



## CHARACTERIZATION OF PHOSPHATE CONVERSION COATING ON MILD STEEL FOR ENHANCED CORROSION INHIBITION MECHANISMS

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### ABSTRACT

This study focuses on the comprehensive characterization of phosphate conversion coatings applied to mild steel, emphasizing their role as inhibitive corrosion mechanisms. Through a multifaceted approach, including micro hardness testing, Spectromaxx Metal Analyzer analysis, and corrosion potentiostat testing, the research delves into the mechanical, chemical, and corrosion-resistant properties of the coated mild steel. Micro hardness testing reveals insights into the coating's impact on the material's resistance to indentation and deformation, providing a foundational understanding of its mechanical behavior. The Spectromaxx Metal Analyzer analysis elucidates variations in the chemical composition, specifically highlighting changes in alloying elements such as copper, which are crucial for corrosion resistance. Corrosion potentiostat testing further substantiates the inhibitive corrosion mechanism, showcasing reduced corrosion rates in various environments for the coated mild steel compared to the uncoated counterpart. The findings contribute to a nuanced comprehension of how phosphate conversion coatings enhance the material's durability and resilience in corrosive settings.

**Keywords:** Coating, Corrosion mechanism, Hardness values, Mild steel inhibitive, Phosphate conversion, Potentiodynamic polarization, Spectromaxx metal analyzer.

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### INTRODUCTION

Steel will always corrode during manufacturing and everyday use. According to estimates, every country experiences yearly economic losses from corrosion exceeding 3% of its GDP[1]. Corrosion is a natural phenomenon that can degrade and destroy metallic structures like mild steel. It is a major concern in several industries, including manufacturing, construction, and the automobile industry. Therefore, it is essential to carry out steel coating protection. Due to their exceptional mechanical qualities and workability, alloy steels are still employed today as great building materials in the appliance and aerospace industries for welded and riveted constructions [2, 3]. Nevertheless, there is still a slight chance of corrosion with these steel elements [4]. Steels' ability to withstand corrosion is frequently increased by the use of phosphate chemical conversion (PCC) coatings [5].

In the production of automobiles, chemical conversion coating is a type of well-known industrial surface pre-treatment that is frequently used to enhance the adhesion of organic coatings on metal surfaces and to temporarily protect metal substrates from corrosion, lubricate them, and provide insulation [1, 6, 7]. Numerous types of chemical conversion coatings exist, including but not limited to chromate, phosphate, molybdate, and vanadate coatings [8, 9, 10]. Among them, chromate coating exhibits a

superior performance [11, 12]. Regrettably, chromium salt poisoning poses a serious threat to human health and the environment [13, 14, 15]. Phosphate coating is one of the coatings that may be applied to the surface of both ferrous and non-ferrous metals that is suitable to the environment. Since it is inexpensive, easy to apply, lubricates well, and adheres to steel, it has been widely used in the surface pretreatment of cars and steel constructions [16, 17, 18]. Because they work so well at strengthening mild steel's resistance to corrosion, phosphate conversion coatings are widely employed.

The protective qualities of the phosphate conversion coating can be more comprehensively understood through the characterization procedure of mild steel. Understanding the ways via which the coating prevents corrosion will be beneficial. As an illustration, the coating might function as a physical barrier, keeping corrosive substances from reaching the metal surface. Furthermore, there's a chance that the coating will chemically react with the metal substrate to create a passivation layer, which will impede the corrosion process. Understanding the inhibitive corrosion mechanism of phosphate conversion coatings can facilitate the optimization of their formulation and application processes. This information can help create corrosion protection plans for mild steel and other metals that are more effective, which will ultimately increase the robustness and lifespan of items made of metal [19].

This study therefore tends to analyze the phosphate conversion coating that is used on mild steel and comprehend how it prevents corrosion. This entails dissecting the coating's morphology, structure, content, and characteristics. To evaluate the coating's properties, technologies including the Spectromaxx metal analyzer, corrosion potentiostat testing, and micro-hardness testing would be used [20].

## MATERIALS AND METHODS

### Sample collection

The Defense Industries Corporation of Nigeria, Kakuri, Kaduna, Heat and Surface Treatment Department, provided a mild steel sample that was 50 x 26 mm (length x breadth) and 3 mm thick that was used for the study. The absence of any surface impurities or pre-existing coatings that might interfere with the characterization procedure was guaranteed for the sample.

### Reagent and equipment

The following reagents were employed in this study: demineralized water, sulfuric acid, steel wool for enhanced corrosion resistance, phosphoric acid ( $H_3PO_4$ ), and pH-adjusting agents: For the best reaction conditions, these were employed to regulate the phosphate solution's pH, it is crucial to remember that the particular chemicals utilized and their quantities can change based on the method, phosphate type, and intended outcomes [21].

Spectromaxx metal analyzer LMF14 model, mild steel, phosphate bath, corrosion prevention stat, microhardness tester HVS-1000Z model, petri dish, measuring cylinder, tongs, beakers, conical flask, funnel, weighing balance, filter paper, and Vernier caliper are among the equipment utilized in this study.

### Material preparation

Before phosphate treatment, the substrate (mild steel) was thoroughly cleaned with a shot blasting machine. A mechanical procedure called short blasting was used to remove contaminants and rust from hard metals prior to surface treatments like chrome plating or phosphate. The metal surface was cleaned and strengthened by spraying steel fragments onto it using a short blasting equipment. Centrifugal force helps the steel shots collide at a high speed, which eliminates impurities and corrosion. After the waste material was collected by a dust collection system, the metal surface was cleansed and ready for further treatments, which improve adhesion and surface roughness [22].

### Preparation of standards

Preparation of the Phosphate Bath: The phosphate bath is a solution that includes the chemicals and additives needed to form the phosphate coating. Phosphoric acid ( $H_3PO_4$ ) was combined with demineralized water (1:9) to create the phosphate solution, which had a pH of 3 to 5 to be slightly acidic.

After that, steel wool was added to help create a phosphate coating [23]. After giving the combination a good stir, the pH was tested and adjusted using pH meter, and the mixture was then left to stabilize in the bath. The ready-to-use bath was applied to metal surfaces to improve their resistance to corrosion. Strict safety measures were taken.

### Experimental procedure (Development of phosphate coating)

The development of phosphate coatings involves various steps to ensure the desired properties and characteristics. These include:

#### a) Sample Preparation

The mild steel samples were cut into Four (4) pairs in a Rectangular shape of 50 x 26 mm and with a thickness of 3mm. It was then cleaned thoroughly using a shot blasting Machine to remove any surface contaminants, oils, or residues. Samples were then rinsed with deionized water and allowed to dry.

#### b) Phosphate Conversion Coating Formation

A phosphate conversion coating solution was prepared. Phosphoric acid ( $H_3PO_4$ ) was mix with demineralized water. This solution often contains phosphate acid, steel wool and demineralized water. The exact composition used was 1:9 (phosphoric acid ( $H_3PO_4$ ): demineralized water)

Steps in Phosphate: In this practical process, metal samples underwent several steps to prepare and enhance their surface for improved adhesion and corrosion resistance.

Step 1, the metal samples were hung and rinsed in Bath-1.

Step 2, the samples were immersed in an etching solution (Bath-2) for 5 minutes and subsequently rinsed with water. This step achieved surface activation, a critical process that enhances the metal's surface properties. The etching solution contained sulfuric acid.

Step 3, the samples were immersed in a Phosphating solution (Bath-3) and heated for 30 minutes at 95°C. This resulted in the formation of a Phosphating layer on the metal surfaces. The resulting color of this layer was gray.

Step 4, the prepared samples were dried in an oven. This drying process firmly adhered the surface layer to the metal, ensuring durability and improved corrosion resistance.

This sequence of steps represents a systematic approach to surface preparation, activation, and Phosphating of metal samples, ultimately resulting in enhanced surface properties for various applications.

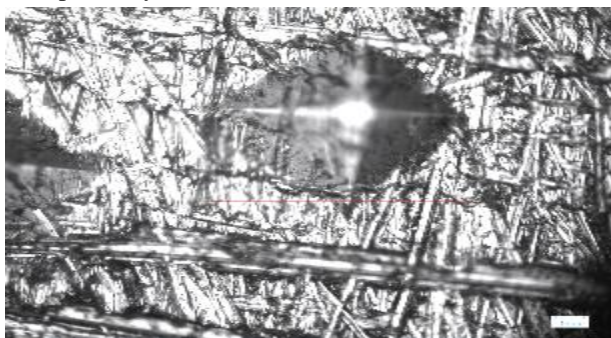
#### c) Surface characterization

Micro hardness tester model HVS-1000Z, Spectromaxx model LMF14, metal analyzer and corrosion potentiostat were used to measure the hardness of small regions, identify the elemental composition and investigate the corrosion behavior of metals respectively. The metal were suspended in

acidic and basic media for 72 hours prior to potentiostat testing.

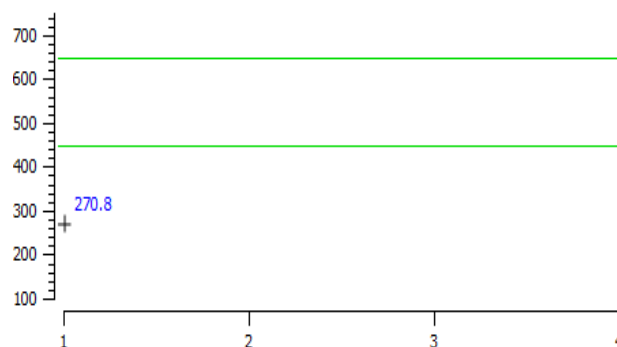
## RESULTS AND DISCUSSION

The results of potentiostat, Spectromaxx metal analyzer model LMF14 and micro hardness model HVS-1000Z for both coated and uncoated mild steel samples are presented in Figure 1-4 and Table 1-3 respectively.



**Figure 1:** Coated Mild Steel HV View: 270.8 HV

The HVS-1000Z type, which has a machine capacity or total case hardness of 550 hardness units, was used for microhardness testing. The study compared the outcomes of coated and uncoated specimens using mild steel samples. Figures 1-4, and Table 1-2 display the micro hardness values, which are expressed in hardness units (HV).

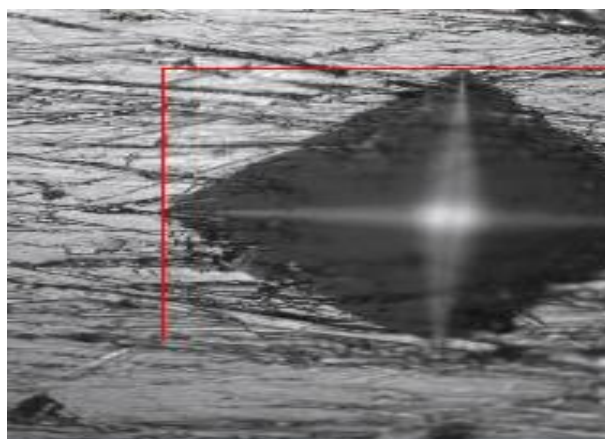


**Figure 2:** Hardness distribution graph for coated mild steel: 270.8 HV

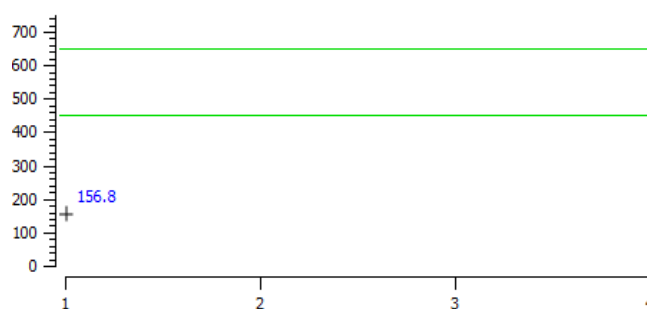
**Table 1:** Statistics for the coated

### STATISTICS

Maximum	270.8(63.2;44.4;25.8)	Minimum	270.8(63.2;44.4;25.8)
Average	270.8(63.2;44.4;25.8)	Std. Dev.	0.0
Cp	0.00	Cpk	0.00



**Figure 3:** Uncoated Mild Steel HV View: 156.8 HV



**Figure 4:** Hardness distribution graph for uncoated mild steel: 156.8 HV

**Table 2:** Statistics for the uncoated

STATISTICS			
Maximum	156.8(50.7; 24.4 ;)	Minimum	156.8(50.7; 24.4 ;)
Average	156.8(50.7; 24.4 ;)	Std. Dev.	0.0
Cp	0.00	Cpk	0.00

The findings from Figures 1 and 2 as well as Figures 3 and 4 above show a notable difference in microhardness between mild steel samples that were coated and those that were not. The coating used on the mild steel surfaces appears to have a significant effect on the material's hardness, based on the reported micro hardness values. The micro hardness of the coated mild steel is 270.8 HV, which indicates that it is harder than the uncoated version, which is 156.8 HV. This enhancement could be ascribed to the coating's protective qualities, which offer more defense against deformation and indentation. he description of the HVS-1000Z model's machine

capacity of 550 hardness units for both coated and uncoated mild steel indicates that the testing parameters were well-suited for the material under consideration, reflecting a meticulous approach to material characterization in line with the standards of precision found in scientific research.

#### **Spectromaxx metal analyzer**

Chemical composition analysis of coated and uncoated mild steel samples was performed using the Spectromaxx Metal Analyzer. Table 3 provides the percentage composition of various elements.

**Table 3:** Chemical composition for coated and uncoated mild steel

S/N	Elements	Coated Mild Steel (%)	Uncoated Mild Steel (%)
1	Carbon (C)	>1.68	>1.68
2	Silica (Si)	0.057	0.015
3	Manganese (Mn)	0.78	0.66
4	Phosphorous (P)	0.051	0.033
5	Silicon (S)	0.084	0.075
6	Chromium (Cr)	0.039	0.039
7	Nickel (Ni)	0.180	0.194
8	Molybdenum (Mo)	<0.0020	<0.0020
9	Aluminum (Al)	0.062	0.033
10	Copper (Cu)	0.031	0.57
11	Cobalt (Co)	0.101	0.109
12	Titanium (Ti)	0.0010	0.0016
13	Niobium (Nb)	0.024	0.043
14	Vanadium (V)	<0.0010	<0.0010
15	Tungsten (W)	0.015	0.043
16	Palladium (Pd)	0.020	0.012
17	Magnesium (Mg)	0.029	0.030
18	Boron (B)	0.015	0.012
19	Tin (Sn)	0.057	0.062
20	Zinc (Zn)	>0.036	>0.036
21	Arsenic (As)	0.070	0.063
22	Bismuth (Bi)	<0.0020	<0.0020
23	Calcium (Ca)	>0.016	>0.016
24	Cerium (Ce)	0.013	0.022

25	Zirconium (Zr)	<0.0015	<0.0015
26	Lanthanum (La)	0.050	0.060
27	Iron (Fe)	<93.3	<95.4

Table 3 depict the elemental composition of both coated and uncoated mild steel samples. The results indicated a significant difference in the chemical makeup of mild steel samples that were coated and those that were not.

#### Elemental composition variation

- i. Carbon (C): Both coated and uncoated mild steel exhibit carbon levels exceeding 1.68%. This is expected for mild steel, as carbon content significantly influences its mechanical properties, such as hardness and strength.
- ii. Silica (Si): The coated mild steel has considerably higher silica content (0.057%) compared to the uncoated sample (0.015%). This elevation is likely a deliberate addition in the coating process to enhance corrosion resistance.
- iii. Manganese (Mn): The coated sample shows higher manganese content (0.78%) than the uncoated (0.66%). Manganese is commonly used as a deoxidizer and can contribute to increased hardenability.
- iv. Copper (Cu): A significant reduction in copper content is observed in the coated mild steel (0.031%) compared to the uncoated sample (0.57%). This reduction indicates a potential improvement in corrosion resistance, as copper is known to promote corrosion [21].
- v. Alloying Elements (Ni, Cr, Mo, etc.): While some alloying elements like nickel (Ni) and chromium (Cr) show minor variations, their overall levels are relatively consistent between coated and uncoated mild steel.

#### Practical implications

Corrosion Resistance: The coated sample's observed

decrease in copper concentration is consistent with predictions of increased corrosion resistance. This is important for situations where environmental factor resistance is essential. Mechanical Properties: The differences in alloying components point to possible improvements in the mild steel coating's mechanical qualities. Hardness, strength, and other mechanical properties may be impacted by these modifications.

#### Correlation with micro hardness results

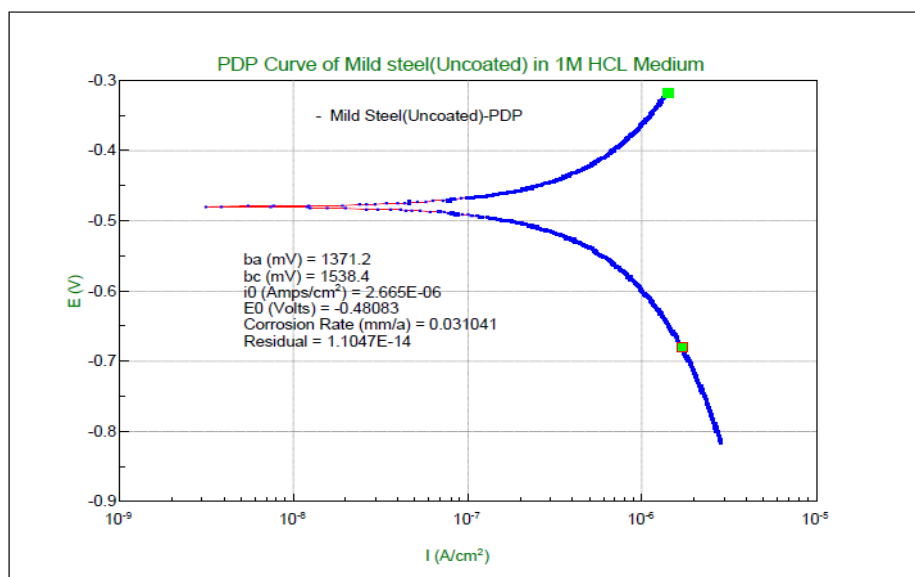
- i. Carbon and Micro Hardness: The higher carbon content in the coated sample is likely contributing to its higher micro hardness. This correlation supports the idea that the coating is influencing the material's resistance to indentation and deformation.
- ii. Copper and Corrosion Resistance: The significant reduction in copper content aligns with an expected improvement in corrosion resistance. This is a promising indication for applications where corrosion is a critical concern.

#### Corrosion potentiostat tester

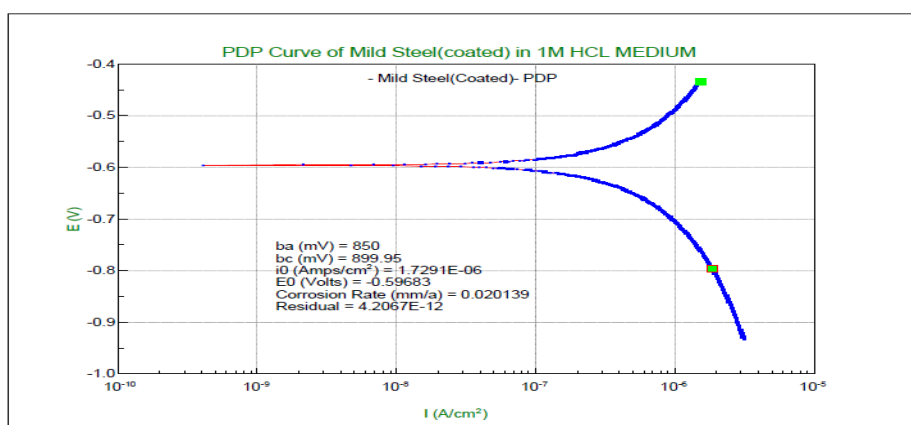
The corrosion potentiostat testing was carried out, and the experimental configuration was as follows:

- i. Sample used, mild steel
- ii. Electrode configuration: Working electrode = sample (mild steel)  
Reference electrode=Ag/AgCl (aqueous)  
Contact electrode = Platinum (Pt.)
- iii. Specifications of solutions
  - a. In Acid medium [HCl], 6.62ml of HCl was dissolved in 100ml of distilled water.
  - b. In Basic medium [NaOH], 40g of NaOH was dissolved in 100ml of distilled water.

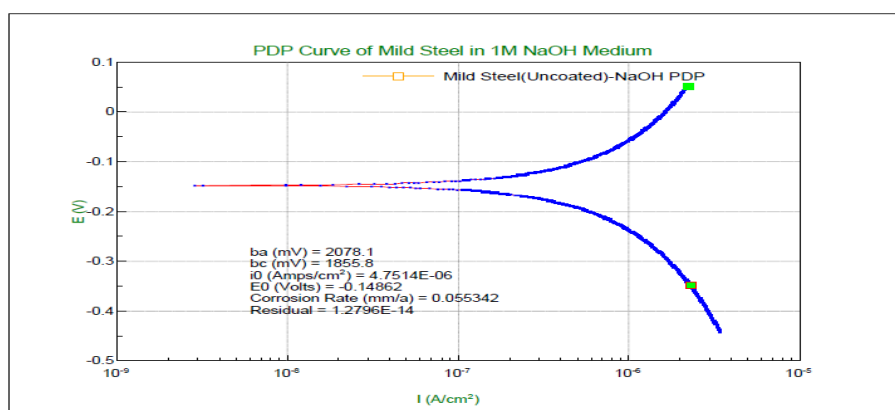
Figure 5-8 displays the findings for both coated and uncoated mild steel in the Acidic and Basic Medium.



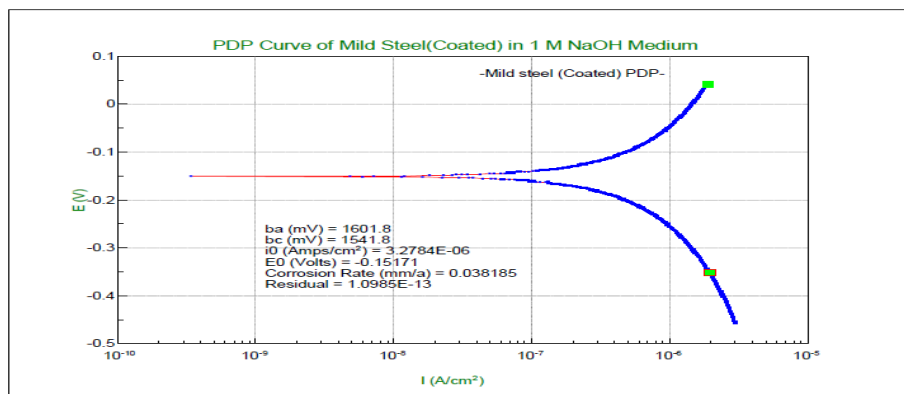
**Figure 5:** Uncoated PDP Curve of mild steel in HCl medium (corrosion rate= $0.031041 \text{ g} \cdot \text{m}^{-2} \cdot \text{d}^{-1}$ )



**Figure 6:** Coated PDP Curve of mild steel in HCl medium (corrosion rate= $0.020139 \text{ g} \cdot \text{m}^{-2} \cdot \text{d}^{-1}$ )



**Figure 7:** Uncoated PDP Curve of mild steel in NaOH medium (corrosion rate= $0.055342 \text{ g} \cdot \text{m}^{-2} \cdot \text{d}^{-1}$ )



**Figure 8:** Coated PDP Curve of mild steel in NaOH medium (corrosion rate= $0.038185 \text{ g} \cdot \text{m}^{-2} \cdot \text{d}^{-1}$ )

Mild steel corrosion rates were evaluated in various conditions by contrasting coated and uncoated samples, as seen in Figures 5, 6, 7, and 8, respectively. The following outcomes were attained:

#### Corrosion Rate in 1 M HCl Medium

Figure 6 shows that the coated mild steel corrodes at a lesser rate ( $0.020139 \text{ gm}^{-2} \cdot \text{d}^{-1}$ ) than its uncoated counterpart ( $0.031041 \text{ gm}^{-2} \cdot \text{d}^{-1}$ ) in a solution of 1 M HCl figure 5. This decrease in corrosion rate indicates that the coating is improving the material's ability to withstand corrosion in acidic conditions. In particular, the decrease in copper concentration, which is known to affect corrosion resistance, is consistent with the observed changes in chemical composition.

#### Corrosion Rate in 1 M NaOH Medium

In 1 M NaOH medium, the coated mild steel exhibits a lower corrosion rate ( $0.038185 \text{ gm}^{-2} \cdot \text{d}^{-1}$ ) at Figure 8 than the uncoated mild steel ( $0.055342 \text{ gm}^{-2} \cdot \text{d}^{-1}$ ) at Figure 7, which is comparable to the results in an acidic medium. This result demonstrates how well the coating works to increase the material's ability to withstand corrosion in alkaline settings. This improved corrosion resistance could be attributed to changes in chemical composition, such as a decrease in copper content.

#### Practical implications

The application of mild steel in corrosive situations is practically affected by the corrosion rate data that were obtained. Because of its proven ability to reduce corrosion, the coating is a useful safeguard for equipment or structures that are subjected to both acidic and alkaline environments. The observed differences in corrosion rates highlight the interaction between environmental factors and material qualities, as they correspond with changes in chemical composition [21].

i. Corrosion Control in Acidic Environments: The coated mild steel's reduced corrosion rate in HCl has real-world implications for applications where materials are subjected to acidic environments. Similar protective coatings could be used by industries

that deal with hydrochloric acid to extend component longevity.

ii. Alkaline Environment Material Selection: The coated mild steel's reduced corrosion rate in NaOH highlights its appropriateness for use in alkaline environments. This knowledge may be essential for industries working with alkaline substances when choosing materials to reduce corrosion-related problems.

#### Effectiveness of the coating

The efficacy of the protective coating in preventing corrosion is demonstrated by the decrease in corrosion rates for both acidic and alkaline conditions. Because it helps to preserve mild steel in a variety of chemical conditions, this efficacy is very remarkable.

## CONCLUSION

Mild steel's mechanical, chemical, and corrosion resistance qualities have been thoroughly examined using corrosion potentiostat testing, Spectromaxx Metal Analyzer analysis, and micro hardness testing. When comparing the coated mild steel to its untreated equivalent, the micro hardness measurements showed a notable improvement in hardness. This improvement implies that the coating helps the material resist deformation and indentation, suggesting that it may be used in applications requiring a higher level of hardness. Spectromaxx Metal Analyzer chemical composition examination revealed differences in important components between mild steel that was coated and mild steel that was not. Significantly, the coated sample's decreased copper content suggests possible increases in corrosion resistance. Subtle modifications in the mechanical and corrosion-resistant properties were observed in other alloying elements. Evaluations of corrosion rates in various settings provided additional evidence of the coating's effectiveness. In comparison to the uncoated sample, the coated mild steel showed reduced rates of corrosion in both acidic and alkaline conditions. These findings support the coating's ability to fend off corrosive attacks and increase the material's resilience

in a range of environmental circumstances. This indicate that phosphate conversion coating could greatly enhance the mechanical and chemical composition of mild steel.

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