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Low-cost mussel inspired poly(catechol/polyamine) coating with superior anti-corrosion capability on copper



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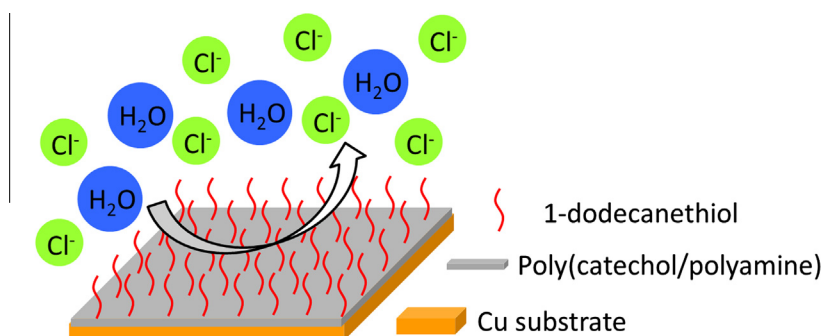
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HIGHLIGHTS

- A uniform, compact and thermal stable biomimetic coatings was obtained.
- The coating exhibits superior long-term anti-corrosion properties.
- The hydrophobicity, good stability and strong wet adhesion endow the remarkable properties.

GRAPHICAL ABSTRACT



Low-cost Mussel-inspired Anti-corrosion Coating

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ABSTRACT

A low-cost mussel inspired approach was developed to produce anti-corrosion coating on copper substrate. Catechol (CA) and polyamine (PA) were spontaneously polymerized to form adhesive coating of poly(catechol/polyamine) (P(CA/PA)) onto copper surface and then P(CA/PA) was grafted by 1-dodecanethiol. The SEM, contact angle, XPS, FTIR and TG results demonstrated the formation of uniform, compact and thermal stable coatings through multiple interactions and chemically grafting. Electrochemical tests indicated of Cu-P(CA/PA)-SH possessed a highest corrosion potential of -81 mV, a lowest corrosion current density of $0.15 \mu\text{A}/\text{cm}^2$, and a highest coating resistance of $57.19 \text{ k}\Omega \text{ cm}^2$, and also exhibit great long-term stability whether in solution immersion or salt spray tests. The remarkable anti-corrosion capability of Cu-P(CA/PA)-SH could be ascribed to the synergistic effect of the hydrophobicity, good stability, and strong wet adhesion of the mussel-inspired coating. This study provides an effective and cheap way for material protection and may give inspiration in the fields of material, biology and medicine relating to surface and interface engineering.

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1. Introduction

Metal corrosion is a common phenomenon and causes a huge loss annually. Finding appropriate ways to reduce metal corrosion is crucial in industry, economy and society [1]. Among the numerous

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corrosion control techniques, organic coating modification, especially employing water repellent coatings, has been demonstrated an effective approach [2,3]. Previously, varieties of hydrophobic or superhydrophobic coatings have been fabricated on metals surfaces and have displayed good corrosion resistance properties [4–8]. However, the long-term anti-corrosion capability of many water repellent coatings was not satisfied due to the weak adhesion between coatings and substrates [9,10]. Thus, it is necessary to enhance the interface adhesion for long-term stable anti-corrosion coatings.

Recently, polydopamine (PDA), polymerized from dopamine, has drawn strong interest owing to its mussel-like outstanding underwater adhesion to many kinds of materials, and also its chemical versatility for diverse secondary reactions [11]. PDA has shown promising potential application in many fields [12–14], including for anti-corrosion coatings. Various superior corrosion resistant coatings were fabricated by using PDA as an adhesive layer and platform for hydrophobic modification [15–17]. Nevertheless, the price of dopamine is rather expensive, thus impede its extensive application. Previously, our group replaced dopamine by a catechol (CA) and polyamine (PA) binary system for the first time [18]. CA and PA can also polymerize in an alkaline solution and the resultant poly(catechol/polyamine) (P(CA/PA)) can deposit on the surfaces of various materials, displaying universal adhesion ability as PDA. The binary system is less than 8% of the cost of dopamine. As mentioned above, the remarkable adhesion ability and chemical versatility are the foundations of wide applications of PDA. Although P(CA/PA) shown similar adhesion ability, the chemical versatility need to be verified to expand its application.

Herein, P(CA/PA) coating was investigated for metal corrosion protection, and copper (Cu) substrate was used as an illustration. P(CA/PA) film was formed on Cu by immersing the substrate in a mixture aqueous solution of CA and PA. The chemical grafting of 1-dodecanethiol on the P(CA/PA) film was employed to produce hydrophobic surface. The chemical structure, morphology and thermal properties of coatings were characterized and analyzed. The anti-corrosion performances of the resultant coatings immersed in salt solution and exposed to salt spray condition were demonstrated.

2. Experimental section

2.1. Materials

Copper (>99.7%) was received from Jiangsu Xiangwei Mechanism Co., Ltd and was machined to deserved sizes. 1-dodecanethiol (97%) was purchased from Aldrich. CA, PA and other reagents were analytical grade, purchased from Beijing Chemical Company, used without further purification. For all experiments, deionized water was used.

2.2. Preparation of coatings

Cu substrate (10 mm × 10 mm × 1 mm) was sealed by epoxy except leaving 1 cm² as analyzed surface. The analyzed area was polished with SiC papers of different grit 350, 600, 1500 and 2000, rinsed by deionized water and ethanol, respectively, and finally blown dried by N₂. Cu substrate for neutral salt spray experiment was machined to 50 mm × 30 mm × 3 mm and was pre-treated as epoxy sealed sample. CA (5 mM) and PA (2.5 mM) were dissolved in deionized water without adding any other agents. The Cu substrates were immersed in the freshly prepared solution at ambient temperature for 18 h. Then, the substrates were taken out, rinsed with deionized water and ethanol, respectively, and dried by N₂. The modified samples were coded as Cu-P(CA/PA).

The hydrophobic modification was performed by adding Cu-P(CA/PA) into deoxygenized solution of 1-dodecanethiol in ethanol (1:100 v:v). After 12 h, the sample was taken out, rinsed with ethanol and then dried with N₂. The 1-dodecanethiol modified samples were denoted as Cu-P(CA/PA)-SH. Due to the spectra obtained from attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy are obscure and the coatings are too few, net P(CA/PA) and P(CA/PA)-SH were prepared for FTIR spectra (KBr) and thermogravimetric (TG) analysis. The free-standing P(CA/PA) film was collected from the interface between air and CA/PA mixed solution (Fig. S1). P(CA/PA)-SH was synthesized according to the above mentioned approach.

2.3. Characterizations

The scanning electron microscopy (SEM) images were observed on a field emission scanning electron microscopy (JSM 7500F, JEOL, Japan) at 5 kV. The contact angle was tested on a Contact Angle Meter (DSA100, Krüss Company, Germany) by a sessile droplet method using 5 μL water. X-ray photoelectron spectrometry (XPS) was performed on an ESCALab 220I-XL spectrometry (VG Scientific) with an Al Kα X-ray source (1486.6 eV). To evaluate the state of Cu after P(CA/PA) deposition, the surface of Cu-P(CA/PA) was etched by Ar ion beam until the Cu element was detected. XPS and X-ray excited Auger [Cu(L3M4,5M4,5)] spectra were recorded. FTIR spectra were recorded on a FTIR Spectrometer (model 1600, Pekin-Elmer Co., USA). The test specimens were obtained by KBr-disk method. TG analysis was carried on a thermogravimetric analyzer (Q600, TA Instruments, USA) under nitrogen atmosphere. The specimen was put in an Al₂O₃ pan and heat from 30 °C to 700 °C with a heat rate of 10 °C/min.

2.4. Corrosion tests

The electrochemical corrosion tests were performed at room temperature in a standard three-electrode cell, containing a saturated calomel reference electrode (SCE), a Pt net counter electrode and a working electrode of bare or modified Cu sheets. The potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) curves were recorded on an electrochemical workstation (Reference 600, Gamry, USA) in 3.5 wt% NaCl solution. Potentiodynamic polarization curves were obtained with a scan rate of 5 mV/s in Tafel model after the open circuit potential (OCP) became stable. EIS measurement was operated at the OCP in the frequency range from 10⁵ to 0.01 Hz with different immersing time. For immersion tests, epoxy sealed Cu substrates (10 mm × 10 mm × 1 mm) were immersed in 200 mL 3.5 wt% NaCl solution at a temperature of 25 ± 2 °C. The immersion duration was three weeks. The concentration of Cu ions in solution during immersion test was detected by a volt-ampere polarograph (797 VA Computrace, Metrohm AG, Switzerland). Neutral salt spray test was carried out in a salt spray test chamber according to the standard ASTM B-117 [19]. Specimens were exposed to a continuous indirect spray of 5 wt% NaCl aqueous solution (pH 6.5–7.2) at a temperature of 35 °C. The fell rate of spray was about 0.013–0.025 mL cm⁻² h⁻¹. The test duration was ten days.

3. Results and discussion

The molecular structures of CA, PA and 1-dodecanethiol, and typical procedure of preparing the anti-corrosion coating are show in Scheme 1. The P(CA/PA) coating was firstly formed via polymerization of CA and PA. Then, the hydrophobic coating was produced by modification of P(CA/PA) with 1-dodecanethiol. The possible interaction of coating with substrate and possible chemical

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