

NICOTINAMIDE-BASED IONIC LIQUID AS A POTENTIAL COPPER CORROSION INHIBITOR

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Abstract: In this paper, the possibility of using newly synthesized ionic liquid based on nicotinamide as a potential inhibitor of copper corrosion in an acidic sulfate medium was investigated. Inhibition efficiency of butyl-nicotinamide, [C₄Nic]Br on copper corrosion in acidic (pH=3) 0.1 mol/dm³ sodium sulphate solution was analyzed using potentiostatic polarization measurements and electrochemical impedance spectroscopy (EIS). It was found that the investigated ionic liquid had inhibitory properties of the corrosion of copper in acidic media. The inhibitory effect of the test compound is manifested by rapid and spontaneous adsorption of [C₄Nic]Br molecules on the copper surface following Langmuir's adsorption isotherm. As a result, the cathodic corrosion current decreases, as well as the polarization resistance of the system, increases compared with the basic solution. The inhibition efficiency of the investigated compound largely depends on the applied concentration.

Keywords: corrosion, copper, inhibitors, ionic liquid.

1. INTRODUCTION

Corrosion is a spontaneous process of materials degradation or changes in their properties due to a chemical or electrochemical reaction with the environment. The occurrence of corrosion is present in various materials, but it causes the biggest problems with metals, significantly reducing their use-value. The consequences of corrosion are numerous and varied, and their effects on the safe, reliable and efficient operations of equipment or structures are often more serious than the simple loss of metal mass. As a result, the costs of corrosion represent a great economic burden on the economy and, according to the latest estimates of experts in the most developed countries, amount to over 3% of global GDP [1]. Given the above, more and more attention in the world is paid to the reduced degree of corrosion of materials and the costs that corrosion cause, by

applying various methods of corrosion protection. Modern methods of corrosion protection are extremely important because, in addition to bringing great material savings, they also enable the development of other, newer and more modern types of protection of materials from corrosion [2].

Copper is one of the most important industrial materials and, as an excellent conductor of heat and electricity, it has an extremely large application in various fields. Copper, like most of its alloys, exhibits good corrosion resistance in both slightly alkaline and weakly acidic environments. The presence of oxidizing agents, complexing agents and a more aggressive environment leads to the dissolution of copper and it is subject to the corrosion process. The aggressive environment prevents the formation of a protective oxide layer on the copper surface, which contributes to the accelerated dissolution of copper and significant corrosion [3].

Reducing the degree of metal degradation can be achieved by using corrosion inhibitors – substances whose presence in the corrosive environment leads to a reduction in the rate of metal corrosion. Modern science strives to find metal corrosion inhibitors that, in addition to the basic properties required of an inhibitor (high efficiency, temperature and time resistance), also meet increasingly rigorous environmental protection requirements (non-toxicity, biodegradability).

Due to its "green" properties, there is a growing interest in the possibility of using ionic liquids as potential inhibitors of metal corrosion in environments with different aggressiveness, as an alternative to toxic organic and inorganic inhibitors [4-8].

Since copper is an important construction material, the possibility of using an ionic liquid, butyl-nicotinamide, $[C_4Nic] Br$, to protect the copper from corrosion in an acid-sulfate medium was investigated in this paper. Potential inhibition properties of $[C_4Nic] Br$ were investigated using polarization and electrochemical impedance spectroscopy measurements.

2. EXPERIMENTAL

In this paper, the possibility of using the ionic liquid butyl-nicotinamide, $[C_4Nic]Br$, (Figure 1) to protect the copper from corrosion in acid-sulfate medium $c(Na_2SO_4) = 0.1 \text{ mol dm}^{-3}$ at $pH = 3$ was investigated.

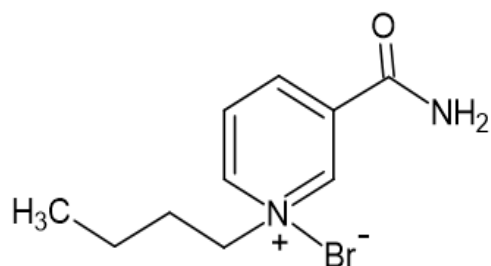


Figure 1. Structure of butyl nicotinamide, $[C_4Nic] Br$

A solution of Na_2SO_4 (0.1 mol dm^{-3}) at $pH = 3$ was used as corrosive medium. Inhibitor solutions were prepared under the same conditions with the addition of butyl nicotinamide in the concentration range of $1 \cdot 10^{-5} \text{ mol dm}^{-3} - 1 \cdot 10^{-3} \text{ mol dm}^{-3}$ by diluting the basic solution ($0.025 \text{ mol dm}^{-3}$) with distilled water and adding the required amount of Na_2SO_4 . In all the tested

solutions, the pH was adjusted to 3 by adding H_2SO_4 dilute. All measurements were performed at 298 K with constant stirring of the solution using a magnetic stirrer.

Electrochemical measurements

A computer-controlled potentiostat, Volta-Lab PGZ 301, was used for electrochemical measurements. The measurements were performed in a three-electrode electrochemical cell. In this system, copper (99.99%) was used as the working electrode, platinum as the counter electrode and the saturated calomel electrode (*SCE*) was used as a reference electrode. Before each measurement, the working electrode was polished with SiC paper (grit sizes of 1200), washed and immersed in the test solution. After a relaxation time of 20 minutes, the corrosion potential of the copper electrode was read, and then the electrode potential was first scanned between corrosion potential and -250 mV/SCE to the cathodic and after that to the anodic direction ($+200 \text{ mV/SCE}$), with a scan rate of 10 mV min^{-1} . Electrochemical impedance measurements were performed at the corrosion potential, in the frequency range $0.01 \text{ Hz} - 10 \text{ kHz}$, with 10 points per decade.

The obtained results were converted into Excel documents and further processed using the Origin 6.1 program. The modelling of the examined electrochemical system was done by applying the nonlinear least-squares method within the Eissa_0.1b program.

3. RESULTS AND DISCUSSION

3.1. Polarization measurements

Potentiostatic polarization measurements were performed at tested ionic liquid's concentration in the range of $1 \cdot 10^{-5} \text{ mol dm}^{-3}$ to $1 \cdot 10^{-3} \text{ mol dm}^{-3}$, at pH value 3 at 298 K. The aim of these measurements was to determine the influence of the concentration of ionic liquid $[C_4Nic]Br$ on its inhibition efficiency as well as to determine the mechanism of its adsorption on the copper surface. Figure 2 shows the polarization curves of copper electrodes recorded in the blank solution ($0.1 \text{ mol dm}^{-3} Na_2SO_4$) and the inhibitor solution ($[C_4Nic]Br$) concentration $1 \cdot 10^{-3} \text{ mol dm}^{-3}$, at pH 3.

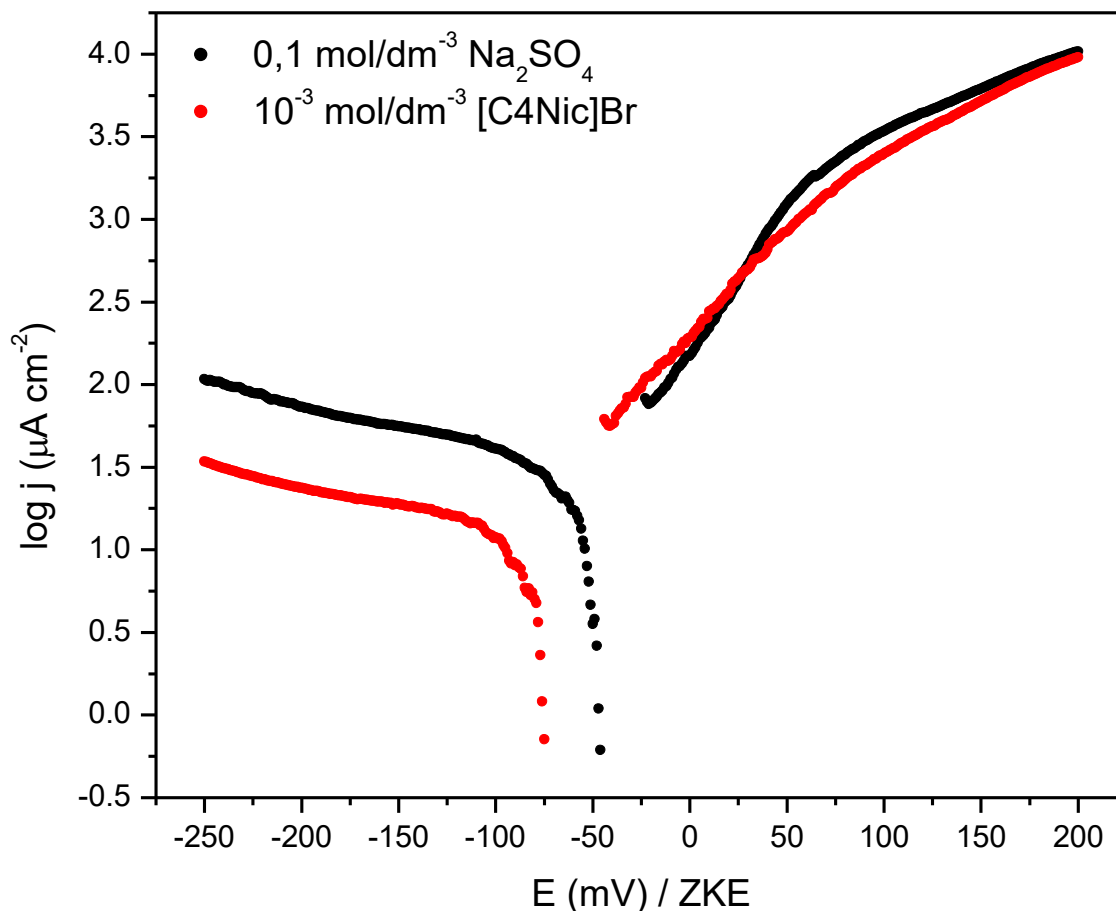


Figure 2. Polarization curves of copper electrodes in Na_2SO_4 solution and $1 \cdot 10^{-3} \text{ mol dm}^{-3}$ inhibitor solution

During the polarization of the copper electrode, the cathode part of the polarization curve describes the process of oxygen reduction, while the anode part refers to the dissolution of the copper under the stated conditions. As can be seen from Figure 2, relative to the blank solution, in the presence of inhibitors the anodic dissolution of copper begins at slightly more negative potential values, while the reduction of oxygen takes place at lower current densities. These results indicate that the applied ionic liquid has an inhibitory effect on the copper corrosion in an acidic environment, whereby $[\text{C}_4\text{Nic}]\text{Br}$ exerts its protective effect on the corrosion of copper mainly by blocking the cathodic reaction of oxygen reduction.

From the obtained experimental data, the corrosion potentials E_{corr} and the corrosion current density j_{corr} were determined using the Tafel extrapolation method. The inhibition efficiency, η ,

of the tested potential inhibitor was calculated based on the difference in current density flowing at the copper electrode in the corrosive and inhibitor solution using the equation:

$$\eta = \frac{j_0 - j}{j_0} \cdot 100 \quad (1)$$

where j_0 and j represent the respective corrosion current densities in the absence and presence of the inhibitor.

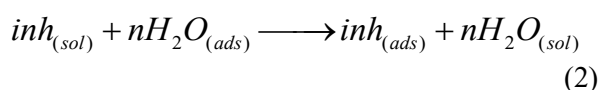
Table 1 shows the obtained experimental results for the corrosion potential, E_{corr} , corrosion current density, j_{corr} , for the blank solution and inhibitor solutions of the applied concentrations, as well as the coverage of the metal surface with adsorbed inhibitor molecules, θ , and degree of inhibition efficiency of ionic liquid, η .

Table 1. Characteristics of polarization curves of copper in the blank solution and the solutions with different inhibitor concentrations

c_{inh} (mol/dm ³)	E_{corc} (mV)	j_{corc} (μA/cm ²)	η (%)	θ
0	-23	32.03	-	-
$1 \cdot 10^{-5}$	-49	27.39	14	0.1449
$5 \cdot 10^{-5}$	-44	23.15	28	0.2774
$1 \cdot 10^{-4}$	-48	19.18	40	0.4010
$5 \cdot 10^{-4}$	-44	16.03	50	0.4995
$1 \cdot 10^{-3}$	-60	15.00	53	0.5316

The data shown in Table 1 indicate that in the presence of an inhibitor there is a slight shift of the corrosion potential to more negative values as well as the inhibitory efficiency of the [C₄Nic]Br depends on the applied concentration. It can be noticed that, with increasing concentration, the inhibition efficiency increases and that the best protection against copper corrosion is achieved at the highest applied concentration of $1 \cdot 10^{-3}$ moldm⁻³.

The organic molecules exert their protective effect on the process of metal corrosion by expelling water molecules and/or ions from inside the electrochemical double layer during their adsorption [9]:



The type of the realized adsorption of the organic molecule on the metal surface depends on several factors: the chemical composition and electronic properties of the inhibitor molecule, the temperature of the corrosion medium, as well as the electrochemical potential on the metal-solution interface, etc. [10, 11]. Under real conditions, it is usually very difficult to determine which of the above factors is most responsible for the type of the achieved adsorption. Theoretically, physical adsorption of the inhibitor molecule is characteristic at

lower temperatures, while chemical adsorption is mainly present at higher temperatures [12].

To find the appropriate adsorption isotherm, i.e., to study the thermodynamic parameters of the adsorption process of [C₄Nic]Br molecules on the surface of the copper electrode, the coverage of the metal surface with adsorbed molecules was first calculated based on the data obtained by potentiostatic polarization measurements [C₄Nic]Br:

$$\theta = \frac{\eta}{100} \quad (3)$$

The obtained values for the copper surface coverage at pH = 3 for different concentrations of the tested inhibitor are shown in Table 1. From the given data, it can be seen that with the increase of the inhibitor concentration, the degree of surface coverage by ionic liquid increases. As expected, the highest degree of coverage was achieved at a concentration of $1 \cdot 10^{-3}$ moldm⁻³.

By processing the obtained experimental results, it was found that the tested inhibitor, [C₄Nic]Br, is adsorbed on the copper surface following the Langmuir isotherm [13, 14]:

$$\frac{c_{inh}}{\theta} = \frac{1}{K_{ads}} + c_{inh} \quad (4)$$

where θ represents coverage of the copper surface, K_{ads} is the equilibrium constant of the adsorption process and c_{inh} is inhibitor concentration.

Figure 3 shows the Langmuir adsorption isotherm (dependence of c_{inh} on the c_{inh}/θ ratio) $[C_4Nic]Br$ on the copper surface at pH 3 in the sulfate medium.

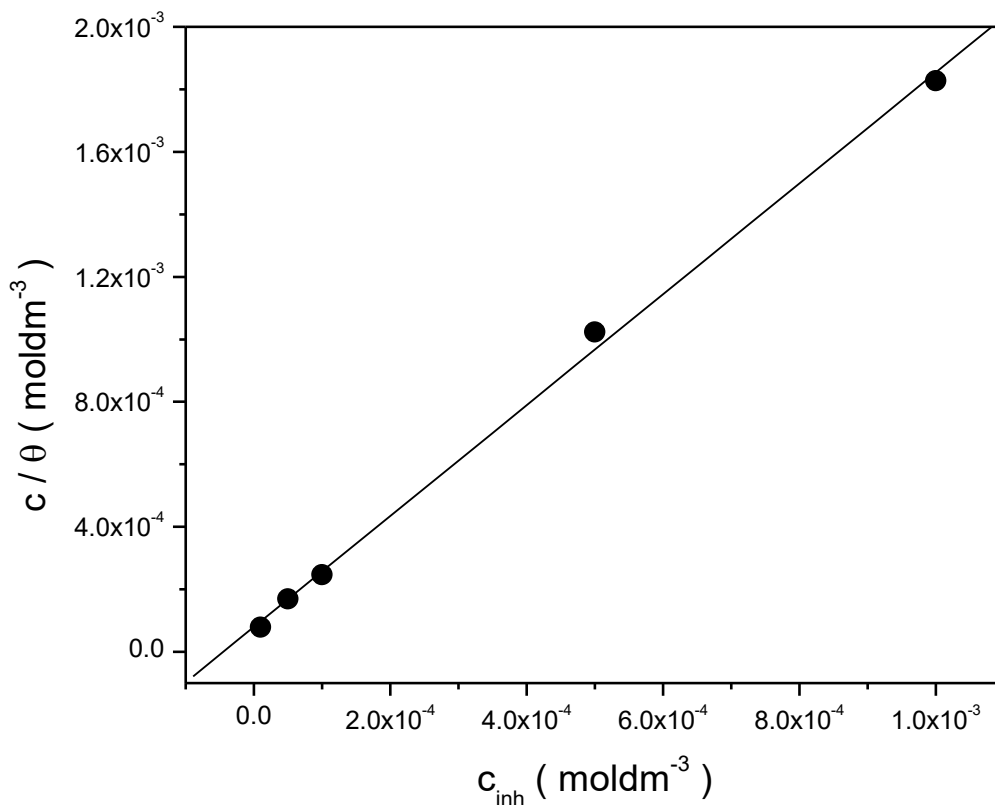


Figure 3. The Langmuir's adsorption isotherm of $[C_4Nic]Br$ on the copper surface

It can be seen from Figure 3 that the obtained dependence is linear, which confirms that the isotherm describes well the adsorption process $[C_4Nic]Br$ on the copper surface. An important thermodynamic parameter of the adsorption process is the free energy of the adsorption, ΔG_{ads} , and it can be calculated by knowing the value of the equilibrium constant of the adsorption process using equation 5 [13,14]:

$$K_{ads} = \frac{1}{55.5} e^{-(\Delta G_{ads}/RT)} \quad (5)$$

Table 2 shows the values obtained for the equilibrium constant of the adsorption process, K_{ads} , regression coefficient, r , calculated based on the Langmuir isotherm as well as the value of the free energy of the adsorption, ΔG_{ads} at a temperature of 298 K.

Table 2. Values of the equilibrium constant of the adsorption process, K_{ads} , regression coefficient, r , and the free energy of the adsorption, ΔG_{ads} for $[C_4Nic]Br$

$\ln K_{ads}$	ΔG_{ads} (kJ/mol)	r
9.54	-33.58	0.9997

As can be seen from the results shown in Table 2, the adsorption constant, K_{ads} , [C₄Nic]Br has a high value, and the free energy of the adsorption, ΔG_{ad} , has a negative value, which indicates the fact that the adsorption of ionic liquid molecules on the copper surface take place quickly, spontaneously with the formation of a stable protective film. The value of the free energy of the adsorption of [C₄Nic]Br molecules may indicate the ongoing type of adsorption. Namely, a value of ΔG_{ads} more positive than -20 kJ/mol indicates that the organic molecule on the metal surface is physically adsorbed, while a value less than -40 kJ/mol is attributed to the presence of a coordination

bond between the metal and the inhibitor molecule [15,16]. The obtained value of ΔG_{ads} for [C₄Nic]Br, -33.58 kJ/mol, indicates that the examined ionic liquid under the given conditions is predominantly bound to the copper surface by physical adsorption.

3.2. Electrochemical impedance measurements

Electrochemical impedance measurements were performed in 0.1 mol/dm³ Na₂SO₄ solution, and in inhibitor solutions, in the concentration range 1·10⁻⁵ mol/dm³ – 1·10⁻³ mol/dm³, at pH value 3, after a relaxation time of 20 minutes (Figure 4).

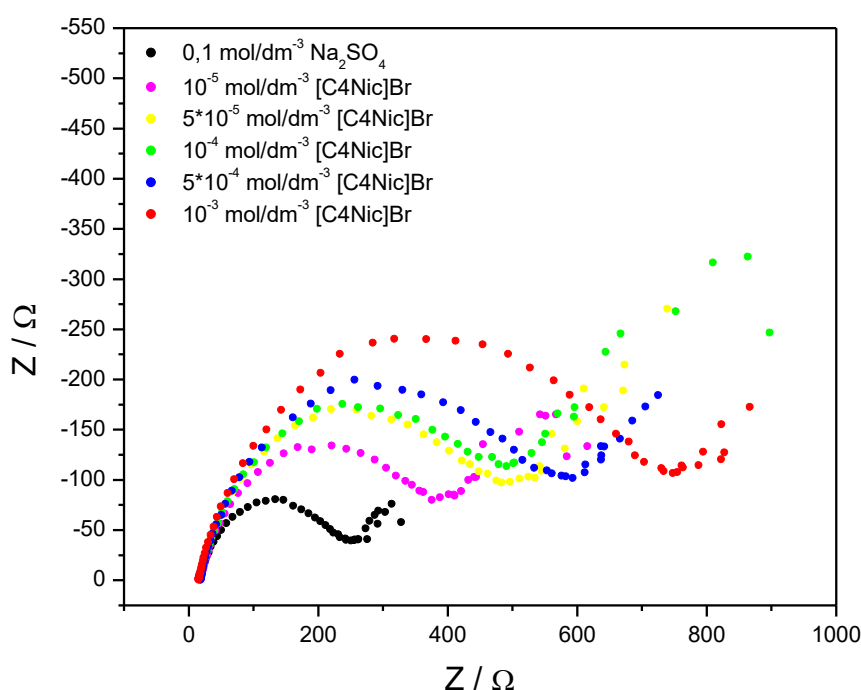


Figure 4. Nyquist plot of the copper electrode in inhibitor solutions of different concentrations and the blank solution

Figure 4 shows that the Nyquist plots in both the basic and inhibitory solutions deviate from the ideal semicircular shape, which is expected based on the literature data for copper in an acidic sulfate medium [17,18]. In a corrosive environment without the presence of inhibitors, in contrast to inhibitor solutions, one semicircle appears on the curve at high frequencies, which is followed by a constant phase angle of 45°, independent of the frequency. This phenomenon is characteristic of systems that are controlled by linear diffusion and can be described by applying the Warburg impedance. Unlike the blank test in the inhibitor solutions, the constant phase angle disappears, which is a sign that the process of dissolving copper

in the presence of inhibitors, unlike the corrosive medium, is no longer diffusion-controlled. On the other hand, in the presence of ionic liquid on the Nyquist plot, another capacitive loop appears. The appearance of the second capacitive loop is a sign that inhibitor molecules form a barrier on the metal surface [19], while the increase of the first capacitive loop with increasing inhibitor concentration indicates that resistance to charge transfer plays a key role in controlling copper dissolution rate [20].

More information on the dynamic processes developing on the surface of the copper electrode in the corrosion and inhibitor solutions can be obtained by modelling the tested electrochemical

system. Figure 5 shows the obtained empirical models of an electrochemical cell in which the physical and chemical properties of the corrosion

system are replaced by the corresponding linear circuit elements for the corrosion solution (Figure 5a) and inhibitor solutions (Figure 5b):

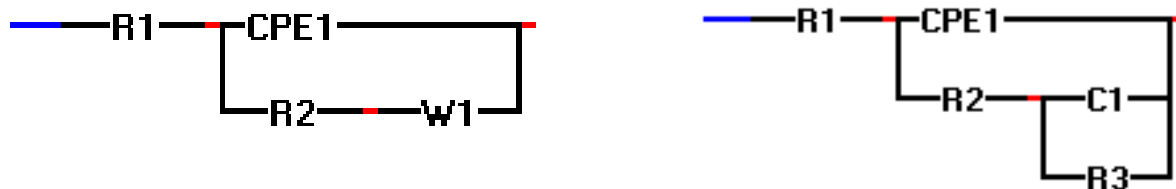


Figure 5. Electrical equivalent circuit for copper electrode (a) in corrosion solution (b) in inhibitor solution

where R_e ($R1$) represents the resistance of electrolyte solution, Q_{ds} ($CPE1$) is the double-layer constant phase elements, R_{ct} ($R2$) is the charge transfer resistance, Z_w ($W1$) represents Warburg impedance, C_{po} ($C1$) is pore capacity of the inhibitor film, R_{po} ($R3$) is resistance to charge transfer through the pores of the inhibitor film.

In modelling, instead of the double-layer capacity, C_{ds} , the double-layer constant phase element, Q_{ds} , was used. Better agreement of experimental data during modelling with a phase constant element than with the capacity of the double layer indicates the presence of inhomogeneity of the electrode surface, which affects the uneven distribution of surface properties of the copper electrode.

Values obtained by modelling the electrochemical system for polarization resistance (R_p),

phase constant element (Q_{ds}), pore capacity of the inhibitor film (C_{po}), charge transfer resistance through the inhibitor film pores (R_{po}), surface inhomogeneity (n) in the basic solution and solutions of the tested ionic liquids at different concentrations and inhibition efficiency (η) are shown in Table 3.

The inhibition efficiency (η) was calculated based on the following equation:

$$\eta = \frac{R_p(inh) - R_p(o)}{R_p(inh)} \cdot 100 \tag{6}$$

where $R_p(o)$ and $R_p(inh)$ represent the charge transfer resistance in the absence and presence of the inhibitor respectively.

Table 3. Impedance parameters and inhibition efficiency of the $[C_4Nic]Br$

C_{inh} (mol dm ⁻³)	Z_w (Ω)	R_p (Ω)	Q_{ds} (F cm ⁻²)	n	η (%)	R_{po} (Ω)	C_{po} (F cm ⁻²)
0	0.9	215	$6.5 \cdot 10^{-2}$	0.80	-	-	-
$1 \cdot 10^{-5}$	-	420	$7 \cdot 10^{-3}$	0.70	49	300	2
$5 \cdot 10^{-5}$	-	530	$7 \cdot 10^{-3}$	0.71	59	320	2
$1 \cdot 10^{-4}$	-	560	$8 \cdot 10^{-3}$	0.70	62	330	2
$5 \cdot 10^{-4}$	-	620	$7 \cdot 10^{-3}$	0.70	65	330	3
$1 \cdot 10^{-3}$	-	780	$7 \cdot 10^{-3}$	0.70	72	330	6

The value of the polarization resistance, R_p , represents a direct measure of the protection of the copper surface from corrosion. A higher value of polarization resistance is a sign of better resistance of the metal surface in relation to degradation processes [21]. The values shown in Table 3 indicate that the presence of the tested ionic liquid leads to an increase in the polarization resistance of the system relative to the blank solution, which is evidence of the tested ionic liquid's inhibitory properties on copper corrosion in acidic sulfate medium. It is also noticeable that the value of the polarization resistance changes as a function of the applied ionic liquid concentration. As expected, with increasing concentration there is an increase in polarization resistance, and thus inhibition efficiency, η . The values obtained for inhibition efficiency calculated based on the polarization resistance using equation 5 are slightly higher than those obtained based on the polarization measurements, which can be attributed to the difference in the applied determination techniques.

Table 3 also shows that the values of the double-layer constant phase element, Q_{ds} , determined in the blank and inhibitor solutions differ by approximately one order of magnitude, with no change in the values of Q_{ds} with a change in the concentration of applied ionic liquid. The phenomenon can be explained by the fact that the presence of ionic liquid leads to changes in the structure of the electrochemical double layer, but it is independent of the applied concentration.

The values of n factors shown in Table 3 indicate that the presence of ionic liquid in the corrosive environment does not lead to an increase in the value of n factors, i.e., to a decrease in the inhomogeneity of the electrode surface.

As can be seen from the data shown in Table 3, the resistance to charge transfer through the pores of the inhibitor film, R_{po} does not depend on the concentration of ionic liquid. This trend can be explained by the compactness of the inhibitory film that forms on the copper surface and does not depend on the concentration of the applied ionic liquid.

It is interesting to note that the values obtained for the pore capacity of the inhibitor film, C_{po} , also do not depend on the concentration at low concentrations of ionic liquid, while there is a significant increase in pore capacity at higher concentrations. This phenomenon can be attributed to the fact that only at higher concentrations of inhibitors a significant amount of ionic liquid is

deposited on the copper surface, which also leads to a significant increase in the pore capacity of the formed film.

4. CONCLUSION

In this work, the inhibitory properties of butyl nicotinamide, $[C_4Nic]Br$ on copper corrosion in an acidic solution of $0.1 \text{ mol dm}^{-3} \text{ Na}_2\text{SO}_4$ at pH 3, were investigated by using potentiostatic polarization measurements and electrochemical impedance spectroscopy.

The results of polarization measurements indicate that the examined ionic liquid has the properties of a corrosion inhibitor of copper under the stated conditions. The inhibition efficiency of the test compound depends on the applied concentration, and it was determined that the best protection against copper corrosion is achieved by applying the highest concentration of inhibitor ($1 \cdot 10^{-3} \text{ mol dm}^{-3}$). Inhibitor molecules achieve a protective function by physical adsorption on the copper surface, which takes place quickly and spontaneously by the mechanism of the Langmuir isotherm, whereby the cathode current density of the corrosion decreases.

The results of electrochemical impedance spectroscopy indicate that in corrosion solution the corrosion rate of copper is diffusion-controlled, while in inhibitor solutions, the charge transfer resistance controls the corrosion rate. As the concentration of the inhibitor increases, the value of the polarization resistance increases, and thus the efficiency of the applied inhibitor.

5. ACKNOWLEDGEMENTS

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ЈОНСКА ТЕЧНОСТ НА БАЗИ НИКОТИНАМИДА КАО ПОТЕНЦИЈАЛНИ ИНХИБИТОР КОРОЗИЈЕ БАКРА

Сажетак: У овом раду испитивана је могућност примене новосинтетизоване јонске течности на бази никотинамида као потенцијалног инхибитора корозије бакара у киселој сулфатној средини. Анализирана је инхибиторска ефикасност бутил-никотинамида $[C_4Nic]Br$ на корозију бакара у киселом ($pH = 3$) раствору натријум-сулфата концентрације $0,1 \text{ mol/dm}^3$, применом поларизационих мерења и електрохемијске импедансне спектроскопије (EIS). Утврђено је да испитивана јонска течност поседује инхибиторске особине на корозију бакара у киселој средини. Инхибиторско дејство испитивано једињење остварује брзом и спонтаном адсорпцијом $[C_4Nic]Br$ молекула на бакарну површину по механизму Лангмирове адсорпционе изотерме, при чему долази до смањења катодне густине струје као и до повећања поларизационог отпора система. Утврђено је да инхибиторска ефикасност испитиване јонске течности у великој мери зависи од примењене концентрације.

Кључне речи: корозија, бакар, инхибитори, јонске течности.

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