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Predicting the Sorption of Mn^{2+} ion from Wastewater onto Zinc Chloride Activated Sawdust using Artificial Neural Network (ANN)

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Abstract
Activated carbon from sawdust was prepared and its textural properties was evaluated using Fourier transform infra-red (FTIR) and scanning electron microscope (SEM) to determine the presence of functional groups and visualize its microstructural arrangement in other to ascertain its potential for the removal of Mn^{2+} ion from wastewater. Batch experimental technique was then employed to evaluate the effects of adsorption variables, namely; initial metal ion concentration, adsorbent dose, pH, and contact time on the sorption of Mn^{2+} ion onto acid activated sawdust. Thereafter, statistical design of experiment (DOE) using central composite design was employed to randomized the levels of selected input parameters in other to produce an experimental design matrix. To model and predict the sorption of Mn^{2+} ion onto acid activated sawdust, artificial neural network (ANN) was employed. To apply the neural network, the input data were first normalized to avoid the problems of weight variation. Thereafter, different training algorithm and hidden neurons were selected and tested to ascertain the optimum number of hidden neuron and the best training algorithm that will produce the most accurate network for predicting the sorption of Mn^{2+} ion. Results obtained show that Levenberg Marquardt Back Propagation training algorithm with 10 hidden neurons in the input and output layer with tangent sigmoid transfer function produced the most accurate prediction network. In addition, artificial neural network gave a strong agreement between the experimental and predicted sorption efficiency of Mn^{2+} ion with coefficient of determination (R^2) value of 0.9893.
Keywords: Activated sawdust, Artificial neural network, central composite design, Levenberg Marquardt Back Propagation training algorithm
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1. Introduction

The discharge of untreated wastewater containing heavy metals such as lead, chromium and manganese into the environment is a public concern (Ilaboya and Izinyon, 2019). Although, some conventional processes exist for treating this wastewater prior to their being discharged into water bodies, adsorption is much easier and highly efficient (Ilaboya et al., 2013, Izinyon et al., 2016). Adsorption, which is an alternative process for heavy metal removal involves the use of commercial activated carbon as adsorbent for wastewater treatment. The adsorption process is challenged by the difficulties of preparing commercial activated carbon and the associated problem of regeneration. Hence, the search for locally existing adsorbents which are also readily available and less difficult to prepare (Ilaboya, 2017).

Unlike organic pollutants, the majority of which are susceptible to biological degradation, heavy metals will not degrade into harmless end products, and their presence in streams and lakes leads to bioaccumulation in living organism, causing health problems in animals, plants and human beings (Shengtao et al., 2011). The assimilation of relatively small amounts of these heavy metals over a long period of time in the human body can lead to chronic toxicity coupled with numerous health challenges such as skin irritation, lung tumor including severe damage to the nervous systems and circulatory system (DWI, 2014, Khurshid and Qureshi, 1984). The toxic effects of lead, chromium and manganese ions in human, especially when present above the threshold limit in the hydrosphere are well documented. The presence of these heavy metals in the environment is of great concern to scientists and engineers because of their toxic nature and other adverse effects posed by the discharge of untreated effluents containing heavy metals on receiving water bodies (Sekar et al., 2004, Badmus et al., 2007, Eba et al., 2010). Since adsorption is a complex process that is influenced by many variables, predicting the optimal working condition requires the use of different classical methods of prediction such as artificial neural network (ANN), adaptive neuro-fuzzy inference systems, fuzzy logic and genetic algorithm. Artificial neural networks (ANNs) belong to a class of models, where difference or differential equation are used to identify a direct mapping between inputs and

outputs without detailed consideration of the internal structure of the physical processes (Sinan et al., 2011, Mohammed et al., 2013). The structural flexibility and ease of implementation of ANN accounts for its selection in this research.

2. Materials and Methods

2.1 Material collection and preparation

Locally available sawdust was collected from sawmill located in Egor Local Govt. Area in Edo State of Nigeria using a washed, clean dried shovel. 2 kg of the sawdust was placed in a fresh black polythene bag and taken to Water Resources and Environmental Engineering Laboratory in the Department of Civil Engineering, University of Benin where the experiment was conducted. First, the sawdust was soaked in a plastic bowl containing 5% hydrogen peroxide and washed with distilled water to remove any carbonaceous and water-soluble impurities. It was dried in hot air oven at 50-70°C for 8 hours, pulverized and screened sieved to obtain geometric sizes of 212 µm before analysis (Mariadas et al., 2012). Carbonization was done using the method recommended by (Ekpete & Horsfall, 2011) with slight modification as follows. 500 g of the pulverized sawdust was placed in a muffle furnace which allows limited supply of air at a temperature of 250^{0C} for 60 minutes. The sample was then placed in a desiccator to cool before it was activated using the method recommended by (Mansfield, 1996) with slight modification as follows: 125 g of the carbonized sawdust was soaked in 250 mL of 5.5M $ZnCl_2$ solution. The mixture was thoroughly mixed until it formed a paste. The paste was then transferred to an evaporating dish which was placed in an oven and heated at 200ºC for thirty minutes. It was then allowed to cool and washed with distilled water to remove the residual salt. Thereafter, it was oven dried at 105^0C for thirty minutes, grind using mortar and pestle and sifted with 106µm Standard Tyler Sieve. The activated sawdust was then characterized before using.

2.2 Equipment used for the experiment

Major equipment's used in this study are presented in Table 1. Minor equipment's include: pH meter, digital weighing balance and hand held conductivity meter. Glass wares include: reagent bottles, conical flask, measuring cylinder, glass funnels and beakers.

Table 1: Equipment Details

S/No	Equipment Name	Model
1	Laboratory Oven	DHG 9101-2A
2	Industrial Furnace	DHG 9101-5A
3	Constant Temperature Water Bath	DHG 3101-6A
4	Hot Plate with Magnetic Stirrer	HJ-3D
5	Scanning Electron Microscope (SEM)	APEX 3020 PSEM 2
6	Fourier Transform Infra-red (FTIR)	FTIR 2000, Shimadzu Kyoto, Japan
7	X-Ray Fluorescence (XRF)	APEX 3022
8	Atomic Absorption Spectrophotometer (AAS)	UNICAM SOLAR 969

2.3: Performance of activated sawdust

2.3.1: Analysis of Microstructures

Scanning electron microscope (SEM) was employed to study the surface characteristics in order to assess the presence of microporous structures on the surface of activated sawdust. Such presentations can provide possible explanations on the adsorbent behaviour and its adsorption potentials (Omisanya et al., 2012).

2.3.2 Functional group analysis

Fourier Transform Infra-Red (FT-IR) spectra of activated sawdust was obtained by using FTIR spectrophotometer (Model: FTIR 2000, Shimadzu Kyoto, Japan). The spectra were employed to determine the presence of functional groups that can influence the adsorption capacity of the sawdust. 150 mg potassium bromide (KBr) disks containing approximately 2 % sawdust was prepared prior to recording the FTIR spectra in the range of 400-4000 cm^{-1} with a resolution of 4.0 cm^{-1} (Dawodu et al., 2012).

2.4 Preparation of aqueous solution

Stock solution of manganese was prepared by dissolving accurate quantity of manganese (II) chloride tetrahydrate ($MnCl_2.4H_2O$) in one liter of distilled water. All working solutions were made by diluting the stock solution with distilled water and the concentration of Mn^{2+} ion present in solution was determined with the aid of Atomic Absorption Spectrophotometer (AAS). A duplicate was analyzed for each sample to track experimental error and show capability of reproducing results. The pH of the working solution was adjusted to the desired value for each

experiment with drop wise addition of 1 M nitric acid (HNO_3) or 1 M sodium hydroxide (NaOH). A comprehensive list of all the chemicals and reagents with the respective minimum assay is presented in Table 2.

Table 2: List of chemicals and reagents

S/No	Chemicals/Reagents	Type	Minimum Assay
1.	Nitric Acid	Analytical	96 %
2.	Sodium Hydroxide	Analytical	96 %
3.	Manganese (II) chloride tetrahydrate	Analytical	96 %
4.	Hydrogen Peroxide	Analytical	96 %
5.	Zinc chloride	Analytical	96 %

2.5 Adsorption studies

250 mL conical flask containing varying dose of adsorbent and 50 mL aqueous solution of the metal was agitated at 150 rpm using mantle fitted with magnetic stirrer for maximum contact time of 120 minutes. The pH value of aqueous solution was kept at the optimum and separation of adsorbent from aqueous solution was done by filtration using 150 mm whatman filter paper. The filtrate was stored in sample cans and placed in refrigerator prior to analysis. The residual metal ion concentration was determined using Atomic Absorption Spectrophotometer (AAS). Amount of Mn^{2+} ion removed during the series of batch investigation was determined using the mass balance equation presented in (Ilaboya et al., 2013) as follows.

$$q = \frac{v}{m}\left[C_0 - C_e\right] \quad (1)$$

Where: q, defines the metal uptake (mg/g); C_0 and C_e: are the initial and equilibrium metal ion concentrations in the aqueous solution [mg/L] respectively; V: is the aqueous sample volume (mL) and m: is the mass of adsorbent used (g). The efficiency of metal ion removal (%) was calculated using the mass balance equation of the form (Badmus et al., 2007).

$$\text{Efficiency (\%)} = \left(\frac{C_0 - C_e}{C_0} \times 100\right) \quad (2)$$

Where: C_0 and C_e are the metal ion concentrations (mg/L) in aqueous solution before and equilibrium adsorption.

2.6 Design of experiment and process optimization

For Mn^{2+} ion adsorption, varied initial metal ion concentration of 4 – 20 mg/L, varied adsorbent dose of 0.2 – 1.0 g, varied pH of 2 – 10 and varied contact time of 24 – 120 mins for a constant adsorption temperature of 27 ± 2^0C were selected. The range and levels of the selected input variables is presented in Table 3

Table 3: Range and Levels of independent variables for Mn^{2+} ion adsorption

Independent Variables	Range and Levels				
	-2	-1	0	+1	+2
Initial metal ion concentration (mg/l)	4	8	12	16	20
pH	2	4	6	8	10
Adsorbent dose (g/L)	0.2	0.4	0.6	0.8	1.0
Contact time (minutes)	24	48	72	96	120

Using the parameters presented in Table 3, a full factorial central composite design comprising of sixteen factorial points, eight axial points and six replicates at the center point resulting in a total of 30 experimental runs as shown in Table 4 was employed to optimize the selected variables.

Table 4: Central composite design showing coded and real variables with observed and predicted Mn^{2+} ion adsorption

Experimental Runs	Coded Values of Variables				Real Values of Variables				Mn(II) Sorption Efficiency (%)	
	X_1 (mg/L)	X_2 (g/L)	X_3 (pH)	X_4 (mins)	X_1 (mg/L)	X_2 (g/L)	X_3 (pH)	X_4 (mins)	Observed	RSM Predicted
1	0	0	0	0	12.00	0.600	6.000	72.000	76.5	76.36
2	0	0	0	0	12.00	0.600	6.000	72.000	76.4	76.36
3	0	0	0	0	12.00	0.600	6.000	72.000	76.4	76.36
4	0	0	0	0	12.00	0.600	6.000	72.000	76.5	76.36
5	0	0	0	0	12.00	0.600	6.000	72.000	76.3	74.93
6	0	0	0	0	12.00	0.600	6.000	72.000	76.4	74.93
7	0	-2	0	0	12.00	0.200	6.000	72.000	75.8	74.80
8	0	+1	0	0	12.00	0.800	6.000	72.000	64.3	65.71
9	0	0	+1	0	12.00	0.600	8.000	72.000	74.3	73.41
10	0	0	-2	0	12.00	0.600	2.000	72.000	75.3	76.60
11	0	0	0	+1	12.00	0.600	6.000	96.000	65.2	64.48
12	0	0	0	-2	12.00	0.600	6.000	24.000	66.7	67.83
13	-1	0	0	0	8.000	0.600	6.000	72.000	67.1	63.33
14	-2	0	0	0	4.000	0.600	6.000	72.000	83.7	87.88
15	+2	+2	-2	+2	20.00	1.000	2.000	120.00	65.4	66.58
16	+2	+2	-2	-2	20.00	1.000	2.000	24.000	63.2	65.22
17	-2	-2	+2	-2	4.000	0.200	10.00	24.000	67.5	66.31
18	+2	-2	+2	+2	20.00	0.200	10.00	120.00	64.5	66.98
19	+2	-2	-2	-2	20.00	0.200	2.000	24.000	71.2	73.34
20	-2	+2	-2	+2	4.000	1.000	2.000	120.00	65.4	64.71
21	-2	-2	+2	+2	4.000	0.200	10.00	120.00	67.3	69.95
22	+2	+2	+2	-2	20.00	1.000	10.00	24.000	64.8	63.34
23	-2	+2	+2	-2	4.000	1.000	10.00	24.000	76.8	76.89
24	-2	-2	-2	-2	4.000	0.200	2.000	24.000	76.1	74.41
25	-2	+2	-2	-2	4.000	1.000	2.000	24.000	79.8	81.45
26	+2	+2	+2	+2	20.00	1.000	10.00	120.00	84.5	80.99
27	+2	-2	-2	+2	20.00	0.200	2.000	120.00	85.4	83.88
28	+2	-2	+2	-2	20.00	0.200	10.00	24.000	74.3	74.12
29	-2	-2	-2	+2	4.000	0.200	2.000	120.00	88.7	85.31
30	-2	+2	+2	+2	4.000	1.000	10.00	120.00	77.8	77.58

2.6 Data generation for ANN modelling

Input data employed for the training, validation and testing were obtained from series of batch experiments based on central composite design of experiment under varied initial metal ion concentration, pH, adsorbent dose and contact time. A full factorial central composite design of experiment with 6 center points and 3 replicates resulted in a total of 90 experimental runs was used as the input data. The data were randomly divided into three subsets to represent the training (60%), validation (25%) and testing (15%). The validation data was employed to assess the performance and the generalization potential of the trained network while the testing data was to test the quality of the network. To avoid the problem of weight variation which can subsequently affects the efficiency of the training process, the input and output data were normalized to a give a weight range of between 0.1 and 1.0 using genetic optimizer.

3. Results and Discussions

Scanning electron micrograph was taken in order to verify the presence of micropores. Scanning electron micrograph of raw and activated sawdust is presented in Figures 1a and 1b.

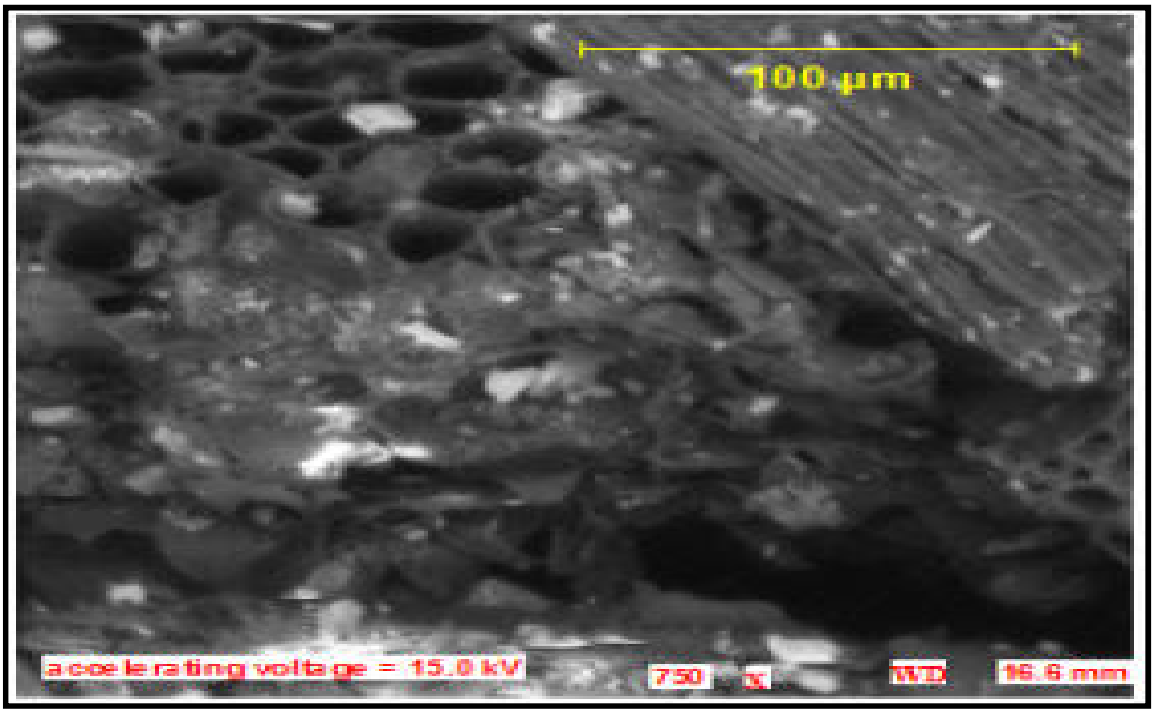

Figure 1a: Scanning electron micrograph of raw sawdust

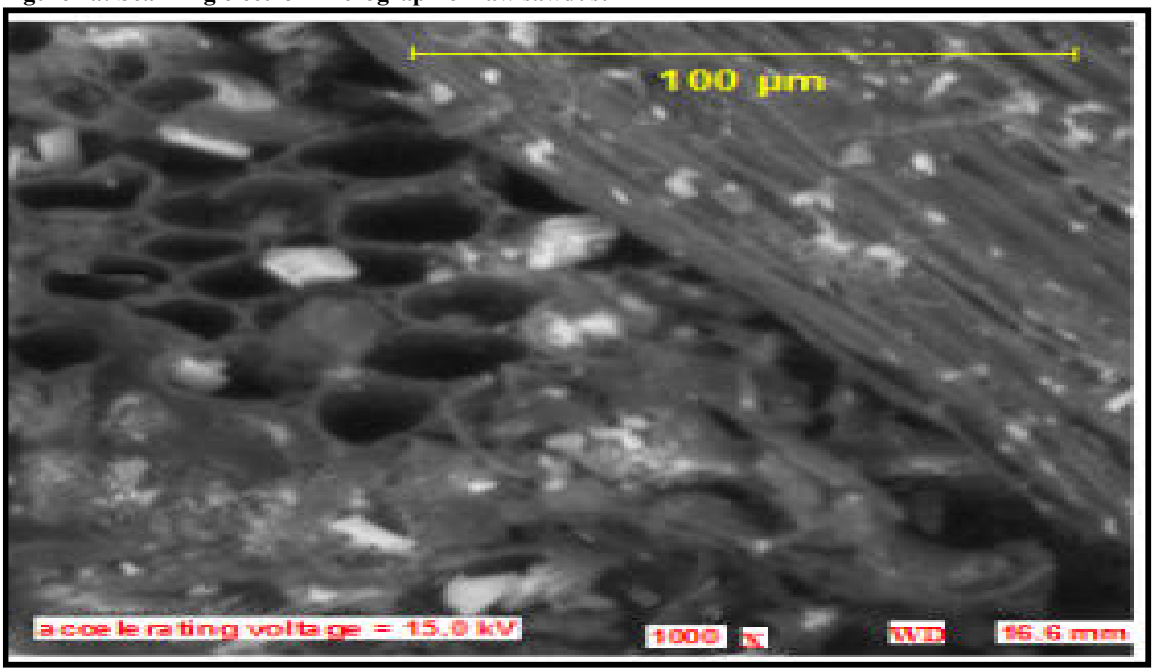

Figure 1b: Scanning electron micrograph of activated sawdust

Larger number of microporous structures observed with activated sawdust indicate a higher surface area hence better adsorption property. This claim is based on the fact that as biosorbent materials present larger numbers of microporous structure, they adsorb higher amount of nitrogen, which resulted to higher surface area and higher adsorption properties. Insight into the nature of functional groups that make up the surface of adsorbent would create a better picture on the adsorption potentials of the material. To identify the functional group's present on the surface of sawdust, Fourier Transform Infrared (FTIR) spectroscopy was used. Figure 2 shows the Fourier Transform Infrared (FTIR) spectra of raw sawdust.

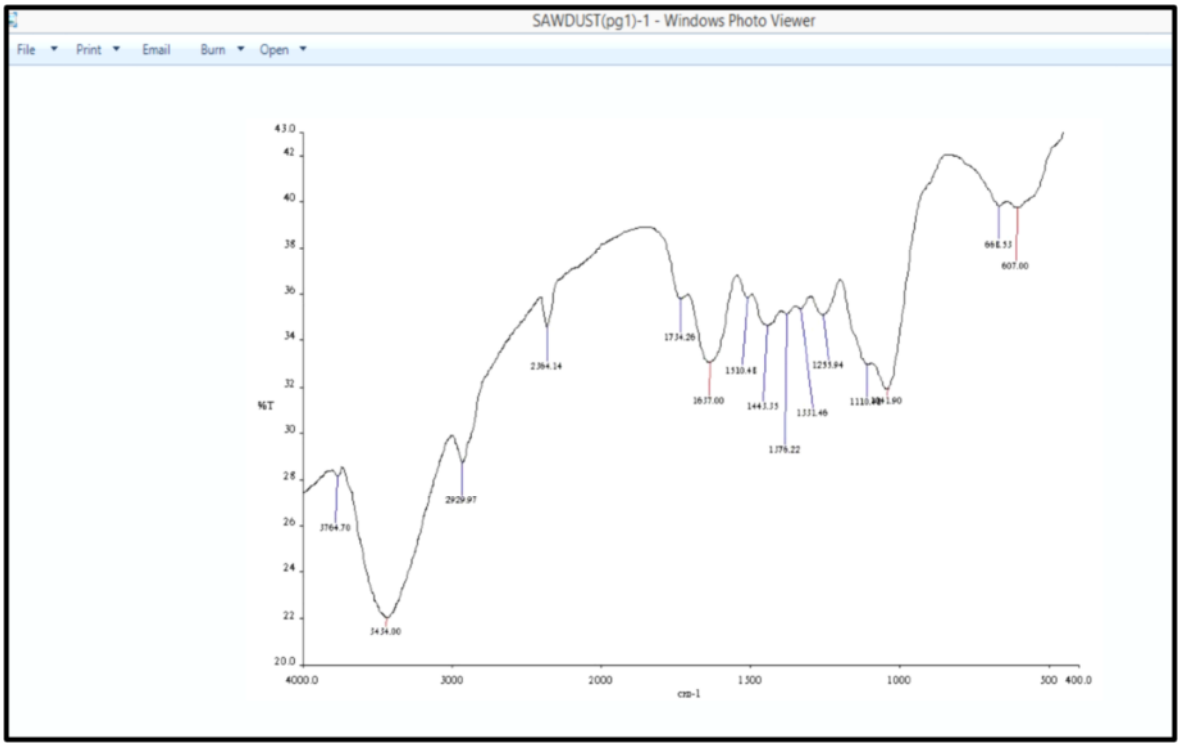

Figure 2: FTIR spectra of raw sawdust

To identify the functional group based on the FTIR spectra, absorption assigned bands from the work of previous researchers was employed to analyzed the spectrum of raw sawdust and result obtained is presented in Table 5

Table 5: Interpretation of FTIR spectrum of raw sawdust

S/No	Wave Number (cm^{-1})	Bond Source
1	3434.00	O-H stretching mode of hydroxyl groups N-H stretch
2	1637.00	N-H bending of amides, C ☰O stretch, carbonyl
3	1510.42	Quinonic and carboxylate groups, N-H bending, C ☰O stretch
4	1445.55	CH_2 and CH_2 bend, pyrones and aromatic group
5	1376.22	Organic phosphate, (P ☰O stretch)
6	1110.24	Organic siloxane or silicone, Si-O-C stretch
7	661.58	Disulphides (C - S stretch)

Result of Table 5 revealed that O-H stretching of hydroxide group and N-H stretching of amides are responsible for Mn^{2+} ion absorption onto zinc chloride activated sawdust. When the effect of varied adsorbent dose was studied at constant initial Mn^{2+} ion concentration of 20mg/l, optimum pH of 7.0, and adsorption contact time of 120minutes under a constant stirring speed of 150rpm, result obtained is presented in Figure 3

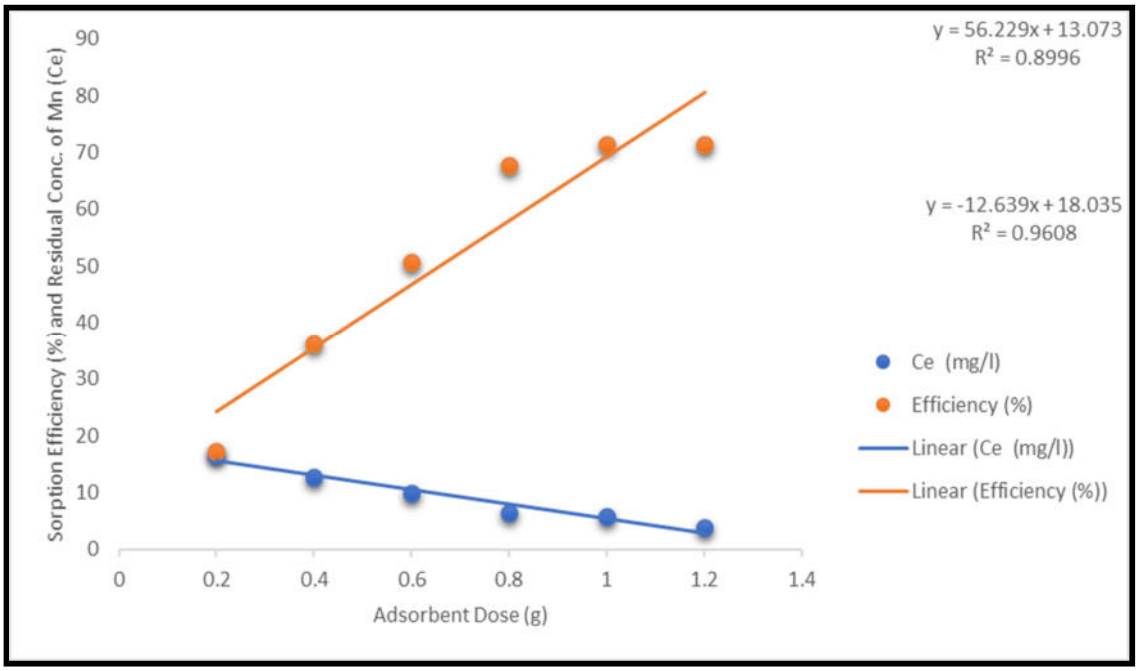

Figure 3: Effect of adsorbent dose on the sorption of Mn^{2+}

Adsorption efficiency increases with adsorbent dose as observed in Figure 3 reaching a maximum efficiency of 71.40%. Higher dosage of adsorbent will increase adsorption efficiency due to more active site and functional groups on the adsorbent surface which the metal could interact with. These functional groups are important in the formation of van der Waals bonding since they played a major role in binding metals to adsorbents during adsorption process. To predict the sorption of Mn^{2+} from wastewater onto activated sawdust, predictive model such as artificial neural network (ANN) is required. Input and output data training resulting in the design of network architecture is of paramount importance in the application of neural network to data modeling and prediction. To obtain the optimal network architecture that possesses the most accurate understanding of the input and output data, two factors were considered. First is the selection of the most suitable training algorithm and secondly, the number of hidden neurons. Based on this consideration, different training algorithm and hidden neurons were selected and tested to determine the best training algorithm and optimum number of hidden neurons that will produce the most suitable network architecture needed to predict the sorption of Mn^{2+} ion onto zinc chloride activated sawdust. Selectivity of the algorithm was based on the calculated coefficient of determination (r^2) and the mean square error (MSE). Table 6 shows the performance of the different training algorithm tested.

Table 6: Selection of optimum training algorithm for ANN modelling

Training Algorithm (Learning Rule)	Training MSE	Cross Validation MSE	R-Square (r^2)
Gradient information (Step)	0.06782	0.06038	0.720
Gradient and weight change (Momentum)	0.05891	0.06628	0.732
Gradient and rate of change of gradient (Quick prop)	0.04489	0.03802	0.733
Adaptive step sizes for gradient plus momentum (Delta Bar Delta)	0.04247	0.00507	0.775
Second order method for gradient (Conjugate gradient)	0.05072	0.08012	0.654
Improved second order method for gradient (Levenberg Marquardt)	0.00018*	0.00027*	0.987*

Result of Table 6 revealed that improved second order method of gradient also known as Levenberg Marquardt Back Propagation training algorithm was the best learning rule and was adopted in designing the network architecture. To determine the optimum number of hidden neurons, different number of hidden neurons were selected to create a trained network using Levenberg Marquardt Back Propagation training algorithm. Performance of the trained network was assessed using the computed mean square error (MSE) and coefficient of determination

(r^2). The number of hidden neuron's corresponding to the lowest MSE and the highest r^2 as presented in Table 7 was selected to design the network architecture.

Table 7: Selection of optimum number of hidden neurons for ANN modelling

S/No	Number of Hidden Neurons	Training MSE	Cross Validation MSE	R-Square (r^2)
1	2	0.0033	0.0072	0.65
2	3	0.0057	0.0133	0.77
3	5	0.0066	0.0692	0.81
4	8	0.0059	0.1107	0.79
5	10	0.0003	0.0004	0.94

Based on the results of Table 6 and 7, Levenberg Marquardt Back Propagation training algorithm having 10 hidden neurons in the input layer and output layer was used to train a network of 4 input processing elements, namely; adsorbent dose, contact time, pH and initial metal ion concentration and 1 output processing element (sorption efficiency). The input layer of the network uses the hyparbolic targent (tan-sigmoid) transfer function to calculate the layer output from the network input while the output layer uses the linear (purelin) transfer function. The number of hidden neuron was set at 10 neurons per layer and the network performance was monitored using the mean square error of regression (MSEREG). The structural definition of the ANN platform include; training function (TRAINLM), adaptation learning function (LEARNGDM) and performance function (MSEREG). Trainlm is a network training function that updates weight and bias values according to Levenberg-Marquardt optimization. Trainlm is often the fastest back propagation algorithm in the MATLAB toolbox, and is highly recommended as a first-choice supervised algorithm, although it does require more memory than other algorithms. For optimum performance and accurate prediction, a learning rate of 0.01, momentum coefficient of 0.1, target error of 0.01, analysis update interval of 500 and a maximum training cycle of 1000 epochs was used. Using these parameters, an optimum neural network architecture presented in Figure 4 was generated to predict the sorption of Mn^{2+} ion onto activated sawdust using back propagation neural network.

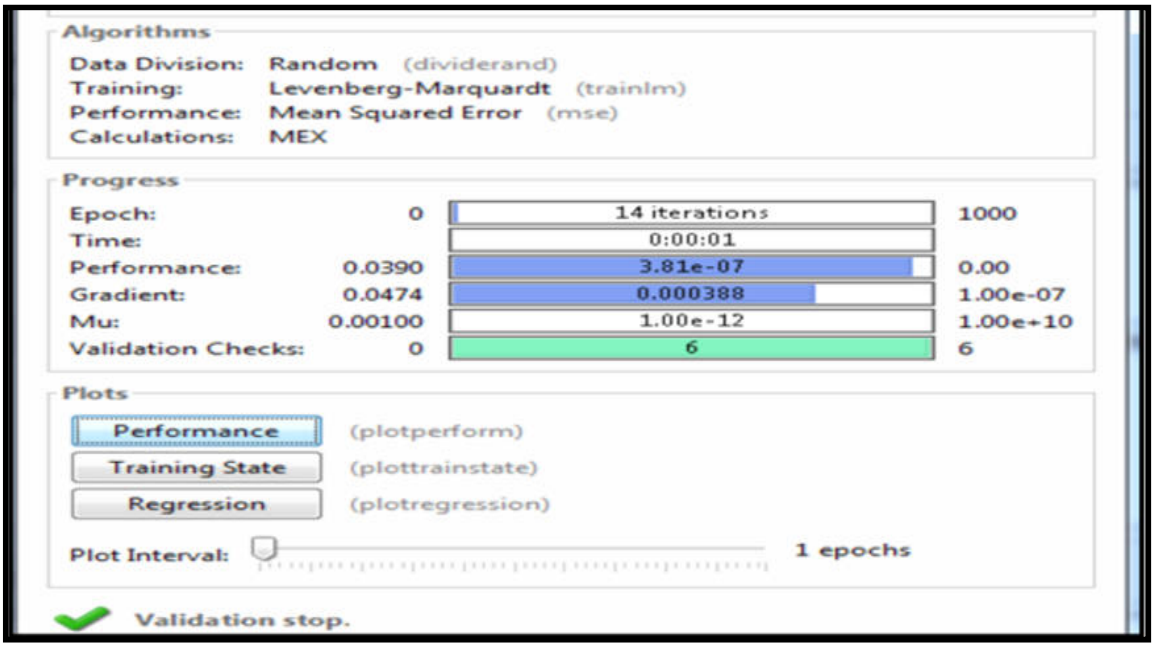

Figure 4: Network training diagram for predicting the sorption of Mn^{2+} ion

From the network training diagram of Figure 4, it was observed that the network performance was significantly good with a performace error of 3.81e-07 which is far lesser than the set target error of 0.01. The maximum number of iteration needed for the network to reach this performance was observed to be 14 iterations which is also lesser than the initial 1000 epochs. The gradient function was calculated to be 0.000388 with a training gain (Mu) of 1.00e-12. Validation check of six (6) was recorded which is expected since the issue of wieght biased had been addressed via normalization of the raw data. A performance evaluation plot which shows the progress of training,

validation and testing is presented in Figure 5

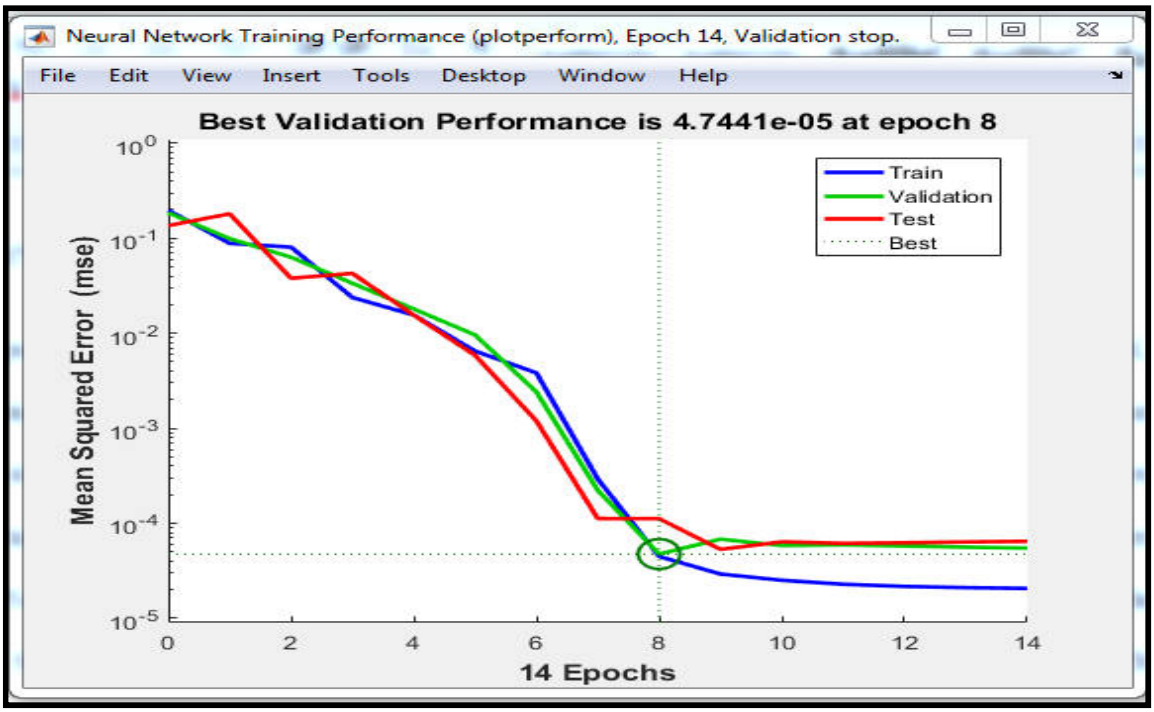

Figure 5: Performance curve of trained network for predicting the sorption of Mn^{2+} ion

From the performance plot of Figure 5, no evidence of over fitting was observed. In addition, similar trend was observed in the behaviour of the training, validation and testing curve which is expected since the raw data were normalized before use. Lower mean square error is a fundamental criterion used to determine the training accuracy of a network. An error value of 4.7441e-05 at epoch 8 is an evidence of a network with strong capacity to predict the sorption of Mn^{2+} ion onto zinc chloride activated sawdust. The training state, which shows the gradient function, the training gain (Mu) and the validation check, is presented in Figure 6

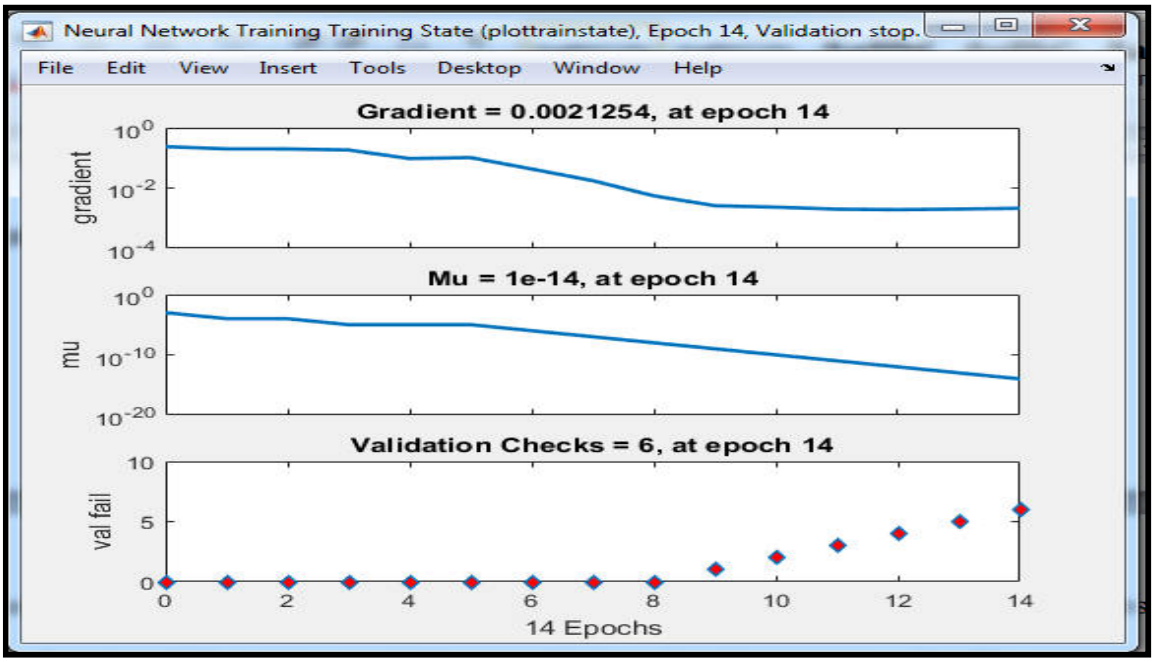

Figure 6: Neural network training state for predicting the sorption of Mn^{2+} ion

Back propagation is a method used in artificial neural networks to calculate the error contribution of each neuron after a batch of data training. Technically, the neural network calculates the gradient of the loss function to explain the error contributions of each of the selected neurons. Lower error is better. Computed gradient value of 0.0021254 as observed in Figure 6 indicates that the error contributions of each selected neuron are minimal. Momentum gain (Mu) is the control parameter for the algorithm used to train the neural network. It is the training gains and its value must be less than unity. Momentum gain of 1.0e-14 shows a network with high capacity to predict the sorption of Mn^{2+} ion onto activated sawdust.

The regression plot which shows the correlation between the input variables (adsorbent dose, pH, contact time and initial metal ion concentration) and the target variable (sorption efficiency) coupled with the progress of training, validation and testing is presented in Figure 7

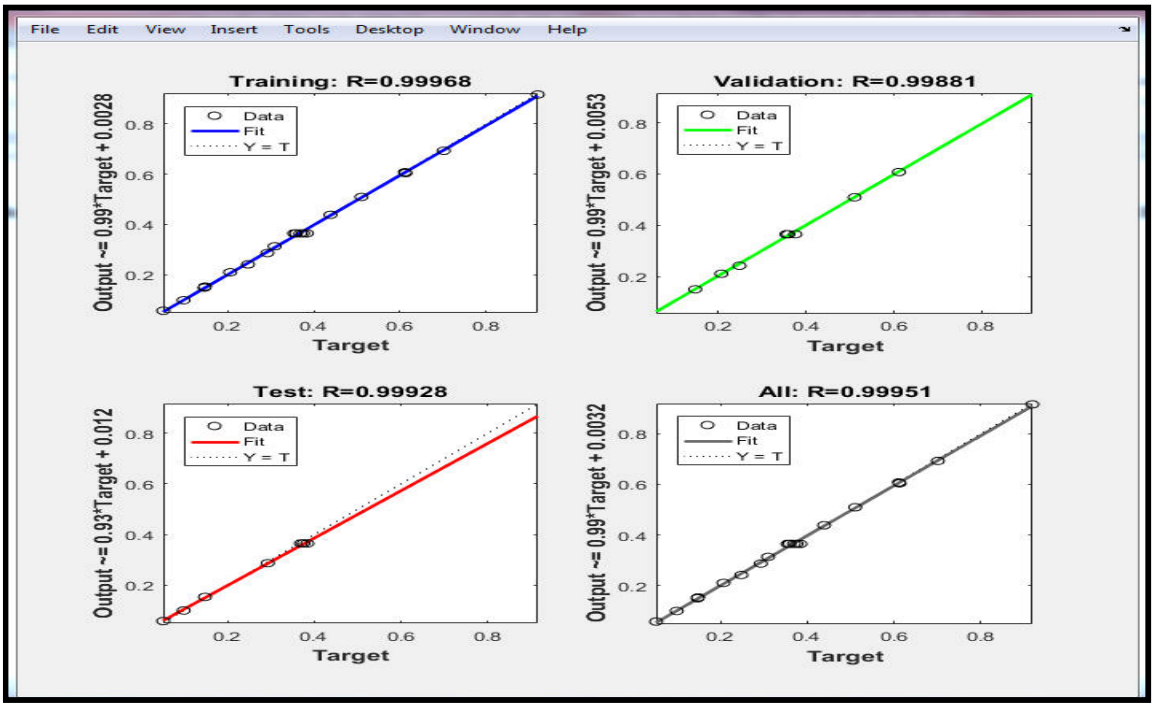

Figure 7: Regression plot showing the progress of training, validation and testing

Based on the computed values of the correlation coefficient (R) as observed in Figure 7, it was concluded thet the network has been adequately trained and can be employed to predict the sorption of Mn^{2+} ion onto solid adsorbent prepared from sawdust. To test the reliability of the trained network, the network was thereafter employed to predict its own values of sorption efficiency using the same set of input parameters (adsorbent dose, pH, contact time and initial metal ion concentration) generated from the central composite design. Based on the observed and the predicted values, a regression plot of outputs was thereafter generated as presented in Figure 8.

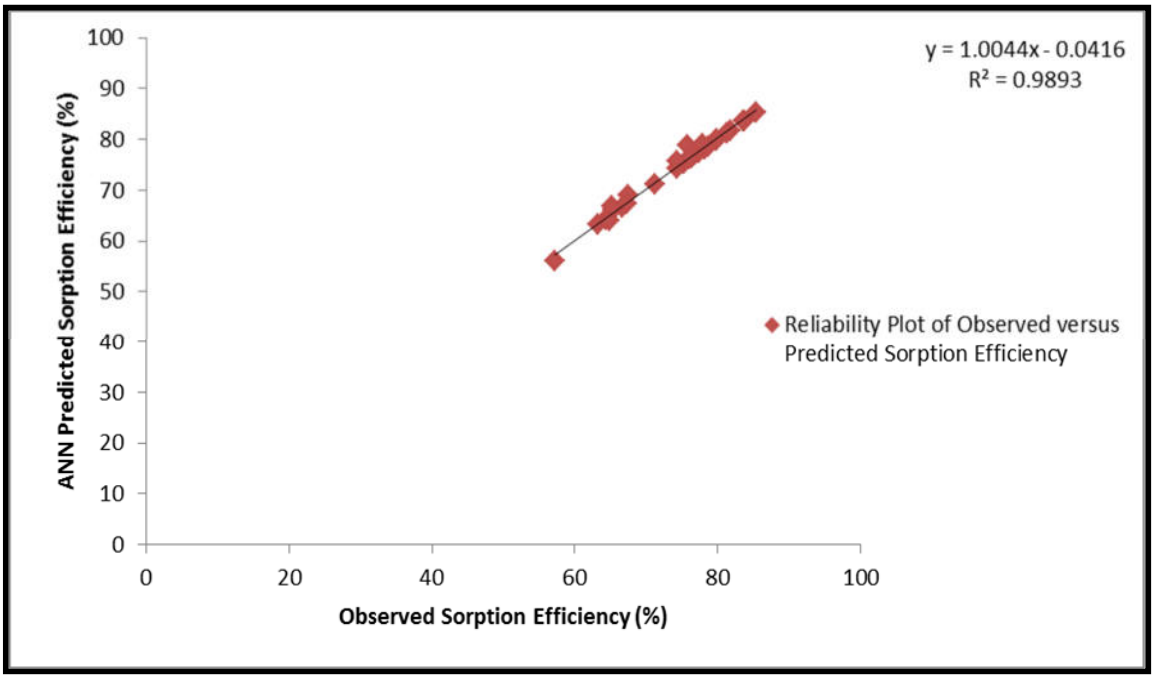

Figure 8: Regression plot of observed versus predicted sorption efficiency

Coefficient of determination (r^2) value of 0.9893 as observed in Figure 8 was employed to draw a conclusion that the the trained network can be used to predict the sorption of Mn^{2+} ion beyond the limit of experimentation.

4. Conclusion

The performance of statistical design of experiment and artificial neural network (ANN) in predicting the sorption of divalent metal ion onto zinc chloride activated sawdust has been successfully implemented and will form the bases for future research in related areas. The optimal training algorithm and number of hidden neurons that will yield a better training of input and output data has been adequately determined to ensure a more effective application of neural network as a predicitive tool for environmental studies and management.

5. References

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