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Investigation and inhibition of Brass in sulphuric acid solutions by 3-Amino 1,2,4 Triazole compound

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Abstract: The inhibition effect 3-Amino 1,2,4 Triazole compound on the corrosion of brass in 1M H_2SO_4 has been investigated by Weight Loss (WL),Temperature effect and electrochemical techniques such as Potentiodynamic polarization studies were used. Results showed that ATA has good inhibition efficiency on the corrosion of bress in 1M H_2SO_4 solution. Polarization measurement indicated that, the studied inhibitor act as a mixed type one. The inhibition efficiency depends on the concentration of inhibitor and reaches 99% at 30^{o}c. The results obtained from the different methods are in good agreement. The SEM and AFM examination of the brass surface revealed that the compound prevented from corrosion by adsorption on its surfaces.
Keywords: Nitric acid, Weight loss, corrosion inhibition, Scanning Electron Microscope(SEM), Electrochemical impedance spectroscopy (EIS).

INTRODUCTION

Brass is extensively used in various industrial operations and the study of its corrosion inhibition is of importance, most investigations on the corrosion of brass have been carried out on. The development of corrosion inhibitor based on organic compounds has much scope in several industries because of their practical usefulness. The molecular structure of organic compounds used as inhibitor has been found to exert a major influence on the extent of inhibition of corrosion [1-4], brass and its alloys are widely used materials for their excellent electrical and thermal conductivities in many applications and recently in the manufacture of integrated circuits [5-6]. The chemical dissolution and electro plating are the main processes used in the fabrication of electronic devices. The most widely used acid solution, so this medium has induced a great deal of research on brass [7-10]. Most of the effective organic inhibitor have hetero atoms containing multiple bonds in their molecules through which they can adsorb on the metal surface, In the present study organic AminoTriazole as inhibitions for the corrosion of brass in 1M H_2SO_4 have been examined using weight loss measurement, Polarization studies SEM and process [11] results are reported and discussed.

The aim of the present investigation was to study the inhibitive action of organic compounds on the corrosion of brass in 1M H_2SO_4 solution.

MATERIALS AND METHODS

Materials and reagents

Brass strips of size 4 X 1 X 0.25 cm, the samples were mechanically polished using different grades of emery sheets, washed with trichloroethylene and triply distilled water and dried.
The organic ATA used in this study were procured from Fluka AG in US, and, Analar grade 1M H_2SO_4 was used for preparing the aggressive solution.

METHODS

Weight loss measurements

Weight loss measurement weight loss measurements were called out described in an earlier work [12] Brass metal strip was immersed in 100ml of inhibited and uninhibited solutions of 1M H_2SO_4 for 2 hours. The inhibition efficiency was calculated from weight loss values obtained in the present and absence of the inhibitors at the end of definite intervals of time

$$\text{I E\%} = \{ (W_O - Wi) / W_O \} \text{ X } 100$$

Where

Wo = weight loss in plain acid
Wi = weight loss in inhibited acid

Temperatures Effects:

The some procedure adopted for weight for studies at temperature of the study was varied from 35^{O}c to 60^{o}c. At the end of each experiment. The specimens were taken out washed both in running tap water and in distilled water. They were dried and their weights were measured. The loss in weight was calculated. Each experiment was duplicated to get good reproducibility. Weight loss measurement were performed in 1M H_2SO_4 with and without the addition of the inhibitor at their best inhibitory concentration percentages inhibitor at various temperature was calculated.

Polarization measurements:

The working electrode was immersed in test solution during 30 minutes until a steady state open circuit potential was obtained. Both anodic and cathodic polarization curves were recorded by potentiodynamically using a corrosion measurement system consisting of a BAS Model 100 K H Z to 10MHZ a comprised elctrochemical analyzer (made in Lafeyette, in USA) PL-10 digital plotter (DMP-40 series the instruments, Division, Hovston, TX, USA) made by a platinum for and Hg/ Hg2Cl2 / 1M H_2SO_4 were used as he auxiliary electrode and reference electrode respectively. Which was controlled by a personal computer[13].

EIS measurements:

The electro chemical impedance spectroscopy were carried out using a transfer function analyzer, with a small amplitude . The double layer capacitance (C_{dl}) and the charge transfer resistance (R_t) were obtained using ac- impedance instrument [14].

Scanning Electron Microscope

The brass immersed in blank and in the inhibitor solution for a period of 2 hours was removed, rinsed with double distilled water, dried and observed in a Scanning Electron Microscope to examine the surface morphology. The surface morphology measurements of the Brass were examined using Hitachi S-3000 computer controlled Scanning Electron Microscope [15].

Atomic Force microscopy

AFM is a powerful technique for gathering of roughness statistics from a verity of surfaces AFM is becoming an accepted method of roughness investigation [16].

Atomic force microscopy provided direct insight into the changes in the surface morphology takes place at several hundred nanometers when topographical changes owing to the initiation of corrosion and formation of protective film on the metal and presence and absence of inhibitors respectively .All atomic force microscopy images were obtained on a pico SPM2100 AFM instrument operating in contact mode in air.

RESULTS AND DISCUSSION

Molecular structure of organic compound used in the present study is given in Fig (1).

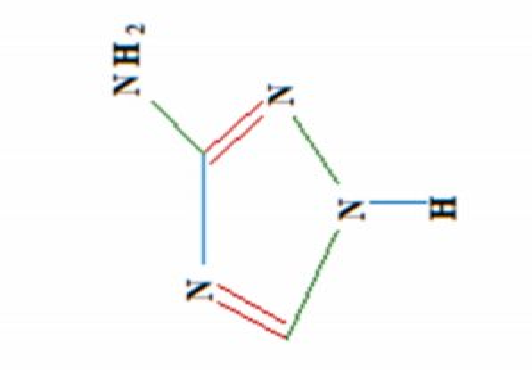

Fig (1) 3-Amino 1,2,4 Triazole (ATA)

Weight loss measurements

The value of percentage inhibition efficiency (% I E), corrosion rate (CR) and surface coverage values (θ) obtained from weight loss method at different concentrations of ATA in 1M H_2SO_4 at 303k .The variation of inhibition efficiency with in increase in inhibitor concentrations is shown in Fig (2). It was observed that DES inhibits the corrosion of brass in 1M H_2SO_4 solution at all concentrations used in study, i.e from 1 mM to 100mM maximum inhibition efficiency was shown at 100mM concentration of the inhibitor in 1M H_2SO_4 at 303k. it is evident from Fig(2) that the corrosion rate is decreased on the addition of ATA and also increase surface coverage.

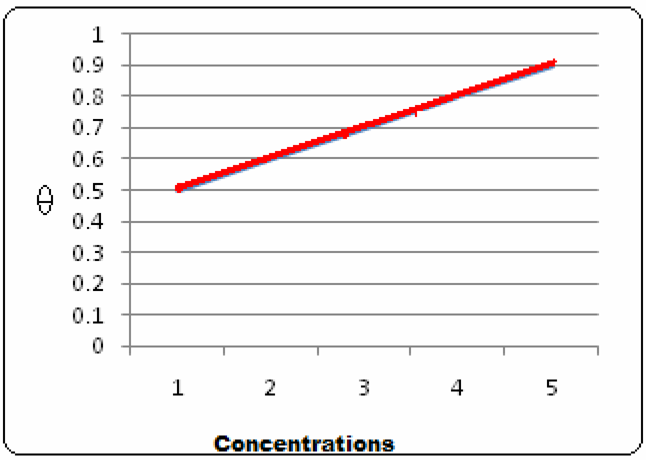

Fig (2) Variation of inhibition efficiency on brass with different concentration of ATA in 1M H_2SO_4at 30° for 2 hrs

Temperatures Effects:

The value of inhibition efficiency obtained from weight loss measurement at the different temperatures of 303k to 333k in 1M H_2SO_4 solution at its best protecting concentration are presented in the Tables – 1. The inhibition efficiency decreased at high temperature.

Table- 1 Corrosion parameters for brass in aqueous solution of 1M H_2SO_4 in absence and presence of optimum concentration of ATA at different temperature for 2 hrs

Temperature (K)	Inhibitor	W (mg/cm2.h)	% I. E
303	Blank	**0. 3912**	**-**
	ATA	**0.0770**	**85.8**
313	Blank	**0.3134**	**--**
	ATA	**0.0239**	**63.18**
323	Blank	**0.5720**	**--**
	ATA	**0.0336**	**35.80**
333	Blank	**1.2031**	**--**
	ATA	**0.5334**	**14.59**

Potentiodynamic polarization measurement

Current – potential characteristics resulting from cathodic and anodic polarization curves of brass 1M H_2SO_4 in the presence and absence of ATA at various concentrations are gives values of corrosion current

(I_{corr}) corrosion potential (E_{corr}) anodic and cathodic Tafel slop (ba,bc) for ATA at various concentration in 1M H_2SO_4

In the case of polarization method the relation determines the inhibition efficiency (% I E) As it is shows in fig-(3) cathodic current – potential curves give rise to parallel Tafel lines indicating that the reaction is under activation controlled the cathodic current density decreases with the concentration of ATA more over a small effect is observed on the anodic portions. This result indicates that ATA is adsorbed on the metal surface on the cathodic sites and hence inhibition occurs, we remark that the inhibitor act on the anodic portion and the anodic current density is reduced. It seems also that the presence of the inhibitor the corrosion potential values in definite direction. These results indicated that ATA act as a mixed type inhibitor.

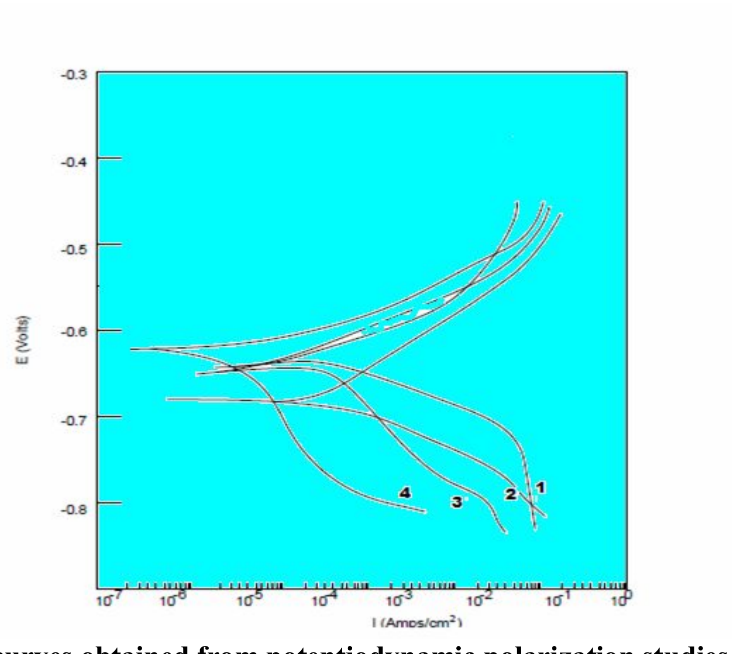

Fig-3 Polarization curves obtained from potentiodynamic polarization studies for Brass in aqueous solution of 1M H_2SO_4 in absence and presence of different concentration of ATA.

Electrochemical Impedance spectroscopy

The results of impedance parameters (R_t and C_{dl}) for inhibitors are given in fig (4), the charge transfer resistance (R_t) value increase with increase in inhibitor concentrations but the value of durable layer capacitance (C_{dl}) decrease due to decrease in local dielectric constant and increase in thickness of the electrical double layer, suggesting that the inhibitor molecules function by adsorption at the metal solution interface. Hence the inhibition efficiency increases with increase in inhibitor concentration.This may be due to the availability of more sites on the metal surface in 1M H_2SO_4 solution because of the lesser adsorption of the ATA on the metal surface.

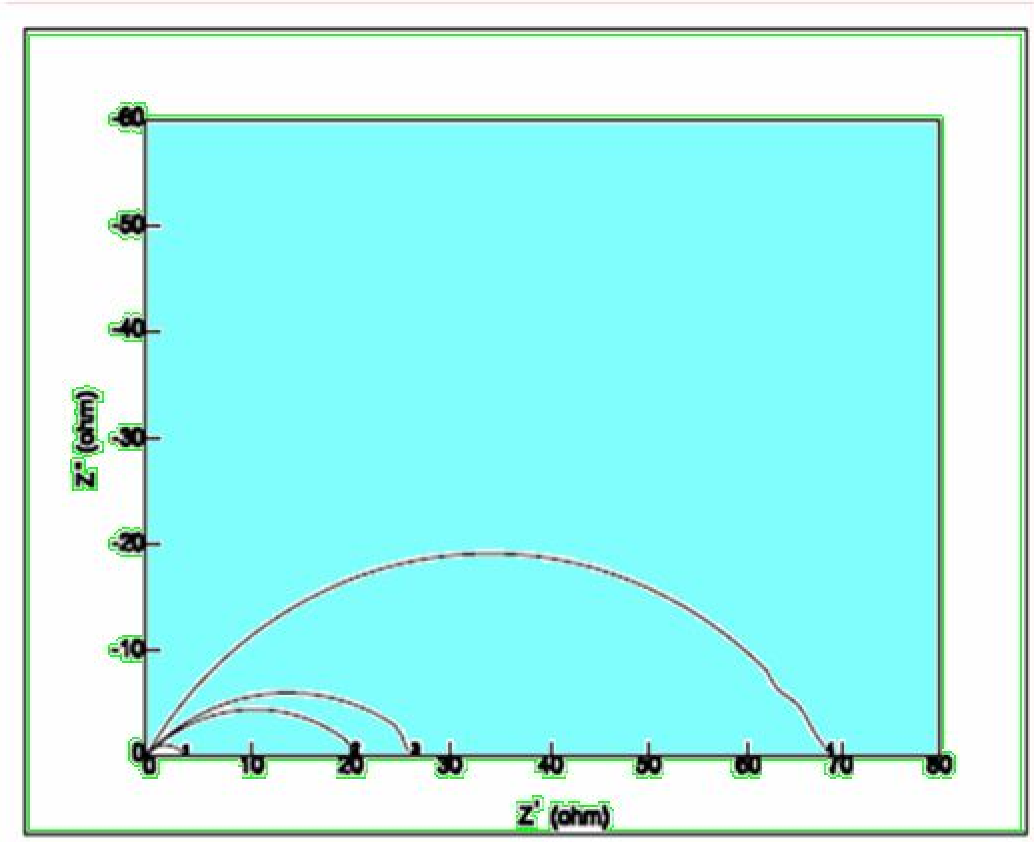

Fig -4 Impedance diagram for corrosion of brass in 1M H_2SO_4 in absence and presence of different concentration of ATA.

SEM analysis of metal surface

The SEM image of magnification (x 1000) of brass specimen immersed in 1M H_2SO_4 for 2 hours in the absence and presence of inhibitor system are shown in fig.5 (a,b).

The SEM micrographs of brass surface immersed in 1M H_2SO_4 in fig.5 (a) Shows the roughness of the metal surface which indicates the corrosion of brass in1M H_2SO_4. Fig.5(b) indicates that in the presence of ATA, the surface coverage increases which inturn results in the formation of insoluble complex on the surface of the metal (ATA inhibitor complex) and the surface is covered by a thin layer of inhibitor which effectively control the dissolution of brass.

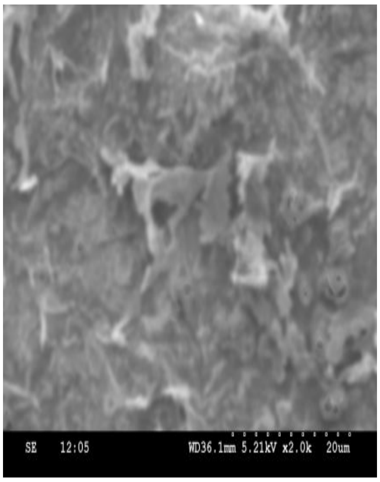

Fig-5 (a) Pure metal + 1M H_2SO_4 Acid

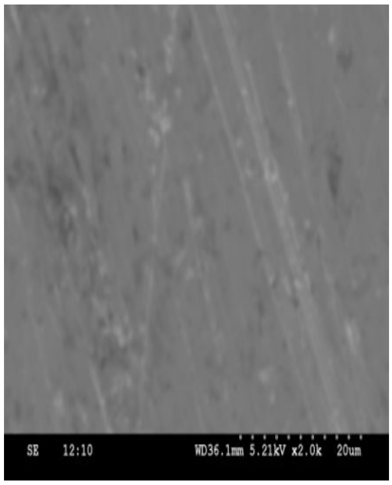

Fig-5 (b) Pure metal+1MH_2SO_4 Acid + Inhibitor (ATA)

Atomic Force Microscopy (AFM) Analysis of Metal Surface

The coated protective films on brass metal are examined with AFM (Atomic Force Microscopy), a powerful technique for the gathering of roughness statistics from a variety of surfaces. AFM is becoming an accepted method of roughness investigation.

Atomic force microscopy provides direct insight into the changes in the surface morphology that takes place at several hundred nano meters, when topographical changes takes place due to corrosion and formation of protective film on the metal surface in the presence and absence of inhibitors respectively.

The two dimensional (2D) and three dimensional (3D) AFM morphologies for polished brass metal surface. Brass metal surface immersed in 1M H_2SO_4 and brass metal immersed in 1M H_2SO_4 containing inhibitor is shown in Fig(6)&(7).

AFM image analysis was performed to obtain the average roughness (Ra). The average deviation of all points roughness profile from a mean line over the evaluation length, Root-mean-square roughness R_q (the average of the measured height deviations taken within the evaluation length and measured from the mean line) and the maximum Peak-to-Valley (P-V) height values (largest single peak-to-valley height in five adjoining sampling heights). R_q is much more sensitive than R_a to large and small height deviations from the mean.

Table 2. AFM Data for brass metal surface immersed in inhibited and uninhibited environments

Samples	RMS (R_q) Roughness *(nm)*	Average (R_a) Roughness *(nm)*	Maximum Peak- to-Valley Height *(nm)*
Polished brass (Control)	250	198	2630
Brass immersed in1M H_2SO_4 blank	665	578	2892
Brass immersed in1M H_2SO_4 containing 0.01M ATA	346	248	2080

Table 2. is a summary of the average roughness (R_a) RMS-roughness (R_q), maximum peak-to-valley height (P-V) value for brass immersed in different environments.

The value of R_q, R_a and P-V height for the polished brass metal surface are 250 nm, 198 nm and 2630 nm respectively. Which shows a homogeneous surface, with some places in which the height is lower than the average depth fig(6).displays the un corroded metal surface.

The RMS roughness, average roughness and P-V height values for the brass metal surface immersed in 1M H_2SO_4 are 665 nm, 578 nm and 2892 nm respectively. These data suggests brass metal surface immersed in1M H_2SO_4 acid a greater surface roughness than the polished metal surface, which shows that the unprotected brass metal surface is rougher due to the corrosion of brass in 1M H_2SO_4 Fig (7). displays corroded metal surface with few pits.

The presence of ATA in 1M H_2SO_4 reduces the R_q by 346 nm from 665 nm and R_a is significantly reduced to 248 nm from 578 nm of brass metal immersed in acid. The P-V also reduced to 2080 nm from 2892 nm. These parameters confirm that the surface appears smoother. The smooth run of the surface is due to the formation of a protective film Fig (8). displays the un corroded metal surface.

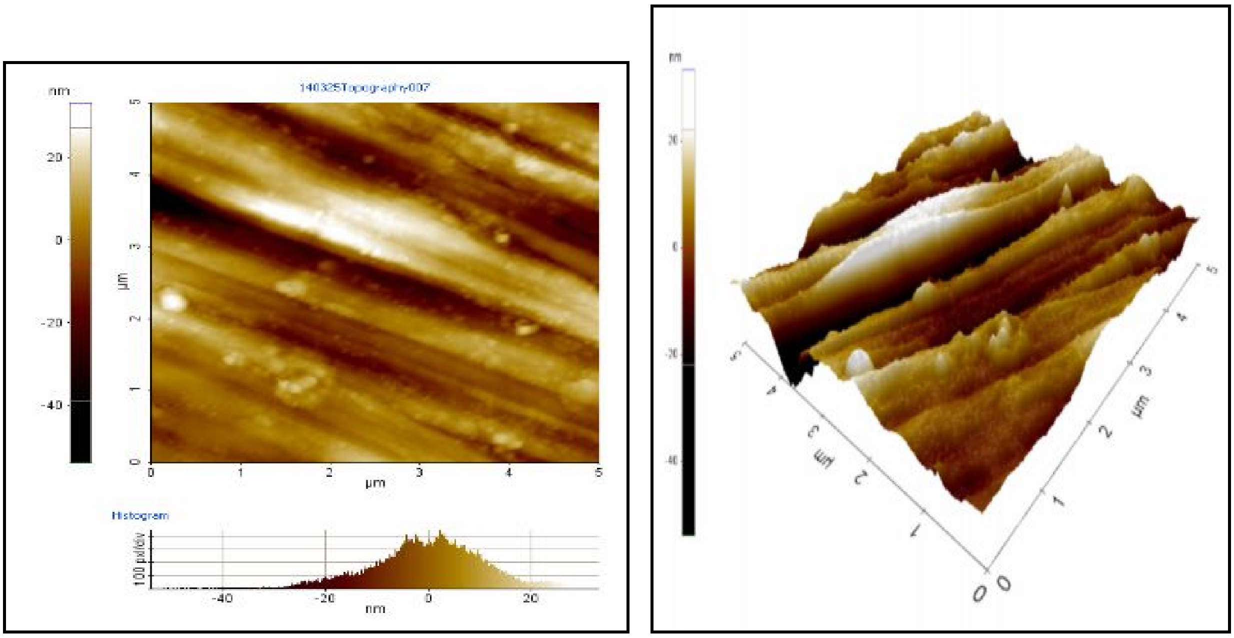

Fig-6, 2D and 3D AFM images of polished brass metal surface

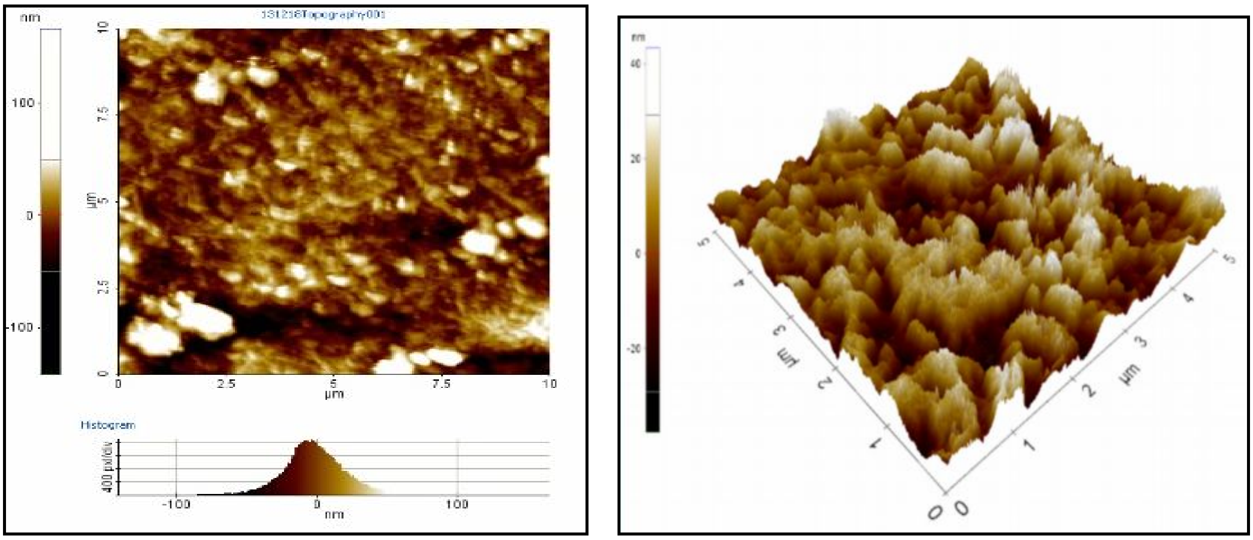

Fig -7. 2D and 3D AFM images of brass immersed in1M H_2SO_4

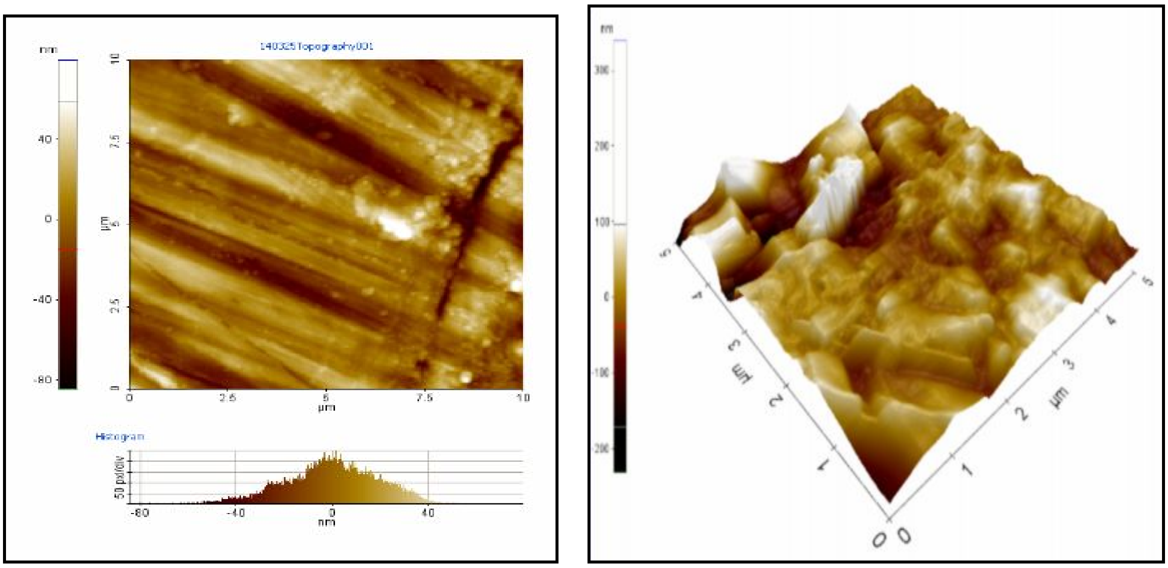

Fig -8, 2D and 3D AFM images of brass metal immersed in1M H_2SO_4 in Presence of ATA.

CONCLUSION

On the basis of the above results the following conclusion can be drawn.

- Results obtained from the experimental data shown that ATA act as an effective inhibitor for corrosion in 1M H_2SO_4 acid.
- The corrosion process was inhibited by adsorption of the organic matter on the brass surface. Inhibition efficiency increases with increase in the concentration of the ATA but decrease with rise in temperature.
- Polarization measurements show that ATA act essentially as a mixed type inhibitor.
- The values impedance parameters justify the impressive performance of organic ATAcompound good corrosion inhibitor.
- The SEM and AFM images confirm the formation of protective layer on the metal surface.

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