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Development and evaluation of an eco-friendly hybrid epoxy-silicon coating for the corrosion protection of aluminium alloys

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ABSTRACT

The present research work is focused on the formulation, synthesis and evaluation of three novel eco-friendly hybrid epoxy–silicon (EP-S) coatings. For comparative purposes, two commercial epoxy (EP) coatings were also evaluated. The protective properties of the coatings were evaluated by means of Electrochemical Impedance Spectroscopy (EIS), also supported by physico-chemical characterization of the coated surfaces by Fourier Transformed Infrared Spectroscopy (FTIR), Atomic Force Microscopy (AFM), as well as Contact Angle (CA) and Energy Surface measurements. The very high impedance (Z) values confirmed the excellent protective properties and the suitability of the novel EP-S coatings to protect 5005 aluminium substrates exposed to a simulated harsh marine environment (10% (w/w) NaCl solution), when compared with commercial EP coatings providing an adequate corrosion protection performance. Surface characterization tests to the hybrid coatings corroborated their excellent barrier properties. The comparison of the IR spectra before and after immersion test in the referred NaCl solution demonstrated the absence of water inside the EP-S coatings. Additionally, the very high water contact angle ($> 90^\circ$) and low surface energy values, confirmed the hydrophobic nature as well as the low wettability of the hybrid coatings. It was concluded that the addition of silane containing an amine functional group allowed obtaining a denser and more effective protective coating.

1. Introduction

Aluminium alloys are the subject of a great industrial and scientific interest due to their high technological value and applicability, especially in the aircraft and aerospace fields. However, they are prone to suffer corrosion in aggressive environments due to the presence of a heterogeneous microstructure (alloy elements such as Cu, Mg, Zn, Si) limiting their applications [1–3]. Coatings protection is considered one of the most successful and cost-effective alternatives to increase the service life of metallic structures including Al alloys.

Snihirova et al. [4] have pointed out that coatings follow two main mechanisms to prevent aluminium corrosion: barrier and smart protection [4]. The first one takes place due to the presence of layers that hinder the entry of aggressive species to the substrate. This is the case of anodization [5,6], conversion coatings [7], sol-gel coatings [2,8], and highly cross-linked organic polymeric coatings (waterborne or solvent-based) [7,9]. However, barrier protection might fail after some time, thus leading to the oxidation of the substrate. Therefore, inhibitive pigments are normally added in order to achieve longer-term protection of the areas under corrosion activity, responding to a smart protection [4,9].

The development of advanced functional and smart coatings for corrosion protection in different technological applications is an issue in the limelight. A recent review on the topic has highlighted the relevance of self-healing and smart coatings by combining multiple functionalities for increased corrosion protection. In this sense, the introduction of functionalities based on encapsulation of corrosion inhibitors, anti-fouling agents and superhydrophobic additives or the modification of organic and hybrid matrices via chemical manipulation are some of the latest developments [9].

Advanced functional coating with low volatile organic compounds (VOCs), based on waterborne formulations and isocyanate-free compositions have been receiving a great attention in the last years [7,9]. Siloxanes-modified chemicals, cyclic carbonates and amines are considered “greener” routes to develop functional barrier coatings for multi-substrate application [7,10,11]. Organic-inorganic hybrid coatings, and more specifically the epoxy-siloxane ones, respond to most of the aforementioned demands such as low VOCs content and isocyanate-

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free composition. Recent studies have demonstrated the excellent anticorrosive performance of these hybrid coatings on steel substrates after being exposed to constant immersion in a 3.5% (w/w) NaCl solution and to salt spray tests, with regard to traditional ones such as polyurethane [12,13]. Analogously, positive results were also concluded for aluminium substrates [9,14,15].

Montemor has pointed out that the addition of different additives (silanes, amines, polyaniline) to modify cross-linking and molecular structure of polymeric matrices (epoxy, polyurethane or acrylic ones) allowed the development of thinner but denser functional coatings, with significantly better barrier properties. This fact has led to an increased corrosion protection, durability, and cost effectiveness [9]. In relation to this, some authors agree that the combination of siloxane-based coatings with epoxy (EP) and acrylic resins have been the most successful alternative, and siloxane concentration decisively modify the desired coating properties [13–17]. A recent investigation has focused on the potential of silanes and amines as cross linkers in order to develop hybrid epoxy-silane coatings with high protective performance for the protection of magnesium alloys used in the aeronautical industry [16]. The addition of silanes with an amine functional group led to a higher corrosion protection performance, promoting the formation of a denser and more cross-linked hybrid matrix, due to the bonding of the amine groups in diethylenetriamine with the glycidyl group in the EP component [16]. Another advantage of siloxane functionalities is the possibility to reduce the quantity of layers, or the thickness of the layers, thus leading to a shorter coating application and cost savings, as reported recently [9].

On the basis of all this information we have aimed at developing and evaluate three novel eco-friendly hybrid epoxy-silicon (EP-S) coatings, functionalized with a type of silane (3-aminopropyltriethoxysilane at different concentrations), for the corrosion protection of AA5005 aluminium alloy submitted to a simulated harsh marine environment (10% (w/w) NaCl solution). The corrosion protection behavior of the hybrid coatings was monitored by means of Electrochemical Impedance Spectroscopy (EIS), and further evidences of the response of the coatings to this extreme marine environment were obtained by Fourier Transform Infrared (FTIR) Spectroscopy, Atomic Force Microscopy (AFM), and Contact Angle (CA). For comparative purposes two commercial EP coatings were also included in the investigation.

2. Experimental

2.1. Materials

2.1.1. Substrate preparation

Metallic panels of 5005 aluminium alloy, with dimensions 100 mm length, 100 mm width and 1 mm thickness, were the samples employed for evaluating the corrosion protection performance of the coatings under study. The aluminium alloy nominal composition is 0.40% Si, 0.70% Fe, 0.20% Cu, 0.10% Mn, 1.80% Mg, 0.10% Cr, 0.25% Zn, 0.10% Ti (weight percentages) and Al balanced with previous elements. Metallic surfaces were cleaned with 99.5% ACS grade acetone (Sigma Aldrich). Then, the metallic surfaces were sanded to 1000 grit sandpaper. Later, samples were kept in the desiccator (for 2 h) till the coating preparation and application.

2.1.2. Coatings composition

Three hybrid EP-S coatings (identified as EP-S1, EP-S2 and EP-S3) and two commercial EP coatings (identified as EP1 and EP2), are the 5 coatings under study. The EP-S coatings were formulated and synthesized in the lab, in contrast to the EP commercial ones, which were bought to two coating manufacturers. The coating selection was carried out considering the aggressiveness of the media (simulated harsh marine environment) in which they would be exposed. Due to a nondisclosure agreement, the identity of the EP coating manufacturer cannot be included. The EP-S coatings are characterized as three different types of two pack EP hybrid coating materials. The polymer base and the curing agent for each EP-S formulation are presented in Table 1. The same filler particulates, additives and solvents were added (with the same concentration) to all EP-S formulations. The filler particulates consisted of titanium dioxide pigments and talcum (D50, 2.5 µm). As additives, a deaerator (Airex 955) and a dispersing additive (Dispers 685) were selected in order to get a foam free coating film and a good particle wetting. Additionally, *n*-butyl acetate solvent was used for adjusting viscosity. The coating raw materials were not treated prior to their involvement in the coating mixture. They were stored at room temperature (23 °C). Compositions of EP-S coatings are summarized in Table 2.

Table 1
Polymer base and curing agent of the epoxy-silicon (EP-S) coatings.

Coating	Polymer base	Curing agent
EP-S1	Silicone epoxy hybrid resin	3-Aminopropyl-triethoxysilane
EP-S2	Liquid epoxy bisphenol-A + A/F − liquid epoxy resin + Low molecular Silicon-epoxy resin	3-Aminopropyl-triethoxysilane + Cycloaliphatic polyamine (type 1) + Cycloaliphatic polyamine (type 2)
EP-S3	Liquid epoxy bisphenol-A + A/F − liquid epoxy resin + Low molecular silicon-epoxy resin	3-Aminopropyl-triethoxysilane + Cycloaliphatic polyamine (type 1) + Cycloaliphatic polyamine (type 1)

Table 2
Chemical composition of the epoxy-silicon (EP-S) coatings (EP-S1, EP-S2 and EP-S3).

Components	EP-S1	EP-S2	EP-S3
Component A		Concentration (g/L)	
Silicone epoxy hybrid resin	64.50	–	–
Liquid epoxy resin is a reaction product of epichlorohydrin and bisphenol-A	–	30.00	30.00
A/F–liquid epoxy resin; reactive diluted	–	15.00	15.00
Low molecular silicon-epoxy resin	–	7.50	12.50
Deaerato (Airex 955)	1.00	1.00	1.00
Dispersing additive (Dispers 685)	1.50	1.50	1.50
Titan dioxide	5.00	5.00	5.00
Talcum	3.00	3.00	3.00
Butyl acetate	9.00	14.85	9.35
Component B			
3-Aminopropyltriethoxysilane	16.00	0.75	1.25
Cycloaliphatic polyamine (Amine number 365–385 mg KOH/g)	–	12.65	12.65
Cycloaliphatic polyamine (Amine number 300–350 mg KOH/g)	–	8.75	8.75
Sum	100.00	100.00	100.00

2.1.3. Coatings manufacturing

All EP-S coatings were manufactured under the same conditions. During the manufacturing process, the component A raw materials were weighed following the order described in the formulation (Table 2). After the initial weighing of talcum, the material was dispersed by an agitator mill for 45 min at 1500 rpm and at 45 °C. After the dispersing step, the viscosity was adjusted by adding *n*-butyl acetate. The component B of the formulations was also manufactured by carefully following the order detailed in the above-mentioned table, separately from component A. Both components (A and B) were manually mixed in one vessel just before coating application.

2.1.4. Coatings application

EP and EP-S coatings application were carried out by the air-less spray and air spray methods, respectively, in order to obtain a uniform coating layer on aluminium substrates. The former method consists of atomizing a coating without using compressed air. The coating is

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