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Effect of Process Time in the Deposition and Corrosion Resistance of Ni-Zn-P Coating Formed on Mild Steel Surface

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ABSTRACT

In this investigation, uniform and full coverage of Ni-Zn-P coatings were electroless deposited successfully on mild steel substrates. The coatings were electroless formed at 85°C for various deposition time (10, 30, 60 and 90 min). The coating morphology were observed via optical microscope and the corrosion resistance of obtained coatings were evaluated by weight loss test in a 3.5% NaCl solution. The results revealed that the coating deposition increased as the process time increased. The optimal uniform coating deposition was formed at deposition time of 60 & 90 min. Accordingly, the anti-corrosion performance was improved and the best corrosion resistance was obtained at process time of 90 min. The weight loss was 0.55 g/m² after 24 h immersion time for the coating formed with process time of 90 min.

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1 INTRODUCTION

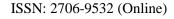
Electroless deposition. Ni-Zn-P coating.

Corrosion resistance.

Keywords:

Nickel.

Ni-Zn electroless coatings have been widely used as a protective coating for metal surfaces, especially for steel [1-4]. They provide variety of desirable properties such as good hardness, wear resistance and corrosion protection [5-8]. Compared to electrodeposition coatings, electroless deposition technique produce coatings with uniform and full coverage regardless of the sample shape.





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The electroless coatings performance depend on many factors including process parameters (deposition temperature, pH of the solution and process time) and its microstructure [9, 10]. The inclusion of zinc on the chemical composition of electroless Ni-Zn-P coating was reported by Abdel Hamid et al. [11]. The codeposition of Zn with Ni-P resulted in transformation of the coating structure from amorphous to crystalline. It is also noted that the corrosion resistance was significantly improved as the percentage of the zinc reached to 7.4% in the coating composition. Isaac Fayomi et al. [12] have investigated the effect of deposition time on the anticorrosion and microstructural properties of Zn-Ni-P coating formed by electrodeposition technique on mild steel surface. They found that, higher corrosion performance was provided as the time of deposition increased. Remarkably, a predominant refine crystal orientation on the morphology was noticed, which produced coatings with free of stresses, porosity, and cracks at the optimum time. Therefore, optimization the electroless deposition parameters such as time and temperature is very important to obtain coating with uniform deposition, full coverage and good corrosion performance. In this study, the Ni-Zn-P coatings were formed on mild steel samples by using electroless deposition method with alkaline bath. The effect of deposition time in the coating formation and corrosion resistance were evaluated and addressed.

2 MATERIALS AND METHODS

Mild steel samples with dimension of $10 \times 15 \times 1.5$ mm and chemical composition as listed in Table 1 were used in this study to produce Ni-Zn-P coatings by electroless plating from alkaline solutions. The chemical composition and operating conditions for the alkaline bath used in this study are shown in Table 2. The pH of the coating solution was hosen (9.5) based on previous literatures which recommended the range of pH should be between 8 and 11 [5,11]. The values of pH higher than 11 and lower than 8 provide coatings with less deposition and coverage. The pH in this study was adjusted by adding a 50 % NaOH solution. The temperature also has a strong effect on the coating deposition, and based on some preliminary experiments and results (not shown here) the temperature fixed at 85°C. This was in agreement with reported study by Abdel Hamid, *et al.* [11], where the best deposition temperature for Ni-Zn-P coating was in the range of 75 to 90°C.

The mild steel samples were firstly abraded with emery SiC papers till 420 grits, and then degreased in acetone and cleaned thoroughly in deionized water respectively. Thereafter, the substrates immersed in the alkaline coating bath for different processing time. Eventually, the coated samples were cleaned thoroughly by deionized water and dried by compressed air. The experimental set up of coating deposition is presented in Fig. 1.

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Element	Composition	
С	0.16	
Al	0.07	
Si	0.168	
Mn	0.18	
Р	0.025	
Cu	0.09	
Fe	balance	

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The surface morphology of the obtained coatings was photographed and observed using optical microscope. The corrosion performance of the obtained coatings was evaluated by total immersion test in an aerated 3.5% sodium chloride solution at room temperature. The calculations were taken by measuring the substrate weight before immersion in the corrosive solution (W'). After completing the test period, the samples were taken out, rinsed with deionized water, dried with compressed air and then weighed again (W"). The weight loss in the sample weight after 24 and 168 hours of immersion, which related to the corrosion performance, was determined according to the following equation [13, 14]:

$$w_{loss} = \frac{w' - w''}{A} \tag{1}$$

where, W_{loss} is the weight loss in the coated sample (g/cm²), and A is the surface area of the substrate (cm²). All the measurements were performed by analytical balance with an accuracy of ± 0.0001 g, and the results were the average of three times.

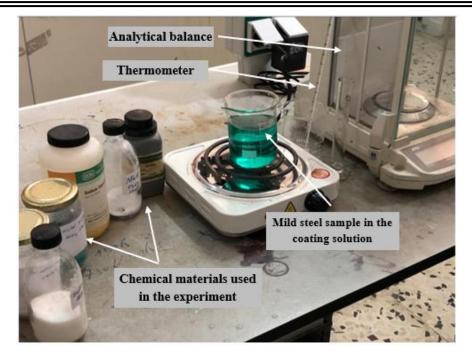


Figure 1. experimental set up for Ni-Zn-P coating deposited on mild steel surface.

Bath composition	(g/l)	Operating conditions	
Zinc sulphate	2.8	T = 85 °C	
Nickel sulphate	26	pH = 9.5	
Sodium hypophosphite	17.5	Time = $10 \sim 90$ min	
Sodium citrate	41	-	
Ammonium chloride	25	-	

Table 2. Chemical composition of the coating solution and operating conditions

3 RESULTS AND DISCUSSION

3.1 Surface morphology of the coatings

Photos of the mild steel substrate before and after coating treatment are shown in Fig. 2. The colour of polished mild steel substrate has changed from shining silver to uniform light-grey after deposition of Ni-Zn-P coating by immersion technique, indicating the formation of a uniform layer on the metal surface. Optical microscope micrographs of bare mild steel surface, and Ni-Zn-P coatings for various process time are presented in Fig. 3. The bare mild steel sample showing rough and scratched surface which resulted from SiC paper grinding. After ten minutes of deposition time of Ni-Zn-P coating the surface morphology of steel surface has changed and presented smooth surface and fine microstructure, indicating the precipitation of the coating started at early stage. As the deposition time increased to 90 min

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the surface of coated substrates demonstrated a very fine coating with a uniform and full coverage. The formation of electroless Ni-Zn-P coating on mild steel surface occurred though some cathodic reactions which start at metal-bath interface. These reactions resulted in reduction of Zn, Ni, and P on the metal surface as follows [11]:

$$Ni^{2+} + 2H_2PO_2 + 2H_2O \rightarrow Ni + H_2PO_3^- + 2H^+$$
(1)

$$2H_2PO_2 \rightarrow H_2PO_3^- + P + OH^- + \frac{1}{2}H_2$$
 (2)

$$Zn^{+2} + 2e \rightarrow Zn \tag{3}$$

3.2 Corrosion resistance of Ni-Zn-P coatings evaluated by weight loss test

The corrosion performance, evaluated by weight loss test, of mild steel sample and Ni-Zn-P coatings obtained at various deposition time are shown in Table 3. It is observed that the deposited coatings provided a barrier protection to the mild steel substrates from the corrosive media of sodium chloride solution. The weight loss of bare steel sample after 24 and 168 h of immersion time was 3.25 and 16.93 g/m2 respectively, whilst it was 0.55 and 5.39 g/m2 for the coating deposited with 90 min of process time. It was also noticed that, as the process time increased the corrosion performance of coated substrates was improved indicating a remarkable effect of process time in the coating deposition and corrosion protection to the mild steel surface and the best corrosion resistance obtained with deposition time of 90 min.

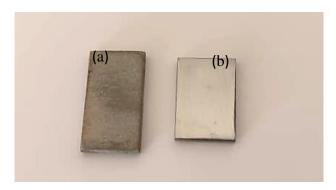


Figure 2. Photos of (a) Ni-Zn-P coating on mild steel surface performed at 85°C for 90 min, and (b) bare mild steel surface.

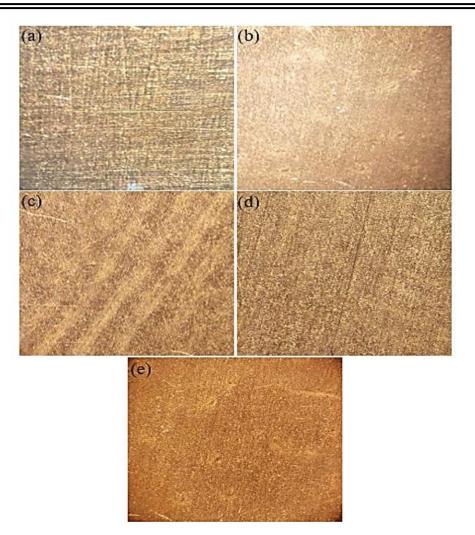


Figure 3. Optical microscope micrographs of (a) bare mild steel surface, and Ni-Zn-P coatings with various process time: (b) 10 min, (c) 30 min, (d) 60 min, and (e) 90 min.

Table 3. Corrosion resistance of Ni-Zn-I	P coatings evaluated by	y immersion in 3.5% NaCl solution
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Substrate	Loss in wei after 24 (g/m^2)	ght Corrosion h after 24 h $(g/m^2.s)$	rate Loss in we after 168 (g/m^2)	ight Corrosion h after 168h (g/m ² .s)	rate
Bare mild steel	3.25	3.76×10 ⁻⁵	16.93	2.80×10 ⁻⁵	
Ni-Zn-P coating obtained with 1 min of processing time	02.85	3.3×10 ⁻⁵	12.06	2.00×10 ⁻⁵	
Ni-Zn-P coating obtained with 3 min of processing time	³⁰ 2.71	1.45×10 ⁻⁵	10.61	1.75×10 ⁻⁵	
Ni-Zn-P coating obtained with 6 min of processing time	⁶⁰ 1.95	2.26×10 ⁻⁵	9.15	1.51×10 ⁻⁵	
Ni-Zn-P coating obtained with 9 min of processing time	000.55	6.37×10 ⁻⁶	5.39	8.91×10 ⁻⁶	

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4 CONCLUSIONS

Ni-Zn-P coatings formed on mild steel surface by electroless method at various deposition time were addressed in this study. The optical microscope micrographs showed the fine and dense coatings were formed at process time of 60 and 90 min. The weight loss test demonstrated that the corrosion resistance of steel substrate was significantly improved after coating formation. The deposition time has a strong effect on the coating formation and its anticorrosion performance. The best coating coverage and corrosion resistance was obtained for Ni-Zn-P coating deposited for 90min.

5 ACKNOWLEDGMENT

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تأثير الزمن في ترسيب ومقاومة التآكل لطبقة النيكل-الزنك-الفوسفور المتكونة على سلح الفولاذ المطاوع

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الملخص

في هذه الدراسة، تم ترسيب طبقة منتظمة وذات غطاء كامل من النيك-الزنك-الفوسفور على سطح عينات من الفولاذ المطاوع. الطبقة ترسبت كيمائيا من غير استخدام التيار الكهربائي عند درجة حرارة 85 م⁶ ولفترات زمنية مختلفة (10، 30، 60، 90 دقيقة). تم فحص سطح العينة عن طريق الميكروسكوب وتم اختبار مقاومة التآكل عن طريق اختبار فقد الوزن في محلول كلوريد الصوديوم بتركيز 3.5%. النتائج أظهرت ان ترسيب الطبقة يزداد كلما زاد زمن العملية وكان أفضل تكوين للطبقة عندما كان زمن العملية 60 و90 دقيقة. وتبعا لذلك تحسنت مقاومة التآكل مع زيادة ترسيب الطبقة وكانت افضل مقاومة للتآكل عندما كان زمن العملية 90 دقيقة. الفقد في الوزن كان مقداره 0.55 جم/م² عندما تكونت الطبقة في زمن قدره 90 دقيقة.

ا**لكلمات الدالة:** الترسيب اللاكهريائي. طبقة النيكل-الزنك-الفسفور. النيكل. مقاومة التآكل.

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