



## Evaluations of Potable Water Tanks Epoxy Coatings Performance Using Electrochemical Impedance Spectroscopic Method

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### Abstract

The aim of this paper is to investigate and discuss the mechanisms of corrosion of epoxy coatings used for potable water tanks. Two distinct types of Jotun epoxy coatings: Tankguard 412 contained polyamine cured epoxy and Penguard HB contained polyamide cured epoxy, were tested and studied using the electrochemical impedance spectroscopic (EIS) method. The porosity of epoxy coatings was determined using EIS method. The obtained results showed that the two epoxy coatings have excellent behavior when applied and tested in potable water of Basrah city. Polyamine is more resistance to water corrosion compared to polyamide curing epoxy and has high impedance values. Microscopic inspection after test showed that the polyamine cured epoxy has blistering while the surface of polyamide cured epoxy showed pitting and blisters.

**Keywords:** Corrosion mechanism , epoxy coats, porosity, potable water, , EIS method.

### 1. Introduction

Large tanks for storing potable water are spread in all locations of Iraq provinces. Storage tanks serve two major purposes, one is to provide storage volume and the other is to provide pressure to the distribution system [1]. Most tanks are built by welding stainless steel such as ASTM 304, AISI 316 and 316 L because it's inert and corrosion-resistant in nature makes it an ideal material for contact with potable drinking water[2].

Lining by epoxy coats are the most effective method used to protect water storage tanks plate from corrosion and have been a cost effective way of preventing leaks. One of the methods used to test the corrosion of the coatings is the electrochemical impedance spectroscopy (EIS) method. Various parameters effect on epoxy coating corrosion can be obtained using EIS test like impedance and porosity of coatings. Porosity is an important parameter that should be assessed to estimate their quality of coatings. Porosity of

the coating is strongly related to the corrosion resistance of coats [3].

A number of papers have been published on the potable water steel tank and coatings. Leidheiser [3] show that the general range of the impedance for good coating  $> 10^6$  ohm.cm<sup>2</sup> whereas poor coatings have impedance of  $< 10^6$  ohm.cm<sup>2</sup>. Tato et al. [4] developed an electrochemical procedure to find the porosity of coatings. They suggest that the porosity can be found by comparing the polarization resistance of the uncoated and coated substrate at constant potential. Ramirez et al. [5] studied corrosion protection of four types of epoxy coating used for navy steel using EIS method. The obtained results show that it is possible to get information concerning the disposal of corrosion under each coating. Mathiesen et al. [6] studied the effect of low chlorine of drinking water in corrosion of 316 steel and show that all corrosion process is a combined by microbial effect on corrosion rate. Havn et al. [7] study the effect of both chloride and temperature on corrosion process of steel 316L in fresh water. They showed that a different

type of corrosion occurs. Francis et al. [8] studied and discussed the selecting of alining used in potable water tanks. They showed that relining of tank bases using fiber glass matt embedded in solvent less epoxy fiber glass provide optimum strength for the corroded bases, and solvent less epoxy resin good protection to the steel base along with ease of applications. Doolittle [9] reviewed some of the long-life coating systems used as lining for the water tank exteriors and interiors. Then showed a relative comparison of salient application and performance properties. Duraisamy et al. [10] prepared and studied corrosion behaviors using EIS method the primer hybrid coating from epoxy resin pigmented and zinc phosphate cured with polyamide. Results indicate that the primer coating had good corrosion resistance. Sara et al. [11] studied using scanning electron microscopy technique the Beeswax-colophony blended coating for drinking water distribution tanks. They found that this blend could be considered as a novel green organic coating and have good corrosion barrier even in aggressive environments.

The current work aimed to evaluate the protecting performance against potable water of two types of epoxy coatings using electrochemical impedance spectroscopy method. Mechanism of corrosion process of epoxy coats in potable water was investigated and discussed in detail.

## 2. Experimental Procedure

### 2.1. Types of Epoxy Coatings

Epoxy coatings consist of two parts that are mixed prior to use in coating application to develop the characteristic required for coatings. These two parts are an epoxy resin which is cross-linked (base) and a curing agent (hardener). Two types of commercial epoxy coatings were used in this studies and one type used as primer undercoats. These coatings are:

A- Penguard Primer COMP. A, PM3192, is a two-pack epoxy primer based on a high molecular weight epoxy resin made by Jotun Henry Clark LTD, UK.

B- Tankguard 412, this is a two component solvent free polyamine cured epoxy coating. This type has thickness in the range 150 to 200  $\mu\text{m}$ .

C- Penguard HB is a high build, two pack polyamide epoxy coating based on epoxy resin with high molecular weight. The Film thickness in the range 80 to 150  $\mu\text{m}$ .

The two coats Tankguard 412 Penguard HB are made by Jotun Paints Company of Emirates LLC,

Sharjah, U.A.E. Refer to these two type of coats as: Tankguard 412 model A, Penguard HB model B.

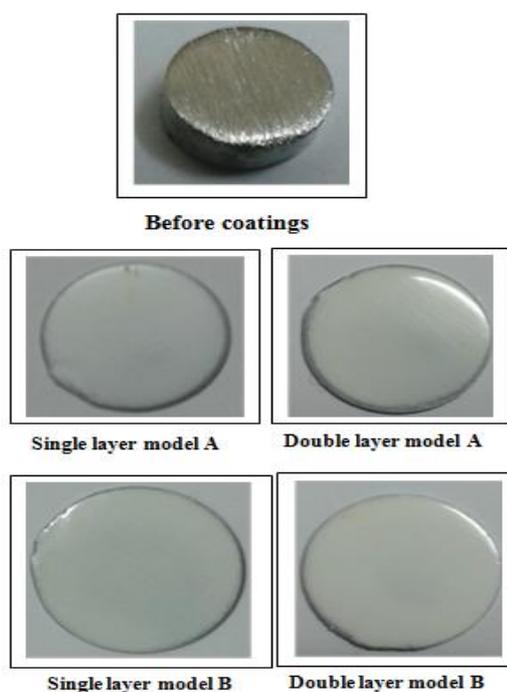
### 2.2. Specimen Preparation and Coats

The steel used to constriction of potable water tanks are those of the chromium-nickel-molybdenum, in particular the stainless steel grade AISI 316 (EN 1.4401), this types is suitable for water storage tanks and its available on the Iraqi market. The addition of a certain percent of molybdenum metal to this type improves the corrosion resistance. Table 1 shows the chemical composition of AISI 316 steel [12].

**Table 1,**  
The Percentages of the Constituent Elements of AISI 316 Steel [12].

Element	Mass %
Cr	0.08
C	0.08 max
Ni	10-14
N	0.1
Mn	2
Mo	2-3
Si	0.75
P	0.045

The tests samples have circular shape with diameter of 2 cm and thickness of 5mm. Figure 1 shows specimens before and after coats.



**Fig. 1. Photograph of Specimens Before and After Coats (Single and Double Layers) for EIS Test.**

For coating process, first, the primer layer was coat and then the two classes of Jotun coatings models A and B were applied by airless spray to steel surface at nominally standard dry film thicknesses as given by manufactured company. The main function of the primer was to maximize adhesion to the coating. The epoxy coating thickness is measured using paint thickness tester (Gain Express Holdings Ltd, China) to ensure the coatings thickness in range of uses. The thickness as average value of the single and double coating layers is 260  $\mu\text{m}$  and 375  $\mu\text{m}$  of the model A and B receptively. The epoxy coated specimens were left to cure for 15 days at 25°C and atmosphere pressure. A total 15 specimens were used for testing.

### 2.3. The Electrochemical Impedance Spectroscopic (EIS) Corrosion Test

Electrochemical Impedance Spectroscopy is considering one of the recent tools which were commonly used in the corrosion test of organic coatings. EIS used to determine the resistance and impendence of coatings and then determine the protective performances of coatings [13]. In EIS method, an AC potential was applied to an electrochemical cell. The current pass through the cell electrodes was measured and from this current the electrochemical impedance was obtained. A small excitation signal is usually used during EIS test.

Small amplitude of sinusoidal excitation signal with variable frequency is superimposed to the corrosion potential has the form [13]:

$$\Delta E = |\Delta E_o| \sin(\omega t) \quad \dots (1)$$

And

$$\omega = 2\pi f \quad \dots (2)$$

Where

f: The radial frequency in (Hz) of the applied signal.

E: Voltage of electrical resistance.

$E_o$ : Amplitude of the excitation signal.

t: Time in second.

This excitation induces a sinusoidal current  $\Delta I$  having  $\Delta I_o$  amplitude with a phase shift  $\phi$  with respect to the applied potential:

$$\Delta I = |\Delta I_o| \sin(\omega t - \phi) \quad \dots (3)$$

These values of a sinusoidal applied voltage  $\Delta E$  and a sinusoidal current  $\Delta I$  can be represented in the complex plane as:

$$\Delta U = \Delta U_{re} + i \Delta U_{im} \quad \dots (4)$$

$$\Delta I = \Delta I_{re} + i \Delta I_{im} \quad \dots (5)$$

At any frequency the impedance is complex number because  $\Delta I$  contains a phase shift  $\phi$  and defined as:

$$\Delta Z = \frac{\Delta E}{\Delta I} = Z_{re} + Z_{im} \quad \dots (6)$$

The impedance can be represented by a modulus  $|Z|$  and a phase angle shift  $\phi$ :

$$|Z| = \sqrt{Z_{re}^2 + Z_{im}^2} \quad \dots (7)$$

$$\tan \phi = \frac{Z_{im}}{Z_{re}} \quad \dots (8)$$

The impedance data can be represented by two methods:

-Nyquist plot: the imaginary part of impedance  $Z_{im}$  is plot as a function of real part  $Z_{re}$ .

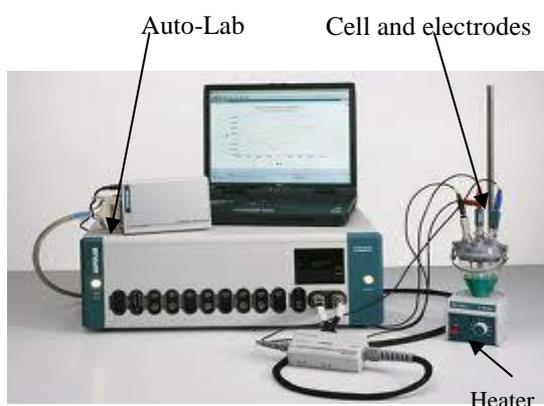
-Bode plot: the Absolut values of impedance ( $\log |Z|$ ) and phase angle  $\phi$  is plot as a function of  $\log$  frequency ( $\log f$ ).

EIS measurements were carried out on coated and uncoated samples exposed to potable drinking water, for periods up to 24 hr. and 25°C. The EIS corrosion test instrument consists from the AUTOLAB PSTAT 30 Potentiostat system computer control operated by the general purpose software (GPES) version 4.9 as shown in Fig.2. This EIS Apparatus foundat chem. Eng. Dept. of Tehran2 University of Iran. The AUTOLAB is connecting to glass cell (0.5L) have three electrodes with a coats specimen as the working electrode; a reference electrode contain silver/silver-chloride saturated solution and a concentric platinum ring as the counter electrode. The impedance measurements in all cases were carried out over a frequency range of 0.1 Hz to 500 Hz using 10mV amplitude of sinusoidal voltage in order to minimize the external interference on the system. The coating surfaces were macroscopic view after test using a computerize Carl Zeiss Jena imaging system Fig.3, provided by microscope and camera smart technical has resolution of 22 Mega pixels.

The materials compositions of Basrah potable water are analysis using the Spectrophotometer and shown in Table 2. The pH and TDS values of potable water tested determine laboratory and have a values of pH = 8.46 and TDS = 115 ppm respectively. The concentration of dissolved Oxygen determined not exceeds of 5 mg/L.

**Table 2,**  
**Materials Analysis of Basrah Liquefaction Water.**

Element	Concentrate
Cr	12 ppm
Cu	0.01 ppm
Ni	0.76 ppm
Al	0.19 ppm
Fe	0.2 ppm
Mn	4 ppm
Fl	1.3 ppm
Na	25 ppm
Cl	715.09 mg/l
Ca	30 mg/l
Mg	12 mg/l



**Fig. 2. EIS Apparatus Test.**



**Fig. 3. Imaging Devices of Simple's Surfaces.**

### 3. Evaluation the Porosity of Epoxy Coatings

Porosity are set of gaps in the coatings that can contain fluid (liquid or gas). Porosity distinguishes in two types: porous cracks and gaps. Among various techniques, the EIS method has been used because it provides quantitative

results via a simple electrochemical apparatus [14].

The Porosities (P %) of the coatings were calculated to assist in comparison and to determine the effective barrier properties of the coatings using the following formula [15] :

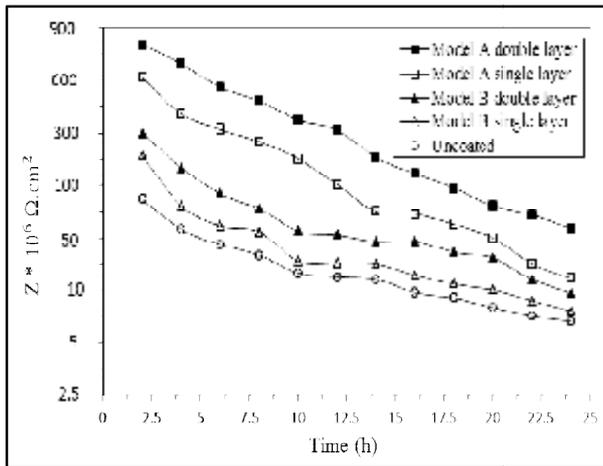
$$P = \frac{R_{uc}}{R_c} * 100\% \quad \dots (9)$$

$R_{uc}$  and  $R_c$  : The polarization resistance of the uncoated and coated metal substrate respectively.

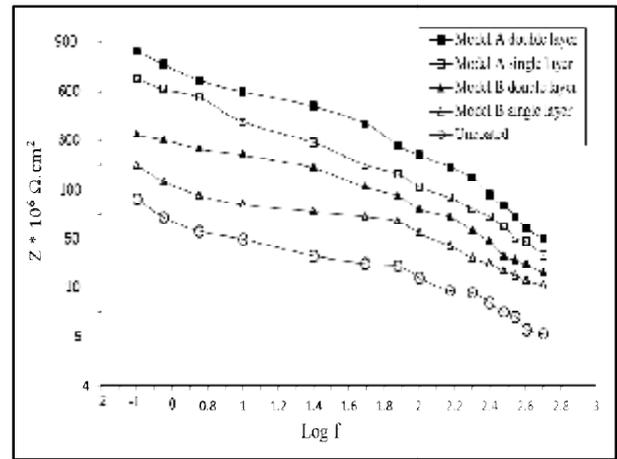
### 4. Results and Discussion

Figure 4 shows the variation of impedance (Z) of the uncoated AISI 316 stainless steel and coatings in case of single and double layers of two types of epoxy coats with time for 24 h of EIS test at frequency 0.1 Hz. As shown, there are continuous decreases in the impedance with time of immersion increased due to diffusion of destructive ions, water molecules and other atoms like chloride through the coating.

As shown the model A double layer initially has very high impedance. However, its impedance has small drops consistently with time, so this means that the corrosion protects and barrier of this coating was high through applications. Model A single layer and the two models of B show large increment at first period of test, this means a large water uptake in the film of coating. After 15 h of test all specimens shows very small decrease increments in impedance. It can be note that the form of the difference in impedance between two layers coats and single layer for model A very large, especially after 24 h of the test. The Z values are 55 and 13 \*10<sup>6</sup> Ω.cm<sup>2</sup> for the case of two layers and single layer for model A respectively. For the model B, the difference of Z between the state of the single and double layers is large at the beginning of the test and then decreases after 14 h after the start of the test. The value of Z of the state of the single layer is 7.8 \*10<sup>6</sup> Ω.cm<sup>2</sup>. While the 316 stainless steel showed Z value of 6.6\*10<sup>6</sup> Ω.cm<sup>2</sup> and the difference between them and the state of the single layer of model B is rather small.



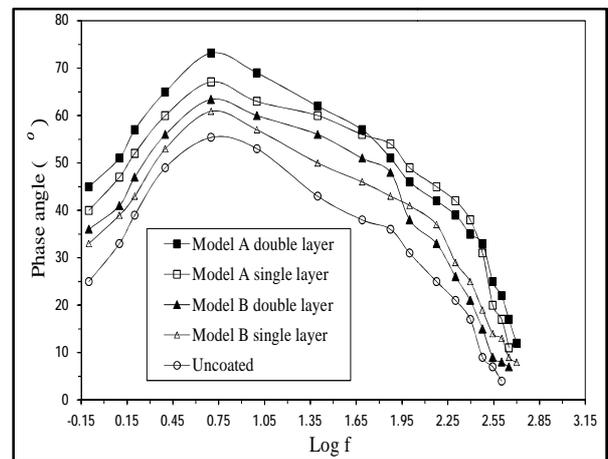
**Fig. 4. Impedance of Specimens of Uncoated Steel and Coats by Single and Double Layers of Two Models as a Function of Time at 0.1 Hz and 25°C.**



**Fig. 5. Comparison of Impedance of Uncoating, Single and Double Layer Coatings of the Two Models as a Function of log f at 25°C.**

Figures 5 and 6 show comparisons of the experiment Bode plot, i.e. logarithm of impedance and the phase angle as function of the logarithm of frequency for the two types of epoxy coats in case of single and double layers of the EIS test. As indicated in Fig.5, when the frequency increased drastically, that value of impedance decreases. This behavior could be due to the rigidity of the epoxy coatings. The Fig.5 consists from two parts, the high-frequency impedance behavior represents the coating characteristics and that of low-frequency part of impedance represents the corrosion reactions occurring at the coating surface.

The initial values of impedance in the low frequency range i.e. 0.1 Hz of the impedance spectra amounting for single and double layers of model A and B are 17.6, 116.5, 15.2 and 13.98 respectively. The value of impedance for ASTM 316 stainless steel is 11.64 at first of test of time of Fig. 5. The maximum phase angle occurred at approximately values 73.2, 67.1, 60.9 and 55.4 respectively. At a small frequency range with phase angles less than 35° could be observed in the Bode-phase diagrams. Despite these observations, phase angles were always smaller than 90°, which would be an indication of a resistance behavior. When the phase angle close to 90°, the sample tends to be have good physical barrier properties. This double layer coat of model A has good corrosion resistance properties compared to model B.

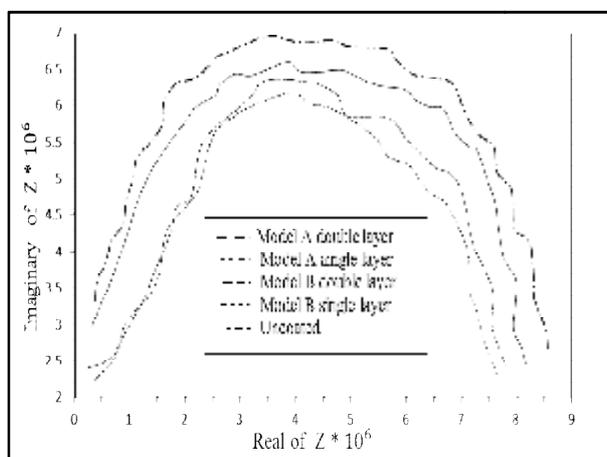


**Fig. 6. Phase Angle in Degrees of Uncoating, Single and Double Layer Coatings of the Two Models as a Function of log f at 25°C.**

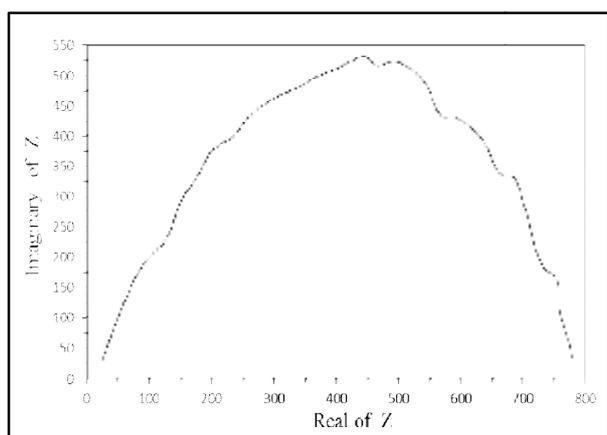
The polarization resistance  $R_p$  is useful as a quantitative parameter to identify the corrosion resistance of coats under certain circumstances. High  $R_p$  of coats means high corrosion resistance while low  $R_p$  means low corrosion resistance. The values of  $R_p$  of coats are determined from Nyquist plots. The Nyquist plots is obtained by plot the imaginary part of the impedance against the real parts and takes the characteristic of semicircle shape at lower frequency values as shown in Fig.7. Due to the nature of semicircle shape, the polarization resistance  $R_p$  is obtains as the point which the extension of semicircle touches the X-axis.

At the high frequency intercept, or close the plot origin, the real axis value gives the potable water resistance. The low frequency intercept of

the real axis provides a summation of the coats polarization resistance and the potable water solution resistance. Therefore, the semicircle diameter will equal to the polarization resistance. To find a point of touch of extension of semicircle with the X-axis from the Nyquist plots utilizing the data of Fig.7 and by using simple computerize curve fitting procedure by Microsoft axial 2010 programs to extrapolation of the relatively high frequency semicircular data to  $R_p$  at the zero frequency restrict. In the same method the polarization resistance of AISL 316 stainless steel was determined and shown in Fig.8. Then use of eq.9 to calculate the porosity of coatings. The results of porosity values, polarization resistance of coatings and uncoats AISL 316 steel are given in Table 3.



**Fig. 7. Nyquist Plot of the Imaginary vs. Real Impedance for the Uncoated and Coats Single and Double Layers of Two Types of Models at 25°C.**



**Fig. 8. Nyquist Plot of the Imaginary vs. Real Impedance for AISI 316 Stainless Steel at 25°C.**

**Table 3, Polarization Resistance and Porosity of the Two Models at 25°C.**

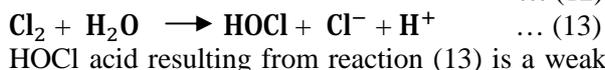
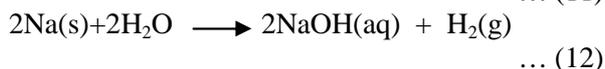
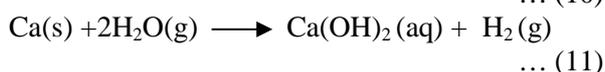
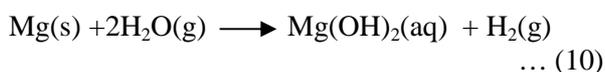
Cases resistance	Quantity	
	Polarization % ( $M\Omega \cdot cm^2$ )	Porosity
Uncoated	783	-----
Model A (Single layer)	8400	9.32
Model A (Double layer)	8750	8.95
Model B (Single layer)	7700	10.17
Model B (Double layer)	7870	9.95

From table 3, it's clear that the model A has lower degree of porosity in the case of single layer 9.32 % while that for model B is 10.17 %. The used of double layer coats reduce the porosity a small values for model B about 0.2 % and model A by about 0.37 %. The difference in polarizations resistance between single and double layers of two types of epoxy coatings is not very large, i.e. in model A  $350 M\Omega \cdot cm^2$  and in model B  $170 M\Omega \cdot cm^2$ . This attributed to that the thicker films of epoxy coats should allow greater amount of water to be absorbed and then increase the release electron from specimen surface during EIS test. The values of coating porosity have been related to the number of pores or capillary channels perpendicular to the substrate surface through which the electrolyte reaches the interface. The low porosity of the model A coating reduces the extent of surface charging, which may contribute to the observed increase in impedance of coats.

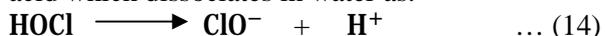
Polyamides curing epoxy coating layers are much more open in terms of their chemical structure because of their large distances between amine groups in the chemical chain. Consequently they are more flexible. This open structure of the curing agent's results in reduces coatings resistance to corrosion process. Polyamines curing epoxy coats are relatively small, low molecular weight compounds as compared with epoxy resins. Thus, the crossing over produced by the reaction of both components generates a three dimensional network that, after solvent

evaporation, settles down on the substrate forming a very smooth surface.

One of the important parameters affecting the corrosion rate of stainless steel and epoxy coats exposed to water is the deposition of soluble salts such as calcium, magnesium and chlorides. From table 2, the Basrah drinking water have various concentrations of calcium, magnesium and sodium salts. Calcium is dissolved in water and initiates limestone deposits, while the magnesium is dissolved and initiates calcium magnesium carbonate  $\text{CaMg}(\text{CO}_3)_2$ . Water that contains high calcium or magnesium salts is less corrosive, because the minerals tend to coat and protect the inside of interior tanks. Water contains sodium salts does not coat the plate of tanks and consequently is more corrosive. Reactions of calcium, magnesium, sodium and chloride with water are given by the following equations:



HOCl acid resulting from reaction (13) is a weak acid which dissociates in water as:



In all reaction above is accompanied by release hydrogen which increase pH values and increase corrosion rate of water.

Figs. 9 show the microscopic inspection of uncoated and coated specimens surface after 6 hr. of EIS test at 0.1 Hz. Fig. 9-B and C., Blisters was observed in the coating surface of both single and double layers of model A. The water absorption by epoxy coats leads to swelling of the coating and when this occurs locally, blisters may form and water may accumulate at the interface. These water accumulations are increased the charge transfer during test and reduced the impedance of coats. Water ions diffuse through coatings makes the coated surface metal in contact with the water. The steel substrate under blaster was lost. Adhesion losses at the metal-coats interface eventually occur, and the onset of metal corrosion reactions will happen at the buried metal surface. Bubbling is usually caused by the air pockets beneath the surface of the epoxy coat. During test, the air underneath coat expands and pushes the layer upward. Fig. 9-D and E, for single and double layer model B, it is appear that the coating surface have some pitting and blisters compared

to model A. The pits have black corrosion product mainly  $\text{Fe}_3\text{O}_4$ .

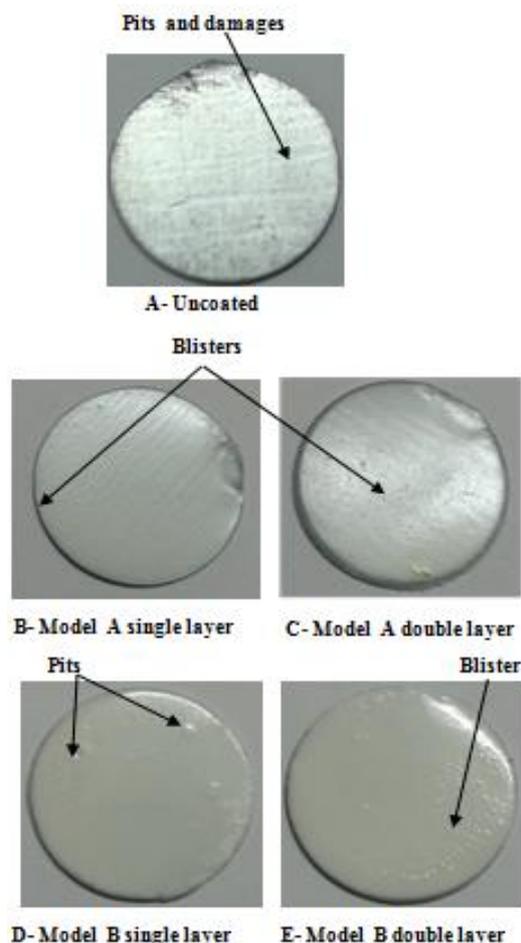
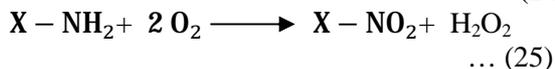
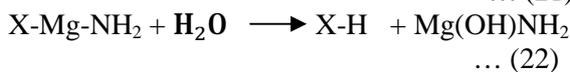
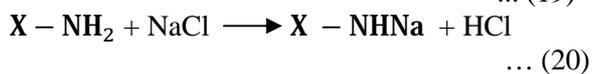
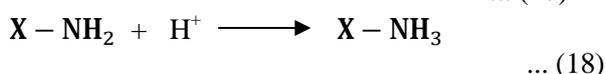
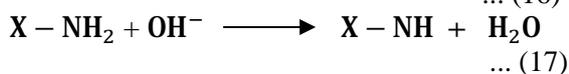
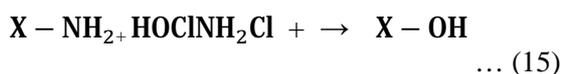


Fig. 9. Surface Morphologies of Specimens Before and After Coats Tested by EIS Method.

### Mechanism of Corrosion

The mechanism of corrosion suggested that the reaction occur between different compositions of water components and epoxy coats directly. This reaction accompanied by release of electrons to water solution and degradation of epoxy coats. The motivational force to corrosion reactions processes are voltages inflicted during EIS test and self-ions tendency to reactions. Fig.10 shows the mechanism of corrosion.

As indicated from Fig.10, water charged is transported through the coating and causes the corrosion process by two methods: convection through the pores and coating imperfections reaching to metal surface and by diffusion through the epoxy coats. The reaction between amines and water components can be represented by the following chemical reactions:



Where X is any function groups.

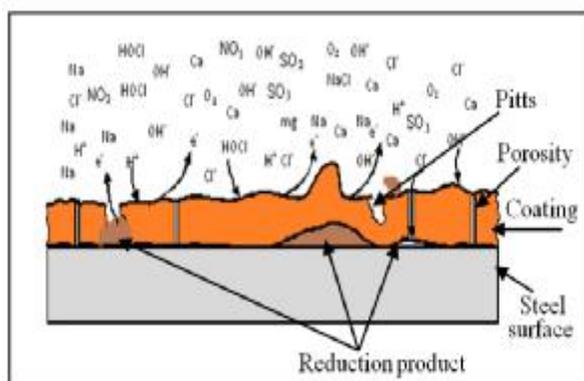
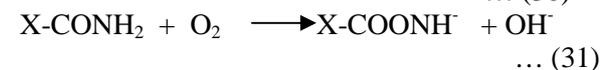
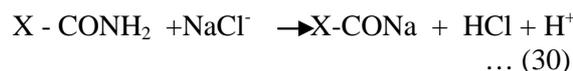
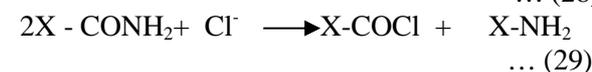
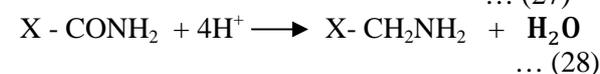
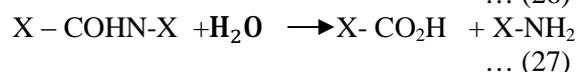
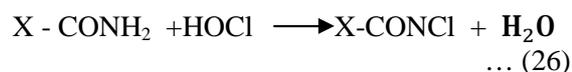


Fig. 10. Proposed Mechanism of Corrosion Process in Epoxy Coating.

The primary amine consists of a nitrogen atom linked to one alkyl group by a weak Van Der Waals bond and to the two hydrogen atoms by hydrogen bonds. Interaction with sodium hydroxide salt frees a strong HCl acid and this increases the pH of the potable water and increases the corrosion rate process. Reaction between amine and magnesium produce X-Mg-NH<sub>2</sub> which is unstable and dissolve in water to Mg(OH)NH<sub>2</sub>. Carbon dioxide dissolved gas reacts with epoxy coating and generates some of carbamate derivative which are unstable compounds which cause low epoxy coats adhesion with metal and water-spotting of coats.

Dissolved oxygen in potable water can destroy the protective coat and produced of oxidant ions but its insoluble forms. Eq.25 shows reaction between polyamine and oxygen and the reaction gives H<sub>2</sub>O<sub>2</sub> which further convert to hydroxide ion or radical oxygen in the water solution. The nitro compound X-NO<sub>2</sub> is analysis to X<sup>-</sup> and NO<sub>2</sub><sup>+</sup>.

Amide (-CONH<sub>2</sub>) is feeble base have a lone pair of electrons on the nitrogen atom. After exposed to water solution, the amide linkages are broken. The long chains which break and can eventually end up with the original monomers. This mechanism can be represented by the following equations:



The amide is hydrolysis in water and produces amine (weak base) and carboic acid (weak acid). These products are reacts and contribute to accelerate the corrosion rates.

## 5. Conclusions

Through the preceding discussion we can conclude the following points:

1. Polyamine curing epoxy is more resistance to water corrosion compared to polyamide curing epoxy and have high impedance values.
2. EIS tests of the two coatings show large change in impedance due to surface adsorption of water; this means that corrosion process appears to be initiated after test began directly.
3. Surfaces of specimens analyzed after the test revealed that the model A has blistering while model B specimens surface shows blisters and pitting.
4. The protection rate of epoxy coats is inversely proportional with coats porosities. The porosity of polyamide epoxy coats larger than polyamine epoxy coats, so it's less corrosion protection rate.

5. The mechanism of the coatings degradation depends on type and percent of impurities contents by water and coating compositions.

### Acknowledgement

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## تقويم أداء طلاء الايبوكسي لخزانات مياه الشرب باستخدام طريقة الممانعة الكهروكيميائية الطيفية

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### الخلاصة

الهدف من هذا البحث، تحقيق أليات التآكل لطلاء الايبوكسي المستخدم في طلاء خزانات المياه الصالحة للشرب ومناقشتها. هناك نوعان مختلفان من طلاء الايبوكسي لشركة جوتن هما : Tankguard 412 تحتوي على ايبوكسي البولي أمين و Penguard HB المحتوي على ايبوكسي البولي اميد تم اختبارها ودرستها باستخدام طريقة الممانعة الكهروكيميائية الطيفية (EIS). وتم تحديد المسامية لطلاءات الايبوكسي باستخدام طريقة EIS. أظهرت النتائج المستحصل عليها أن كلا الطلائين اظهرا أداء ممتاز عند تطبيقهما واختبارهما في المياه الصالحة للشرب من مدينة البصرة. لكن الطلاء المحتوي على البوليامين هو الأكثر مقاومة للتآكل مقارنة مع الطلاء الذي يحتوي مادة البولي اميد وامتلك أيضا قيمة اعلى من الممانعة. الفحص المجهرى بعد الاختبار بين أن السطح المطلي بطلاء الايبوكسي البوليامين اظهر بثور وحببيات على السطح بينما السطوح المطلية بطلاء مادة البولي اميد اظهر تنقرا وتجاويف على السطح.