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Enhancing the Corrosion Protection of Mild Steel using Nickel-Voltage-Controlled Electrodeposition

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Abstract
Tailoring the electrodeposition parameters to yield high performance coating for corrosion control in mild steel is important for its sustainability as an effective corrosion mitigation measure. Nickel film was electrodeposited on mild steel for corrosion inhibition at varied coating voltages. The influence of the coating voltage on the surface morphology with elemental percentage composition and the corrosion resistance of the coating was examined using scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS) and potentiodynamic polarization method respectively. Results obtained showed that the electrodeposited nickel film impacted an impressive corrosion inhibitory property on mild steel when compared to the uncoated samples. The study also indicated that corrosion rate of 0.0074996 mm/yr, 0.0005916mm/yr, and 0.00440 mm/yr where obtained with coating voltage values of 2, 4 and 7Volts respectively. This indicates that the coating voltage has significant effect on the corrosion resistance potential of nickel film. The optimal corrosion resistance performance of the coating material was achieved using coating voltage of 4Volts which yielded corrosion inhibition efficiency of 97.7%. The morphology of the samples deposited using the optimal setting indicated smoother surfaces with minimum corrosion affected areas in contrast to the one coated using extremely low and high voltage values. Thus, an optimal electrodeposition voltage at which nickel-coated mild steel exhibits significantly enhanced corrosion resistance has been identified. More also the correlation between the electrodeposition voltage, microstructural characteristics and the corrosion protective film integrity has been established. Hence, optimizing a single electrochemical parameter-coating voltage, is a cost- effective approach for enhancing the durability and sustainability of mild steel in corrosive environments for industrial applications.

Keywords: Corrosion, electrodeposition, coating voltage, surface morphology, protective film.

1. Introduction

Mild steel is an engineering material that has a lot of scientific and industrial applications. Its relative cost effectiveness, availability and impressive performance account for such wide applications. However, it is often prone to corrosion attack (Chima, 2021). Hence, it is obvious that the corrosion of mild steel remains a major challenge facing industries across the globe (Mohammed, *et al.*, 2022; Setyowati, *et al.*, 2021). Therefore, corrosion mitigation is a major concern to every researcher in the industries in order to ensure that the service life of a steel component is guaranteed. The metal surface could be modified through protective coating in order to improve its corrosion resistance potentials using scientific techniques (Fatahiamirdehi, *et al.*, 2024; Kaya, *et al.*, 2024; Ozkan, *et al.*, 2024). Electrodeposition of nickel film has been widely adopted as an effective strategic technique for corrosion control because of its cost effectiveness, impressive corrosion resistance performance and sustainability over a wide range of time (Yan, *et al.*, 2025; Cui, *et al.*, 2024). The nickel coating is effective in creating a physical barrier, which can isolate the steel from the corrosive medium and at the same time offer sacrificial protection under some certain conditions. The corrosion resistance performance of nickel coated film is highly dependent on some of its characteristics such as its surface morphology, grain structure and adhesion capability. On the other hand, the film characteristics is a function of the electrodeposition process parameters among which is coating voltage (Anderson, *et al.,* 2024; Tseluikin, *et al.,* 2021; Nunez-Perez, *et al.,* 2024).

The coating voltage controls the ion reduction rate, hydrogen evolution, and nucleation and grain growth mechanisms during the process of electrodeposition. Extremely low coating voltage could slow down the deposition rate which could give rise to thinner and incomplete coatings (Wijanarka, *et al.,* 2020). In contrast, excessive high voltage can yield rapid deposition that is associated with hydrogen gas evolution. This could lead to porous, rough and defected films. Therefore, the need to optimize the coating voltage becomes paramount if dense, good adherent and defect free nickel film that offers optimum corrosion resistance must be achieved Saha, *et al.,* 2018).

In the past, the electrochemical and mechanical attributes of mild steel towards electro-plating with Zn-Al has been studied and it was revealed that Al introduction into the coatings altered both aforementioned properties of mild steel (Popoola et al. 2012). Also, the electrochemical deposition of zinc on mild steel was investigated using cyclic voltammetric and chronopotentiometry techniques (Fazazi et al. 2019). Recently, electrodeposition of Zn and Zn-$CaCO_3$ composite on mild steel purposely to resist corrosion in biodiesel storage tank was examined. The result showed enhancement of the substrate's corrosion performance for the mild steel coated with Zn-$CaCO_3$ (Fardilah et al. 2025). Many of the previous studies focused primarily on bath composition or current density. However, there is little or no systematic research investigation that have explained the effect coating voltage variations on the properties of the film and the corresponding corrosion resistance behavior. Considering the fact that the performance of the coated material is highly dependent on these process parameters, it is obvious that the insight on how to optimize the coating process has been omitted (Setyowati, *et al;* 2021). Therefore, establishing how specific coating voltages influence the microstructure and uniformity of the nickel layers, which directly governs corrosion resistance performance is paramount for optimal protective film design in order to tailor the deposition parameters to yield high performance coating for corrosion control in mild steel.

Therefore, this study sought to coat nickel on mild steel substrate using electrodeposition method under varied coating voltages. The influence of this process factor on the nickel film characteristics which consequently affect the corrosion resistance was examined using characterization techniques which include scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS), x-ray diffraction (XRD), and EIS and potentiodynamic polarization. The study therefore offers a good insight on the comprehensive understanding of the coating voltage with film property and corrosion performance relationship that have identified the optimal deposition condition for effective corrosion mitigation in mild steel.

2.0 Materials and methods.

2.1 Materials

The materials used in this study include nickel chloride, nickel sulphate, boric acid nickel initial bright, nickel anode, chromic acid and tetra oxo sulphate IV acid. The equipment used for the electrodeposition process include the nickel plating bath, and double compartment economic rinsing bath, rectifier, compressed air to bubble the nickel electrolyte and drying machine. The electrochemical test was done using electrochemical analyzer equipment with Model: CHI604E while the microstructural characterization were achieve using scanning electron microscopy (SEM) and energy dispersion spectrometer (EDS).

2.2 Methods

2.2.1 Sample Surface Pre-treatment Process

Prior to the electrodeposition process, metal coupons were cut to a convinent sizes of 40 x 45cm. They were further subjected to pre-coating operations involving cleaning, descaling and pickling. These ensured that samples are free from dirt and rust that will prevent bonding or adhesion of the coating film on the metal during deposition process. Cleaning was done by polishing while pickling was carried out by dipping samples in a pickling solution containing 70 g/l of hydrogen chloride acid solution within a period of 30 minutes at ambient temperature condition. They were thereafter rinsed in water sufficiently enough to avoid introducing the pickling solution into the plating electrolyte. They samples were further washed using a surfactant by dropping them into the electrolyte which removed all the contaminant substances on their surfaces.

2.2.2 Electrodeposition Process

The electroplating process was achieved using ASTM A967 standards.The experimental set-up was made of an electroplating bath containing the nickel electrolyte, the plating nickel (anode), the work piece to be plated (mild steel coupon) which served as the cathode. Both the cathode and the sacrificial anode were immersed into the electrolyte.

When voltage of 7volt was passed through the rectifier, the metal salt in aqueous solution was observed to undergo ionization leading to oxidation taking place at the anode and reduction at the cathode were the coupons were uniformly deposited with nickel for 10mins. The operational temperature for the nickel plating was maintained at 80°C with a pH of 5.0-6.0.

2.2.3 Electrochemical Test

The corrosion characteristics of the samples of mild steel were determined using the potentiodynamic polarization resistance method which conformed to ASTM G199 – 09 (2014 Standard Guide for Electrochemical Measurement). The Tafel polarization method was used to determine the Tafel plots, open circuit potential (OCP) and electrochemical impedance spectroscopy (EIS). The potential was scanned from -1.0V to +1.0Vat a scan rate of 0.25 mV/s. The open circuit test was made to run for a period of 3600 seconds. The potential waveform was applied as a function of time. The linear polarization resistance (Ωcm^2) was determined using electrochemical method by recording the corresponding values of current as function of the applied voltage. The change in the applied voltage with respect to change in the measured current gave the value of polarization resistance (Rp). The values of the logarithm of current was ploted as a function of potential to get the Tafel plots shown in figures 1 - 4.The gradients of the Tafel curves for both the cathodic and anodic sides were considered as the cathodic Tafel slope (βc) and anodic Tafel slope (βa) respectively. Thereafter, Stern Geary equation was used to determine other parameters which include the Stern-Geary constant (B), corrosion current density, corrosion rate, and corrosion inhibition efficiency of the coating are shown in equation1 – 4. The Faraday's law expressed in terms of thickness loss over time which is presented in equations 5 was used to determine the corrosion depth penetration expressed in terms of thickness loss over time (Srivastava *et al;* 2006). The results obtained are shown in Table 1.

$$B = \frac{\beta a \beta c}{2.303(\beta a+\beta c)} \quad (1)$$

$$Corrosion\ current\ densit(\mathrm{Icorr}) = \frac{B}{Rp} \quad (2)$$

$$Corrosion\ Rate = \frac{\mathrm{KIcorrM}}{\rho} \quad (3)$$

$$Corrosion\ inhibition\ efficiency = \frac{\mathrm{Icorr(Control\ sample)} - \mathrm{Icorr(Coated\ samplel)}}{\mathrm{Icorr(Control\ sample)}} \quad (4)$$

$$Corrosion\ penetration\ depth = \frac{\mathrm{KIcorrM}}{nF\rho} \quad (5)$$

Where B is Stern-Geary Constant, Rp is Polarization resistance inA/cm^2, K is conversion factor

represented as 3.27×10^{-3}, M represents the atomic molar mass of steel in g/mol = 55.85 g/mol, n

represents the number of electrons of the corrosion reaction in steel, ρ represents the density of steel ($7.87g/cm^3$) and F represent Faraday's constant = 96,500 C/mol.

2.0.1 Microstructural Characterization

The morphology of both the coated and uncoated samples of the mild steel coupon was examined after being subjected to a corrosion test using a scanning electron microscope with energy dispersive spectroscopy (SEM/EDS) analysis under high-vacuum evaporation at 5000 x. The SEM microscope produced the image of the sample by scanning the surface with a focused beam of electrons in a vacuum at a point within a specific time. It is the interaction of the electron beam with the atoms of the specimen at various depths that formed the image and the constituent elements. The phase composition analysis was done using computerized X-ray diffractometer that uses Cu Kα radiation at a scan speed of 4^0/min 2Θ which reveal the phase constituents in the composite metals.

3.0 Result and Discussion

The results obtained in this study are presented in figures 1 – 5, Table 1 and Plates 1 – 6.

3.1 Corrosion Study.

The electrochemical study of the corrosion process for both uncoated and uncoated samples of the mild steel were studied using Tafel polarization curves presented in figures 1 – 4. The SEM micrographs shown in plate 1 – 4 were used to study the morphology of the corroded metal surfaces and confirm the results which indicate the severity of the corrosion on each sample subjected to aggressive corrosion condition. The results presented in these figures and plated also were used to explain the effect of coating parameter such coating voltage on the corrosion resistance potential of the coated samples.

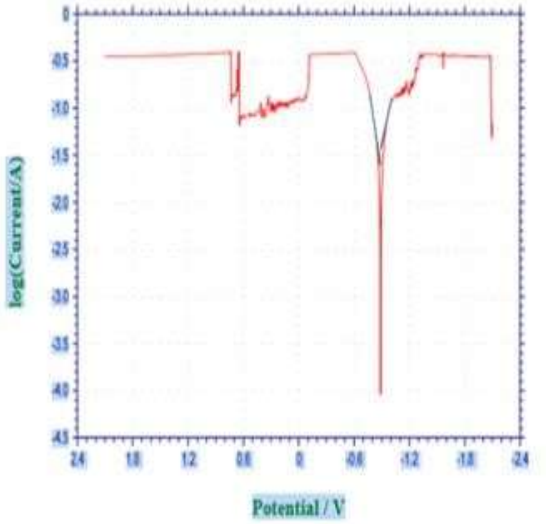


Figure 1: Tafel Plot for Control Sample

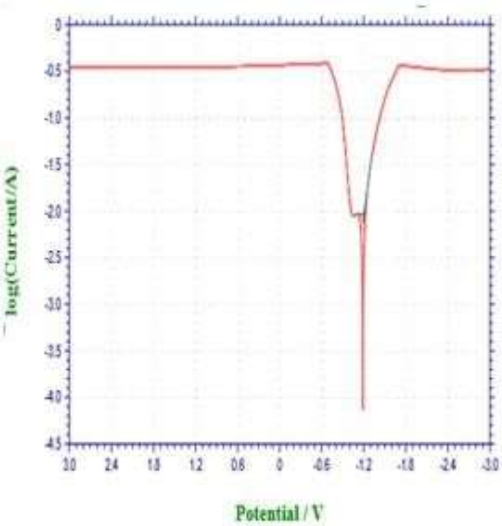


Figure 2: Tafel Plot for Sample Coated with 2V

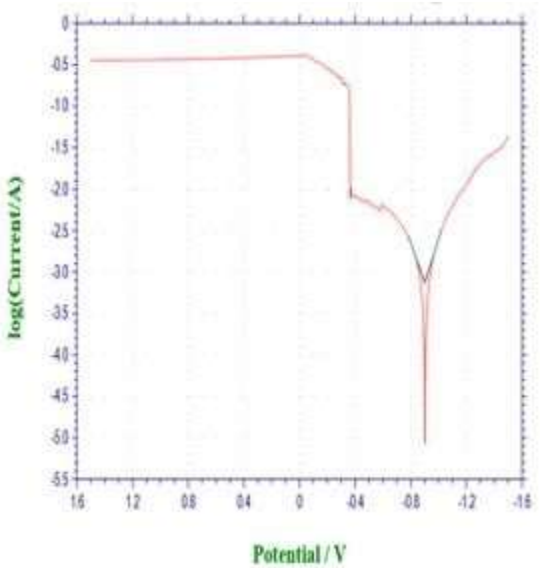


Figure 3: Tafel Plot for Sample Coated with 4V

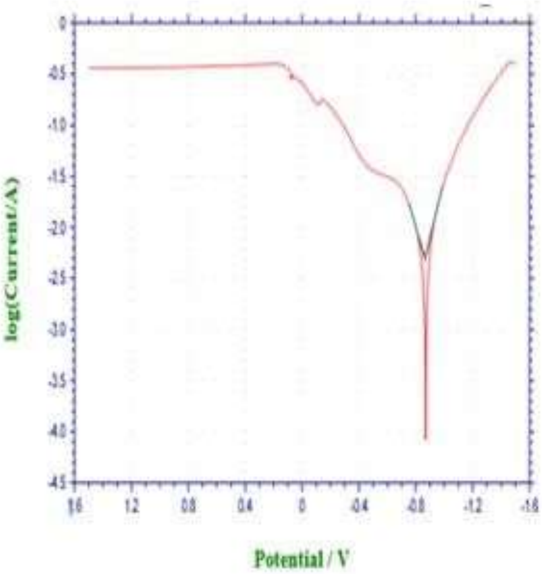


Figure 4: Tafel Plot for Sample Coated with 7V

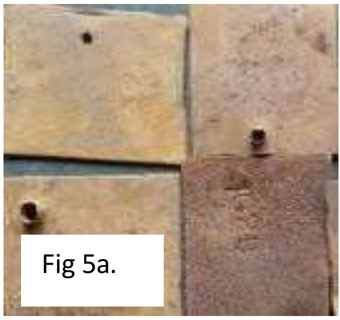


Fig 5a.

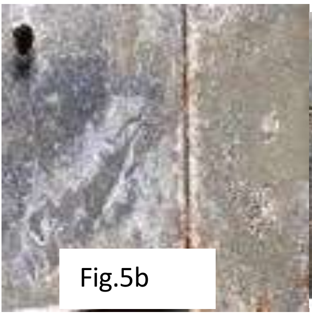


Fig.5b


Fig 5c

Figure 5a is uncoated sample subjected to corrosion, figure 5b is coated sample subjected to corrosion while figure 5c coated sample that was not subjected to corrosion.

3.1.1 The Tafel Polarisation Analysis

Table 1: Potentiodynamic Polarization Parameter for Samples

Sample	Anodic slope (βa)	Cathodic slope (βc)	Stern-Geary constant (B)	Corrosion current density (A/cm^2)	Linear polarization resistance (Ωcm^2)	Corrosion rate (mm/yr)	Corrosion penetration depth (mm/yr)	Corrosion Inhibition Efficiency (%)
Uncoated sample	6.499	4.479	1.15137	1.15137	1	0.026716	1.38 x 10^{-7}	
Sample coated at 4V	4.775	5.363	1.0968	0.02550	43	0.000592	3.065 x10^{-9}	97.7
Sample coated at 7V	4.808	5.782	1.1398	0.18977	6	0.00440	2.281 x10^{-8}	83.49
Sample coated at 2V	6.106	5.811	1.2928	0.32321	4	0.007499	3.885 x10^{-8}	71.92

Figure 1 indicates the polarization curves of the control of mild steel which was not coated with nickel film while the Tafel plots of the nickel coated samples which were processed at different voltages of 4, 7 and 2Volts were presented in figures 2 – 4. The Tafel plots were used to determine the anodic and cathodic slopes alongside with corrosion potential and corrosion current density for each of the samples tested for corrosion resistance. The electrochemical parameters obtained from the potentiodynamic curves are presented in Table 1. From the results obtained, it was observed that a combination of cathodic and anodic slopes of

4.479 and 6.499 were obtained for the uncoated sample while the respective values of (5.363, 4.775), (5.782, 4.808) and (5.811, 6.106) were obtained for samples coated at the voltage values of 4, 7 and 2Volts. Cathodic slope measures the rate the current decayed after the voltage was turned off. Hence, it was observed that for the uncoated sample, there was a relatively fast decay in the current which resulted in a lower cathodic slope of 4.479. In contrast, the coated samples were associated with slower decay in the current, which yielded higher cathodic slopes ranging from 5.363 – 5.811 as seen in the results presented in Table 1. The trend observed in the coated samples is traced to the fact that the electrodeposited nickel coating prevented the steel samples from reverting back to its original oxide form as fast as it ought to have done if the corrosion process proceeded in its normal way. Consequently, a longer time was taken for the current to decay.

In contrast, the anodic slope measured the rate at which the current rises after the voltage was turned on. The comparison of the anodic slopes of the uncoated sample with reference to the coated ones shows that for uncoated sample, a relatively fast rise in the current was noted. At the same time, for the coated sample, a slower rise in current resulted to a lower anodic slope. This is due the presence of the barrier created by the coating on the metal which prevented the current from rising at a faster rate. In this way, the size of the slope on either of the Tafel plot decided the reaction rate which consequently determined the extent of corrosion rate. Notwithstanding, some other factors such diffusion and experimental condition that affect reaction mechanism was believed to have affected the corrosion behavior of the composite metal.

Furthermore, the effect of the coating voltage on the corrosion resistance potential of the coated samples was observed by comparing the corrosion current density values obtained using varied coating voltage values. The comparison indicates that the respective corrosion current density values of 0.3232, 0.02550 and 0.18997A/cm^2 were obtained using 2, 4 and 7Volts. This implies that the corrosion resistance potential of the coated samples increased with increase in the coating voltage from 2V till a certain threshold value of 4V, above which it began to depreciate as observed at 7Volt value. This is based on the fact that a lower value of the corrosion current is associated with higher and better corrosion resistance capacity. This was affirmed by the corresponding corrosion rate values of 0.0074996, 0.0005916, and 0.00440 mm/yr obtained with the coating voltage values of 2, 4 and 7Volts respectively. Hence, though electrodeposited nickel impacted good corrosion resistance quality on mild steel within the range of coating voltage studied, the optimal processing voltage was obtained at 4V which yielded the lowest corrosion rate value with corresponding corrosion inhibition efficiency of 97.7%.

Thus, it is evident from the results obtained in this study that coating voltage controls the rate of nickel ion reduction, and nucleation mechanism during deposition. Hence, at low voltage such as 2Volts, the deposition rate must have been sluggish which is assumed to have resulted in thinner or incomplete coatings that gave rise to a low corrosion resistance performance. More also, this could be traced to the fact that low voltage generates more particles that are not properly melted as observed by Setyowati, et *al;* 2020.This could affect the adherent property of the coating which consequently yields low bonding strength. Besides, it is strongly believed that such mixture of melted and unmelted particles gave rise to non-homogeneous coated film on the metal surface which could introduce pores at the metal surface. When the pressure of corrosion fluid becomes high on the surface of the metal, the coating film cracked as a result of the surface defect, and give way for the fluid to have contact with the metal being protected. This led to rise in corrosion rate.

In contrast, the coating voltage value of 7Volts was considered excessively high above the minimum threshold value which was observed to have induced rapid deposition accompanied by hydrogen evolution that led to porous, rough and brittle film. Therefore, optimizing the coating voltage is very paramount to achieve dense, adherent and defect free nickel film that offers superior corrosion resistance. This was confirmed by the SEM images of the samples shown in plates 2-4. The SEM micrograph of samples coated using excessively lower and high voltage values were found with more corrosion pits with larger corrosion affected areas appearing darker which is a clear indication of the occurrence of higher corrosion rate unlike the one coated using optimal voltage value that showed a smooth uniform homogeneous surface.

Generally, it was noted that the electrodeposited nickel coating studied was effective in corrosion protection on mild steel. This is traced to the fact that the electrodeposited nickel film acted as a physical barrier between the substrate (mild steel) and the corrosive environment (acid). Nickel on its own resists oxidation and chemical reaction. Hence, in this way, it has impacted its protective quality on the coated steel. It also form stable and adherent passive oxide layers as nickel oxide which were observed on the SEM micrographs. This oxide layer is believed to prevent the metal surface from being exposed from the corrosive environment. Besides, it also functioned as self- healing which reformed minor corrosion damages that enhanced the performance of the coating in corrosion mitigation. More also, from electrochemical point of view, nickel possess a high electrochemical stability that iron which makes it unlikely to be prone to electrochemical reaction leading to corrosion. Hence, its coating on substrate's surface is an effective for corrosion protection in mild steel. Notwithstanding, it was observed that coating voltage governs the rate of nickel ion deposition and its nucleation mechanism during deposition. Therefore, optimizing the coating voltage is necessary for the sustainability of an effective corrosion mitigation design.

3.1.2 Morphology and Microstructural Characterization

Plate 1 and 2 show the SEM images and the EDS spectrum of the uncoated and coated samples respectively. The nickel film layer deposited on the surface of the mild steel is shown in the SEM micrograph in light ash cloudy form and the composition of constitute elements reflected in the EDS spectrum. It was observed from the SEM image of the coated sample showed a smoother and more homogeneous film on the surface compared to the rough surface in the uncoated sample. The surface of the uncoated sample appeared darker with the presence of more corrosion pits shown as deep dark spots compared to the lighter colour of the coated metal surface. This showed clearly that corrosion rate decreased on the coated sample. The SEM micrographs shown in Plate 2- 4, also confirmed the effect of the coating voltage on the corrosion rate of the coated samples. Comparison of the morphologies of the surfaces shows that Plate 2 indicates a relatively heavier corroded surface with more severely pitted surface which is an evidence of extensive localized corrosion and significant material degradation as confirmed in the experimental results. The SEM micrograph also revealed that Plate 3 has relative more extensive surface irregularities and surface cracking, indicating accelerated corrosion processes compared to Plate 4.The EDS spectrum indicated the presence higher percentage composition of nickel as the coating element in the coated sample. The improved nickel composition is believed to have offered higher corrosion resistance observed on the electrochemical test.

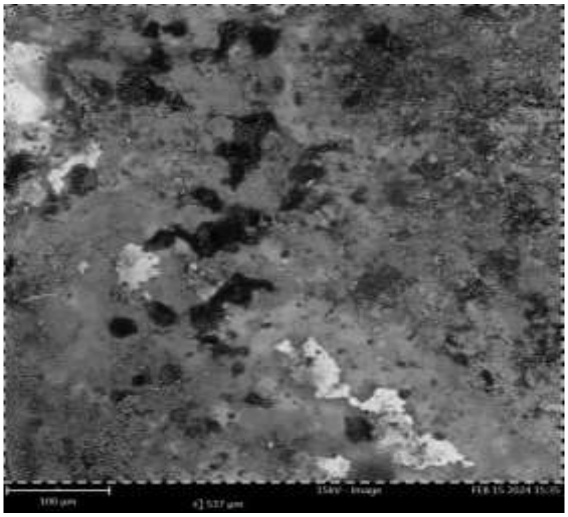


Plate 1: Control

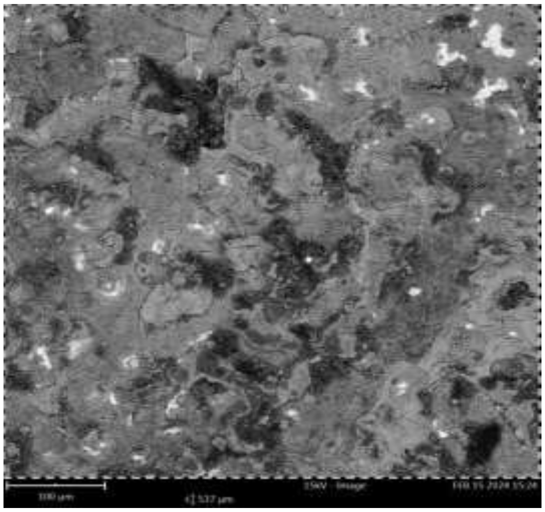


Plate 2: Sample coated at 2V

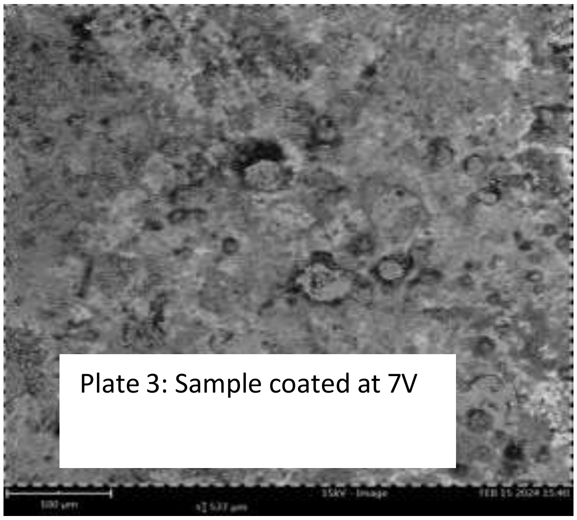


Plate 3: Sample coated at 7V

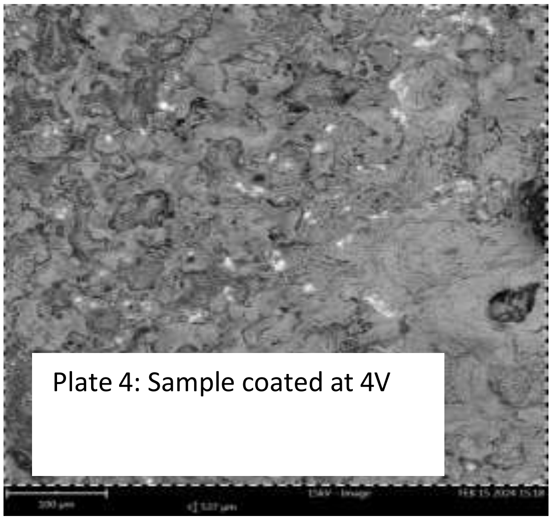


Plate 4: Sample coated at 4V

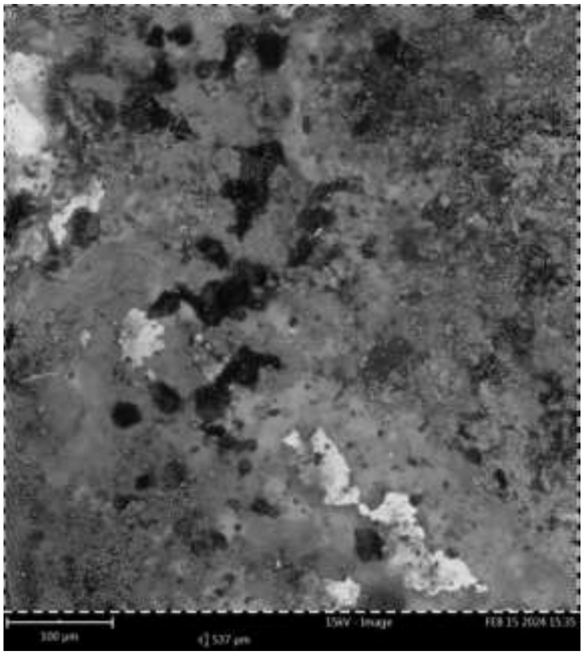
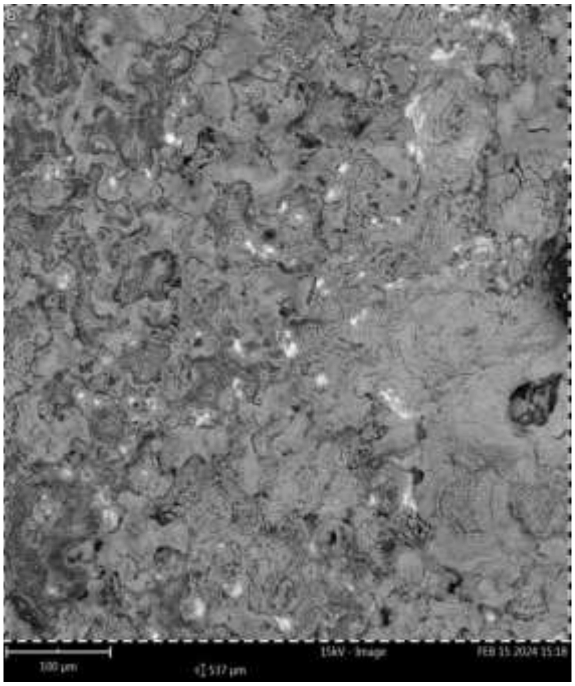


Plate 5 SEM/EDS Micrograph of control sample

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	30.45	73.52
6	C	Carbon	6.86	4.53
14	Si	Silicon	6.66	3.48
48	Cd	Cadmium	1.43	3.00
16	S	Sulfur	4.41	2.63
30	Zn	Iron	2.51	2.61
13	Al	Aluminium	5.17	2.59
12	Mg	Magnesium	5.67	2.57
19	K	Potassium	2.51	1.82
15	P	Phosphorus	1.92	1.11
28	Ni	Nickel	0.12	0.25
20	Ca	Calcium	1.41	1.05
22	Ti	Titanium	0.00	0.00



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	33.76	74.03
6	C	Carbon	6.65	4.54
11	Na	Sodium	3.34	2.64
16	S	Sulfur	1.86	2.05
48	Cd	Cadmium	0.43	1.66
17	Cl	Chlorine	1.04	1.27
14	Si	Silicon	1.09	1.05
13	Al	Aluminium	1.11	1.03
19	K	Potassium	0.74	1.00
12	Mg	Magnesium	0.96	0.80
20	Ca	Calcium	0.48	0.66
15	P	Phosphorus	0.54	0.57
28	Ni	Nickel	4.38	8.70
22	Ti	Titanium	0.00	0.00

Plate 6 SEM/EDS Micrograph of sample coated with nickel

4.0. Conclusion

The corrosion inhibitory characteristics of electrodeposited nickel film on mild steel have been established in a voltage optimized coating process. The mild steel surfaces coated using extremely low and high coating voltages demonstrate a relatively higher severity of corrosion attack than the one coated using optimal process parameter. The increased in corrosion rate, more advanced pitting morphology, and higher corrosion current density as measured by the electrochemical analysis correlate with the SEM microstructural analysis. The coating voltage directly influenced the rate of nickel ion deposition on mild steel substrate and its nucleation mechanism during deposition which determine the quality, surface morphology and protective ability. A comprehensive analysis of the relationship between coating voltage, film properties, and corrosion performance of electrodeposited nickel on mild steel revealed that an optimal coating voltage of 4V yielded the most protective film characteristics with superior corrosion resistance that achieved maximum corrosion inhibition efficiency of 97.7%. These findings confirm that optimizing the deposition voltage significantly enhances the corrosion protection performance.

5.0 Recommendation

In this study, it is recommended that moderate coating voltage of 4Volts should be used in electrodeposition process of nickel film on mild steel for corrosion protection. It is also recommended that further studies should the effect of multi-parameters on corrosion behavior of mild steel.

Acknowledgements

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Nomenclature

(βa) = Anodic slope

$(\beta c) =$ Cathodic slope

(Icorr) = Corrosion density (CR), mm/yr (IE) = Corrosion Inhibition efficiency, % (Rp) = Polarization resistance, Ωcm^2

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