



Green approach to fabrication of a super-hydrophobic film on copper and the consequent corrosion resistance



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ARTICLE INFO

Article history:

Received 31 May 2013

Accepted 29 November 2013

Available online 5 December 2013

Keywords:

A. Copper

B. EIS

B. SEM

B. Polarisation

C. Interfaces

ABSTRACT

A green approach was used to fabricate super-hydrophobic film with capsaicin (N-(4-hydroxy-3-methoxybenzyl)-8-methylnon-trans-6-enamide), pungent ingredient of cayenne. The film is composed of capsaicin that coordinates with Cu(II) through π orbital of benzene ring. Super-hydrophobic property of film can be attributed to nanospherical pores of its structure and low surface energy of its hydrocarbon chain. Super-hydrophobic film shows >99.9% efficiency as a barrier that inhibits corrosion of underlying Cu without losing its stability within a wide potential range. This performance can be attributed to isolating action and insulating property of air trapped in film.

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1. Introduction

Copper (Cu) is widely used in chemical and micro-electronics industries because of its excellent thermal and electrical conductivities [1]. However, Cu is an active metal that does not resist corrosion well [2].

Wettability of a solid surface is an important factor that influences corrosion process. Compared with hydrophilic surfaces, hydrophobic surfaces exhibit better inhibitory effect on substrate corrosion. Recently, artificial super-hydrophobic surfaces (water contact angle larger than 150°), the concept of which was derived from water-repellent nature of lotus leaf, have attracted considerable attention because of their potential application in corrosion protection [3–12]. Low surface energy and rough structure are required for fabrication of super-hydrophobic films. Numerous methods have been proposed to fabricate super-hydrophobic films on metal surfaces; most of these methods, such as electrochemical deposition, involve modification of self-assembled films with low surface energy on rough metal surfaces [13,14]. However, self-assembled films cannot resist attraction of corrosive ions and hydration action well. Exposure of a substrate under destroyed self-assembled film accelerates corrosion process. This phenomenon limits application of super-hydrophobic films in corrosion protection. To date, very few methods and chemicals are preponderant in fabrication of super-hydrophobic films for corrosion protection.

With increasing public awareness on environmental protection, demand for environmentally friendly corrosion protection techniques has become urgent. Extracts of plants, such as *Phyllanthus amarus* [15], *Salvia officinalis* [16], bamboo [17], and others [18–24] were reported to act as effective inhibitors to metal corrosion. Capsaicin, the pungent active ingredient of cayenne, produces painful sensations upon cutaneous application by activating the transient receptor located on the peripheral terminals of nociceptors [25]. Capsaicin can inhibit generation of reactive oxygen species in rat peritoneal macrophages [26], and induce apoptosis [27,28] and electron transfer in bovine heart mitochondrial complex I [29]. To the best of our knowledge, the present study is the first to report application of capsaicin in corrosion protection.

In this study, capsaicin was used to fabricate super-hydrophobic film by electrolysis. The resultant film was characterised by scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS), and its corrosion protection property was evaluated by electrochemical measurements. Formation mechanism and corrosion resistance mechanism of super-hydrophobic film were proposed based on these results.

2. Experimental

2.1. Reagents and materials

Cu (≥ 99.5 wt.%), NaCl and ethanol were purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. and used as received. Milli-Q water (Milli Q, USA) was used in all experiments. N-(4-hydroxy-3-methoxybenzyl)-8-methylnon-trans-6-enamide (95 wt.%)

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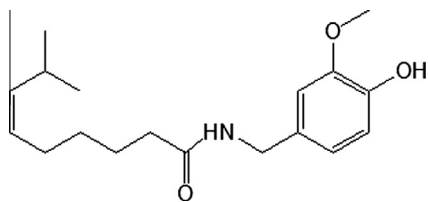


Fig. 1. Molecular structural formula of capsaicin used.

(HMMTE) extracted from cayenne was purchased from Qingdao Haida Chemical Co., Ltd. Its molecular formula is shown in Fig. 1.

2.2. Fabrication of super-hydrophobic film

Cu foil with an area of 1 cm^2 was abraded with emery paper up to 400 grade, degreased with ethanol, and then blow dried. Super-hydrophobic film was grown in a two-electrode cell, with Cu as anode and a platinum wire as cathode. A 0.02 M HMMTE/ethanol solution was used as electrolyte. Film was grown by applying a cell voltage of 5–30 V between anode and cathode from 30 min to 2 h at room temperature. The constant voltage was supplied by an uninterrupted DC power system purchased from Shenzhen Parwa Technology Co., Ltd. After electrolysis, Cu foil was brought out, washed with ethanol, and then dried in nitrogen flow.

2.3. Surface characterisation

Morphologies of samples were characterised with SEM (JSM-6700F). Chemical composition information was obtained by XPS on a Thermo ESCALAB 250 photoelectron spectrometer equipped with an Al-anode at a total power dissipation of 150 W (15 kV, 10 mA). Water contact angle was measured with a contact angle metre (JC2000C1) at ambient temperature. Quantum chemical calculations were performed by GAUSSIAN 03 software using B3LYP functional and a 6-31G^{*} basis set.

2.4. Electrochemical experiments

Electrochemical experiments, including open circuit potential (OCP)– t curves, electrochemical impedance spectra (EIS), and polarisation curves, were obtained with a computer-controlled electrochemical system (CHI 760C, CH Instruments Inc.) in 3.5 wt.% NaCl solution at room temperature. These experiments were performed in a three-electrode cell, with a platinum wire as counter electrode, Cu/filmed Cu as working electrode, and Ag/AgCl (3 M KCl) as reference electrode. EIS experiments were carried out at frequency range of 10^5 Hz to 10^{-2} Hz at open circuit potential with amplitude of perturbation voltage $\pm 5 \text{ mV}$. The system was assumed to achieve a stable state after sample was immersed into test solution for an hour. Thus, all samples were immersed in 3.5 wt.% NaCl solution for an hour before EIS measurement. EIS results were analysed by fitting data using Zsimpwin software. Each test was repeated for three times to verify repeatability of results, meanwhile the mean values and standard deviations of electrochemical parameters are reported. Polarisation curves were recorded at a sweep rate of 1 mV s^{-1} .

3. Results and discussion

3.1. Morphology and wettability of film

Fig. 2 presents wettability and morphology of Cu electrolysed under cell voltage ranging from 5 V to 30 V for 2 h in 0.02 M HMMTE/ethanol solution. As shown in Fig. 2a, bare Cu surface is relatively smooth, except for some nicks, and presents a hydrophilic property with a water contact angle of $52.0 \pm 5^\circ$. After electrolysis under a cell voltage of 5 V, contact angle of water on Cu increases to $78.5 \pm 3^\circ$, and no obvious deposits can be found on Cu surface (Fig. 2b). After electrolysis under a cell voltage of 15 V, some deposits form on Cu surface, and filmed Cu presents a hydrophobic property with a contact angle of $93.3 \pm 3^\circ$ (Fig. 2c). After electrolysis under a cell voltage of 30 V, a film with a porous

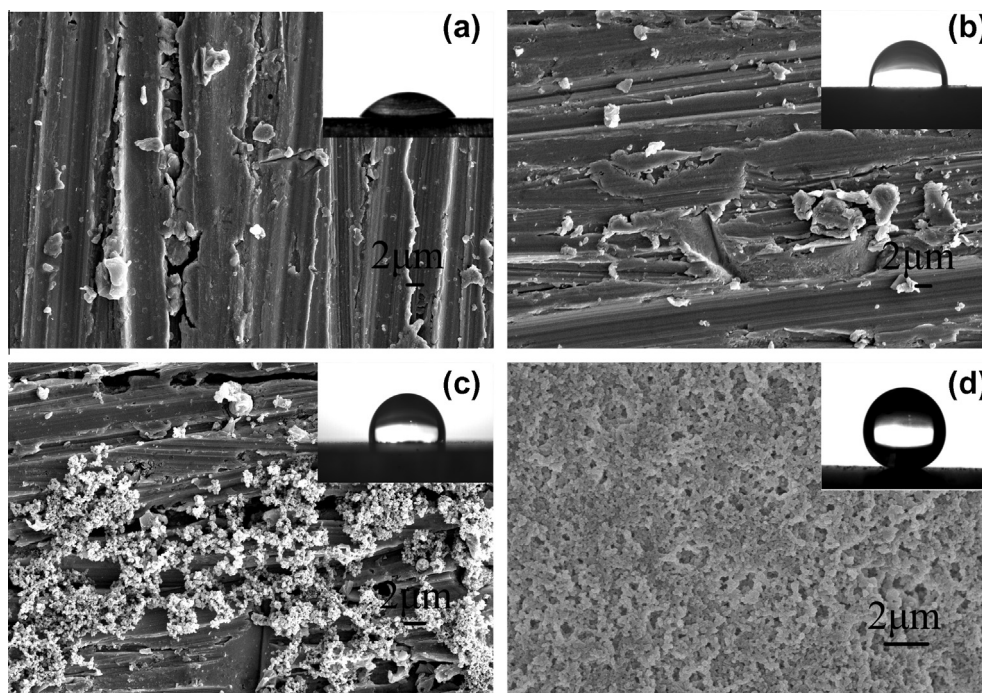


Fig. 2. Wettability and morphologies of (a) bare Cu and Cu electrolysed under cell voltage of (b) 5 V, (c) 15 V, (d) 30 V for 2 h in 0.02 M HMMTE/ethanol solution.

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