Experimental and Theoretical Studies of 1,3-diaminopropylene Derivatives as Potential Antioxidative Agents for Copper Nanoparticles in Hydrothermal System

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Abstract

Novel 1,3-diamino-propylene derivatives as potential corrosion inhibitors for copper nanoparticles has been studied by density functional theory (DFT) calculations, Fourier transform infrared spectra (FTIR), thermogravimetric and Zeta potential analysis. 1,3-Dimorpholin-propylene was considered to be the most promising inhibitor for copper nanoparticles. Thermogravimetric results indicated the oxidation temperature of copper nanoparticles decreased from 286.50 °C (blank) to 270.1 °C (inhibitor). FTIR spectra indicated the addition of inhibitor could optimize the adsorption behavior of surfactant molecules on the surface of copper nanoparticles. Zeta potential results showed 1,3-dimorpholin-propylene could improve the stability of copper nanoparticles (~0 mV for blank, and -2.5 mV for inhibitor).

Keywords: Copper nanoparticles; Corrosion; DFT; Zeta potential


1. Introduction

Copper nanoparticles, one of the most important nanomaterials for metallurgy industry, have been the focus subject of scientific studies in recent few years. Owing to their special properties in optics, electrics, calorifics and size-dependent effects, copper nanoparticles are widely used in microelectromechanical systems, printed wiring board fabrication, conducting materials, premium lubricant and catalyst [1-6]. However, the application of copper nanoparticles has been confined for their susceptibility to oxidation in an aggressive medium [7, 8]. Therefore, improving their activity and stability are vital to explore their application in many areas that are dominated by the use of gold, silver and platinum nanoparticles [9]. To date, one of the most promising techniques is the use of organic substances as corrosion inhibitors for nanomaterials [10-13]. Generally, organic substances containing heteroatoms (e.g., O, N and S) can be used as corrosion inhibitors [14]. The organic molecules may be adsorbed on the surface of nanomaterials via the bond between the lone pair electrons and/or p-electrons and the unoccupied orbital of metal, forming a protective thin film [15]. As a result, the corrosive attack of anions on the defects of nanomaterials is reduced in solution. Numerous investigations suggested that the protective action of organic substances was related to the molecular structures, the planarity and the lone pair electrons in the heteroatoms [16-19]. During the development of novel corrosion inhibitors for nanomaterials, the quantum chemical calculation is still the most classical and traditional approach involving isolated organic substances [20-22]. According to the theoretical calculations, it may give some basic valuable parameters for huge organic substances at low cost and understand the reactivity behavior of organic substances and their protective effect for nanomaterials [23, 24]. Due to the specificity of organic substances and the variety of reaction systems, the choice of a potential corrosion inhibitor for a particular system seems to be in demand.

1,3-Diaminopropylene is low-toxicity amphiphilic compounds with outstanding reducing ability (C=C bond), water solubility and thermal stability, exhibiting potential inhibition property for nanomaterials. In this paper, we propose a 1,3-diaminopropylene-assisted hydrothermal method for the synthesis of active and stable copper nanoparticles. The synthetic procedure
is easy and straightforward. Scheme 1 illustrates the inhibition procedure for growing copper nanoparticles in the presence of 1,3-diaminopropylene.

Scheme.1 Schematic illustration of the inhibition procedure for copper nanoparticles (NPs) in hydrothermal system.

2. Experimental

2.1 Computational details

Calculations were performed using a Gaussian 98 program in the gas phase. Exchange and correlation calculations were done by the theoretical B3LYP/6-311 G(d,p) model. The structure optimization and the vibration analysis of the investigated structures were done in order to determine whether they corresponded to a minimum potential energy state. For the molecular structures of the investigated inhibitors, the possible active sites with copper nanoparticles have been discussed in the light of frontier molecular orbital theory and Mulliken charges.

2.2 Materials

Fabrication of copper nanoparticles was carried out by a hydrothermal method. Typically, a mixture of 40 mL NaOH (0.2 M) and 20 mL Vitamin C (Vc, 0.3 M) was gradually added into a solution of 20 mL CuSO$_4$·5H$_2$O (0.2 M) and 10 mL cetyltrimethylammonium bromide (CTAB, 0.01 M) in the absence and presence of inhibitors at room temperature. After reaction, the resulted solution was transferred into a Teflon cell and elevated to 80-180 °C for 6 h. When the mixture was naturally cooled to room temperature, red sediments can be obtained. The formed powders were washed by deionized water, ethanol and naturally dried for 24 h at room temperature.

2.3 Characterization

The morphology of copper nanoparticles was observed by scanning electron microscopy (SEM, FEI-Sirion200), and its composition was characterized by an X-ray diffractometer (XRD, Germany, Omicron, Cu Kα radiation, λ=1.5418 Å). The reactivity of copper nanoparticles was by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) in air. The adsorption behavior of surfactant molecules (CTAB) in the absence and presence of inhibitors was analyzed by Fourier transform infrared (FTIR, Bruker, Equinox 55, Germany). Zeta potential analyzer (NICOMP 380 ZLS, Santa Barbara, California, USA) was used to analyze the stability of copper nanoparticles.

3. Results and discussion

The scanning electron microscopy (SEM) image in Fig. 1a shows the products are composed of numerous polyhedron particles (~1 μm) and a few irregular nanoparticles. These small nanoparticles have a tendency to melt or partially melt (red arrow), resulting in the interparticle coalescence which was related to the surface melting of copper nanoparticles at high temperature [25]. The X-ray diffraction (XRD) pattern in Fig. 1b shows that copper nanoparticles can be synthesized at a wide temperature range (80-180 °C). The diffraction peaks at 2θ=43.4, 50.5 and 74.3 can be indexed as (111), (200) and (220) planes of cubic copper (JCPDS no.1-1241) structure. The rather sharper diffraction peaks indicated the high crystallinity of the copper nanoparticles. Fourier transform infrared (FTIR, Fig. 1c) spectra confirmed the adsorption behavior of CTAB on the surface of copper nanoparticles was improved by the addition of 1,3-dimorpholin-propylene. The characteristic peaks at 566 cm$^{-1}$ can be assigned to the vibration of copper nanoparticles. Adsorption peaks around at 3452, 1637, 1564, 1415, 1096 and 1038 cm$^{-1}$ can be assigned to the adsorption peaks of CTAB molecules. All these features show that the chemical integrity of CTAB is maintained during the formation of copper nanoparticles at 160 °C. When 1,3-dimorpholin-propylene was added. The peak shape of CTAB around at 1637 and 1564 cm$^{-1}$ changed significantly due to the influence of stretch vibration of C=C bond (1636 cm$^{-1}$) by the 1,3-dimorpholin-propylene molecule, implying that the addition of 1,3-dimorpholin-propylene has optimized the adsorption behavior of CTAB molecules. This will lead to the formation of stable screening layer on the surface of copper nanoparticles, inhibiting their oxidation in a high temperature aggressive medium. In order to evaluate the activity of synthesized copper nanoparticles, thermogravimetric analysis and differential scanning calorimetry (TGA/DSC) were carried out. Fig. 1d shows the TGA/DSC curves of synthesized copper nanoparticles in the absence and presence of 1,3-dimorpholin-propylene. The sample weight decreases at temperature below 300 °C with a comparatively slow rate, which might be connected with the mass loss of organic capping shells on the surface of copper nanoparticles. Smaller mass loss of organic capping shells implies the higher crystallinity of copper nanoparticles formed at 160 °C. The initial oxidation temperature of copper nanoparticles decreased from 286.5 °C (blank) to 270.1 °C (1,3-dimorpholin-propylene), identifying that the fact that the 1,3-dimorpholin-propylene could inhibit the oxidation of copper nanoparticles in the hydrothermal condition. Over 300 °C, the DSC curve shows two remarkable exothermic peaks at approximate 305 °C and 434 °C, corresponding to the oxidation of copper nanoparticles from Cu$^{2+}$ to CuO: Cu$^{2+}$ + 1/2O$_2$ → CuO; CuO + 1/2O$_2$ → 2CuO. When the temperature was elevated to 531.6 °C, all of the products are converted to CuO. The effect of 1,3-dimorpholin-propylene on the stability of copper nanoparticles

was evaluated by Zeta potential measurements. The synthesized copper nanoparticles were dispersed in ethanol via ultrasonic condition (30 min). Figure 1e shows the Zeta potential of copper nanoparticles in the absence and presence of 1,3-dimorpholin-propylene. Initially, the Zeta potential abruptly decreased and reached a relatively steady value thereafter. The Zeta potential was found to be ~0 mV without corrosion inhibitors, and ~2.5 mV for 1,3-dimorpholin-propylene. This result revealed that the stability of copper nanoparticles can be improved by the addition of 1,3-dimorpholin-propylene (1,3-dimorpholin-propylene is one of the most promising corrosion inhibitors in the series of 1,3-diamino-propylene derivatives (f)).

In order to clarify the electronic and inhibition properties of 1,3-dimorpholin-propylene, we have a theoretical studies for 1,3-diamino-propylene In the present study, the investigated compounds include: (1) 1,3-diamino-propylene, (2) 1,3-dimethylamino-propylene, (3) 1,3-diethylamino-propylene, (4) 1,3-dimethylethylamino-propylene, (5) 1,3-dipropylamino-propylene, (6) 1,3-dimorpholin-propylene, (7) 1,3-dipyrrolidineamino-propylene, and (8) 1,3-dipyrroleamino-propylene. The optimized structures of 1,3-diamino-propylene derivatives are shown in Fig. 2. When the H-atoms in N_1 and N_2 of compound-1 were replaced by different alkyl groups (compounds 2 to 5), the nucleophilic sites of nitrogen atoms may be shadowed by the adjacent carbon and hydrogen atoms of the substituents. This means that these molecules are not favoring the adsorption on metallic surfaces. For the molecules of compound-7 and compound-8, the H-atoms in N_1 and N_2 are substituted by the heterocyclic...
Aromatic amines. It is observed that the molecular structures exhibit an unequal configuration because the two substituents of heterocyclic aromatic amines are not located in a common plane. The difference of the symmetrical adsorption centers (N_1 and N_2) may facilitate the configuration change of molecules. As far as the compound-6 (1,3-dimorpholin-propylene) is concerned, its arc structure and symmetrical adsorption centers (O_1 and O_2, N_1 and N_2) which are favoring the adsorption on metallic surfaces, especially for the nanomaterials with a spherical shape. Generally, the inhibition efficiency of an organic compound was related not only to its spatial molecular structures but also to their molecular electronic properties [26]. Table 1 shows the quantum chemical parameters of the investigated compounds. The global hardness (η) and the energy gap (ΔE) between the E_{HOMO} and E_{LUMO} were calculated by the following equations:

$$\eta = \Delta E / 2$$

$$\Delta E = E_{LUMO} - E_{HOMO}$$

We should mention that the energy of E_{HOMO} that is

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**Table 1.** Quantum chemical parameters of 1,3-diamino-propylene derivatives.

<table>
<thead>
<tr>
<th>Molecules</th>
<th>HOMO (eV)</th>
<th>LUMO (eV)</th>
<th>ΔE (eV)</th>
<th>Total energy (a.u.)</th>
<th>η (eV)</th>
<th>μ (debye)</th>
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<td>0.105</td>
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<td>0.212</td>
<td>-464.443</td>
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Table 2. Mulliken charges of 1,3-diamino-propylene derivatives.

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<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<td>-0.266</td>
<td>-0.288</td>
<td>-0.265</td>
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<td>-0.307</td>
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<td>0.128</td>
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<td>C_10</td>
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<td>-0.355</td>
<td>-0.205</td>
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<td>-0.316</td>
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<td>O_1</td>
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Fig. 3 Molecular HOMO orbital for (a) 1,3-diamino-propylene, (b) 1,3-dimethyl amino-propylene, (c) 1,3-diethylamino-propylene, (d) 1,3-dimethylamino-propylene, (e) 1,3-dipropylamino-propylene, (f) 1,3-dimorpholin-propylene, (g) 1,3-dipyrrolidineamino-propylene and (h) 1,3-dipyrroleamino-propylene.
often related to the electron donating ability of organic substances. An increase in the values of $E_{\text{HOMO}}$ may facilitate the adsorption and therefore improve the inhibition efficiency. The values of $E_{\text{LUMO}}$ are correlated with the ability of the molecule to accept electrons. Therefore, lower values of $\Delta E$ may provide good inhibition efficiency, because the energy to remove an electron from the last occupied orbital will be low. The lower value of $\Delta E$ has, the higher stability is for the formed stable film on the metal surface. It is interesting to observe that there is a minimum value of $\Delta E$ for compound-8 in the investigated compounds, implying a greater efficiency in being used as corrosion inhibitors. Global hardness is another parameter that provides important information about the reactive behavior of organic substances. According to the calculated data (Table 1), the reactivity order of 1,3-diamino-propylene derivatives is:

$$8 > 7 > 3 > 4, 5 > 6 > 2 > 1$$

It is evident that compound-8 presents a minimum hardness value, whereas compound-1 exhibits a maximum value. For the compounds 3, 4, 5, 6 and 7, there are slight changes in the global hardness when the mother structure of compound-1 has been substituted by different groups.

Dipole moment ($\mu$) is an important parameter that reflects the hydrophobic character of corrosion inhibitors. It was found that compound-1 has a maximum value of $\mu$ (2.177), suggesting compound-1 is not favoring the accumulation of inhibitive molecules on the metal surface. This means that the corrosive ions (such as Cl\(^-\), NO\(^3\) or SO\(^2^-\)) in solution are easily arrived at the defects of metal substrate, inducing the chemical dissolution of nanomaterials. For the compound-4, it has a minimum value of $\mu$ (1.070), implying a greater adsorption efficiency in being used as inhibitive agent. Generally, the effectiveness of a corrosion inhibitor was related with the physical/chemical properties of the protective film. If the inhibitive molecules are easily adsorb on the metal surface, at the initial reaction stage, the competitive adsorption among the inhibitive molecules may result in the formation of porous complex film. The diffusion process of metal cations and aggressive anions into and from the screening layer is faster. In this way, the compound with high adsorption efficiency (compound-8) commonly can’t be acted as effective corrosion inhibitor. The hydrophobic property of the investigated compounds has the following order:

$$1 > 5 > 6 > 2 > 3 > 8 > 7 > 4$$

The Mulliken charges of all the compounds are shown in Table 2. Except for C\(_3\) atoms, other carbon atoms and heteroatoms (N and O) are all presenting negative charge feature. This suggests 1,3-diamino-propylene derivatives may serve as potential corrosion inhibitor for nanomaterials due to their stronger nucleophilic properties. For the molecule of compound-1, the charges of N\(_1\) (-0.704) and N\(_2\) (-0.790) have the largest negative values. It is shown that the negative charges of N\(_1\) and N\(_2\) (compounds 2 to 8) has a decrease trend owing to the replacement of the H-atoms. It is interesting to observe that there are six atoms present a considerable excess of negative charges for the molecules of compound-2 (-0.403, -0.520, -0.357, -0.351, -0.366 and -0.371 for N\(_1\), N\(_2\), C\(_4\), C\(_5\), C\(_6\) and C\(_7\)), compound-3 (-0.439, -0.524, -0.484, -0.493, -0.508 and -0.502 for N\(_1\), N\(_2\), C\(_8\), C\(_9\), C\(_10\) and C\(_11\)), compound-4 (-0.419, -0.515, -0.365, -0.380, -0.546 and -0.518 for N\(_1\), N\(_2\), C\(_4\), C\(_6\), C\(_8\) and C\(_9\)) and compound-7 (-0.387, -0.615, -0.410, -0.395, -0.355 and -0.356 for N\(_1\), N\(_2\), C\(_8\), C\(_9\), C\(_10\) and C\(_11\)), whereas for the compound-5 there are eleven active sites (-0.307, -0.418, -0.534, -0.344, -0.322, -0.334, -0.316, -0.525, -0.524, -0.523 and -0.531 for C\(_1\), N\(_1\), N\(_2\), C\(_8\), C\(_9\), C\(_10\), C\(_11\), C\(_12\), C\(_13\), C\(_14\) and C\(_15\)). For the compound-8, only two of the nitrogen atoms present an excess of electrons (-0.531 and -0.636 for N\(_1\) and N\(_2\), respectively). For the compound-6, four active sites display a considerable excess of negative charge (-0.419 and -0.542 for N\(_1\) and N\(_2\), -0.471 and -0.466 for O\(_1\) and O\(_2\)). As for the coordination with the metallic surfaces, more active sites of organic substances are not favoring the formation of stable protective film due to the competitive adsorption centers were susceptible to undergoing configuration change. This means that the protective film is porosity, and cations mobility in the screening layer is more unrestricted. According to the molecular structures, energy difference, dipole moment and Mulliken charges, compound-6 (1,3-dimorpholin-propylene) are prone to undergo nucleophilic attacks and form hydrophobic protective film on metallic surfaces against the attack of aggressive anions.

Fig. 3 showed the HOMO orbital for 1,3-diamino-propylene derivatives. As for compound-1, the HOMO is located in the N\(_1\), C\(_2\)=C\(_3\) and N\(_2\). For compounds 2, 3, 4, 5 and 7, the HOMO is located in C\(_2\)=C\(_3\), N\(_2\). For compound-8, the HOMO is located at C\(_2\)=C\(_3\) and the pyrrole ring attached to N\(_1\) and N\(_2\) atoms. For compound-6, the HOMO is located in C\(_2\)=C\(_3\), N\(_2\) and O\(_2\) atoms. As far as compound-6 is concerned, its arc steric structure (Fig. 3f) and active sites (C\(_2\)=C\(_3\), N\(_2\) and O\(_2\)) may facilitate their adsorption on the surface of nanostructured materials, which confirms that compound-6 may act as an effective corrosion inhibitor for nanomaterials.

In conclusion, active and stable copper nanoparticles were synthesized by a 1,3-diaminoproplylene-assisted hydrothermal method. According to the correlation between the reactivity of organic substances and their steric structures, 1,3-dimorpholin-propylene exhibits the most promising inhibition effect for copper nanoparticles due to its arc structure and active site (C\(_2\)=C\(_3\), N\(_2\) and O\(_2\)) easily facilitate their adsorption on the surface of copper nanoparticles. FTIR analysis shows the addition of 1,3-dimorpholin-propylene could improve the adsorption behavior of CTAB molecules on the surface of copper
nanoparticles. The TGA/DSC data confirms the oxidation temperature of copper nanoparticles decreased from 286.5 °C (blank) to 270.1 °C (1,3-dimorpholin-propylene), identifying that 1,3-dimorpholin-propylene could effectively inhibit the oxidation of copper nanoparticles in the hydrothermal condition. The synthesized copper nanoparticles may be exploited as possible candidates for catalyst application.

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