

# Contribution to a Comparative Study of the Corrosion Inhibiting Effect of Some Azoles, to Protect the Cu70-30Ni Alloy in Aerated NaCl 3% in Presence of Ammonia

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## ABSTRACT

Some azoles were tested such as 3-amino-1,2,4-triazole (ATA), 3-4'-bitriazole -1,2,4 (BiTA) and 2-Mercaptobenzimidazole (MBI) against Cu-30Ni alloy corrosion in 3%NaCl polluted by ammonia using potentiodynamic measurements and electrochemical impedance spectroscopy and non-electrochemical techniques (scanning Electron Microscopy (SEM)) studied surface morphology has been used to characterize electrode surface. This study permitted to follow the evolution of the inhibitory effect of some azoles, on Cu-30Ni alloy corrosion in 3% NaCl polluted by ammonia and indicate that the tested inhibitors act as a good mixed-type inhibitor retarding the anodic and cathodic reactions. An increase of the inhibitors concentration leads to a decrease of corrosion rate and inhibition efficiency increase.

**Keywords:** Cu-30Ni Alloy, Electrochemical Impedance Spectroscopy, Ammonia, Inhibition, Azole

## 1. Introduction

Organic inhibitors are largely used with success to protect copper and copper alloys against corrosion. Many papers reported that heterocyclic compounds, such as azole, reveal marked inhibition efficiency [1-12]. In a neutral chloride medium, Otmacic and Stupnišek-Lisac examined the inhibiting effect of non-toxic imidazoles derivatives, and showed that those containing phenyl groups showed a better performance [7]. El Issami *et al.* studied the inhibiting effect of triazoles (triazole, amino-triazole and diamino-triazole) in hydrochloric acid, and they found that the diamino-triazole has the best performance [8]. For substituted uracils with two azole radicals, the grafting of thiol groups increased substantially the inhibiting effect [9]. Huynh *et al.* examined the alkyl esters of carboxybenzotriazole in sulphate medium at different pH. In weakly acidic to neutral solutions, they stated the formation of polymeric complex that hinders corrosion of copper [10]. Laachach *et al.* confirmed also that 3-amino-1,2,4-triazole (ATA) exhibits an ex-

cellent protective effect on the corrosion of Cu-30Ni in NaCl solution because of the complex film with copper oxide and triazoles formed at the metal surface increases its stability [11,12]. These experiments were performed in the absence of pollutants on copper electrode. Es-Salah *et al.* examined the inhibiting effect of ATA and Bitriazole (BiTA) on the corrosion of Cu-30Ni alloy in 3% NaCl in presence of ammoniac, and they found that when the ATA and BiTA were added in the solution, a remarkable inhibiting effect was observed when its concentration is higher than 1 mM [13,14]. 2-Mercaptobenzimidazole (MBI), as an inhibitor, has been studied on the inhibition of mild steel in sulphuric acid solution [15], steel in hydrochloric acid [16,17], copper and its alloys in hydrochloric acid [18], mild steel in phosphoric acid [19].

Cu-Ni alloys are frequently employed in marine applications. Al-Hachem and Carew studied the influence of pollutants such as chlorine and ammonia at low concentration (5 ppm) in natural seawater. Both ammonia

and chlorine increase slightly the corrosion rate of Cu-30Ni [20]. Macdonald *et al.* [21,22] investigated the corrosion of this alloy in the deoxygenated seawater in presence of sulphide. They showed that the presence of sulphide accelerates the corrosion rate of this alloy. In contrast, Syrett *et al.* [23] showed that sulphide increased the corrosion rate in presence of oxygen only. Other authors [24-26] studied the effect of ammoniac. They remarked that this pollutant favours a selective dissolution of Cu-Ni alloys through the formation of copper complexes. In fact, copper-ammoniac complexes make unstable the surface layer constituted of corrosion products, which generally protect the alloy against the corrosion [27,28].

The present investigation discusses the results obtained in studying the inhibiting effect of 2-Mercaptobenzimidazole (MBI), 3-4'-bitriazole -1,2,4 (BiTA) and 3-amino-1,2,4-triazole (ATA), against Cu-30Ni alloy corrosion in 3% NaCl polluted by ammoniac. The tested compounds are presented in **Figure 1**.

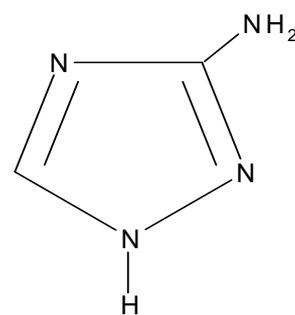
## 2. Experimental Conditions

A classic electrochemical cell with three-electrode configuration was used in this study: a platinum grid as a counter electrode, a rotating disk of Cu-30Ni as working electrode, and Ag/AgCl in 3 M KCl (SSE) as a reference electrode. All potentials in this paper are referred to this later electrode.

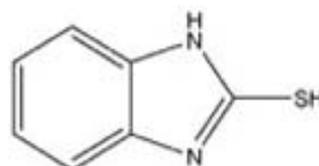
The working electrode was made of cylinder rod of Cu-30Ni (Goodfellow) of 12.5 mm in diameter. A cylinder rod of about 1 cm height was fixed to a stainless-steel shaft, and then the lateral part was covered with a cathaphoretic epoxyamine base paint (PPG; WT724 + P962). First, the paint was deposited at a constant voltage of 180 V during 4 min, and then cured at 180°C for 30 min. After that, the electrode was embedded into an epoxy resin (Buhler; Epoxyure), and worked out to the cylinder shape, the outer diameter of which was 21 mm. Only the cross-section of the alloy rod embedded in the epoxy resin was used to form a rotating disk electrode. The cathaphoretic coating allowed avoiding any infiltration of electrolyte between the metal and epoxy resin interface. Just before each experiment, the electrode surface was polished by emery-paper up to 1000 grade.

The corrosion test solution was prepared with de-ionized water and reagent grade chemicals: 3 wt% NaCl + 0.05 M NH<sub>4</sub>OH + 0.05 M NH<sub>4</sub>Cl. The pH of this buffer solution was 9.25. MBI and ATA (Fluka; Purum) were used as received without any special reaction or transformation, BiTA was synthesized.

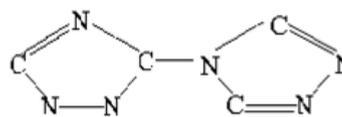
The potentiodynamic measurements were performed using a PGP201 potentiostat/galvanostat. The electrode was immersed for 30 min in the test solution in free cor-



3-amino-1,2,4-triazole (ATA)



2-Mercaptobenzimidazole (MBI)



3-4'-bitriazole -1,2,4 (BiTA)

**Figure 1. Compounds investigated.**

rosion conditions. Impedance (EIS) measurements were done using an EG & G apparatus (model 6310), with a frequency interval ranging from 100 kHz to 10 mHz.

The surface morphology of the electrode was examined with a scanning electron microscope (SEM; Leica Stereoscan 440).

## 3. Experimental Results and Discussion

In the context of a study published elsewhere [13], we limit ourselves here to have the comparative results at 1mM of each inhibitor.

The morphology and corrosion products covering the electrode surface were first analyzed by SEM in absence and in presence of examined inhibitors. Then, the kinetics of corrosion inhibition was examined with polarization curves and electrochemical impedance spectroscopy (EIS).

### 3.1. Surface Film Formed upon Cu-30Ni Surface by Corrosion

**Figure 2(a)** presents the surface morphology after 24 h of immersion in ammonia containing NaCl 3% solution in absence of inhibitors (reference solution). It can be seen that the alloy surface is covered by spongy corro-

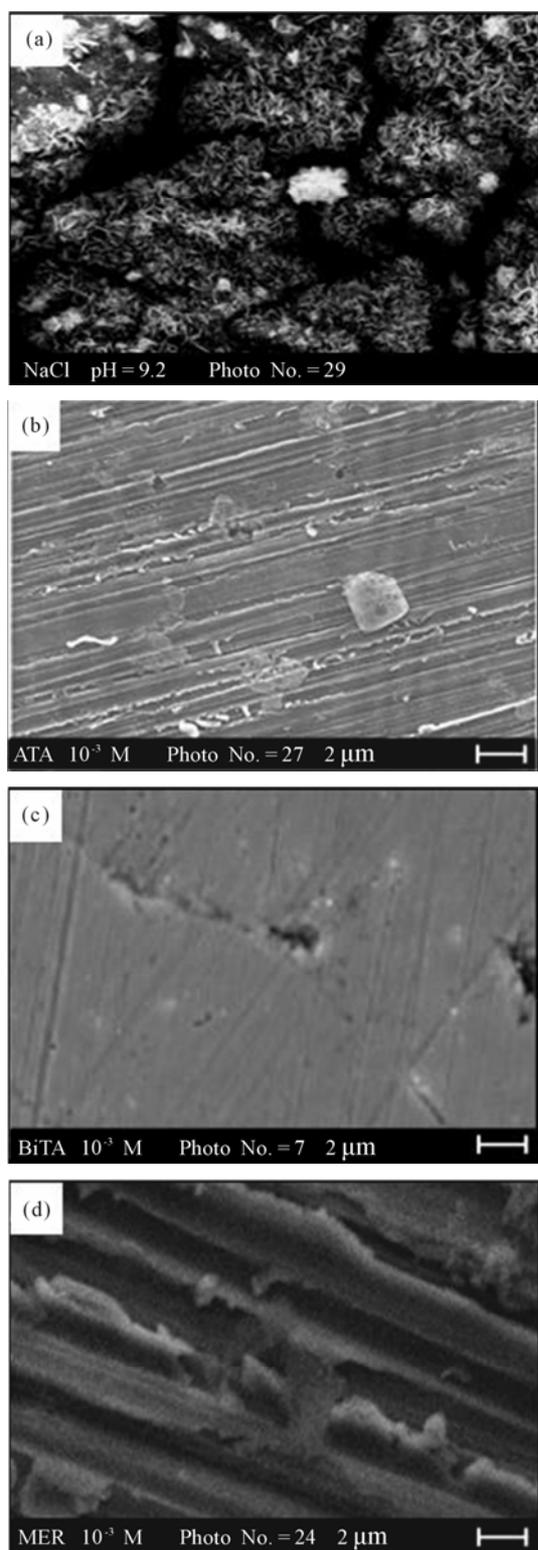


Figure 2. SEM picture of Cu-30Ni electrode surface after 24 h immersion in 3% NaCl pH 9.25 (corrosion test solution), (a) Blank, (b) ATA, (c) BiTA, (d) MBI.

sion products. In contrast, in presence of inhibitors (**Figures 2(b-d)**), almost no corrosion is revealed, and the grooves due to the initial surface abrasion remain clearly visible after 24 h immersion. Some precipitates observed are NaCl crystals appeared because of insufficient surface rinsing. The comparison of these four figures reveals a marked inhibiting efficiency of inhibitors.

The corrosion rate will be evaluated quantitatively first by polarization curves then by electrochemical impedance spectra.

### 3.2. Polarization Curves

Cathodic and anodic polarization curves of Cu-30Ni alloy corrosion in 3% NaCl polluted by ammoniac in the absence and presence of the examined inhibitors, were plotted after 30 min of immersion time at free corrosion potential. The effect of the tested inhibitors was studied. Values of associated electrochemical parameters such a corrosion potential ( $E_{corr}$ ), cathodic Tafel slop ( $b_c$ ), corrosion current density ( $I_{corr}$ ) and inhibition efficiencies ( $E\%$ ) for the tested inhibitors at the concentration  $10^{-3}$  M are given in **Table 1**.

#### 3.2.1. Cathodic Curves

Cathodic curves of Cu-30Ni alloy in aerated 3% NaCl solution polluted by ammonia, without and with the tested inhibitors are shown in **Figure 3**. A small current peak, at  $-0.36$  V versus Ag/AgCl, can be noticed when immersed in the reference solution. This peak may correspond to the reduction of corrosion products formed at the open-circuit conditions before potential sweep [13,29]. In presence of the tested inhibitors we note a decrease of the current density values in the vicinity of the corrosion potential and more particularly in presence of MBI. In the context of a detailed study published elsewhere [13,14], an increase of the inhibitors concentration leads to a decrease of corrosion rate, this decrease is accompanied by the disappearance of the peak raised in the absence of inhibitors. We also note a large domain of linearity indicating a modification of the cathodic process kinetic control.

The corrosion current density was determined by Tafel extrapolation of the current density-potential curve after correction for diffusion, by using the following relation [30,31]:

$$\frac{1}{I} = \frac{1}{I^*} + \frac{1}{I_1} \quad (1)$$

where:

- $I$ : current density at mixed process.
- $I^*$ : corrected current density.
- $I_1$ : limited current density.

**Table 1. Effect of ATA, BiTA and MBI on the electrochemical kinetics of Cu-30Ni in 3% NaCl pH 9.2.**

Solution	$b_c$ (mV/dec)	$E_{corr}$ (mV <sub>Ag/AgCl</sub> )	$I_{corr}$ ( $\mu$ A/cm <sup>2</sup> )	E (%)
Blank	-267	-298	91.3	-
1 mM BiTA	-213	-214	4.5	95
1 mM ATA	-198	-217	1.7	98.1
1 mM MBI	-158	-195	0.1	99.8

The inhibiting efficiency  $E$  in per cent was calculated according to the following expression:

$$E = 100 \frac{i_{corr}^0 - i_{corr}}{i_{corr}^0} \quad (2)$$

where  $i_{corr}^0$  and  $i_{corr}$  stand, respectively, to the corrosion current density without and with inhibitor.

### 3.2.2. Anodic Curves

In the anodic domain (Figure 4), the addition of the tested inhibitors to the corrosion test solution decreases markedly the rate of alloy dissolution. The current peak, observed at -200 mV in absence of the inhibitors, disappears in the presence of the tested inhibitors.

The anodic curves presents a passive domain that extends on 350 mV for MBI, which is clearly observed compared to blank essay. This effect can be explained by the fact that the product tested act by adsorption on the surface of the material and contributes to an establishment of anodic film formation. This passivity is broken with anodic overpressures higher than 160 mV for MBI. This effect can be allotted to the destruction or the desorption of film formed by the examined inhibitors on the surface of the electrode.

Comparatively, the study of these obtained results enabled us to arise the following observations:

- The tested inhibitors inhibit both the anodic and cathodic process. Their effect as a mixed type inhibitors.
- The classification of these inhibitors according to their inhibition efficiency obtained from cathodic and anodic polarizations curves gives:

$$MBI > ATA > BiTA$$

These results make it possible to note an important protective effect of the tested inhibitors against Cu-30Ni alloy corrosion as of the addition of 1mM.

### 3.3. Electrochemical Impedance Spectroscopy (EIS)

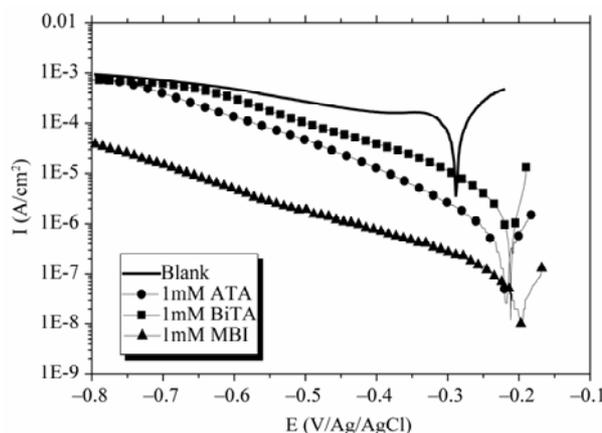
Impedance diagrams were plotted to obtain more information about the electrochemical process which carried at the Cu-30Ni electrode in 3% NaCl solution polluted by ammonia in presence of BiTA, ATA and MBI.

### 3.3.1. Effect of the Tested Inhibitors

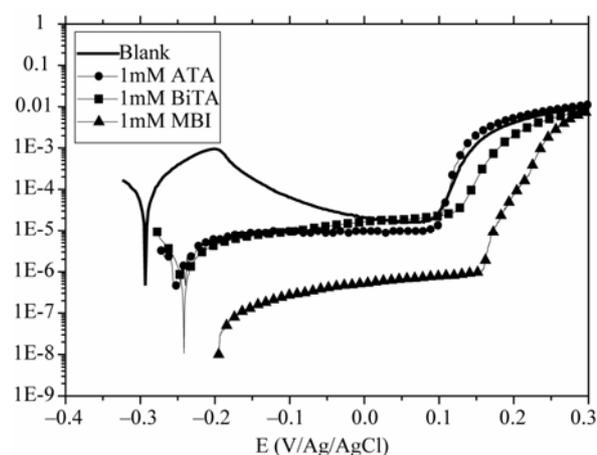
The impedance diagrams in Nyquist plot for different inhibitors are after 30 min of stabilization period presented in Figure 5. The high frequency part of impedance is displayed with an enlarged scale in the insert.

Though not clearly separated, these diagrams may be split into two capacitance loops. However, in contrast to the case of copper electrode [32], with the working electrode, the addition of inhibitors does not induce a new time constant, they remained always two. In the context of a detailed study published elsewhere [14,29], the impedance spectra in absence of inhibitors present two capacitive loops, the first loop can be attributed to a charge transfer process.

In the presence of inhibitors, we note that the imped-



**Figure 3. Cathodic polarization curves of Cu-30Ni in 3% NaCl in presence of ammonia (pH 9.25) without and with the tested inhibitors at the concentration  $10^{-3}$  M,  $\Omega = 1000$  rpm;  $|dE/dt| = 30$  m/Vs.**



**Figure 4. Anodic polarization curves of Cu-30Ni in 3% NaCl in presence of ammoniac (pH 9.2) without and with the examined inhibitors at the concentration  $10^{-3}$  M,  $\Omega = 1000$  rpm;  $|dE/dt| = 30$  m/Vs.**

ance display of the electrode in inhibitors containing the solution changes in shape and size, other it can be noticed that the impedance modulus increased dramatically in presence of tested inhibitors. The presence of two capacitive loops seems to indicate a diffusion contribution to the beginning of the experiment. At high frequency loop, it is found that the charge transfer resistance  $R_t$  value increased in presence of the tested inhibitors, whereas the double layer capacity value found to be decreased (Table 2). The decrease in capacity value was due to the adsorption of inhibitor molecules on the metal surface and act as barrier to the oxygen diffusion process.

### 3.3.2. Effect of the Rotation Speed

Figure 6 presents the impedance spectra in presence of BiTA, ATA and MBI, when the electrode rotation speed is limited to 200 rpm and 1000 rpm after 30 min of immersion. No marked effect of rotation speed. This confirms the appearance of a broad tafelien field in the cathodic domain, when the tested inhibitors were added, on the one hand, and makes it possible to allot the first loop has a charge transfer process. As expected from the effect of rotation speed [13,14,29], the diffusion does not modify the electrode kinetics at low cathodic or at the corrosion potentials.

### 3.3.3. Influence of Time Immersion

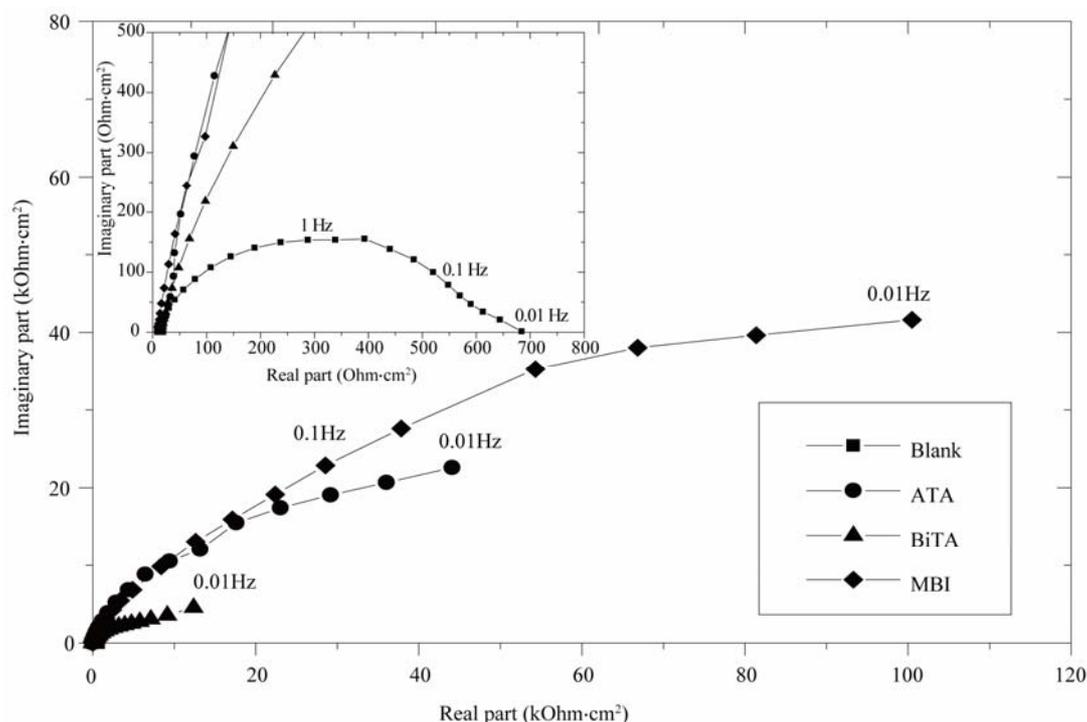
Figure 7 shows the impedance diagrams obtained without and with  $10^{-3}$  M of BiTA, ATA and MBI at the cor-

**Table 2. The results of non-linear values regression for the impedance spectra presented in Figure 5.**

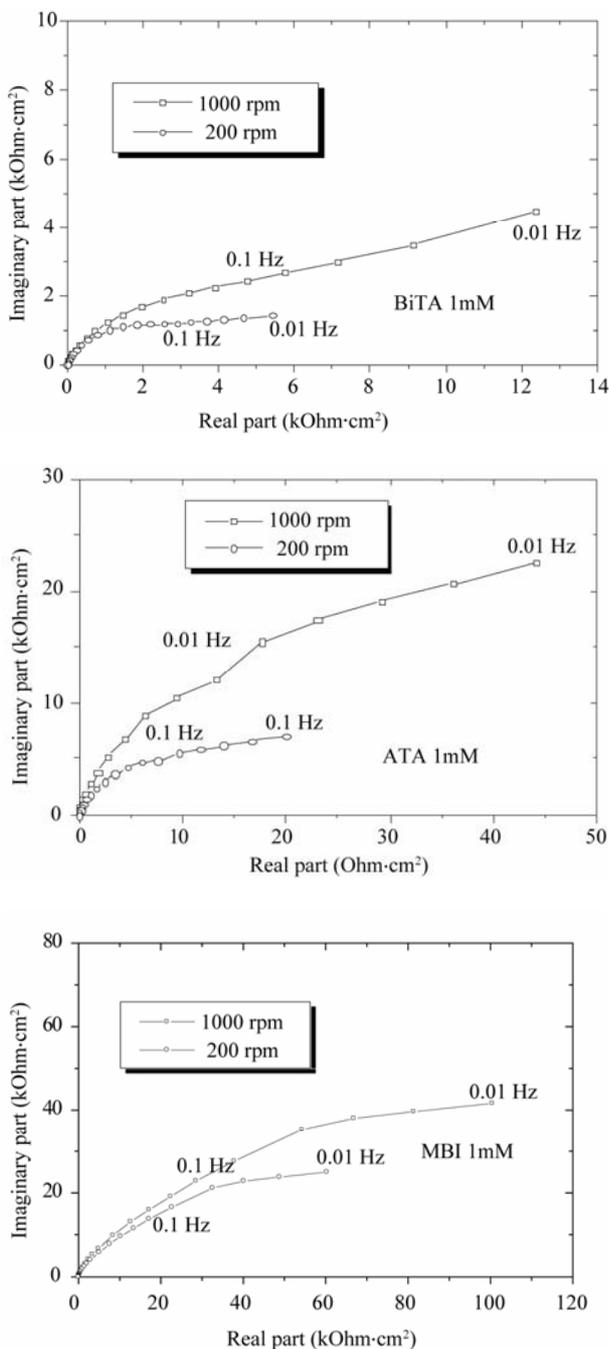
Solution	$R_t$ ( $k\Omega \cdot cm^2$ )	$C_d$ ( $\mu F/cm^2$ )	$E'$ (%)
Blank	0.450	145	-
1 mM BiTA	7.25	87.2	94
1 mM ATA	11.16	17	96
1 mM MBI	34.5	6.6	97

**Table 3.  $R_t$  and  $C_d$  changes with respect to immersion period in the test solution added of 1 mM of BiTA, ATA and MBI for  $\Omega = 1000$  rpm.**

Solution	Time (h)	$R_t$ $k\Omega \cdot cm^2$	$C_d$ $\mu F/cm^2$	E (%)
Blank	0.5	0.450	145	-
	2	0.372	171	-
	4	0.196	123	-
	24	0.177	90.1	-
1 mM BiTA	0.5	4.6	138	90
	2	11.5	43	96.7
	4	12	46	98.3
	24	14	10	98.7
1 mM ATA	0.5	11.2	17	96
	2	17.3	3.3	97.8
	4	21.1	5.3	99
	24	33	1.1	99.4
1 mM MBI	0.5	34.5	6.6	97.1
	2	45.6	3.5	99.2
	4	57.6	4.8	99.6
	24	125	1.3	99.7



**Figure 5. Impedance diagrams of Cu-30Ni in the corrosion test solution in absence and presence of BiTA, ATA and MBI after 30 min of stabilization period.  $\Omega = 1000$  rpm;  $|dE/dt| = 30$  mVs.**



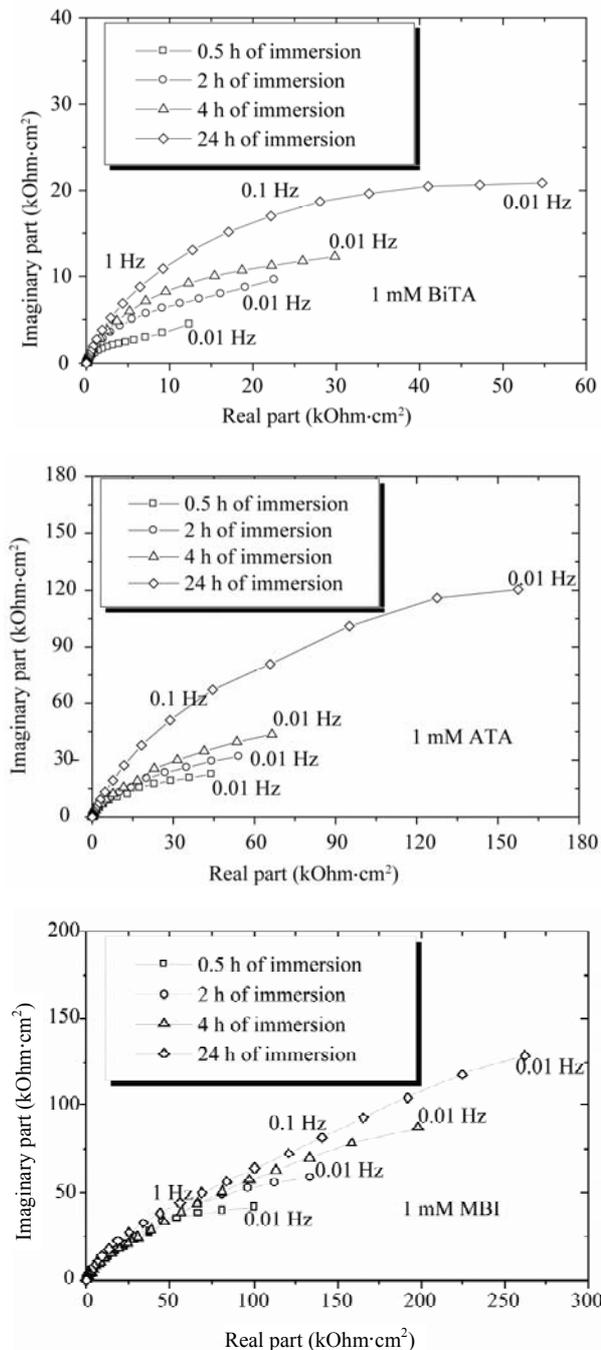
**Figure 6.** Impedance diagrams of Cu-30Ni in the corrosion test solution in presence of BiTA, ATA and MBI after 30 min of immersion at different rotation speed.

rosion potential after the Cu-30Ni was exposed to the solution for different times.

The evolution of the characteristic parameters associated with the capacitive loop with time is summarized in Table 3. The value of the associated capacity was calcu-

lated from the relation  $C = 1/(2\pi fR_t)$ , where  $f$  is the characteristic frequency in the maximum of the loop and  $R_t$  is the diameter of the first capacitive loop.

The various diagrams of Figure 6 show a capacitive behavior of the interface in the field of frequency exam-



**Figure 7.** Impedance diagrams of Cu-30Ni in the corrosion test solution in presence of 1 mM of tested inhibitors exposed for different times.  $\Omega = 1000$  rpm.

values of polarization resistances can be classified as ordered. As can be seen in this figure, the impedance diagrams in the Nyquist plot become larger with time. The increase of the polarization resistance with the immersion period is often reported for the inhibiting action of heterocyclic on copper corrosion [5-7,9,10]. However the ordering is to an order ascending:

$$R_{p(\text{MBI})} > R_{p(\text{ATA})} > R_{p(\text{BiTA})}$$

The capacitive behavior seems is not affected with immersion period. The  $C_d$  values observed in presence of examined inhibitors correspond to those usually allotted to the double layer.

If we interpret the action of the inhibitors by the variation of charge transfer resistance  $R_t$ , the inhibition efficiencies evaluated using the following relation:

$$E = 100 \frac{R_t^i - R_t^0}{R_t^i} \quad (3)$$

where  $R_t^i$  and  $R_t^0$  are respectively charge transfer resistance with and without inhibitor, corroborate those obtained with the stationary curves of polarization.

In the whole cases, the protective effectiveness is higher than 99%. Furthermore, the inhibiting efficiency tends to increase with immersion period. Compared with the solution without ammoniac [11], it was observed that protective effect of inhibitors is reinforced, and the corrosion current becomes smaller in spite of Cu-NH<sub>3</sub> complex formation.

#### 4. Conclusions

The comparative analysis of the results obtained in this work with the examined inhibitors shows that:

The ATA, BiTA and MBI act at the same time on the reactions anodic and cathodic of the process of corrosion. Indeed the cathodic domain is characterized for the examined inhibitors, by decrease of the current density values in the vicinity of the corrosion potential and more particularly in presence of MBI and disappearance of the cathodic peak, recorded in the absence of inhibitors. The anodic polarization curves show a decrease of the current density values with disappearance the current peak recorded in the absence of the examined inhibitors. The values of charge transfer resistances raised of the impedance diagrams show the same evolution in time for all tested inhibitors, but with different values. The calculated inhibiting effectiveness remains important for all inhibitors.

The kinetic effect of the tested inhibitors is two-fold: in one hand, their presence suppresses completely the oxygen evolution reaction, and merely the hydrogen evolution reaction is taking place at the electrode surface.

On the other hand, the latter is reduced at least by one order of magnitude when 1 mM was added into the test solution. What shows that the use of only one compound is very sufficient to ensure an important protection of alloy in the corrosive medium used, that shows that the use of only one compound can be satisfactory to ensure an important protection of alloy in the studied corrosive medium?

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