Enhancement of Corrosion Protection of UV Curable Coatings in the Presence of Aliphatic and Aromatic Amine Compounds

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CORROSION inhibitors based on aromatic amine; 4-carboxy aniline (CA) and aliphatic amine; acrylamide (AA) were prepared. CA and AA amine compounds allowed reacting with epoxidized sunflower oil (ESO) at $130^{o}C$ for 3h to prepare amine adducts ESO/ CA and ESO/ AA, respectively. The adducts were characterized by IR-spectroscopy and oxirane content determination. Different ratio of prepared adduct were added in coating formulations based on epoxy acrylate oligomers to evaluate them as corrosion inhibitors on metal substrates under UV radiation. Corrosion resistance tests and wt loss values were measured for all cured films, in addition to the physical and mechanical measurements. It was found that the coating films containing CA have high resistance for corrosion of metal than AA and the optimum concentration is 0.5%.

Keywords: Corrosion inhibitors, coating, metal substrates, wt loss

Corrosion of metals is a major industrial problem that has attracted many investigators (Abiola and Oforka, 2002). Various attempts have been made to prevent the destructive effect of corrosion on metals and alloys. Organic coatings are employed in order to protect the steel structures against corrosion (Gonzlez-Garc *et al.,* 2007). Volatile organic compounds (VOCs) in polymeric coatings cause health hazards and toxicity problems (Wang *et al.,* 2007). To overcome such problems many acts and regulations have been imposed on paints and coatings industries (Bandres *et al.,* 2011 and Pirvu *et al.,* 2010) ensuring the use of eco-friendly materials, like vegetable oil based polymeric coatings (Boruah *et al.,* 2012), waterborne polymer coatings (Zafar *et al.,* 2008) and UV curable coatings (Xu *et al.,* 2012). UV curable coatings can be applied as a potential alternative for VOC producing solvent borne polymer coatings.

Curable coatings show other useful characteristics over conventional solvent borne polymers like rapid curing rate, solvent free, low energy requirement, excellent properties of products and good applicability. However, most of the organic coatings cannot provide long term corrosion protection properties due to their poor resistance against electrolyte diffusion. Water molecules diffuse into the coating/metal interface leading to the decrease of coating adhesion. Moreover, the coating deterioration may also occur as a result of electrolyte diffusion though coating porosities and defects. This would be responsible for the severe decrease in the coatings barrier properties (Liu *et al.,* 2009 and Ramezanzadeh *et al.,* 2011). Therefore, different studies have been performed in order to find proper methods to improve the anticorrosion properties of the organic coatings. In the past, chromates were the most commonly used inhibitive pigments. However, due to their high toxicity, many studies have been devoted to the development of more environmentally acceptable organic coatings. One of the most convenient and economic methods was the use of inhibitors, which can slow down corrosion rate or protect metal from corrupting, though used in a very small amount (Obot and Obi-Egbedi 2010 and Sherif *et al.,* 2010).

There are so many different kinds of organic corrosion inhibitors, such as imidazoline, amino acids, pyridine, alkynol, amides and thiourea (Bobina *et al.,* 2013, Kosari *et al.,* 2014, Sadeghi Meresht *et al.,* 2012 and Yan *et al.,* 2013). Most of organic inhibitors contain nitrogen, sulphur and oxygen heteroatoms, which can be adsorbed on the metal surface because they are able to establish coordinative interactions between unshared electron pairs in the molecule with the metal (Ashassi-Sorkhabi *et al.,* 2006, Tang *et al.,* 2013 and Qian *et al.,* 2013). P-aminoazobenzene derivatives and sulphidated poly (acrylamide-vinyl acetate), as a new corrosion inhibitors for carbon steel in acidic media were studied by some authors (Noor El-Din and Khamis, 2015 and Shihab and Al-Doori, 2014). Although many heterocyclic compounds have been successfully used as corrosion inhibitors in several metallic systems, most of them were toxicity and non-biodegradable (Eddy and Mamza, 2009). With the trends of environmental protection, researches were focused on the environmentally friendly corrosion inhibitors. Natural oils are considered to be one of the most important classes of renewable resource which were non-toxic, relatively cheap and easily available. They can be obtained from naturally occurring plants, such as sunflower, cotton, linseed.

The aim of the present study was to prepare and evaluate the corrosion inhibitors based on reaction of environment friendly ESO with AA and CA. Different amounts of prepared inhibitors were added in UV curable coating formulations based on epoxy acrylate oilgomer. The coating formulations were applied on a steel substrates and the behaviour of the steel/coating interface was characterized by corrosion resistance tests and wt loss measurements.
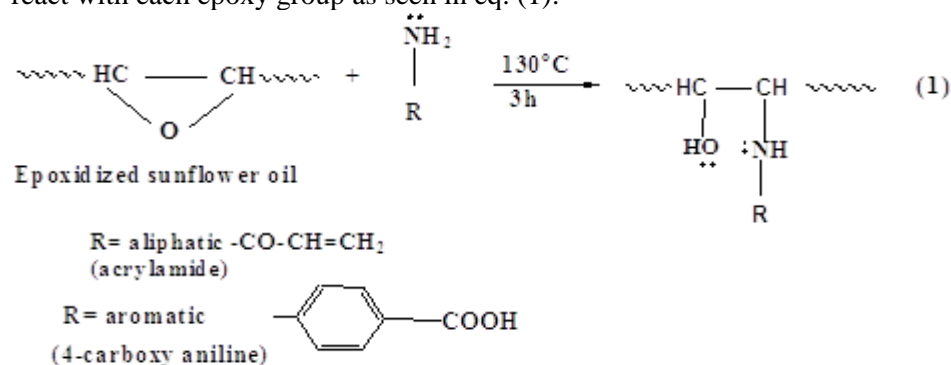
Experimental

Materials

Mild steel panels (80mm× 70mm× 2mm) were provided from the Iron and Steel Co. (Egypt). The steel sample has the following composition (wt %): Fe (97.7), C (0.19), Si (0.415), Mn (1.39), P (<0.005), S (<0.005), Cr (0.026), Mo (0.018), Co (0.429) and Cu (0.0481). Samples were polished with sand papers of 600, 800 and 12,000 grades followed by acetone degreasing. Epoxy acrylate (EA) under the trade name EBECRYL 645 and 2- hydroxy ethyl methacrylate (HEMA) were obtained from Cytec Surface Specialties (Drogenbos, Belgium). CA and AA were supplied from Sigma-Aldrich Chemie Gmbh Munich, Germany. ESO having oxirane oxygen content 6% and Castor oil was delivered from Paint and Chemical Industry (Egypt). Benzophenone as a photoinitiator was offered by Ciba Speciality Chemicals.

Techniques

Preparation of the aliphatic and aromatic amine/ ESO adducts

The reaction of aliphatic and aromatic amine with ESO was carried out in sealed ampoules under an inert atmosphere at 130°C for 3h. The amounts of amine added to ESO were calculated on the basis that one mole of amine would react with each epoxy group as seen in eq. (1):

R= aliphatic -CO-CH=CH$_2$
(acrylamide)

R= aromatic
(4-carboxy aniline)

Characterization of the prepared adducts

The oxirane oxygen content was measured for both ESO and the prepared adduct. The sample was titrated against 0.1N HBr in a glacial acetic acid solution in the presence of crystal violet indicator until a bluish green end point. The oxirane oxygen content was determined using equation below:

$$\text{Oxirane oxygen content (\%)} = LN \times 1.6/ W$$

Where, L is the volume of HBr solution, N is the normality of HBr solution and W is the wt of the sample.

Fourier transform infrared spectrometry (FT-IR) was used for scanning and measuring the transmittance spectra of the prepared adducts at a resolution of $4cm^{-1}$.

Preparation and curing of formulations by UV irradiation

Eleven coating formulations (L1–L11) were prepared using EA as binder 70%, HEMA as diluent 20%, castor oil as plasticizer 7% and benzophenone as initiator 3%. L1 was blank and free from any prepared adducts. The formulations L2–L11 were prepared with the incorporation of different concentrations of the prepared CA and AA/ ESO adducts. The formulations were applied as a thin film on different substrates, such as glass, tin, and carbon steel metal, with a film applicator (100μm) according to ASTM D 823-07. The curing of the coating was carried out by irradiation standard UV lamp (Type: EMITA VP-60 (made in Poland), 180W mercury lamp, 220V, 50Hz and Monochromatic filter (λ= 320nm) were used to provide the required irradiation wavelength). In this study the UV irradiation was done at a constant distance (10cm) for various times intervals in the path of UV lamp with dose rate= 23.7 KJ/ m^2.

Testing and evaluation

The formulated coatings were tested and evaluated according to well-known standard methods. The measurement of film hardness was carried out with a Wolff-Wilborn pencil hardness tester according to ASTM D 3363 (2000). Bending tests were done according to ASTM D 522-93a with 2 mm diameter mandrels. The adhesion of the cured films was measured according to ASTM D 3359-97. Alkali- and acid-resistance tests were carried out according to ASTM D 1647-89 and ASTM B 287-74, respectively. Corrosion scratch tests

were carried out according to ASTM D 1654-92 (2000). Blister-resistance tests were carried out according to ASTM D 714-87 (2000). Rusting was determined according to ASTM D 610-01. The wt loss of coated steel was measured according to ASTM D 2688-94 (1999).

Results and discussion

Preparation and characterization of the prepared aromatic adducts

FT-IR spectra of ESO, ESO/ CA and ESO/ AA interaction are shown in Fig. 1. The IR spectrum of ESO showed a characteristic peaks at 846 and 1742cm^{-1} are attributed to the epoxy group and C=O group, respectively, as shown in Fig. 1. Also, a strong band at 2916cm^{-1} due to aliphatic C-H attached to the ester group was present.
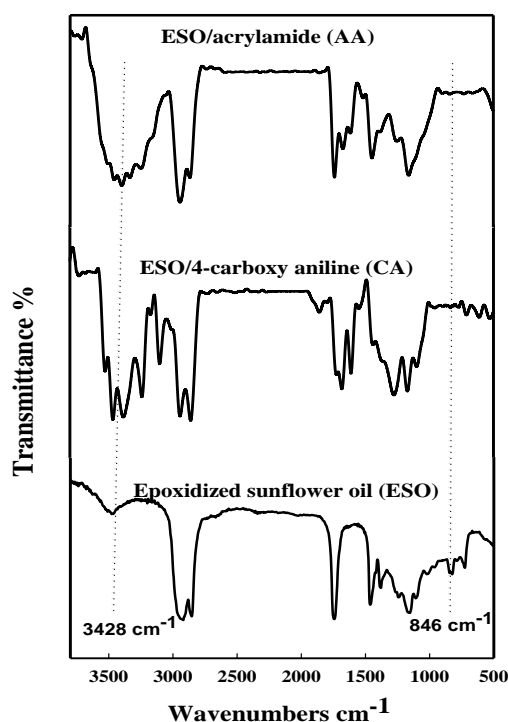

Fig. 1. IR spectra of ESO, ESO/CA and ESO/AA.

IR chart spectrum of the prepared aromatic and aliphatic amine adducts, which are shown in Fig. 1, proved that the epoxy group of the ESO was consumed during the reaction with the CA and AA, where the band of the

epoxy group, which appeared at 846cm^{-1}, had completely diminished. Moreover, new characteristic bands of secondary amino groups (one band) and hydroxyl groups appeared; they appeared as an overlapped broad peak at 3200-3500cm^{-1}. In addition, a characteristic band near 1860cm^{-1} due to p-substitution and a band of C-N appeared at 1180cm^{-1} for ESO/ CA and strong band at 1670cm^{-1} due to aliphatic C=C and a band of C-N appeared at 1186cm^{-1} for ESO/ AA. These observations indicated that the epoxy/ amine reactions took place successfully. On the other hand, the oxirane oxygen contents of the ESO and their reaction products with CA and AA were estimated, and the obtained results are given in Table 1. It was found that the oxirane oxygen content of ESO/ CA and ESO/ AA adducts had a low value compared to the 6% per mol found in the starting material ESO. This means that almost all of the oxirane oxygen (>99%) participated in the reaction.

TABLE 1. Oxirane oxygen content (mol %) of the prepared inhibitors.

Prepared inhibitors	Index	Oxirane oxygen content (mol %)
Epoxidized sunflower oil	ESO	6
4-carboxy aniline adduct	ESO/ CA	0.11
acryalmide adduct	ESO/ AA	0.18

Group 1: Anticorrosion performance evaluation of ESO/ CA adduct

The aim of this application was to study the efficiency of the prepared aromatic (ESO/CA) and aliphatic amine (ESO/AA) adducts as corrosion inhibitors for carbon steel and to select the optimum concentration of the prepared amine adducts with the best performance as a corrosion inhibitor. To evaluate ESO/CA as corrosion inhibitor, six coating formulations based on EA 70%, HEMA 20%, castor oil 7% and benzophenone 3% were prepared, in which formula L1 was selected as a blank. Different concentrations of ESO/CA adduct (0.1-0.9g) were added to coating formulations (L2-L6) as shown in Table 2. It was found that, no changes in the physico-mechanical properties of cured films in all formulations as a result of inhibitor addition. Where, all of the cured films of the investigated formulations showed good adhesion (4B) and excellent flexibility (bending). Very low decreases in the hardness (from 6H to 5H) of the cured films were observed with increasing concentration of inhibitors compared to the blank (6H). In addition, all of the coated glass panels had very good chemical resistance in acid, alkali, and water for 24h without considerable defects.

TABLE 2. Composition and properties of coating formula of group 1.

Composition and properties	Blank	Group 1				
	L1	L2	L3	L4	L5	L6
Epoxy acrylate EA (%)	70	70	70	70	70	70
HEMA (%)	20	20	20	20	20	20
Castor oil (%)	7	7	7	7	7	7
Benzophenone (%)	3	3	3	3	3	3
ESO/ CA adduct (g)	-	0.1	0.3	0.5	0.7	0.9
Hardness*	6H	6H	6H	6H	5H	5H
Adhesion**	4B	4B	4B	4B	4B	4B
Bending (2mm mandrel)	pass	pass	pass	pass	pass	pass
Acid, alkali, water resistance	v.g.***	v.g.	v.g.	v.g.	v.g.	v.g.

*Lead pencils supplied with the unit, softest to hardest, are as follows:

9B, 8B, 7B, 6B, 5B, 4B, 3B, 2B, B, HB, H, 2H, 3H, 4H, 5H, 6H, 7H, 8H, 9H

**The adhesion of the cured films decreases in the following descending.   order: 5B> 4B> 3B> 2B> B.

***Very good.

The corrosion test results of the coated steel panels of the group 1 after immersion in artificial seawater up to 28 days are provided in Table 3. and represented photographically in Fig. 2 for the different concentrations of ESO/CA inhibitor.

Table 3. and Fig. 2. show that the coating formulation L4 which contains 0.5 g of ESO/CA give the best corrosion protection for steel, where no sign of blisters (rating 10) with a little degree of rusting (rating 9) were observed, and the adhesion of the cured film around the scribe was unaffected (rating 10). More than 0.5g, the corrosion will appear with some loss of adhesion of the cured films around the scribe.

TABLE 3. Corrosion resistance tests of the coated steel panels of group 1.

Test	Blank	Group 1				
	L1	L2	L3	L4	L5	L6
Degree of rusting[1]	4	7	8	9	7	6
Degree of blistering[2]	8F	10	10	10	10	10
Failure at scribe[3]	8	8	9	10	9	8

1- Rating of rust as area percentage; it is graded on a scale from 10 to 0, where 10 < 0.01%   and 0, greater than 50%.

2- Graded on a scale from 10 to 0, where 10 no blistering and 0 for largest blisters and frequency denoted by F, M, MD, and D (few, medium, medium dense and dense).

3- Rating of corrosion or losing of paint extending from the scribe; it is graded on a scale from 10, no creepage to 0, > 16 mm.
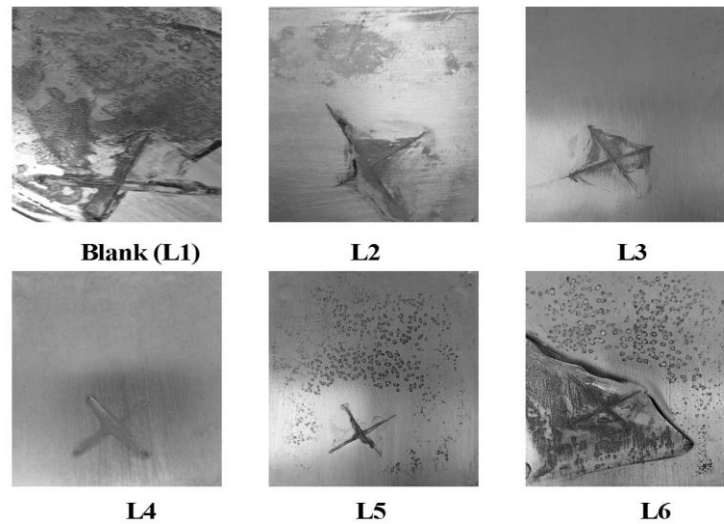
Fig. 2. Corrosion of metal panels coated with formulations of group 1 after 28 days of immersion time.

The wt loss results of the coated steel as a function of immersion interval times in artificial seawater for 60 days were determined and are plotted in Fig. 3.
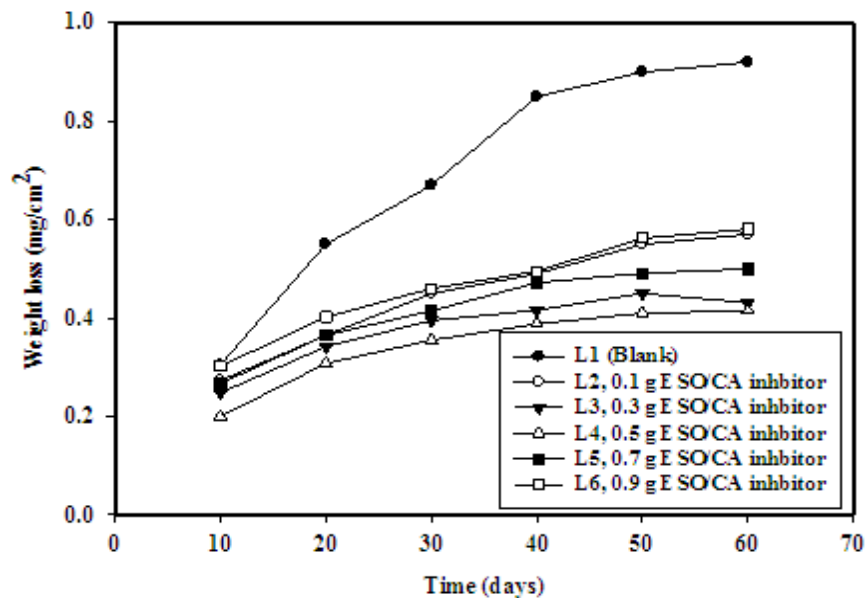

Fig. 3. The wt loss values of the coated metal plates of group 1 during immersion in artificial seawater.

It was evident that the wt loss results were consistent with observations of the corrosion resistance results of group 1, as illustrated in Fig. 3. It can be seen from this figure that the coated steel panels with formulations L3 and L4, which contained 0.3 and 0.5g of the ESO/ CA inhibitor, showed the lowest wt losses, 0.431 and 0.415mg/cm^2, respectively, after 60 days of immersion in artificial seawater. On the other hand, formulations L5 and L6 showed relatively higher wt losses with time.

Anticorrosion performance evaluation of ESO/ AA adduct

In the same trend of the previous group, six coating formulations, in which formula L1 was selected as a blank, were prepared. The prepared ESO/AA adduct was added in different concentrations (0.1-0.9g) with regular increments of 0.2g to the blank. The physico-mechanical and chemical properties of cured films were not affected significantly with the addition of the prepared ESO/AA inhibitor.

All cured films showed high adhesion and excellent flexibility (bending). Moreover, the hardness of the cured films decreased very slightly with increasing concentration of the prepared inhibitor (from 6H to 5H). Also, all of the prepared cured films passed the acid-, alkali-, and water-resistance tests.

The corrosion resistance tests results of the immersed cured films for 28 days are given in Table 4 and represented photographically in Fig. 4. The results show a significantly superior performance of coating formula L9, which contained 0.5g of the ESO/ AA inhibitor and in which a bright metal surface with a very slight degree of rusting (rating 9) was noticed, and the adhesion of the cured film around the scribe was affected in a little degree (rating 9). Moreover, no sign of blisters on the cured films (rating 10) was observed. For the coating formulas L10 and L11 which contain beyond the optimum concentration, the corrosion and failure around the scribe were appeared.

TABLE 4. Corrosion resistance tests of the coated steel panels of group 2.

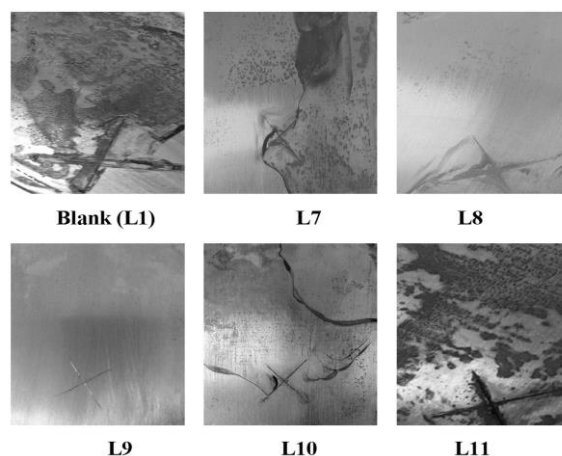| Test	Blank	Group 2				
	L1	L7	L8	L9	L10	L11
Degree of rusting	4	7	8	9	6	5
Degree of blistering	8F	10	10	10	10	10
Failure at scribe	8	8	9	9	8	7

Fig. 4. Corrosion of metal panels coated with formulations of group (2) after 28 days of immersion time.

With regard to wt loss measurements as a function of immersion time for 60 days, it was evident that the wt loss results were parallel with the obtained corrosion resistant results of group 2, as illustrated in Fig. 5. On the basis of this finding, we concluded that the coated steel panels with formulations L8 and L9, which contained 0.3 and 0.5g of the ESO/ AA inhibitor, showed the lowest wt losses, 0.430 and 0.426mg/ cm^2, respectively, and give the best corrosion protection for carbon steel. On the other hand, the wt loss values started to increase with higher inhibitor concentration, especially in formula L10 and L11.

On the other hand, the wt loss values of the ESO/CA and ESO/AA at optimum concentration (0.5g) after immersion 60 days can be illustrated in Fig. 6. It can be seen from this figure that the prepared ESO/CA adduct was the best corrosion inhibitor which have the lowest wt loss value than ESO/AA adduct.

The results of the previous two groups may be explained as follows:

1- At optimum concentration of the prepared inhibitor (0.5g), inhibitor molecules may physically or chemically be adsorbed on the corroding metal surface forming a layer that functions as a barrier, thus protecting the metal from corrosion (Bouklah *et al.,* 2006). It has been commonly recognized that an organic inhibitor usually promotes the formation of a chelate on a metal surface by transferring electrons from the organic compound to the metal and forming a coordinate covalent bond during the chemical adsorption (Ajmal *et al.,* 1994).
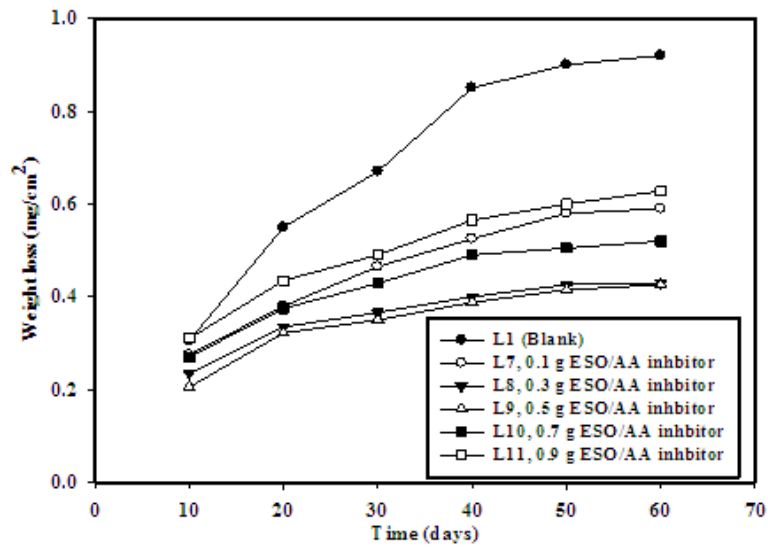
Fig. 5. The Wt loss values of the coated metal plates of group 2 during immersion in artificial seawater.

2- By increasing inhibitor concentration beyond the optimum level, the corrosion inhibition decreased because of the random distribution of the prepared inhibitor, which may have bloomed to the surface and attracted water molecules to the cured film through its hydrophilic groups.
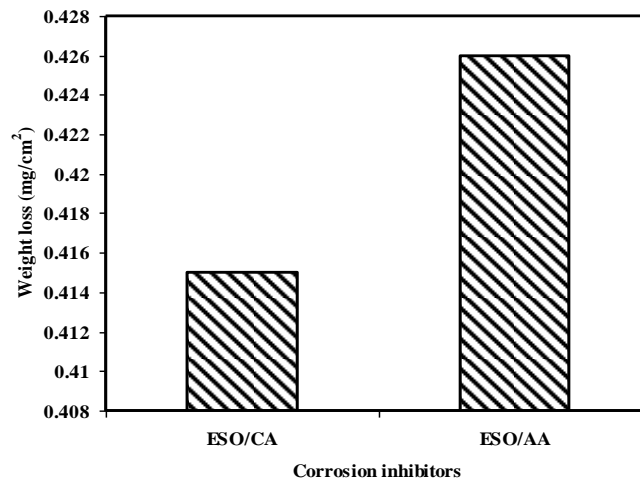

Fig. 6. Wt loss values of the coated metal panels of ESO/ CA and ESO/ AA adducts at 60 days artificial sea water.

Corrosion-inhibition efficiency of the prepared inhibitors

The corrosion-inhibition efficiencies (I's) of the curable coating formulations containing the prepared inhibitors were calculated from eq. (2), and the obtained results are represented graphically in Fig. 7.

$$I\,(\%) = \ 100\,[(W_o - W_i)/\,W_o] \qquad (2)$$

Where W_o and W_i are the wt loss values in the absence and presence of the prepared inhibitor after 60 days, respectively.

The obtained results prove that the formulation containing 0.5g of the ESO/ CA inhibitor played important role on the enhancement of the corrosion protection properties of the epoxy coating on the steel surface than ESO/AA inhibitor and this is due to the presence of high electron density on the nitrogen atom of amino group through resonance process in the benzene ring of CA than resonance in AA. The higher electron density in CA leading to increase the coordinate bond strength through chemisorption between the prepared ESO/CA inhibitor and the metal surface which, in turn, increased the inhibition efficiency of the ESO/CA inhibitor than ESO/AA inhibitor.
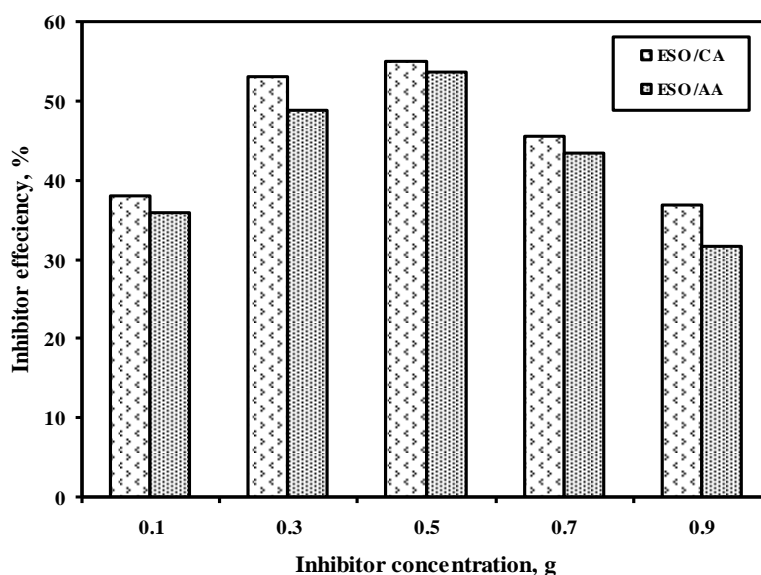

Fig. 7. Efficiency of the prepared two inhibitors depending on their concentrations in curable formulations

Conclusion

Two corrosion inhibitors based on aliphatic amine AA and aromatic amine CA were prepared via reaction with ESO at 130°c for 3h. The two inhibitors were added in different concentrations (0.1-0.9g) in epoxy acrylate formulations under UV-irradiation. The results showed that the physico-mechanical and chemical properties of coating films were not affected significantly with the addition of the prepared inhibitors and the concentration (0.5g) gave the best corrosion protection for carbon steel for two inhibitors. It was found that the efficiency of prepared ESO/ CA inhibitor higher than prepared ESO/ AA inhibitor for protection of steel surface against corrosion.

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تحسين خواص الحماية من تآكل الدهانات على سطوح الحديد و المعالجــة بالاشـعة الفـوق البنفسـجية فـى وجـود مركبـات اليفاتية وأروماتية

عيسى محمد موسى ، و حمدى راضى

قسم الكيمياء الاشعاعية ، المركز القومى لبحوث وتكنولوجيا الاشعاع ، ص.ب. ٢٩ مدينة نصر ، مصر.

تم تحضير مركبات اليفاتية و أروماتية ذات وزن جزيئ عالى بتفاعل زيت عباد الشمس المبوكسد و مركب الاكريلاميد و مركب ٤ـ كاربوكسى أنيلين كلا على حده عند درجه حرارة قدرها ١٣٠م° و لمده ٣ ساعات. تم التاكد مـن التفاعـل السـابق بقيـاس كـلا مـن الاشـعة تحـت الحمـراء و المحتـوى الايبوكسى للمركبات المحضرة. تمت إضافة تركيزات مختلفة من المركبات المحضرة فى خلطات دهانات من الايبوكسى أكريلات ومعالجتها بالاشعة الفوق البنفسجية لتقيمهـا كمـواد مانعـة للتآكـل و أيضـا الحصـول علـى أفضل تركيز للمركبات المحضرة.

و قد أظهرت النتائج ان إضافة هذ المركبات لايؤثر على الخواص الكيميائية و الميكانيكية للدهان و أن أفضل تركيز للمركبات المحضرة كانت ٠.٥ جم. و قد وجد أن كفاءة حماية سطوح الحديد من التآكل فى وجود مركب ٤ـ كاربوكسى أنيلين أفضل من مركب الاكريلاميد.