina, Bangladesh, Chile, China, India, Mexico and the United States of America. Drinking-water crops irrigated with contaminated water and food prepared with contaminated water are the sources of exposure. People who smoke tobacco can also be exposed to the natural inorganic arsenic content of tobacco because tobacco plants essentially take up arsenic naturally present in the soil[1]. The immediate symptoms of

acute arsenic poisoning include vomiting, abdominal pain and diarrhea. These are followed by numbness and tingling of the extremities, muscle cramping and death in extreme cases. The International Agency for Research on Cancer (IARC) has classified arsenic and arsenic compounds as carcinogenic to humans and has also stated that arsenic in drinking-water is carcinogenic to human. There are a number of options to reduce levels of arsenic in drinking-water[2]. Many methods have been proposed for removal of excessive As(III) from waste water such as adsorption, coagulation, ion exchange, precipitation, electrolysis, and reverse osmosis[3,4,5,6,7]. Most of these methods suffer from some disadvantages such as high capital and operational cost, limited tolerance to pH change, incomplete metal removal and high cost of reagent and energy requirements. Adsorption technique is quite trendy due its simplicity and high efficiency, as well as the ease of use of a wide range of adsorbents[8,9]. Adsorption has been treated as a potential technology for removal of toxic heavy metals from industrial waters using microbial biomass[10,11,12,13]. The main advantages of this technique are the reusability of biomaterial, low operating cost, improved selectivity for specific metals of interest, removal of heavy metals from effluent irrespective of toxicity, short operation time, and no production of secondary compounds which might be toxic[14,15]. The present study is aimed at selection of a low cost adsorbent which can adsorb arsenic from aqueous solution. Detailed batch studies with the selected adsorbent, *Azadirachta indica, Corriandrum sativum, Ocimum sanctum, Emblica officinalis* have been carried out in the earlier investigation[16,17]. The tribulations of our bionetwork are increasing with the encroachment in technology. Techniques used for deduction of heavy metals like lime coagulation, reverse osmosis, chemical precipitation, ion exchange and solvent extraction are expensive and non-environmental friendly as compared to adsorption. Adsorption is one of the easiest safest and most expenditure effective methods for the removal of metals. The foremost advantage of an adsorption system of silica implanted zinc oxide nanoparticle are less investment in terms of both initial cost and simple designed easy operation and has no effect of toxic substance compared to conventional chemical treatment process. There is a vital requirement for development of innovative but low cost processes by which heavy metals can be removed. Escalatingattentiveness towards green chemistry and biological processes has led to the connotation and practicability of an eco-friendly approach for the synthesis of ZnO nanoparticle ingrained on activated silica using *Acalypha indica* as an adroit adsorbent for removal of As(III) using Artificial Neural Network.

Materials and Methods

Preparation of the adsorbent ZnO-NPs-AS-*AI*

The introductory material Zinc acetate dihydrate (99% purity) and sodium hydroxide (pellet 99%) used was supplied by Sigma-Aldrich chemicals, India. Green synthesis scheme was used to prime down ZnO-NPs-AS-*AI* structure. Aqueous leaf extract of *Acalypha indica* was stirred for 30 min and 1g of Zinc acetate dihydrate was added under dynamic stirring. After 1hr stirring 10 g of activated silica was introduced into the above solution. Addition of drops of aqueous NaOH resulted in a white aqueous solution at pH 10-12. This was then positioned in a magnetic stirrer for 2Hrs. The activated silica surrounded ZnO nanoparticle were then filtered and washed with double distilled water. The synthesized ZnO-NPs-AS-*AI* was maintained at 60ºC for 12 hrs. A mortor was used to homogeneously ground the sorbent .The proposed sorbent were stored in air at room temperature.

Characterisation of the adsorbent

Pore size, surface area and pore volume of the sorbent were determined by single point Brunauer, Emmett and Teller (BET) N_2 sorption procedure. Elemental analysis and external morphology of the sample were characterized by scanning electron microscope (SEM) (LEO 1530FEGSEM). The surface characteristic and particle size distribution of ZnO-NPs-AS-*AI* was investigated using Particle Size analyser (PSA, Zetasizer Ver.6.32). The X-Ray powder diffraction pattern of the as- synthesized sample was recorded on an X-ray diffractometer (XRD, PW 3040/60 Philips X'Pert, Holland) using Cu (Kα) radiation (λ =1.5416 A˚) operating at 40 kv and 30 mA with 2θ ranging from 10- 90˚.

Batch adsorption tests and Input parameters

Common isotherms are used in this investigation to generate different series of adsorption data that will serve as learning data sets to fit artificial neural networks. The four types of isotherms are Langmuir, Freundlich, Temkin and BET. Langmuir isotherm applies to localized adsorption of monolayer surface coverage assuming that each adsorbed molecule occupies one adsorption site. Freundlich isotherm is a semi-empirical equation which is widely used to represent adsorption equilibrium data for low to intermediate range

of concentration. Temkin isotherm describes the behavior of adsorption systems on heterogeneous surfaces. Specific surface area, pore volume and pore size of the sample were determined by means of N_2 adsorption-desorption at -195.629 C° using BET analysis. The physisorption and chemisorptions kinetic rate equation is determined using Pseudo first and second order rate equation. Parameters such as concentration, dosage, contact time, pH and agitation have been evaluated by means of portable instruments and analyzed in the laboratory. Sequence of batch adsorption experiments were conducted to resolve the effect of adsorbent dosage, initial concentration of As(III) ions, initial pH, operating agitation speed and contact time on adsorption performance of ZnO-NPs-AS-*AI* used as adsorbent materials in this study. Therefore various adsorbent dosages of 0.5, 1 - 5.5 g were introduced into 250mL flasks with 20mL solution containing 0.005, 0.075,0.01 - 0.1 N of As(III) ions. The flasks were then placed in an orbital shaker and agitated up to a total contact time of 70 min at an agitation speed of 50, 100 - 450 rpm. Samples were taken at predetermined time intervals (10, 20, 30, and 120 min) and then separated by centrifugation. Concentration of As(III) ions aqueous phases were analyzed volumetrically. Batch experiments were carried out in a pH range of 1–8 to establish the effect of initial pH on adsorption. Each experiment was performed in duplicate to observe the reproducibility and the mean value was used for each set of values. Percentage of As(III) ions removal being the output parameter of the ANN model was considered as a measure of adsorption efficiency of ZnO-NPs-AS-*AI*. The efficiency of percentage removal was calculated as follows:

$$\% \text{ Removal} = (C_0\text{-}C_e) \times 100 / C_0$$

$$q_e = (C_0\text{-}C_e) \times V / W$$

Where C_o and C_e are initial and equilibrium concentration of As(III) respectively, q_e the amount of arsenic adsorbed, V the volume of the solution and W the weight of the adsorbent used.

Definition of the ANN model

In this study Neuro Solution 7(R2011a) MATLAB® mathematical software was used to predict the sorption efficiency. Ninety five experimental sets were used to develop the ANN model. Data statistics and model variables are presented in **Table** 1. A three-layer ANN with tangent sigmoid transfer function (*tansig*) at hidden layer and a linear transfer function (*purelin*) at output layer was used. The data gathered from batch experiments was divided into input matrix [p] and target matrix [t].

Table 1. Experimental statistics and model variables for adsorption of As(III)

Variables	Range
Input layer[P]	
Initial arsenic concentration (N)	0.005-0.1
Adsorbent dosage (g)	0.5-4.5
Initial pH	1-8
Contact time (min)	10-120
Agitation speed (rpm)	50-450
Output layer [t]	
Removal (%)	36-98

A three-layered ANN with sigmoid axon transfer function and linear purelin was used for input hidden and output layers. The data gathered from batch experiments were divided into five input matrix. The three layered sigmoid and purelin network represents functional relationship between inputs and output, provided sigmoid and purelin layers has enough neurons. Levenberg-Marquardt algorithm is fastest training algorithm for network of moderate size used in the present. Feed-Forward Back Propagation (FFBP) neural networks were utilized for training of experimental data set in some application works related to drying technology[18,19,20]. A typical FFBP neural network consists of the following layers: input layer, hidden layer and output layer as illustrated in Figure 9. For studying by FF network, back propagation (BP) learning algorithm is normally used. Finally, some analysis of the network response was carried out. The entire data set was put through the network and a linear regression between the network outputs and the corresponding targets was performed. Principal component analysis (PCA) followed the division of the original 95 data into training, validation and test subsets. One fourth of the data was taken for the validation set, one fourth for the test set and one half for the training set. Therefore 67, 14 and 14 samples were used for the training, validation and test subsets respectively. The experimental data was loaded into the workspace at random for each subset. During training, the output

vector is computed by a forward pass in which the input variable (concentration, dosage, time, pH and agitation) is propagated forward (Table 1) through the network to compute the output (percentage removal) of each unit.

Results and Discussion

Experimental characterization of ZnO-NPs-AS-AI

Figure 1 shows emblematic peak primarily at about 12˚, 20˚ for silica and 32˚, 34˚, 36˚ for indicative nano-crystalline nature of ZnO-NPs-AS-*AI* in combination. It can be seen that all of these peaks are well matched[21] with that of Zincite phase (JCPDS CARD NO: 36- 1451). The crystalline sizes of the ZnO-NPs-AS-*AI* prepared at 60˚C for 12 hours were observed to be 80nm which was determined by Debye Scherer's formula according to the equation ($d=k\lambda /\beta \cos\theta$), where λ is the wavelength of incident X-ray which is the Full Width Half Maximum (FWHM) of diffracted peak and θ is diffracted angle. Figure 2 shows the SEM micrograph of homogeneous shape and size for ZnO-NPs-AS-*AI* at X30,000 which is the image of ZnO-NPs-AS-*AI*. The SEM image of ZnO-NPs-AS-*AI* is found to have the size ranging 500 nm. The EDX spectrum (Figure 3) shows the peak only for the presence of Zinc, Oxygen, Silicon elements in the as-prepared ZnO-NPs-AS-*AI*. The percentage of zinc, silica and oxygen is found to be 100% which proves that it is completely free from impurities.

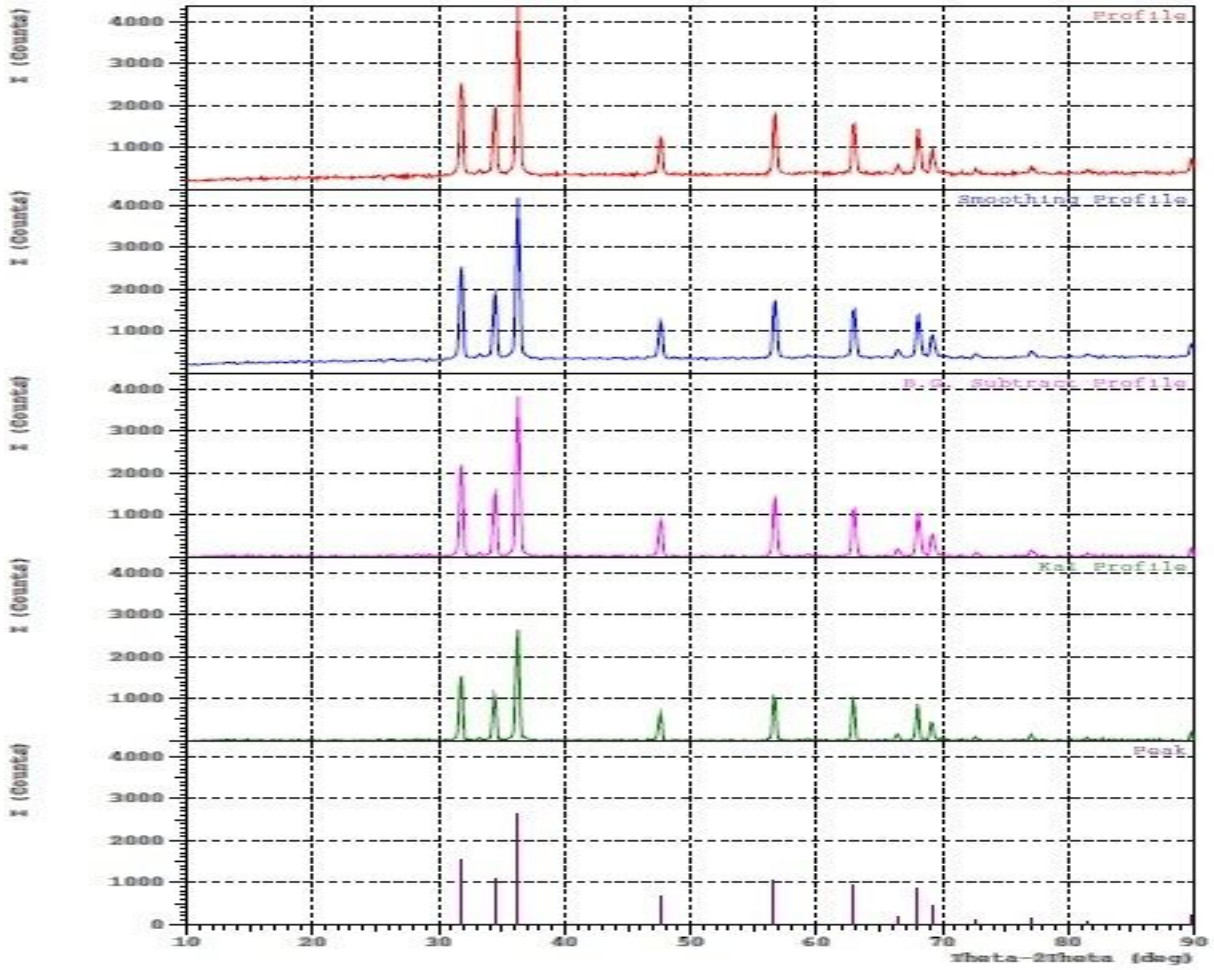

Figure 1 Distinctive XRD Pattern of ZnO-NPs-AS-*AI*

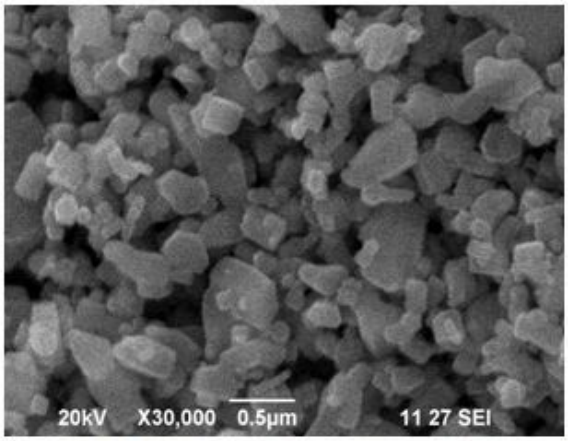

Figure 2 SEM Magnification of ZnO-NPs-AS-*A*/

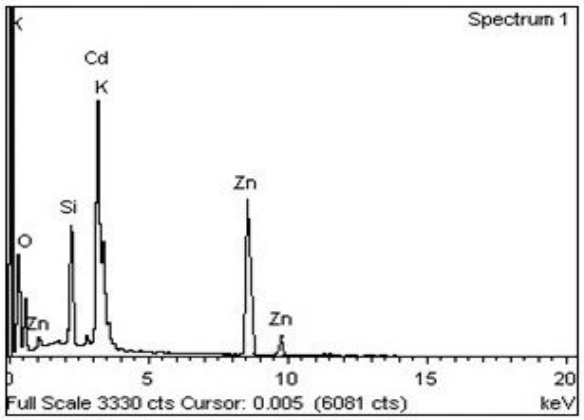

Figure 3 EDX spectrum of ZnO-NPs-AS-*A*/

Influence of five input parameters on adsorption

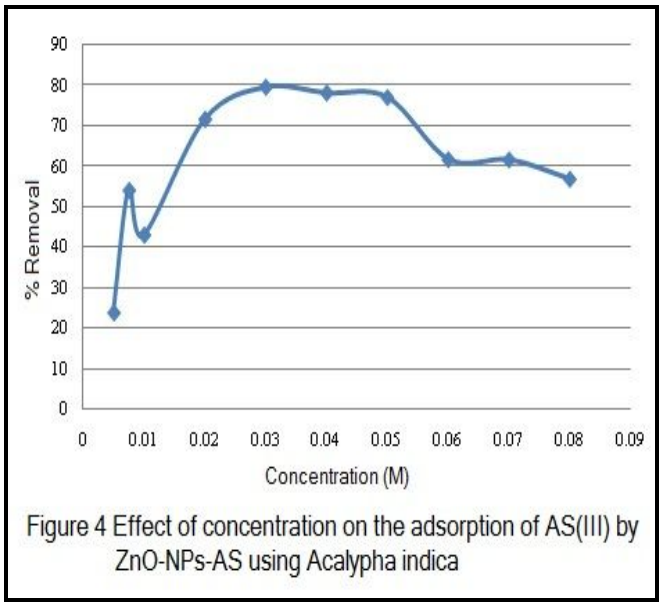

Figure 4 Effect of concentration on the adsorption of AS(III) by ZnO-NPs-AS using Acalypha indica

The effect of concentration on the sorptive removal of As(III) is presented in Figure 4. The results show gradual decrease in adsorption with increase in concentration from 0.005N to 0.05N and then constant. The decrease in percentage of adsorption may be caused by the lack of sufficient surface area to accommodate much more As(III) ions available in the solution. Hence at higher concentration lower adsorption yields were observed because of the saturation of the adsorption sites. Similar trend is also noted by other researchers[22].

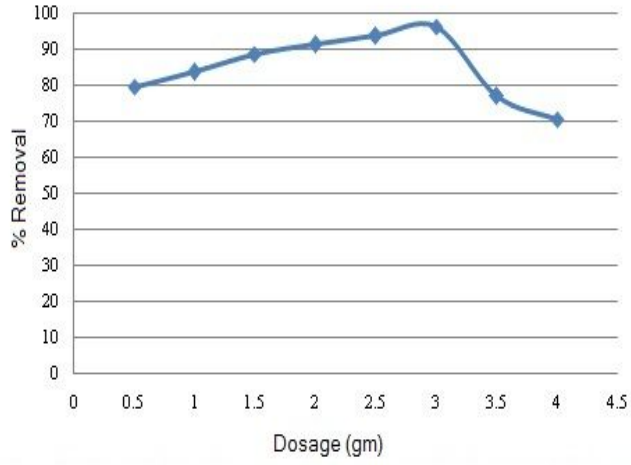

Figure 5 Effect of adsorbent dosage on the adsorption of AS(III) by ZnO-NPs-AS using Acalypha indica

Effect of adsorbent dosage on the adsorption of As(III) is studied by changing the ZnO-NPs-AS-*AI* dosage from 0.5gm to 4.5gm and the initial concentration of As(III) was fixed as 0.05N. Figure 5shows an increase in adsorption from 0.5gm to 3gm due to greater availability of the surface area. At higher concentration of the adsorbent any further addition of the adsorbent beyond 3gm did not cause any significant change in the adsorption. This may be due to over lapping of adsorption sites as results of overcrowding of adsorbent particle. The maximum removal of As(III) was obtained in the adsorbent dose of 3gm which is in accordance with other researchers[23].

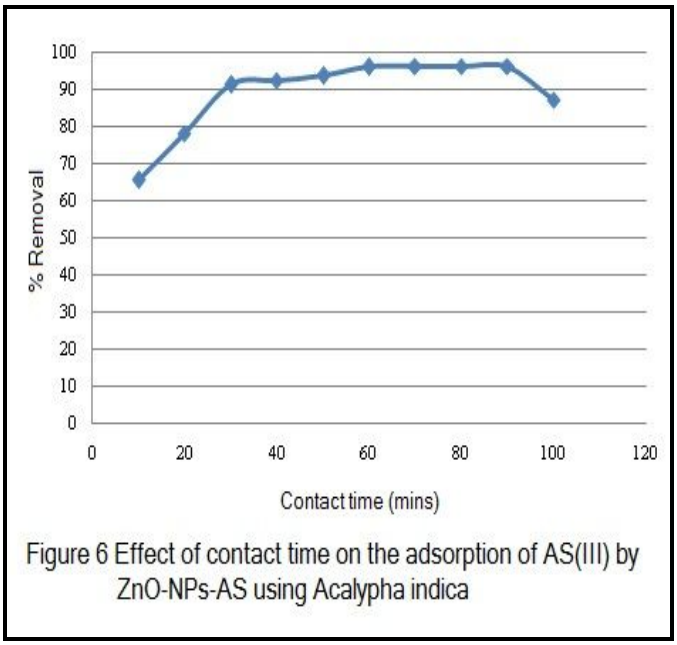

Figure 6 Effect of contact time on the adsorption of AS(III) by ZnO-NPs-AS using Acalypha indica

The studies involving different contact times help in determining the uptake capacities of the As(III) at varying time intervals keeping the amount of the adsorbent is fixed as 3g at room temperature. The purpose of studying the effect of time on adsorption is to establish the equilibrium reaction time between adsorbent and As(III). The adsorption experiment was carried out using contact time ranging from 10mins to 120 mins and the results are depicted in the Figure 6. It was observed that metal adsorption occurred rapidly. The adsorption efficiency of As(III) increased gradually with increasing contact time up to 70 mins and reached a plateau afterwards and there was no change in adsorption and the equilibration time is 70 mins. The data showed that time is a significant factor contributing largely to the adsorption under different sets of condition as time is required for As(III) to diffuse in to the ZnO-NPs-AS similar results were observed by other reporters[24] for As(III) on to different adsorbents.

The pH is one of the important parameter controlling the removal of metal. It is clear from the Figure 7 that pH has a significant role on adsorption of metal using the synthesized ZnO-NPs-AS-*AI*. It was found that

increasing the pH of the solution from 1 to 8 increases the percentage removal of metal. This is due to the decline in the competition between proton and metals species for surface sites. Basic pH was also attempted but it could not be investigated due to precipitation[25].

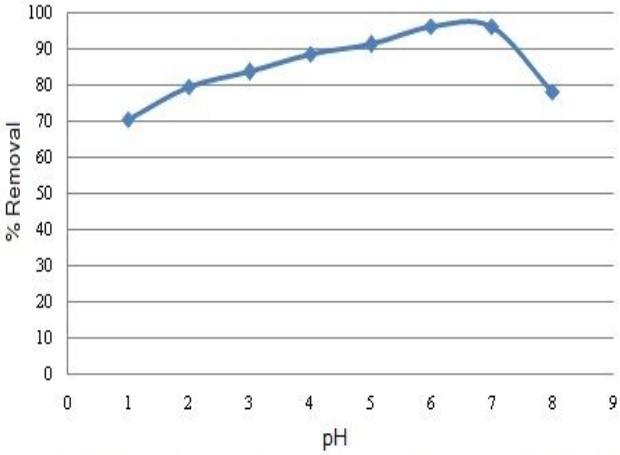

Figure 7 Effect of pH on the adsorption of AS(III) by ZnO-NPs-AS using Acalypha indica

The effect of agitation speed on the adsorption rate was investigated by changing the speed in the range 50 to 450 rpm. The rate of As(III) removal was very significant from 50 rpm to 300 rpm. Increase in agitation makes the particle to collide with each other with the greater speed resulting in detachment of loosely bound ions also they did not get appropriate time to interact with each other as shown in Figure 8. By increasing the speed beyond 350 rpm there was no further increase in adsorption. This is because all the binding sides have been utilized and no binding sides were available for further adsorption[26]. An increasing agitation rate may reduce the film boundary layer surrounding the ZnO-NPs-AS-*AI*.

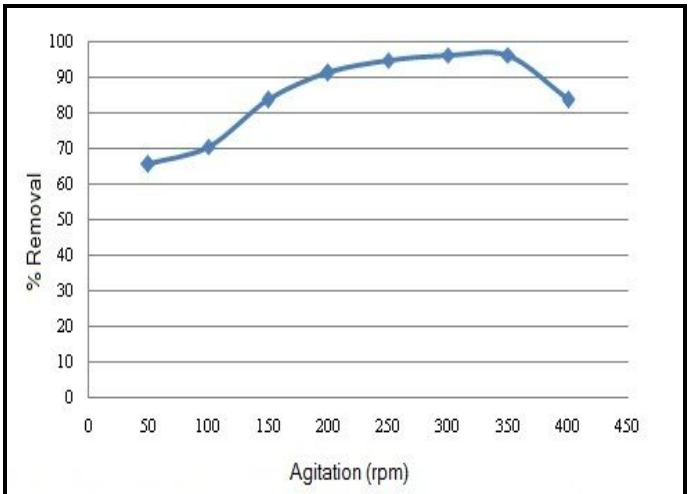

Figure 8 Effect of agitation speed on the adsorption of AS(III) by ZnO-NPs-AS using Acalypha indica

Optimisation of ANN

The prediction and removal efficiency of As (III) ions from aqueous system using ZnO-NPs-AS-*AI* are made in the range of metal concentration with which experiments have not been conducted. A training set of ninetyfive experimental data sets was selected to develop the model. ANN model based on three layers recurrent Back Propagation Algorithm (BPA) for the experimental data was applied to train the neural network. During training, the output vector is computed by a forward pass in which the input variable (concentration, dosage, time, pH and agitation) is propagated forward through the network to compute the output (percentage removal) of each unit (Table 1). The output is then compared with the desired vector which resulted into error signal for each output unit .To minimize the error appropriate adjustments were made for each of the weights of the network. After several such iterations, the network was trained to give the desired output for a given five

input variable (Figure 9). The best possible network structure was determined as three layer with 20 hidden neurons (Figure 9) and 1000 epochs with unsurpassed validation performance and least mean squared error describing the dynamics of As(III) in aqueous solutions with output one. The available 95 data set are divided into 67 data sets for training and 14 data sets for testing and validation. When the training for 67 data sets has been completed testing for 14 data has done to examine the performance of the derived network as testing is the primary approach for verifying that inputs produced the appropriate output as 1. The iteration continues until the error sum is converged.

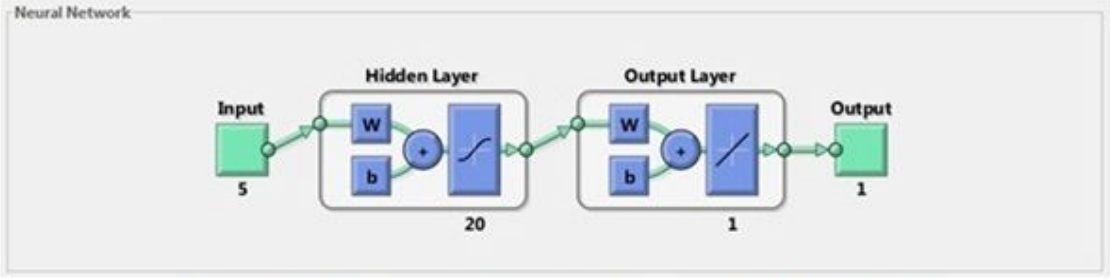

Figure 9 ANN architecture with *tansig* and *purelin* function

In the field of machine learning a confusion matrix also known as a contingency table or an error matrix is a specific table layout (Figure 13) that allows visualization of the performance of an algorithm typically a supervised learning. Each column of the matrix represents the instances in a predicted class, while each row represents the instances in an actual class. This demonstrates how a confusion matrix can be used to assess the performance. All dominant diagonal elements represented in the green and blue cells (67, 14, 14) on the confusion matrix represents the classified data with 100 percentage performance while the off-diagonal (red cells) represents the misclassified data.

Figure 10 Contigency table represents instances in a predicted class and actual class

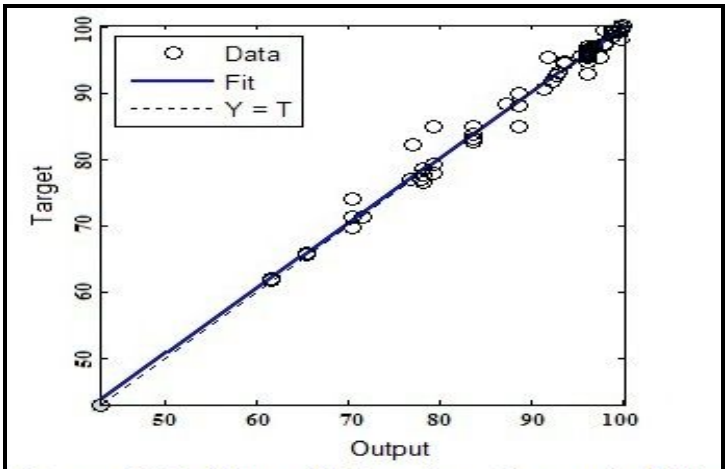

Figure 11 Training plot for adsorption probability

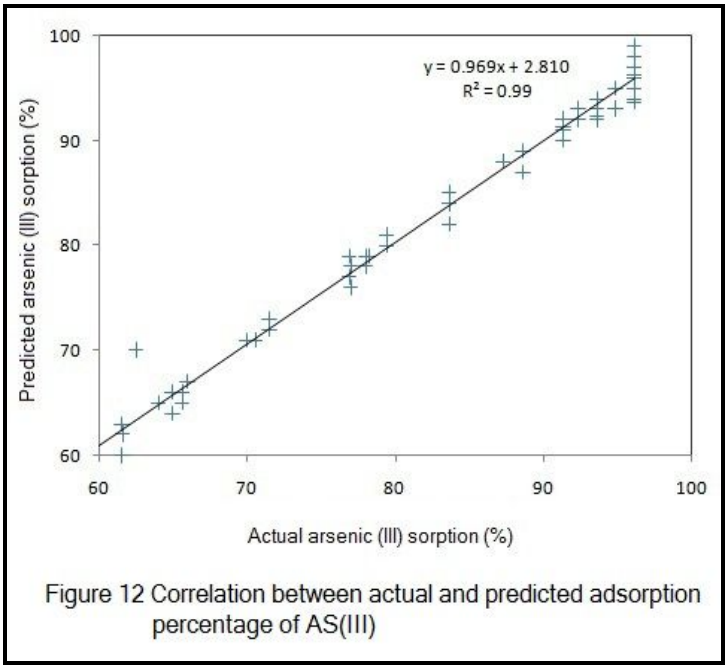

Figure 12 Correlation between actual and predicted adsorption percentage of AS(III)

A total of 95 points have been used to train the neural network, of which 67 points are chosen for training and 14 points are chosen for validation and 14 points for testing. Total iteration number was set as 11 at 1000 epochs for the learning algorithms and the performance goal is set at 10^4. A Feed-Forward BP was used for modeling the experimental design for predicting the removal capacity of As (III). The experimental design used in this research work was based on one factor experiment at a time. The network is tested with different number of neurons to find the optimal number of neurons at the hidden layer by observing the mean squared error. Twenty neurons are selected in the hidden layer when mean square error starts decreasing. Learning and momentum parameters are set at 0.20 and 0.10 respectively during the training phase. During training phase the output vector is computed by a forward pass in which the input is propagated forward through the network to compute the output value of each unit. The output vector is then compared with the desired vector which resulted into error signal for each output unit. In order to minimize the error appropriate adjustments were made for each of the weights of the network. After several such iterations the network was trained to give the desired output for a given five input vector. Then network is trained till minimum root mean square error is observed. A root mean square error of 0.227 is observed at epoch number 1000. Training was stopped at this point and weights have been frozen for network to undergo testing phase. A high degree of correlation between actual and predicted sorption efficiency of 100% observed is shown in Figure 11 & 12. Coefficient of determination (Figure 11 & 12) $R^2 = 0.992$ is obtained for training data set. When the network is well trained testing of the network with testing data set is carried out. The prediction ability of the developed network model for responses of experimental data not forming part of the training set. During testing phase output of the data is not presented to the network. The correlation coefficient (Figure 11 & 12) confirms the degree of linear dependence of two random variables. An added variable plot illustrates the incremental effect on the response of percentage removal by removing the effects of all other terms. The slope of the fitted line is the coefficient of the linear

combination of the specified terms projected onto the best-fitting direction(R=0.99). The adjusted response includes the constant intercept terms and averages out all other terms.The studies conclude (Table 2) that ANN approach is quite efficient in modeling complex adsorption phenomenon.

Table 2. Performance evaluations of various isotherms and kinetics with ANN

S.No	Models	R^2	BLE
1.	Artificial Neural Network (Training)	0.992	y=0.98x+1.6
2.	Langmuir	0.989	y=12.97x+1.507
3.	Freundlich	0.962	y=-0.087x-1.141
4.	Temkin	0.960	y=-0.008x+0.068
5.	Pseudo first order	0.452	y=0.008x-2.827
6.	Pseudo second order	0.603	y=185.7x-1376
7.	Correlation of actual and predicted	0.990	y=0.969x+2.810
8.	BET	0.990	------

R^2 - Correlation coefficient, BLE - Best Linear Equation.

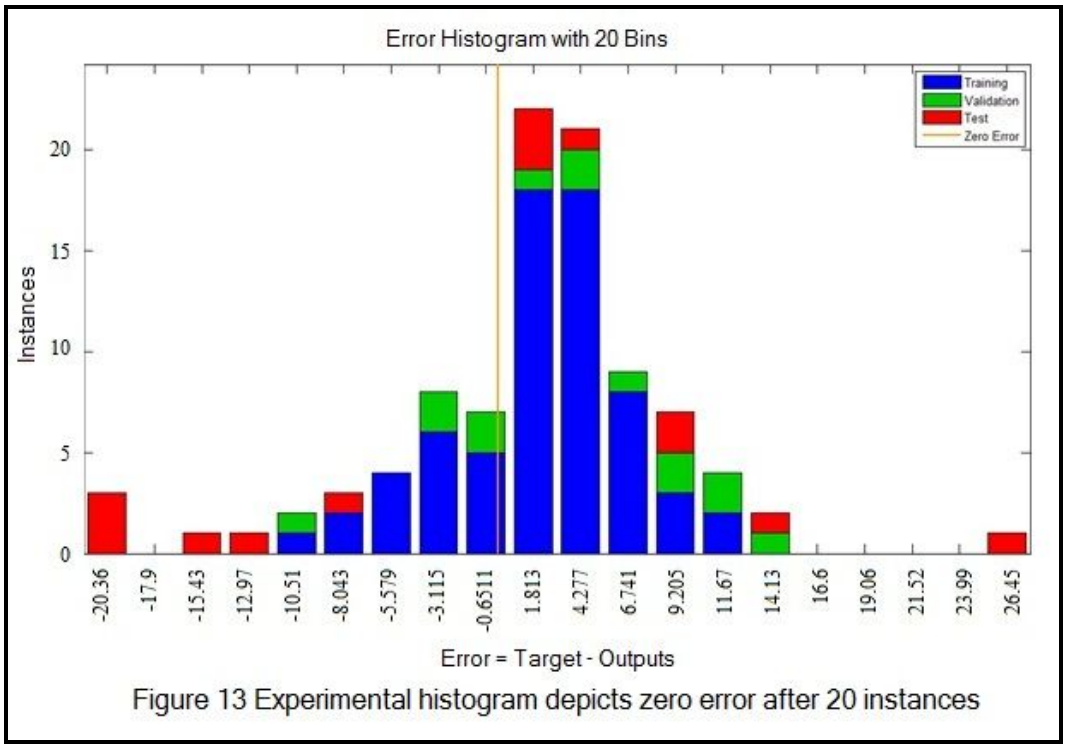

Figure 13 Experimental histogram depicts zero error after 20 instances

This error histogram graphically represents (Figure 13) the distribution of data and a plot of the number of data points in each 20 bin. Experimental histogram depicts zero error after repeated trials of 20 instances. These studies conclude that ANN approach is quite efficient in modeling complex adsorption phenomenon.

Conclusion

This green approach confirmed the effect of various operational parameters on the adsorption of As(III) ions onto ZnO-NPs-AS-*AI* was investigated and optimized. Batch adsorption experiments showed that optimal operating conditions were determined to be pH of 6, an adsorbent dosage of 3.0 g, an As(III) concentration of 0.05N and agitation speed 350 rpm. A contact time of 70 min was found to be sufficient to achieve equilibrium. Findings of the experimental study clearly indicated that the removal of As(III)ions from aqueous solutions could be effectively improved up to about 98% by using ZnO-NPs-AS-*AI*. The maximum adsorption capacity of the ZnO-NPs-AS-*AI* in As(III) removal was found to be 3.4 g/L. The equilibrium data have been analyzed using correlation coefficients of Langmuir, Freundlich, and Tempkin isotherms (R^2 = 0.989, 0.962, and 0.960) in which Langmuir isotherm is more fitted than Freundlich and Tempkin isotherm. From the results it is accomplished that the maximum adsorption corresponds to a saturated monolayer of As(III) molecules on the

adsorbent surface with constant energy and strong dependence. The data were analyzed using pseudo-first order, pseudo-second order and intra-particle equation in which pseudo-second order (R^2 = 0.603) shows moderate dependence than pseudo-first order (R^2 = 0.452) where intra particle diffusion was not the rate controlling step. The proposed ANN model showed a precise and an effective prediction of the experimental data with a good correlation coefficient of 0.99 for five operating variables. The optimal neuron number for the LMA was determined to be 20 hidden neurons with MSE of 0.22. The sensitivity analysis showed that MSE values decreased as the number of variables used in the ANN model increased. The present outcome recommend that ZnO-NPs-AS-*AI* synthesized in a inventive green method may be used as an economical and effectual adsorbent for the confiscation of As(III) ions from aqueous solutions.

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