



INHIBITING MILD STEEL CORROSION IN SULPHURIC ACID WITH AFRICAN SPINACH STEM WASTE: EFFECTS, MECHANISM, AND ARTIFICIAL NEURAL NETWORKS MODELLING

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ABSTRACT

The utilization of environmentally friendly plant extract to inhibit the corrosion of metal materials has been widely researched; however, the derivation of such corrosion inhibitors from plants that are not in direct confrontation to human food chain is desirable, and thus formed the focus of this study. This study gravimetrically investigated the corrosion inhibition property of African Spinach stem waste extract on Gauge 16 mild steel in 1.0 H₂SO₄ medium for an integrated waste to wealth purpose. The study also determined the adsorption isotherm characteristics of the extract. Additionally, Artificial Neural Networks (ANN) was employed to model and predict the experimental observation through network optimization of neuron size and amount. The results showed that the plant extract effectively inhibited the corrosion progression of mild steel, achieving a 95.12% inhibition rate at a 0.5 g/ml inhibitor concentration, 1-day immersion, and 30°C 1.0 H₂SO₄ medium temperature. However, the inhibition effectiveness decreased with increasing immersion time and 1.0 H₂SO₄ medium temperature. Langmuir and Freundlich model provided reasonable fit to the experimental data (R² of 0.954 and 0.937, respectively) with adsorption constants (K_{ads}) of 7.498 and 8.892, respectively, indicating a moderate affinity between the extract and the metal surface. The Freundlich model's surface heterogeneity parameter (n) was 1.233, implying a relatively high degree of nonlinearity in the adsorption behavior. The standard free energy change of adsorption (ΔG_{ads}°) values for both models were negative, confirming the spontaneous nature of the process. The ANN with a 3-3-1 topology exhibited the highest coefficient of determination (R² = 0.9884) at 25 epochs without over-fitting, demonstrating the effectiveness of ANN. These showed that African Spinach stem waste's extract effectively inhibited mild steel corrosion, and ANN model adequately represented the process for future design purposes.

Keywords: African Spinach fibrous stem waste; adsorption isotherm; mild steel; Sulphuric acid

Introduction

The widespread use of mild steel in industries is attributed to its favorable properties and cost-effectiveness (Umoren and Ekanem, 2010). The selection of mild steel for various applications is crucial due to its sustainable characteristics, including recyclability and a

lower carbon footprint compared to thermosetting plastic (Zubairu *et al.*, 2021). Despite these merits, mild steel is prone to corrosion, particularly in environments characterized by high moisture or chemical exposure (Umoren and Ekanem, 2010). In light of these challenges, Scientists and



Engineers are developing effective measures to enhance the corrosion resistance of mild steel, especially in aggressive environments. This is expected to progress for more localized and sustainable solutions.

Corrosion is a natural electrochemical process, it is a complex chemical reaction influenced by various factors such as moisture, oxygen, temperature, and presence of corrosive substances. It also involves the transfer of electrons from a material surface (metal) to the environment, causing the material to lose its structural integrity and properties over time (Ayeni *et al.*, 2014). Metal corrosion can have detrimental effects such as economic loss, structural failure, leakages, and downtimes; therefore, understanding and mitigating it is crucial.

A number of products, which are either synthetic or non-synthetic based, have been employed to prevent or control corrosion; however, the application of non-synthetic plant derived corrosion inhibitors remains a cheap, environmentally friendly, biodegradable, and effective solution. The antioxidant richness in plants enables their corrosion inhibition capabilities. More also, plant-derived corrosion inhibitors align with the principles of green chemistry, which aim to minimize the use of hazardous substances and reduce the environmental impact of chemical processes (Zakeri *et al.*, 2022). Green inhibitors can be extracted from various parts of plants, including leaves, stems, seeds, and roots (Umoren *et al.*, 2011). The plant-derived corrosion inhibitors act through various mechanisms, such as adsorption onto the metal surface, formation of a protective film or barrier, inhibition of electrochemical reactions, or neutralization of corrosive species (Asafa *et al.*, 2022). The

specific mechanism of plant derived corrosion inhibitor depends on the chemical composition of the specific plant extract (Zubairu *et al.*, 2021). A golden rule in plant selection for corrosion inhibitor extraction is for the plant not to be in confrontation to the human food supply chain.

African spinach, a leafy vegetable indigenous to Africa and belonging to the Amaranthaceae family, is known for its high antioxidant content (Olubanjo and Alade, 2018). Rich in flavonoids and other bioactive compounds (Adeosun *et al.*, 2016), African spinach leaves are commonly used in soup preparation in Nigeria. However, the process of plucking leaves and extracting the succulent stems for soup results in a significant amount of fibrous stem waste, which poses a challenge in households and restaurants. While animals like goats can consume the African spinach fibrous stem waste, the uneaten portions are often disposed haphazardly, contributing to unpleasant odors and environmental pollution as the waste decays. To address this issue, it is proposed that such waste be repurposed for integrated use, particularly in the extraction of corrosion inhibitors. This not only presents an environmentally friendly solution but also contributes to the development of cost-effective products.

Modeling and prediction are key concepts in Science and Engineering. Modeling entails creating a representation from existing data or patterns, while prediction involves estimating future outcomes based on this representation (Adeyi *et al.*, 2018). The representation captures essential features and relationships within a system or process, facilitating analysis and understanding of complex systems under different conditions (Onwude *et al.*, 2016). These tools enable design



optimization, risk mitigation, performance enhancement, cost reduction, quality improvement, and sustainability in various applications.

Amongst the currently utilized modeling methods, artificial neural network (ANN) continues to soar due to its accuracy, easy implementation on commercial software and non-mathematical nature (Yazdani *et al.*, 2013). ANN is inspired by the structure and functioning of biological neural networks in the brain (Adeyi *et al.*, 2018) and it is a subset of machine learning algorithms that is designed to recognize patterns, make predictions, and perform complex tasks by learning from example (Onwude *et al.*, 2016). ANN is composed of layers, and interconnected nodes called artificial neuron or simply neuron, which is the basic building block that receives inputs, performs computation, and produces output. Each input is multiplied by a weight value, the weighted inputs are summed, and the sum is passed through an activation function to introduce non-linearity, propagating the output to the next layer of neurons. Neurons play a crucial role in information processing and decision-making within ANN (Yazdani *et al.*, 2013). This study utilizes ANN for modeling and predicting the observed experimental data for improved future design, understanding and product specification.

In related studies, Pruthviraj *et al.*, (2013), amongst others reported effective corrosion inhibition of mild steel by *Clerodendrum phlomidis* leaves with brilliant results. However, a study on the corrosion inhibition capability of African Spinach fibrous stem waste is scarce. Therefore, the objectives of this study are to extract corrosion inhibitor from African Spinach fibrous stem waste, test

the corrosion inhibition effectiveness of the extract on mild steel and utilize ANN to model and predict the observed experimental corrosion inhibition properties.

Materials and Methods

Materials and equipments

Gauge 16 (0.16 cm) mild steel was obtained locally in Ogbomoso Township (8.133°N latitude and 4.245°E longitude), Oyo state, Nigeria. Chemicals including Sulphuric acid, ethanol and acetone were purchased from Ojota chemical market in Lagos, Nigeria. African Spinach fibrous stem wastes were collected from restaurants in Ogbomoso. Distilled water was also procured.

A dryer (SG-90526 - model), water bath (Mermmet model), digital weighing balance (Lito XG, ± 0.01 g precision), locally made burr grinder, sieve, mechanical shear cutter, XRF metal analyzer, Soxhlet extractor and emery papers were also utilized for sample preparations.

Methods

Mild steel coupon preparation

The elemental composition (wt.%) of the mild steel were C, Si, Mn, P, S, Cu and Fe equals to 0.13, 0.18, 0.39, 0.40, 0.04, and 0.025, respectively as determined with XRF metal analyzer. Mild steel coupons of dimension 3 x 4 cm each were cut out and polished for a smoother surface using mechanical shear cutter and emery paper. Thereafter, the mild steel coupons were cleaned with 95% ethanol and acetone successively, followed by storage in airtight container.

Preparation of African Spinach fibrous stem waste



A 500 g of African Spinach fibrous stem waste were hand shredded and rinsed in distilled water to remove sand and other foreign materials, followed by air-drying for 30 min. The air-dried plant stem wastes were then subjected to oven drying in Stangas oven at 80°C until constant weight (this took about 7 h). The dried stem wastes were pulverized using burr grinder, manually sieved to less than 1 mm particle sizes and stored in a glass bottle to prevent hygroscopic moisture absorption.

African Spinach fibrous stem waste crude extract preparation

The widely used extraction process as also utilized by Zubairu *et al.*, (2021), Chaudhary and Tak, (2022) and Ezugha and Aralu, (2023) was adopted in this study with slight changes. A 300 g of dried and ground African Spinach fibrous stem waste was boiled in 500 ml of ethanol using Soxhlet extractor for 2 h. After boiling, the ethanol extract was allowed to cool naturally. The cooled and filtered crude extract was then heated at 65 – 68 °C for 20 min in conical flask placed in water bath to remove the ethanol. The crude extract was then stored in airtight bottle to prevent hygroscopic activities.

Mild steel corrosion test

The basic and widely utilized gravimetric corrosion test method was adopted (Zubairu *et al.*, (2021), Oyewole *et al.*, (2021), Asafa *et al.*, (2022) and Ezugha and Aralu (2023). Six conical flasks containing 100 ml of 1.0 H₂SO₄ chemical each were prepared. The first flask had no plant extract dilution while the second to sixth flask had 0.1, 0.2, 0.3, 0.4 and 0.5 g/ml of crude extract dilution, respectively. A set of eight prepared mild steel coupons of equal weight and sizes were immersed totally

in each of the six conical flasks for the experimentation, and were monitored daily.

The gravimetric tests were conducted in two experimental batches of non-elevated (30°C) and elevated (50°C) temperature. The non-elevated temperature experimental batch implied that the conical flasks containing samples and corrosive medium were kept at 30°C for only 5 h and at room temperature (27 - 28°C) for 19 h within every 24 h immersion time. Similarly, the elevated temperature implied that the conical flasks containing samples and corrosive medium were kept at 50°C for only 5 h and at room temperature (27 - 28°C) for 19 h within every 24 h immersion time. After every 24 h immersion time, a sample from each of the conical flasks was removed with glass rod, washed with distilled water, ethanol and acetone in successively, and reweighed. The initial and 24 hourly weights of samples were utilized for computation. The experiments in the two experimental baths were terminated on day eight (that is 192 h). Experiments were done in triplicate and the average values were utilized for analysis.

To draw inference from the experimental observations, weight loss and inhibitor efficiency were determined using Eqn. 1-2 in accordance with the work of Ayeni *et al.*, (2014), Oyewole *et al.*, (2021) and Martins *et al.*, (2022).

$$W = W_1 - W_2 \quad (1)$$

Where W is the weight loss (g), W_1 is the initial weight of the specimen (g), W_2 is the final weight of specimen (g) after immersion in corrosive medium or solution.

$$IE = \left[\frac{(W_0 - W)}{W_0} \right] \times 100 \quad (2)$$



Where IE is the inhibition efficiency (%), W_0 is the weight loss of the specimen in the corrosive solution that does not have inhibition, W is the weight loss of the specimen in the corrosive solution that has inhibitor.

Adsorption isotherm

Adsorption isotherms are useful to understand the mechanism of heterogeneous organo-electrochemical reactions involved in a solid surface, making the degree of surface coverage (O) a useful consideration for understanding the mechanism of inhibitor adsorption. Eqn. 3 represents the surface coverage in accordance with the study of Oshoghomo *et al.*, (2020)

$$O = 1 - \left[\frac{W}{W_0} \right] \quad (3)$$

Where O is the Degree of surface coverage, W is weight loss of the specimen in a corrosive solution that has inhibitor, W_0 is the weight loss of specimen in a corrosive solution that does not have inhibitor.

The most frequently used adsorption isotherms are the Temkin, Langmuir, Freundlich, Hill de Boer, Parsons, Flory Huggins, Dhar-Flory Huggins, and Bockris Swinkles. In comparison to each other, the correlation coefficients (R^2) are usually applied to determine the best-fitted or performing isotherm (Ating *et al.*, 2010).

In this study, experimental data (non-elevated temperature experimental batch only) were fitted to Langmuir and Freundlich isotherms using Eqn. (4) – (5)

$$\log \left(\frac{\theta}{1-\theta} \right) = \log C + \log K_{ads} \quad (4)$$

Where, C is the concentration of the inhibitor, O is a fraction of surface coverage area or

surface coverage and K_{ads} is the adsorption equilibrium constant.

$$\log \theta = \log K_{ads} + \frac{1}{n} \log C, \text{ where } n \text{ is the interaction parameter} \quad (5)$$

Where, n is the adsorption intensity or surface heterogeneity

ANN modeling and prediction

To have a simple and accurate ANN model, the selection of an appropriate topology cannot be overemphasized (Adeyi *et al.*, 2018). In this study, a feed-forward structure, comprising of hyperbolic-tangent-sigmoid transfer function for the hidden layer, linear transfer function for output layer and Levenberg–Marquardt training algorithm was selected to investigate the optimum ANN topology (optimum value of the input and hidden layer of the ANN structure) that predict the inhibition efficiency (IE) data in this study. Data were partitioned into training (50 %), validation (25 %), and testing (25 %) portion, respectively. A mean of three separate computations is reported for each investigated ANN topology to enable reproducibility. Optimization of ANN topology was decided with highest value of coefficient of determination in accordance with Eqn. (6).

Coefficient of determination (R^2) = 1 –

$$\left(\sum_{i=1}^N \frac{(\text{Pred},i - \text{Exp},i)^2}{(\text{Pred},i - \text{AverageExp})^2} \right) \quad (6)$$

Where, Pred,i is the i th predicted value, Exp,i is the i th experimental value. AverageExp is the mean experimental value and N is the number of observation.

Results and Discussion

Extract inhibitor efficiency on mild steel



The time and temperature dependent inhibition efficiency of mild steel samples in non-inhibited sulphuric acid medium and

varied extract concentration inhibited sulphuric acid medium over time are represented in Fig. 1.

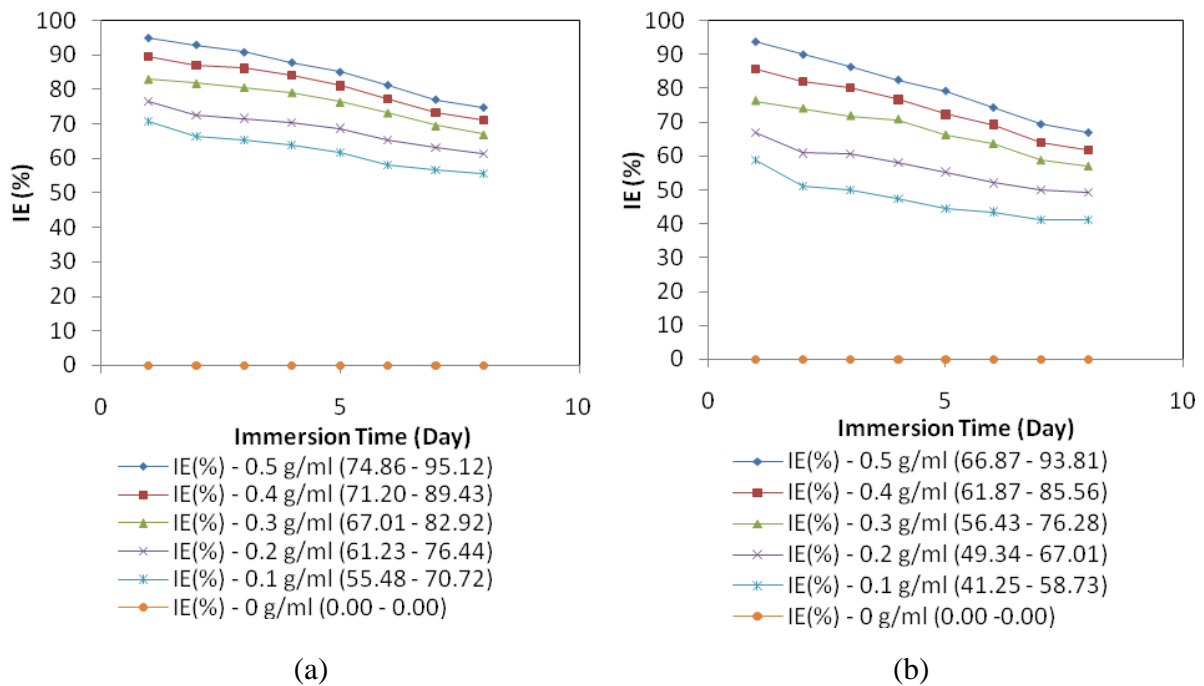


Fig. 1 Inhibition efficiency of mild steel in inhibited and un-inhibited H_2SO_4 (a) $30\text{ }^\circ\text{C}$ and $50\text{ }^\circ\text{C}$

The figure showed that the African Spinach stem waste extract effectively inhibited the corrosion progression of mild steel samples when the inhibited sulphuric acid medium is compared to un-inhibited sulphuric acid medium. The highest inhibition efficiency of 95.12% was noticed at 0.5 g/ml of extract at mild temperature and least inhibition efficiency of 49.34% was noticed at 0.2 g/ml at elevated temperature.

Furthermore, the figure showed that corrosion inhibition efficiency decreased as immersion time progressed for all concentrations. This implies an increased metal degradation as a function of time and temperature increment. The observation implied that a possible initially formed protective layer on the metal

surface by the extract breaks down with time and temperature increment and consequently exposing the metal to the corrosive medium more directly (Zakeri *et al.*, 2022). As a result, the corrosion process becomes significant, leading to increased corrosion progression. This result is consistent with the report of Asafa *et al.*, (2022) and Zakeri *et al.*, (2022).

In addition, the figure showed increased inhibition efficiency as the concentration of the extract increased. This is attributed to the availability of reactants in the corrosion process. When the concentration of the extract in the corrosion environment increased, there is more of it available to participate in the reaction and this reduced the rate at which



corrosion occurs. Similarly, when the concentration of extract is reduced, there is decreased inhibition and therefore corrosion inhibition reduced. This suggests an inverse relationship between concentration and corrosion progression (Ayeni *et al.*, 2014). Zand and Hoveidi (2016) also reported a close result.

Adsorption isotherms

Isotherms are utilized to comprehend the adsorption behavior of inhibitors on metal

surfaces. The Langmuir and Freundlich isotherms were applied in this study due to their wide utilization and effectiveness to explain the adsorption mechanisms. The isotherm, R^2 values, slopes, intercepts, and adsorption constants aid in understanding the adsorption process and the connection between the inhibitor's concentration and its adsorption on the metal surface (Umoren *et al.*, 2011). The adsorption isotherm constants derived by the two applied models in this study is depicted in Table 1.

Table 1 Adsorption values derived for extract's corrosion inhibitor

Model type	R^2	Slope	Intercept	K_{ads}	n	ΔG_{ads}°
Langmuir	0.954	1.589	0.875	7.498	Nil	15.093
Freundlich	0.937	0.811	0.949	8.892	1.233	15.519

The coefficient of determination (R^2) signifies the goodness-of-fit of the isotherm to the experimental data. It ranges between 0 and 1 and values closer to 1 indicates a better fit. In this case, both Langmuir and Freundlich isotherms exhibited high R^2 values, indicating reasonably good fits to the experimental data.

The K_{ads} represents adsorption constant that quantifies the strength of the interaction between the adsorbate and the adsorbent surface. It provides information about the affinity of the inhibitor for the metal surface. In this study, the K_{ads} of both isotherm demonstrated that the extract interacts with the mild steel surface, resulting in a reduction in corrosion phenomenon. For Freundlich isotherm, the provided K_{ads} value of 1.233 suggests a moderate affinity between the inhibitor and the metal surface. Higher K_{ads} values indicate stronger adsorption and potentially more effective corrosion inhibition.

The surface heterogeneity or the adsorption process (n) provides insights into the nonlinearity or variability of the adsorption behavior. If $n = 0$, it implies more surface heterogeneity; if $n < 1$, it indicates chemisorption; however, if $n > 1$, it indicates cooperative adsorption. In this case, the adsorption process of the extract onto the mild steel surface exhibits cooperative adsorption. This value of 8.892 suggests a relatively high degree of nonlinearity or variability in the adsorption behavior. This indicates that the adsorption process may not follow a simple, uniform mechanism and may be influenced by various factors.

The standard free energy change of adsorption (ΔG_{ads}°) indicates the spontaneity and thermodynamic favorability of the adsorption process. The negative values of ΔG_{ads}° reveals that the adsorption of inhibitor molecules on mild steel surfaces is a spontaneous process. The value of ΔG_{ads}° around -20 kJ/mol or lower supports the electrostatic interaction (physisorption)



between the adsorbent and adsorbate. This result is in agreement with the work of Ating et al., (2010) and Chaudhary and Tak (2022).

Data description for ANN modeling

The statistical descriptions of the output experimental data used for ANN modeling and prediction in this study are summarized in Table 2.

Table 2 Descriptive statistics of corrosion inhibition efficiency data

Description	Inhibition Efficiency (%)
Mean	51.82
Standard Error	3.15
Median	63.96
Mode	0
Standard Deviation	32.82
Sample Variance	1077.20
Kurtosis	-1.03
Skewness	-0.73
Range	95.12
Minimum	0
Maximum	95.12
Sum	5597.31
Count	108
Confidence Level (95.0%)	6.26

The mean inhibition efficiency was 51.83%, indicating the average level of inhibition across the dataset. The standard error of 3.16 suggests some variability in the sample means. The median inhibition efficiency of 63.96% suggests that the middle value is relatively high, indicating that a significant portion of the data has relatively high inhibition efficiency. The mode of 0 suggests the presence of samples with no inhibition which is in tune with the experimental data. The high standard deviation (32.82) and sample variance (1077.20) indicate a considerable spread or dispersion in the data. The negative kurtosis (-1.04) indicates a relatively flat distribution with fewer outliers compared to a normal distribution. The negative skewness (-0.73) suggests a slightly

skewed distribution to the left, meaning that the tail of the distribution extends more to the left than the right. The range of inhibition efficiency is substantial at 95.12%, indicating a broad spectrum of inhibition levels. The minimum value of 0 suggests the presence of samples with no inhibition, while the maximum of 95.12% indicates the highest observed inhibition efficiency. The sum of inhibition efficiency values (5597.32) and the count of 108 confirm the total and number of observations, respectively. The confidence level with a 95.0% confidence interval (6.26) gives an idea of the precision of the mean estimate. A wider confidence interval indicates greater uncertainty in the true population parameter. Therefore, the experimental data is reliable for modelling. A



similar data statistical description for data transparency and appropriateness for adaptive neuro fuzzy inference system intelligent modelling was reported by Adeyi et al., (2021) and Adeyi et al., (2022).

ANN simulation

The results of the ANN structure optimization for accurate modeling and prediction of the corrosion inhibition behavior in this study are summarized in Table 3. The optimization was performed by considering the simulation efficiency of each applied ANN topology. ANN topology is the engine for the modeling and prediction activity of the network neuron.

Table 3 Optimization of the computational structure

S/N	Topology	Epoch No	R	R ²
1	3-1-1	9	0.9808	0.9620
2	3-2-1	18	0.9808	0.9620
3	3-3-1	18	0.9900	0.9801
4	3-4-1	15	0.9785	0.9574
5	3-5-1	20	0.8485	0.7201
6	3-6-1	10	0.9881	0.9763
7	3-7-1	11	0.9848	0.9698
8	3-8-1	9	0.9801	0.9605
9	3-9 1	6	0.9688	0.9386
10	3-10-1	8	0.9652	0.9316
11	3-1-1-1	24	0.9811	0.9626
12	3-1-2-1	15	0.9800	0.9604
13	3-1-3-1	15	0.9807	0.9230
14	3-1-4-1	11	0.9804	0.9612
15	3-1-5-1	13	0.6798	0.4621
16	3-1-6-1	42	0.9806	0.9616
17	3-1-7-1	20	0.8732	0.7625
18	3-1-8-1	11	0.9876	0.9754
19	3-1-9-1	15	0.9681	0.9372
20	3-1-10-1	14	0.9785	0.9575
21	3-2-1-1	10	0.9837	0.9677
22	3-3-1-1	25	0.9942	0.9884
23	3-4-1-1	11	0.7020	0.4928
24	3-5-1-1	13	0.9899	0.9799
25	3-6-1-1	13	0.7123	0.5075
26	3-7-1-1	8	0.7618	0.5803
27	3-8-1-1	13	0.5972	0.3566
28	3-9-1-1	11	0.6054	0.3665

The table showed that the highest epoch number of 42 epochs occurred at topology 3-

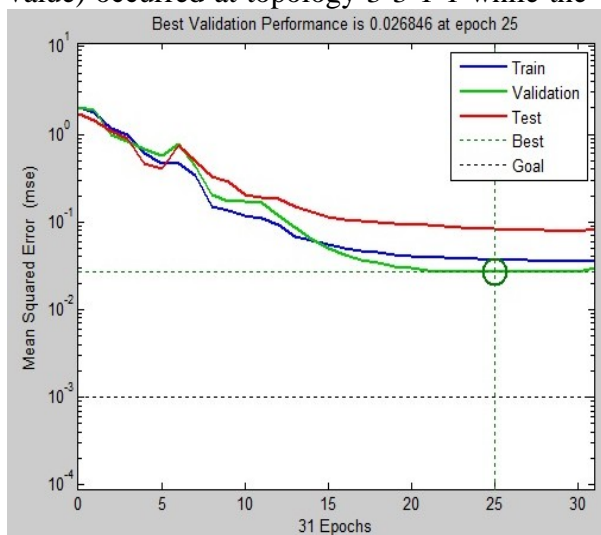
1-6-1 while the least number of 6 epochs occurred at topology 3-9 1. The table also



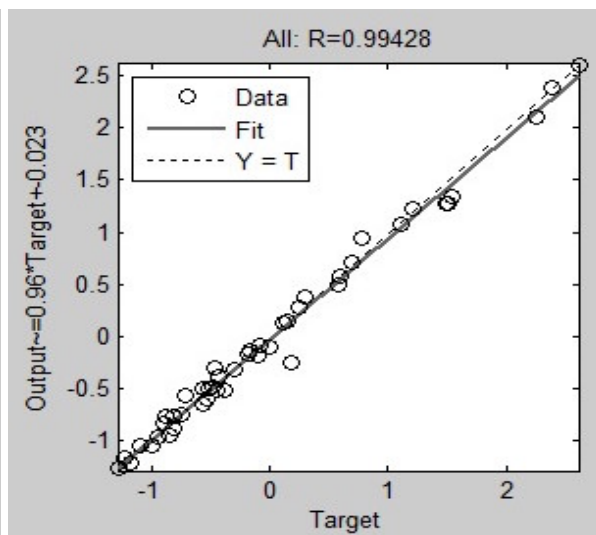
showed that high epochs are associated with high R^2 values. High epoch number is a signal to high computer memory utilization during computation which may not be desirable. In intelligent modeling, investigation of optimum epoch number helps in preventing over-fitting or memorization, therefore, low epoch with appreciable R^2 value (value close to unity) is desirable. The table further showed that the highest model efficiency (R^2 value) occurred at topology 3-3-1-1 while the

least model efficiency occurred at topology 3-8-1-1. It could therefore be established that ANN topology 3-3-1-1 was optimum, and best used in modeling and prediction of the corrosion efficiency inhibition data in this study.

In addition, Fig. 2 (a) and (b) depicts the performance of the best ANN topology (3-3-1-1) in modeling and predicting the corrosion inhibition efficiency data.



(a)



(b)

Fig. 2 ANN (a) performance and (b) the prediction efficiency

Fig. 2 (a) showed that training, validation and testing errors decreased during simulation until 25 epochs or iteration before the tendency of an increase in error was noticed (see the small circle in Fig. 2 (a)). This means that a further increase in epoch number will make the built ANN network memorize will lead to over-fitting. The parity plot or prediction efficiency of the best ANN topology in Fig. 2 (b) shows a strong relationship between the experimental and simulated data as observed from the cluster of data around the regression lines. This shows

that insignificant differences existed between the experimental and ANN simulated data. In a related study, Amodu et al., (2022) reported R^2 of 0.9621 – 0.9886 for ANN modeling of the corrosion inhibition of mild steel in 1 M HCl by *Musa paradisiaca* peel extract.

Conclusion

The study gravimetrically investigated the corrosion inhibition property of African Spinach stem waste's extract on Gauge 16 mild steel in 1.0 H_2SO_4 medium. The inhibitor adsorption isotherm was also studied



using Langmuir and Freundlich absorption isotherms. Based on the results, it was concluded that the extract is effective for mild steel corrosion inhibition with moderate affinity between the extract and the metal surface. The activity of the extract was also found out to be spontaneous in nature. Furthermore, the Artificial Neural Networks (ANN) model represented the experimental observation efficiently. Therefore, African Spinach stem wastes can be converted to a valued extract product for mild steel corrosion inhibition.

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