Research Article

Corrosion Inhibition of Aluminium brass in natural sea water by substituted benzotriazoles derivatives

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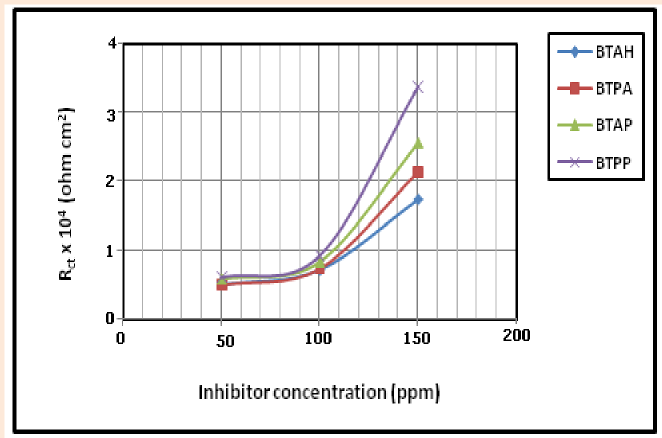
Abstract

New class of four substituted benzotriazole derivatives namely 2-(1H-benzotriazol-1-yl) acetohydrazide (BTAH), 2-(1H-benzotriazol-1-yl)phenyl acetohydrazides (BTPA), 2-(1H-benzotriazol-1-yl) acetohydrazides pyrazolidine dione (BTAP) and 2-(1H-benzotriazol-1-yl) aceto hydrazides phenyl pyrazolidine dione (BTPP) have been synthesized in the laboratory. Benzotriazole derivatives as corrosion inhibitor of aluminium brass in natural sea water were studied. The inhibition effect of benzotriazole derivatives on Al-brass corrosion was studied. The inhibition effect of the BTA derivatives on the aluminium brass corrosion in natural sea water was studied using gravimetric measurements, potentiodynamic polarization technique, potentiostatic current transient techniques and electrochemical impedance spectroscopy methods. The potentiodynamic polarization curve shows that BTA derivatives inhibit both anodic and cathodic reactions at all concentration. EIS results indicate that as the additive concentration is increased, the charge transfer resistance increases whereas double-layer capacitance decreases. Scanning electron microscopy is used to identify the nature of the protective film formed on the metal surface.



Keywords: benzotriazole, Al-brass alloy; potentiodynamic polarization; electrochemical impedance spectroscopy, SEM

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Introduction

Copper and its alloys are widely used in many industrial fields, especially in marine applications [1]. Al and its alloys are used in a wide range of industrial applications for different aqueous solutions, so it represents an important category of technological materials [2, 3]. The relatively low cost and high resistance toward corrosion of Al–brass preferred its use for many years in tubes production for sea water, cooled heat exchangers and piping. This metal and its alloy have the tendency to form a stable oxide film naturally or by anodisation [4, 5]. Generally, Al-oxide consists of a thin barrier film adjacent to the metal surface (25 nm thick) which is covered by a thicker porous oxide layer. In aggressive media as chloride solution, a localized corrosion can occur which leads to a break down of the passive layer and a pit formation [6, 7].

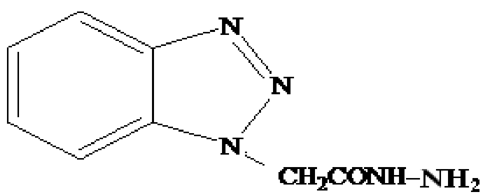
The inhibition of copper corrosion in neutral or slightly alkaline solutions is one of the most hectic tasks for cooling water system operations. A study of mechanistic action of corrosion inhibitors has relevance both from the point of view of a search for new inhibitors and also for their effective usage [8]. Many authors have investigated the corrosion control of copper in various media using large numbers of organic and inorganic compounds. Results showed that organic compounds, especially those containing nitrogen [9-11] or sulphur [12] gave a very good inhibition for copper corrosion in different media. It is well known that triazole types of organic compounds are good corrosion inhibitors for many metals and alloys in various aggressive media [13, 14]. Many mechanisms have been proposed for the inhibition of metal corrosion by organic inhibitors which take place via adsorption on the metal surface [15, 16]. The adsorption process leads to an effective blocking of the active sites of metal dissolution and/or hydrogen evolution, which results in a considerable decrease in the corrosion rate.

The purpose of the present study is to assess the inhibition efficiencies of new corrosion inhibitors viz., 2-(1H-benzotriazol-1-yl) acetohydrazide (BTAH), 2-(1H-benzotriazol-1-yl)phenyl acetohydrazides (BTPA), 2-(1H-benzotriazol-1-yl) acetohydrazides pyrazolidine dione (BTAP) and 2-(1H-benzotriazol-1-yl) aceto hydrazides phenyl pyrazolidine dione (BTPP). Weight-loss method and electrochemical studies such as potentiodynamic polarization, impedance spectroscopy and current transient techniques were used. Accelerated leaching studies were carried out by inductively coupled argon plasma–atomic emission spectroscopy.

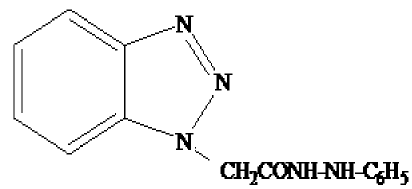
Materials and Methods

Materials

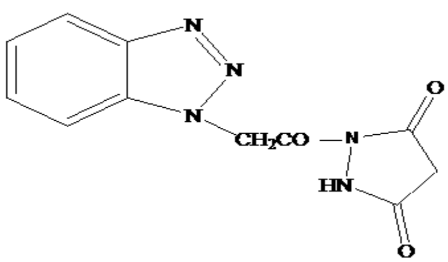
The material used for this study was Al-brass alloy supplied in the form of sheet and the chemical composition (weight percent) of the alloy was 78.25 Cu, 14.65% Zn, 4.5% Al, 0.6 % Mn, 1.5% Mg, 0.1% Cr and 0.5% Si as analyzed by optical emission spectrophotometry. The natural sea water was collected near National Thermal Power Station, Ennore, Chennai, India. The substituted benzotriazole derivatives employed in the present investigation were prepared according to the earlier reports [17] and their structures are shown in **Figure 1**.



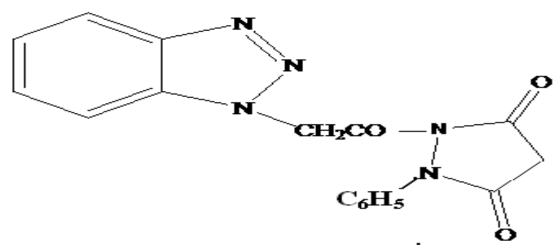
2-(1H-benzotriazol-1-yl) acetohydrazide



2-(1H-benzotriazol-1-yl)phenyl acetohydrazide



2-(1H-benzotriazol-1-yl) acetohydrazides pyrazolidine dione



2-(1H-benzotriazol-1-yl) acetohydrazides phenyl pyrazolidine dione

Figure 1 Structures of substituted benzotriazoles derivatives

Synthesis of Substituted Benzotriazole Derivatives

2-(1H-benzotriazol-1-yl) acetohydrazide (BTAH)

In a 250ml iodine flask, an ethanolic solution of ethyl 1-H benzatriazol-1-yl acetate (0.01M) and hydrazine hydrate (20 ml) is stirred for 4 hours at room temperature and then refluxed on a water bath for 3 hours in a 250 ml round bottom flask, the solution is kept overnight in a 250 ml beaker and then excess solvent was removed under reduced pressure. The solid mass so obtained is washed with cold water and recrystallised from ethanol.

Synthesis of 2-(1H-benzotriazol-1-yl)phenyl acetohydrazides (BTPA)

In a 250ml iodine flask, an ethanolic solution of ethyl 1-H benzatriazol-1-yl acetate (0.01M) and phenyl hydrazine hydrate (20 ml) is stirred for 4 hours at room temperature and then refluxed on a water bath for 3 hours in a 250 ml round bottom flask, the solution is kept overnight in a 250 ml beaker and then excess solvent was removed under reduced pressure. The solid mass so obtained is washed with cold water and recrystallised from ethanol.

Synthesis of 2-(1H-benzotriazol-1-yl) acetohydrazides pyrazolidine dione (BTAP)

In a 250ml round bottomed flask, 2-(1-H-benzotriazol-1-yl) aceto hydrazide (0.01M) is dissolved in ethanol (50ml) and diethyl propanedioate (0.01M), glacial acetic acid (2-3 drops) were added. The reaction mixture is refluxed for 6 hours. Then the reaction mixture is kept in an open china dish for 3 days. The so obtained crystals were filtered and recrystallised from ethanol.

2-(1H-benzotriazol-1-yl) acetohydrazides phenyl pyrazolidine dione (BTPP)

In a 250ml round bottomed flask, 2-(1-H-benzotriazol-1-yl) phenyl aceto hydrazide (0.01M) is dissolved in ethanol (50ml) and diethyl propanedioate (0.01M), glacial acetic acid (2-3 drops) were added. The reaction mixture is refluxed for 6 hours. Then the reaction mixture is kept in an open china dish for 3 days. The so obtained crystals were filtered and recrystallised from ethanol.

Methods

Gravimetric Measurements

The experiments were carried out with Al-brass alloy specimens of dimension 3 cm x 2 cm x 0.2 cm. The panels were polished mechanically with silicon carbide papers from 120 to 1200 grit. The panels were degreased in acetone, thoroughly washed with double distilled water and dried. The polished alloy specimens were weighed and immersed in duplicate, in 500 ml natural seawater in the absence and presence of benzotriazole derivatives at different concentrations, for a period of 30 days. Then these specimens were reweighed after dipping for 2 to 3 min in 10% sulfuric acid at room temperature. Later, these specimens were scrubbed with a bristle brush under running water, degreased with acetone, dried and weighed.

Potentiodynamic Polarization Studies

The polarization studies were carried out with Al-brass strips having an exposed area of 1 cm^2. The cell assembly consisted of Al-brass as working electrode, a platinum foil as counter electrode and a saturated calomel electrode (SCE) as a reference electrode with a Luggin capillary bridge. Polarization studies were carried out using an Electrochemical work station (Model: CHI 760C, CH Instruments, USA) at a scan rate of 1mV/s. The degreased working electrode is then inserted into the test solution and immediately cathodically polarized at -1.0 V (SCE) for 15 minutes to reduce any oxides on the brass surface [18]. The cathodic and anodic polarization curves for Al-brass in the test solution with and without various concentrations of the inhibitors were recorded at a scan rate of 1 mV/s. The inhibition efficiencies of the compounds were determined from corrosion current densities using the Tafel extrapolation method.

Electrochemical AC impedance studies

AC impedance measurements were conducted at room temperature using an Autolab with Frequency Response Analyzer (FRA), which included a potentiostat model PGSTAT 12. An AC sinusoid of ±10 mV was applied at the corrosion potential (E_{corr}). The frequency range of 100 kHz to 1 mHz was employed. The Al-brass specimen with an exposing surface area of 1 cm^2 was used as the working electrode. A conventional three electrode electrochemical cell of volume 100 ml was used. A saturated calomel electrode was used as the reference and platinum plate electrode was used as the counter. All potentials are reported versus SCE.

Potentiostatic Current Transient Techniques

The current transient of brass specimen as a function of immersion period in the test solution with and without the addition of inhibitors was recorded at a preset potential of -100 mV.

Scanning Electron Micrscopy (SEM)

The Al-brass specimens were immersed in various test solutions for a period of 15 days. After this period, the specimens were taken out and dried. The nature of the film formed on the metal surface was analyzed by the scanning electron microscopy.

Results and Discussion

Weight-loss Measurements

The weight loss measurements are the best known and simplest of all corrosion monitoring techniques. The corrosion rates and inhibition efficiencies of Al-brass with different concentrations of BTAH, BTPA, BTAP and BTPP in natural sea water at room temperature (30^oC) are given in **Table 1**. The variation of inhibition efficiency with different concentration of the inhibitors in natural sea water is shown in the **Figure 2**. The corrosion rate (CR) and percentage inhibition efficiency (IE %) were calculated using the following equation [19].

$$CR\ (mmpy) = \frac{87.6 \times W}{D \times A \times T}$$

$$IE\% = \frac{CR_{(bl)} - CR_{(inh)}}{CR_{(bl)}} \times 100$$

where W is the weight-loss, D is the density, T is the immersion time, A is the area of the specimen and $CR_{(inh)}$ and $CR_{(bl)}$ are the corrosion rate of Al-brass in the presence and absence of inhibitors respectively. The corrosion rate decreases with increase in the concentration of the inhibitor. The inhibition efficiency increases with the increase in the concentration of the inhibitor upto the optimum level, thereafter it was found to decrease slightly, which is due to the interaction between adsorbed molecules at the sites. The extent of inhibition depends on the nature and concentration of the inhibitor. The optimum concentration was evaluated on the basis of inhibition efficiency and it was found to be 150ppm for BTAH, BTPA, BTAP and BTPP in natural sea water. The inhibitors have shown the maximum inhibition efficiency of 84.18, 85.59, 90.63 and 92.35 respectively for BTAH, BTPA, BTAP and BTPP in natural sea water. It can be seen that the values of inhibition efficiency for brass obtained using substituted benzotriazoles in natural sea water follow the order.

BTPP > BTAP > BTPA > BTAH

The inhibition of corrosion by these compounds can be attributed to their adsorption on the metal surface because of the following interactions.

- The interaction between the π-electrons of the benzotriazole ring and the positively charged metal surface, and
- The interaction between the lone pair of electrons of the atoms of nitrogen and the positively charged metal surface

The order of inhibition shown by these compounds can be mainly attributed to the electron releasing tendencies (+I effect) of different substituents present in the benzotriazole derivatives. An increase in the size of the molecule of these compounds can lead to more surface coverage and thereby more corrosion inhibition. Inhibition of corrosion of Al-brass in natural sea water can be explained in the following way. The adsorption of benzotriazole derivatives on the surface of brass leads to the formation of a protective layer of Cu(I)-chloride complex on the surface of brass. Actually the formation of a benzotriazole film starts with the chemisorption of the inhibitor molecule on to the slightly oxidized areas of the copper surface. The adsorption of benzotriazole molecules on the oxidized parts of the copper surface was found to occur much faster than on bare metal zones. The film formed in this way has a limited hydrophobic action, which succeeds in protecting brass in the corroding medium by blocking main reaction centers on the metal surface.

Table 1: Weight loss measurements of Al-brass at different concentrations of BTAH, BTPA, BTAP and BTPP in natural sea water

Inhibitor Concentration (ppm)	Weight Loss (mg)	Corrosion Rate x 10^{-2} (mmpy)	Inhibition Efficiency (%)
Blank	138.1	13.46	-
BTAH			
50	74.47	7.26	46.06
100	50.78	4.95	63.22
150	21.85	2.13	84.18
200	21.95	2.14	84.10
BTPA			
50	66.88	6.52	51.56
100	42.37	4.13	69.32
150	19.90	1.94	85.59
200	20.11	1.96	85.43
BTAP			
50	55.91	5.45	59.51
100	37.34	3.64	72.96
150	12.93	1.26	90.63
200	13.23	1.29	90.41
BTPP			
50	52.11	5.08	62.26
100	29.65	2.89	78.53
150	10.57	1.03	92.35
200	10.77	1.05	92.20

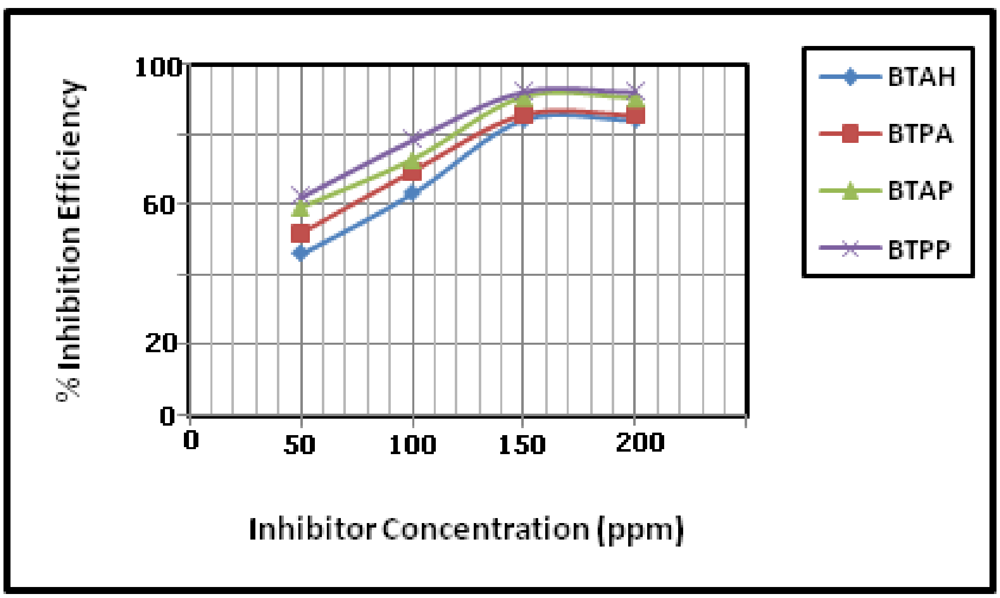


Figure 2 Variation of inhibition efficiency with different concentration of BTAH, BTPA, BTAP and BTPP in natural sea water

Potentiodynamic Polarization Studies

The cathodic and anodic polarization curves of Al-brass in natural sea water containing optimum concentration of BTAH, BTPA, BTAP and BTPP are shown in **Figure 3**. It is evident that in the presence of inhibitor, the cathodic and anodic curves were shifted towards noble direction and the shift was found to be dependent on inhibitor concentration. A **Table 2** illustrates the corresponding electrochemical parameters. The corrosion potential (E_{corr}) values were shifted in the presence BTAH, BTPA, BTAP and BTPP, which clearly indicated that the inhibitors control the anodic and cathodic reactions and thus act as mixed-type inhibitors. The corrosion current density (I_{corr}) decreased with increasing concentrations of the inhibitors. The corrosion rates [20] and inhibition efficiencies [21] were calculated from polarization curves using the following equation.

$$CR = \frac{3.27 \ x \ 10^{-3} \ x \ I_{corr} \ x \ EW}{D}$$

$$IE\% = \frac{I_{corr} - I_{corr\,(inh)}}{I_{corr}} \ x \ 100$$

Where CR is the corrosion rate (mmpy), D is the density (g cm^{-3}), EW is the equivalent weight of the specimen, IE is the inhibition efficiency and $I_{corr\,(inh)}$ and I_{corr} are corrosion current density in the presence and absence of inhibitors respectively.

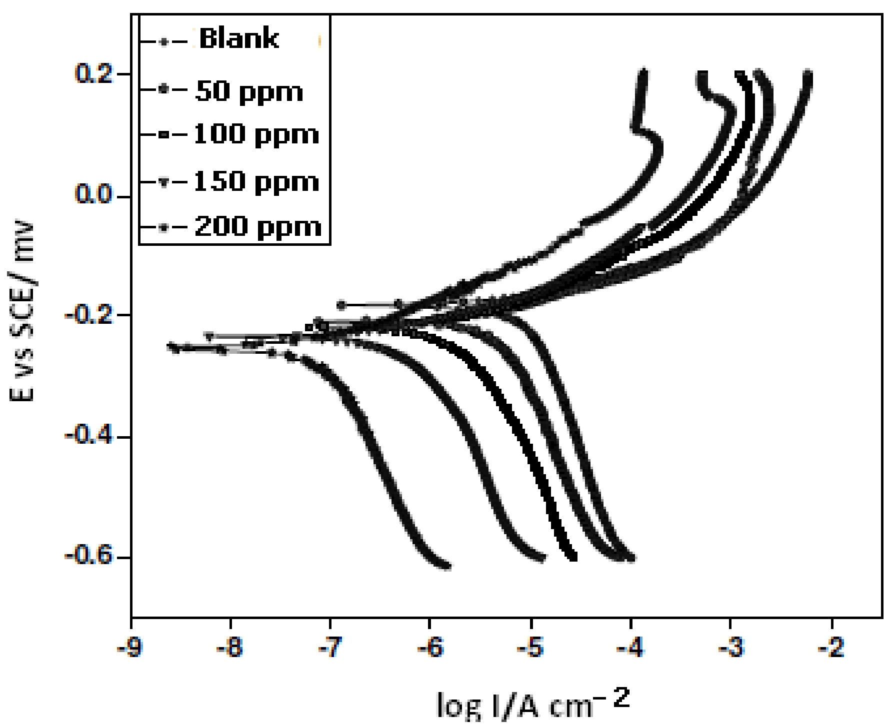


Figure 3 Polarization curves of Al-brass in natural sea water containing optimum concentrations of BTAH, BTPA, BTAP and BTPP

The values of cathodic Tafel slope (β_c) and anodic Tafel slope (β_a) of benzotriazole derivatives are found to change with inhibitor concentration, which clearly indicates that the inhibitors controlled both the reactions. The inhibition efficiency of BTAH, BTPA, BTAP and BTPP in natural sea water attained a maximum value of 84.59%, 86.14%, 89.87% and 91.56% at 150ppm concentration respectively. The values of inhibition efficiency increase with increasing concentration of inhibitor, indicating that a higher surface coverage was obtained in the solution with the optimum concentration of inhibitor. The corrosion rate of Al-brass in natural sea water was found to be 12.20 x 10^{-2} mmpy and it was minimized by adding the inhibitors to a lower value of 1.88 x 10^{-2} mmpy, 1.69 x 10^{-2} mmpy, 1.24 x 10^{-2} mmpy and 1.03 x 10^{-2}mmpy due to the adsorption of BTAH, BTPA, BTAP and BTPP on the metal surface respectively. A comparison of the values of inhibition efficiency obtained by weight loss measurements and polarization studies bring out clearly the fact that there is a fairly good agreement between these values. It is also found that all the compounds function more effectively on the corrosion inhibition of Al-brass in natural sea water.

Table 2 Tafel polarization parameters for the corrosion of Al-brass natural sea water at different concentrations of BTAH, BTPA, BTAP and BTPP

Inhibitor Concentratio (ppm)	I_{corr} (μA cm^{-2})	- E_{corr} (mV vs. SCE)	β_a (mV dec^{-1})	β_c (mV dec^{-1})	Corrosion rate x 10^{-2} (mmpy)	Inhibition Efficiency (%)
Blank	9.67	181	63	-116	12.20	-
BTAH						
50	5.02	213	78	-93	6.33	48.11
100	3.43	223	94	-82	4.33	64.51
150	1.49	263	126	-59	1.88	84.59
200	1.50	265	122	-62	1.89	84.51
BTPA						
50	4.83	210	83	-86	6.09	50.05
100	3.21	218	99	-73	4.05	66.80
150	1.34	257	131	-56	1.69	86.14
200	1.37	258	130	-58	1.73	85.83
BTAP						
50	4.22	208	87	-85	5.32	56.36
100	2.87	217	103	-69	3.62	70.32
150	0.98	251	139	-53	1.24	89.87
200	1.02	252	136	-56	1.28	89.45
BTPP						
50	4.03	230	92	-82	5.08	58.36
100	2.63	229	107	-66	3.32	72.70
150	0.82	208	145	-45	1.03	91.56
200	0.83	188	142	-48	1.05	91.39

Electrochemical Impedance Spectroscopic Studies

Electrochemical impedance spectroscopic technique is a powerful tool in the investigation of the corrosion and adsorption phenomena. This method explains effectively the corrosion and passivation phenomena of metals and alloys. The impedance diagrams represented in Nyquist plot obtained at open-circuit potential after one hour immersion in natural seawater in the presence and absence of benzotriazole derivatives are presented in **Figure 4**. The percent inhibition efficiency (IE %) of Al-brass was calculated as follows [22]:

$$\text{IE}\,\% = \frac{(R_{ct})^{-1} - (R_{ct(inh)})^{-1}}{(R_{ct})^{-1}} \times 100$$

Where, $R_{ct(inh)}$ and R_{ct} are the charge-transfer resistance values with and without inhibitors respectively.

The calculated parameters obtained from equivalent circuit fitting analysis in the absence and presences of different concentrations of inhibitors in natural seawater are given in **Table 3**. The variation of R_{ct} values with different concentrations of inhibitor are presented in the **Figure 5**.

These Nyquist plots obtained for Al-brass in the presence of different concentrations of inhibitor exhibited different shapes, which indicated the change in corrosion mechanism due to the presence of inhibitors. It can be seen from the table that with increase in inhibitor concentration, R_{ct} value increased and C_{dl} value decreased. The decrease in C_{dl} value can result from a decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, suggests that the benzotriazole derivatives function by adsorption at the metal-solution interface [23]. The decrease in C_{dl} value upon increase in inhibitor concentration was due to reduced access of charged species to the brass surface, as inhibitor has adsorbed and a good persistent layer of the same was formed on the Al-brass surface. The change in R_{ct} and C_{dl} values was caused by the gradual replacement of water molecules by the anions of the NaCl present in seawater and adsorption of the organic inhibitor molecules on the metal surface, reducing the extent of dissolution [24].

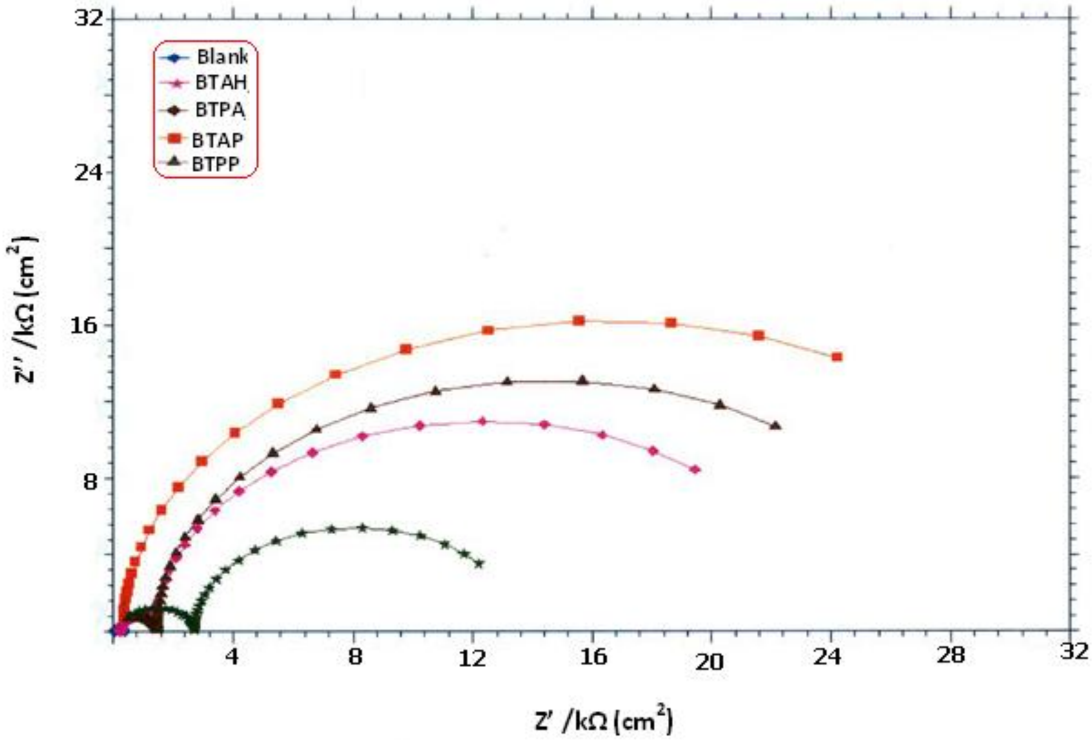


Figure 4 Nyquist diagram of Al-brass in solution containing optimum concentrations of HMBT, natural sea water

Table 3 Electrochemical impedance data of Al-brass in natural seawater containing different concentrations of BTAH, BTPA, BTAP and BTPP

Inhibitor concentration (ppm)	R_{ct} x 10^4 (Ω cm^2)	C_{dl} (μF cm^{-2})	R_F x 10^4 (Ω cm^2)	C_F (μF cm^{-2})	IE (%)
Blank	0.242	6.427	0.143	9.438	-
BTAH					
50	0.491	2.326	0.456	3.168	50.70
100	0.709	0.751	0.872	0.935	65.86
150	1.745	0.096	1.894	0.137	86.13
BTPA					
50	0.502	2.178	0.496	2.853	51.79
100	0.736	0.684	0.932	0.713	67.11
150	2.134	0.082	2.463	0.094	88.65
BTAP					
50	0.576	1.873	0.568	2.275	57.98
100	0.824	0.527	1.213	0.592	70.63
150	2.562	0.069	2.916	0.071	90.55
BTPP					
50	0.598	1.684	0.632	1.943	59.53
100	0.913	0.406	1.451	0.478	73.50
150	3.380	0.051	4.453	0.057	92.83

The value of Faradic resistance (R_F) increases with increase in concentration showing that BTAH, BTPA, BTAP and BTPP stabilize the corrosion products on the metal surface. As the concentration of the inhibitor increases, the inhibitors get adsorbed effectively on the Al-brass surface which increases the R_F values and decreases the C_F values.

The molecular structure normally determines the type of adsorption on brass surface [25]. Benzotriazole derivatives viz., BTAH, BTPA, BTAP and BTPP show differences in their inhibition efficiency due to the difference in their molecular structures. Of the benzotriazole derivatives studied, BTPP has the highest inhibition efficiency and this corresponds well with the polarization measurements. The results obtained from EIS measurements are in good agreement with that obtained from both potentiodynamic polarization and gravimetric measurements.

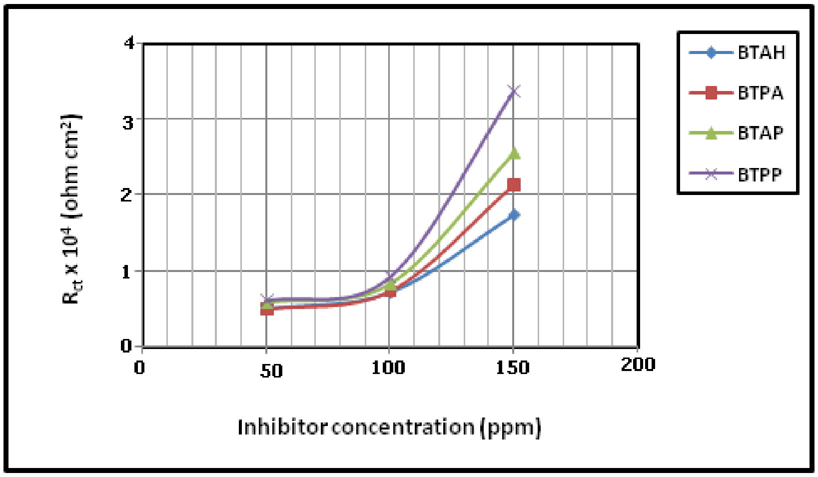


Figure 5 Variation of R_{ct} values with different concentration of BTAH, BTPA, BTAPand BTPP in natural sea water

Potentiostatic Current Transient Techniques

Figure 6 shows the current-time relationship of the Al-brass specimen in natural sea water with and without the optimum concentration of BTAH, BTPA, BTAP and BTPP at the applied potential of -100 mV. During the initial 60 seconds, there was an abrupt decrease in the current and a slower decrease thereafter. After one minute there was no remarkable change in current and a steady value was obtained. Evidently, the intensity of metal dissolution was comparatively low in the presence of inhibitors. Among the inhibitors studied, BTPP shifted the corrosion current of Al-brass to a lower value and thus effectively retards the metal dissolution in natural sea water.

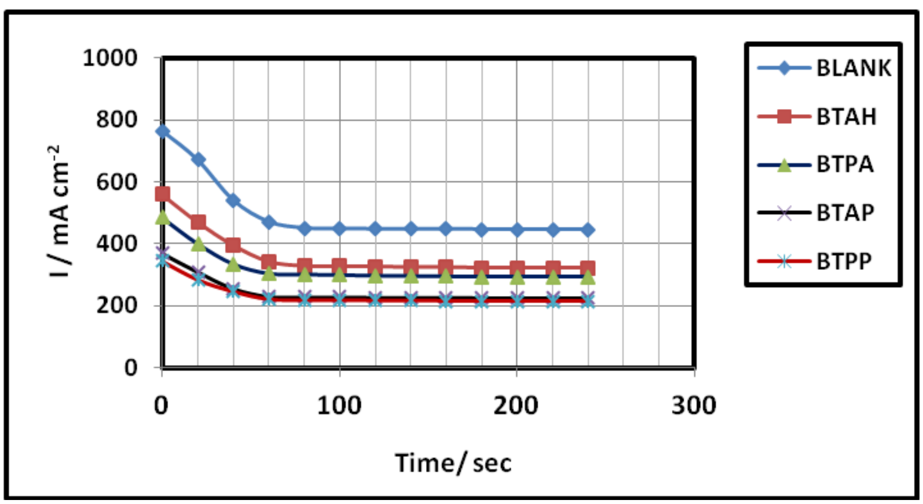


Figure 6 Potentiostatic current transient curves of Al-brass in natural sea water containing optimum concentration of BTAH, BTPA, BTAP and BTPP

Scanning Electron Microscopy

In **Figure 7A** shows the SEM image of Al-brass surface after immersion in natural sea water for 15 days. The micrograph reveals that the surface is highly damaged in absence of the inhibitor. In Figure7 B, C, D and E shows the image of another Al-brass surface immersed in natural sea water for the same period in the presence of BTAH, BTPA, BTAP and BTPP. From this micrograph, it is clear that all the studied inhibitors exhibit a good protective film on Al-brass surface. This is attributed to the involvement of the compounds in the interaction with the active sites of the metal surface. This results shows that an enhanced surface coverage of the metal so that there is a decrease in the contact between metal and the aggressive medium.

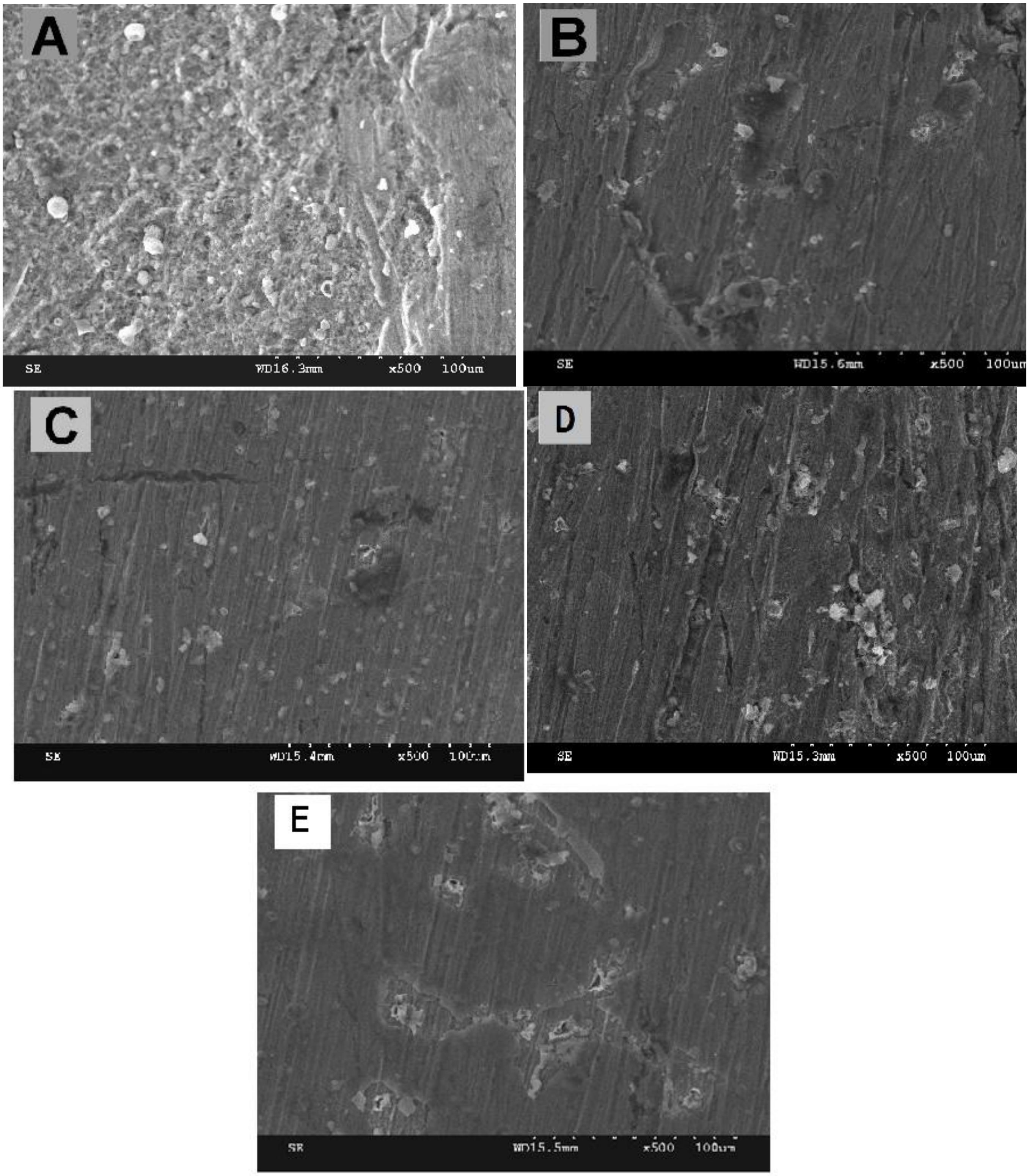


Figure 7 SEM micrographs of Al-brass immersed in natural sea water with BTA derivatives (A) Blank, (B) BTAH, (C) BTPA, (D) BTAP and (E) BTPP

Conclusions

- The benzotriazole derivatives show an inhibiting effect on Al-brass alloy corrosion in natural sea water. From the gravimetric measurements, it is showed that the inhibition efficiency increases with the increase of inhibitor concentration.
- Potentiodynamic polarization curves show that benzotriazole derivatives inhibit both anodic and cathodic reactions at all concentration, which indicate mixed type inhibitor.
- Electrochemical impedance spectroscopy results indicate that as the additive concentration increases, the charge transfer resistance increases whereas double-layer capacitance decreases.
- Current transient studies showed that the inhibitors shifted the corrosion current to a lower value and thus effectively retard the dissolution of Al- brass.
- SEM micrographs clearly proved that the inhibition is due to the formation of an insoluble stable film through the process of complexation of the organic molecules on the metal surface.

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