

Research Article

Artificial Neural Network Modelling of Corrosion Inhibition of Mild Steel in Marine Environment Using Epoxy-Nickel Oxide Nanocomposite Coatings

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Abstract

The corrosion inhibition of mild steel in 3.5 wt. % NaCl in the absence and presence of epoxy coatings containing NiO nanoparticles with concentrations of 1.0, 2.0, 3.0 and 5.0 wt. % respectively was studied using the gravimetric technique for a duration of 60 days at room temperature and varying temperatures ranging from 30 to 60 °C for 5 hours. The Nickel oxide nanoparticles with average particle size was 23 nm were synthesized by the chemical precipitation technique followed by calcination in a muffle furnace for 3 hours at a temperature of 300 °C. Results from the study reveal that epoxy-Nickel oxide nanocomposite coatings are effective green corrosion inhibitors for mild steel in 3.5 wt. % NaCl under different operating conditions and at temperatures within the range of 30 to 60 °C. A predictive model based on the Artificial Neural Network (ANN) was developed to study the relationship between the input variables (exposure time, inhibitor concentration and Temperature) and output variables (Corrosion Rate and Inhibition Efficiency). The ANN model was based on the Multilayer Perceptron algorithm with input layer comprising of 3 factors and 23 units. Hyperbolic tangent was used as the activation function for the hidden layer which was made up of 3 units. The output layer with two dependent variables was made up of 2 units. Corrosion test data obtained from 80 experimental runs were successfully modelled using ANN with minimal errors. 56 cases corresponding to 70% of test data were used for training the network and 24 cases corresponding to 30% of test data was used for testing the efficacy of the network. The model had sum of squares error of 0.981, average overall relative error of 0.018 for the training component and values of 3.190 and 0.043 for the sum of squares error and average overall relative error respectively for the testing component.

Keywords

ANN Modelling, Nickel Oxide Nanoparticles, Nanocomposite Coatings, Mild Steel, Corrosion, Green Inhibitors, 3.5 wt. % NaCl

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1. Introduction

Corrosion of pipelines and other engineering infrastructure made from mild steel is a global problem and constitutes major economic and environmental losses to the oil and gas industries, chemical processing industries, food processing industries and desalination plants operating within the marine environment. Corrosion which is the gradual removal of the material surface due to interaction with its environment is an electrochemical process [1] and accounts for losses of about 3-5% of the world's GDP annually [2, 3]. Development of strategies such as the use of protective coatings, inhibitors, cathodic protection, anodic protection, pigging of pipelines, appropriate materials selection and design to combat this menace has been the subject of many research efforts [4-7]. The use of corrosion inhibitors is very common among researchers due to their availability, low-cost, ease of use and environmental compatibility [8, 9, 1]. Among the corrosion inhibitors used successfully in the past, plant extracts have been the most reported in literature. However, these extracts have the challenges of need for constant replacement and tendency to be carried away by corrosive fluid or leached into solution. The ability of metal oxide nanoparticles to overcome the aforementioned limitations has increased their demand as corrosion inhibitors and several metal oxide nanoparticles such as titanium oxide [10], nickel oxide [11], zinc oxide [12], silver oxide [13] have been used successfully as corrosion inhibitors. ZnO has excellent anti-corrosive properties due to its ability to react with moisture to form stable zinc hydroxide and zinc carbonate, which acts as a protective barrier. ZnO is also effective in protecting against UV radiation, offering a dual advantage for both corrosion and UV resistance. However, ZnO's performance can degrade under highly alkaline conditions, and it might not be as stable as other metal oxides in the long term, particularly in more complex or extreme corrosive environments. TiO₂ is highly stable and offers excellent UV resistance, which makes it ideal for outdoor applications. It also has photocatalytic properties that can break down organic pollutants, enhancing self-cleaning and anti-corrosion capabilities. TiO₂ is also highly stable in both acidic and alkaline conditions. While TiO₂ coatings offer superior stability and UV protection, their corrosion inhibition properties may not be as pronounced as NiO or ZnO, especially in highly aggressive environments where metal-specific reactions are more favorable.

Nickel oxide nanoparticles is of particular interest due to its unique combination of hardness, high surface area to volume ratio, longevity, chemical and thermal stability in aggressive salt environments and synergistic effect with other corrosion inhibitors [14-16]. The efficacy of 3-Mercaptopropyl triethoxysilane (MPTES)-modified NiO nanoparticles in varying concentrations ranging from 1 to 3% embedded in epoxy resin as a corrosion inhibitor for mild steel in sea water was studied by using electrochemical impedance spectroscopy (EIS) and scanning electrochemical microscopy (SECM). Findings from the study revealed that the introduction of NiO nano-

particle in the epoxy coating enhances the charge transfer resistance (R_{ct}) as well as the film resistance (R_f). Epoxy-MPTES modified NiO nanocomposite coated steel significantly enhanced protective capacity of epoxy resin by preventing the transportation of H₂O, O₂ and corrosive ions into the epoxy resin and lowered the chances of degradation and blistering of the coating film [17]. The potential of epoxy/1.8 ZnO-NiO as an effective inhibitor for corrosion protection of steel in 3.5 wt. % NaCl environment was studied using electrochemical impedance spectroscopy (EIS) and Potentiodynamic Polarization (PDP) measurements. Results from the electrochemical testing confirmed that improved anticorrosion performance was exhibited by the EP/1.8 ZnO-NiO-epoxy coating compared to the pure epoxy, EP/1 ZnO-NiO coatings due to the higher charge and resistance for the epoxy/1.8-ZnO-NiO nanocomposite coated steel [18]. In an attempt to produce more environmentally benign corrosion inhibitor, sustainable vegetable (soy) oil-based epoxy polymer blends (ESODG), and their nickel oxide (NiO) based organic-inorganic hybrid (ESODG-PA-NiO) nanocomposites were formulated as anticorrosive coatings. Addition of small amount of NiO nanoparticle in epoxy blend remarkably improved the hydrophobic characteristics, corrosion inhibition, metal surface adhesion, scratch hardness, and abrasion resistance properties of the hybrid composite coatings. The excellent anticorrosive performance of ESODG- PA-NiO hybrid was attributed to the electrostatic interaction between NiO and ESODG-PA, which provide additional locking/sealing effect and rectified polymeric artifacts and can be used as a surface decorative/ protective anticorrosive coating [19]. Corrosion is a complex process with many variables at play at the same time [20]. Mathematical modelling is used to study the contribution of each of the process variables to the entire corrosion process [21]. In this study, NiO nanoparticles prepared by chemical precipitation technique were embedded in epoxy resin and used as nanocomposite coatings for corrosion inhibition of mild steel in 3.5 wt. % NaCl at different operating conditions. Corrosion rate and inhibition efficiency were monitored using weight loss technique in absence and presence nanoscale NiO with epoxy coating. The relationship between process parameters including exposure time, temperature and inhibitor concentration were correlated using ANN modelling of test data.

2. Methodology

2.1. Metal Preparation and Procedure

Mild Steel with dimensions of 2 cm x 3 cm x 0.2 cm and total surface area of about 14 cm² was used as the working electrode. The composition of the mild steel used is tabulated in Table 1.

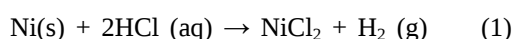
Table 1. Chemical composition of mild steel used.

Metal	C	Mn	P	Si	S	Cu	N	Cr	Fe
Composition (wt. %)	0.14	0.48	0.017	0.18	0.005	0.03	0.007	0.79	98.43

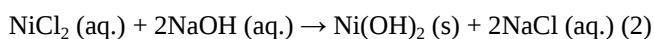
Before use, the specimens were degreased with acetone and abraded sequentially with emery papers ranging from 220 to 1200 grades respectively. This was closely followed by rinsing with running water, drying and rinsing with acetone. The cleaned specimens were preserved in a desiccator over silica gel until they were ready to be used for experiments. The blank specimen, pristine epoxy coated specimen and specimens coated with epoxy resin containing varying concentrations of 1, 2, 3 and 5 wt. % NiO respectively were completely submerged in 400 cm³ of 3.5 wt. % NaCl for a duration of 60 days at intervals of 5 days at room temperature (25 °C). Subsequently, the specimens were submerged in 250 cm³ of 3.5 wt. % NaCl for a duration of 5 hours in the absence and presence of epoxy resin and the nanocomposite coatings at temperatures of 30, 40 and 60 °C respectively. The specimens were thoroughly washed with water after each experiment, dried with cotton wool, rinsed with acetone, dried with a hand dryer and subsequently weighed with a high precision electronic analytical weighing balance.

2.2. Synthesis of Nickel Oxide Nanoparticles

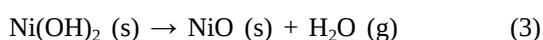
Pure solid nickel crucible was dissolved in 330 ml of 12M concentrated Hydrochloric acid (HCl) containing 37% wt. % HCl to form nickel chloride in aqueous solution accompanied by the liberation of hydrogen gas according to the reaction:



The conversion of nickel chloride (NiCl₂) into nickel oxide (NiO) was achieved by titrating the nickel chloride against 2M NaOH solution obtained by dissolving 80 g of NaOH pellets in 1 litre of distilled water which resulted in the precipitation of nickel hydroxide Ni(OH)₂ as a green solid according to the reaction:



The green precipitate of Ni(OH)₂ was filtered and washed several times, dried in air and subsequently converted into nickel oxide (NiO) through a heating process by calcination for three hours in a muffle furnace at a temperature of about 300 °C. The reaction below represents the dehydration of nickel hydroxide to form nickel oxide and water vapour.



2.3. Characterization of Nickel Oxide Nanoparticles

The characterization of the synthesized NiO nanoparticles was done using a combination of XRD and SEM-EDX techniques. Structural analysis was performed using Thermo Scientific ARL X'TRA X-ray Diffractometer (XRD) with serial number 197492086, a product of Thermo fisher Scientific Company Switzerland. The elemental composition of the synthesized nanoparticles was studied using the JEOL JSM 7600F Scanning Electron Microscope.

2.4. Preparation and Application of Coatings on Mild Steel Specimens

The coatings were prepared using the method adopted by Khodair et al. [20] with slight modification. The coatings for the mild steel specimens were produced by mixing epoxy resin based on diglycidyl ether of bisphenol A (DGEBA) with an amine hardener, tetraethylenepentamine (TEPA) in the ratio of 2: 1 by weight. Varying concentrations of 1, 2, 3 and 5 wt. % NiO were added to the coating mixture and the contents were mechanically stirred for about 30 minutes and ultrasonicated for about 15 minutes for homogenization. Well cleaned mild steel specimens suspended by polymeric threads were fully dipped into the coating mixture and subsequently sun dried for 48 hours for full curing.

2.5. Characterization of Coatings

The presence of epoxy resin, tetraethylenepentamine hardener and nickel oxide nanoparticles in the coating mixture were detected using Fourier Transform Infrared Spectroscopy, FTIR [PerkinElmer Instrument, Spectrum Two FT-IR Spectrometer (LiTaO₃ Detector) with serial number 122344].

2.6. Corrosion Rate Measurements

The corrosion inhibition data for mild steel in the presence and absence of epoxy-nickel oxide nanoparticles for 60 days of immersion in 3.5 wt % NaCl was obtained using the gravimetric method. Corrosion rate was calculated using the formula:

$$\text{CR} = \frac{W}{At} \quad (4)$$

where CR is the Corrosion rate ($\text{gm}^{-2}\text{day}^{-1}$), A is the surface Area (m^2) and t is the exposure time (days). The corrosion inhibition efficiency was calculated using the formula:

$$\% \text{ I. E.} = \frac{CR^0 - CR^i}{CR^0} \times 100 \quad (5)$$

where % I. E. = percentage inhibition efficiency, CR^0 = Corrosion rate of uncoated sample,
 CR^i = Corrosion rate of coated sample.

2.7. Development of ANN Model for Corrosion Test Data

Artificial Neural Network (ANN) modelling of corrosion test data was carried out using the neural network tool of IBM SPSS Statistics version 27 software. The Multilayer Perceptron algorithm was adopted for this study for the modelling of data obtained from 80 experimental runs with 56 cases constituting 70% of test data used for training the network and 24 cases corresponding to 30% of test data was used for testing the efficacy of the network. The independent (input) variables used were inhibitor concentration (wt. % NiO), exposure time (hr) and temperature ($^{\circ}\text{C}$) while the dependent (output) variables were corrosion rate ($\text{gm}^{-2}\text{day}^{-1}$) and inhibition efficiency (%). The input layer consisted of 23 units with 1 hidden layer and 3 units in the hidden layer 1. The three input variables (inhibitor concentration, exposure time, and temperature) are the main predictors used to estimate the corrosion rate and inhibition efficiency, which are the dependent (output) variables. The input layer of an ANN typically corresponds to the number of independent variables. Each input variable gets mapped to one unit (or neuron) in the input layer. In this case, there are 3 input variables so the input layer consists of 3 units. However, the input layer in this case consists of 23 units in total because of combination of input variables as illustrated in the network architecture (Figure 1). Sometimes, additional combinations or interactions of input variables (such as product or ratio of temperature and concentration) are added to capture non-linear effects between them. Hyperbolic tangent was adopted as the activation function for the hidden layer. Hyperbolic tangent is a highly effective activation function and was adopted because it provides a balanced, non-linear output with a smooth gradient, is suitable for regression tasks, and allows for the effective handling of both positive and negative corrosion rate values. In comparison to other activation functions, tanh is particularly well-suited for environments where the data is continuous and exhibits complex, non-linear behavior, like corrosion prediction in marine environments using composite coatings. The network information for the ANN model of corrosion test data is illustrated in Figure 2.

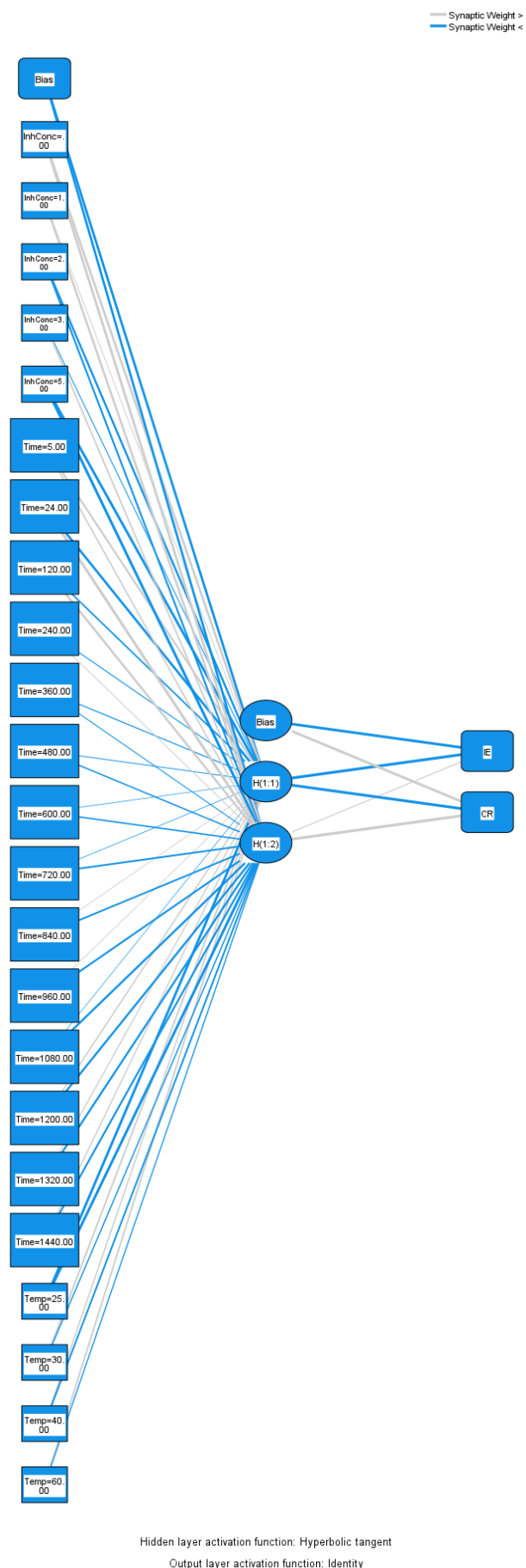


Figure 1. ANN Architecture for the predictive model.

Network Information			
Input Layer	Factors	1	Inh. Conc.
		2	Time
		3	Temp.
	Number of Units ^a		23
Hidden Layer(s)	Number of Hidden Layers		1
	Number of Units in Hidden Layer 1 ^a		3
	Activation Function		Hyperbolic tangent
Output Layer	Dependent Variables	1	CR
		2	IE
	Number of Units		2
	Rescaling Method for Scale Dependents		Standardized
	Activation Function		Identity
	Error Function		Sum of Squares

a. Excluding the bias unit

Figure 2. Network information for modelling of corrosion test data using ANN.

3. Results

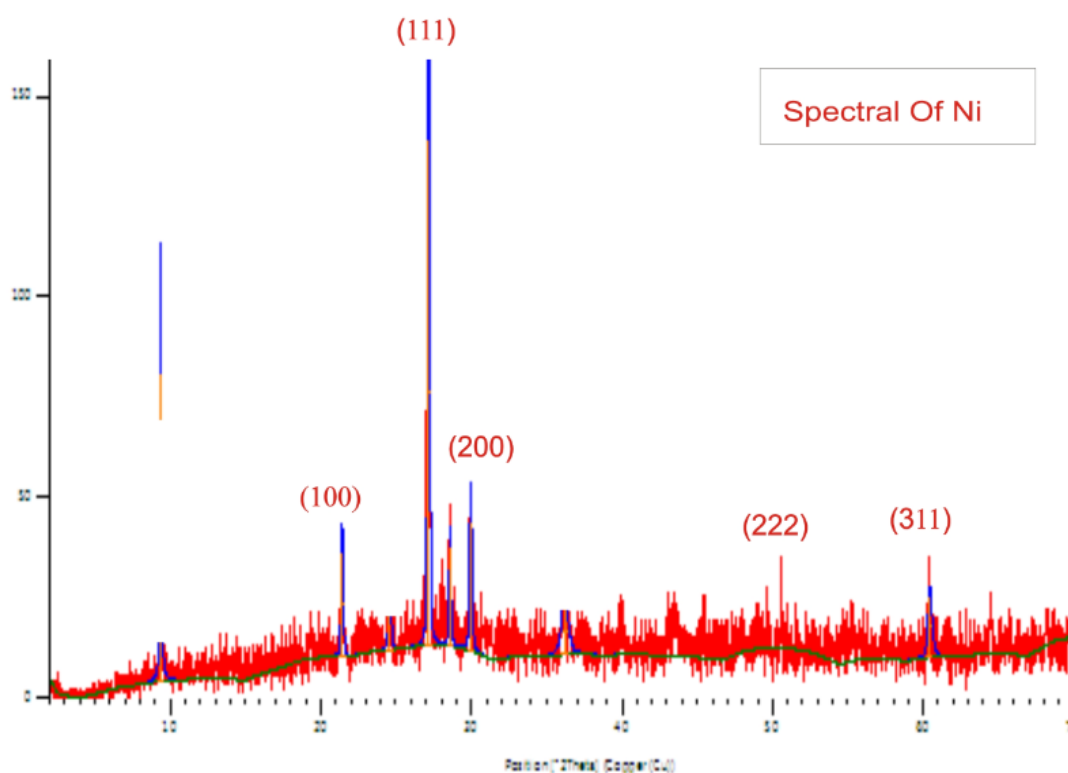


Figure 3. XRD Analysis of synthesized nickel oxide nanoparticles.

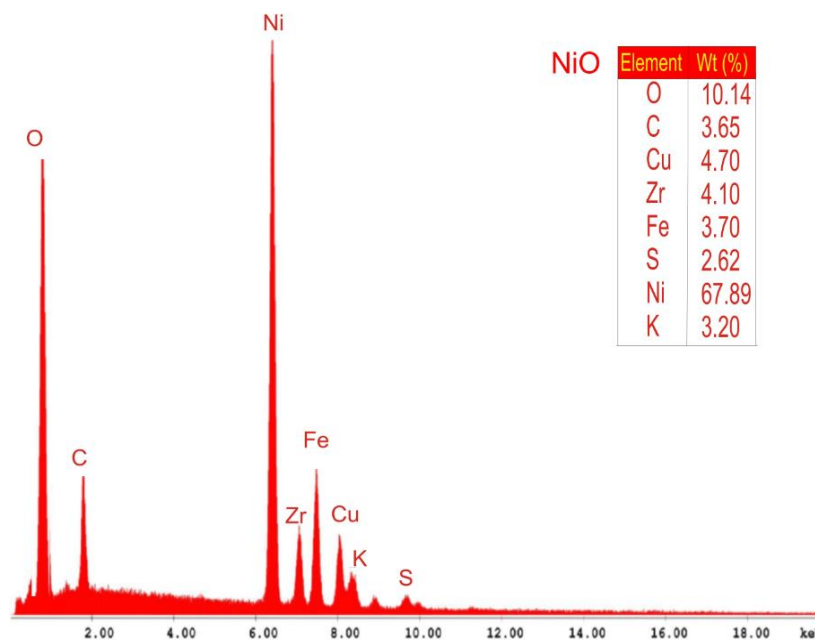


Figure 4. EDX Analysis for synthesized nickel oxide nanoparticles.

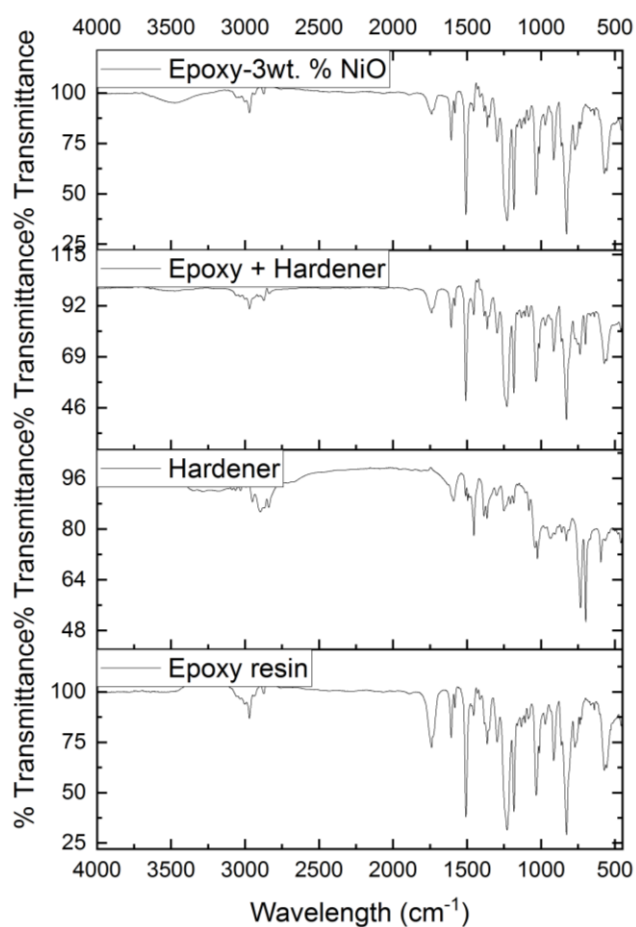


Figure 5. FTIR spectrum of epoxy resin, hardener and Epoxy-3 wt. % NiO nanocomposite coating.

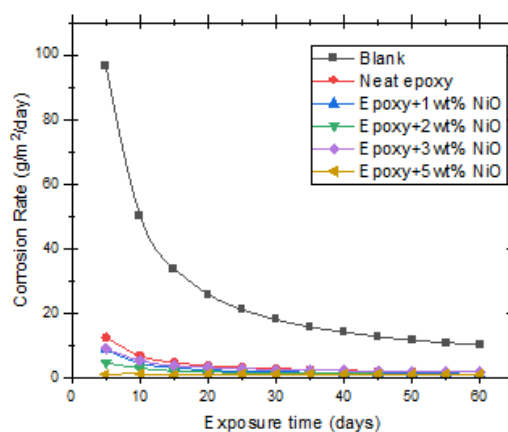


Figure 6. Corrosion rate of uncoated and coated mild steel in 3.5 wt % NaCl.

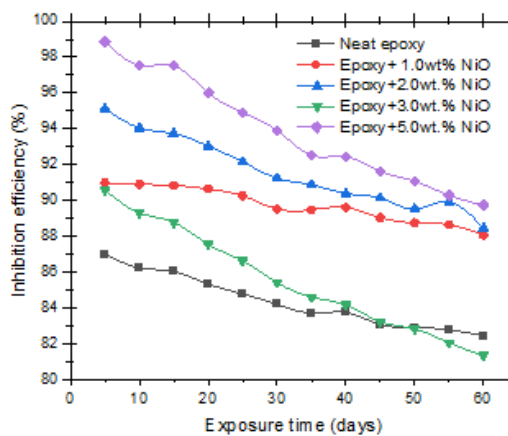


Figure 7. Inhibition efficiency of uncoated and coated mild steel in 3.5 wt % NaCl.

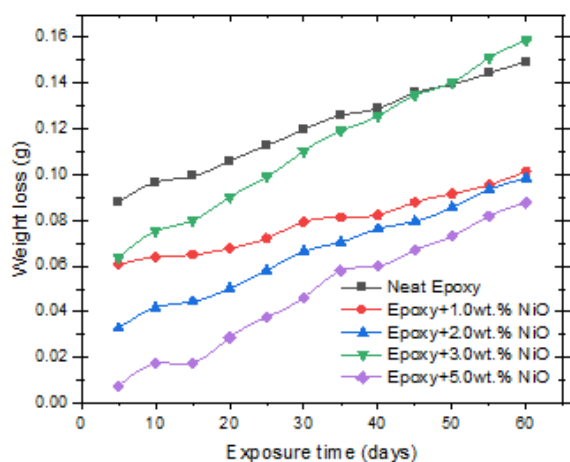


Figure 8. Weight loss of uncoated and coated mild steel in 3.5 wt. % NaCl.

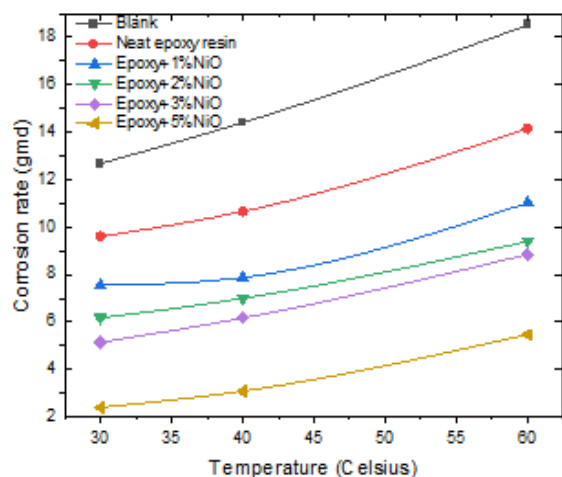


Figure 9. Effect of Temperature on corrosion rate.

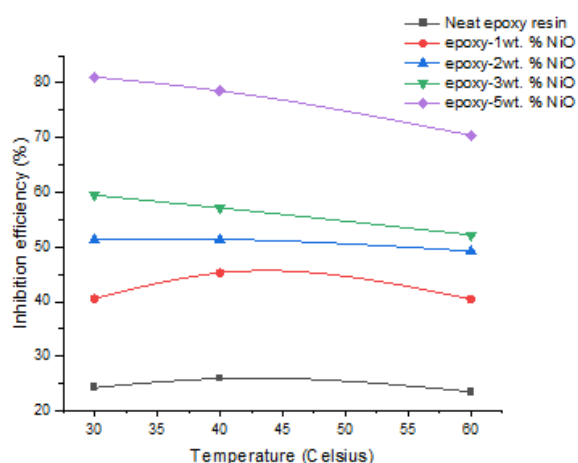


Figure 10. Effect of Temperature on Inhibition efficiency.

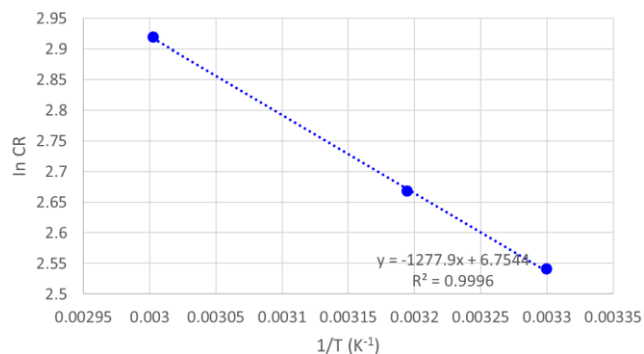


Figure 11. Arrhenius plot for blank MS in 3.5 wt. % NaCl at 30 - 60 °C.

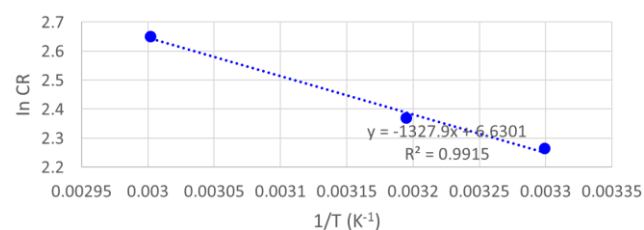


Figure 12. Arrhenius plot for neat epoxy resin coated MS immersed in 3.5 wt. % NaCl at 30 - 60 °C.

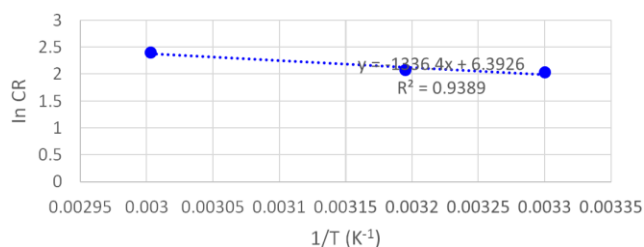


Figure 13. Arrhenius plot for epoxy-1% NiO coated MS immersed in 3.5 wt. % NaCl at 30 - 60 °C.

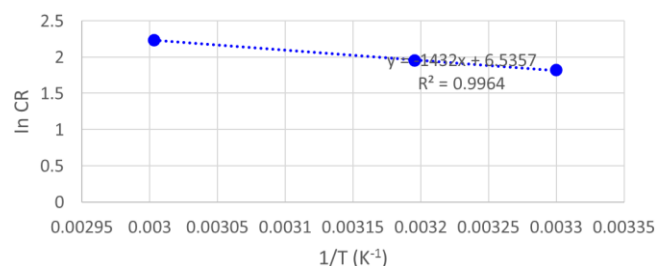


Figure 14. Arrhenius plot for epoxy-2% NiO coated MS immersed in 3.5 wt. % NaCl at 30 - 60 °C.

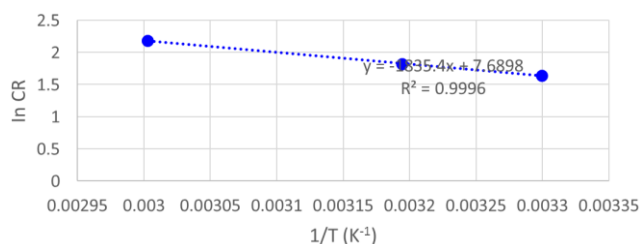


Figure 15. Arrhenius plot for epoxy-3% NiO coated MS immersed in 3.5 wt. % NaCl at 30 - 60 °C.

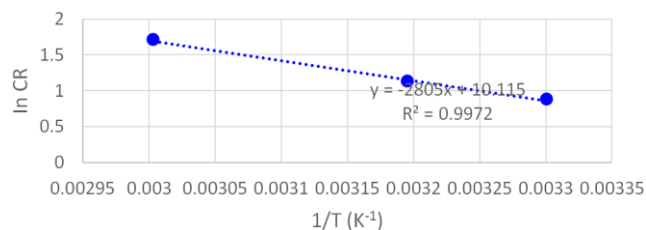


Figure 16. Arrhenius plot for epoxy-5% NiO coated MS immersed in 3.5 wt. % NaCl at 30 - 60 °C.

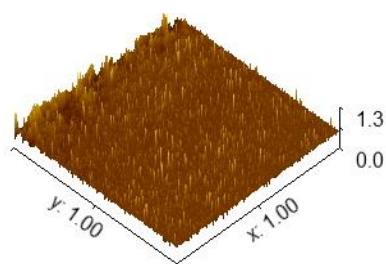


Figure 17. 3-D AFM micrograph of blank sample in 3.5 wt.% NaCl.

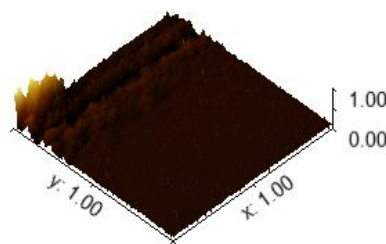


Figure 18. 3-D AFM micrograph of epoxy resin coated sample in 3.5 wt.% NaCl.

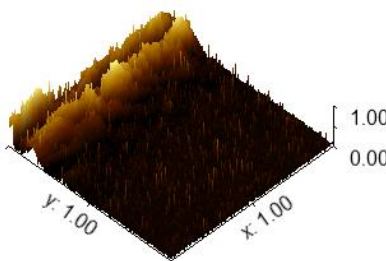


Figure 19. 3-D AFM micrograph of epoxy-1.0 wt. % NiO coated Mild steel sample in 3.5 wt.% NaCl.

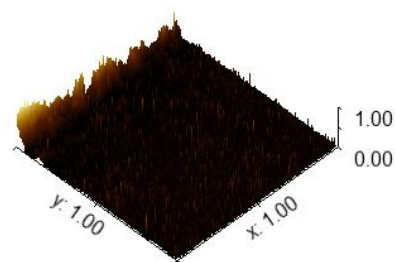


Figure 20. 3-D AFM micrograph of epoxy-3.0 wt. % NiO coated Mild steel sample in 3.5 wt.% NaCl.

Model Summary			
Training	Sum of Squares Error		.981
	Average Overall Relative Error		.018
	Relative Error for Scale Dependents	CR	.012
		IE	.023
	Stopping Rule Used	1 consecutive step(s) with no decrease in error ^a	
Testing	Training Time		0:00:00.01
	Sum of Squares Error		3.190
	Average Overall Relative Error		.043
	Relative Error for Scale Dependents	CR	.040
		IE	.055

a. Error computations are based on the testing sample.

Figure 21. Model summary for modelling of corrosion test data using ANN.

Parameter Estimates					
		Predicted			
		Hidden Layer 1		Output Layer	
Predictor		H(1:1)	H(1:2)	IE	CR
Input Layer	(Bias)	-.793	-.646		
	[lnhConc=.00]	.543	.857		
	[lnhConc=1.00]	.115	.516		
	[lnhConc=2.00]	-.487	-.414		
	[lnhConc=3.00]	-.144	.298		
	[lnhConc=5.00]	-1.303	-1.913		
	[Time=5.00]	.476	.279		
	[Time=24.00]	-.732	2.147		
	[Time=120.00]	-.328	.533		
	[Time=240.00]	-.176	.123		
	[Time=360.00]	-.199	-.160		
	[Time=480.00]	-.141	-.276		
	[Time=600.00]	-.024	-.283		
	[Time=720.00]	-.032	-.408		
	[Time=840.00]	.106	-.405		
	[Time=960.00]	.102	-.453		
	[Time=1080.00]	-.014	-.510		
	[Time=1200.00]	.273	-.492		
	[Time=1320.00]	.205	-.478		
	[Time=1440.00]	.246	-.445		
[Temp=25.00]	-1.002	-1.334			
[Temp=30.00]	.353	-.255			
[Temp=40.00]	.262	-.391			
[Temp=60.00]	.341	-.212			
Hidden Layer 1	(Bias)			-2.015	1.642
	H(1:1)			-2.736	-2.152
	H(1:2)			.199	4.158

Figure 22. Parameter estimates for ANN Model.

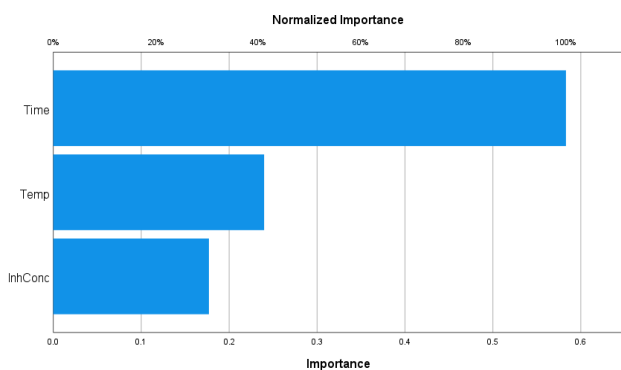


Figure 23. Normalized Independent variable importance.

Independent Variable Importance		
	Importance	Normalized Importance
Inh. Conc.	.177	30.4%
Time	.583	100.0%
Temp.	.240	41.1%

Figure 24. Independent variable importance.

ANN modelling of test data showed very high correlation between obtained experimental data and predicted values by ANN with little residuals and deviation as illustrated in Figures 25 to 28.

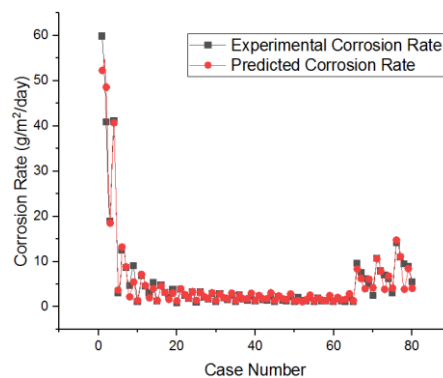


Figure 25. Comparison of experimental and predicted corrosion rates.

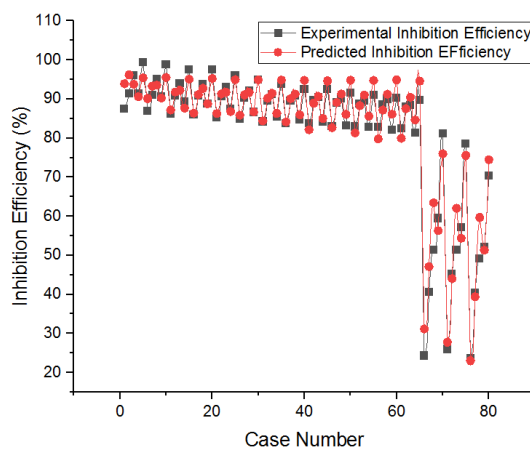


Figure 26. Comparison of experimental and predicted Inhibition Efficiencies.

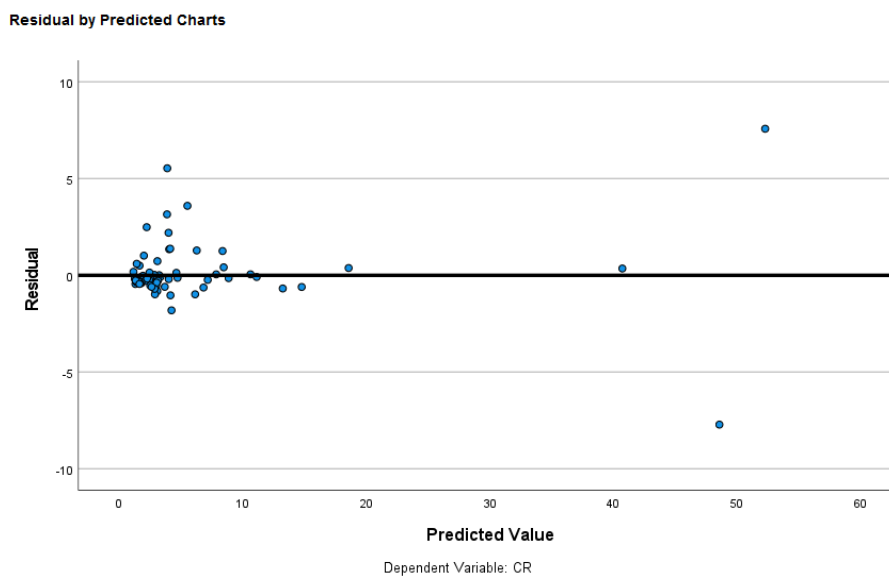


Figure 27. ANN chart for residual versus predicted corrosion rate.

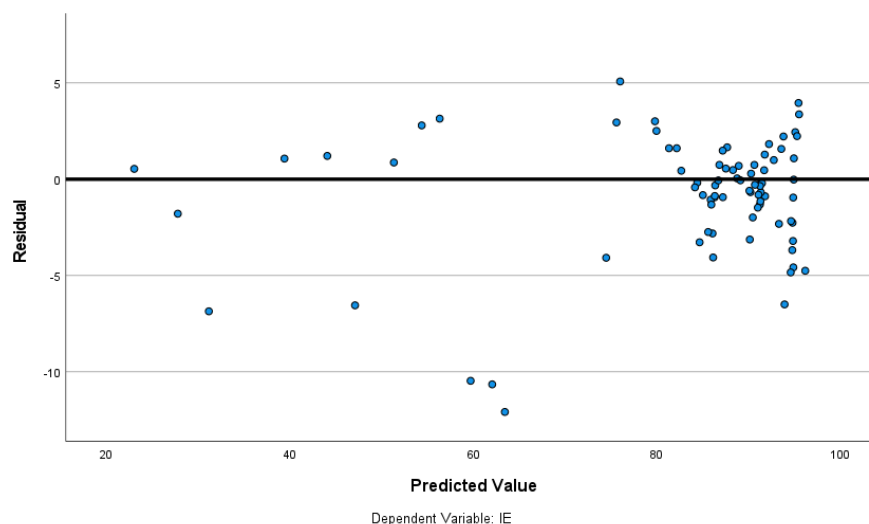


Figure 28. ANN chart for residual versus predicted inhibition efficiencies.

Table 2. Computed activation energy values for the various samples.

Sample description	Slope of graph (K)	E_a (J/mol)	E_a (kJ/mol)
Blank MS	-1 277.90	10 624.46	10.62
Epoxy coated MS	-1 327. 90	11 040.16	11.04
Epoxy-1% wt. % NiO coated MS	-1 336. 40	11 107. 50	11.10
Epoxy-2 wt. % NiO coated MS	-1 432.00	11 988.79	11.99
Epoxy-3 wt. % NiO coated MS	-1 835. 47	15 260.10	15.26
Epoxy-5 wt. % NiO coated MS	-2 805.00	23 320.77	23.32

Table 3. Surface Roughness values of selected samples.

SAMPLE	Average value	RMS roughness (Sq)	RMS (grainwise)	Mean roughness (Sa)	Maximum Peak height (Sp)	Maximum Peak depth (Sv)	Maximum height (Sz)
Blank	0.1854	0.07872	0.07872	0.04283	0.8146	0.1854	1.0000
Epoxy coated MS	0.1206	0.05295	0.05295	0.02655	0.8794	0.1206	1.0000
Epoxy-1 wt. % NiO coated MS	0.1047	0.1432	0.1432	0.0919	0.8953	0.1047	1.0000
Epoxy-3 wt. % NiO coated MS	0.0452	0.06408	0.06408	0.03146	0.9548	0.0452	1.0000

4. Discussion of Results

4.1. Structural Analysis of NiO NPs

The X-ray diffraction pattern for the synthesized nickel

oxide nanoparticles is shown in Figure 3. The (100), (111), (200), (222) and (311) planes corresponding to diffraction angles of 21.5°, 27.5°, 30.7°, 51° and 60.9° respectively detected at major diffraction peaks confirmed the synthesis of nickel oxide with a lattice constant of 4.1749 Å (DB Card number 01-076-6122). The average crystallite size was cal-

culated to be about 23 nm which is in close agreement with other reported works [22, 23] using the Debye-Scherrer formula [24] which is given as:

$$D = \frac{\text{Shape factor } X \text{ x-ray wavelength}}{\text{Full width at half maximum, FWHM } X \cos \Theta} \quad (6)$$

4.2. Compositional Analysis of NiO NPs

The EDX pattern of the synthesized nickel oxide nanoparticles shown in Figure 4 revealed that the composition of the sample is made up of 67.89% Ni and 10.14% O with a small concentration of other elements such as C, Cu, Zr, Fe, S and K with composition of 3.65% C, 4.70%, 4.10%, 3.70%, 2.62% and 3.20% respectively. The presence of Carbon can be attributed to the residue formed from the calcination process while the presence of other elements can be attributed to contamination from the crucible or possibly reagents used for the synthesis. Impurity control can be enhanced by ensuring that the crucible is of high purity (preferably >99.9% Ni) and is well-cleaned before use since any surface oxidation on the crucible could also lead to contamination. Although HCl is commonly used for dissolving nickel, it can contain trace impurities depending on its source, the use of high-purity, analytical-grade HCl can reduce contamination. Similarly, the use of analytical-grade NaOH and preparing the solution using deionized water would reduce contaminants like sulfur (S), potassium (K), or sodium (Na). The use of double-distilled or deionized water to avoid trace amounts of metals like Fe, Cu, or K, can enhance the purity of the final product. Calcination in an inert atmosphere (such as a furnace with a controlled nitrogen or argon flow) can help prevent contamination from atmospheric gases like sulfur (S), which might lead to the formation of nickel sulfides (NiS), or carbon (C), which can lead to the formation of NiO in the presence of carbon impurities.

4.3. Characterization of Coating Mixture

The FTIR spectra for the epoxy resin, hardener and nanocomposite coating are shown in Figure 5. For epoxy resin, the peaks observed within the range 2970.71 to 2872.83 cm^{-1} were assigned to Stretching of C-H of CH_2 and CH aromatic and aliphatic chains [25] while the peak bands observed at 1738.52 to 1607 cm^{-1} were assigned to the Stretching C=C of aromatic rings. Other peaks detected at 1581.93 cm^{-1} , 1032.33 cm^{-1} , 914.31 cm^{-1} , 827.07 cm^{-1} and 770.84 cm^{-1} were attributed to the occurrence of Stretching of C-C of aromatic, Stretching of C-O-C of ethers, Stretching of C-O of oxirane group, Stretching of C-O-C of oxirane group and Rocking of CH_2 respectively [25, 26]. For the hardener, observed peaks at 3284.25 cm^{-1} ; 2949.70 - 2896.12 cm^{-1} ; 1591.20 cm^{-1} ; 1453.12 cm^{-1} ; 1207.25 cm^{-1} ; 1039.52 cm^{-1} ; 732.75 - 594.87 cm^{-1} ; 459.45 cm^{-1} were attributed to the occurrence of N-H stretching of amines or amides; C-H stretching vibration; C=C aromatic bending or stretching; C-H bending; C-N stretching;

C-O stretching; C-H out of plane bending and Metal-ligand bending respectively [27, 28]. The NiO nanoparticles had peaks at 3350 cm^{-1} and 1624 cm^{-1} due to the presence of Oxygen-Hydrogen bonding [29]. Other peaks observed at 944 cm^{-1} and 579 cm^{-1} were assigned to Nickel-Oxygen Tetrahedral Bonding and Metal-Oxygen stretching respectively [14, 29].

4.4. Corrosion Rate

Figures 6 to 8 give a summary of the corrosion rate, corrosion inhibition efficiency and weight loss of uncoated and coated mild steel with varying concentrations of epoxy-nickel oxide nanocomposites immersed in 3.5 wt. % NaCl for 60 days at room temperature. The effects of the variation in the inhibitor concentration and exposure time had very significant influence on the metal weight loss. In general, Figure 8 reveals that increase in the inhibitor concentration resulted into a decrease in metal weight loss, at room temperature which is an indication that the inhibitor retards the corrosion reaction process. Hence, epoxy-5 wt. % nickel oxide inhibitor concentration appeared to be the most promising inhibition concentration, in terms of the inhibitive effect. The increase in weight loss of the metal with increase in the exposure time implies that corrosion is a continuous and time dependent process. The resistance to corrosion process exhibited by the epoxy-nickel oxide nanocomposites can be explained in terms of the ability of the inhibitor to get adsorbed at the reaction sites (cathode and anode) on the metal surface by blocking the metal pores and forming a thin layer thereby preventing the free flow of electrons for corrosion reactions [30]. The corrosion resistance is also enhanced by the excellent barrier property of epoxy resin as well as the polar functional groups present in epoxy resin which serve as adsorption sites to the metal substrate [31]. The uncoated mild steel exhibited the highest corrosion rate of 96.47 $\text{g/m}^2/\text{day}$ in 3.5 wt. % NaCl within the first five days of immersion due to surface exposure, immediate electrolyte contact leading a series of electrochemical reactions which initiate corrosion, formation of corrosion products, surface roughness, disruption of protective films and oxygen availability. The electrochemical reactions involve the anodic reaction i.e. oxidation of mild steel which is primarily composed of iron which leads to release of Fe^{2+} ions into the solution and cathodic reaction i.e. reduction reactions typically involving water and oxygen. The ions and hydroxides formed often lead to the creation of corrosion products which can accelerate further corrosion if they do not effectively passivate the surface. Upon immersion, any pre-existing surface oxide layer can be disrupted thereby exposing fresh metal to the electrolyte which increases the corrosion rate. The high concentration of ions in NaCl enhances conductivity, allowing for rapid electrochemical reactions. Freshly exposed mild steel may have microstructural irregularities that can act as localized corrosion sites (anodic sites) heightening localized attack. The progressive reduction

of corrosion rate from 96.47 g/m²/day in 3.5 wt. % NaCl within the first five days to 10.15 g/m²/day in 3.5 wt. % NaCl after 60 days of immersion can be attributed to passivation, depletion of reactive species, stabilization of corrosion products and inhibition by passive films. Over time, corrosion products may form a layer that can partially protect the mild steel surface, slowing corrosion rates. As the corrosion process continues, the concentration of reactive species may decrease, reducing the driving force for the electrochemical reaction. The formation of stable corrosion products can reduce the diffusion of ions to the metal surface. If less soluble corrosion products form, they can adhere to the steel surface, leading to the establishment of passive film behaviour. The relatively low corrosion rate of 12.54 g/m²/day observed for the mild steel sample coated with only epoxy resin within the first five days of immersion in 3.5 wt. % NaCl can be attributed to the barrier properties of epoxy resin. The epoxy coating acts as a physical barrier, limiting direct contact of the salt solution with the underlying metal, drastically reducing initial corrosion rates. The mild steel samples coated with epoxy-nickel oxide nanocomposites containing 1, 2, 3 and 5 wt. % of nanoparticles exhibited corrosion rates of 8.69, 4.69, 9.09 and 1.09 g/m²/day respectively which were all lower than the corrosion rate observed for mild steel sample coated with only neat epoxy resin as well as the uncoated mild steel sample within the same period of exposure. This shows that nickel oxide nanoparticles have imparted some electrochemical properties that can enhance the overall resistance to corrosion. Nanoparticles can also alter the charge distribution and surface characteristics, potentially inhibiting localized corrosion rates. It can be seen from Figure 7 that the inhibition efficiency generally increases with an increase in the wt. % of NiO in the epoxy-nickel oxide nanocomposite coatings but decreases with prolonged exposure time. This can be attributed to prolonged exposure to saline conditions, stress, moisture, mechanical wear, leaching of nanoparticles into the solution, loss of contact of nanoparticles with the substrate and possible delamination which can compromise the protective properties of the coatings.

4.5. Effect of Temperature

The effect of temperature on the corrosion rate and corrosion inhibition efficiency of mild steel in 3.5 wt. % NaCl using epoxy-nickel oxide nanocomposites is illustrated in Figures 9 and 10 respectively. It is evident from Figure 9 that the increase in temperature significantly increased the corrosion rates of the coated and uncoated samples. The effect of temperature on corrosion rate is governed by an Arrhenius type relationship [32] shown in equation 7.

$$CR = A \exp \left(-\frac{E_a}{RT} \right) \quad (7)$$

Where CR is the corrosion rate, R is the universal gas constant, T is the absolute temperature and A is a

pre-exponential factor.

Computed values of activation energy, E_a from Arrhenius plots for the various samples (Figures 11 to 16) using the linear form of equation 7 were 10.62, 11.04, 11.10, 11.99, 15.26 and 23.32 kJ/mol for the blank MS sample, neat epoxy coated MS sample, epoxy-1.0 wt. % coated MS sample, epoxy-2.0 wt. % coated MS sample, epoxy-3.0 wt. % coated MS sample and epoxy-5.0 wt. % coated MS sample respectively. This trend indicates that less energy barrier is to be overcome in the blank sample for corrosion to take place compared to the neat epoxy resin coated sample and higher activation energy is required to initiate corrosion in samples protected by epoxy-NiO nanocomposite coatings. The decrease in inhibition efficiency of NiO-epoxy coatings at elevated temperatures can be attributed to a combination of factors and mechanisms including accelerated corrosion processes due to increased electrochemical activity; degradation of the coating structure, leading to microcracking, delamination, or loss of coating adhesion; increased permeability of the coating at higher temperatures, allowing chloride ions to penetrate and reach the mild steel substrate; alteration of the NiO particles' dispersion or surface interactions, which weakens their corrosion-inhibiting properties; thermal softening of the epoxy resin, increasing its susceptibility to ion penetration and decreasing the barrier properties.

Epoxy coated Mild steel samples immersed in 3.5 wt. % NaCl exhibited the least percentage inhibition efficiencies of 24.29%, 25.97% and 23.55% at temperatures of 30 °C, 40 °C and 60 °C respectively while Mild steel samples coated with Epoxy- 5 wt. % NiO had the highest percentage inhibition efficiency values of 81.07%, 78.54% and 70.39% at temperatures of 30 °C, 40 °C and 60 °C respectively and highest activation energy of 23.32 kJ/mol due to the enhanced corrosion resistance induced by the presence of Nickel oxide nanoparticles in epoxy resin.

4.6. Surface Roughness Analysis

The surface roughness values obtained from AFM Characterization of selected samples after 60 days of immersion in 3.5 wt. % NaCl are presented in Table 3 while the AFM micrographs are illustrated in Figures 17 to 20. The progressive reduction of surface roughness values from the blank with average value of 0.1854 to the pure epoxy coated mild steel with average value of 0.1206 and subsequently to the epoxy-NiO nanocomposite coatings containing 1.0 and 3.0 wt. % NiO with average values of 0.1047 and 0.0452 respectively confirm that the corrosion rate is reduced as the concentration of NiO is increased in the epoxy resin. Lower surface roughness values generally indicate better protection against corrosion, as a smoother surface is less prone to localized corrosion or degradation which implies that lower surface roughness values generally enhance percentage inhibition efficiency. As NiO concentration increases in the epoxy resin, the surface roughness decreases significantly which

suggests that the NiO nanocomposites are contributing to a more uniform, smooth surface during immersion in 3.5 wt.% NaCl, indicative of better corrosion protection. The inhibition efficiency of 82.46% after 60 days of immersion in 3.5 wt. % NaCl for pure epoxy resin coated MS sample increases when 1.0 wt. % NiO is added to the epoxy resin, reaching a peak of 88.07% after 60 days of immersion in 3.5 wt. % NaCl. This is likely because the 1.0 wt. % NiO concentration provides a balance between optimal coating thickness, barrier properties, and smoothness (as seen in the relatively low surface roughness of 0.1047 μm). For the 3.0 wt. % NiO nanocomposite, the inhibition efficiency decreases to 81.38%, despite the surface roughness reaching its lowest value of 0.0452 μm suggesting that while the coating is very smooth, the higher NiO concentration might have introduced other factors such as excessive thickening, porosity, or adhesion issues that reduce its overall corrosion protection compared to the 1.0 wt. % NiO coating. The decrease in inhibition efficiency for 3.0 wt. % NiO might also be due to changes in the coating's structural integrity at higher NiO concentrations, where the coating may become too rigid or less flexible, making it more prone to cracking and reduced protection over time.

4.7. ANN Modelling of Test Data

Corrosion test data obtained from 80 experimental runs were successfully modelled using ANN with minimal errors (Appendix 1). 56 cases corresponding to 70% of test data were used for training the network and 24 cases corresponding to 30% of test data was used for testing the efficacy of the network. The ANN model was statistically significant with Mean Absolute Error of 0.7803 and 2.2058 for corrosion rate and inhibition efficiency respectively while the Mean Square Error (MSE) was 2.5460 and 10.4478 for corrosion rate and inhibition efficiency respectively. The model had sum of squares error and average overall relative error of 0.981 and 0.018 respectively for the training component and values of 3.190 and 0.043 for the sum of squares error and average overall relative error respectively for the testing component (Figure 21). The parameter estimates are illustrated in Figure 22. ANN modelling of corrosion test data revealed that inhibitor concentration had a normalized importance contribution coefficient to the corrosion rate and inhibition efficiency of 0.177 corresponding to 30.4% while exposure time played a more significant role with a normalized importance contribution coefficient to the corrosion rate and inhibition efficiency of 0.583 corresponding to 100% whereas temperature had a normalized importance contribution coefficient to the corrosion rate and inhibition efficiency of 0.240 corresponding to 41.1% (Figures 23 and 24). ANN modelling of test data showed very high correlation between obtained experimental data and predicted values by ANN with little residuals and deviation as illustrated in Figures 25 to 28. Residuals are the differences between the actual values (observed data) and the predicted values (from the model). They provide insight into

how well the model is fitting the data and if any patterns or issues exist that could affect the predictions. Since the residuals are randomly scattered around zero, with no discernible pattern, this indicates that the ANN model is performing well and has captured the underlying trend in the data which implies that the model is accurately predicting the corrosion rates and inhibition efficiencies across various conditions, without systematic biases related to factors like temperature, inhibitor concentration, or exposure time. The few deviations observed at lower and higher concentrations of NiO nanoparticles are likely due to a combination of factors, including inadequate surface coverage at low concentrations, nanoparticle aggregation at high concentrations, temperature effects, and model limitations. The nonlinear nature of corrosion inhibition means that both very low and very high concentrations can lead to reduced effectiveness, with an optimal concentration range where the inhibition efficiency is maximized.

5. Conclusion

The synthesis of NiO nanoparticles via chemical precipitation technique with average crystallite size of 23 nm was confirmed by X-ray Diffraction (XRD) and Energy Dispersive X-ray Analysis (EDX).

The corrosion rate of mild steel in 3.5 wt. % NaCl generally decreased with increasing exposure time and increasing inhibitor concentration but increased with rise in temperature.

The maximum inhibition efficiency of 99.4% was obtained after 24 hours for inhibitor concentration of 5.0 wt. % NiO in epoxy resin at a temperature of 25 °C while the corrosion rate reduced from 59.86 to 3.07 $\text{g}/\text{m}^2/\text{day}$.

Surface Roughness values obtained from Atomic Force Microscopy (AFM) of epoxy coatings containing NiO nanoparticles were significantly lower than the pure epoxy-coated and blank steel samples in 3.5 wt. % NaCl.

Values for corrosion rate and percentage inhibition efficiency obtained from experiments were close to the corresponding values predicted by the Artificial Neural Network (ANN) model.

The ANN model was statistically significant with Mean Absolute Error of 0.7803 and 2.2058 for corrosion rate and inhibition efficiency respectively while the Mean Square Error (MSE) was 2.5460 and 10.4478 for corrosion rate and inhibition efficiency respectively.

The ANN model revealed that the independent variable importance was of the order Exposure Time > Temperature > Inhibitor Concentration with coefficients of 0.177, 0.583 and 0.240 for inhibitor concentration, exposure time and temperature respectively.

Abbreviations

ANN	Artificial Neural Network
CR	Corrosion Rate

E _a	Activation Energy
FTIR	Fourier Transform Infrared Spectroscopy
GDP	Gross Domestic Product
IE	Inhibition Efficiency
MS	Mild Steel
MSE	Mean Square Error
NPs	Nanoparticles
SEM-EDX	Scanning Electron Microscopy-Energy Dispersive X-ray Analysis
TEPA	Tetraethylenepentamine
UV	Ultraviolet radiation
XRD	X-ray Diffraction

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Conflicts of Interest

The authors declare no conflicts of interest.

Appendix

Table A1. Summary of Corrosion Rate and Inhibition Efficiency data modelled by ANN.

Case No.	Inhibitor Conc. (wt. % NiO)	Time (hours)	Temp. (°C)	Experimental Corrosion Rate (g/m ² /day)	Experimental Inhibition Efficiency (%)	Predicted CR (g/m ² /day)	Predicted IE (%)	Error in Prediction (CR)	Error in Prediction (I. E.)
1	0	24	25	59.86	87.42	52.29	93.92	7.57	-6.5
2	1	24	25	40.86	91.42	48.58	96.18	-7.72	-4.76
3	2	24	25	18.93	96.02	18.55	93.8	0.38	2.22
4	3	24	25	41.07	91.37	40.72	90.63	0.35	0.74
5	5	24	25	3.07	99.4	3.67	95.44	-0.6	3.96
6	0	120	25	12.54	87	13.22	90.13	-0.68	-3.13
7	1	120	25	8.69	90.99	8.84	93.31	-0.15	-2.32
8	2	120	25	4.69	95.14	2.2	93.57	2.49	1.57
9	3	120	25	9.09	90.56	5.5	90.27	3.59	0.29
10	5	120	25	1.09	98.87	1.36	95.5	-0.27	3.37
11	0	240	25	6.91	86.26	7.15	87.2	-0.24	-0.94
12	1	240	25	4.57	90.91	4.7	91.8	-0.13	-0.89
13	2	240	25	2.99	94.05	1.98	92.23	1.01	1.82
14	3	240	25	5.37	89.32	4.02	87.67	1.35	1.65
15	5	240	25	1.24	97.53	1.38	95.09	-0.14	2.44
16	0	360	25	4.73	86.04	4.6	86.36	0.13	-0.32
17	1	360	25	3.1	90.85	3.15	91.13	-0.05	-0.28
18	2	360	25	2.12	93.74	1.62	92.75	0.5	0.99
19	3	360	25	3.8	88.79	3.07	88.75	0.73	0.04
20	5	360	25	0.84	97.52	1.29	95.28	-0.45	2.24
21	0	480	25	3.79	85.35	3.99	86.29	-0.2	-0.94
22	1	480	25	2.42	90.65	2.7	91.33	-0.28	-0.68

Case No.	Inhibitor Conc. (wt. % NiO)	Time (hours)	Temp. (°C)	Experimental Corrosion Rate (g/m ² /day)	Experimental Inhibition Efficiency (%)	Predicted CR (g/m ² /day)	Predicted IE (%)	Error in Prediction (CR)	Error in Prediction (I. E.)
23	2	480	25	1.8	93.04	1.87	91.76	-0.07	1.28
24	3	480	25	3.22	87.56	3.31	86.82	-0.09	0.74
25	5	480	25	1.03	96.02	1.38	94.94	-0.35	1.08
26	0	600	25	3.22	84.8	3.21	85.87	0.01	-1.07
27	1	600	25	2.06	90.27	2.23	91.08	-0.17	-0.81
28	2	600	25	1.66	92.16	1.82	91.7	-0.16	0.46
29	3	600	25	2.83	86.64	3.11	86.7	-0.28	-0.06
30	5	600	25	1.08	94.9	1.38	94.92	-0.3	-0.02
31	0	720	25	2.85	84.22	2.83	84.4	0.02	-0.18
32	1	720	25	1.89	89.53	2	90.2	-0.11	-0.67
33	2	720	25	1.58	91.25	1.75	91.46	-0.17	-0.21
34	3	720	25	2.63	85.44	3.01	86.31	-0.38	-0.87
35	5	720	25	1.1	93.91	1.36	94.86	-0.26	-0.95
36	0	840	25	2.57	83.73	2.43	84.15	0.14	-0.42
37	1	840	25	1.66	89.49	1.75	90.09	-0.09	-0.6
38	2	840	25	1.44	90.89	1.77	91.25	-0.33	-0.36
39	3	840	25	2.43	84.62	2.98	85.94	-0.55	-1.32
40	5	840	25	1.18	92.53	1.37	94.79	-0.19	-2.26
41	0	960	25	2.3	83.77	2.48	82.16	-0.18	1.61
42	1	960	25	1.47	89.62	1.77	88.93	-0.3	0.69
43	2	960	25	1.36	90.4	1.77	90.69	-0.41	-0.29
44	3	960	25	2.24	84.19	3.06	85.02	-0.82	-0.83
45	5	960	25	1.07	92.45	1.37	94.63	-0.3	-2.18
46	0	1080	25	2.16	83.1	2.4	82.66	-0.24	0.44
47	1	1080	25	1.4	89.05	1.75	89.11	-0.35	-0.06
48	2	1080	25	1.26	90.14	1.63	91.29	-0.37	-1.15
49	3	1080	25	2.14	83.25	2.84	86.07	-0.7	-2.82
50	5	1080	25	1.07	91.63	1.32	94.84	-0.25	-3.21
51	0	1200	25	1.99	82.93	1.4	81.32	0.59	1.61
52	1	1200	25	1.31	88.77	1.13	88.3	0.18	0.47
53	2	1200	25	1.22	89.53	1.54	91.01	-0.32	-1.48
54	3	1200	25	2	82.85	2.6	85.59	-0.6	-2.74
55	5	1200	25	1.04	91.08	1.31	94.76	-0.27	-3.68
56	0	1320	25	1.88	82.8	1.9	79.79	-0.02	3.01
57	1	1320	25	1.24	88.66	1.45	87.18	-0.21	1.48
58	2	1320	25	1.21	89.93	1.35	91.24	-0.14	-1.31
59	3	1320	25	1.96	82.07	2.49	86.14	-0.53	-4.07

Case No.	Inhibitor Conc. (wt. % NiO)	Time (hours)	Temp. (°C)	Experimental Corrosion Rate (g/m ² /day)	Experimental Inhibition Efficiency (%)	Predicted CR (g/m ² /day)	Predicted IE (%)	Error in Prediction (CR)	Error in Prediction (I. E.)
60	5	1320	25	1.06	90.3	1.24	94.88	-0.18	-4.58
61	0	1440	25	1.78	82.46	2.07	79.95	-0.29	2.51
62	1	1440	25	1.21	88.07	1.52	87.52	-0.31	0.55
63	2	1440	25	1.17	88.47	1.62	90.46	-0.45	-1.99
64	3	1440	25	1.89	81.38	2.87	84.66	-0.98	-3.28
65	5	1440	25	1.04	89.75	1.33	94.59	-0.29	-4.84
66	0	5	30	9.6	24.29	8.35	31.15	1.25	-6.86
67	1	5	30	7.54	40.54	6.26	47.09	1.28	-6.55
68	2	5	30	6.17	51.34	3.97	63.43	2.2	-12.09
69	3	5	30	5.14	59.46	6.12	56.32	-0.98	3.14
70	5	5	30	2.4	81.07	4.21	76	-1.81	5.07
71	0	5	40	10.66	25.97	10.61	27.76	0.05	-1.79
72	1	5	40	7.88	45.27	7.83	44.06	0.05	1.21
73	2	5	40	7	51.39	3.85	62.04	3.15	-10.65
74	3	5	40	6.17	57.15	6.8	54.36	-0.63	2.79
75	5	5	40	3.09	78.54	4.13	75.59	-1.04	2.95
76	0	5	60	14.15	23.55	14.76	23.01	-0.61	0.54
77	1	5	60	11.02	40.46	11.11	39.39	-0.09	1.07
78	2	5	60	9.4	49.22	3.87	59.69	5.53	-10.47
79	3	5	60	8.85	52.19	8.44	51.32	0.41	0.87
80	5	5	60	5.48	70.39	4.1	74.47	1.38	-4.08

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