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Evaluation of the corrosion protection performance of mild steel coated with hybrid sol-gel silane coating in 3.5 wt.% NaCl solution



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ARTICLE INFO	A B S T R A C T					
Keywords:	This study aims at studying the effect of silane hydrolysis time and concentration on the corrosion protection					
Corrosion protection	performance of an eco-friendly hybrid silane coating based on tetraethylorthosilicate (TEOS) and trimethox-					
Silane coating FT-IR Salt spray EIS	ymethylsilane (TMOMS) on mild steel substrate. By electrochemical impedance spectroscopy (EIS) and salt spray test the corrosion evaluations were done. Results of Fourier transform infrared (FT-IR), thermogravimetric analysis (TGA) and water contact angle tests measurements revealed the effective impact of the hydrolysis time and silane concentration on the curing process of the coating. Results revealed that the mixture of 50% silanes (TEOS/ TMOMS: 50/50 w/w) hydrolyzed for 24 h resulted in the highest corrosion resistance. The enhancement in protective performance of the hybrid coating was connected to the better condensation reaction, denser Si O Si naturely formation and leas hydrophilicity of the coating.					

1. Introduction

Metal and related alloys such as mild steel, aluminum, copper and magnesium are highly susceptible to corrosion in aggressive environments. For this sake, the extension of their service life has been the subject of many investigations in recent decades and it is still snowballing [1–6]. A variety of strategies have been used to protect metals from corrosion among them cathodic protection [7,8], and application of organic coatings [9–11], corrosion inhibitors [12–16] and conversion coatings [17,18] are the most useful methods. Conversion coatings such as chromate have been considered as a conventional surface pre-treatment method to promote the organic coatings adhesion to metal surface. However, due to the environmental aggressiveness and toxicity of chromates, the application of this coating is strongly prohibited in recent years [19–23]. Therefore, various green alternatives have been proposed in order to develop environmental friendly coatings [24–29].

In the past decades, the protection behavior of a variety of silane sol-gel coatings such as TMOS (tetramethoxysilane) [30], GPTMS (3-gly-cidoxypropyl-trimethoxysilane) [26,30], VTMOEO (tris(2-methoxyethoxy)vinylsilane) [31], APS (3-aminopropyltriethoxysilane) [32], MPTES ((3-mercaptopropyl)triethoxysilane) [33] and TEOS (tetra-ethylorthosilicate) [18,34] has been largely studied as attractive alternatives to the chromate. These coatings are a rapidly expanding technology resulting from its main advantages: good compatibility and high adhesion to the wide range of inorganic and organic interfaces, minimal

environmental impact and competitive price [35,36]. Another important feature of these coatings is the barrier performance against electrolyte diffusion to the substrate, which is based on the establishment of a dense layer with a three-dimensional network of siloxane [37–39]. Nevertheless, the silane sol-gel coatings have some disadvantages, i.e. thickness limit and crack ability [35,40]. Therefore, they suffer from detriment worsening barrier properties in the presence of micro-cracks or small defects in the coating. Inclusion of corrosion inhibitors such as rare earth based components [17,41], nanoparticles like graphene [24] and clay [39,42] into the silane coating has become a promising approach to overcome the above-mentioned drawbacks. In other words, the liking of the protective film to build cracks and pores can be diminished using this strategy.

Up to now, the corrosion resistance behavior of silane coatings deposited onto steel substrate has been reported in several papers [43–47]. Also, a few review papers have been periodically published, giving an up-to-date description of state of the art contributions [48–50]. The interest for sol-gel coatings applied on steel is focused on developing the coating with higher critical thickness and better protective performance using the mixture of silanes [19,32,50–54]. It is also necessary to guarantee the stability of the solutions to obtain homogeneous sole and get coatings with superior properties on the mild steel surface. It was demonstrated by Ramezanzadeh et al. that the silane coating made of a mixture of TEOS and APS provides better corrosion protection performance than the coating based on either one of

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Composition of the mild steel sample.

Elements	Fe	C	Si	Mn	Р	S	Cr	Мо	Со	Cu
wt%	balance	0.19	0.415	1.39	> 0.005	> 0.005	0.026	0.018	0.0559	0.0429

Table 2

Brief description of various coated samples (hydrolysis time: 24 h, curing temperature: 90 °C).

Sample	Percentage of silane (TEOS/ TMOMS: 50/50 w/w)	Percentage of water (70 %) and alcohol (30 %)
А	30	70
В	50	50
С	70	30

the silanes. Also, they showed that the mixture of 70/30 of TEOS/APS resulted in a more consecutive and compact film formation on the steel surface [32]. TEOS, due to its relatively slow, controllable rate of reaction and low cost (compared to other reagents), has been extensively used in such systems.

Several factors such as curing time, aging, concentration, pH and chemical structure of the silanes are the main parameters that influence the properties of the final coating [51,52,55].

The scope of the present work is designing an effective and ecofriendly corrosion protective silane coating on mild steel substrate through studying the effects of hydrolysis time and silane concentration in the silane coating corrosion resistance enhancement. A favored corrosion protection is the result of an accurate design. For this purpose, the hybrid silane coating was prepared by mixing the TEOS and TMOMS (Trimethoxymethylsilane) mixture. It is worth to notice that since no organic solvent is needed for the process under investigation, this technology is "eco-friendly". The influence of the hydrolysis time and silane concentration on the curing process and the chemistry of the prepared eco-friendly silane sol–gel coatings were investigated by FT-IR and contact angle measurements. Moreover, the corrosion studies were done by EIS and salt spray test.



Fig. 2. Isothermal TGA results at 90 $^\circ C$ for the silane solutions hydrolyzed for 24 and 48 h.

2. Experimental

2.1. Raw materials and sample preparation

Ethanol (98%), acetic acid and synthetic grades of organosilanes including TEOS and TMOMS were obtained from Merck Co. (Germany). Mild steel panels (with dimension of $10 \text{ cm} \times 7.5 \text{ cm} \times 0.2 \text{ cm}$) were provided from Foolad Co. (Iran). The chemical composition (wt.%) of the mild steel are tabulated in Table 1. Before the experiment, all the samples were abraded with 150, 600, 800 and 2000 grads SiC papers and then degreased by acetone (Merck) and rinsed with deionized water.



Fig. 1. FT-IR spectra of the silane coatings hydrolyzed for 24 and 48 h.

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