Int. J. Electrochem. Sci., 10 **(2015)** 10821 - 10831

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

Effect of Processing Factors and Ambient Conditions on Phytic Acid Conversion Coating on Zinc by Atmospheric Corrosion Monitor Sensor

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Received: 18 September 2015 / *Accepted:* 10 October 2015 / *Published:* 4 November 2015

Both the effect of forming conditions on corrosion resistance of phytic acid conversion coating on zinc in air and the influence of ambient factors (temperature and relative humidity) on service of conversion coating were studied by SEM, EDS, ACM and SKP. Zinc-graphite ACM sensors and zinc plates were treated with phytic acid solution at different processing time and pH values. The results showed that longer processing time and alkalinity phytic acid solution weaken the corrosion resistance of treated zinc. Self-assembled monolayer of zinc phytate provided the best protection. The corrosion resistance of conversion coating with phytic acid solution was reduced when the ambient temperature or relative humidity is high values, which due to the zinc phytate bond strength with zinc matrix and co-adoption water on it.

Keywords: Phytic acid; Zinc; atmospheric corrosion monitor; atmospheric corrosion;

1. INTRODUCTION

Zinc has been extensively used as a coating to protect metals for its behaving as barrier and sacrificial anodes [1]. However, zinc is electrochemical active as a result of high corrosion rate in some environments, which reduce its service life [2]. In the past years, demand on improving corrosion resistance of metal coating has increased [3-8]. One of the most effective methods is to form a conversion coating on the surface of zinc. Chromate conversion coating can provide exceptionally good corrosion resistance [9], but the treatment solution containing hexavalent chromium compounds is harmful to the environment, which has been restricted and forbidden to be used in many countries and regions [10]. The seeking of environmentally-friendly substituents of chromium is needed. A few studies have been developed, including Permanganate [11], phosphate [12], phosphate-permanganate [13], stannate [14], molybdate [15], tungstate [16], rare earth [17], nickel [18], bismuth [19], silanes [20], polymer [21] and phytic acid [22].

Phytic acid is a naturally occurring material that is nontoxic, biocompatible, and green to the environment [23]. Phytic acid with the particular structure, consists of 24 oxygen atoms, 12 hydroxyl groups and 6 phosphate carboxyl groups, has a powerful chelating capability with many metal ions [24-27]. When the metal atoms or ions on the surface of metals react with the active groups of phytic acid to form chelate compounds, the complex compounds will be deposited on the surface of metals to form a steady chemical conversion layer to improve the corrosion resistance [28]. There are some literatures about the corrosion resistance of conversion coating on magnesium alloys [28-30] and copper alloys [31-34]. The results show that the corrosion resistance of coating conversion treatment with phytic acid is improved obviously. There are also a few works investigated conversion coating on zinc and zinc alloys. Bikulcius et al. [26] found that the phytic acid conversion coating can evidently improve the corrosion resistance of Zn–Co alloys, and the corrosion current density of phytic acid conversion coating is smaller than that of chromate conversion coating. While El-Sayed et al. [35] investigated conversion coating on galvanized steel by effect of phytic acid and the results show that the corrosion current density of phytic acid conversion coating is 1/3-1/2 that of untreated coating. Liu et al. [36,37] acknowledged the corrosion resistance of phytic acid conversion coating is inferior to that of chromate conversion coating.

The results of phytic acid conversion coating on zinc differing in corrosion resistance are related to many factors. The effect of forming conditions on corrosion resistance of conversion coatings on zinc has been less studied. The influence of ambient factors on corrosion behavior of conversion coating on zinc has not studied. Moreover, all the aforementioned studies were processed in solution when zinc conversion coating is used in air. Atmospheric Corrosion Monitor (ACM) has been proposed as an effective method to study the corrosivity of metals in air due to several distinct advantages such as quantitative, direct and automatic measurement of the corrosion rate [38,39]. There have been no reports on corrosion monitoring for conversion coating on zinc surface using ACM.

In the present work, an ACM sensor, which is graphite and phytic acid conversion zinc electrodes couple type, was used to investigate the effect of both of chelating time and pH of phytic acid on the conversion coating's corrosion resistance. It was also used to investigated the effect of environment factors (e g. RH and temperature) on the conversion coating behavior. In order to detected the difference of corrosion resistance between untreated and conversion zinc, Scanning Kelvin probe was used in this paper.

2. EXPERIMENTAL

2.1. Features of ACMs

The structure of the ACM sensor (developed by the Tianjin University–Liceram Joint Laboratory) is in the sandwich form, and there is a porous insulating film (0.02 mm thick) between the tested Zn specimen and conductive graphite film. The schematic illustration of the Zn–Graphite coupling type ACM sensor is shown in Fig. 1. This galvanic current passing between Zn and graphite has been found to show a good relationship with the corrosion rate of metals [38,39]. Therefore, the corrosion process of Zn with conversion coating can be monitored by measuring the galvanic current.

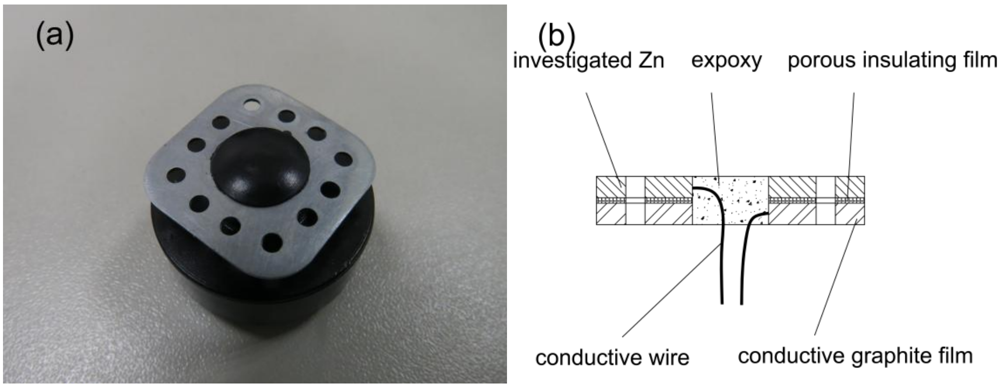

Figure 1. (a) Zn-graphite ACM sensor detecting corrosion used in the present work, and (b) the schematic diagram of the ACM sensor.

2.2. Sample preparation

The conversion solution containing 1mM of phytic acid was prepared by dissolving the appropriate amount by mass in deionized water. The phytic acid was a chemical reagent, purity=70%, and was purchased from Aladdin Chemistry Co., Ltd., China. The pH value of the phytic acid solution was adjusted by diluted NaOH solution to 3.08, 6.94 and 11.07. The pH values of solutions were measured by PHB-4 pH Meter (INESA Scientific Instrument Co., Ltd, Shanghai).

Zinc electrodes of ACM sensors and Zinc plate samples, used as substrates, were made of high purity (99.99%) zinc. Zinc electrodes of ACM sensors were mechanically abraded with SiC paper up to 400 grit. Zinc plates were abraded with SiC paper up to 1000 grit, following 2.5 μm alumina was used to polish the zinc plates until shiny mirror-like surface was obtained. All the zinc electrodes of ACM sensors and zinc plates were washed with absolute ethanol in an ultrasonic bath for 10 minutes, and dried with cold air. The conversion coatings on zinc electrodes were formed by immersing the zinc electrodes in the phytic acid solutions at room temperature. Phytic acid solution was dropped onto the zinc plate surface. The time of immersion or droplet staying were chosen as 1.5 min, 15 min and 60 min. After conversion, the zinc electrodes and zinc plates were washed using running deionized water and absolute ethanol, dried with cold air for 10 minutes and then measured.

2.3. Measurement

2.3.1. ACM laboratory corrosion test

The phytic acid conversion Zn-graphite ACM was placed inside a chamber with controlled humidity or temperature. The temperature was set at 10, 30 and 50 ℃, when RH variation was set as a constant value of 80%. When Temperature was set as a constant value of 30 ℃, the RH was set at 40%, 60%, 80%, and 90%, respectively. The ACM sensor was kept detecting for a few hours at a pair of RH and temperature values.

2.3.2. Surface analyses and characterization

The surface morphology of samples was observed by field-emission scanning electron microscopy (FESEM, SUPPA 55, ZEISS, Germany). The characterization of the composition of elements in the conversion coatings on zinc plates was detected by energy dispersive spectrometer (EDS, X-Max, OXFORD, England).

Scanning Kelvin probe (SKP, AMETEK 7230, USA) was used to detected the difference in the electrochemical activity on zinc plate surface with both of untreated and conversed area by phytic acid. The distance between the 500 μm diameter SKP needle and the zinc plate surface was in the range of 100 μm. The distance was kept constant during measurement. The vibration amplitude and the frequency were 30 μm and 100 Hz respectively. All the SKP measurements were performed at the ambient relative humidity (64%) and temperature (26 ℃).

3. RESULTS AND DISCUSSION

3.1. Influence of processing factors on conversion coating

3.1.1. Influence of immersion time in phytic acid solution

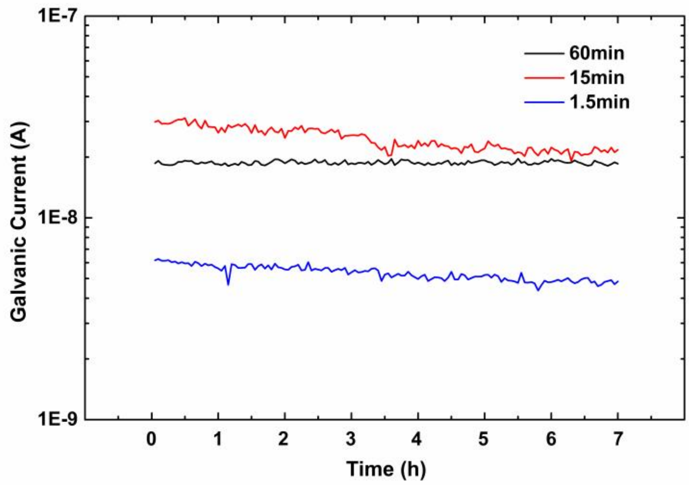

Figure 2. Galvanic current of ACM sensor with phytic acid (pH=3) conversion for different immersion time.

Fig. 2 shows the galvanic current data detected by ACM sensors inside a chamber with controlled humidity 80% and temperature 30 ℃. Zinc electrodes of these ACM sensors were treated by phytic acid solution (pH=3) for 1.5 minutes, 15 minutes and 60 minutes respectively. The galvanic current value of zinc electrode for immersion time of 1.5 minutes is the smallest of them, which is about 1/3 value of that immersion time 60 minutes and 1/5 value of that immersion time 15 minutes. It means that the conversion coating with immersion time 1.5 minutes has the best protection, while the conversion coating with immersion time 15 minutes has the worst protection.

In order to investigated the difference of these conversion coatings with phytic acid for different immersion time, surface morphological analysis of treated zinc plate was conducted. Fig. 3 presents the SEM images of the conversion coatings. Table 1 lists the chemical composition of these coatings determined by EDS. The SEM morphology of the conversion coating with 60 minutes immersion time shown in Fig. 3(a) reveals that there are network-like wide cracks and discontinued bulk coating. The bulk coating contains 30.19 wt% phosphor verified by EDS in table 1, while the bottom of cracks contains 4.18 wt% phosphor. The conversion coating being immersed in phytic acid for 15 minutes has fewer and narrower cracks, and the phosphor weight percent of both coating and cracks is less too. There are no cracks on the conversion coating immersing for 1.5 minutes and the conversion coating contains 2.45 wt% phosphor. It can be seen that the conversion coating of 1.5 minutes is thin and may be self-assembled monolayer (SAM) [34,35,40]. The geometrical feature of the specimen surface could affect the current density distribution during the chemical reactions [41-45], thus influencing the corrosion rate. According to the detected data with ACM sensors shown in Fig. 2, the SAM of 1.5 minutes has the best protection capability.

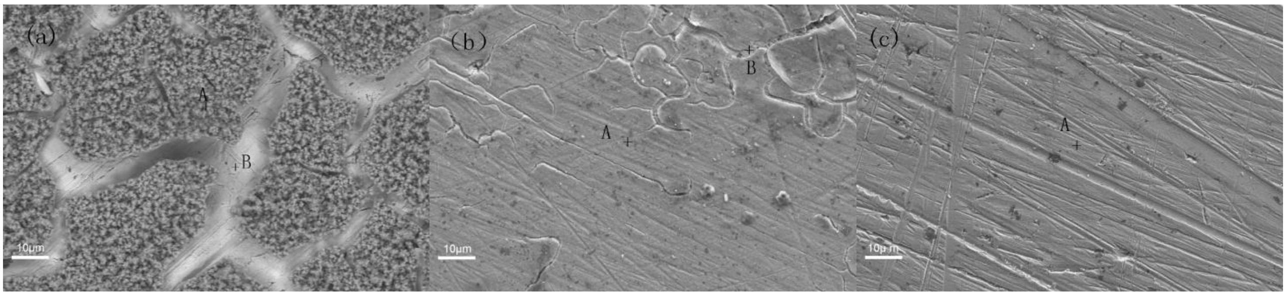

Figure 3. SEM images of conversion coating in phytic acid solution (pH=3) for different immersion time. a 60 min, b 15 min, c 1.5 min

Table 1. The content of various elements in the phytic acid coating for different time.

	(a)A	(a)B	(b)A	(b)B	(c)A
Element	Wt%	Wt%	Wt%	Wt%	Wt%
C K	8.66	6.73	3.03	7.27	5.19
O K	24.44	12.71	12.23	5.12	7.11
Si K	-	6.09	-	1.05	-
P K	30.19	4.18	6.59	1.56	2.45
K K	3.88	-	-	-	-
Ca K	1.13	-	-	-	-
Zn L	31.70	70.28	78.15	85.00	85.25
total	100.00	100.00	100.00	100.00	100.00

Fig. 4 shows characteristic surface potential images obtained with SKP when phytic acid droplet staying on zinc plate for 15 minutes and 1.5 minutes. The droplet can not be staying on zinc plate for more than 15 minutes for evaporation. The potential range between untreated area and coating area of zinc plate with phytic acid droplet staying for 15 minutes shown in Fig. 4(a) is 500 mv, while that with staying 1.5 minutes shown in Fig. 4(b) is about 300 mV. And there is a potential pit occupying a big percent of the potential peak area of the former. The pit is the place where the cathode reduction of proton took place. It can be concluded that the coating with 15 minutes has lower electrical activity than that with 1.5 minutes, and it has serious non uniformity, which lead to bad protection. Though the potential range of SAM coating with 1.5 minutes is little, the potential distribution is uniform, and the conversion coating has good protection.

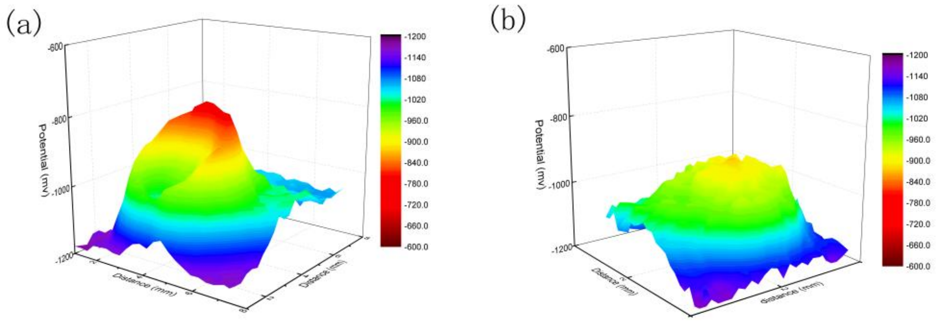

Figure 4. SKP images of zinc plate with phytic acid (pH=3) coating area for: (a) 15 min, (b) 1.5min.

3.1.2. Influence of pH of phytic acid solution

The detected data by ACM sensors with untreated and conversed zinc electrodes by different pH phytic acid solution for 1.5 minutes are shown in Fig. 5. It can be seen that the pH of phytic acid solution has effect on protection capability of conversion coating from the four lines in Fig. 5. The treated ACM sensor by phytic acid solution with pH=11 has the biggest galvanic current and the treated surface can’t give valid protection. The treated ACM sensor by phytic acid with pH=7 detected the smallest galvanic current. The conversion coating with near neutral phytic acid solution has the best protection, which is similar to the effect of phytic acid on magnesium alloy [46].

Fig. 6 shows the surface morphology of treated zinc plates under different pH values. The corresponding compositions are listed in table 2. The morphology and content of phosphor on conversion coating under pH=7 are similar to conversion coating under pH=3 with immersion time 1.5 minutes. There are many deep holes on the treated zinc surface under pH=11 and the phosphor does not been detected on it. A literature [47] has said the forming rate of conversion coating on magnesium alloy is low when phytic acid is alkalinity. Valid conversion coating has not been formed on zinc surface in 1.5 minutes.

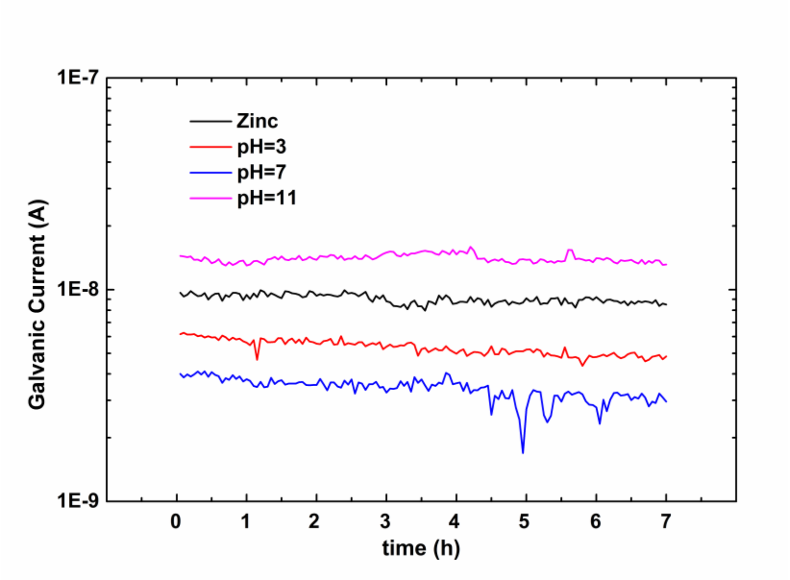

Figure 5. Galvanic current of ACM sensors were detected under different pH values.

But holes were formed on zinc surface when zinc being immersed in phytic acid under pH=11. It is concluded that zinc dissolved in some pots and uniformity conversion coating can't be formed on zinc surface. So, zinc does not be protected and has big corrosion rate. It is coincide with the result of Fig. 5.

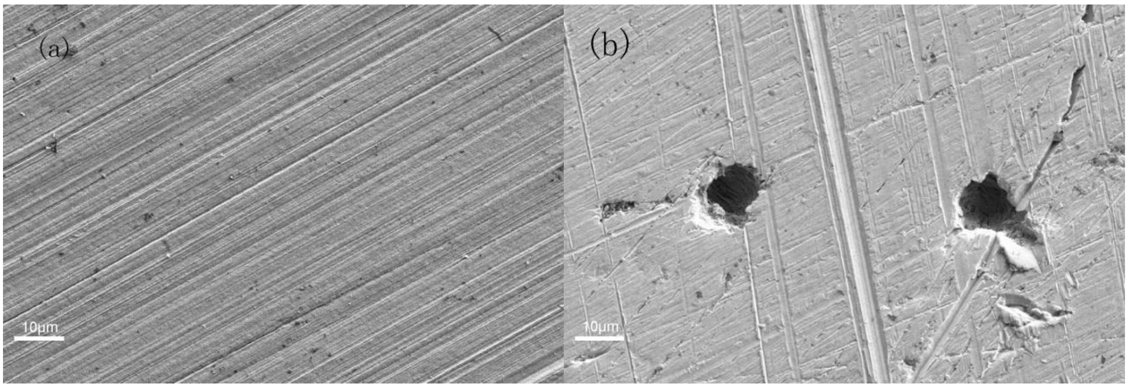

Figure 6. SEM images of conversion coating in phytic acid solution for 1.5 min at different pH.

Table 2. The content of various elements in the phytic acid coating at different pH.

	(a)	(b)
Element	Wt%	Wt%
C K	14.19	3.16
O K	9.59	4.75
Si K	33.31	-
P K	2.53	-
Zn L	40.38	92.10
total	100.00	100.00

In order to further know the effect of pH on electrochemical activity of phytic acid conversion coating, SKP was chosen to investigate phytic acid droplet treating surface of zinc plate. Fig. 7 shows the surface potential map of conversion coatings under different pH values. The potential range of conversion coating under pH=7 is about 300 mV shown in Fig. 7(a) which is similar to that under pH=3 with immersion time 1.5 minutes. But the former has no obvious potential pit on potential peak area. It means that there is more proton reduction processing of zinc in phytic acid solution under pH=3, which can form very thin defects. The potential range of conversion coating under pH=11 is about 200 mv shown in Fig. 7(b). It is the smallest one among those under different pH values. It can be seen that the conversion coating formed on zinc surface is too thin to improve corrosion resistance obviously. The more terrible thing is that there are a lot of holes where is the pitting corrosion processing on the conversion coating, which improve the corrosion rate of zinc. This is coincide with galvanic current in Fig. 5.

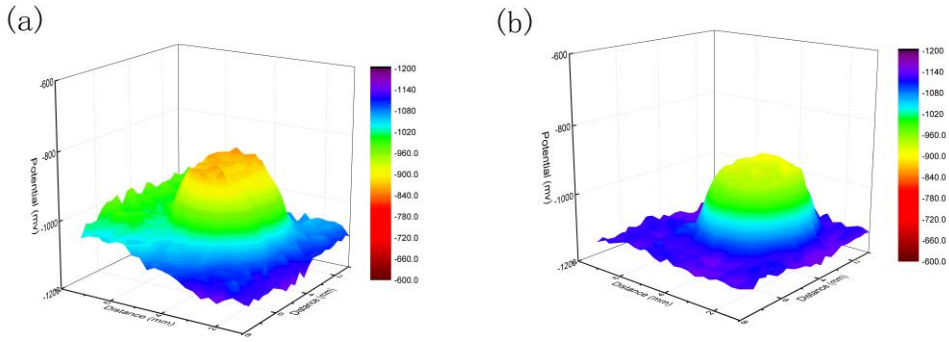

Figure 7. SKP images of zinc plate with phytic acid coating area for 1.5 min in different solution pH. a pH=7, b pH=11

3.2. Influence of ambient conditions on conversion coating behavior

3.2.1. Influence of ambient temperature

It is known that temperature is one of the main factors influencing the phytic acid coating formation on metals [48,49]. Temperature can influence the service of conversion coating. In order to invest it, ACM sensors with conversion coating were used to monitor the corrosion rate of zinc. The detected data is shown in Fig. 8 with different ambient temperature. The galvanic current at 50 ℃ is the biggest, and both of those at 10 ℃ and 30 ℃ are the same. Phytic acid can chelate metal cations and form stable compounds [26,27]. Furthermore, Liu et al. [48] found that the conversion coating formed in phytic acid solution at 60 ℃ could effectively improve the corrosion resistance of magnesium alloys. It means that zinc phytate is stable under 50 ℃ with the high galvanic current. Zinc phytate must have low bonding strength with matrix under 50 ℃ and there must be co-adoption water [24].

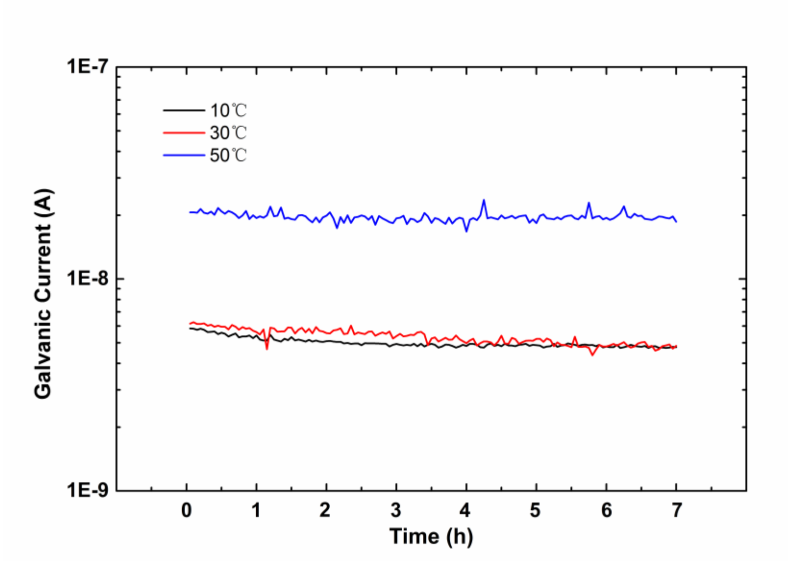

Figure 8. Galvanic current of ACM sensor, treated by phytic acid (pH=3), at different temperature.

3.2.2. Influence of relative humidity

Relative humidity is another environment factor influencing material corrosion rate. Fig. 9 shows the galvanic current detected by ACM sensors under different humidity. Both of the galvanic currents under 40% and 60% coincide and that under 80% is bigger. That means the performance of conversion coating on zinc under low humidity is unchanged. The highest galvanic current under 90% is about 2 orders of magnitude higher than the lowest one under 40% and 60%. The influence of relative humidity on corrosion resistance is higher than that of temperature, especially in high humidity value. In this condition, there is thick electrolyte film formed on the conversion coating. Zinc phytate is still a stable chelate compound, but the corrosion resistance of treated zinc is reduced. It is also relate to the low bonding strength of zinc phytate with matrix and co-adoption water, and the thick electrolyte film under high humidity enhances the corrosion rate.

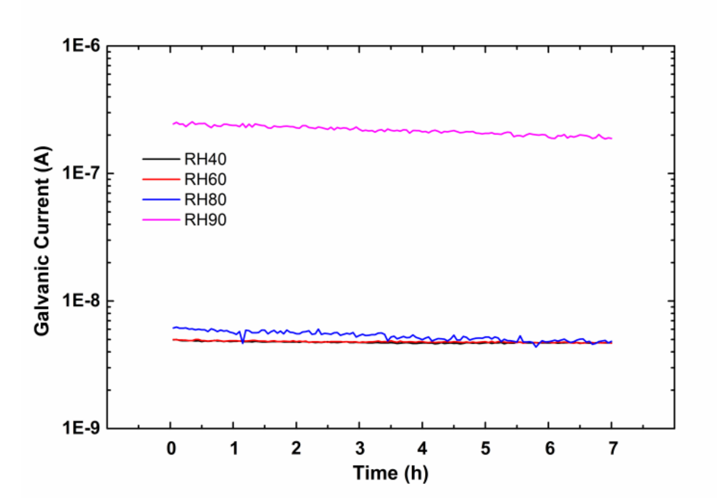

Figure 9. Galvanic current of ACM sensor, treated by phytic acid (pH=3), at different Relative humidity.

4. CONCLUSIONS

The present study attempts to use ACM in air to detect corrosion rate of conversion coating on zinc by immersing in phytic acid solution. SKP was used to investigate the difference between treated and untreated area on one zinc plate. The corrosion resistance of conversion coating on zinc will be weakened when extending immersion time and using alkalinity phytic acid solution. Cracks appeared and got bulky when prolonging the processing time by phytic acid. The uniformity conversion coating is being formed slowly and zinc is dissolved in pits by alkalinity phytic acid. There are many processing factors influencing the conversion coating and forming many kinds of conversion coatings, but only the zinc phytate SAM can provide effectively protection. The performance of conversion coating affected by ambient temperature and relative humidity was also investigated. High temperature or humidity reduces the protection ability of conversion coating on zinc. The zinc phytate bond strength with zinc matrix is the ultimate reason, and further study is needed for the mechanism.

ACKNOWLEDGEMENTS
This work was supported by the State Grid Corporation of China and Tianjin University–Liceram Joint Laboratory.

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