

Corrosion of copper in aerated acidic pickling solutions and its inhibition by 3-amino-1,2,4-triazole-5-thiol

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Abstract

Corrosion of copper in aerated acidic chloride pickling (0.5 M HCl) solutions and its inhibition by 3-amino-1,2,4-triazole-5-thiol (ATT) have been investigated using electrochemical techniques and weight-loss measurements, along with Raman spectroscopy. Electrochemical measurements for copper after varied immersion periods of 0, 24, and 48 h showed that the presence of ATT and the increase of its concentration significantly decrease cathodic, anodic, corrosion (j_{CORR}) currents and corrosion rates (K_{CORR}), as well as the dissolution currents at 300 mV vs Ag/AgCl, while increasing polarization resistance (R_p), degree of surface coverage (θ) and inhibition efficiency (IE%) to a great extent. Weight-loss measurements after different immersion periods of 6 to 48 h revealed that the dissolution of copper decreased to a minimum and the corresponding IE% increased with increasing ATT concentration. The detection of ATT molecules on the copper surface by Raman spectroscopy indicated that inhibition of copper corrosion is achieved by strong adsorption of ATT molecules onto the copper surface.

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1. Introduction

Copper is commonly used as a material in heating and cooling systems owing to its excellent thermal conductivity, good corrosion resistance and mechanical workability. Scale and corrosion products have a negative effect on heat transfer and cause a decrease in heating efficiency of the equipment. Periodic descaling and cleaning in hydrochloric acid pickling solutions are thus necessary. Consequently, corrosion of copper and its inhibition in hydrochloric acid solutions have attracted the attention of several investigators [1–6].

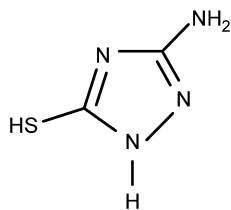
It is generally believed that corrosion inhibitors effectively eliminate the undesirable destructive effects of aggressive media and prevent copper dissolution. The inhibiting action of these inhibitors is usually attributed to their interactions with the copper surface via their adsorption [7]. However, the adsorption of an inhibitor on a metal surface depends on the na-

ture as well as the surface charge of the metal, the adsorption mode, its chemical structure, and the type of the electrolyte solution [8].

We have been studying surface properties of metals [9], their corrosion reactions [10], and effects of organic additives on its corrosion inhibition [1,11,12] in different media. In the earlier work [10], the corrosion of copper at different concentrations of NaCl in absence and presence of NaHCO_3 has been studied using electrochemical techniques and XPS investigations. More recently [1,11], the inhibition of copper corrosion by *N*-phenyl-1,4-phenylenediamine (NPPD) [1,11a], 2-amino-5-ethylthio-1,3,4-thiadiazole (ATD) [11b,11c], and 2-amino-5-ethyl-1,3,4-thiadiazole (AETDA) [11d] in 3% NaCl and 0.50 M HCl, was reported. NPPD, ATD, and AETDA were found to be good mixed-type inhibitors for copper corrosion by strongly adsorbing on the copper surface with the inhibition efficiency increasing with increasing concentration. FT-IR, Raman, and UV-visible absorption spectroscopies, as well as SEM and EDX investigations revealed that the molecules of these compounds adsorb on the copper and form a complex not only with

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Scheme 1. 3-Amino-1,2,4-triazole-5-thiol (ATT).

the copper chloride complex on the surface but also in the solution.

The present paper reports on the corrosion and corrosion inhibition of copper after varied exposure periods of time (0–48 h) in aerated 0.5 M hydrochloric acid solutions by 3-amino-1,2,4-triazole-5-thiol (ATT, Scheme 1). ATT is expected to be a good inhibitor for copper corrosion in HCl solutions because it is a heterocyclic compound containing a variety of donor groups.

2. Experimental procedure

ATT (Sigma–Aldrich, 95%), hydrochloric acid (HCl, Glassworld, 32%), and absolute ethanol (C_2H_5OH , Merck, 99.9%) were used as received. An electrochemical cell with a three-electrode configuration was used; a copper rod (Cu, Goodfellow, 99.999%, 5.0 mm in diameter), a platinum foil, and an Ag/AgCl electrode (in saturated KCl) were used as a working, counter, and reference electrodes, respectively. The copper electrode was first polished successively with metallographic emery paper of increasing fineness of up to 600 grits and further with 5, 1, 0.5, and 0.3 μm alumina slurries (Buehler). The electrode was then washed with doubly distilled water, degreased with acetone, washed using doubly distilled water again and finally dried with tissue paper.

Electrochemical experiments were performed by using a Schlumberger SI 1286 electrochemical interface potentiostat-galvanostat. For potentiodynamic polarization experiments the potential was scanned from -600 to 700 mV at a scan rate of 1 mV/s. Potentiostatic current-time experiments were carried out by setting the potential at 300 mV for 120 min. Impedance measurements were made using a Solatron SI 1255 HF frequency response analyzer along with the potentiostat-galvanostat. The instruments were controlled by the FRA-3.5 software program between 100 kHz and 0.05 Hz with an ac wave of ± 5 mV peak-to-peak overlaid on a dc bias potential, and the impedance data were obtained at a rate of 10 points per decade change in frequency. All electrochemical measurements were carried out at an open circuit potential after varied immersion periods of 0, 24, and 48 h of the copper electrode in the test electrolyte.

The weight loss experiments were carried out using rectangular copper coupons (Goodfellow, 99.999%) having the dimensions of 3.0 cm length, 1.0 cm width, and 0.20 cm thickness with an exposed total area of 7.6 cm^2 . The coupons were polished and dried as the copper rods were, weighed (m_1), and then suspended in 50 cm^3 solution of 0.5 M HCl with and without the desired concentrations of ATT for different exposure periods (6–48 h). At the end of a run, the samples were rinsed

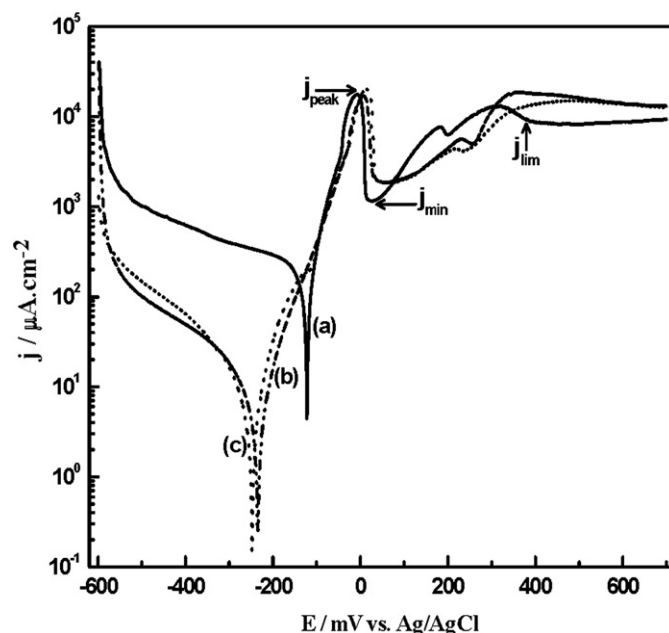


Fig. 1. Potentiodynamic polarization curves for copper electrode in aerated 0.5 M HCl after varied immersion periods of 0 (a), 24 (b), and 48 h (c). Potential scan rate 1 mV/s.

with distilled water, dried and weighed again (m_2). All weight-loss measurements were performed in triplicate and the maximum standard deviation in the observed weight loss was $\pm 2\%$. The loss in weight (Δm , mg m^{-2}), the corrosion rate (K_{Corr} , $\text{mg m}^{-2} \text{h}^{-1}$), and the percentage of the inhibition efficiency (IE%) over the exposure time were calculated as reported in previous work [11a].

Raman spectra were measured using a Jobin-Yvon T64000 Raman spectrometer operated in single spectrograph mode with a holographic dispersive grating of 600 grooves/mm. The samples were analyzed in the back-scattering mode on the microscope stage of an Olympus confocal microscope attached to the spectrometer using a long working distance 20 \times objective. The detector used was a liquid nitrogen cooled charge coupled device (CCD) detector. A 647.1 nm holographic notch filter was used to remove the Rayleigh-scattered light. The entrance slit width was 100 μm giving a resolution of 2 cm^{-1} .

3. Results and discussion

3.1. Potentiodynamic polarization

In order to study the effect of immersion time on Cu corrosion in aerated 0.5 M HCl solution, we carried out potentiodynamic polarization experiments. The potentiodynamic polarization curves of the copper electrode in aerated 0.50 M HCl solutions after varied immersion times of 0 (a), 24 (b), and 48 h (c) are shown in Fig. 1. It is clearly seen from Fig. 1 that the anodic currents at all immersion periods display three distinct regions as reported in our previous work [1,11a]: In brief, these are the Tafel region at lower over-potentials extending to the peak current density (j_{peak}) due to the fast dissolution of copper into Cu^+ , then slow oxidation of Cu^+ to Cu^{2+} ; the re-

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