Self Assembling Mono Layers of Nano Films by Oxalic Acid

P.Satyabama [1], P.DesiyaKiruban[2], S.Rajendran[3]

[1], Visting faculty, Department of chemistry, University College of Engineering –Dindigul, Dindigul-624622, Tamilnadu, India.

[2] , Department of civil engineering, University College of Engineering –Dindigul, Dindigul, Tamilnadu, India

[3.]Corrosion Research Centre, Department of Chemistry, RVS School of Engineering and Technology, Dindigul - 624005,Tamilnadu, India.

Abstract

The inhibition efficiency of oxalic acid in controlling corrosion of Aluminium in an aqueous solution at pH 10, in the absence and presence of Zn^{2+} has been evaluated by weight loss method. The formulation consisting of 250 ppm of oxalic acid and 50 ppm of Zn^{2+} has 95% corrosion inhibition efficiency. A synergistic effect exits between oxalic acid and Zn^{2+}. The mechanistic aspects of corrosion inhibition have been evaluated by polarization study and AC impedance spectra. In the presence of inhibitor, linear polarization resistance increases;corrosion current decreases; charge transfer resistance value increases; impedance value increases and double large capacitance decreases . The protective film has been analyzed by UV and Flouresence spectra.

Keywords: *Corrosion inhibition, Aluminium at pH10, oxalic acid, synergistic effect*

1. INTRODUCTION

Aluminum is one of the metals which used in different human activities and many of important applications, where it isthe second most abundant metal after iron, it has a low atomic mass and negative value of standard electron potential, aluminum potentially attracts as an anodic material for power sources with high energy density. It is used in construction, packing and transportation because of its strength and electrical conductivity. Aluminum is used in electronics due to it is super purity [1]Aluminum and its alloys have a remarkable economic and attractive materials for engineering applications owing to its low cost, light weight, high thermal and electrical conductivity. The interest of the materials arises from their importance in recent civilization. Inhibition of metal corrosion by organic compounds is a result of adsorption of organic molecules or ions at the metal surface forming a protective layer. This layer reduces or prevents corrosion of the metal. The extent of adsorption depends on the nature of the metal, the metal surface condition, the mode of adsorption, the chemical structure of the inhibitor, and the type of corrosion media[2]. To prevent the attack of acid, it is very important to add a corrosion inhibitor to decrease the rate of Al dissolution in such solutions. Thus,many studies concerning the inhibition of Al corrosion using organic substances are conducted inacidic and basic solutions [3-7].

The present work is undertaken

i. To evaluate the inhibition efficiency of oxalic acid in controlling corrosion of aluminium immersed in an aqueous solution at pH 10, in the absence and presence of Zn^{2+} using the mass loss method

ii. To study the mechanistic aspects of corrosion inhibition by potentiodynamic polarization and AC impedance spectra

iii. To analyze the protective film by UV-visible absorption spectroscopy and Fluorescence spectra

II. METHODS AND MATERIALS

A. Preparation of specimens

Commercial aluminium specimens of dimensions 1.0 x 4.0 x 0.2cm, containing 95% pure aluminium were polished to mirror finish, degreased with trichloroethylene, and used for the Mass-loss method.

B. Weight loss method

Three aluminium specimens were immersed in 100mL of the solution at pH 10 and various concentrations of the inhibitor in the absence and presence of Zn^{2+} for a period of 1 day. The weight of the specimen before and after immersion was determined using shimadzu balance AY62.Inhibition efficiency (IE) was calculated from the relationship.

$$IE = 100 [1-W2/W1)] \%$$

Where W1 and W2 are the corrosion rates in the absence and presence of the inhibitor, respectively.

C. UV – visble spectrum

A spectrophotometer can be either *single beam* or *double beam*. In a single beam instrument (such as the Spectronic 20), all of the light passes through the sample cell. I_o must be measured by removing the sample. This was the earliest design and is still in common use in both teaching and industrial labs. In a double-beam instrument, the light is split into two beams before it reaches the sample. One beam is used as the reference; the other beam passes through the sample. The reference beam intensity is taken as 100% Transmission (or 0 Absorbance), and the measurement displayed is the ratio of the two beam intensities. Some double-beam instruments have two detectors (photodiodes), and the sample and reference beam are measured at the same time. In other instruments, the two beams pass through a beam chopper, which blocks one beam at a time. The detector alternates between measuring the sample beam and the reference beam in synchronism with the chopper. There may also be one or more dark intervals in the chopper cycle. In this case, the measured beam intensities may be corrected by subtracting the intensity measured in the dark interval before the ratio is taken.

D. Fluorescence spectra

In fluorescence spectroscopy, the species is first excited, by absorbing a photon, from its ground electronic state to one of the various vibrational states in the excited electronic state. Collisions with other molecules cause the excited molecule to lose vibrational energy until it reaches the lowest vibrational state of the excited electronic state. In a typical fluorescence (emission) measurement, the excitation wavelength is fixed and the detection wavelength varies, while in a fluorescence excitation measurement the detection wavelength is fixed and the excitation wavelength is varied across a region of interest.

Fluorescence spectroscopy is used in, among others, biochemical, medical, and chemical research fields for analyzing organic compounds. There has also been a report of its use in differentiating malignant skin tumors from begin.

III. RESULTS AND DISCUSSION

A. Analysis of result of weight loss method

The corrosion rate of aluminium in an aqueous solution at pH 10 (dil NaOH) in the absence and presence of inhibitors obtained by weight loss method are given in Table 1. The inhibition efficiency are also given in this table.

Table -1

Corrosion rates of Aluminium in an aqueous solution at pH 10 (dil NaOH),in the absence and presence of inhibitor system and the inhibition efficiencies (IE) obtained from weight loss method.

Inhibitor system : oxalic acid (OA)and Zn^{2+}

oxalic acid ppm	Zn^{2+} system ppm	Corrosion rate mdd	Inhibition efficiency %
0	0	23.48	--
50	0	6.52	70
100	0	6.10	72
150	0	5.63	74
200	0	5.12	75
250	0	4.80	78

Table -2

Corrosion rates of Aluminium in an aqueous solution at pH 10 (dil NaOH),in the absence and presence of inhibitor system and the inhibition efficiencies (IE) obtained from weight loss method.

Inhibitor system : oxalic acid (OA) and Zn^{2+}

oxalic acid ppm	Zn^{2+}system Ppm	Corrosion rate Mdd	Inhibition efficiency %
0	50	23.48	--
50	50	2.58	89
100	50	2.11	91
150	50	1.87	92
200	50	1.40	94
250	50	1.17	95
0	50	19.25	18

B. Analysis of UV –visible absorption spectra

UV-visible absorption spectrum of an aqueous solution containing oxalic acid and Al^{3+} (Aluminium Sulphate) is shown in Fig.1 A peak appears at 420nm .Is indicate that a complexes from between Al^{3+} and oxalic acid Fig.1

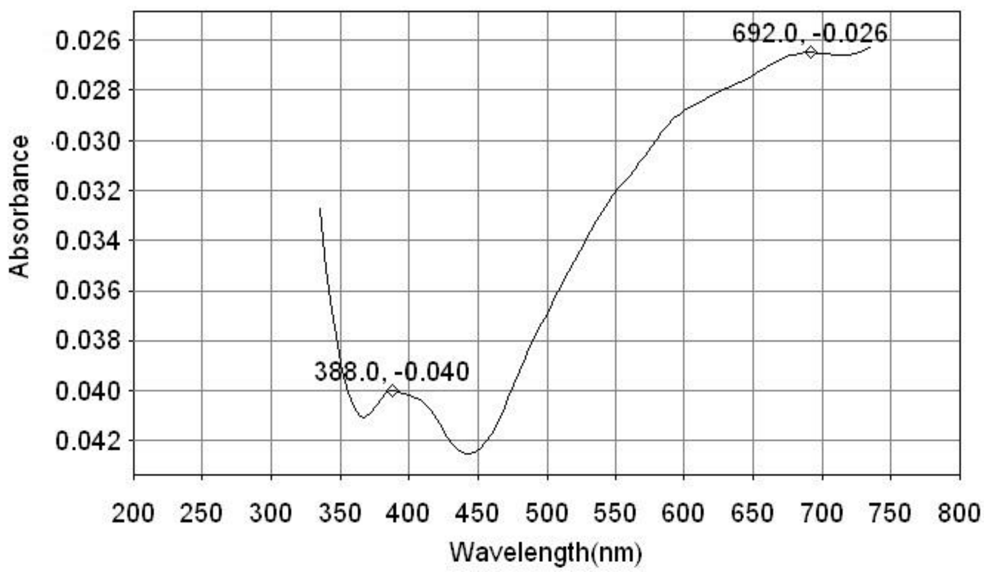


Fig.1 UV – visible absorption spectrum of an solution containing Oxalic acid and Al^{3+}

C. Analysis of Fluroscence spectra

The Fluroscence spectrum ($\lambda^{excitation}$ = 300nm) Fluroscence spectrum of an aqueous solution containing Al^{3+} and adipicacid (Al^{3+}-Oxalic acid) is shown in Figure 2a. Apeak appears at 392.5nm. The fluroscence spectrum of ($\lambda^{excitation}$ = 300nm) protective film formed on aluminium metal surface after immersion in the solution containing 250ppm of **Oxalic** acid and 50ppm Zn^{2+} solution after for 1 day is shown in figure 2b. A peak appears at 425.0nm.The peak matches which that of aluminium **Oxalic** acid complex. Hence it is confirm that the protective film formed on the metal surface is Aluminium and **Oxalic** acid complex ion

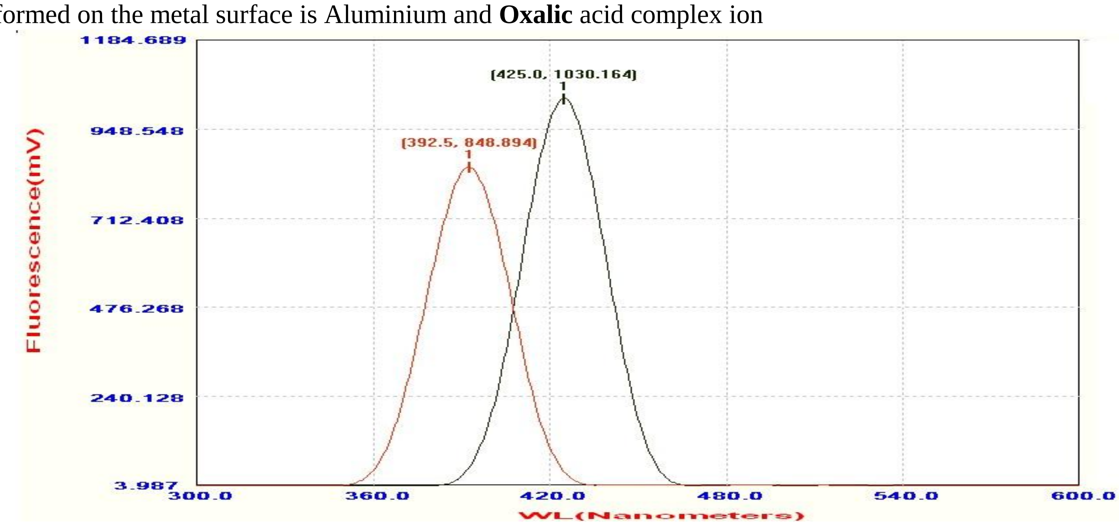


Fig.2a Fluroscence spectrum of an solution containing Oxalic acid and Al^{3+}

Fig.2b Fluroscence spectrum of an solution containing250ppm Oxalic acid and 50ppm Zn^{2+}

D. Analysis of SEM and EDAX spectra

The SEM and EDAX spectra have been used to investigate the smooth of the surface of the protective film formed on the metal surface during corrosion inhibition study. The SEM image of polished metal is shown in Figure 3(a) a .The EDAX spectrum is also shown in Figure 4(b). The weight percentage of aluminium is given in Table 3.The SEM image of aluminium metal immersed in an aqueous at the corrosive medium (aqueous solution at PH -10) is shown in Figure 3 (b). The EDAX spectra is also shown in Figure 4(b).The weight percentage of various elements is given in Table 5 and 6.The SEM image of protective film formed on the metal surface after immersed in pH -10 of the solution containing 250ppm of oxalic acid and 50ppm of Zn^{2+} is shown in Figure 3 (c). The EDAX spectra is also shown in Figure 4(c). The weight percentage of various elements is given in Table 7and 9

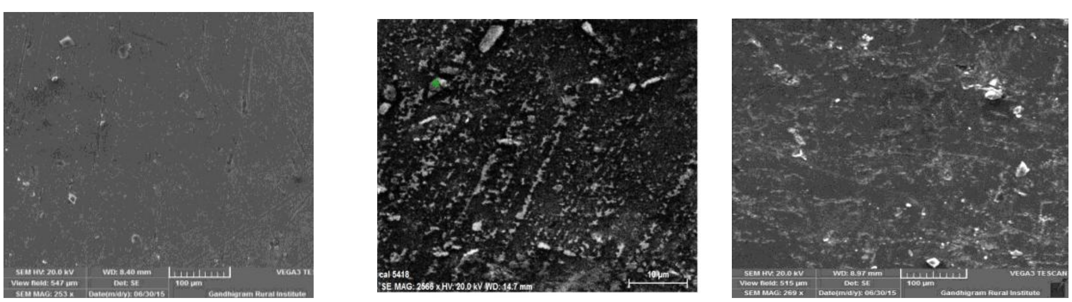
It is absorbed that SEM image of polished metal is smooth. When the metal is placed in the corrosive medium the surface become smooth. The image also indicated present of crystal of Al^{3+} /Al complex formed on the metal surface. The EDAX spectra reviewing the presence of aluminium for polished metal weight percentage OF aluminium 1.65% for the metal immersed in corrosive medium the weight percentage of aluminum is higher (3.5%) .This is due to the facts the corrosive medium has contract aluminium and exposable more aluminium. In the dealing the weight percentage of aluminium for the inhibitor is less than that for the corrosive medium system. This is due to the fact adipic acid and Zinc hydroxide have been absorbed on the metal surface and prevented corrosion.

Table-3 Data derived from SEM image polished metal

El	AN	Series	unn. [wt.%	C norm. [wt.%]	C Atom. [wt.%]	C Error	(1Sigma) [wt.%]
Al	13	K-series	85.33	88.89	78.95		4.12
O	6	K-series	8.51	8.86	17.68		3.11
C	8	K-series	2.15	2.24	3.36		0.79

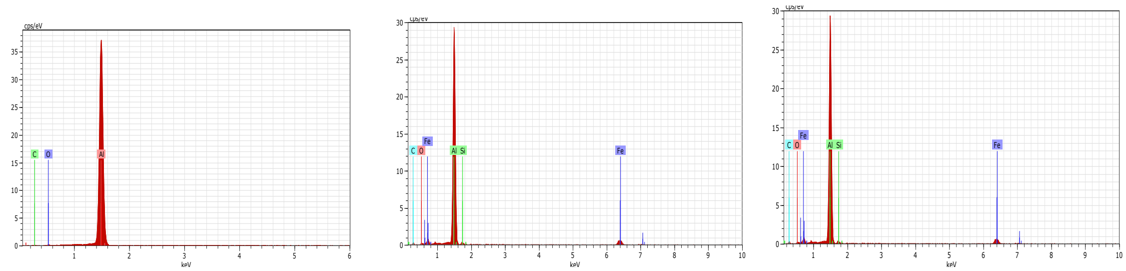
Table-4 Data derived from SEM image Metal in corrosive medium (OA)

El	AN	Series	unn. [wt.%	C norm. [wt.%]	C Atom. [wt.%]	C Error	(1Sigma) [wt.%]
Al	13	K-series	77.17	71.52	62.91		3.80
C	6	K-series	14.86	13.77	27.20		6.73
Fe	26	K-series	11.41	10.57	4.49		0.55
O	8	K-series	3.21	2.97	4.41		1.60
Si	14	K-series	1.20	1.16	0.98		0.15



(c) polished metal **(d) Metal in corrosive medium** **c) Metal in presence of inhibitors**

Fig 3 : SEM images of the surface of Aluminium metal (OA)



(e) polished metal **(f) Metal in corrosive medium** **c) Metal in presence of inhibitors**

Fig 4: EDX images of the surface of Aluminium metal (OA)

4. CONCLUSION

#.The present study lead to the following conclusions

#.Inhibition efficiency of Oxalic acid in controlling corrosion of aluminium in distillate water in the absence and presence of Zn^{2+} has been evaluated by weight loss method

#.UV –visible spectra reveal that the protective film consists of Al^{3+} and oxalic acid complex

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