



Fabrication of silane coating with improved protection performance using *Mentha longifolia* extract

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ABSTRACT

This work reports incorporation of *Mentha longifolia* in a silane coating to improve active corrosion protection. Fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA) were employed to characterize the chemical structure of extract. Electrochemical impedance spectroscopy (EIS) revealed that superlative corrosion resistance of silane primer was achieved at 200 ppm inhibitor concentration. DC polarization and open circuit potential (OCP) measurements confirmed the EIS results. The effect of inhibitor concentration on the crosslinking density was evaluated by FTIR and its subsequent effect on the coating wettability was assessed by water droplet contact angle method. The protective performance of the artificially defected bilayer coating system (silane + epoxy) assessed by EIS and surface analysis showed an improvement in the anticorrosion properties of silane formulated with 200 ppm extract due to the corrosion reaction restriction. Solution phase studies by electrochemical test and SEM indicated participation of a protective layer on the surface.

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1. Introduction

Application of coatings is a promising strategy in the corrosion protection of metallic substrates, particularly steel structures in numerous fields such as pipelines, constructions, automotive, household, etc. Epoxy coatings as a favorable physical barrier between the substrate surface and corrosive media have been widely employed to improve the corrosion resistance of steels [1–6]. In spite of presenting suitable barrier effect, coatings are permeable and eventually aggressive species find their way to the metal surface, leading to a decrease in the coating adhesion and progress of the underfilm corrosion [7]. Thus the protective function of coatings depends highly on the adhesion properties of coating/substrate interface. Organofunctional silane films have been successfully used as an effective adhesion promoter to enhance the bonding of organic coatings to metallic substrates [8–12]. In addition, they have also been reported to offer corrosion protection for various substrates [12–17]. The silane compounds used for coating application comprise at least one non-hydrolyzable functional group (amino, epoxy, etc.) that chemically interact with overlying organic coating and also hydrolyzable portions which are responsible for the formation of covalent bonds with the substrate surface. The primary

protective mechanism of a hybrid silane coating is to develop a barrier siloxane network, obtaining through condensation of silanol (Si–OH) groups [18–20]. The paramount drawback of silane pre-treatments is to provide no corrosion protection after penetration of aggressive species to the substrate/coating interface [21–24].

One of the ways offering active protection is to add corrosion inhibitors to the silane coating formulation. Some inorganic and organic corrosion inhibitors have been already examined by this group to enhance the protective function of silane sol-gel coatings on steel and aluminum substrates in sodium chloride solution. In the case of inorganic inhibitor, the positive impact of zinc cation on the active protection of an ecofriendly silane coating composed of methyltriethoxysilane, γ -glycidoxypropyltrimethoxysilane and tetraethoxysilane applied on mild steel was connected to the precipitation of a layer composed of zinc hydroxide and oxide at the sol-gel coating/substrate interface. No significant effect on the coating barrier properties was observed in the presence of Zn^{2+} [25]. The optimum concentration of $Ce(NO_3)_3$ enhancing the corrosion protection of the three-component silane film on Al was also determined elsewhere [26]. Furthermore, zinc cation and 2-mercaptobenzoxazole were incorporated in silane coating offering a significant corrosion resistance to the mild steel substrate in sodium chloride solution due to the inhibition synergism of the organic and inorganic inhibitors. The behavior was mainly linked to the formation of zinc hydroxide as well as complexes between the mercaptobenzoxazole molecule and Zn and Fe cations at the coating/metal interface [27].

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Due to the toxicity of synthetic organic inhibitors, many attempts have been done to find green alternatives which are biocompatible, economic, readily renewable and have the lowest side effect for environment [28]. Most parts of plants such as leaf, fruits peel, etc., are rich of the compounds that give them the capability to be used as corrosion inhibitor for various substrates in different electrolytes [29–31]. The effective role of green inhibitors in enhancement of the corrosion resistance of metallic substrates in acidic solutions is frequently reported in the literature. However, there are a few researches related to formulation of protective coatings with the green inhibitors. For instance, Motalebi et al. [32] determined the most effective concentration of henna extract in polyvinyltrimethoxysilane (PVTMS) coating to improve the corrosion resistance of stainless steel 316L in simulated body fluid (SBF) solution. The corrosion protection in the presence of henna extract was mainly connected to the formation of complexes between iron cations and lawsone as the main henna component as well as modification of the coating defects.

The *Mentha longifolia* (ML), also known as wild mint or horsemint, belongs to lamiaceae family, which is an herbaceous perennial plant. ML itself has not been employed as a corrosion inhibitor yet; however, the usage of other species of lamiaceae family such as *Mentha spicata* or *Mentha rotundifolia* has been reported by some authors only for acid solutions. Znini et al. [33] indicated that *Mentha spicata* oil which contains carvone as a major constituent act as a mixed-type inhibitor through physisorption on the surface of steel in 1 M HCl and provided 97% inhibition efficiency at the concentration of 2 g/l. Ansari et al. [34] showed the adsorption of the oxygenated component of *Mentha rotundifolia* including pulegone and menthone leading to proper inhibition of mild steel towards 1 M HCl medium.

The present study intends to formulate a water-based hybrid silane film with *Mentha longifolia* (ML) to control the corrosion of mild steel in a sodium chloride solution. To this end, firstly the extract was characterized, then its corrosion inhibition in solution phase on the bare steel was evaluated. Afterwards, the protective performance of single layer silane coating containing ML extract and bilayer system composed of silane primer and epoxy top-coat were studied.

2. Experimental

2.1. Materials

Mild steel panels with the dimensions 3×6 cm and the chemical composition (in wt.%: Fe: 97.7, C: 0.19, Si: 0.415, Mn: 1.39, $p < 0.005$, S < 0.005, Cr: 0.026, Mo: 0.018, Co: 0.0559, Cu: 0.0429, Nb: 0.0481) were purchased from Foolad Mobarakeh Co, Iran. The ML powdered extract was obtained from Ebn-e-Masouyeh pharmaceutical Co, Iran. The silane precursors, methyltriethoxysilane (MTES), γ -glycidoxypropyltrimethoxysilane (γ -GPS) and tetraethoxysilane (TEOS) [17,27,35,36] were prepared from Aldrich. Polyamide hardener (crayamid 115), epoxy resin (E6) and butyl acetate were supplied respectively by Cray Valley Co, Khouzestan Petrochemical Co and Sigma-Aldrich.

2.2. Sample preparation

The steel samples were first abraded with abrasive papers of 400 to 1000 grit size and degreased with acetone. In order to activate the surface of samples prior to applying silane film, an alkaline treatment was carried out through 7-min dipping in 25 g/l NaOH solution at 55 °C. Then, the samples were rinsed in distilled water and dried with compressed air. In order to prepare the 10 wt. % silane coating solution, the mixture of three silane precursors with a weight ratio of 1/1/1 was added to distilled water

with pH 3 adjusted by acetic acid and stirred for 24 h at ambient temperature. Before the 24-h stirring, different concentrations (100, 200 and 400 ppm) of ML were also added to the mixture. Finally, the surface pretreated samples were immersed in the silane solution for 2 min at ambient temperature. The curing temperature and time were 150 °C and 30 min, respectively.

In order to prepare topcoat, the epoxy resin and polyamide hardener were mixed in a 1.13/1 (w/w) ratio. Butyl acetate was added to dilute the coating material before air-spray application on the silanized and bare metallic substrates. The epoxy-polyamide coated samples left at 25 °C for 24 h then placed in an oven at 80 °C for 40 min to ensure complete curing. The dry film thickness measured by an Elcometer digital coating thickness gauge Model 456 was $45 \pm 5 \mu\text{m}$. Finally, an artificial scratch with an approximate 5 mm length was made by a sharp blade on the samples. Before making scratches, the samples were exposed to salt spray for 24 h, then left at ambient temperature for 3 h.

2.3. Methods

2.3.1. FTIR

The FTIR analysis of the chemical structure of ML extract as well as silane coating was performed by a Perkin-Elmer spectrum instrument through KBr pellet preparation method within 4000–450 wavenumber range. To prepare the powder of silane coatings, small droplets of the silane solution were placed on a flat Teflon sheet and subsequently cured at 150 °C for 30 min in an oven. Then, the dried flakes were finely powdered.

2.3.2. Thermogravimetric analysis (TGA)

To perform TGA analysis using Mettler-TGA/DSC instrument a heating ramp of 10 °C/min was chosen to rise temperature from 25 °C to 700 °C under a nitrogen atmosphere.

2.3.3. SEM

A SEM-EDX Model Camscan mv2300 was employed to study the morphology and composition of bare steel surface after exposure to extract saline solution and the surface morphology and cross-section of silane coatings. The morphology and composition of scratched and vicinity areas after corrosion test was also studied by SEM-EDX.

2.3.4. Contact angle

Wettability of silane coating was analyzed through measuring the contact angle of water droplets (4 μL) by employing OCA 15 plus.

2.3.5. Electrochemical measurements

An electrochemical cell including the sample under study as working electrode, saturated calomel reference electrode (SCE) and platinum counter electrode was used to perform the EIS and polarization tests. In the electrochemical measurements, the area of bare metal, silanized sample and sample with silane/epoxy coating exposed to the electrolyte was 1.0 cm², 1.0 cm² and 2.0 cm², respectively. The rest areas including edges and backside of the samples were sealed using a beeswax colophony mixture.

2.3.5.1. Open circuit potential (OCP). To measure the OCP values during 12 h immersion, the sample under study along with a saturated calomel reference electrode were connected to a Sanwa digital multimeter model 800a.

2.3.5.2. EIS. Using an ACM-Gill instrument, the EIS measurements were performed in a frequency range of 10 kHz to 10 mHz at open circuit potential (OCP) with a sin wave of 10 mV perturbation. The AC impedance spectra were analyzed using Zview3.1c software.

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