The Use of Neural Network for Modeling of Waste Activated Sludge for Solubilization and Biodegradability Enhancement Using Ozonolysis Process

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***Abstract*—This study investigated the application of ozonolysis pre-treatment of waste activated sludge for solubilization and biodegradability enhancement of municipal waste activated sludge (WAS). The effect of operational parameters such as initial pH, ozone dosage and ozone duration were studied to optimize the conditions of total suspended solids (TSS) reduction percentage. Based on the experimental results that optimum ozone dosage of 90 mg/min for ozonation duration of 60 min and initial pH of 11 were found significant to save on the energy requirements and carbon contents of the pre-treated WAS. The configuration of the backpropagation neural network (NN) was three-layer (3:10:1) with tangent sigmoid transfer function (*tansig*) at hidden layer with 10 neurons, linear transfer function (*purelin*) at output layer and Levenberg-Marquardt backpropagation training algorithm (LMA). NN predicted results are very close to the experimental results with correlation coefficient (R) of 0.997 and MSE 0.1050. The results showed that neural network modeling could effectively predict and simulate the behavior of the ozonolysis process.**

Keywords: Neural network; Ozonolysis; waste activated sludge; biodegradability enhancement, solubilization

I. Introduction

Waste activated sludge (WAS) is considered as a major by-product of the activated sludge treatment process [1]. The WAS consists mainly of biomass from cell growth and decay throughout activated sludge treatment (AST) process as well as traces of odorous and volatile organic compounds, and toxic chemicals. If is not stabilized can pose the risk of secondary environmental pollution [2]. The enhancement of anaerobic digestion (AD) process for WAS stabilization, reduction, and energy recovery in the form of methane gas has evoked great interest [3]. However, AD of WAS suffers high retention times, low methanogenic production and an overall 30-35% dry solids degradation [4]. The insoluble and biorecalcitrant proteinaceous biomass cell walls and extracellular polymeric substances (EPS) in WAS hinders the initial rate determining hydrolysis step of AD. During hydrolysis, enzyme-mediated solubilization of insoluble organic materials, the rapture of hard cell walls and degradation of EPS occur releasing biodegradable organic compounds for the ensuing acidogenesis step [5]. A long retention time resulting in the consumption of a large amount of energy is, therefore, required for the hydrolysis of WAS, limiting the effective application of AD in the treatment and stabilization of WAS [6]. To enhance the AD process and meet the strict regulation on environmental protection, implementation of a pre-treatment method, that would improve the hydrolysis step is mandatory [2]. Various methods for sludge solubilization including chemical treatment using acids and alkalis, mechanical disintegration, and advanced oxidation processes (AOPs) have been successful applied to date [6]. Ozonolysis is a promising technology for the pre-treatment of WAS before anaerobic digestion for stabilization and energy recovery. Among these methods, ozonolysis, which is an AOP is considered most effective for sludge disintegration, because it does not lead to significant increase in salt concentration and has no chemical residues in comparison to order pre-treatment techniques [4]. Through ozonation, organic matter can be released from the sludge into the solution as a result of the breaking and solubilization of the hard cellular membrane [6]. The role of ozonolysis pre-treatment is to partially oxidize the biorecalcitrant component of WAS to release biodegradable products [2]. Through ozonolysis, low soluble volatile organic compounds (VOCs), can be oxidized and solubilized as well thus contributing to the improved biodegradability. To avoid over-oxidation of the released biodegradable components, the ozonolysis dose and time have to be optimized [4, 7]. The treatment of wastewater by AOPs is quite complex, since the process is influenced by various factors. Due to complexity of the process, it is difficult to be modeled and simulated using convention mathematical modeling. NNs are now applied in numerous areas of science and engineering and considered as promising tool because of their simplicity towards simulation, prediction and modeling [8]. NNs were applied to solve environmental engineering problem in biological and physico-chemical wastewater treatment [9]. However, few studies on application of NN in advanced oxidation processes have been reported [9,10]. The present work examined the implementation of NN for the prediction and simulation of ozonolysis process for the pre-treatment of

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waste activated sludge for solubilization and biodegradability enhancement of municipal waste activated sludge. The NN modeling outputs were compared with the experimental data.

II. Materials and Methods

The following chemicals: Sodium thiosulphate, phenol, bovine serum albumin (BSA), methanol, phosphoric acid, Bradford reagent, Coomassie brilliant blue, potassium iodide, sulphuric acid, hydrochloric acid, silver sulfate, potassium dichromate, starch, and sodium hydroxide were analytical grade and all purchased from Merck Limited, South Africa. The WAS was sourced from a secondary settler of a local municipal wastewater treatment plant in Vanderbijlpark, South Africa. The WAS was filtered through a 450µm microfilter to remove large solid particles then further analyzed to determine its physical and chemical characteristics. The waste activated sludge was treated in a 4 L ozone reactor batch-wise for sludge solubilization and improved biodegradability. For every batch experiment, 2 L of WAS was transferred to the reactor then subjected to ozonolysis. Gaseous ozone was generated from air using an ozone generator coupled to an air compressor. The ozone generator employed the corona discharge principle, where a high voltage corona mechanism converts the oxygen in air to ozone [11]. The resulting ozone air mixture was then bubbled in an up-flow mode in the ozone reactor via a gas diffuser. The ozone concentration in the mixture was determined following a volumetric method based on the reaction between potassium iodide and ozone [4]. Previous studies have shown that the ozonolysis process is dependent on parameters such as initial pH, ozone dosage and reaction time [7]. To obtain different ozone dosage, the ozone flow rate was controlled using a manual valve and the flow measured using an air rotameter. For instance, an air flow rate of 2 L/min generated air-ozone mixture with a dosage of 45 mg O_3 per minute supplied to the reactor. Air flow rates of 4, 6 and 8 L/min generated dosages of 90, 135 and 180 mg O_3 per minute. The unutilized ozone gas from the reactor was destroyed by passing through the KI solution [4]. The schematic diagram of the ozonolysis is given in Fig. 1.

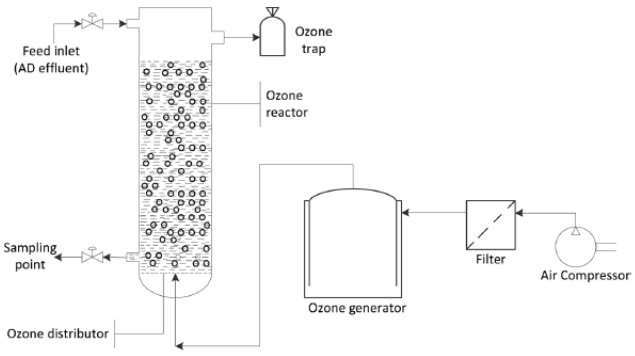

Fig. 1. Schematic representation of the ozone unit [7]

The average oxidation state (AOS) of the WAS during ozonolysis was used to estimate the degree of oxidation of the substrate containing the oxidation by-products as well as the initial compounds. The AOS was calculated by [4]:

$$AOS = 4 \times \frac{TOC - \frac{12}{32} \times COD_T}{TOC} \quad (1)$$

Where COD_T and TOC are the total chemical oxygen demand and total organic carbon, respectively. The values for AOS vary from -4 (for CH_4 the most reduced state for C) to +4 (for CO_2 the most oxidized sate for C). In an oxidation process, however, the increment observed in AOS (ΔAOS), can be calculated using Eq. (2).

$$\Delta AOS = AOS_f - AOS_i = 4 \times \left(\frac{COD_{Ti}}{DOC_i} - \frac{COD_{Tf}}{DOC_f} \right) \times \frac{12}{32} \quad (2)$$

Where i and f refer to initial and final values, respectively. The ΔAOS is of more importance as it indicates the degree of oxidation [11, 12].

The amount of ozone in the inlet and outlet gas streams was determined for the different flow rates by titrating the ozonated KI solutions with potassium thiosulphate solution. Ozone transfer, which is the amount of ozone used up in the reaction was determined as follow:

$$O_3T = \frac{O_3U}{O_3S} \times 100 \quad (3)$$

Where, O_3T, O_3S and O_3U are the ozone transfer (%), supplied and utilized ozone (mg), respectively. The utilized ozone was determined from the difference obtained between the ozone supplied to the reactor and the ozone coming out the reactor (off gas). Aliquot samples were withdrawn and analyzed for pH, total organic carbon (TOC), COD, BOD, total suspended solids (TSS), total solids (TS), dissolved organic carbon (DOC) total volatile solids (VSS), nitrates, phosphates, proteins, carbohydrates and chlorides. The COD, BOD, TSS, TS and VSS were analyzed according to the standard methods of analysis [4].

A. Neural network (NN)

Neural network is an information processing system that is inspired by the learning algorithm biological nervous systems. Neural networks are known for their ability of learning, simulation and prediction of data. NNs are nonlinear statistical techniques and very flexible [8]. The network consists of numerous individual processing units called neurons and commonly interconnected in a variety of structures. The strength of these interconnections is determined by the weight associated with neurons. The multilayer feed-forward net is a parallel interconnected structure consisting of input layer and includes independent variables, number of hidden layers and output layer [9, 13]. In this study, a three-layered (3:10:1) backpropagation algorithm neural network with tangent sigmoid transfer function (*tansig*) at hidden layer and a linear transfer function (*purelin*) at output layer was used. Neural network Toolbox V4.0 of MATLAB mathematical software was used for TSS reduction percent prediction. Data (120 experimental sets) were obtained from our previous study [4] and were divided into input matrix and target matrix. The input variables were ozone dosage, initial pH and ozone duration. The corresponding TSS reduction percent was used as a target. The data sets were divided into training, validation and test subsets, each of which contained 60, 30 and 30 data, respectively.

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III. Results and Discussion

A. Neural Network model

Three important aspects that must be determined in design procedure of NN are as follows [14]:

- Data distribution in three subsets (training, validation and testing).
- Selection of neurons' transfer functions.
- Selection of NN structure
- Selection of training algorithm.

A.1. Data distribution (training, validation and testing)

Neural network multilayer perceptron (MLP) feed forward model was used in this study. The total of 120 experimental data were randomly divided into three subsets of training, validation and testing for developing NN model. Sixty training data were used to update the network weights and biases. In order to check the generality of network prediction and to prevent the data overfitting, 30 validation data were applied. In the first few epochs of training, errors of both training and validation data are reduced. After several epochs, the error of training data decreases while that of validation data increases. As a result, the network is overtrained and its generality decreases. Hence, the training process must be continued until the validation data error decreases. Testing data set are used to test the generality of trained network via unseen patterns (experimental data which are not used in training procedure). The network generalizes well when it sensibly interpolates these new patterns. Termination of training procedure at a proper time, when the minimum validation error is achieved, results in a generalized predictor network. The data set were used to feed the optimized network in order to test and validate the model.

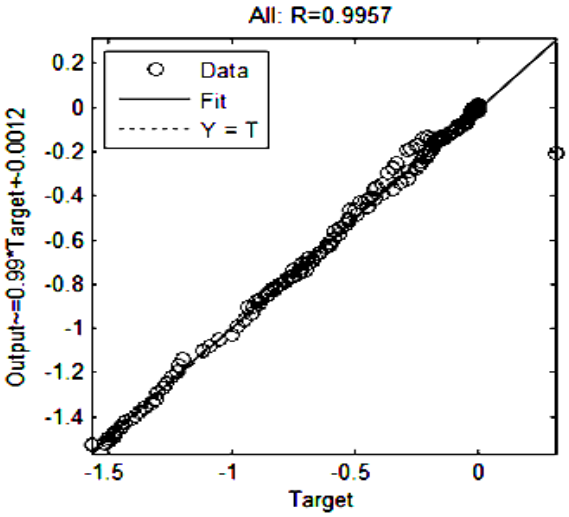

Fig. 2. Comparison between predicted and experimental values of the output

Fig. 2 shows a comparison between experimental and predicted values for All (training, validation and testing) using NN model. The figure contains two lines, one is the perfect fit Y = T (predicted data = experimental data) and the other is the best fit indicated by a solid line with best liner equation Y = 0.99T + 0.0012, correlation coefficient (R) 0.9957.

A.2. Training algorithm and transfer function

The MLP networks were created in the neural network toolbox of MATLAB with *newff* function. Performances of different training algorithms were studied for a specified network with three layers. Due to the convergence speed and the performance of network to find better solution, the Levenberg-Marquardt training method was selected as a proper training algorithm in agreement with the literature [8, 10]. Another important factor in NN design is the type of transfer functions. NNs owe their nonlinear capability to the use of nonlinear transfer functions [14]. Different transfer functions can be used for neurons in the different layers. Different transfer functions were examined in each layer, separately and with respect to the mean squared error (MSE) of testing data, the proper transfer functions were chosen. MSE was calculated using Eq. (4):

$$MSE = \frac{\sum_N \left(DR_{cal} - DR_{\exp}\right)^2}{N} \quad (4)$$

Where DR_{cal} and DR_{exp} denote calculated and experimental values, respectively. N is the number of validation and training data.

The most widely used criteria including MSE, correlation coefficient (R), and mean squared relative error (MSRE) for training, validation and testing data sets are presented in Table I.

TABLE I
Stastical criteria for evaluation of NN model

	Training data	Validation data	Testing data	Total NN
MSE	0.0170	0.2580	0.3490	0.1050
R	0.9940	0.9940	0.9960	0.9956
MSRE	0.0030	0.0010	0.0120	0.0040

In probability theory and statistics, R indicates the strength and direction of a linear relationship between two variables. In general statistical usage, R refers to the departure of two variables from independence. A number of different coefficients are used for different situations. The best known is the Pearson product-moment correlation coefficient as follows:

$$R = \frac{\sum_N \left(DR_{cal} - DR_{cal,ave}\right)\left(DR_{\exp} - DR_{\exp,ave}\right)}{\sqrt{\sum_N \left(DR_{cal} - DR_{cal,ave}\right)}\sqrt{\sum_N \left(DR_{\exp} - DR_{\exp,ave}\right)}} \quad (5)$$

R^2 can have only positive values ranging from R^2 =+1.0 for a perfect correlation (positive or negative) down to R^2 = 0.0 for a complete absence of correlation. The advantage of R is that it provides ta measure of the strength of the correlation. It can be said that R^2 represents the proportion of the data that is the closest to the line of best fit.

Another measure of fit is MSRE which is calculated by the following Eq. (6).

$$MSRE = \frac{1}{N}\sum_N \left(\frac{DR_{cal} - DR_{\exp}}{DR_{\exp}}\right)^2 \quad (6)$$

According to data presented in Table 1, excellent fitness of NN predicted values with experimental data was confirmed. Among different transfer functions available in MATLAB. Log sigmoid function was selected for all neurons due to its better prediction performance than other transfer functions. The log sigmoid function is bounded between 0 and 1, so the input and output data should be normalized to the same range as the transfer function used. In other words, the logarithmic sigmoid transfer function gives scaled outputs (DR) in this range (0-1).

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A.3. NN structure

Network structure has significant effects on the predicted results. The number of input and output nodes, as mentioned before, is equivalent to the number of input and output, respectively. However, the optimal number of hidden layers and the optimal number of nodes in each layer, are case dependent and there is no straightforward method for determination of them. Neural network feed forward with one hidden layer and sufficiently large neurons can map any input to each output to an arbitrary degree of accuracy. However, Flood and Kartam [15] reported that many functions are difficult to approximate well with one hidden layer. They revealed that use of more than one hidden layer provides greater flexibility and enables the approximation of complex functions with fewer neurons. Baughman and Liu [16] found out that adding a second hidden layer improves the network prediction capability significantly without having any detrimental effects on the generalization of the testing data set. However, adding a third hidden layer results in a prediction capability similar to that of two hidden layer network, but it requires longer training times due to its more complex structure. In this study, the configuration of the backpropagation neural network (NN) giving the smallest mean square error (MSE) was three-layer (3:10:1). Neural network with tangent sigmoid transfer function (*tansig*) at hidden layer with 10 neurons, linear transfer function (*purelin*) at output layer and Levenberg-Marquardt backpropagation training algorithm (LMA) was adopted (see Fig.3).

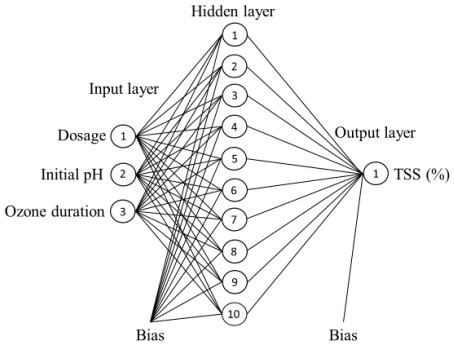

Fig. 3. Optimized NN structure

B. Characterization of waste activated sludge

The physical and chemical characteristics of the obtained waste activated sludge are given Table II. The WAS had a soluble COD (COD_S) of 1400 mg/L which was 20% of the total COD (COD_T) indicating that up to 80% of the COD was in the suspended solids. The DOC value of 122 mg/L also indicated a low concentration of the organic compounds in soluble form. With the DOC of 122 mg/L, a soluble COD value of 600 mg/L was expected. However, the higher soluble COD value of 1408 mg/L reported was partly contributed to by the oxidation of ammonia, soluble proteins (20 mg/L) and other soluble inorganic compounds present in the WAS. The BOD_S:COD ration of 0.1 confirmed the poor biodegradability of the WAS. The low biodegradability is attributed to the proteinaceous biomass cell wall which hinders the rate determining hydrolysis step of AD. Therefore, ozonolysis should be applied for the solubilization of the biorecalcitrant biomass cells.

TABLE I
PHYSICOCHEMICAL CHARACTERISTICS OF MUNICIPAL WASTE ACTIVATED SLUDGE [4]

Parameter	Value
pH	6.7
TSS	12.4
CODT	6860
CODS	1408
DOC	122
BODs	901
TS	65.3
VSS	42.1
BOD5:COD	0.1
Sulphate	1.8
Phosphate	67
Chloride	55
Soluble proteins	20

C. Effect of ozone dosage

Ozone dosage is very important in optimization the ozonolysis process [4]. The effect of ozone dosage is achieved at varying ozone flowrate. Low ozone dose would achieve low solubilization while an over-dose would lead to mineralization of already solubilized organics and a high concentration of ozone in the off-gas. As a result, the process will not be economical. After 60 min of ozonolysis, the highest TSS reduction of 53% (12.4-5.6 mg/L) was achieved at a flow rate of 90 mg/min, while the lowest flow rate of 45 mg/min removed 47% of TSS. A corresponding observation was made with the evolution of DOC with the highest increase of 25% (from 120 to 152 mg/L) attained at the 90mg/min flow rate. An increase in ozone flow rate from 45 to 90 mg/min led to an increase in ozone concentration in the reactor which adequately dissolved the organic suspended solids leading to the reduced TSS.

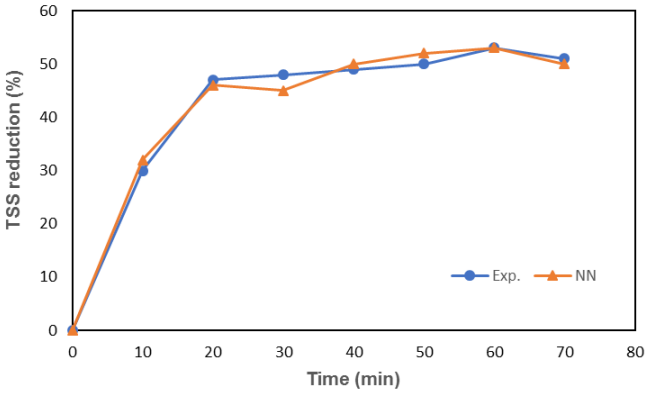

Fig. 4. Comparisons between NN outputs and experimental data for flow rate 45 mg/min

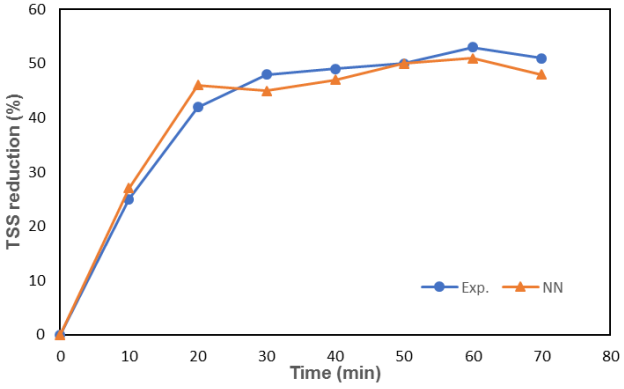

Fig. 5. Comparisons between NN outputs and experimental data for flow rate 90 mg/min

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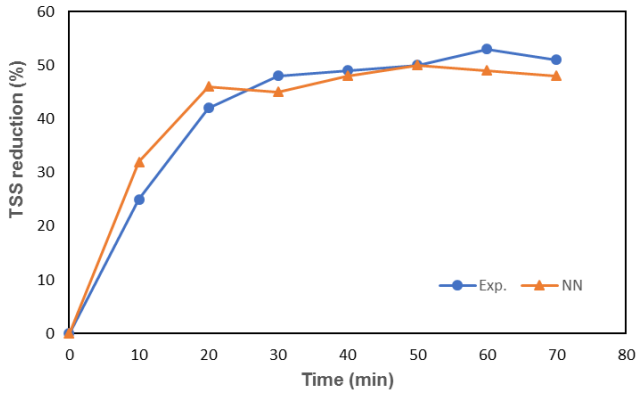

Fig. 6. Comparisons between NN outputs and experimental data for flow rate 135 mg/min

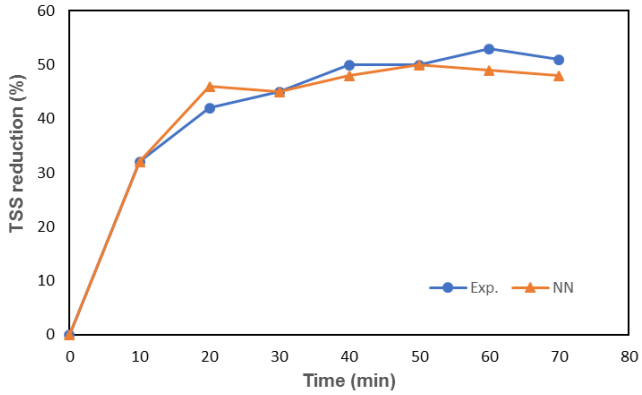

Fig. 7. Comparisons between NN outputs and experimental data for flow rate 180 mg/min

However, as the flow rate increased further, the TSS reduction decreased despite the increasing amount of supplied O_3 molecules. At high ozone flow rates above the optimal dose, the supplied gas forms larger bubbles which significantly reduces gas hold-up resulting in reduced contact time between O_3 and the suspended solids leading to reduced efficiency [17]. In terms of the relation between the experimentation data and the predicted values of TSS reduction by the NN model, Figs. 4, 5, 6 and 7.

D. Effect of initial pH

The ozonolysis process is highly affected by the pH of the substrate [4]. Highest TSS reduction of 47% (12.4-6.5 mg/L) was obtained at pH 11 as compared to 36% and 19% reductions at pHs of 7 and 3, respectively. Similarly, the highest increase in DOC evolution of 55% (122-190 mg/L) was achieved at pH 11 and the lowest value of 23% at pH 3. The reduction in TSS and increase in DOC are attributable to the solubilization of the suspended organic matter. Under acidic conditions, ozone selectively attacks parts of organic compounds containing C=C double bonds, aromatic rings, and negatively charged centers/sites containing atoms such as N, P, O, S [18]. The selective attack leads to partial solubilization hence the low increase in DOC [4]. The performance of ozone is superior under alkaline conditions because its half-life increases from 15 min, which it decomposes leading to the formation of the highly react with all the organic compounds hence the highest increase in DOC at occurred pH 11 [11]. In terms of the relation between the experimentation data and the predicted values of TSS reduction by the NN model, Figs. 8, 9 and 10.

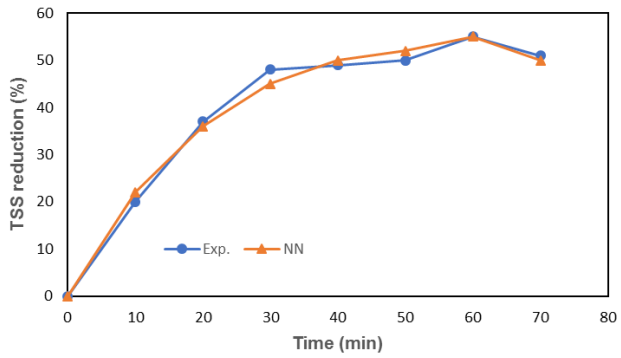

Fig. 8. Comparisons between NN outputs and experimental data for pH 3

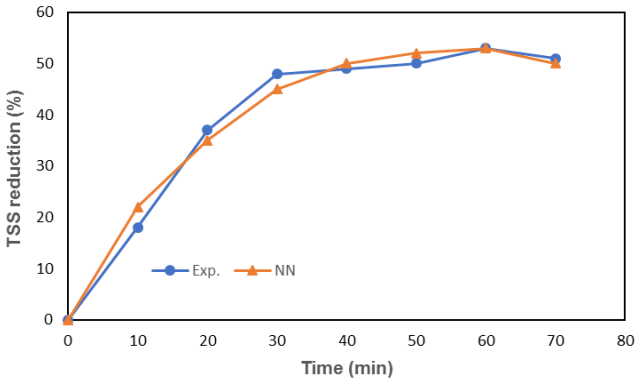

Fig. 9. Comparisons between NN outputs and experimental data for pH 7

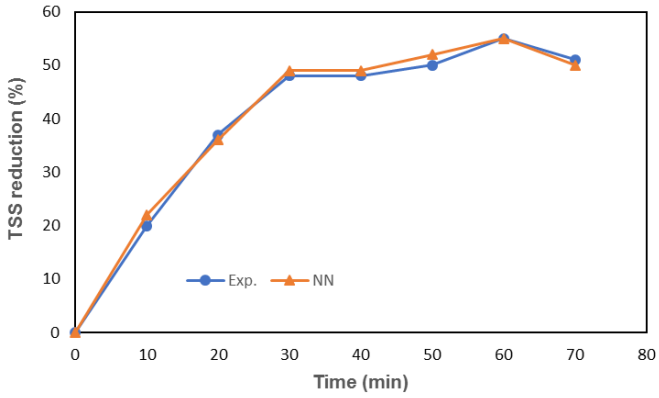

Fig. 10. Comparisons between NN outputs and experimental data for pH 11

E. Change in COD, biodegradability, and ozone transfer

Ozonolysis of the WAS carried out at the optimal conditions of pH 11 and ozone flow rate of 90 mg/min. The work done by Otieno et al. [4] shown the changes in total COD (COD_T) and soluble (COD_S), pH, BOD5:COD ratio, AOS, and ozone transfer. The COD_T decreased by 42% after 2h of ozonolysis. Consequently, the COD_S increased by 41% after 60 min of ozonolysis then reduced with additional ozonolysis time. The decrease in COD_T and the subsequent increase in COD_S was attributed to the solubilization of the suspended solids (sludge) present in the WAS. However, the extended ozonolysis period past the 60th minute could have led to mineralization of the already solubilized organic matter hence the observed decrease in COD_S [4]. Also, the low reduction in COD_T during the extended period from the 80th minute indicates that most of the oxidizable organic solids had already been solubilized within the first 60 min.

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Based on these findings, the duration of ozonolysis should be minimized to reduce excessive energy consumption and depletion of carbon if anaerobic digestion is to be applied down-stream to the ozonolysis process [20]. The ozone transfer efficiency decreased with increasing duration of ozonolysis after every 20 min interval. Improved biodegradability due to ozonolysis was confirmed by the increases in AOS from-1.5 to 0.9. The BOD_5:COD ration increased from 0.1 to 0.21 while the pH decreased from 11 to 9.68. The increase in oxidation state could be attributed to the solubilization of the organic solids by degradation of the aromatic groups and formation of aliphatic structures with functional groups such as-COOH, -OH and -CHO as indicated by the FTIR analysis done by Otieno et al [4] shown that aliphatic structures contain higher oxidation state carbons than aromatic carbons. Moreover, the decrease in basicity could be due to the formation of the easily biodegradable acidic intermediate compounds such as acetic acids and humic substances [21].

IV. Conclusion

The developed neural network model successfully tracked the nonlinear behavior of the ozonolysis process for the pre-treatment of waste activated sludge (WAS) for sludge solubilization and to improve biodegradability. Based on batch experiment test results showed that optimal operating conditions were determined to be an initial pH 11, ozone dosage 90 mg/min and ozone duration of 60 min. The configuration of the backpropagation neural network (NN) was three-layer (3:10:1) with tangent sigmoid transfer function (*tansig*) at hidden layer with 10 neurons, linear transfer function (*purelin*) at output layer and Levenberg-Marquardt backpropagation training algorithm (LMA). Neural network predicted results are very close to the experimental results with correlation coefficient (R) of 0.9957, MSE 0.1050 and MSRE 0.0040.

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