Libyan Journal of Medical and Applied Sciences LJMAS
Online ISSN: 3006-1113
Volume 3, Issue 3, 2025, Page No: 187-193
Website: https://ljmas.com/index.php/journal/index

Study of the Effect of Immersion Time in Zinc Phosphate Solution on the Corrosion Protection Efficiency of Steel

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Received: July 12, 2025 Accepted: September 25, 2025 Published: September 29, 2025

Cite this article as: A, A, Alfasi., F, F, ALjaray., K, Abdalla, S, Ghidhan. (2025). Study on the Effect of Immersion Time in Zinc Phosphate Solution on the Corrosion Efficiency Protection of Steel. Libyan Journal of Medical and Applied Sciences (LJMAS). 2025;3(3):187-193

Abstract:

zinc phosphate layers were synthesized on steel surfaces through an electroless deposition method under controlled temperature and pH conditions. The deposition process was conducted for immersion periods of 30, 60, 90, and 120 minutes in the phosphate bath. The surface morphology of the coatings was examined using optical microscopy, and their corrosion resistance was evaluated by weight loss tests carried out within a 3.5% sodium chloride medium. The results revealed a gradual improvement in both coating thickness and surface coverage with increasing immersion time. Dense and uniform layers were obtained after 90 and 120 minutes, with the highest corrosion resistance achieved at 120 minutes. After 24 hours of exposure, the specimen treated for 120 minutes exhibited a minimal weight loss of 0.33 g/m^2, confirming its superior protective performance.

Keywords: Chemical electroless plating, Zinc-phosphate layers, Corrosion Resistance, Steel substrate.

دراسة تأثير زمن الغمر في محلول فوسفات الزنك على كفاءة حماية الفولاذ من التآكل

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الملخص

تم ترسيب طبقات من فوسفات الزنك على أسطح من الفولاذ المطاوع باستخدام تقنية الترسيب الكيميائي غير الكهربائي تحت ظروف مضبوطة من درجة حرارة والحموضة، مع أزمنة ترسيب مختلفة 30، 60، 90، و120 دقيقة. جرى فحص الخصائص الميكروسكوبية للطلاءات بواسطة المجهر الضوئي، بينما تم تقييم مقاومتها للتآكل من خلال قياس الفقدان في الوزن داخل محلول كلوريد الصوديوم بتركيز 3.5%. أظهرت النتائج تحسناً تدريجياً في سُمك الطلاء وتغطية السطح مع زيادة زمن الترسيب. كما تم الحصول على طلاءات مدمجة ومتجانسة عند 90 و120 دقيقة، حيث سُجلت أفضل مقاومة للتآكل بعد 120 دقيقة من الترسيب. وبعد 24 ساعة من الغمر، أظهر العينة المطلية لمدة 120 دقيقة فقداناً وزنيًا لم يتجاوز 0.33 جم /م^2، مؤكداً كفاءتها العالية في الحماية.

الكلمات المفتاحية: الترسيب اللاكهربائي، طبقة الزنك فوسفات، مقاومة التآكل. الفولاذ

Introduction

Corrosion poses a critical problem in numerous industrial applications, particularly within construction and manufacturing, where iron and steel remain the preferred materials due to their excellent mechanical strength and widespread engineering use worldwide [1–2]. Despite these advantages, their susceptibility to degradation in aggressive environments limits their long-term performance. To address this issue, researchers have developed various strategies to improve steel durability. Among these approaches, conversion coatings based on zinc phosphate are frequently employed to provide enhanced surface protection and increase resistance to corrosive damage [3]

Zinc phosphating serves as a key method for pretreating metal surfaces, applicable to both ferrous and non-ferrous materials. Its popularity stems from its cost-effectiveness, fast processing, and the ability to improve surface properties such as resistance to corrosion and wear, adhesion, and lubrication. This treatment method is particularly valuable in industries including automotive manufacturing, industrial processing, and household appliances [4–7].

Zinc phosphate layers are typically created on metal surfaces through immersion in solutions containing zinc and phosphate ions, along with accelerating agents such as nitrate and nitrite [9]. These agents enhance the partial dissolution and oxidation of the metal, which facilitates the formation of insoluble phosphate deposits [12]. During this chemical phosphating process, insoluble phosphate compounds form on the surface through reactions between zinc, phosphate, and sometimes iron ions. The main deposited phase, hopeite ($Zn_3(PO_4)_2 \cdot 4H_2O$), a hydrated zinc phosphate, constitutes the primary layer formed during chemical phosphating, and its precipitation occurs due to interactions between zinc and phosphate ions, particularly under alkaline conditions generated by hydrogen evolution at the metal–solution interface, as represented by the equation: [11].

$$3Zn^{2+} + 2PO_4^{3-} + 4H_2O \rightarrow Zn_3(PO_4)_2.4H_2O \qquad (hopeite)$$

Furthermore, when iron ions are present in the bath, a secondary phase known as phosphophyllite can also form alongside hopeite, according to:

$$2Zn^{2+} + Fe^{2+} + 2PO_4^{3-} + 4H_2O \rightarrow Zn_2Fe(PO_4)_2.4H_2O \quad (phosphophyllite)$$

These reactions result in the formation of protective phosphate layers on the metal surface, enhancing corrosion resistance and improving the adhesion of subsequent coatings. The precise composition and proportion of these phases depend on the solution chemistry and the presence of metallic ions such as iron [12].

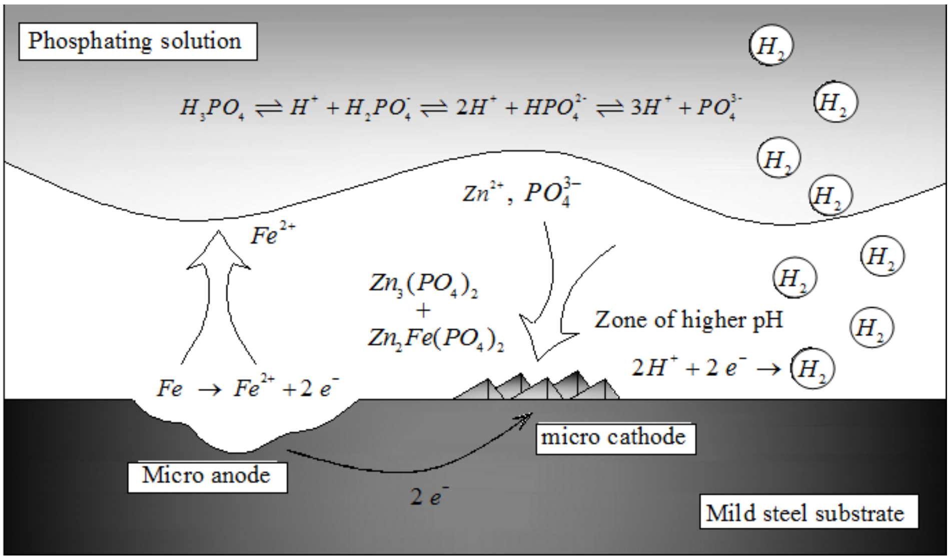

Figure 1 . Schematic illustration of the zinc phosphating mechanism on the steel surface

This research examines the performance of zinc phosphate coatings under controlled experimental conditions, specifically maintaining a constant temperature of 65 ºC and a solution pH of 2.8. The study primarily focuses on assessing how the duration of deposition affects the quality of the resulting coatings.

The effectiveness of electroless coatings depends on multiple factors, particularly the key process parameters including deposition temperature, solution pH, and deposition duration, as well as the resulting microstructural characteristics [5,6]. Among these, the length of deposition plays a vital role because it directly impacts the coating’s morphology, thickness, and adhesion. In the current study, deposition intervals of 30, 60, 90, and 120 minutes were carefully analyzed to determine the optimal duration that maximizes corrosion resistance.

Experimental

In this study, Pieces of mild steel from Iron and Steel Company of Misrata-Libya, measuring 15 × 10 × 2 mm were employed in the tests, with their elemental analysis provided in Table 1, were selected to prepare phosphate coatings using electroless plating from an acidic solution. The bath composition and experimental parameters are summarized in Table 2. The pH of the solution was adjusted to 2.75 with a 50% NaOH solution, consistent with prior studies recommending a pH range of 1.75–2.75 for optimal deposition [7,8]. The deposition temperature was maintained at 65 ºC based on preliminary experiments and literature findings [8].

Before initiating the phosphating process, the mild steel specimens were mechanically polished using silicon carbide abrasive papers up to 400 grit to ensure a smooth surface finish. They were then ultrasonically cleaned in acetone to remove contaminants, followed by rinsing with deionized water. Immediately after preparation, the samples were immersed in the phosphating bath for different deposition times under controlled temperature conditions, while the bath pH was carefully adjusted using a 50% NaOH solution. Once the coating was completed, the specimens were rinsed again with deionized water and dried using compressed air. The schematic representation of the experimental setup is illustrated in Fig. 2, and the coating weight was determined based on the procedure described in [9,10].

$$W_{m(coat)} = \frac{w_{mf} - w_{mi}}{A_{SS}}$$

Here, $W_{m(coat)}$ represents the weight mass of the phosphate coating on the metal, expressed (g/m²). The term w_{mi} refers to the mass of the specimen before phosphating, w_{mf} corresponds to the mass after the coating process, and A_{SS} indicates the total surface area of the coated sample.

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Table 1. Elemental Analysis of Mild Steel

Element	Composition (weight %)
C	**0.171**
Si	**0.022**
Mn	**0.711**
P	**0.017**
Cu	**0.007**
Al	**0.065**
Fe	**Balance**

Table 2. Chemical Characteristics and Working Conditions of the Phosphating Solution

Phosphate Bath Composition	Concertation (g/L)	Bath Processing Parameters
H_3PO_4 (85%)	15	**T=60~65 ºC** **PH=2.75~2.80** **Time = 30 ~ 120 min**
ZnO	2.5	
HNO_3 (70%)	25	

The phosphate-coated specimens were immersed in a 3.5% sodium chloride solution at room temperature. The initial mass of each sample (w_a) was measured prior to immersion, and the specimens were exposed individually according to the designated immersion periods. Upon completion of the immersion durations (24 and 48 h), the samples were removed, thoroughly dried, and reweighed (w_b). The corrosion-induced mass loss over 24 and 48 hours was subsequently calculated using the following equation [11,12].

$$W_{m(loss)} = \frac{w_{ma} - w_{mb}}{A_{SS}}$$

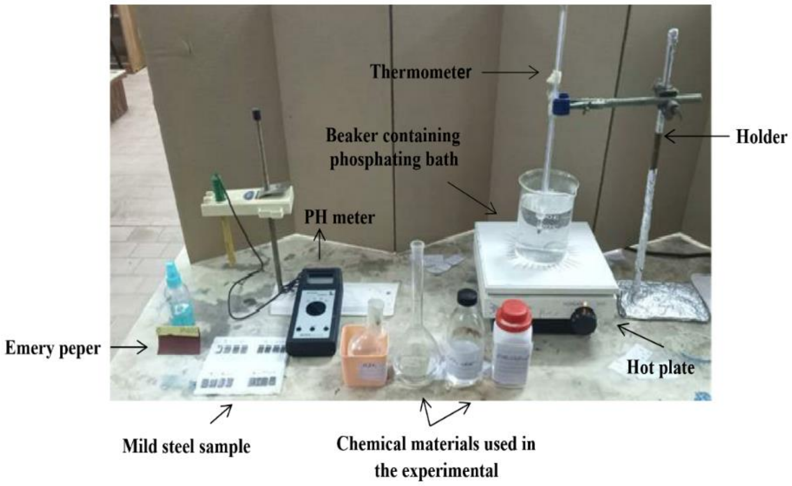

Figure 2. Phosphating Process Setup for Mild Steel Samples

Here, $W_{m(loss)}$ represents the mass lost by the coated specimen, which serves as an indicator of the corrosion of the specimen's coating, expressed (g/m²). And A_{SS} Total Surface Area of the Specimen. The weights were measured using a high-precision analytical balance, as illustrated in Fig. 3

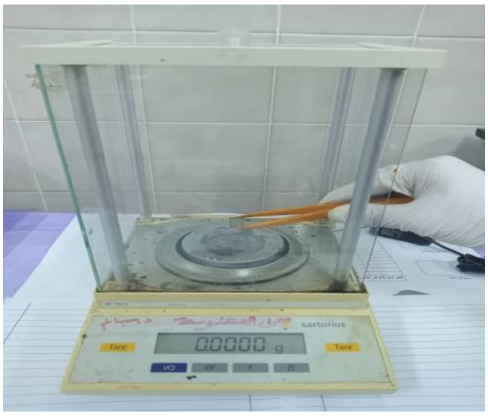

Figure 3. Analytical Balance

Results And Discussion

Microscopic and macroscopic image of phosphate –coated surfaces

As illustrated in Fig. 4, the mild steel surface exhibited a distinct change in appearance after the phosphating treatment. The shiny metallic silver of the polished substrate was replaced by a homogeneous dark-grey shade, confirming the formation of a continuous zinc phosphate layer. The complete surface coverage reflects the uniformity and density of the deposited coating.

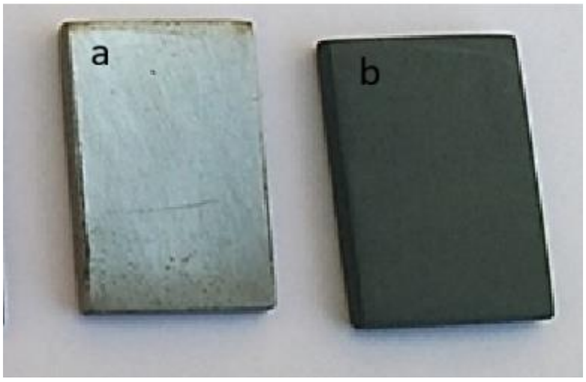

Figure 4. Optical images of mild steel: (a) before phosphating treatment (b) after phosphating treatment

Figure 5 (a–e) presents the morphological evolution of the steel surface with increasing immersion times in the phosphate bath. In the untreated state (a), the surface appears relatively smooth and free of deposits. After 30 min (b), small and sparsely distributed phosphate nuclei are observed, marking the onset of coating formation. At 60 min (c), the number of crystals increases and becomes more evenly distributed, producing a denser layer compared with the earlier stage. By 90 min (d), crystal size and density show significant growth, resulting in overlapping crystals and a more compact film. After 120 min (e), the surface is almost entirely covered with large, well-developed phosphate crystals, forming a continuous and protective layer. These observations indicate that longer immersion times promote higher coating density and thickness, with full layer development occurring at 120 min, which is expected to enhance corrosion resistance.

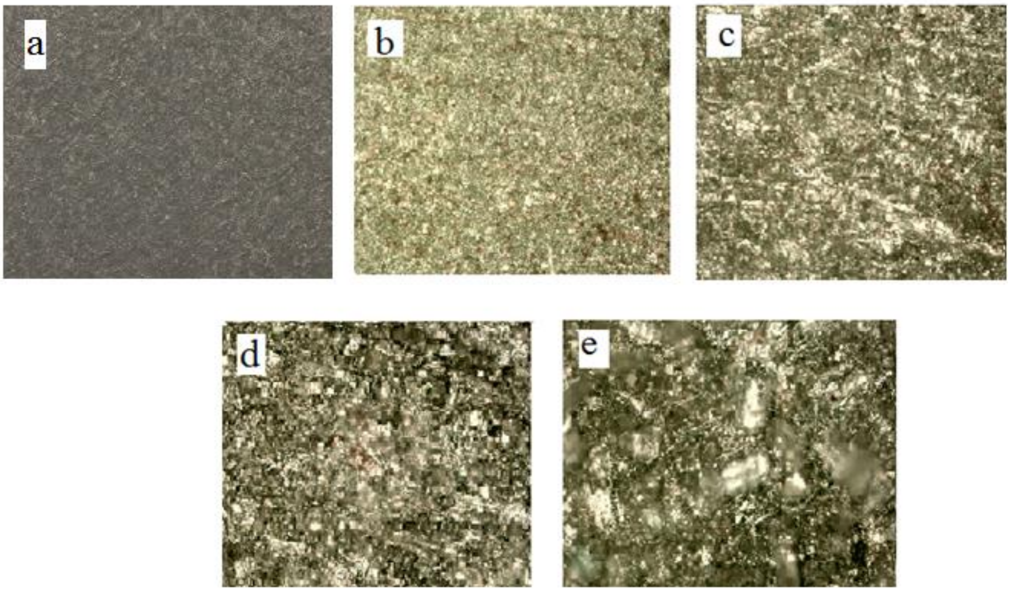

Figure 5. Optical microscopy images illustrating the surface of mild steel: (a) untreated substrate (b) specimen coated with zinc phosphate after 30 min, (c) after 60 min, (d) after 90 min, and (e) after 120 min of immersion.

Evaluation of Phosphate Coating Weight

The data presented in Figure 6 show that the phosphate coating weight progressively increased as the deposition time was extended from 30 to 120 minutes. This suggests that longer treatment times not only stimulate the nucleation of phosphate crystals but also promote their subsequent growth and densification on the metal surface.

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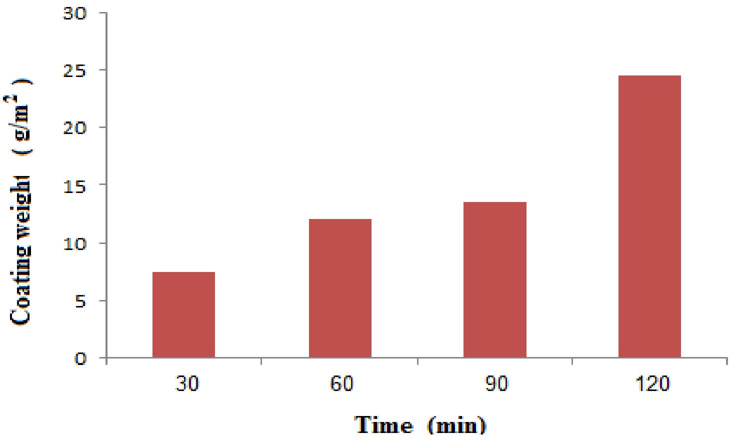

Figure 6. Effect of Immersion Time on the Weight mass of the Phosphate Coating on Steel.

Assessment of Corrosion Resistance Using Immersion Testing

Table 3 presents the results of the immersion test in a 3.5% sodium chloride solution, assessed through weight loss measurements, for mild steel and phosphate coatings applied at various deposition times. The data indicate that the phosphate coatings acted as an effective barrier, protecting the mild steel substrates from the corrosive effects of the sodium chloride solution. For the uncoated mild steel, weight loss after 24 and 48 hours of immersion was 4.33 and 7.33 g/m², respectively, whereas the coated sample with a deposition time of 120 minutes exhibited significantly lower weight loss of 0.33 and 0.66 g/m². Moreover, the results show that increasing the deposition time enhanced the corrosion resistance of the coatings. Overall, the phosphate layer provided excellent protection against corrosion, with the highest resistance observed at 120 minutes of deposition.

Table 3. Evaluation of Phosphate Coatings' Corrosion Behavior in 3.5% NaCl Solution

Base Material	$W_{m(loss)}$ **after 24 hours (g/m^2)**	$W_{m(loss)}$ **after 48 hours (g/m^2)**
Mild steel without coating	4.33	**7.33**
Zinc-phosphated layer applied for 30 minutes	2	**4**
Zinc-phosphated layer applied for 60 minutes	1.833	**3.66**
Zinc-phosphated layer applied for 90 minutes	1.333	**2.66**
Zinc-phosphated layer applied for 120 minutes	0.33	**0.66**

Conclusions

This work focused on forming zinc phosphate layers on mild steel substrates through an electroless deposition method at various time intervals. The results indicated that the duration of deposition strongly influences the microstructure and corrosion resistance of the layers. Samples treated for 90 and 120 minutes displayed a compact and uniform morphology, whereas the optimal surface coverage and highest corrosion protection were achieved after 120 minutes. These findings underscore the importance of controlling the deposition period to enhance the protective performance of zinc phosphate layers.

Acknowledgments

The authors sincerely thank Al-Merqib University – Libya for their support and for providing the necessary facilities to carry out this research. Special appreciation is also extended to the Department of Chemical Engineering for their guidance and assistance throughout the study.

References

1. Sankara Narayanan, T., & Seshadri, S. (2008). Electro- and electroless plated coatings for corrosion protection.
2. Liu, Y., Xu, J., Zhang, J., & Hu, J. (2017). Electrodeposited silica film interlayer for active corrosion protection. Corrosion Science, 120, 61–74.
3. Sankara Narayanan, T. S. N., Jegannathan, S., & Ravichandran, K. (2006). Corrosion resistance of phosphate coatings obtained by cathodic electrochemical treatment: Role of anode – graphite versus steel. Progress in Organic Coatings, 55(4), 355–362.
4. Wang, C. M., Liau, H. C., & Tsai, W. T. (2006). Effects of temperature and applied potential on the microstructure and electrochemical behavior of manganese phosphate coating. Surface and Coatings Technology, 201(6), 2994–3001.
5. Rattanawaleedirojn, P., et al. (2017). Factors affecting the corrosion resistance of electroless Ni-Zn-P coated steel. In Key Engineering Materials (Vol. 728, pp. 39–44). Trans Tech Publications.
6. Maniam, K. K., & Paul, S. (2020). Progress in electrodeposition of zinc and zinc–nickel alloys using ionic liquids. Applied Sciences, 10(15), 5321.
7. Abdalla, K., et al. (2013). The influence of deposition temperature on the morphology and corrosion resistance of zinc phosphate coating on mild steel. Advanced Materials Research, 626, 183–189.
8. Abdalla, K., et al. (2013). The effect of zinc phosphate coating morphology on its corrosion resistance on mild steel. Advanced Materials Research, 626, 569–574.
9. Abdalla, K., et al. (2017). Characteristics of zinc phosphate coating activated by different concentrations of nickel acetate solution. Metallurgical and Materials Transactions A, 48(2), 771–779. https://doi.org/10.1007/s11661-016-3850-1
10. Abdalla, K., & Zuhailawati, H. (2021). Corrosion performance and morphological analysis of activated zinc phosphate coating formed on steel surface. Anti-Corrosion Methods and Materials. Advance online publication. https://doi.org/10.1108/ACMM-03-2021-2462

11. Ibrahim, H., et al. (2021, December 14–16). Effect of process time in the deposition and corrosion resistance of Ni-Zn-P coating formed on mild steel surface. In Proceedings of the 4th Conference on Engineering Science and Technology (CEST 2021), Zliten, Libya.
12. Alfasi, A. A., et al. (2024). Surface morphology and anticorrosion properties of zinc phosphate coating deposited with various concentrations of zinc oxide. Journal of Materials Science and Engineering, 1(1).