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Investigation of Corrosion Resistance and Surface Properties of Carbon Steel Treated with Phosphate Coating in Aqueous Media

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Keywords:
Carbon steel corrosion; Immersion behavior; Phosphate coating; Pitting corrosion; Surface roughness.

Highlights:

- *The threat of corrosion is particularly pronounced in the case of carbon steel exposed to acidic or salt-laden environments.*
- *This study employs a zinc phosphate coating enriched with nickel carbonate to bolster the corrosion resistance of carbon steel.*
- *Adding nickel carbonate substantially enhanced the steel corrosion resistance, substantiated by weight loss and electrochemical tests.*
- *The present findings potentially extended the operational life of carbon steel in industrial settings.*

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Abstract: Carbon steel is widely used in industrial applications due to its mechanical properties and high availability. Zinc phosphate conversion coating is commonly used in industry for surface pretreatment. It enhances corrosion resistance and improves adhesion between metal surfaces and subsequent treatments. The practical aspect of the study involved treating carbon steel samples with zinc phosphate at 90 °C using nitric acid, zinc oxide, phosphoric acid, and sodium carbonate. Then, nickel carbonate was added to the same metal samples for phosphating. The results revealed that adding nickel carbonate effectively improved the corrosion resistance of the samples, evaluated using weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy techniques. Furthermore, surface properties, particularly surface roughness, were assessed using scanning electron microscopy (SEM). The samples were tested before and after exposure to three corrosive media, including hydrochloric acid with pH 3, seawater with 3.5% sodium chloride, and sodium hydroxide with pH 11. The SEM showed the effect of the corrosive solutions on the zinc phosphate coating layer, in the HCl solution dissolving it and exposing carbon steel. Also, there was localized corrosion with pits and erosion. 3.5% NaCl exposure made the surface rough and deposited pitting corrosion products. NaOH immersion for 15 mins formed a uniform, dense, crystalline corrosion-resistant layer. The coating transformed to light gray, indicating improved properties. Also, incorporating nickel carbonate in treating carbon steel can substantially enhance its corrosion resistance.

دراسة مقاومة التآكل والخواص السطحية للفولاذ الكربوني المطلي بطبقة حماية من الفوسفات في الاوساط المائية

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الخلاصة

تناقش الدراسة حماية الصلب الكربوني من التآكل في ثلاث اوساط مائية مختلفة وذلك من خلال معالجة عينات الصلب الكربوني بمحلولي فسفتة باستخدام حمض النيتريك، اكسيد الزنك، حمض الفسفوريك وكربونات الصوديوم للنوع الاول. وتمت إضافة كربونات النيكل إلى المحاليل وفسفتة نفس عينات المعدن للحصول على طبقة حماية من الفوسفات مختلفة عن طبقة الحماية الأولى. تم قياس معدل التآكل لأنواع العينات الثلاث بطريقتين، الاولى هي الطريقة الوزنية (weight loss) بغمر نوعي العينات في الاوساط الثلاث ولفترة زمنية استغرقت ثلاثة اشهر تم خلالها حساب الوزن المفقود , والثانية هي الطريقة الكهروكيميائية حساب التآكل (Tafel) كما تم دراسة الخصائص السطحية باستخدام المجهر الماسح الالكتروني (SEM) وذلك للعينات المفسفتة مع كربونات النيكل والتي اظهرت اعلى مقاومة تآكل, حيث أظهرت النتائج أن إضافة كربونات النيكل قد ساهم بشكل فعال في تحسين مقاومة التآكل للعينات مقارنة مع العينات الاخرى، الاوساط المائية المستخدمة في الدراسة هي حمض الهيدروكلوريك بتركيز pH 3 وماء البحر بنسبة ٣,٥٪ كلوريد الصوديوم وهيدروكسيد الصوديوم بتركيز pH 11. كما بينت نتائج الفحص المجهري تأثير تلك الاوساط على الطبقة السطحية للعينات وتاثيراتها على المعدن من خلال وجود تنقرات سطحية ناجمة عن هجوم اوساط التآكل على سطح المعدن وتبين تأثر لون وشكل طبقة الحماية، كما وتثبت نتائج الدراسة إلى أن إضافة كربونات النيكل الى محاليل الفسفتة لمعالجة الصلب الكربوني تعزز بشكل كبير مقاومته التآكل. بالإضافة إلى ذلك، تقدم هذه الدراسة معلومات قيمة حول الخواص السطحية وسلوك عينات الصلب في اوساط التآكل المذكورة.

الكلمات الدالة: التآكل التنقري، الخشونة السطحية، تآكل الصلب الكربوني، فسفتة الحديد.

1.INTRODUCTION

Corrosion presents a major hurdle for various industries and can result in expenses on par with natural disasters like floods, tornadoes, and earthquakes [1]. Carbon steel is a popular choice due to its cost-effective strength. Nevertheless, when exposed to acidic environments or salt without adequate protection, steel pipes, and applications become susceptible to corrosion. The financial consequences and maintenance expenditures underscore the importance of implementing improved control methods. Preventing corrosion has become an integral part of the design process, with the primary goal to achieve cost-effective durability and ensure industrial integrity. Surface treatments provide effective approaches for mitigating corrosion. Considering the corrosion effects on the lifespan of equipment is crucial in industrial designs. Failure to do so can result in catastrophic losses of billions of dollars [2]. Corrosion occurs due to chemical or electrochemical reactions between a metal and its environment, which can adversely affect its properties [3]. Two main reasons behind corrosion are of utmost importance: economics and safety [4]. Direct losses include the cost of replacing corroded structures, while indirect losses involve efficiency loss, product contamination, environmental pollution, and work shutdown. Hence, economic factors are significant motivators for corrosion research [5]. Corrosion inhibitors are crucial in reducing the corrosion rate and protecting metals and alloys from deterioration. They create a protective barrier that isolates the metal from its environment [6-8]. Corrosion inhibitors are considered the first line of defense against corrosion [9]. These inhibitors can be classified based on their chemical nature, such as organic or inorganic coatings [10]. Phosphate conversion coating is a chemical process that involves depositing a layer of insoluble metal phosphates onto the surface of a metal substrate. Typically, this process includes immersing the metal part in a solution containing phosphate ions that react with the metal surface to form a metal phosphate insoluble layer [11]. This layer provides corrosion protection resistance and enhances the adhesion of subsequently applied coatings, such as paint or powder coatings [12]. Phosphate conversion coatings are commonly used in automotive, aerospace, and military industries to enhance the durability and performance of metal parts [13]. Phosphating can be easily applied to various metals like steel [14], aluminum [15], galvanized steel [16], and magnesium [17]. The phosphate coatings' corrosion resistance depends on various factors, including the coating thickness, surface roughness, and the composition of the surrounding environment. In aqueous media, corrosion can occur due to dissolved oxygen, chloride ions, and other corrosive agents. However, phosphate coatings can act as a barrier to prevent diffusing these agents into the metal substrate, thereby reducing the corrosion rate [18, 19]. Surface roughness is to coatings' adhesion and performance [20]. In the case of phosphate coatings, surface roughness can influence the coating thickness uniformity, surface texture, and topography [21]. Moreover, the surface roughness of the substrate can impact the adhesion of the coating, as a rough surface provides more mechanical interlocking sites for the coating to adhere to [22]. Al-Swaidani [23] applied zinc phosphate coating to reinforcing steel. It was found that the porosity decreased, and the necessary driving force for achieving a thicker coating increased. Jiang and Cheng [24] developed zinc phosphate coatings on building bars to study their stability and corrosion behavior in an alkaline solution. Zinc phosphate (ZP) was brush-coated onto building steel in a one-step process. Positive outcomes included plate-like morphology, typical hopeite and minor phosphophyllite phases, and enhanced corrosion resistance. Abdalla et al. [25] evaluated the coating effectiveness, and an immersion test was conducted in a 3.5% NaCl solution. The results revealed that a higher concentration of zinc in the phosphating bath improved corrosion protection. Ali et al. [26] used different zinc phosphating solutions with Na_3PO_4, $NaNO_3$, and $NiCO_3$ as accelerators. Enhanced corrosion resistance was achieved with a maximum of 80 g/L $NaNO_3$. Meanwhile, superior corrosion protection was provided by 1 g/L $NiCO_3$. Mixed accelerators produced finer grains and reduced the corrosion current to 1.83 mA at 80 g/L $NaNO_3$ and 5 g/L $NiCO_3$. Ayoola et al. [27] investigated the potential of calcium-modified zinc phosphate coating as a corrosion inhibitor for steel. Numerous studies have examined the corrosion resistance properties of zinc phosphate coatings on various substrates. The findings indicated that these coatings offered effective protection against corrosion. Factors, such as adding inhibitory ions and nanoparticles, have been explored to enhance the corrosion resistance of zinc phosphate coatings. By comprehending the factors that influence the corrosion resistance and surface roughness of phosphate coatings, researchers can develop new coating formulations and optimization techniques to enhance the coated materials' performance in corrosive environments. In this study, two zinc phosphate coatings were prepared with and without nickel carbonate to investigate their stability, corrosion resistance, and surface properties in different aqueous media containing hydrochloric acid with a pH of 3, seawater with 3.5% sodium chloride, and sodium hydroxide with a pH of 11.

2.EXPERIMENTAL PROGRAM

2.1.Material

The samples used in the present study were Carbon steel plates with dimensions of (2mm×15mm×15mm) according to (ASTM-G 71-30). The studied Carbon steel samples' chemical composition is shown in Table 1.

2.2.Sampling Configuration

- The Samples were a braded in sequence on (180, 220, 400, 600, and 1000) grades. Then, the samples were drilled into a (1.5mm) diameter hole for the structural solution and the base of an electrochemical cell.
- The specimens underwent a series of cleaning steps. First, they were washed with tap water, then distilled water, then subjected to alkaline etching using a Sodium Hydroxide solution with a concentration of 5wt %, then etched with 10% concertation HCl solution. The specimens were rinsed again with distilled water.
- Phosphate coating samples: The zinc phosphate layer to cover the surface of the carbon steel specimen was performed by phosphate acid, zinc oxide, nitric oxide, and sodium carbonates, as shown in Table 2. The sample's surface was prepared by immersing them for 10min in an alkaline degreasing solution (solution at a temperature of 80 °C). Then, they were immersed for 5min in an acid-picking solution (at room temperature). Thus, the samples were immersed in the phosphate bath solution for 20min at (90) ± 2°C. At the end of the phosphate process, the samples were dried in distilled water at (90-95) °C and then dried by hot air [28].

Table 2 The Zinc Phosphate Bath Carbon Steel Samples.

Composition	ST1	ST2
H3PO4	400 ml	400 ml
ZnO	160 g	160 g
HNO3	142 ml	142 ml
NaPO4	3 g	3 g
NiCO3	-----	3 g

- Electrolyte preparation: The aqueous media were prepared as HCl solution at pH=3, 3.5% NaCl, and NaOH at pH=11.

2.3.Experimental Sets

2.3.1.Weight Loss Analysis

This is a simple method for measuring corrosion rates. It involves immersing a carbon sample steel in HCl, NaCl, and NaOH solutions for various time intervals (24, 72, 168, 720, 1440, and 2160) hours. The corrosion rate, C.R, was calculated using the following formula [29]:

$$CR = \frac{(K\times W)}{(A\times T\times D)} \tag{1}$$

Table 1 Chemical Composition of Carbon Steel Sample.

Element	Fe	Mo	Cu	Mn	Cr	Si	C	Other
Weight %	98.5041	0.2481	0.0331	0.5752	0.0239	0.2464	0.3534	

2.3.2.Electrochemical Polarization

The corrosion current density (i corr) is a kinetic parameter representing the corrosion rate under specified equilibrium conditions. The corrosion rate (CR mm/y) in a given environment is directly proportional to its corrosion current density (icorr) as Eq. (2) [30]:

$$CR = \frac{mm}{y} = 3.27 \times Icorr.e/p \qquad (2)$$

and the efficiency is calculated by formula [31]:

$$PE\% = (\frac{CR_o - CR}{CR_o}) \times 100 \qquad (3)$$

3.RESULTS AND DISCUSSION

3.1.First Set of Results

3.1.1.Results of Weight Loss in HCl Solution

Figure 1 illustrates the corrosion rate for coated and uncoated samples in HCl solution. The high initial corrosion rates observed in the active corrosion region for samples (ST) and (ST1) can be attributed to the intense interaction between the metal surface and the HCl medium. In contrast, the low corrosion rate observed for (ST2) suggested effective isolation of the metal surface by the protective layer. Then, the corrosion rate decreased, promoting surface passivation. The corrosion rate was (0.2199) mm/y for uncoated ST, (0.168) mm/y for ST1, and the corrosion rate was (0.1076) mm/y for ST2. The efficiency of protection layer PE % was 23.6% for ST1 and 51.06% for ST2.

3.1.2.Weight Loss in NaCl Solution

Figure 2 illustrates the corrosion rate in the NaCl medium. The initial high corrosion rate values observed in the active corrosion region can be attributed to the strong interaction between the metal surface and the NaCl medium. The corrosion rate decreased with establishing a protective layer, promoting a surface passivate (ST) had a corrosion rate of 0.2136 mm/y, while (ST1) corrosion rate of 0.1167 mm/y with a 45.36% efficiency of the protective layer. (ST2) had a corrosion rate of 0.0268 mm/y with an 87.45% efficiency of the zinc phosphate layer [25].

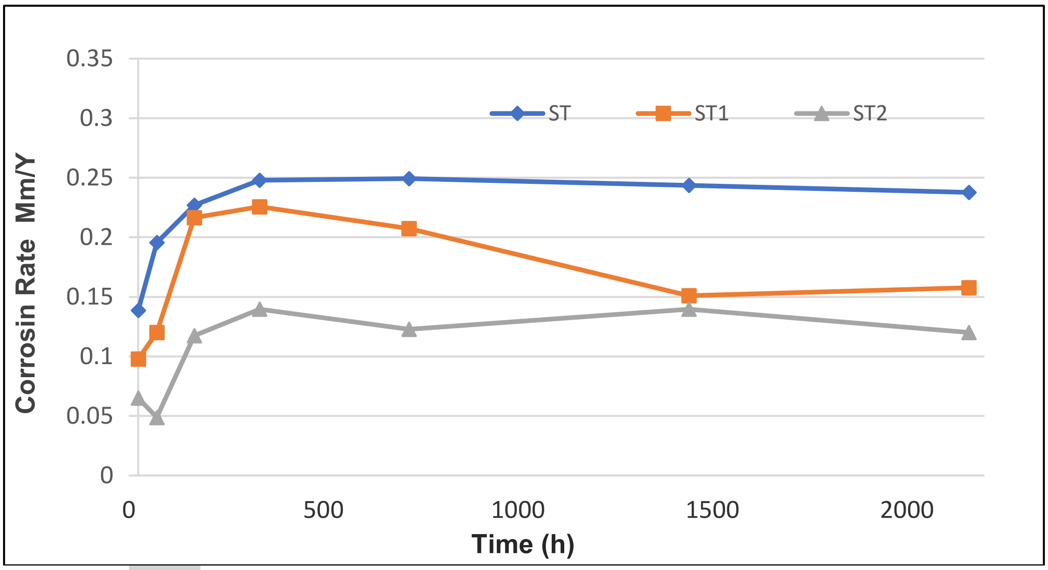

Fig. 1 Corrosion Rate for Coated and Uncoated ST in HCl Solution.

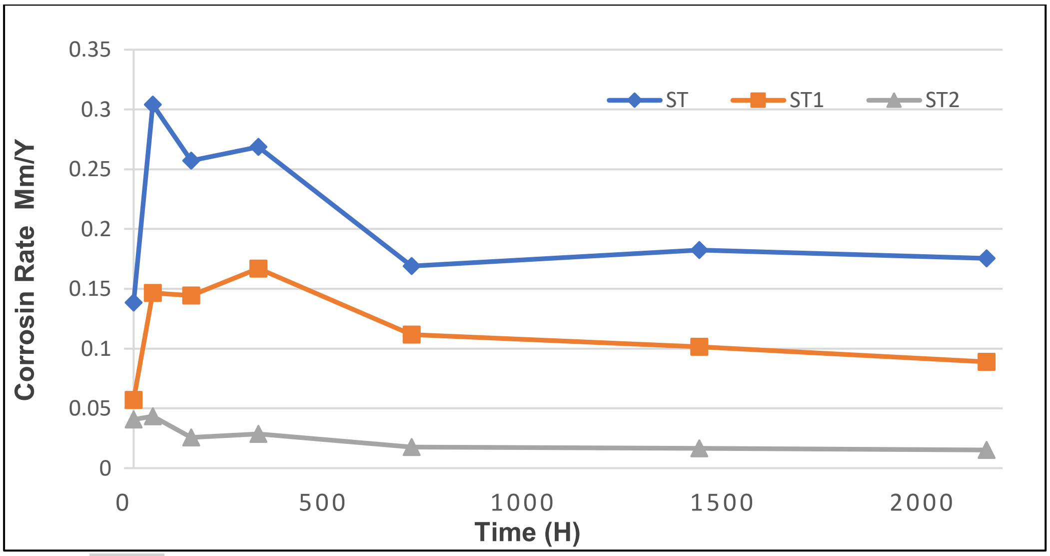

Fig. 2 Corrosion Rate for Coated and Uncoated Samples in NaCl Solution.

3.1.3.Weight Loss in NaOH Solution

Figure 3 illustrates the corrosion rate in a NaOH medium. The initial rapid corrosion observed in the active corrosion region can be ascribed to the interaction between the metal surface and the NaOH medium. As a protective layer formed, the corrosion rate diminished, facilitating the surface passivity's establishment. The average corrosion rate was (0.0297) mm/y for uncoated ST. The ST1 and ST2 corrosion rate were (0.0068) mm/y and (0.0026) mm/y, respectively. The efficiency of protection layer PE was (77.10%) and (91.24 %), respectively.

3.2. Second Set of Results

3.2.1.Polarization in HCl Solution

Table 3 indicates the corrosion rate (Icorr) is lower for the phosphate-coated sample ST1 than the uncoated ST, and it is even lower for ST2, indicating the effectiveness of the phosphate process in safeguarding the metal surface against corrosion. These measurements were performed to investigate the zinc phosphate coating ST1 and ST2 effect on the corrosion resistance in HCl solution compared to uncoating sample ST. The corrosion rates were (0.2515), (0.2049), ST2 (0.1157) mm/y for ST, ST1, and ST2, respectively. The corrosion resistance of the carbon steel surface improved by depositing a phosphate layer for ST1 and ST2 Phosphate coatings, which provide an insoluble crystalline layer that reacts chemically with metal to protect against corrosion.

3.2.2.Polarization in NaCl Solution

Table 4 shows that zinc phosphate coating reduced the steel's corrosion current density (Icorr), particularly for ST1 and ST2 coatings. These results indicated that the zinc phosphate coating, especially ST2, greatly enhanced corrosion resistance, with 48% protection efficiency for ST1 and 84.1% for ST2 in a 3.5% NaCl solution. The zinc phosphate coating effectively isolated the metal surface in a corrosive NaCl environment.

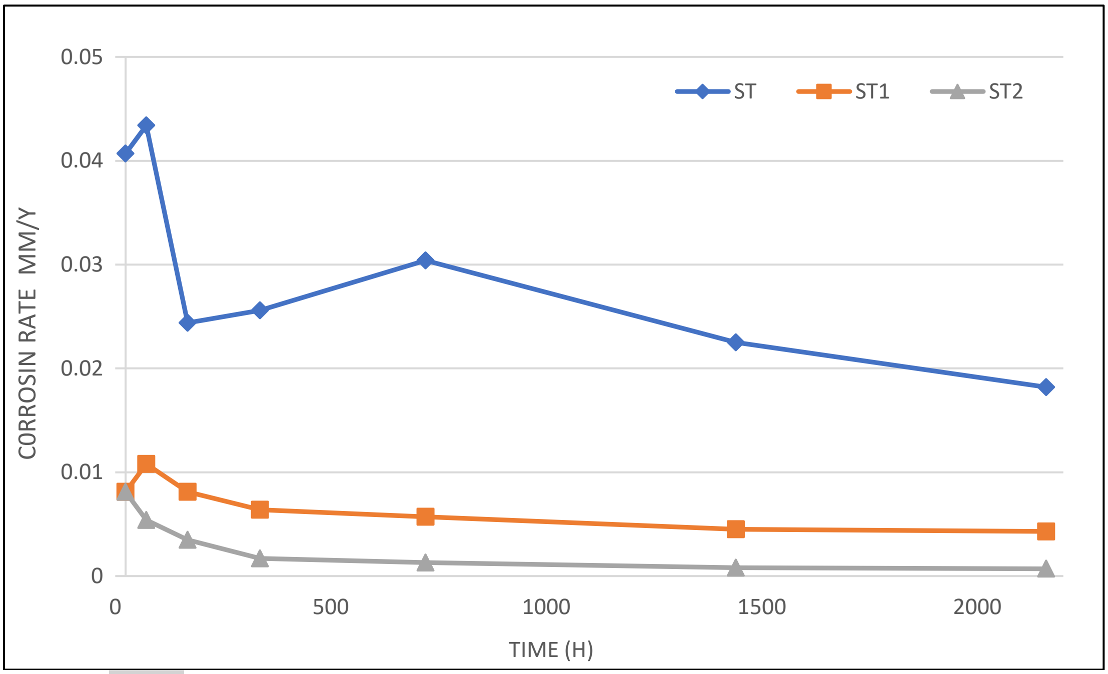

Fig. 3 Corrosion Rate for Coated and Uncoated Samples in NaOH Solution.

Table 3 Corrosion Parameters for ST, ST1, and ST2 in HCl Solution.

Samples	T (k)	$-E_{corr}$ mV	I_{corr} mA/cm²	B_c mV/dec	B_a mV/dec	C.R mm/y	R_P(Ω/cm²)	PE%
ST	300	431.5	0.02166	174	204	0.2515	836.2	
ST1	300	522.4	0.017640	272	210	0.2049	1295	18.52%
ST2	300	465.4	0.009958	150	134	0.1157	1415	53.99%

Table 4 Parameters for ST, ST1, and ST2 in 3.5% NaCl Solution.

Sample	T (k)	$-E_{corr}$ mV	I_{COOR} mA/cm²	B_c mV/dec	B_a mV/dec	C.R mm/y	R_PΩ/cm²	PE%
ST	300	414.4	0.01858	220	200	0.2158	1087	
ST1	300	464.5	0.009658	116	119	0.1122	1174	48%
ST2	300	410.7	0.002953	101	142	0.0343	3870	84.1%

These measurements investigate the effect of zinc phosphate coatings, ST1 and ST2, on corrosion resistance in a 3.5% NaCl solution compared to uncoated sample ST. The corrosion rates for ST, ST1, and ST2 were (0.2158) mm/y, (0.1122) mm/y, and (0.0343) mm/y, respectively [23]. The results demonstrated that depositing a phosphate layer on a steel surface can improve corrosion resistance. Phosphate coatings created an insoluble crystalline layer that reacted chemically with the surface to provide a protection layer.

3.2.3.Polarization in NaOH Solution

Table 5 displays data that revealed decreased carbon steel corrosion current density (Icorr) when coated with zinc phosphate. The decrease was greater for ST2-coated samples than for ST1-coated samples, suggesting that the zinc phosphate coating, particularly ST2, can considerably enhance corrosion resistance, exhibiting a protection efficiency of 66.45% for ST1 and 85.46% for ST2 in NaOH solution, indicating that the zinc phosphate coating can more effectively isolate the metal surface in the corrosive NaOH environment. These results suggested that the quality of the zinc phosphate coating, especially ST2, is good and can effectively provide corrosion protection in NaOH solution. The corrosion rates for ST, ST1, and ST2 were recorded as (0.01699) mm/y, (0.00561) mm/y, and (0.00247) mm/y, respectively. It was observed that the zinc phosphate coatings' quality, ST1, and ST2, was good as they significantly reduced the corrosion rate of carbon steel in a NaOH solution. The corrosion rates for ST1 and ST2 were much lower than the uncoated sample ST, indicating the coatings' effectiveness in providing corrosion protection. The lower corrosion rate of ST2 than ST1 suggested that ST2 offered a higher-quality coating in this specific environment. Table 6 displays data on the corrosion rate in (mm/y) and (mpy) units by calculation method Weight loss and Tafel.

The two methods, i.e., the weight loss method and the Tafel method, provide corrosion rate values quite close to each other and generally indicate the effectiveness of the zinc phosphate coating in reducing corrosion on the tested samples, with ST2 showing the highest level of corrosion protection.

3.3.Surface Properties

The surface morphology of carbon steel coatings by zinc phosphate was studied by SEM technique. Scanning microscopy analysis before immersion in aqueous media. Figure 4 revealed that the protective coating layer on the steel surface was compact, well crystallized, and provided complete coverage. The coating layer appeared homogeneous, with a uniform gray. The coating demonstrated a large surface area, indicating effective overlapping at the microscopic level, resulting in a layer composed of numerous small needle-shaped crystals. Measurements of the phosphate layer crystals obtained from the phosphate bath showed varying sizes, ranging from 7.7µm to 1.321µm. Notably, the ST2 samples exhibited a decrease in phosphate crystal size, suggesting an influence of the bath on crystal size reduction.

3.3.1.Coating Surface Effected in HCl Corrosive Solution

The acidic nature of the HCl effect was shown by SEM. The zinc phosphate layer's dissolution leaves the underlying carbon steel exposed to a corrosive environment. The SEM images showed small localized areas of corrosion where the surface of the coating layer was pitted or eroded, as shown in Fig. 5. These areas may appear as small craters or holes in the surface of the coating layer and areas of intact coating surrounding them. The severity and extent of the pitting depend on the HCl solution's concentration and temperature. The SEM test of the sample showed that corrosion was formed and a structure gives the crust a small porosity as pitting corrosion after 15 minutes of immersion in HCl solution.

Table 5 Corrosion Parameters for ST, ST1, and ST2 in NaOH Solution.

Sample	T (k)	$-E_{corr}$ (mV)	I_{corr} (mA/cm²)	B_c (mV/dec)	B_a (mV/dec)	C.R (mm/y)	R_P(Ω/cm²)	PE%
ST	300	532.9	0.003291	121	120	0.01699	7938	
ST1	300	457.1	0.000483	81	122	0.00561	19360	66.45%
ST2	300	282.4	0.000213	47	71	0.00247	25610	85.46%

Table 6 Corrosion Rates for ST, ST1, and ST2 in Three Solutions.

Corrosive solution	Sample	C.R. Weight loss method		C.R. Tafel method	
		mm/y	mpy	mm/y	mpy
HCl	ST	0.2199	8.657	0.2515	9.901
	ST1	0.1680	6.616	0.2049	8.066
	ST2	0.1076	4.236	0.1157	4.555
NaCl	ST	0.2136	8.409	0.2158	8.496
	ST1	0.1167	4.590	0.1122	4.417
	ST2	0.0268	1.055	0.0343	1.350
NaOH	ST	0.0297	1.169	0.0169	0.665
	ST1	0.0068	0.267	0.0056	0.220
	ST2	0.0026	0.102	0.0024	0.094

3.3.2.Coating Surface Effected in NaCl Corrosive Solution

The SEM analysis evaluated the surface morphology of the coating layer after exposure to 3.5% NaCl. The surface appeared rougher and more irregular than before exposure, and the coating layer was thinner due to the reaction. As seen in Fig. 6, small pitting corrosion products were deposited on the sample's surface, and a structure caused the crust to have a small porosity as a pitting corrosion at some area position [27]. The coating provided a good protection layer in corrosive media, as shown in Fig. 6. The Ni^{2+} ions increased the alkaline stability of the zinc phosphate layer.

3.3.3. Coating Surface Effected in NaOH Corrosive Solution

The SEM analysis of ST2 appeared as a uniform and dense layer after 15min of immersion at room temperature in NaOH solution. The coating layer also showed a crystalline structure, indicating the formation of a stable and good corrosion-resistant layer. The protective layer underwent a color transformation to a light gray shade. This color change can be attributed to forming a distinct chemical composition or the presence of specific phases within the coating. The light gray shade referred to a successful transformation and implied a favorable alteration in the coating's properties, as shown in Fig. 7. The alkaline stability of phosphate layers was increased by Ni^{2+} ions.

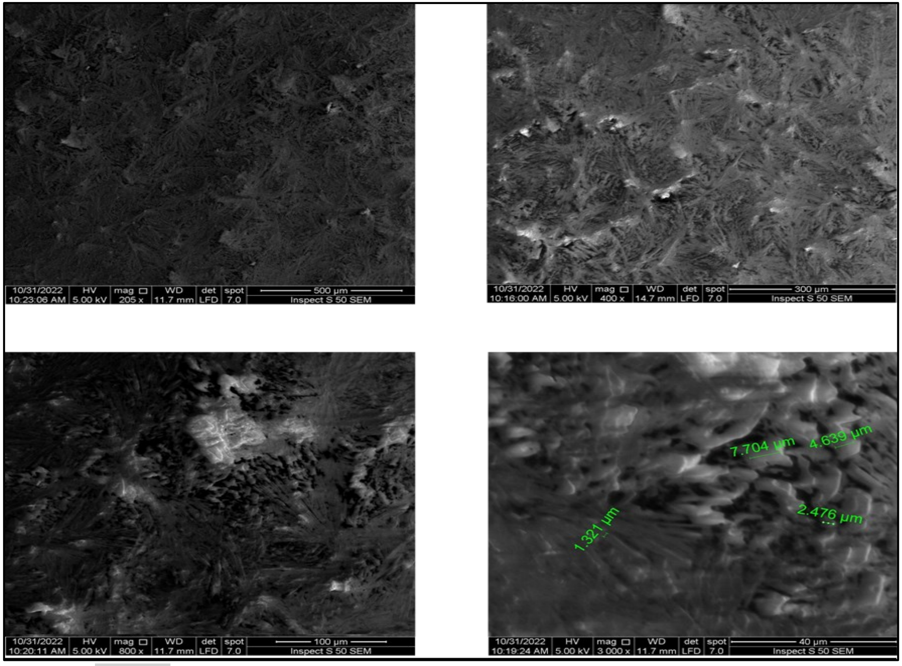

Fig. 4 Coated ST2 Sample by Zinc Phosphate Coating Layer.

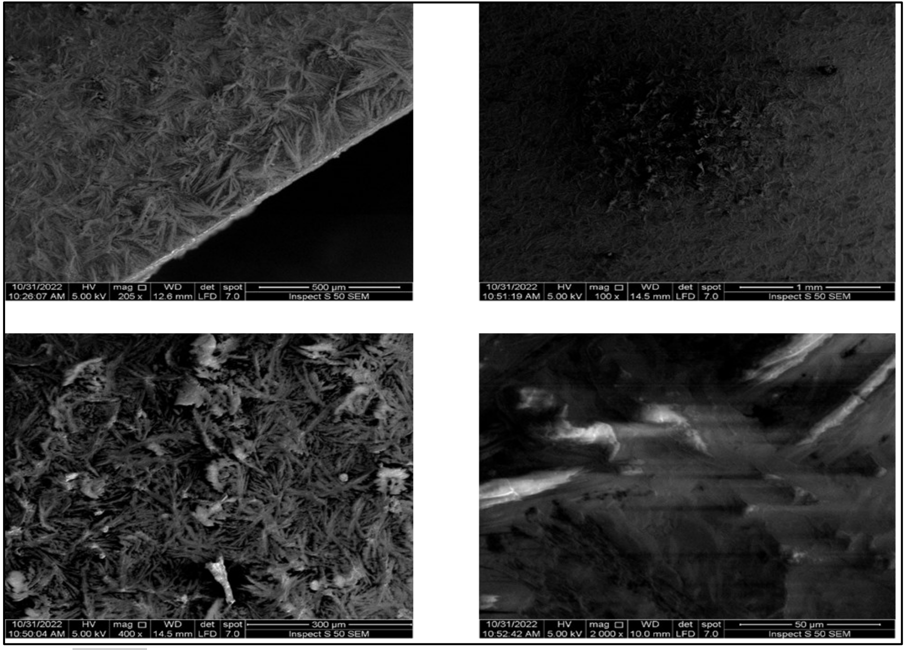

Fig. 5 SEM Images for the ST2 after Immersion in HCl Solution.

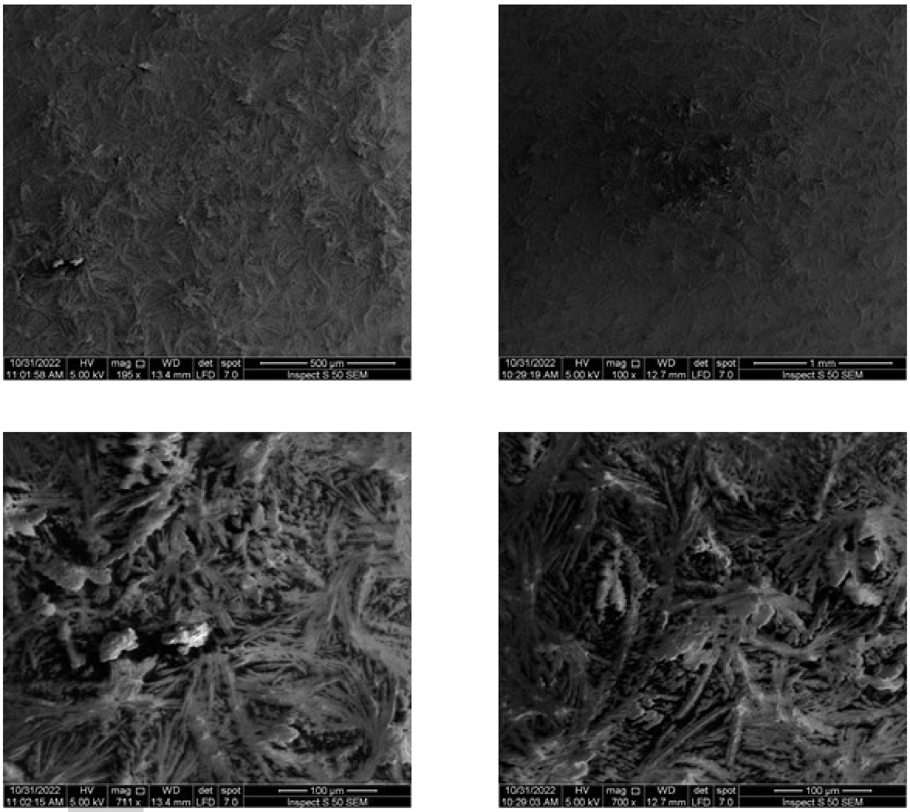

Fig. 6 SEM Images for the ST2 after Immersion in NaCl Solution.

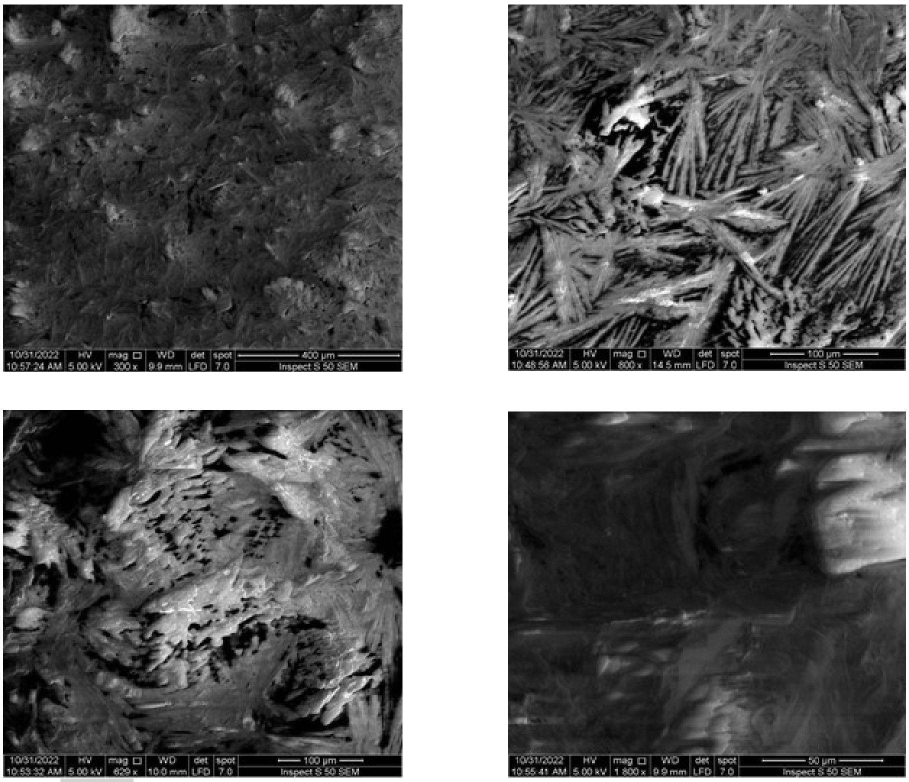

Fig. 7 SEM Images for the ST2 after Immersion in NaOH Solution.

4.CONCLUSIONS

The main conclusions of the present study could be summarized as follows:

- Good corrosion resistance was achieved through zinc phosphating using the standard phosphate bath without any additives (ST1). The protective layer (ST1) demonstrated efficiencies of 23.6%, 45.36%, and 77.10% as determined by the weight loss method, and 18.52%, 48%, and 66.45% as determined by the polarization method in HCl, NaCl, and NaOH solutions, respectively.
- Good corrosion resistance was achieved with the zinc phosphating bath containing nickel carbonate NiCO3 (ST2), with efficiencies of 51.06%, 87.45%, and 91.24% determined by the weight loss method, and 53.99%, 84.15%, and 85.46% determined by the polarization method for the protective layer (ST2) in HCl, NaCl, and NaOH solutions, respectively.
- Adding nickel carbonate NiCO3 in treating carbon steel baths using zinc phosphate improved its corrosion resistance more effectively than standard phosphate.
- The (SEM) showed a compact, well-crystallized protective coating layer on the steel surface, offering complete coverage with minimal porosity, indicating a reduction in phosphate crystal size due to the NiCO3 additive.
- Zinc phosphate is more effective protection in the (NaOH) environment.

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NOMENCLATURE

ΔW	Mass loss in grams
A	Exposed surface area in cm^2
t	Time of exposed in hours
D	Density in g/cm3
CR	Corrosion rate (mm/y)
I_{corr}	Corrosion current density μA/cm2.
e	Equivalent weight (atomic weight / valance).
P	Density of metal g/cm3.
T(k)	Temperature in Kelvin
E_{corr}	Corrosion Potential (mV)
Bc	Beta Cathodic (mV/dec)
Ba	Beta Anodic (mV/dec)
R_P	Corrosion Resistance (Ω/cm2)
PE%	Coating Efficiency

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