University of Belgrade, Technical Faculty in Bor

29th International Conference Ecological Truth & Environmental Research

EcoTER'22

Proceedings

Editor
Prof. Dr Snežana Šerbula

21-24 June 2022, Hotel Sunce, Sokobanja, Serbia

University of Belgrade, Technical Faculty in Bor

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PREFACE

In today's world, the environment has been endangered by the use of outdated technology, fossil fuels and environmental law violations. Therefore, environmental and many other scientists all over the world have been concerned about finding sustainable technology in resolving these issues. That is why environmental research and ecological truth are at the focus of the 29th International Conference Ecological Truth & Environmental Research 2022 (EcoTER'22), which will be held in Sokobanja, Serbia, 21–24 June 2022. On behalf of the Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Conference.

We hope to convey the message of the conference, which is that a transformation of attitudes and behavior would bring the necessary changes. This is also an opportunity for the participants who are experts in this field to exchange their experiences, expertise and ideas, and also to consider the possibilities for their collaborative research.

The 29th International Conference Ecological Truth & Environmental Research 2022 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology, the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica, the University of Zagreb, Faculty of Metallurgy – Sisak, the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Association of Young Researchers, Bor.

These proceedings include 85 papers from the authors coming from the universities, research institutes and industries in 6 countries: Bulgaria, Italia, Albania, Bosnia and Herzegovina, Montenegro and Serbia.

As a part of this year's conference, the 4th Student section – EcoTERS'22 is being held. We appreciate the contribution of the students and their mentors who have also participated in the Conference.

Financial assistance provided by the Ministry of Education, Science and Technological Development of the Republic of Serbia is gratefully acknowledged by the Organizing Committee of the EcoTER'22 conference.

The support of the Platinum donor and their willingness and ability to cooperate have been of great importance for the success of EcoTER'22. The Organizing Committee would like to extend their appreciation and gratitude to the Platinum donor of the Conference for their donation and support.

We appreciate the effort of all the authors who have contributed to these Proceedings. We would also like to express our gratitude to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the Conference participants for their support to EcoTER'22. Sincere thanks go to all the people who have contributed to the successful organization of EcoTER'22.

Prof. Snežana Šerbula,

President of the Organizing Committee

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PURINES AS GREEN CORROSION INHIBITORS

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Abstract

Metals present one of the major groups of materials used for all kinds of different applications in various environments. Often those environments pose a threat regarding the stability and preservation of metals and they undergo corrosion. Corrosion processes lead to several consequences that can have a detrimental effect on the environment such as metals release due to dissolution, leakage of various chemicals that are stored in reservoirs or transported via pipelines. These processes should be diminished, and one of the ways of metals protection is the application of corrosion inhibitors. There is a great number of the potential inhibitors tested, however, some of them have undesired effects on the environment and health. Purines present bioorganic compounds that show a tendency to interact with metals and form complexes that can adsorb on the surface of metal and that characteristic indicates that potentially they can efficiently inhibit corrosion. This is confirmed within numerous studies and those results are presented and discussed. Most of them are done using copper with the addition of purine or adenine, however, other derivatives are studied as well. Common conclusion is that the increase in the number of heteroatoms and molecular size leads to inhibition efficiency increase.

Keywords: corrosion inhibitors, environmentally friendly, purine

INTRODUCTION

Wide world applications of metal materials in various environments, that present media of different degree of corrosiveness, might account for significant release of metals. Exposure of metals to different chemical agents, such as solutions of acids, salts or alkalis, in industrial applications might lead to their corrosion. On the other hand, natural surroundings can also cause similar effects. For instance marine environment is considered to be very corrosive [1,2]. However, having in mind some examples such as offshore wind farms [3], antifouling coatings based on metal oxides [4] etc., the need for metal exploitation in sea water becomes obvious. Moreover, the unintentional depositions of metals in the form of shipwrecks contribute to the metal content as well [5]. Atmospheric corrosion in coastal areas is also significant [6]. Rain water in the urban environment induces release of metals, also the behavior of metals differs in the cases when they are present in bulk and in alloys and that has to be considered when the effects on the environment and health are studied [7]. According to Bertling *et al*. [8] most of the copper that is released to runoff water from patinated copper roofs is retained on solid surfaces and soil [8]. Similar findings are reported by Wallinder *et al*. [9] regarding stainless steel. Consideration of all the above mentioned data points to the fact that the cycle of metals in the environment is very important to study and understand in order to have a clear picture of the amounts of metals that can be found in the environment

and the effect that they can have. High amounts of metals due to dissipative releases can have a significant adverse effects on the environment quality, and on the other hand present the loss of resources. Detailed study regarding copper is presented by Lifset *et al*. [10].

On the other hand, as seen in the paper published by Kirchgeorg *et al*. [3], the corrosion protection systems can also be a source of metals and organic compounds. Already mentioned marine environment requires efficient corrosion protection, however, it should be carefully designed in order to protect environment from polluting releases, and Rossini [4] pointed out the need for more environmentally friendly coatings. When corrosion inhibitor reaches the environment, water, soil etc. it can also have an effect on the growth and development of the living organisms such as plants, and on the uptake of the other present elements. This is clearly presented in the study published by Liu *et al*. [11] regarding the effect of widely used corrosion inhibitor benzotriazole. Hence, numerous eco-friendly corrosion inhibitors are studied by now [12–16].

One of the groups of compounds that can be considered as bioorganic compounds and have a potential to be used as green environmentally friendly corrosion inhibitors is group of purines. Purines are components of various biochemical compounds, among others, they are found in DNA and RNA, so they are present in human body and involved in very important metabolic processes. On the other hand purine and adenine and their derivatives have a tendency to react with metal ions and form complexes. Structures and the atoms that are involved in coordination between metal and organic part of the complexes differ depending on the form of organic molecule since it can be in the form of neutral molecule, anion and cation [17,18]. Based on the possibility to react and form bonds with metals purine and derivatives are considered to have a potential to inhibit corrosion of metals. Another very specific area of metals application is the area of biomaterials. Hence, back from the 80s [19], the metals release from dental biomaterials became an important topic which importance just grow during time [20]. Pourbaix [21] started studying the appearance of corrosion of metals used as biomaterials in 1984, and the research still continues nowadays [22]. One of the applications of copper as biomaterial is in the construction of intrauterine devices (IUD) [23–26]. Due to their origin and characteristics, since some purine and adenine derivatives may be used in various medical treatments [27,28], these compounds are considered as the potential inhibitors of corrosion of metallic biomaterials in real or simulated body fluids, as well [29–32]. The broad area of possible applications and characterization of these compounds as environmentally friendly and non-toxic indicate that they should receive adequate attention.

DISCUSSION

The most often studied compounds belonging to this group are purine and adenine. Their structure is presented in figure 1. They contain imidazole and pyrimidine ring, whereas adenine also has amino group in its molecule. Purine and adenine as copper and brass corrosion inhibitors were studied in several different media such as chloride [33,34], sulfate [35–38] and nitrate [39] solutions. The results can be seen in Table 1 and the mechanism of their action is described as formation of adsorbed protective layer, $[Cu\text{-}INH]_{ads}$, on metal surface.

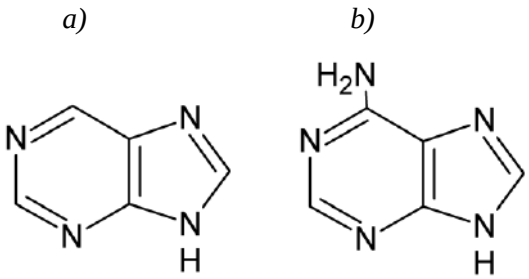

Figure 1 *Molecular structure of a) purine; b) adenine*

In chloride solution reaction mechanism is proposed as follows, depending on the starting form of copper [33,34]:

$$CuCl_{ads} + INH \leftrightarrow [Cu\text{-}INH]_{ads} + Cl^- + H^+ \quad (1)$$

$$Cu + INH - e^- \leftrightarrow [Cu\text{-}INH]_{ads} + H^+ \quad (2)$$

$$nCuCl_2^- + nINH \leftrightarrow (Cu\text{-}INH)_n + nH^+ + 2nCl^- \quad (3)$$

$$[Cu\text{-}INH]_{ads} + (Cu\text{-}INH)_n \leftrightarrow [(Cu\text{-}INH)_m]_{ads}, \text{ where } m = n+1. \quad (4)$$

The proposed model of inhibitor action includes adsorption via Cu-N bond or interaction of π electrons of aromatic ring with Cu or electrostatic interactions enhanced by adsorption of negatively charged chloride ions. More detailed adsorption mechanism is provided later based on new theoretical findings.

Table 1 *Copper and brass corrosion inhibition efficiency obtained using purine and adenine*

Inhibitor	Concentration, M	Medium	Inhibition efficiency, %	Reference
Purine	$1\cdot10^{-2}$	1 M NaCl	76[a]	[33]
Adenine	$1\cdot10^{-2}$	1 M NaCl	92[a]	[34]
Purine	$1\cdot10^{-2}$	0.5 M $NaNO_3$	90[a]/91[b]	[39]
Adenine			91[a]/96[b]	
Purine	$1\cdot10^{-2}$	0.5 M Na_2SO_4 pH 6.8	91[b]	[35]
Adenine			94[b]	
Purine		0.5 M Na_2SO_4 pH 1.0	78.6[b]	
Adenine			88.1[b]	
Purine	$1\cdot10^{-2}$	0.5 M Na_2SO_4 pH 6.8	76[a]	[36]
Adenine			91[a]	
Purine	$1\cdot10^{-2}$	0.5 M Na_2SO_4 pH 7.0	91.08[a]	[37]
		0.5 M Na_2SO_4 pH 9.0	88.88[a]	
Purine	$1\cdot10^{-2}$	0.5 M Na_2SO_4 pH 7.0	85.2[a]	[38]
		0.5 M Na_2SO_4 pH 9.2	91.0[a]	

Inhibition efficiency values obtained using: [a] potentiodynamic polarization, [b] weight loss.

Petrovic *et al*. [40] studied purine (PU), adenine (AD) and 6-benzylaminopurine (BAP) as copper corrosion inhibitors in weakly alkaline solution containing chloride ions (0.1 mol dm^{-3} $Na_2B_4O_7$, 0.05 mol dm^{-3} NaCl). The experimental results, presented in the Table 2, indicated that the increase in molecular size and weight leads to the increase of the inhibition efficiency. Since the molecules studied may have different forms depending on the pH value of the media it has to be considered when the mechanism of the interaction with metals surface is

proposed. According to Scendo [33], in weakly alkaline media PU is in the form of neutral molecule, whereas AD and BAP can also be found in anion form [34,39,41]. In that case adenine tends to react with Cu(I) ions and complex Cu(I)-adenine can be formed [42]. Hence, the proposed mechanism can be one of the following:

$$Cu^{+} + INH_{(ads)} \leftrightarrow [Cu(I)\text{-}INH]^{+} \quad (5)$$

$$Cu^{+} + INH^{-}_{(ads)} \leftrightarrow Cu(I)\text{-}INH \quad (6)$$

Analysis of the data regarding sulfate solutions presented in Table 1 confirms the conclusion that inhibition efficiency depends on the pH value. Purine and adenine show superior protection in neutral media, however they can be efficiently used also in acidic [35] or weakly alkaline solutions [37]. The mode of adsorption can be described by Langmuir adsorption isotherm [34–39]. In this media reaction mechanism can be proposed as follows [37]:

$$4Cu + 4PU_{(ads)} + O_2 \leftrightarrow 4Cu(I)\text{-}PU + 2H_2O \quad (7)$$

Both inhibitors influence cathodic and anodic reactions so they are considered to be mixed type inhibitors. The look of the brass surface, Figure 2, after polarization without and with the addition of purine can elucidate the need for inhibitor application and proper choice of inhibitor.

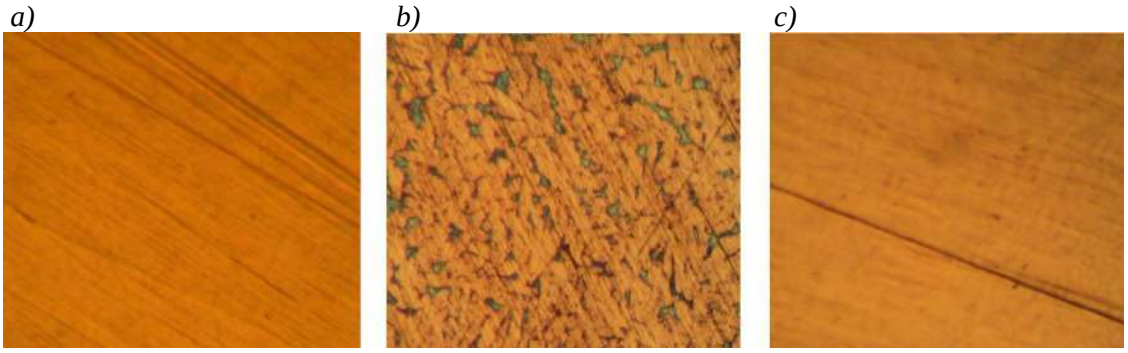

Figure 2 *Brass surface after polishing a) after chronoamperometric measurements at 0V vs. SCE in weakly alkaline 0.5 mol/dm^3 Na_2SO_4 solution, b) and after chronoamperometric measurements at 0V vs. SCE in weakly alkaline 0.5mol/dm^3 Na_2SO_4 solution with 1·10^{-2} mol/dm^3 purine c) (magnified 200x) [38]*

The possibility to enhance the effect of adenine by the addition of cetyltrimethylammonium bromide (CTAB) is studied in synthetic sea water [43]. As can be seen from the data, Table 2, beneficial effect of CTAB is confirmed by the experimental results.

Based on the all of the results mentioned above, it was logical to broaden the research and provide some more insight into how the molecular structure influences the inhibition efficiency values and guidance for adequate inhibitor choice. Petrović Mihajlović *et al*. [44] applied imidazole, purine, adenine and 6-benzylaminopurine as corrosion inhibitors, and the expected trend was confirmed since the inhibition efficiency increased as following: imidazole < purine < adenine < 6-benzylaminopurine. Electrochemical experiments were substantiated by quantum chemical calculations and correlation of some parameters with

inhibition efficiency is noticed. When structurally similar compounds are studied lower value of ΔE ($\Delta E = E_{LUMO} - E_{HOMO}$) and decrease of global hardness (η) or increase of dipole moment (μ) indicate that it can be expected that compound can perform as better corrosion inhibitor.

Table 2 *Copper corrosion inhibition efficiency obtained using purine and derivatives*

Inhibitor	Concentration, M	Medium	Inhibition efficiency, %	Reference
Adenine	$1\cdot10^{-3}$	Synthetic seawater	84.35[a]	[43]
	$5\cdot10^{-3}$		92.17[a]	
CTAB	15 ppm		68.70[a]	
Adenine + CTAB	$1\cdot10^{-3}$ M + 15 ppm		91.30[a]	
Purine	$1\cdot10^{-3}$	0.1 M $Na_2B_4O_7$ + 0.05 M NaCl	78.38[a]	[40]
Adenine	$1\cdot10^{-3}$		78.65[a]	
6-benzylaminopurine	$1\cdot10^{-3}$		83.78[a]	
Purine	$5\cdot10^{-3}$	Synthetic seawater	91.91[a]	[44]
Adenine	$5\cdot10^{-3}$		92.17[a]	
6-benzylaminopurine	$5\cdot10^{-3}$		94.43[a]	
Purine	$1\cdot10^{-2}$ in NaCl 3h	SUF	85[b]	[30]
Adenine	$1\cdot10^{-2}$	BM3	88.10[a]/89.69[c]	[32]
2,6-diaminopurine	$1\cdot10^{-2}$		87.18[a]/90.10[c]	
Adenine + potassium sorbate	$1\cdot10^{-2}$ M + 0.01%		91.17[a]	
2,6-diaminopurine + potassium sorbate	$1\cdot10^{-2}$ M + 0.01%		93.02[a]	
			IE% at -0.1 V vs SCE	
Purine	$1\cdot10^{-3}$, 1 h	SUF	61.20[a]	[29]
	$1\cdot10^{-3}$, 3 h		98.61[a]	
	$1\cdot10^{-2}$, 1 h		98.10[a]	
	$1\cdot10^{-2}$, 3 h		99.18[a]	

Inhibition efficiency values obtained using: [a]potentiodynamic polarization, [b]polarization resistance, [c]electrochemical impedance spectroscopy.

However, quantum chemical calculations are considered to be insufficient to provide dipper understanding of metal-inhibitor interactions so corrosion inhibition mechanism of these compounds was further studied by Kumar *et al*. [45] using density functional theory (DFT) and reactive force fields (ReaxFF). They studied the adsorption of imidazole, purine, adenine and 6-benzylaminopurine on Cu (111) surface and the interactions that occur between the surface and inhibitor molecules. The results of quantum chemical calculations were in accordance with our calculations and experimental findings. Adsorption studies indicated that all of the tested compounds do adsorb on Cu surface while the interaction energy increases from imidazole towards 6-benzylaminopurine, whereas higher values are obtained for parallel orientation of adenine and perpendicular orientation for all other compounds. Charge distribution indicates that adenine and Cu (111) surface interact via electrostatic and van der Waals interactions, whereas adsorption of imidazole, purine and BAP occurs via, one or in the

case of BAP two, Cu-N covalent bond formation and weak van der Waal's and H-Cu interactions.

Purine and adenine also have the ability to inhibit copper corrosion in the hydrocarbon medium [46] and under the high temperature are humidity conditions that can be found in the production of chips and LSI devices [47]. In both cases thin protective inhibitor film is formed on copper surface.

Non-toxic characteristics inspired some other investigations as potential inhibitors for applications in human organisms. Alvarez *et al*. [29,31] studied the effect of purine [29], 6-mercaptopurine (6-MP) [31] and pterin (PT) [31] on corrosion of copper in simulated uterine fluid (SUF). This could find its application for the treatment of intrauterine devices IUDs that are used for birth control. In both cases inhibitor was applied as pretreatment before testing in SUF. The desired effect of reduction of burst release and providing of continual release of small dose that enables contraception effect is achieved by a complex mechanism including adsorption of inhibitor and chloride ions described by the following reactions [31]:

$$Cu(6\text{-}MP)_{ad}+Cl^{-}\rightarrow Cu(Cl)_{ad}+6\text{-}MP+e^{-} \quad (8)$$

$$Cu+H_2O\leftrightarrow Cu(OH)_{ad}+H^{+}+e^{-} \quad (9)$$

$$Cu+Cl^{-}\leftrightarrow Cu(Cl)_{ad}+e^{-} \quad (10)$$

Purine pretreatment and its adsorption on copper surface in aqueous and NaCl media was studied in more detail by Alonso *et al*. [30]. Quartz crystal microbalance (QCM) was applied in order to directly monitor mass change due to purine adsorption. Interestingly greater increase of mass is noticed in chloride solution and in the presence of lower concentration of purine. However, an explanation is also proposed. In the presence of high purine concentration it adsorbs on the pure Cu surface, whereas in the presence of lower purine concentration and chloride ions, chloride compounds of copper are formed that further react with purine and form Cu(I)-purine complex. X-ray photoelectron spectroscopy (XPS) measurements confirmed these assumptions since there is a difference in the composition of products on copper surface. In the presence of high $1\cdot10^{-2}$ mol dm^{-3} - $1\cdot10^{-3}$ mol dm^{-3} purine concentration purine chemisorption on pure copper surface occurs and $(Cu(0)\text{-}PU)_{ads}$ forms. When purine concentration is lower such as $1\cdot10^{-4}$ mol dm^{-3} there are compounds of copper and chlorine beside the $[Cu(I)\text{-}PU]_{ads}$ compound.

Petrovic Mihajlovic *et al*. [32] studied the possibility to apply purine compounds, adenine and 2,6-diaminopurine (DAP), as copper corrosion inhibitors in simulated blood plasma (BM3). This medium is complex regarding its composition, however dominant ions and molecules that interact with copper are oxygen, hydrogen and chloride, so the products are copper ions, oxides, hydroxides and chlorides. When adenine or 2,6-diaminopurine are present in the solution peaks that correspond to copper oxidation are either shifted towards more positive potentials or disappear if the concentration is high enough. Reason for such behavior can be found in the reaction and formation of complexes Cu(I)-AD/Cu(I)-DAP at lower potentials, and Cu(II)-AD/Cu(II)-DAP at higher potentials due to oxidation of Cu(I) to Cu(II) [44,48]. Higher concentration of AD or DAP enables better surface coverage by adsorbed molecules and thus the oxidation of copper is hindered [30,34,44]. The adsorption of inhibitor molecules on copper surface was also confirmed by scanning electron microscope

coupled with energy dispersive spectroscopy (SEM-EDS), Figure 3. As already seen before, further improvement of inhibition efficiency can be provided if the mixture of inhibitors that show synergistic effect is used. In this case potassium sorbate was used since it is safe compound regarding health and is frequently used as food additive and preservative [49,50].

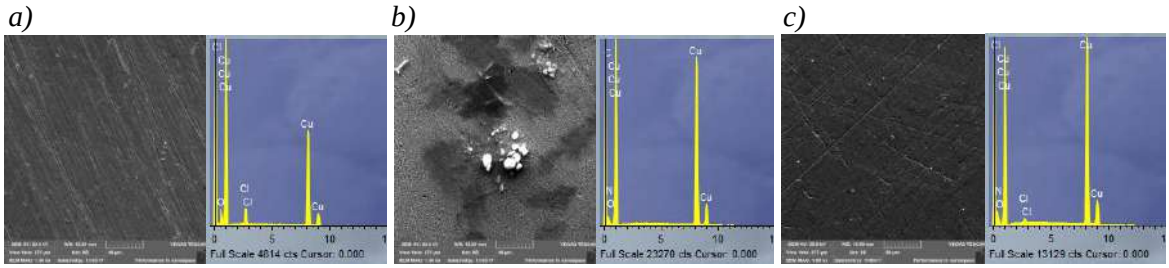

Figure 3 *SEM images and EDS spectrum of copper surface obtained after 7 days immersion in a) BM3 solution and with the addition of b) $1 \cdot 10^{-2}$ M adenine, c) $1 \cdot 10^{-2}$ M 2,6-diaminopurine [32]*

CONCLUSION

Purine compounds, including purine, adenine, 6-mercaptopurine, 2,6-diaminopurine and 6-benzylaminopurine, showed good properties as inhibitors of corrosion of copper and its alloy, brass. They are efficient in numerous media such as chloride, sulfate, nitrate solutions, synthetic seawater, and synthetic body fluids such as blood plasma and uterine fluid. The obtained inhibition efficiency depends among other conditions on the pH value of the test media. Their influence is a results of interaction with metal surface via adsorption and complex formation. The adsorption is confirmed using surface analysis methods such as SEM and the mode of adsorption and most probable orientations and places for interaction are proposed using theoretical calculations. Inhibition efficiency rises as the molecular size and number of heteroatoms increase. The values of inhibition efficiency can be further improved by the addition of compounds providing synergistic effect such as CTAB and potassium sorbate. These inhibitors can be used as the addition to the corrosive media or as a pretreatment.

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