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RESEARCH

Performance Optimization of Electroless Coating on AH36 Steel Using Surfactant, Nickel Source and Reducing Agent

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A B S T R A C T

Electroless Ni–P coatings play a crucial role in improving the corrosion resistance, wear performance, and service life of marine-grade AH36 steel. In this study, the electroless coating process was systematically optimized to maximize coating thickness by controlling key bath parameters. The primary aim was to evaluate the influence of surfactant type, nickel source concentration, and reducing agent concentration on coating thickness. A Taguchi L27 orthogonal array was employed to design experiments, and coating thickness was selected as the response using a larger-is-better criterion. The results reveal that surfactant type has the most significant effect, followed by nickel source and reducing agent concentrations. Among the tested surfactants, the zwitterionic surfactant exhibited superior performance, producing a uniform coating thickness of approximately 25 µm, representing a notable improvement compared to conventional conditions. SEM cross-sectional analysis confirmed uniform and defect-free deposition, while EDS verified the formation of a medium-phosphorus Ni–P alloy. The optimized process demonstrates strong potential for reliable protective coatings in marine and structural applications. The optimized process parameters are zwitterionic surfactant at CMC level of 0.018 g/L, nickel source of 35 g/L, reducing agent of 30 g/L. The significance of this study lies in achieving a higher and more uniform electroless Ni–P coating thickness of about 25 µm (25% increase) on AH36 steel. This improvement is mainly due to the optimized use of a zwitterionic surfactant at its critical micelle concentration, along with higher nickel and reducing agent levels.

1. INTRODUCTION

Electroless coating is only a key method for putting metal layers on surfaces without using electric current. This technique works well especially for the complex shapes and gives uniform coating that sticks properly. Moreover, as per various systems, electroless Ni–P coatings are widely known for their excellent hardness, corrosion resistance, wear resistance, and ability to work as diffusion barriers [1]. The surface of a material acts as the primary interface between the bulk material and the surrounding environment, strongly influencing corrosion, wear, and overall durability. For marine-grade steels like AH36, aggressive environmental interactions initiate surface degradation long before bulk failure occurs [2]. These properties are important for AH36 steel, which is a marine-grade structural steel. Moreover, this steel is often exposed to harsh seawater conditions. AH36 steel gets damaged by corrosion without protective treatment, which further reduces its strength and life itself. The electroless nickel coatings give a low-cost way to make things stronger and stop rust. These coatings help to make parts last longer when compared to the uncoated material [3]. However, the coating performance depends only on process parameters like surfactant type, nickel source amount, and reducing agent amount. Optimizing these parameters is critical to get coatings with maximum thickness, uniformity, and the same long-term reliability [4]. Also, surfactants are the same key components that control how the plating solution wets the surface and keeps the bath stable during electroless plating. The anionic surfactants like SDS (sodium dodecyl sulfate), cationic surfactants like CTAB (cetyltrimethylammonium bromide), and zwitterionic surfactants (C14SB) all work differently with the same substrates and reactants [5].

Further, several studies have actually used statistical and AI based methods to definitely improve coating performance. Mukhopadhyay and Sahoo [6] surely showed that electroless Ni–Cu–P coatings greatly reduced chloride corrosion in Fe-600 rebars. Moreover, these coatings provided significant protection against damage. The improved coatings show better corrosion resistance and lower current flow. This research shows that multi-objective optimization tools improve corrosion resistance by controlling the same interconnected bath parameters. Vijayanand et al. [7] study examined basic methods and provided straightforward results. Authors found that the surfactant 3-DMAPS at 0.049 g/L concentration made Ni-B coatings smoother by reducing surface roughness. The same effect happened when they used the critical micelle concentration level. The regression and back propagation neural network models got very high accuracy in predictions (R^2 > 0.98), showing that computer modelling can find the best surfactant amounts with only very good precision. Surfactants make the coating more uniform and improve the microstructure by spreading out hydrogen bubbles and making the grains finer, which gives the same better results. The reinforced nanocomposite coatings are only another important direction for research. Shozib et al. [8] provided important findings in their research work, their study used simple methods to examine the main research questions effectively. The study further confirmed that TiO_2 incorporation significantly enhanced the coating properties. As per the comparison between response surface methodology and machine learning models, Extra Trees algorithms showed better prediction accuracy with R^2 value around 94.5%. Afuwape et al. [9] studied Ni–Sn–P composite coatings with SnO reinforcement on mild steel, which is the same approach used for protective coating research. Basically, their results showed corrosion rates of 0.000343 mm/year, which is the same as saying it's much lower than bare steel. The analysis actually confirmed that protective films formed on the surface. Adding tin oxide definitely made the material last longer in salty environments. These studies show a common pattern where bath chemistry optimization, nanoparticle reinforcement, and computational modelling are key factors that further improve coating performance itself [10].

This study optimize nickel source, reducing agent, and surfactant amounts at the critical micelle concentration using only Taguchi methods. The novelty of the present work lies in the systematic optimization of electroless Ni–P coating thickness on AH36 marine steel using a Taguchi L27 design combined with surfactant chemistry at critical micelle concentration. Unlike earlier studies that primarily focus on corrosion or hardness, this study quantitatively demonstrates the dominant role of zwitterionic surfactants in achieving significantly higher and more uniform coating thickness, validated through SEM–EDS analysis, thereby offering a reliable and reproducible route for industrial-scale marine applications.

2. EXPERIMENTAL DETAILS AND PROCEDURES

As per the procedure, electroless Ni-P baths were made using pure chemicals in distilled water. The reagents used were of analytical grade regarding quality standards. We are seeing that the 150 mL working bath contained only 4.5 g nickel sulfate, 6 g sodium hypophosphite, 3.75 g trisodium citrate, and 15 g ammonium chloride, which were dissolved one by one under magnetic stirring. A surfactant was added to further improve wetting and adhesion properties. The various types of surfactants are SDS (Sodium Dodecyl Sulfate, Critical Missile Concentration (CMC) 1.2 g/L), CTAB (Cetyltrimethylammonium Bromide, CMC 1.6 g/L) and Zwitterionic surfactant (3-(N,N-dimethylmyristylammonio) propanesulfonate, C14SB) premixed with distilled water at a CMC value of 0.018 g/L and gently stirred until stable foam appeared before adding to the bath. Moreover, this process ensured proper activation at its CMC value. Basically, the AH36 marine steel of composition (0.17 wt. % C, 1.22 wt. % Mn, 0.017 wt. % Ti, 0.012 wt. % P, 0.39 wt. % Si, 0.042 wt. % Al, 0.016 wt. % Cu, tr Cr and balance Fe) was cleaned step by step using acetone, distilled water, ethanol, and 95% diluted sulfuric acid, then immediately put into the preheated electroless bath to prevent the same surface from getting oxidized again. Tin oxide is added into the electrolyte of 1g in all the bath and to maintain the pH in the electrolyte ammonia solution is added as required. The coating mechanism of Ni-P on AH36 steel are given in the following equations [11]. The variable process parameters of electroless coating are shown in the figure 1.

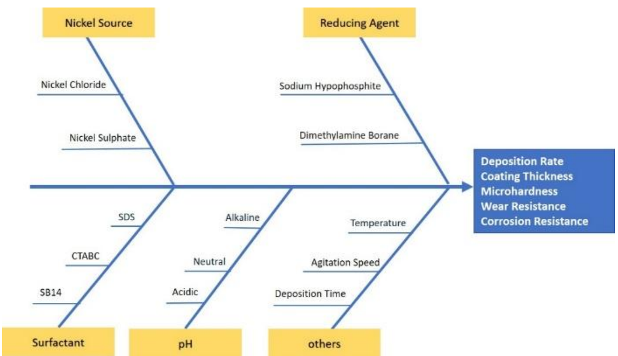


Fig. 1. Fishbone diagram for the electroless coating process.

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + 2H^+ + 2e^-,\ E^0 = 0.50\ V \quad (1)$$

$$Ni^{+2} + 2e \rightarrow Ni^0,\ E^0 = -0.25\ V \quad (2)$$

$$2H^+ + 2e \rightarrow H_2,\ E^0 = 0.000\ V \quad (3)$$

$$H_2PO_2^- + 2H^+ + e \rightarrow P + 2H_2O,\ E^0 = 0.50\ V \quad (4)$$

Taguchi L27 orthogonal array with three factors and three levels was used to study the effects of surfactant type, nickel source concentration, and reducing-agent concentration on coating response. This design further helped to understand how each factor itself affects the coating quality. The 27 trials covered all combinations with balanced representation to estimate main effects properly. This approach further minimized the experimental runs itself. Further, S/N analysis was done for larger-is-better characteristics, and response tables were made to rank which factors have the same level of influence on means and S/N ratios. As per the analyses, surfactant was the most important factor, followed by nickel source and reducing agent regarding their influence. Further, all depositions were conducted in a glass reactor with temperature control under constant magnetic stirring to further prevent particle settling and boundary layer depletion itself. As per the experimental setup, bath temperature was kept constant at the fixed value during each run. Regarding coating time, it was maintained at 1 hour for all trials to ensure proper comparison across the L27 matrix. Based on the thorough literature survey, the process parameter is carefully selected to get the more influence on the coating thickness of electroless plating. The parameters and its levels are Parameter A, Surfactant (SDS, CTAB, C14SB at its CMC value), Nickel Source (25, 30, 35 g/L), Reducing Agent (30, 40, 50 g/L). After deposition, the samples were removed and rinsed well with distilled water, then dried with warm air. The sample bring down to room temperature before measuring their weight and thickness. The selected process parameters and the L27 orthogonal array for the optimization process are given in the table 1.

Table 1. L27 Orthogonal array for the optimization of process parameters.

Trail	Surfactant at CMC in (g/L)	Nickel Source (g/L)	Reducing Agent (g/L)	Coating Thickness (Micron)	SNRA
1	SDS	25	30	15	23.52
2	SDS	25	40	16	24.08
3	SDS	25	50	17	24.60
4	SDS	30	30	18	25.10
5	SDS	30	40	19	25.57
6	SDS	30	50	20	26.02
7	SDS	35	30	22	26.84
8	SDS	35	40	20	26.02
9	SDS	35	50	21	26.44
10	CTAB	25	30	15	23.52
11	CTAB	25	40	17	24.60
12	CTAB	25	50	16	24.08
13	CTAB	30	30	18	25.10
14	CTAB	30	40	15	23.52
15	CTAB	30	50	16	24.08
16	CTAB	35	30	18	25.10
17	CTAB	35	40	19	25.57
18	CTAB	35	50	20	26.02
19	Zwitterionic	25	30	17	24.60
20	Zwitterionic	25	40	20	26.02
21	Zwitterionic	25	50	19	25.57
22	Zwitterionic	30	30	21	26.44
23	Zwitterionic	30	40	24	27.60
24	Zwitterionic	30	50	25	27.95
25	Zwitterionic	35	30	20	26.02
26	Zwitterionic	35	40	22	26.84
27	Zwitterionic	35	50	23	27.23

The coating thickness (T) can be expressed as the following equation (5):

$$T = \frac{w \times 10^4}{dA} \tag{5}$$

Where, T is the coating thickness (microns), w is the weight gain (g), d is the density of the deposit (g/cm^3) and A is the surface area of deposition (cm^2) [12]. This method was applied uniformly to all 27 trials to yield a comparable thickness response for Taguchi analysis.

3. RESULTS AND DISCUSSIONS

As per the main effects plots for means and S/N ratios are depicted in the figure 2 and 3, surfactant type has the strongest effect on coating thickness. The zwitterionic surfactant gives the highest mean and S/N ratio response regarding comparison with SDS and CTAB. Basically, when we increase nickel concentration from 25 to 35 g/L, the deposition becomes much better, and the same happens with higher reducing agent at 50 g/L but the effect is less. It is observed that the "larger-is-better" criterion confirms these trends only, with factors ranked as: Surfactant > Nickel Source > Reducing Agent. The best conditions are using zwitterionic surfactant at CMC value, with 35 g/L nickel source and 50 g/L reducing agent.

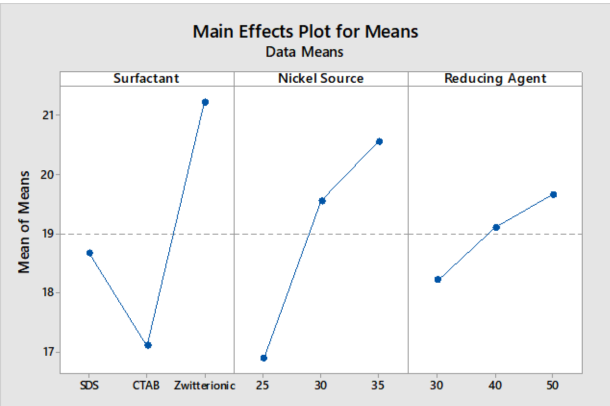


Fig. 2. Main effects plots for Means.

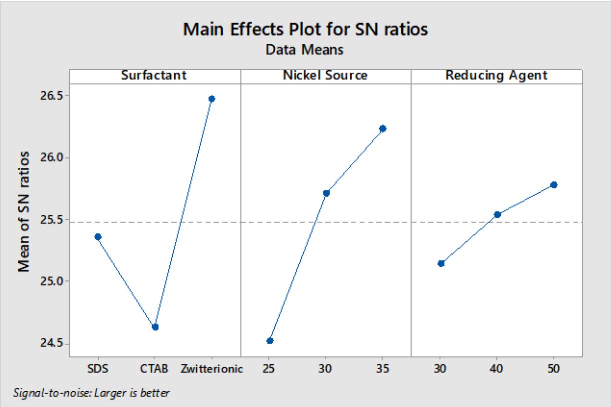


Fig. 3. Main effects plots for SN ratios.

As per the interaction plots for means and S/N ratios, the better understanding obtained regarding the combined effect of surfactant, nickel source, and reducing agent on electroless coating performance. The main effects plots show the importance of each factor is individually. Moreover, the interaction plots reveal how different parameter combinations can increase or decrease the coating responses [13]. Also, as per these findings, it is understand that the statistical significance of factors and also the chemical and physical mechanisms regarding deposition on AH36 steel [14]. The plots clearly show that the zwitterionic surfactant performs better than SDS and CTAB at all nickel and reducing agent concentrations. This surfactant itself gives superior results further confirming its effectiveness over the other two types. When nickel concentrations are higher at 30-35 g/L, the zwitterionic surfactant gives much better coating thickness and S/N response. This actually happens because of its two charged groups, which definitely help to strongly stabilize nickel ions and improve nanoparticle dispersion. SDS and CTAB make clusters that only block ion movement, but the zwitterionic surfactant keeps the surface stable and helps more nuclei form with uniform growth. Basically, the interaction plots show that zwitterionic surfactants perform much better than other surfactants when the reducing agent are included more, and this difference becomes the same pattern it keeps getting bigger. This shows that the zwitterionic surfactant further helps in faster reduction of nickel ions by making uniform distribution of hypophosphite at the substrate interface itself [15]. Also, SDS and CTAB actually show different results each time, and CTAB definitely becomes more unstable when nickel amounts change. The best coating performance comes only from using zwitterionic surfactant with high nickel amount (35 g/L) and high reducing agent amount (50 g/L) together. The three-factor interaction results clearly show this combination gives optimal results. Under these conditions, the coating actually shows maximum thickness and definitely maintains the most stable S/N ratio. This clearly highlights both efficiency and reproducibility. The interaction plots actually show that surfactant has the main effect, but higher nickel and reducing agent concentrations definitely make it work better. The surfactant and nickel source interaction has the strongest effect, followed by surfactant and reducing agent, while nickel source and reducing agent show the same lowest impact. The surfactant controls how ions are available and spread out, and this becomes the same critical factor when reagent concentrations increase. The zwitterionic surfactant works better because it reduces surface tension and helps the nanoparticles spread evenly on the steel surface. The same surfactant also makes the active chemicals stay stable in the solution. The dual charge functionality prevents micelle overgrowth and aggregation. Further, this ensures nickel ions and hypophosphite remain available near the substrate surface [16]. Further, as per the process, dense nucleation and faster growth occur, regarding which smoother coatings with less defects are formed. SDS and CTAB have limitations due to their single charge type, which further causes uneven adsorption and poor reproducibility. The micelle formation itself creates depletion zones that affect the results. Basically, the interaction analysis shows that getting the best electroless Ni–P coating on AH36 steel needs the same carefully adjusted conditions. Basically, among the three factors we studied, surfactant type shows the strongest main effects and the same pattern for interactive effects. As per the study results, zwitterionic surfactant with high nickel and hypophosphite levels gives the best coating speed and thickness. Regarding process reliability, this combination also shows the most stable performance. We are seeing that surfactant chemistry plays an important role in electroless deposition, and this only provides a way to design strong coatings for marine use [17]. The interaction plots for SN ratio and Means are given in the figure 4 and 5.

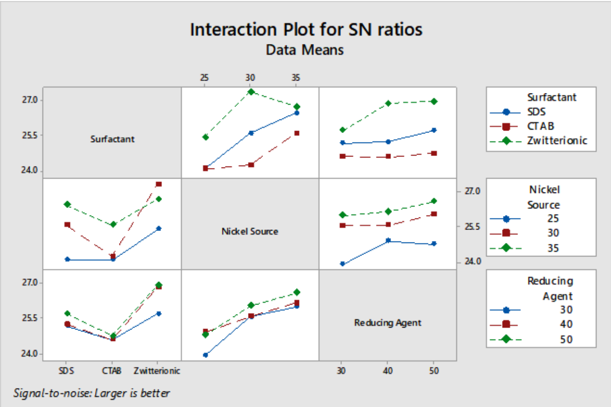


Fig. 4. Interaction Plot for SN ratios.

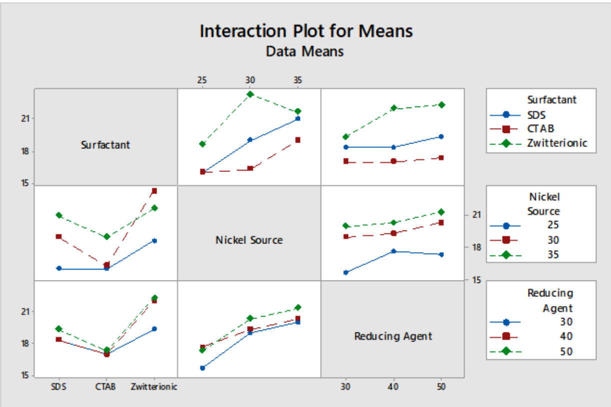


Fig. 5. Interaction Plot for Means.

The Taguchi L27 design shows how surfactant type, nickel concentration, and reducing agent affect the coating thickness on AH36 steel. The same response tables help identify the most important factors and best conditions for electroless Ni-P coating. Table 2 surely shows that surfactant is the most important factor with delta value of 4.11, followed by nickel source at 3.67 and reducing agent at 1.44. Moreover, this gives the ranking order as Surfactant > Nickel Source > Reducing Agent. As per the results, Level 3 surfactant showed the highest mean thickness of 21.22 µm. Regarding performance comparison, the zwitterionic surfactant performed better than SDS (18.67 µm) and CTAB (17.11 µm). The zwitterionic surfactant helps more material deposit by keeping ionic species stable in the electrolyte, reducing surface tension, and making nucleation happen the same way everywhere. In the same way, the nickel source actually worked best at Level 3 (35 g/L) with 20.56 µm thickness, showing that more nickel ions help coating formation. The reducing agent achieved maximum 19.67 µm thickness at Level 3 (50 g/L), actually proving that higher sodium hypophosphite concentration helps the process but definitely has smaller effect than surfactant and nickel source. Table 3 strongly supports these results, where the surfactant shows the highest delta value of 1.85, which is more than nickel source at 1.72 and reducing agent at 0.64 only. This confirms that surfactant not only increases the mean thickness but also makes the process more stable and repeatable. The best S/N ratio was found at Level 3 of surfactant (26.48 dB), then Level 3 of nickel source (26.24 dB) and Level 3 of reducing agent (25.78 dB). Regarding the coating process, this combination gives maximum thickness and best reliability. It is observed from the both tables that all three factors help in the deposition process, but the surfactant shows the strongest effect only. It gives the best mean performance and reduces variance most, making it the most important factor in electroless coating. Moreover, as per the study, zwitterionic surfactant works better with high nickel and reducing agent levels regarding uniform mixing of materials and stable coating conditions. This combination ensures proper ion movement and gives superior results. Therefore, the best parameter settings from this analysis are surely zwitterionic surfactant (Level 3), nickel source at 35 g/L (Level 3), and reducing agent at 50 g/L (Level 3). Moreover, these settings together give maximum coating thickness, better adhesion, and improved reproducibility. The response tables surely confirm that surfactant type is the main factor controlling coating quality [18]. Moreover, nickel and reducing agent concentrations work together to improve deposition, showing the order of importance as Surfactant > Nickel Source > Reducing Agent.

Table 2. Response table for signal to noise ratios.

Level	Surfactant	Nickel Source	Reducing Agent
1	25.36	24.51	25.14
2	24.62	25.71	25.54
3	26.48	26.24	25.78
Delta	1.85	1.72	0.64
Rank	1	2	3

Table 3. Response table for means.

Level	Surfactant	Nickel Source	Reducing Agent
1	18.67	16.89	18.22
2	17.11	19.56	19.11
3	21.22	20.56	19.67
Delta	4.11	3.67	1.44
Rank	1	2	3

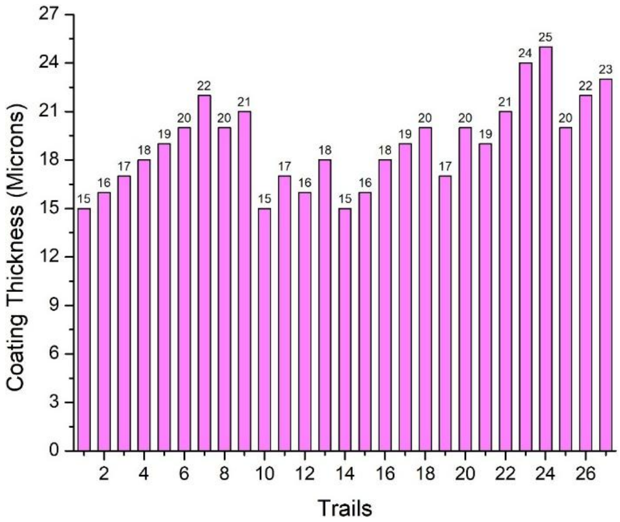


Fig. 6. Coating thickness of coated substrates.

As per the bar chart showing in the figure 6 coating thickness across 27 Taguchi trials, surfactant type, nickel source concentration, and reducing agent concentration clearly affect deposition performance. Regarding the results, these three factors have direct influence on the coating thickness obtained. The thickness values actually range from 15 to 25 µm, with early trials showing lower values and later trials showing higher ones. Trial 24 shows the highest thickness at 25 µm. Further, this trend surely supports the findings in the response tables, where surfactant emerged as the most dominant factor. Nickel source and reducing agent followed in importance. Tests with the zwitterionic surfactant gave better coating thickness (20–25 µm) compared to SDS and CTAB, which stayed below 20 µm. The zwitterionic surfactant itself showed further improvement in coating quality. When nickel source concentration increased from 25 g/L to 35 g/L, deposition efficiency itself improved. Higher reducing agent levels at 50 g/L further enhanced coating uniformity and thickness, though this parameter showed less influence than the other two factors. Trial 24 with zwitterionic surfactant, 35 g/L nickel, and 50 g/L reducing agent gave the maximum coating thickness, confirming the same optimization results shown in the document. The chart and results surely show that surfactant type is the most important factor for good coating quality [19]. Moreover, nickel and reducing agent work together to improve deposition, confirming the order: Surfactant > Nickel Source > Reducing Agent [20].

As per the Taguchi design results, we found the best process settings and then did a confirmation test regarding the accuracy of these optimized conditions. This step was actually essential to ensure that the predicted coating performance from statistical analysis could definitely be reproduced in practice. To check how well the coating is working, we are seeing that the coated sample was only cut into sections and then examined under microscope for detailed study. The thickness of the deposited layer was actually measured using a SEM. This method definitely gave precise cross-sectional measurements. The SEM images gave accurate coating thickness measurements and also showed uniform deposition as per the analysis. Regarding the coating quality, the images revealed consistent thickness throughout the surface. As per the confirmation analysis, the experimental results matched with Taguchi optimization findings regarding the selected process parameters. This validation stage showed that the chosen parameters were effective for achieving good coating quality.

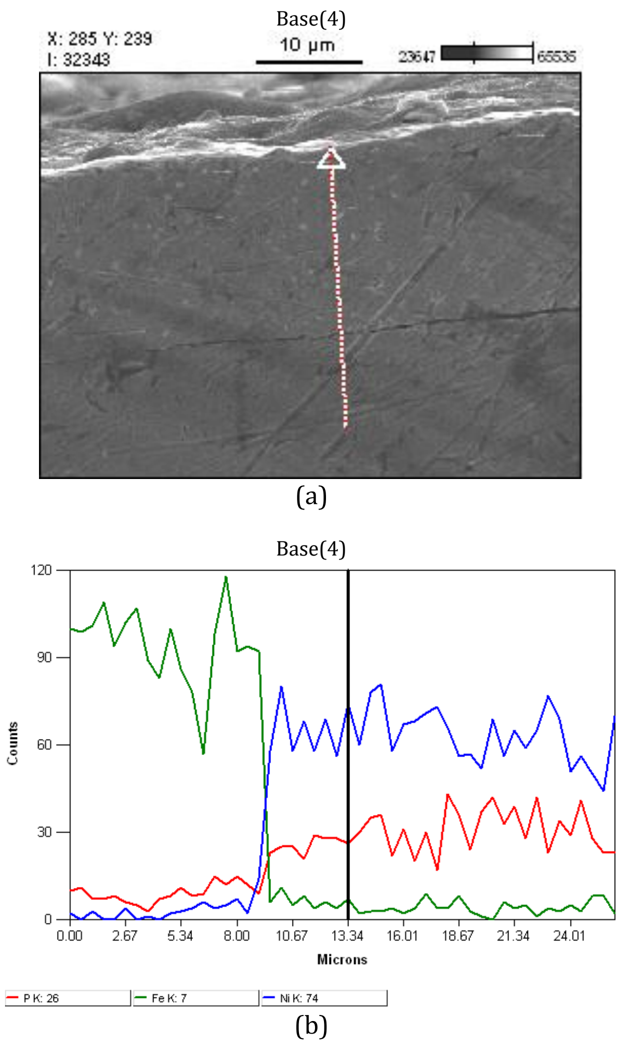
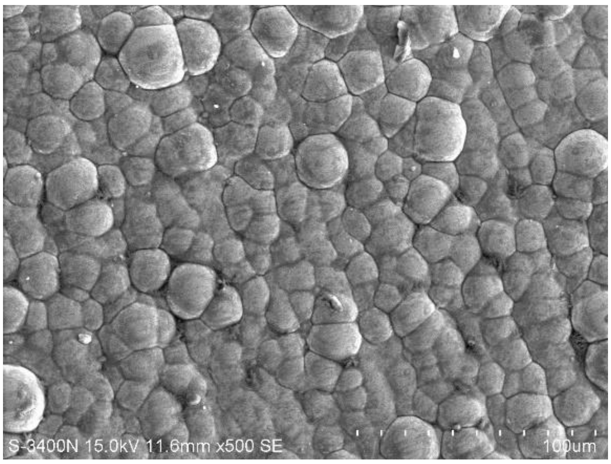
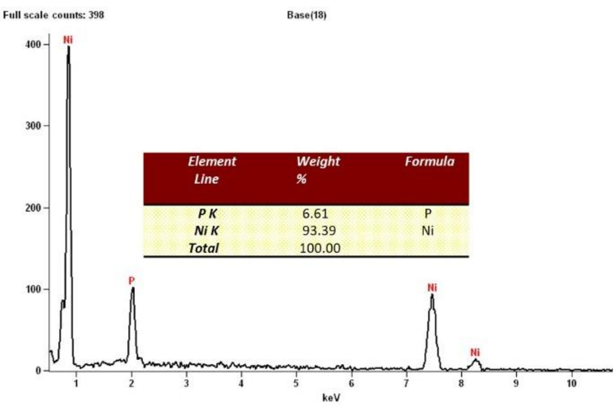


Fig. 7. Coating thickness of optimized condition A3B3C3.

The SEM cross-sectional image in figure 7 shows the coating layer deposited on the substrate. Moreover, the layer formation is clearly visible in the analysis. Basically, the coating thickness is the same as 25 µm, which we can see from the transition zone in the energy dispersive spectroscopy (EDS) line scan. Basically, the element mapping shows Nickel and Phosphorus are present in the coating area, and Nickel intensity increases significantly after crossing from the base material to the coating region. Further, further, the Iron (Fe) signal from the substrate drops sharply at this interface itself, confirming the boundary between the steel substrate and the electroless Ni–P coating. Further, the Ni and P signals are surely uniform across the coating thickness, showing consistent deposition. Moreover, this indicates homogeneous coating formation without major defects or gaps. The SEM-EDS analysis confirms the coating made with best settings has good sticking and uniform thickness only at micrometer level [21]. Optimized surfactant chemistry enhances ion dispersion and suppresses agglomeration, leading to finer, more uniform, and defect-free coating microstructures.



(a)



Element Line	Weight %	Formula
P K	6.61	P
Ni K	93.39	Ni
Total	100.00	

(b)

Fig. 8. Surface morphology elemental identification of (A3B3C3) optimized condition.

The coatings surface morphology was assessed using a SEM, specifically the Hitachi Model S-3400N, operated at an accelerating voltage of 15 kV. Elemental analysis of the deposits was conducted with an attached EDS system. As per the SEM analysis, the figure 8, surface shows a thick, bump-like structure that is typical for electroless Ni-P coatings. This indicates that the coating grew evenly regarding the optimized process conditions. As per the surface analysis, the nodular morphology is beneficial regarding enhanced surface area. This contributes to better wear and corrosion resistance. The EDS spectrum further confirms the coating's elemental composition, with Nickel (Ni) itself being the main element at 93.39 wt.%. We are seeing Phosphorus (P) content at only 6.61 wt.%. This composition matches well with the expected Ni–P alloy phase itself obtained under optimized conditions. Further, these conditions were derived through Taguchi analysis. We are seeing that phosphorus in this range only indicates formation of medium-phosphorus electroless nickel coating. This coating gives good balance between hardness, corrosion resistance, and structural strength. The predicted Taguchi results actually matched well with the experimental EDS data. This definitely proves that the chosen process parameters work effectively to make uniform and strong coatings with consistent composition.

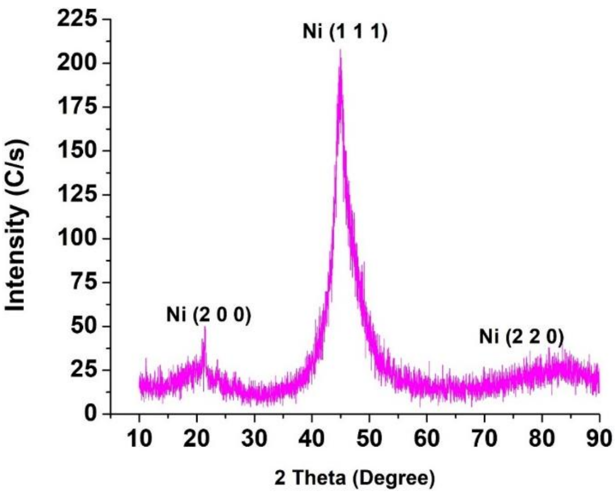


Fig. 9. X-Ray diffraction pattern of optimized condition sample.

Phases and structures were identified using XRD with a Rigaku Ultima IV diffractometer featuring a copper anode. The XRD patterns were documented over a 2θ range from 10° to 90°, with a step size of 0.02° per second. The X-

Ray Diffraction (XRD) pattern of the electroless Ni-P coating in figure 9 shows a broad and strong peak at around 2θ ≈ 44–46°, which matches the (111) plane of FCC nickel. Moreover, this peak indicates the basic crystal structure of the deposited nickel layer. This dominant reflection surely confirms that nickel is the main crystalline phase in the coating. The JCPDS number is 00471049 and the Crystallite size of the developed coating is 22.15 nm. Moreover, this finding indicates the successful formation of nickel crystal structure. The peak actually becomes wider, which definitely shows that nano-crystalline domains are present in an amorphous matrix. This is actually a typical characteristic of medium-phosphorus electroless nickel coatings. Further, when phosphorus is added to the nickel structure, it surely breaks the regular arrangement and makes the crystal particles smaller. Moreover, this process increases the amorphous nature of the material. Basically, along with the main (111) peak, you can see weak reflections for (200) and (220) planes at around 51–52° and 76°, but these are less intense due to partial amorphization. The same pattern shows that the secondary peaks are much weaker than the primary one. We are seeing that the XRD results show the coating has mainly nano-crystalline Ni-P structure with strong direction along the (111) plane only. This structure is good for better hardness, corrosion resistance, and surface life [17].

4. CONCLUSIONS

This work actually showed how to optimize electroless Ni-P coatings on AH36 steel using Taguchi method. The results definitely proved that this approach works well for coating optimization. As per the analysis of means and signal-to-noise ratios, surfactant type is the main factor that controls coating thickness. Regarding performance, zwitterionic surfactants work best because they stabilize nickel ions, improve wetting, and help uniform nucleation. Nickel source concentration ranked second, where 35 g/L gave maximum ion availability for deposition itself. Further, the reducing agent at 50 g/L helped faster reduction and better coating uniformity. The best combination actually used zwitterionic surfactant, 35 g/L nickel source, and 50 g/L reducing agent. This definitely gave maximum thickness of 25 µm, confirmed by SEM cross-section study. EDS actually confirmed that a medium-phosphorus Ni–P alloy was formed. This alloy definitely provides good balance between hardness, corrosion resistance, and durability. Basically, the study shows that optimizing process parameters is vital for getting reliable electroless coatings on marine structural steels, which ensures the same enhanced service life in harsh environments. This study is highly suitable for marine structures, shipbuilding components, offshore platforms, and coastal infrastructure, where enhanced corrosion resistance and durability are critical.

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