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OPTIMIZING ELECTRODEPOSITION PARAMETERS FOR NI-MO COMPOSITE COATINGS: A STUDY, CORROSION RESISTANCE AND SURFACE MORPHOLOGY WITH NANOPARTICLE INCORPORATION

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Abstract

The demand for corrosion-resistant coatings, exploration of novel materials and techniques to enhance protective layers' performance. Electrodeposition of metal-nanoparticle composites offers a promising solution to meet these requirements. Among these composites, nickel-molybdenum (Ni-Mo) coatings have shown increase in corrosion enhansment and mechanical properties. This study focuses on optimizing the electrodeposition parameters to fabricate Ni-Mo improved corrosion resistance composite coatings and surface morphology. To get good coating characteristics, a nickel bath solution with varying concentrations of nanosized molybdenum particles was prepared. The composition of critical parameters was carefully investigated. Additionally, sodium dodecyl sulfate (SDS) as a surfactant was studied to understand its different characteristics on the coating's surface features. Characterization techniques, ie, scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX), were employed to analyze the surface morphology and elemental mixture of the coatings. Tafel extrapolation and impedance spectroscopy were used to verify electrochemical corrosion behaviour. To evaluate the coatings' corrosion resistance.

Key words: Corresion, Electrodeposition, Surface Morphology

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1. Introduction:

Electrodeposition is common employed technique for producing metal-nanoparticle composite coatings with increase in the properties for various industrial applications. Among these composites, nickel-molybdenum (Ni-Mo) Coatings have sparked considerable interest for superior corrosion resistance and mechanical properties [1]. The incorporation of nanosized molybdenum particles into the nickel matrix can further improve the coating's performance, making it a promising solution for protecting substrates from corrosive environments.

In this study, we aim to optimize the electrodeposition process parameters to achieve the desired characteristics of Ni-Mo composite coatings. By carefully controlling factors like bath mixture, pH, temperature, density of current, duty cycle, rotation speed, and deposition time, we can tailor the coating's properties and morphology. Furthermore, the influence of sodium dodecyl sulfate (SDS) as a surfactant will be investigated to assess its influence on the coating's surface features [2].

2. Characterization Results of Nanoparticles Composite Coatings

"Mild steel substrates with an exposed surface area of 3.5cm² x 2.5cm² were utilized for the Ni-Mo the electrodeposition procedure. The electrodeposition was conducted using a nickel bath solution that contains different amounts of nanosized Mo particles. The bath was developed by analytical grade chemicals and Millipore water. Prior to the electrodeposition, the bath solution underwent stirring at 1000 rpm using a magnetic stirrer and was subsequently sonicated with a probe sonicator [3].
The electroplated nickel electrolytic solution is shown in Table 4.1:

Table 1: Optimized nickel electrolytic solution

Composition (g/L)	Concentration (g/L)
NiSO4	65
MoSO4	7.5
Na2SO4	25
H3BO3	25
pH	3.75
Temperature (°C)	55°C
Current Density (mA/cm²)	62.5
Duty Cycle	80%
Temperature	55°C
pH	4.2
Rotation Speed	110 rpm
Deposition Time	90 minutes
Current Density	8 A/cm²

The nickel bath solution utilized for the electrodeposition contained different amounts of nanosized Mo particles. The specific composition of the nickel electrolytic solution was carefully chosen to achieve the required properties and characteristics in the plated deposit. Nickel Sulfate (NiSO4): Nickel sulfate is a crucial component as it provides the source of nickel ions necessary for the metal coating of nickel onto the mild steel substrates. The concentration of 65 g/L was likely determined the desired plating thickness and quality. Molybdate (MoSO4): Molybdate is added as nanosized particles to the electrolytic bath to incorporate molybdenum into the deposited layer. The concentration of 7.5 g/L indicates the specific amount of molybdenum desired in the final plated coating [4].

Sodium Sulfate (Na2SO4): Sodium sulfate is often used as a supporting electrolyte in electrodeposition processes to enhance the conductivity of the electrolytic bath. Its concentration of 25 g/L helps maintain the necessary ionic strength. Boric Acid (H3BO3): Boric acid is added electrolyte solution to act as a pH buffer, helping to stabilize the pH of the solution during electrodeposition. A concentration of 25 g/L serves to maintain the desired pH level during the process. pH refers to the level of acidity or alkalinity in the electrolytic bath solution used for the Ni-Mo electrodeposition process. In this case, the pH value is 3.75. The pH plays a crucial role in the electroplating process as it influences the rate of electrodeposition, the standard of the plated layer, and the distribution of metal ion on the substrate. Maintaining the appropriate pH level is essential to accomplish the desired characteristics of the plated coating [5].

The temperature is specified as 55°C. The temperature significantly affects the kinetics of the electroplating reaction, with higher temperatures often leading to faster deposition rates. Additionally, temperature can impact the adhesion and morphology of the plated layer. The chosen temperature of 55°C indicates a high-temperature electroplating process that may be suitable for specific applications requiring specific characteristics in the plated material.The duty cycle in the context of electrodeposition refers to the percentage of time the electroplating current is active during the plating process. In our provided Table 1, the duty cycle is specified as 80%. This parameter becomes particularly significant in pulse plating techniques, where the current is cyclically switched ON and OFF. With an 80% duty cycle, it means that the electroplating current is active for 80% of each cycle [6, 7]. The duty cycle plays a crucial role in influencing the thickness and properties of the plated layer. Therefore, optimizing the duty cycle is vital to achieve the desired results in the electrodeposition process. By adjusting the duty cycle, we can effectively control the electroplating process and achieve coatings with specific characteristics and properties tailored to our requirements.Rotation speed represents the speed at which the mild steel substrates are rotated during the electrodeposition process. In the table, the rotation speed is specified as 110 rpm (revolutions per minute). Rotating the substrates during plating helps ensure a more uniform distribution of metal ions and promotes better adhesion of the plated layer. The chosen rotation speed of 110 rpm indicates a moderate rotational rate that is optimal for achieving the desired plating characteristics. Deposition time refers to the duration for which the substrates are exposed to the electroplating process [8]. In this case, the deposition time is 90 minutes. The coating time significantly influences the thickness of the plated layer; usually, a longer deposition period results in a thicker deposit. However, the deposition time should be carefully chosen to avoid overplating and to achieve the desired characteristics.

3. Method Electrodeposition for Ni-Mo Composite Coating

In this study, we have successfully fabricated Ni-Mo composite coatings using an electrodeposition method. For the process, we employed mild steel substrates with an exposed area of 3.5 cm² x 2.5cm² as the base material. The nickel bath solution used for the electrodeposition contained nanosized Mo particles in different amounts [9]. To ensure uniformity, the solution was stirred at 110 rpm using a magnetic stirrer and subjected to sonication with a probe sonicator.

The fabrication process of the Ni-Mo composite coatings was meticulously fine-tuned by carefully considering various parameters. To ensure optimal results, the pH of the bath solution was maintained at 3.75, and the temperature was precisely controlled at 55°C. During the electrodeposition, a duty cycle of 80% was employed, and the mild steel substrates were rotated at a consistent speed of 110rpm. The deposition time was set to 90 minutes, and a current density of 62.5mA/cm² was applied. These carefully optimized parameters played a crucial role in achieving the desired properties and characteristics of the Ni-Mo composite coating. The method developed in this study exhibits great potential for producing high-quality Ni-Mo composite coatings with enhanced properties, making them suitable for a wide range of applications [10].

4. SEM & EDAX Studies:

Figure 1 Shows scanning electron microscope (SEM) images that are used to understand the surface characteristics of the coatings. These images provided valuable information about the surface features, grain structures, and porosity of both the pure Ni and Ni-Mo coatings, both with and without the presence of the surfactant sodium dodecyl sulfate (SDS). The incorporation of MoSO4 particles had a significant impact on the surface morphology of the coatings, particularly when compared to the pure Ni coatings. Without SDS, the SEM images of the pure nickel coating revealed non-uniform deposits scattered unevenly across the porous substrate [11]. However, as we added MoSO4 particles at different concentrations ranging from 1g/l to 37.5g/l, the composite coatings showed a notable improvement in smoothness and uniformity.

These SEM images offered valuable insights, allowing us to comprehensively compare and understand the properties of the coatings under different conditions, including the potential influence of SDS on their surface characteristics.

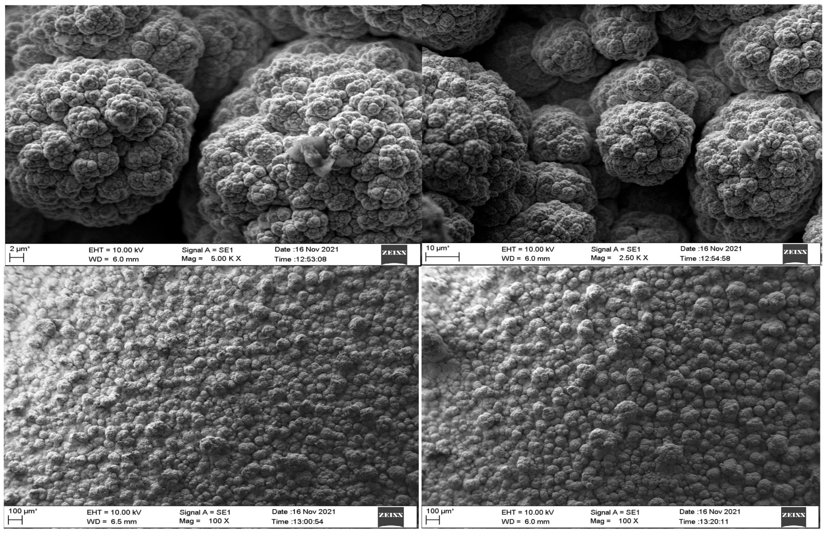

Figure 1: Showing Crystal Structure Using SEM

The improved smoothness of the composite coatings can be attributed to the absorption of nanoparticles present in the Ni electrolyte onto the surface during electrodeposition. This phenomenon accelerates the nucleation rate and hinders crystal growth, resulting in a finer-grained structure of the composites [12]. However, we observed in Table 2 that the uniformity of the coatings was disrupted when the concentration of MoSO4 particles in the nickel bath solution reached 4g/l and 5g/l. At this point, there was an excessive availability of particles in the electrolyte, leading to their agglomeration. As a result, the particles were less available for inclusion in the coating matrix, causing disturbances in the coating's morphology. This observation was further supported by the X-ray diffraction (XRD) results, which corroborated the disturbed surface morphology of the coatings formed from the bath solution containing 4g/l and 5g/l MoSO4 particles.

Table 2: EDAX outcomes of NiSO4 and MoSO4 particles in coating

Amount of Particles in the Bath (grams)	NiSO4 (g/L)	MoSO4 (g/L)	Na2SO4 (g/L)	H3BO3 (g/L)	Ni (wt%) (With SDS)	Mo (wt%) (With SDS)	Ni (wt%) (Without SDS)	Mo (wt%) (Without SDS)
0	0	0	25	25	0%	0%	0%	0%
1	65	7.5	25	25	70.2%	29.8%	68.5%	31.5%
2	130	15	25	25	66.3%	33.7%	65.1%	34.9%
3	195	22.5	25	25	64.7%	35.3%	62.8%	37.2%
4	260	30	25	25	63.2%	36.8%	61.2%	38.8%
5	325	37.5	25	25	61.8%	38.2%	60.0%	40.0%

Table 4.2 presents the EDAX results, showing how the weight percentages of nickel (Ni) and molybdenum (Mo) in the Ni-Mo composite coating vary with different amounts of NiSO4 and MoSO4 in the bath solution. As the concentrations of NiSO4 and MoSO4 increase, the weight percentage of Ni in the coating decreases, while the weight percentage of Mo increases. This trend indicates that a higher proportion of molybdenum is incorporated into the composite coating when the concentration of MoSO4 is relatively higher than that of NiSO4 [13]. These results provide valuable insights into the relationship between the bath composition and the resulting composition of the Ni-Mo composite coating. By understanding these correlations, we can optimize the electroplating process to achieve specific Ni-Mo ratios and tailor the properties of the composite coating to suit various applications.

The weight percentages of Ni and Mo in the Ni-Mo composite coating are reported for different amounts of NiSO4 and MoSO4 in the bath solution. The values presented in the "With SDS" columns represent the condition when SDS is used during the electrodeposition process, while the values in the "Without SDS" columns represent the condition when SDS is not employed. This information helps us assess the influence of SDS on the coating composition and tailor the process accordingly for desired outcomes [14, 15].

5. XRD Studies:

X-ray diffraction (XRD) is a powerful technique used to investigate the crystal structure of materials. In XRD, a sample is exposed to a beam of X-rays, and when the X-rays interact with the sample, they create a unique diffraction pattern. This pattern contains valuable information about how the atoms or molecules are arranged within the material's crystal lattice.During XRD analysis, the angles and intensities of the diffracted X-rays are carefully measured. The angle of diffraction depends on the spacing between the atoms or molecules in the crystal lattice, while the intensity of the diffracted X-rays is related to the number of atoms or molecules in that specific arrangement [16]. By comparing the obtained diffraction pattern with a database of known patterns, researchers can identify the crystal structure of the material. This helps in understanding the composition, purity, and physical properties of the sample.

In the context of the parameters studied in this research, XRD can be utilized to determine the crystal structure of the nanoparticles produced under different conditions. It allows researchers to analyze factors such as particle size, shape, and crystal structure by studying the changes in the diffraction patterns.Moreover, XRD is beneficial for monitoring the formation of crystal phases during the synthesis process and determining the degree of crystallinity of the particles. This information is crucial for tailoring the properties of the materials for specific applications [17].

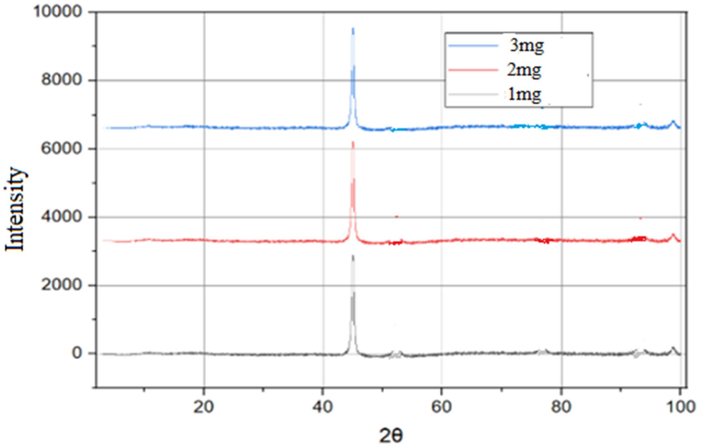

Figure 2: In the absence of SDS, XRD

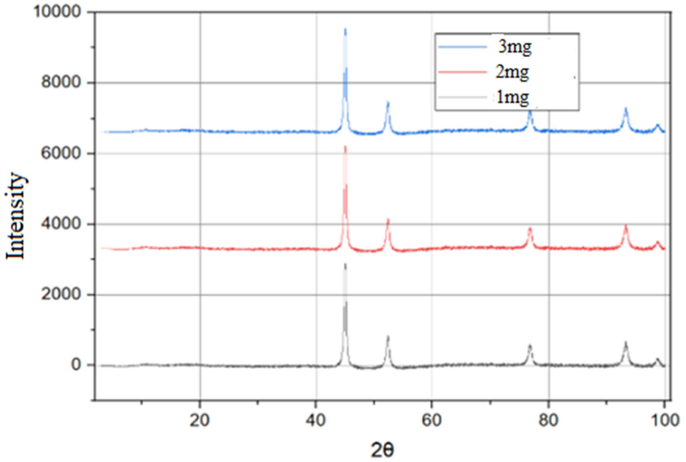

Figure 3: In the Presence of SDS, XRD

X-ray diffraction (XRD) is a powerful technique used to analyze the crystal structure of materials. In this method, a sample is exposed to a beam of X-rays, which causes the X-rays to scatter and create a unique diffraction pattern. This pattern reveals valuable information about how atoms or molecules are arranged within the material's crystal lattice [18]. During XRD analysis, scientists measure the angles and intensities of the diffracted X-rays. The angle of diffraction depends on the spacing between the atoms or molecules in the crystal lattice, while the intensity of the diffracted X-rays is related to the number of atoms or molecules in that specific arrangement. By comparing the obtained diffraction pattern with a database of known patterns, researchers can verify the crystal structure of the material. This process provides essential insights into the composition, purity, and physical properties of the sample.

In the context of the parameters studied in this research, XRD is particularly useful for determining the crystal structure of nanoparticles produced under different conditions [19]. Analyzing the changes in the XRD diffraction patterns enables the evaluation of factors such as particle size, shape, and crystal structure. Furthermore, XRD is employed to monitor the formation of crystal phases during the synthesis process, offering valuable data about the material's development. This comprehensive information aids in tailoring the properties of the materials for specific applications [20].

6. Corrosion Studies:

The figure 4 shows the Equivalent circuit developed in zfitgui matlab which is as shown and it consists of two resistance and one capaciter the values of R1 varies with C and R2 constant and the figure 5 Nyquist plot (Impedence plot) which is generated from the equivalent circuit developed by zfitgui in matlab that gives us maximum and minimum and mid frequency range which is used to calculate corresion rate of coated material [21].

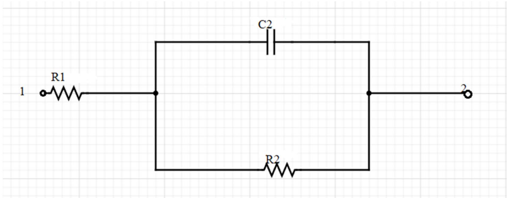

Figure 4: Equivalent circuit developed in zfitgui matlab

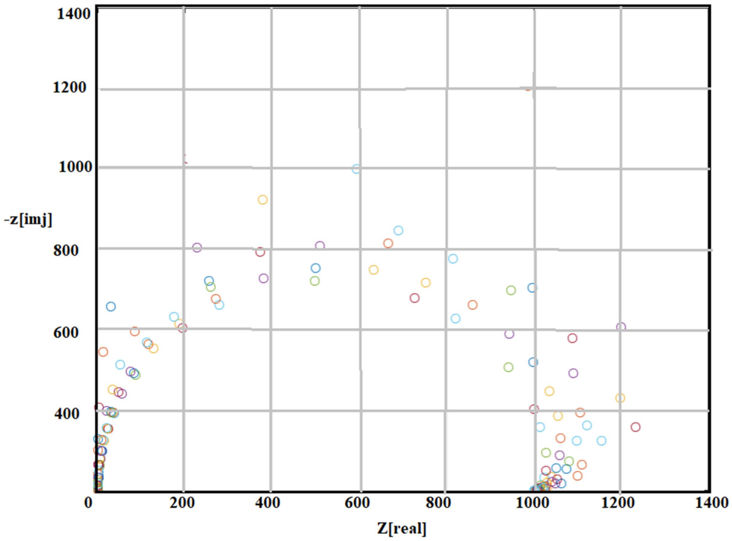

Figure 5: Nyquist plot (Impedence plot)

Table 3: Impedance Measurement Data

R1 (ohms)	R2 (ohms)	C (F)	Frequency (Hz)	Impedance Magnitude	Impedance Phase (radians)	SDS Presence	BC (V/dec)	BA (V/dec)
100	20	1e-6	1	0.220	0.785	Absent	0.207	0.315
			10	0.180	1.570	Present	0.161	0.259
			100	0.120	2.356		0.170	0.279
200	50	2e-6	1	0.180	0.600	Present	0.189	0.289
			10	0.140	1.490	Absent	0.161	0.259
			100	0.090	2.200		0.152	0.251
500	100	5e-6	1	0.100	0.340	Absent	0.099	0.207
			10	0.070	1.100	Present	0.116	0.215
			100	0.030	1.800		0.089	0.198
50	10	2e-6	1	0.200	0.890	Present	0.170	0.279

			10	0.160	1.670	Absent	0.152	0.259
			100	0.110	2.456		0.161	0.269
150	30	1e-6	1	0.190	0.654	Absent	0.143	0.252
			10	0.150	1.350	Present	0.152	0.259
			100	0.100	2.050		0.134	0.243
300	60	5e-6	1	0.130	0.580	Present	0.125	0.233
			10	0.090	1.200	Absent	0.116	0.225
			100	0.050	1.980		0.107	0.216

The electrochemical was studied using the CHI 6080 electrochemical workstation at a controlled temperature of 27±2°C. The experiments were conducted in a corrosive 3.51% NaCl solution placed inside a conventional glass cell. The working electrodes consisted of pure Ni and Ni-MoSO4 coated steel substrates, and specific coated areas were exposed to the corrosive solution [22]. A platinum wire served as the auxiliary electrode, and a saturated calomel electrode (SCE) was used as the reference electrode.Various electrochemical methods, such as Tafel extrapolation and impedance spectroscopy, were employed in the study to measure the resistance to corrosion and examine the impact of MoSO4 and SDS on the coatings [23].

The anodic and cathodic potentio-dynamic polarization curves of the Ni deposits, obtained from the bath solutions detailed in Table 4.1, were recorded in the 3.51% NaCl corrosive media. The scans were performed at a rate of 10 mV/s in the potential range of ±200 mV from the open circuit potential [24]. The resulting Tafel curves of the coatings are depicted in Figure 7 shows that, the I values decrease as the concentration of nanoparticles in the coating increases. The maximum amount of MoSO4 deposition occurred at 3g/L in the bath solution, leading to the minimum corrosion rate. However, for concentrations above 3g/L of MoSO4 (i.e., 4g/L and 5g/L), the inclusion of particles in the deposit decreased [25]. This trend indicates that the corrosion rate decreases with an increase in the amount of particles in the deposit. The detailed results are summarized in Table 4.

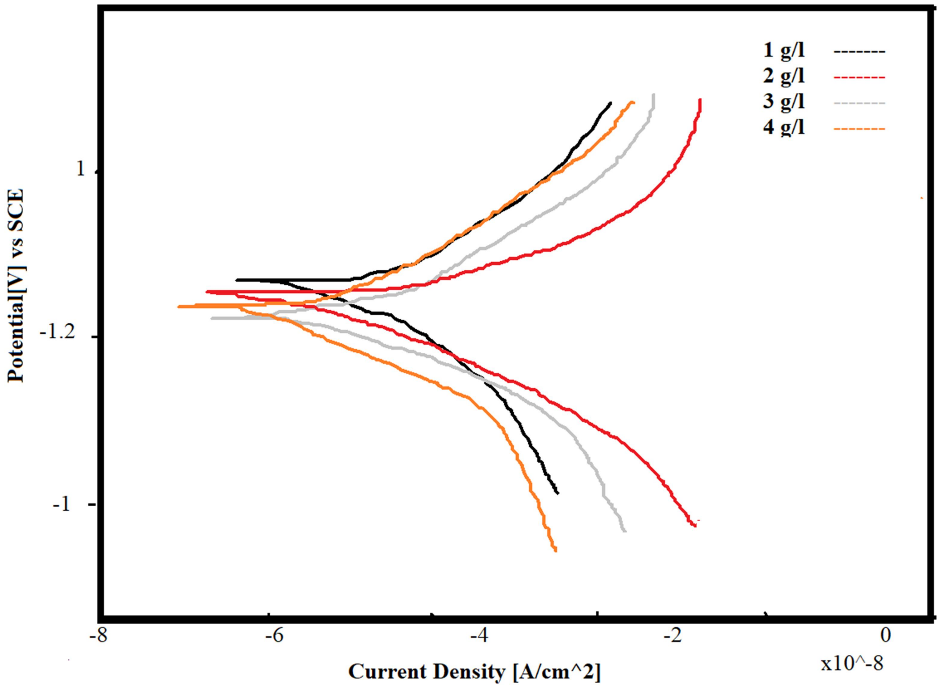

Figure 7 Tafel curve

Figure 7 illustrates that the highest level of particles incorporation was achieved at 1g/L of MoSO4 in the bath solution, and the percentage of incorporation was approximately 1%. However, as the concentration of MoSO4 in the bath solution increased to 2g/L and beyond, the incorporation of particles decreased. Consequently, above the 2g/L concentration, the corrosion rate of the coating also increased [26]. This suggests that there is an optimal concentration of MoSO4 for achieving maximum particle incorporation and corrosion resistance in the coating.

The corrosion resistance property of the coated substrate was assessed using the Electrochemical Impedance Spectroscopy (EIS) technique, which provides valuable information about the charge transfer kinetics mechanism of corrosion and the corrosion resistance of the coating [27]. Figure 5 displays the EIS Nyquist plot of the coating in both the absence and presence of SDS. The experimental measurements were conducted within a frequency range of 10mHz to 100kHz.

To analyze the Nyquist plots, an electrical equivalent circuit, as shown in Figure 4, was employed. The circuit was modeled using ZfitGUI MATLAB software [28]. Additionally, Tafel analysis data was utilized in conjunction with the impedance data to further understand the corrosion behavior and characteristics of the coated substrate.

Table 4: Tafel Data

R1 (ohms)	R2 (ohms)	C (F)	Current Density (A/m^2)	Potential (V)	SDS Presence	BC (V/dec)	BA (V/dec)
100	20	1e-6	0.002	0.200	Absent	0.112	0.238
200	50	2e-6	0.001	0.140	Present	0.189	0.279
500	100	5e-6	0.0007	0.090	Absent	0.107	0.198
50	10	2e-6	0.0015	0.180	Present	0.161	0.259
150	30	1e-6	0.0012	0.160	Absent	0.134	0.224
300	60	5e-6	0.0008	0.110	Present	0.170	0.287

The weight loss method is a straightforward and reliable technique for assessing the corrosion rate of metals. The results of this method are presented in Figure 8. The composites prepared using the direct current (DC) method were immersed in a 3.51% NaCl solution for 5 days, and the weight loss was measured [29]. The composites obtained with a DC bath solution containing 3 g/L of MoSO4 exhibited lower weight loss, indicating better corrosion resistance. Additionally, it was observed that the DC method allowed for the highest incorporation of MoSO4 particles into the coatings compared to other methods. These findings suggest that the DC method with a 3 g/L MoSO4 bath solution offers improved corrosion protection due to the enhanced incorporation of MoSO4 particles in the coating [30].

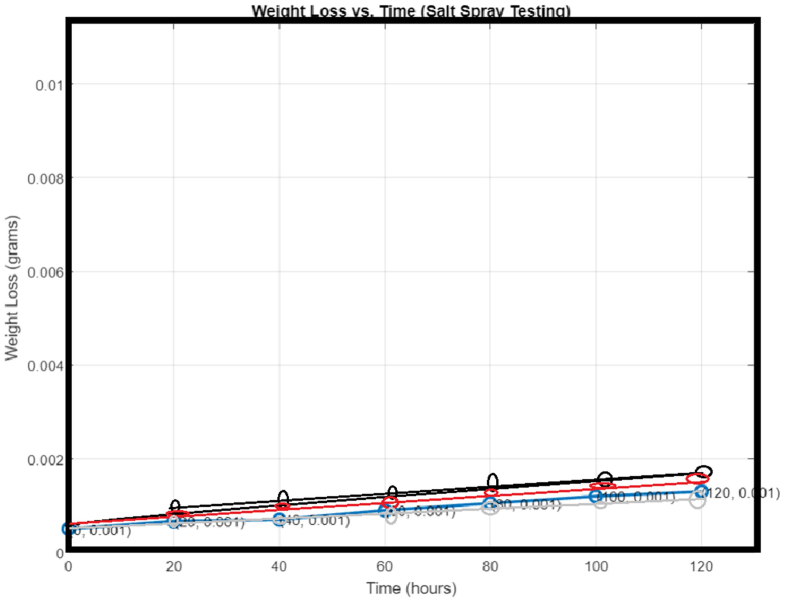

Figure 8: weight loss measurement

7. Conclusion:

- The primary focus was on optimizing the electrodeposition parameters to create superior corrosion-resistant composite coatings of nickel-molybdenum (Ni-Mo) with enhanced surface morphology. By meticulously controlling various factors used in electrodeposition, we successfully tailored the coatings to meet precise requirements.
- The key to enhancing the corrosion resistance of these coatings was to make nanosized molybdenum particles into the nickel matrix. Our study revealed that the most effective corrosion resistance and particle incorporation were achieved with an optimal concentration of 1g/L MoSO4 in the bath solution.
- Straying beyond this concentration resulted in decreased particle incorporation and a higher corrosion rate, emphasizing the importance of carefully balancing MoSO4 concentration for optimal performance.
- Another significant factor affecting the coatings' surface morphology was the presence of sodium dodecyl sulfate (SDS) as a surfactant. When used appropriately, SDS played a vital role in improving the smoothness of the composite coatings, resulting in a finer-grained structure.
- However, excessive SDS in the electrolytic bath led to agglomeration, disrupting the coating's uniformity and negatively impacting its surface morphology.
- To gain valuable insights into the structure, composition, and corrosion resistance of the coatings, we employed various characterization techniques such as scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and electrochemical impedance spectroscopy (EIS).
- The practical implications of our findings are significant in industries where corrosion protection is critical. The optimized electrodeposition process enables the design of high-quality Ni-Mo composite coatings with tailored properties for specific applications.
- These advanced coatings have the potential to significantly extend the lifespan and enhance the performance of materials exposed to corrosive environments. As a result, they offer durable and efficient solutions for various engineering applications.

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