RESEARCH ARTICLE

EVALUATION OF NEW CLASS OF BENZOTRIAZOLE DERIVATIVES AS CORROSION INHIBITOR FOR BRASS-MM55 IN NATURAL SEA WATER BY ELECTRO ANALYTICAL TECHNIQUES

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Abstract

The corrosion behavior of brass-mm55 in the presence of two organic inhibitors that belong to the benzotriazole derivatives namely N-[(benzylidene hydrazino)-propyl]-benzotriazole (BPBT) and N-[(4-oxo-2-phenyl-1,3-thiazolidineimino)-propyl]-benzotriazole(OPBT) has been investigated in natural sea water. Weight-loss measurements, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were applied to analyze the effect of the organic compounds on the corrosion inhibition of brass. Polarization studies showed that these inhibitors were found to act as mixed type for brass in sea water. It suppresses the cathodic and anodic reactions rates and it renders the open circuit potential to more noble directions. Changes in the impedance parameters (charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl})) are related to adsorption of organic inhibitors on the metal surface, leading to the formation of a protective film. Solution analysis was used to calculate the dezincification factor. All studies clearly show that the substituted benzotriazoles inhibits the corrosion of brass in natural seawater and the value of inhibition efficiency increases with increasing concentration of inhibitors.
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Keywords: brass-mm55, sea water, polarization, impedance spectroscopy, ICP-AES, benzotriazole, derivatives, dezincification..

Introduction

In recent times, there has been much interest in the Copper-based alloys have a long history of service in marine environments. In general, they exhibit an attractive combination of properties, e.g., good machinability, good resistance to corrosion and bio-fouling and superior thermal and electrical conductivities (Karpagavalli and Rajeswari 1999). In view of their good machinability, they are available in a wide range of products. The most prominent among the copper-based alloys is the Cu-Zn alloys which are widely used for condenser and heat exchanger tubes in various cooling water systems (Moliere et al., 1990). Brass is susceptible to a corrosion process known as dezincification and this tendency increases with increasing zinc content of the brass (Warraky 1997; ,Kolotyrin 1980). During the past decade, many techniques have been used to minimize the dezincification and corrosion of brasses. One of the techniques for minimizing corrosion is the use of inhibitors. The effectiveness of the inhibitor varies with its concentration, the corrosive medium and the surface properties of the alloy. Many inhibitors have been used to minimize the corrosion of brass in different media. Particularly, heterocyclic organic compounds containing nitrogen, sulphur and/or oxygen atoms

are often used to protect metals from corrosion. Among them, azoles have been intensively investigated as effective copper corrosion inhibitors. Benzotriazole (BTA), for example, has been studied and found to have excellent inhibition properties in several corrosive environments (Zhang et al.,2004). This molecule. The effectiveness of BTA has been related to theformation of a [Cu+ BTA_]n film and the film formed is considered to be insoluble and polymeric. Fenelon and Breslin (2001) studied the formation of BTA surface films on Copper, Cu-Zn alloy and Zn in chloride solution. The electrochemical impedance data were consistent with the formation of a polymeric BTA-containing layer for all three systems. Both ex situ and in situ (Hashemi and Hogarth 1988) spectroscopic techniques attribute the inhibitive action of these triazoles mainly to the formation of an insoluble protective layer of copper-triazole polymeric complex (e.g. $(Cu(I)BTA)_n$ in case of BTAH) and also to their chemisorption on the metal surface. Aramaki et al (1991) proposed the inhibition mechanisms of benzotriazole derivatives on copper in sulphate solution at several values of pH by an impedance technique and surface enhanced Raman scattering spectroscopy. Protective films of cuprous complex polymers of the inhibitors were readily formed on the copper surface at high pH values and noble potential. Tromans and Silva (1997) studied the anodic behaviour of copper in chloride/Tolyl triazole and chloride/benzotriazole solution. Both inhibitors exhibited adsorption behaviour in the active region whose copper was dissolving as cuprous chloride complex systems ($CuCl_2$). Adsorption of Tolyl triazole was more pronounced had more effect on the apparent Tafel slope. At higher potentials, both inhibitors caused on active to passive transition by reacting with $CuCl_2^-$ to form films of cuprous tolyl trriazole (CuTTA) and cuprous benzotriazole (CuBTA) on an oxide free surface. Nagiub and Mansfeld (2001) studied the corrosion behaviour of 26000 brasses in artificial sea water using EIS and ENA techniques. BTA, gluconic acid sodium salt and polyphosphoric acid sodium salt were evaluated as corrosion inhibitors.   Zhang et al (2004) investigated the protective action of bis-(1-benzotriazolymethylene)-2,5-thiadizoly)-disulphide on copper in chloride media and concluded that the inhibitors effectively control corrosion. El-Azhar et al (2001) investigated the corrosion inhibition of mild steel by the new class of inhibitors [2,5-bis(n-pyridyl)-1,2,4-thiadiazoles] in acid media using weight loss and electrochemical impedance spectroscopy. Results obtained reveal that these compounds are mixed

type inhibitors and behave better in 1M HCl than 0.5M H_2SO_4. Shih and Tzou et al (1991) studied the effect of benzotriazole on the stress corrosion cracking and the electrochemical polarization of 70/30 brass in fluoride solutions. Brass is polarized cathodically in the presence of BTA, which is considered as a cathodic inhibitor.

Experimental details

Materials

The composition of brass-mm55 used in the present study is given in Table 1. The brass alloy specimens were taken from the same brass-mm55 alloy sheet. The chemical composition of the seawater is analyzed by analytical technique, whose composition is given in Table 2. The pH of the seawater is 6.8. The inhibitors N-[(benzylidene hydrazino)-propyl]-benzotriazole (BPBT) and N-[(4-oxo-2-pheny l-1,3- thiazolidineimino) - propyl]-benzotriazole(OPBT) were synthesized according to the reported procedures (Adesh Dubey et al.2011) and their structures are shown in the scheme 1.

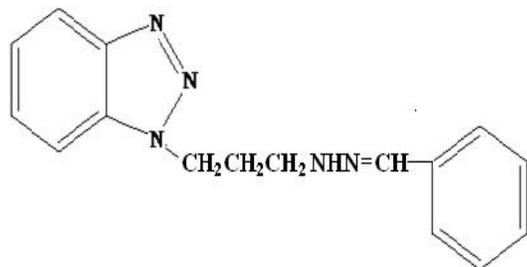### Synthesis of N-[(benzylidene hydrazino)-propyl]-benzotriazole (BPBT)

A mixture of N-(hydrazino propyl)-benzotriazole (2g, 0.01 mol) and benzaldehyde (1.11g, 0.01 mol) in methanol (20 mL) in the presence of a catalytic amount of glacial acetic acid was refluxed for 5.3 hrs. The solvent was removed under reduced pressure to and the resulting crude product was purified by passing it through a chromatographic column packed with silica gel using chloroform: methanol (8:2 v/v) as eluant. Resulting purified product was recrystallized by chloroform to give compound.

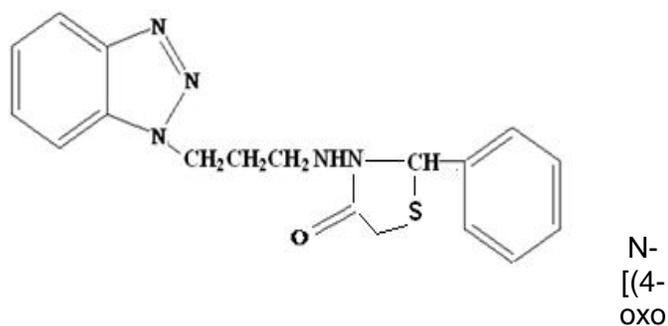### Synthesis of N-[(4-oxo-2-phenyl-1,3-thiazolidineimino)-propyl]-benzotriazole (OPBT)

A mixture of compound N-[(benzylidene hydrazino)-propyl]- benzotriazole(BPBT) (1g, 0.003 mol) and SHCH2COOH(0.331g 0.003 mol) in methanol (20 mL) containing a pinch of anhy. ZnCl2 was first stirred for about 2 hours followed by refluxing on a steam bath for about 6 hrs. The recation mixture was cooled and excess of solvent was evaporated under reduced pressure. The solid crude product was purified by passing it through a chromatographic column packed with silica gel using chloroform: methonal (8:2v/v) as eluant and again purified by recrystallisation from ethanol to

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give compound N-[(4-oxo-2-phenyl-1,3-thiazolidineimino)-propyl]-benzotriazole.

Scheme 1.

N-[(benzylidene hydrazino)-propyl]- benzotriazole (BPPT)

N-[(4-oxo-2-phenyl-1,3-thiazolidineimino)-propyl]-benzotriazole(OPBT)

Table 2.1 Chemical composition of brass–mm55 (in wt.%)

Alloy	Cu	Zn	Mn	Fe	Al	Pb, Ni, Cr, As, Co & Sr
Composition	55	39.34	4	1	0.58	Trace amounts

Table 2.2 Composition of natural sea water

Species	Concentration (mg/ lit)
Dissolved oxygen	0.62
Chloride	29,974
Sulphate	543
Bicarbonate	76
Phosphate	0.59
Silicate	4.52
Nitrate	0.623
Nitrite	0.04

Methods

Weight-loss measurements

The experiments were carried out with brass specimens of dimension 4 cm x 2.5 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, dried and weighed. Then the panels were immersed in 300 ml of artificial sea water with and without the addition of inhibitors. After immersion for a definite period (45 days) the panels were taken out, washed with distilled water, dried and the changes in weights were noted. Triplicate measurements were carried out for each experiment.

Polarization studies

The polarization studies were carried out with brass specimen having an exposed area of 1 cm^2. The cell assembly consisted of 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 a Vibrant potentiostat/ galvanostat model No. VSM/CS/30 at a scan rate of 1mV/s.. The degreased working electrode was 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 (Ravichandran and Rajendran, 2005). The cathodic and anodic polarization curves for brass specimen in the artificial sea water with and without various concentrations of the inhibitors were recorded between -500 to 500 mV at a scan rate of 1 mV/s. The inhibition efficiencies of the compounds were calculated from corrosion current densities using the Tafel extrapolation method.

Electrochemical Impedance Spectroscopic (EIS) Studies

Electrochemical impedance spectroscopic (EIS) studies were carried out at open circuit potential using a potentiostat/galvanostat (Model PGSTAT 12, AUTOLAB, The Netherlands B.V.) with frequency response analyzer (FRA). Brass specimens with an exposed surface area of 1 cm^2 were used as the working electrode. The impedance measurements were carried out at an open circuit potential (OCP), after 30 min immersion of the brass electrode in the corrosive medium. The impedance spectra were acquired in the frequency

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range of 100 KHz – 50 mHz with a 10 mV amplitude sine wave generated by a frequency response analyzer.

In Nyquist plot, the imaginary component (-Z'' ohm, out-of-phase) of impedance was plotted as a function of the real component (Z' ohm, inphase). The impedance spectra were modeled by fitting these data with an equivalent circuit. The software {EQUIVCRT} modeled by B. A. Boukamp was used in this study for EIS data modeling. The impedance spectra were fitted using a non-linear least square (NLLS) fitting procedure for brass in the absence and presence of inhibitors in natural seawater.

Solution analysis by inductively coupled atomic emission spectroscopy (ICP-AES)

ICP-AES is a spectroscopic technique with moderate to low detection limits (0.2-100 ppb). Samples are introduced into the system through a nebuliser with argon gas and are dissociated into its constituent atoms and ions. These are then excited by the plasma and a characteristic radiation is emitted for each element as it falls back to the ground state. The intensity of the emission is proportional to the concentration of the element and quantitative analysis is carried out by reference to calibration curves.

The elements detected by ICP-AES in this work are shown below along with the characteristic wavelengths chosen for each element viz Cu and Zn at 325 nm and 214 nm respectively. The concentration of Cu and Zn in the electrolytes, after the polarisation experiments in the presence and absence of 300 ppm studied inhibitors, was determined by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICPAES). An ICPAES (ARCOS from M/s. Spectro, Germany) was used to measure the amount of dissolution of zinc and copper from the brass surface. The dezincification factor (z) was calculated using the equation (Marshakov, 2005).

$$z = \frac{(Zn / Cu)_{soln}}{(Zn / Cu)_{alloy}}$$

where, $[C_{Zn}/C_{Cu}]_{sol}$ and $[C_{Zn}/C_{Cu}]_{alloy}$ are the ratios between the concentrations of zinc and copper in the solution and in the alloy respectively.

Results and discussion

Weight-loss method

The corrosion rates and inhibition efficiencies of brass with different concentrations of BPBT and OPBT in artificial sea water at room temperature (30°C) are given in Table 3.The inhibition efficiency increases with increase in concentration of the inhibitors. The maximum IE% of each compound was achieved at 300ppm, and a further increase in concentration showed only a marginal change in the performance of the inhibitor. The optimum of concentration of the inhibitors was 300ppm and OPBT was superior to BPBT.

It is well known that the inhibitive action of organic compound containing S, N and/or O is due to the formation of a co-ordinate type of bond between the metal and the lone pair of electrons present in the additive. The tendency to form co-ordinate bond and hence the extent of inhibition can be enhanced by increasing the effective electron density at the functional group of the additive. In aromatic or heterocyclic ring compounds, the effective electron density at the functional group can be varied by introducing different substituents in the ring leading to variations of the molecular structure.

Based on the results, both BPBT and OPBT showed better inhibition efficiencies, due to the presence of heteroatom such as N, O, S and electrons on aromatic nuclei. When compared to BPBT, OPBT showed highest inhibition efficiency, which may be due to the presence of S atom and high molecular weight and possessed by the OPBT molecule. The higher inhibition efficiency of the organic compounds are due to the basis of donor-acceptor interactions between the   electrons of the inhibitor and the vacant d-orbital of copper surface or an interaction of inhibitor with already adsorbed chloride ions.

Potentiodynamic Polarisation Studies

The potentiodynamic polarisation curves of brass in natural seawater in the presence and absence of different concentrations of BPBT and OPBT are shown in Figure 1 and 2. Electrochemical kinetic parameters obtained by extrapolation of Tafel lines are presented in Table 4. The presence of different concentrations of BPBT and OPBT reduce the anodic and cathodic current densities, and the suppression in current increases as the inhibitor concentration increases; this indicates the inhibiting

Table 3. Inhibition efficiency at different concentrations of BPBT and OPBT of brass-mm55 in natural sea water

Inhibitor concentration (ppm)	Corrosion rate x 10^{-2} (mmpy)	Inhibition efficiency (%)
Blank	13.94	-
BPPT		
100	6.78	51.36
200	4.12	70.44
300	1.56	88.81
400	1.61	88.46
OPBT		
100	5.21	62.62
200	2.63	81.13
300	0.94	93.25
400	0.96	93.11

Table 4. Electrochemical parameters and inhibition efficiency for corrosion of brass-mm55 in natural sea water containing different concentrations of BPBT and OPBT

Inhibitor Concentration (ppm)	E_{corr} (mV vs. SCE)	b_a /mV (dec)$^{-1}$	b_c /mV (dec)$^{-1}$	I_{corr} /~A cm^{-2}	Corrosion Rate / mmpy x 10^{-2}	Inhibition Efficiency / %
Blank	-344	59	-126	9.93	12.53	-
BPBT						
100	-275	74	-96	4.92	6.21	50.45
200	-248	89	-64	3.38	4.26	66.00
300	-227	116	-47	1.23	1.55	87.61
400	-221	123	-52	1.24	1.56	87.51
OPBT						
100	-258	84	-87	4.07	5.13	59.01
200	-241	97	-56	2.76	3.48	64.95
300	-214	128	-41	0.84	1.06	91.54
400	-209	125	-44	0.86	1.08	91.34

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effects of the two substituted BTA compounds. It can be shown from the table that with increasing inhibitor concentration, corrosion current density (I_{corr}) decreases and inhibition efficiency (IE) increases.

The values of cathodic Tafel slope bc and anodic Tafel slope ba were found to change with increasing inhibitor concentration, which indicated that the inhibitors control both anodic and cathodic reactions. The inhibitors act as relatively mixed type for brass (Fouda et al 2010). The slight shifts of E_{corr} values towards positive direction are found in the presence of various concentrations of the substituted benzotriazole derivatives in natural seawater. Small changes in the corrosion potential (Ecorr) values are due to the result of the competition of the anodic and the cathodic inhibiting reactions, of the brass surface condition.

The inhibition efficiency (IE) was calculated using the following equation (Ravichandran et al., 2003).

$$I.E.\% = \frac{I_{corr} - I_{corr(inh)}}{I_{corr}} \times 100$$

Where $I_{corr(inh)}$ and I_{corr} are corrosion current density in the presence and absence of inhibitors respectively.

The inhibiting property of the studied BTA derivatives is attributed to their ability to chemisorb on the surface of brass forming few layers of self-assembled films, as sulphur containing compounds are reported to form self-assembled protective layer on the surface of brass. The highest inhibition efficiency was observed for OPBT as it has additional sulfur atom with lone pair of electrons. The values of inhibition efficiency increase with increase in inhibitor concentration, indicating that a higher surface coverage was obtained in a solution with higher inhibitor concentration. It was confirmed that with increase in inhibitor concentration, the corrosion rate decreases. The corrosion inhibition efficiency of OPBT is higher than that of BPBT.

Electrochemical Impedance Spectroscopic Studies

The corrosion behaviour of brass in natural seawater in the presence of BPBT and OPBT is investigated by the EIS method at 303 K after immersion for 1 h. The Nyquist plots of brass in natural seawater in the presence and absence of

different concentrations of BPBT and OPBT are shown in Figure 3 and 4. The Nyquist plots are significantly changed on addition of inhibitors, the impedance of the inhibited system increased with inhibitor concentration. The parameters obtained by fitting the equivalent circuit (Fig. 5) and the calculated inhibition efficiency are listed in Table 5. The impedance spectra of brass in the presence inhibitors showed a capacitive behavior throughout the measured frequency range. The most pronounced and highest R_{ct} was observed for OPBT. R_{ct} increases with increasing concentration of all inhibitors. It can be seen that by increasing the concentration of BTA derivatives, C_{dl} values tend to decrease and the inhibition efficiency increases. The decrease in the C_{dl} values is due to the adsorption of studied inhibitors on the metal surface. The decrease in C_{dl}, which result from local dielectric constant decrease and/or an increase in the thickness of the electrical double layer, suggest that these molecules act by adsorption on the metal/solution interface. It is clear that R_{ct} increases and C_{dl} decreases as the inhibitor concentration increases. The decrease in C_{dl} could be attributed to the adsorption of the inhibitor, forming protective adsorption layer (Babic-Samardzija et al., 2005). A high charge-transfer resistance is associated with a slower corroding system (Hassan et al., 2007). It is clear that the highest values of R_{ct} observed for OPBT than BPBT, suggesting the enhanced inhibitor performance.

The faradaic resistance that linked to the redox reaction involving corrosion process increases and the faradaic capacitance decreases simultaneously with increase in concentration of all the inhibitors. From this it can be concluded that the corrosion products are less susceptible to redox process with increase in concentration of the inhibitors and give better protection efficiency to brass surface. It can be seen that R_f values increased and C_f values decreased for all inhibitors. It is attributed to the increase of true surface area which is partly due to the formation of the corrosion products and also to the roughening of electrode surface. The value of resistance and capacitance involved here are very high and it may be related to the redox process taking place at the electrode surface. The R_f increased and C_f decreased, may be due to the blocking of the surface. The inhibition efficiency increased with increase in concentration for all the inhibitors. The comparative study of the corrosion inhibition of BTA-type organic compounds indicates that OPBT has much significant effect. The investigations proved that the effectiveness of the
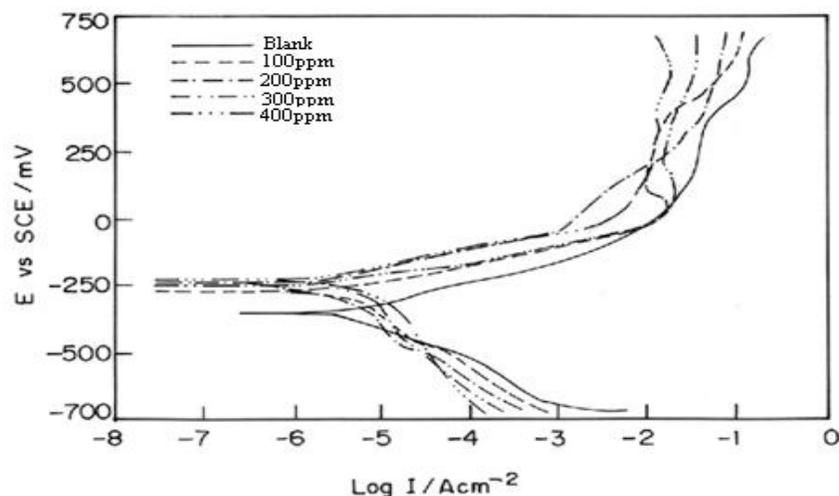
Fig. 1. Polarisation curves of brass-mm55 in natural sea water containing different concentrations of BPBT
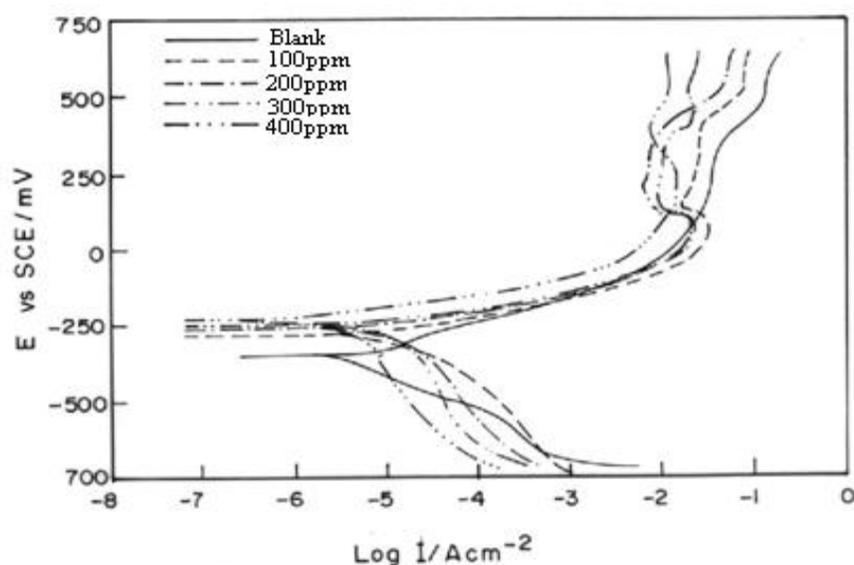

Fig. 2. Polarisation curves of brass-mm55 in natural sea water containing different concentrations of OPBT

Table 5. Impedance Measurements of brass-mm55 in natural sea water containing different concentrations of benzotriazole derivatives after 1 hr immersion.

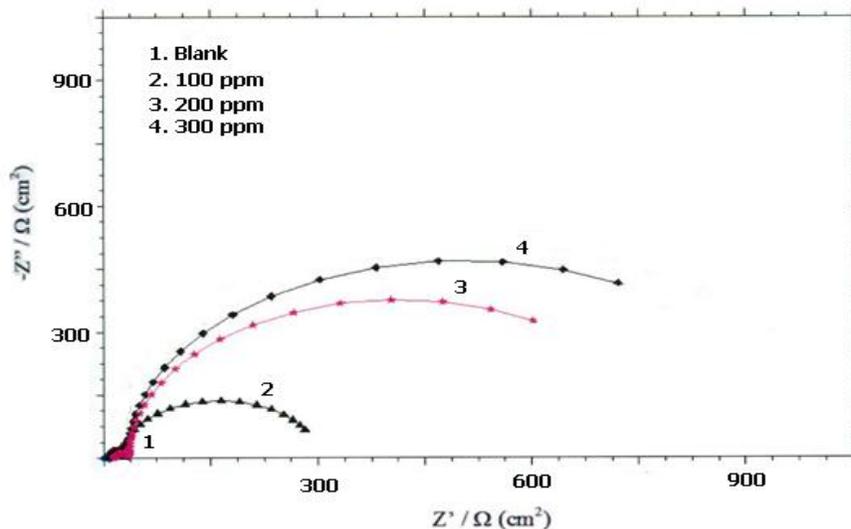| Inhibitor Concentration(ppm)	R_{ct} x 10^4 (ohm cm^2)	C_{dl} (µ Fcm^{-2})	R_f	C_f	Inhibition Efficency (%)
Blank	1.42	0.84	0.03	22.54	-
BPBT					
100	3.28	0.25	12.64	0.425	56.70
200	10.64	0.095	143.7	0.062	86.65
300	14.38	0.068	193.4	0.046	90.13
OPBT					
100	3.75	0.18	16.32	0.315	62.13
200	13.28	0.076	167.8	0.052	89.31
300	18.83	0.052	230.7	0.028	92.46

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Fig. 3. Nyquist diagrams of brass-mm55 in natural sea water containing different concentrations of BPBT after immersion of 1h.
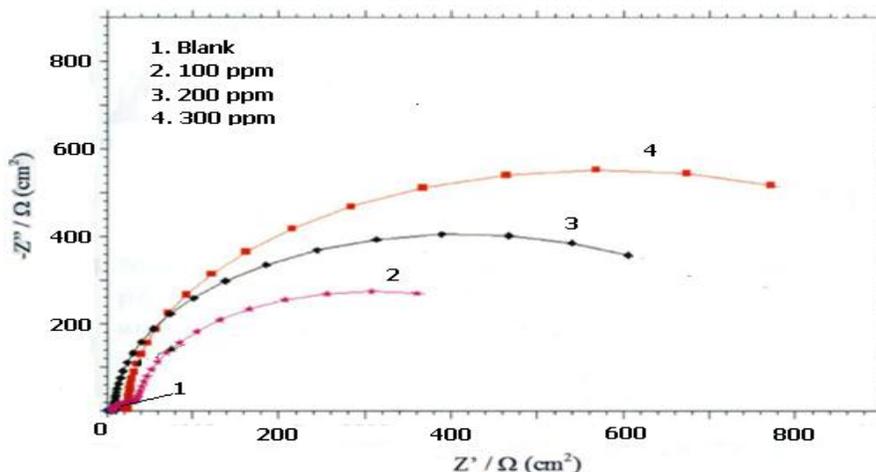

Fig. 4. Nyquist diagrams of brass-mm55 in natural sea water containing different concentrations of OPBT after immersion of 1h.
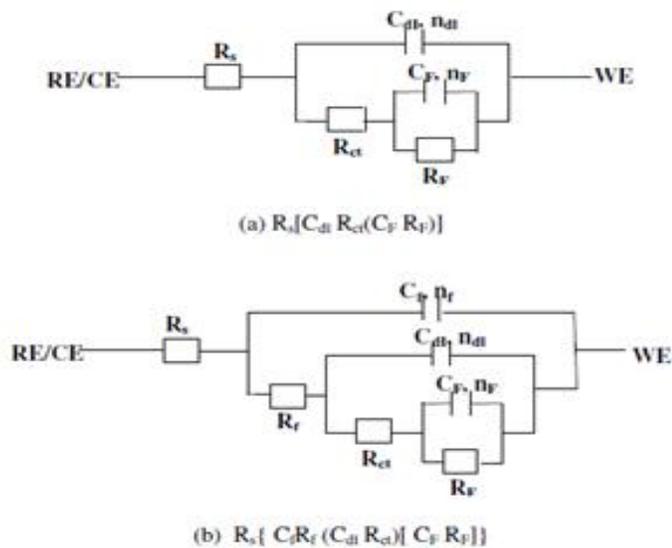

Fig. 5 Equivalent circuit diagrams for brass in (a) absence and (b) presence of

inhibitors

Table 5. Effect of optimum concentrations of BPBT and OPBT on the dezincification of brass-mm55 in natural sea water.

Inhibitors	Solution analysis		Dezincification factor (z)	Percent inhibition	
	Cu (ppm)	Zn (ppm)		Cu (%)	Zn (%)
Blank	0.78	20.63	36.98	-	-
BPBT	0.21	2.27	15.11	73.07	88.99
OPBT	0.085	0.64	10.53	89.10	96.90

BTA derivatives as corrosion inhibitors depends on their molecular structure, particularly on their molecular size and electronic effects of the substituent in the molecule. The IE% calculated from EIS show the same trend as those estimated from polarisation measurements.

Solution analysis by inductively coupled atomic emission spectroscopy

The concentrations of copper and zinc in solutions containing 300ppm of the BTA derivatives after polarisation measurements were determined from inductively coupled plasma atomic emission spectroscopic (ICP-AES) analysis. The dezincification (z) factors for brass in the absence and presence of 300ppm of BPBT and OPBT in natural seawater were calculated from the ICP-AES data and the results are given in Table 5.

The results showed that both copper and zinc were present in the electrolyte in very small quantities and the copper to zinc ratio was found to be lesser than that of the bulk alloy. This is due to the surface barrier arising out of the growth of surface film of inhibitor on the metal surface as well as the corrosion product Cu_2O and ZnO. It is clear from the table that dezincification was much higher in the absence of inhibitors, while dezincification was much lower in the presence of 300ppm concentration of BPBT and OPBT. This indicated that the BPBT and OPBT were able to minimize the dissolution of both Zn and Cu. These values correlate with the corrosion rate and inhibition efficiency obtained by electrochemical methods.

Conclusion

1. Both **BPBT** and OPBT show good inhibition efficiency in natural sea water. The %IE of OPBT was higher than that of BPBT.

2. The polarization data indicate suppression of both the partial corrosion processes in the presence of BPBT and OPBT. These inhibitors behave as mixed type. They decrease the anodic reaction rate more strongly than the cathodic reaction rate and renders the open circuits potential of brass more positive in natural sea water.
3. The inhibitors easily adsorb on the brass surface at the corrosion potential and form a protective complex with the Cu (I) ion, controlling brass from corrosion.
4. Electrochemical impedance spectroscopy shows that R_{ct} values increase, while C_{dl} values decrease in the presence of BPBT and OPBT.
5. Solution analysis reveals that the BPBT and OPBT excellently prevent the dezincification of brass in natural sea water.

Acknowledgement

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