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# Colloids and Surfaces A



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# Formation of a hydrophobic and corrosion resistant coating on manganese surface via stearic acid and oleic acid diethanolamide



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# GRAPHICAL ABSTRACT



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#### ABSTRACT

A novel hydrophobic passivation coating was fabricated on the surface of electrolytic manganese by stearic acid (SA) and oleic acid diethanolamide (ODEA). ODEA, which forms micelles in aqueous solution, can promote largely dissolution of insoluble stearic acid to form stable passivation solution. A series of influence parameters were investigated and the optimized process conditions were as follows: the concentration of SA and ODEA were  $3 g/L$  and  $10 g/L$ , respectively; and the immersion time was 5 min. Under the optimized condition, the anticorrosion properties of the coating were characterized by electrochemical impedance spectroscopy and polarization test. The results showed that the corrosion potential and corrosion current density decreased obviously, and the polarization resistance at 0.01 Hz increased from 20.89 to 467.74 Ohm cm<sup>2</sup>. FT-IR spectroscopy and Gaussian calculations showed that SA and ODEA simultaneously acted on the surface of manganese to form a hydrophobic coating, which increased the contact angle from 41° to 113°.

#### 1. Introduction

Manganese is a relatively active chemical properties of transition metal, with the atomic number 25 in the periodic table. As an important metal element, manganese has an extraordinary significance in the alloy, medicine, batteries industry, electronic component and scientific researches [1–[3](#page--1-0)]. The preparation methods of manganese metal are generally divided into pyrometallurgical and hydrometallurgical processes, both of which use pyrolusite and rhodochrosite as the main raw material [\[4\]](#page--1-1). The hydrometallurgical process mainly refers to the electrolytic method, and it is the most important preparation method of the industrial production of manganese metal due to the fewer

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impurities and higher purity of obtained manganese metal. However, manganese metal is easily oxidized in the air for its low potential, resulting in reduced purity and performance of the manganese product [[5](#page--1-2)]. Therefore, some measures must be taken to protect electrolytic manganese to improve its anti-corrosion performance. In industry, potassium dichromate is often used as a passivator to form a passivation coating on the metal surface, and the passivation coating has good wear resistance and self-healing ability. Nonetheless, the utilization of hexavalent chromium is very harmful to the ecological environment and human existence for its strong carcinogenicity and toxicity [\[6,](#page--1-3)[7](#page--1-4)]. At present, numerous inorganic and organic chromium-free passivation agents have been developed to replace potassium dichromate, such as molybdates [8–[10\]](#page--1-5), titanium salts [\[11](#page--1-6),[12](#page--1-7)], tungstates [\[13](#page--1-8)–15], phytic acid [16–[18\]](#page--1-9), silane [19–[21\]](#page--1-10), chitosan and so on. However, there are still many drawbacks along with these methods, such as high cost, general anti-corrosion effect and complicated fabrication process.

Inspired by the microstructure of the leaf surfaces of lotus leaves, red rose petals and other plant leaves, the hydrophobic coating has been successfully fabricated in many metallic materials [\[22](#page--1-11)–28]. With low surface energy and high surface roughness, the hydrophobic surfaces are considered as promising coating to improve the corrosion resistance of metals. For example, R. Jain and R. Pitchumani [[28\]](#page--1-12) reported a durable and corrosion-resistant hydrophobic zinc coating that was electrodeposited with a zinc acetate electrolyte and modified with stearic acid (SA). Karthik et al. [\[29](#page--1-13)] fabricated superhydrophobic composite coating on aluminum surface via electroless copper deposition and surface modification with laurylamine. Zheng et al. [[30\]](#page--1-14) fabricated a hydrophobic coating on the surface of magnesium alloy via one-step hydrothermal method, which showed great improvement in corrosion behavior. However, studies on the preparation of hydrophobic coating on electrolytic manganese to improve corrosion performance are rarely reported.

SA is widely used in the preparation of hydrophobic surfaces because of its non-toxic, easily available and hydrophobic properties [\[31](#page--1-15)]. Although SA has many benefits, it is insoluble in aqueous solution. Fortunately, surfactants can make molecules that are insoluble enter the micelles that form within themselves, thereby achieving solubilization. In our work, SA and oleic acid diethanolamide (ODEA) were used simultaneously to form the passivation solution. The surfactant ODEA not only acted as a solubilizer to promote the dissolution of SA, but also combined with SA resulting in the formation of negatively charged stable micelles in solution, thereby enhancing the stability of the passivation solution. In addition, both SA and ODEA are inexpensive and harmless to the environment.

In this paper, an environmentally friendly method was used to fabricate a hydrophobic passivation coating on electrolytic manganese metal via cheap SA and ODEA. Scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FT-IR) were used to determine the morphologies and chemical compositions of the coating. The wettability of the coating surface was measured through the contact angle. And the corrosion performance of the coating was characterized by electrochemical method. The effects of various factors on the properties of the passivation coating and the formation mechanism of the passivation coating were also investigated. Herein, we show a simple way to improve the corrosion resistance of electrolytic manganese, and hope that this simple method without any environmental toxicity can accelerate the application of electrolytic manganese and may be of some help to other metal.

## 2. Experimental

#### 2.1. Material and reagents

All the reagents used in the experiment were analytical grade purity except for the chemical grade purity of the ODEA, and all experiments were carried out by distilled water.

#### 2.2. Fabrication of passivation coating

#### 2.2.1. Preparation of electrolytic manganese

The stainless steel was polished mechanically by sandpaper of #600, #800 and #1200, then soaked in absolute ethanol for 2 min and rinsed with distilled water to eliminate the surface pollution. For the electrowinning experiment, 304 L stainless steel with a geometric area of 2.75  $\times$  3.82 cm (unilateral effective area of 10.505 cm<sup>2</sup>) was used as the cathode and the Pb-Sn-Sb-Ag alloy plate was used as the anode. Then, 60 ml of the catholyte and anolyte were added to the cathode tank and anode tank, respectively, wherein the anolyte was 120 g/L (NH<sub>4</sub>)  $_2$ SO<sub>4</sub> and the catholyte was 25 g/L Mn<sup>2+</sup>, 120 g/L (NH<sub>4</sub>) $_2$ SO<sub>4</sub> and  $0.05$  g/L SeO<sub>2</sub>. In order to improve the current efficiency, the pH of the catholyte was adjusted to about 7 with sulfuric acid and aqueous ammonia. Pure manganese was obtained on the cathode plate after electrodeposition for 1.5 h at a bath temperature of 36 °C and a constant cathode current density of  $350 \text{ A/m}^2$ .

#### 2.2.2. Passivation of electrolytic manganese

A certain amount of SA and ODEA were added to a beaker, and ODEA served as a solubilizer to increase the solubility of SA in the water. The SA and ODEA were fully mixed to form a paste under magnetic stirring at 80 °C. Then, a small amount of 80 °C hot water was added to mix with the mixture to form passivation solution. After that, the cathode plate after 1.5 h of electrodeposition was taken out and rinsed with distilled water to remove the remaining electrolyte. Next, the cathode plate with pure manganese was immersed in the passivation solution for a certain period of time, rinsed with distilled water and finally dried.

### 2.3. Electrochemical experiments

The electrochemical measurements were conducted in 3.5 wt% NaCl aqueous solution at room temperature using electrochemical workstation (CHI604D). The corrosion tests were performed in a threeelectrode cell, composed of a platinum electrode as the counter electrode, Mn modified with passivation coating as the working electrode, and AgCl electrode as the reference electrode. Before the test, the area of 0.09 cm<sup>2</sup> was reserved as an effective detection zone with the rest area sealed with F01-1 novolac. In addition, the test sample was immersed in the test solution for about 20 min until the open circuit potential (OCP) reached stability before starting the test. The Tafel polarization curves were achieved by scanning from  $-300$  to  $+300$  mV versus the OCP at scan rate of 2 mV/s. For the electrochemical impedance spectroscopy (EIS), measurements were performed with a  $5\,\mathrm{mV}$  sinusoidal signal at the frequency range of  $10^{\,-2} \sim\! 10^{\text{-}5}\,\mathrm{Hz}$  with  $7$ points for each order of magnitude. All the results were analyzed by the analyst software after the test.

#### 2.4. Surface characterization

Microstructure of surfaces was characterized by using a MIRA 3 LMU field emission scanning electron microscope at an accelerating voltage of 20 kV. Infrared spectroscopy test was performed on a Nicolet6700 Intelligent Fourier Transform Infrared Spectrometer. Taking distilled water as the test solution, the contact angle of different samples was determined using JC2000 contact angle tester at ambient temperature.

## 2.5. Zeta potential test

It is an effective way to use Zeta potential test to determine the stability of the colloidal solution system. In this experiment, the ZetaPALS (Brookhaven, America) was used to evaluate the stability of the passivation liquid system, and each sample was tested 8 times to obtain the average value.

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