

# MICRO/NANOSTRUCTURED ZNO-BASED SUPERHYDROPHOBIC STEEL SURFACE WITH ENHANCED CORROSION PROTECTION

Nguyen Thi Phuong Nhung<sup>1</sup>, Nguyen Thi Ngoc Tien<sup>1</sup>, Nguyen Hoang Luong<sup>1</sup>  
Tran Thu Hang<sup>2</sup>, Nguyen Van Kiet<sup>1</sup>, Nguyen Phan Anh<sup>1</sup>

<sup>1</sup>Petrovietnam University

<sup>2</sup>Petrovietnam Manpower Training College

Email: nhungntp@pvu.edu.vn

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## Summary

This paper presents a simple process of fabricating a superhydrophobic carbon steel surface with high water repellence and good corrosion resistance. The steel surface was covered by thin ZnO layers via various methods of sol-gel method, hydrothermal synthesis, and electrochemical deposition, then further functionalised with methyltrichlorosilane. Morphological features observed by a scanning electron microscope (SEM) show that ZnO layers are composed of micro/nanostructures. Furthermore, the chemical composition of these films has been analysed by an energy dispersive X-ray spectroscopy (EDX). An optimum experimental condition, based on the type of Zn deposition method, has been obtained to provide superhydrophobic properties with a water contact angle of  $152 \pm 2^\circ$ . In a 3.5 wt% NaCl solution, the corrosion resistance properties of the superhydrophobic ZnO-coated steel are found to be very good with an anti-corrosion efficiency of 93.12%.

**Key words:** Superhydrophobic, ZnO electrodeposition, micro and nanostructures, corrosion resistance.

## 1. Introduction

Carbon steel materials are widely used in the oil and marine industries due to its reasonable corrosion resistance, good strength, and high durability. However, carbon steel is generally susceptible to localised corrosions caused by Cl<sup>-</sup>, leading to massive economic losses every year. In fact, carbon steel corrosion is an oxidation process where the metal is the anode in an electrochemical cell and the electrolyte is the environment. In the past decades, various strategies have been developed to control and prevent the occurrence of carbon steel corrosion, where the basic concept is to remove one or more of the electrochemical cell components (anode, cathode, environment). The most popular method is to coat a steel surface with an anti-corrosive layer to provide a barrier between the steel surface and the corrosive environment. However, in the case of hydrophilic coating, it leads to steel corrosion because of the contact between the corrosive solution and the steel/coating interface. In contrast, in the case of

hydrophobic coating (unlike water), the repellent ability against the water of hydrophobic surface can reduce the contact area of corrosive liquid and the surface and hinder the migration of corrosive ions to the steel surface, leading to a decrease in the corrosion rate of material. In recent years, superhydrophobic steel surfaces (having a water contact angle higher than  $150^\circ$ ) have attracted numerous research groups because of their features as a special anti-corrosion coating [1 - 3].

According to the micro/nano composite structures of natural surfaces such as lotus leaves, butterfly wings, and fish scales [4], researchers found that surface chemical composition and surface roughness were the key factors contributing to the formation of superhydrophobic surfaces. There are various methods to build superhydrophobic surfaces, including electrodeposition, anodic oxidation, chemical etching, and sol-gel methods. Most of the methods are based on the combination of (i) roughening the surface of low surface energy material, and/or (ii) modifying a rough surface by depositing low surface energy material. Chen et al. made the superhydrophobic steel surface by using polyurethane/nano-Al<sub>2</sub>O<sub>3</sub> coating. The superhydrophobic aluminium-



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coated steel surface shows excellent anticorrosive performance with a contact angle of 151°. Xiang et al. fabricated a superhydrophobic surface on steel surface by combining two steps, including the formation of porous ZnO coating and modification of myristic acid. Chongwei et al. also built the superhydrophobic steel surface by a two-step process, involving the etching step with Piranha solution and fluorination treatment. The superhydrophobic steel surface exhibits high resistance to water, acid solution, and alkaline solution [5, 6].

In this work, the superhydrophobic surface of carbon steel was prepared in two steps: the carbon steel substrate was first covered by micro and nanostructured ZnO to increase the surface roughness, and then followed by a chemical modification with methyltrichlorosilane to reduce the energy surface thanks to  $-CH_3$  group on the steel surface. One of the reasons to choose ZnO to create the superhydrophobic carbon steel is that ZnO layers have not only been widely used to prevent the corrosion of the steel surface but also easily modified chemically. In this study, micro and nanostructured ZnO coating was introduced into the carbon steel surface by using three coating protocols such as nanoparticles coating by sol-gel method, electrodeposition, and hydrothermal growth. Micro and nanostructured ZnO was deposited on the steel surface by one of three methods: sol-gel method, hydrothermal synthesis, and electrochemical deposition. The micro and nanostructure, wettability, and chemical composition of superhydrophobic coating were evaluated by scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDX), and contact angle measurement. Moreover, the anti-corrosion property of coating was investigated via potentiodynamic polarisation curve acquired by electrochemical experiment in 3.5 wt% NaCl solution (using electrochemical equipment SP-300 system).

## 2. Materials and methods

### 2.1. Materials

Methyltrichlorosilane, ethanol, acetone,  $H_2SO_4$ ,  $H_2O_2$ ,  $NH_3$ , and  $Zn(CH_3COO)_2$  are obtained from Sigma-Aldrich. In this study, two kinds of steel substrates, API 5L X65 and CT3 are used. The X65 steel is obtained from VINA Tan Phat company and has the following composition: Fe (98.8%), C (0.12%), Mn (0.69%), Si (0.11%), Cr (0.02%), and Ni (0.01%). The CT3 steel is from China and consists of Fe (42.89%), C (0.14%), Mn (11.12%), Si (0.13%), Cr (0.02%), and Zn (0.51%).

### 2.2. Preparation of superhydrophobic steel surface

The fabrication process of the superhydrophobic carbon steel surface includes 2 steps: first, a thin layer of micro and nanostructured ZnO is deposited on the steel substrate, then this is followed by chemical treatment with methyltrichlorosilane.

#### 2.2.1. Formation of micro and nanostructured ZnO coating

The steel surfaces are cut into 0.1 cm x 1 cm x 0.5 cm for the X65 substrate, and 1.5 cm x 3.0 cm x 3.0 cm for the CT3. The substrates are then polished by sandpaper (100, 200, and 600 grit) and subsequently degreased in acetone and ethanol, and finally rinsed with distilled water.

There are 3 methods to prepare the micro and nanostructured ZnO coating on the steel substrate: (i) sol-gel method, (ii) hydrothermal synthesis, and (iii) electrochemical deposition.

- Sol-gel method: Solution of ZnO seed layer is prepared by using the sol-gel method. Zinc acetate dihydrate (ZAD) is dissolved in absolute ethanol. Using a dropper, the 0.048 M ZAD solution is dropped onto a cleaned steel surface and left for 15 seconds, followed by a rinse with absolute ethanol; this step is repeated 15 times. The substrate is then annealed on a hot plate at 350°C for 1 hour. More details of the growth conditions can be found in the article by Suvindraj Rajamannickam [11].

- Hydrothermal synthesis: Zinc acetate dihydrate is dissolved in 250 ml of distilled water; 0.3 ml of ammonium hydroxide is then added to this solution and stirred at room temperature for 30 minutes. The steel substrate with the seed that was prepared previously by the same sol-gel method is placed vertically in this aqueous solution for 1 hour at 90°C. More details of the growth conditions can be found in the article by Wided Chebil et al. [2].

- Electrochemical deposition: The steel substrate is firstly dipped into 0.1 M HCl solution for 30 seconds before the zinc electrodeposition process is conducted for different time durations: 5 mins, 15 mins, 30 mins, and 60 mins. More details concerning this process are described in our previous article [10].

A two-electrode cell is used, in which the cathode is the steel substrate, the anode is a Zn metal sheet (0.2 x 2 x 5 cm) and the electrolyte is deionised water. A constant voltage of 1 V is applied between the two electrodes to grow the Zn layer in different durations. After electrolysis, the substrate is cleaned with deionised water and then

dried. Finally, the substrate is annealed in furnace at 250°C for 120 minutes to form a ZnO thin film coating on the steel surface.

### 2.2.2. Surface functionalisation by silanisation

The ZnO-coated steel substrates are UV/O<sub>3</sub> treated for 30 minutes to remove any organic contaminants and to generate surface hydroxyl -OH groups. The activated surface is then directly dipped into a solution containing 10 vol% methyltrichlorosilane in ethanol for 12 hours. The substrate is rinsed 3 times with ethanol and further dried under a gentle nitrogen flow, and finally kept in a clean oven at 120°C for 10 minutes.

### 2.3. Sample characterisations

Morphology and composition of the thin film is characterised by JEOL JSM-7600F SEM, X-ray microanalyser by Oxford Instruments EDS. The wetting properties of all substrates are determined by measuring static water contact angle with OCA - DataPhysics Instruments at 3 positions on each surface using 5 µl distilled water.

### 2.4. Corrosion resistance test

The corrosion tests are carried out at room temperature in a 3.5 wt% NaCl solution using the SP-300 system. The exposed coating area is 0.785 cm<sup>2</sup>. Ag/AgCl and pure graphite electrodes are used as the reference and counter electrodes, respectively. Prior to the potentiodynamic polarisation test, the samples are stored in the solution for 1 hour to establish the open circuit potential (OCP). The potential of the electrodes is swept at a rate of 0.166 mV/s, ranging from an initial potential of -250 mV vs.  $E_{\text{corr}}$  to a final potential of -200 mV Ag/AgCl. Electrochemical impedance spectroscopy (EIS) is conducted using an SP-300 system with a commercial software programme. The amplitude of the sinusoidal peak - peak perturbation is 10 mV. The frequency varies from 100 kHz to 10 mHz.

## 3. Results and discussion

### 3.1. Surface morphology and wettability

Figure 1 reveals the SEM results of the surface morphology of carbon steel coated with and without different micro and nanostructured ZnO layers: (i) steel surface without coating ZnO layer (CTs-1); (ii) steel surface coated with ZnO particles by sol-gel method (CTs-2); (iii) steel surface with ZnO layer deposition by hydrothermal method (CTs-3); and (iv) steel surface with ZnO layer by electrochemical deposition.

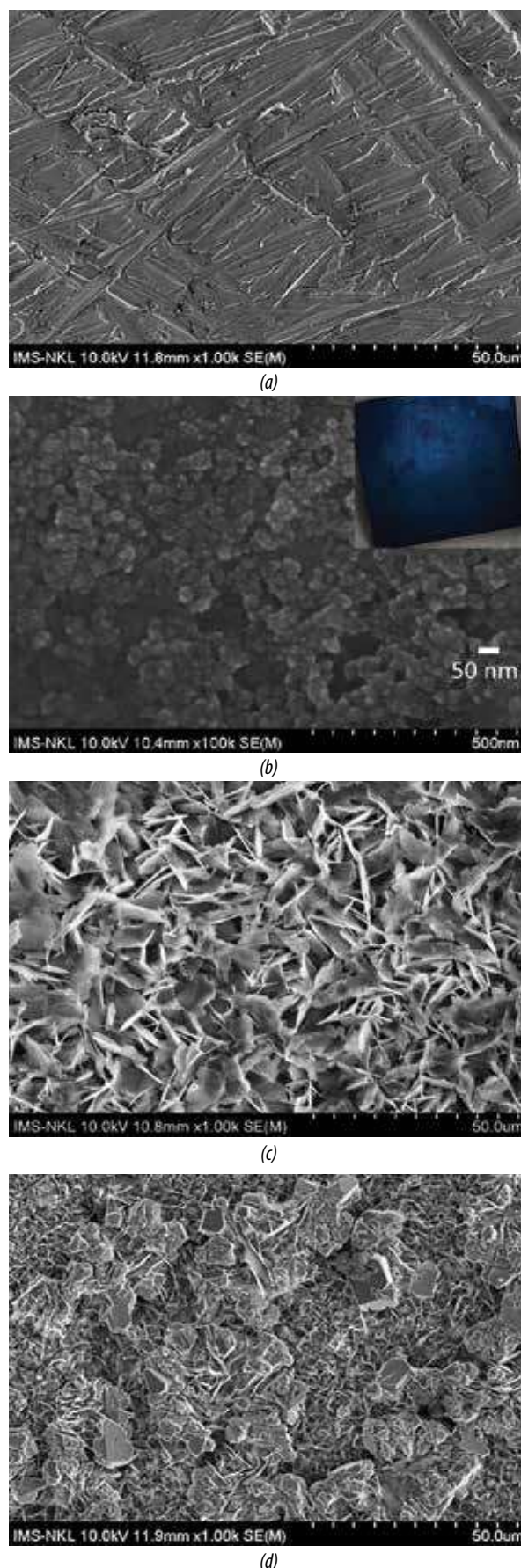


Figure 1. SEM morphology of (a) CTs-1, (b) CTs-2, (c) CTs-3, (d) CTs-4.



After chemical modification with methyltrichlorosilane, their hydrophobic ability is evaluated via water contact angle measurements. The results are shown in Figure 2.

As shown in Figure 1a, the ZnO-uncoated surface of CTs-1 is rough with some minor scratches, holes, and other defects. After chemical modification with silane, the contact angle increases from  $63 \pm 2^\circ$  to  $93 \pm 2^\circ$  (Figure 2).

According to the treatment with ZnO particles by sol-gel method, there are several nanoparticles of around 10 - 20 nm located on the CTs-2 substrate (Figure 1b). The corner of Figure 1b is a photograph of a steel surface with a ZnO seed layer. The bluish tinge appears after the seed layer is annealed. However, these particles are not distributed homogeneously, and the water contact angle is smaller,  $73 \pm 2^\circ$  (Figure 2). It can be explained that the scratches, holes, and other defects on the original surface are filled with ZnO particles, making the surface less rough and more hydrophilic after chemical modification with silane.

When the CTs-2 substrate is immersed into the  $\text{NH}_3$  solution, ZnO particles are transformed into a plate-like structure on CTs-3 (Figure 1c). A staggered coating distribution is also observed on the sample surface; the thickness and size of the coating are uniform and there is a gap between the lamellas. The structure of this composite interface greatly improves the surface roughness. Its hydrophobic property changes dramatically with a water contact angle of  $142 \pm 2^\circ$ .

In terms of electrochemical deposition, CTs-4 is covered with a new structure of two layers (Figure 1d).

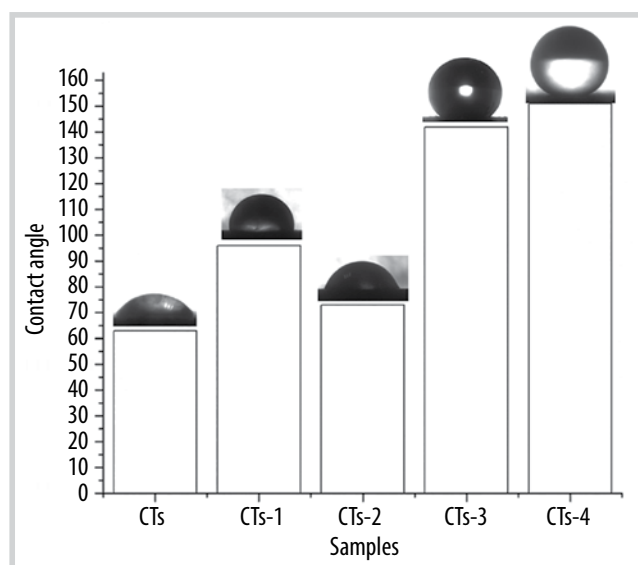


Figure 2. Contact angle of different samples: CTs, CTs-1, CTs-2, CTs-3, CTs-4.

The top layer presents a large amount of accumulated microparticles on the surface while the bottom layer possesses a plate-like structure. In this case, air can fill in these pores, preventing water droplets from entering and increasing the contact angle of the substrate. The surface becomes superhydrophobic with a contact angle of  $151 \pm 2^\circ$ .

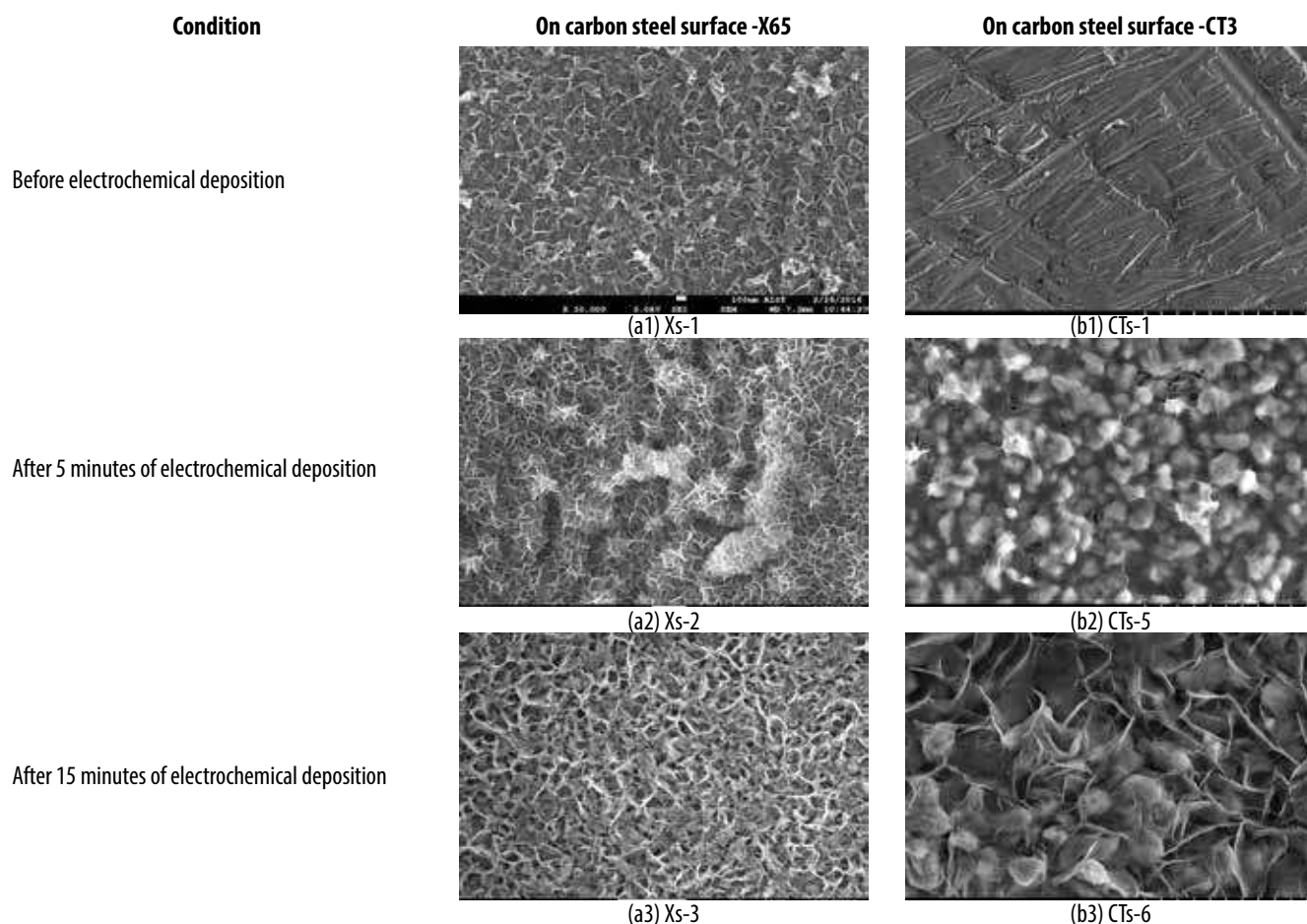
In conclusion, micro and nanostructured ZnO can be formed on the steel surface by 1 of the 3 methods: deep coating to form the particles, hydrothermal growth, and electrochemical deposition. Each coating method results in different steel surface morphology. Compared to the other two, the electrochemical deposition offers a feasible method to control the coating thickness and the surface morphology. Therefore, the electrochemical deposition will be used in this investigation on superhydrophobic surface.

### 3.2. Effect of substrate type on formation of nanostructure

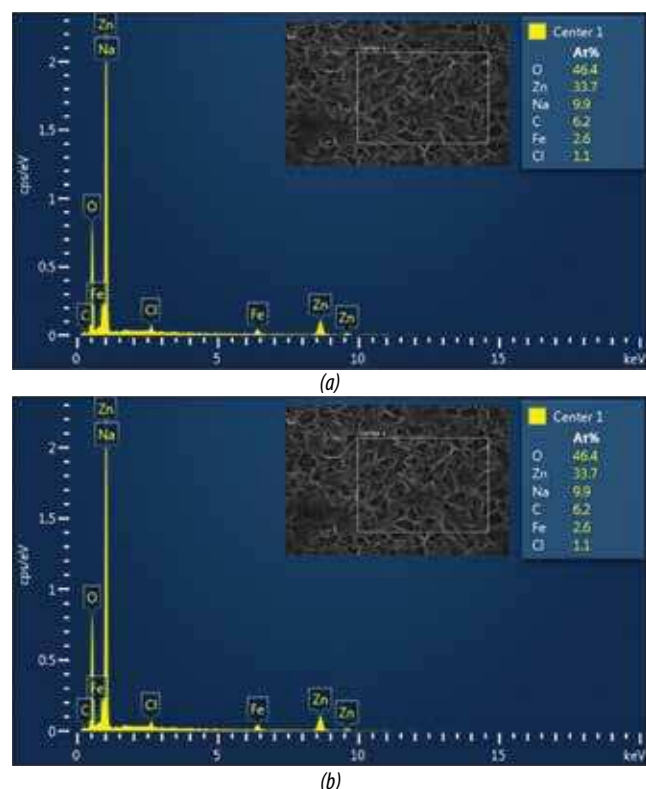
To evaluate the influence of substrate types on ZnO micro and nanostructure in the electrodeposition method, 2 carbon steel surfaces of CT3 and X65 substrates are considered. Figure 3 indicates the SEM results of carbon steel surface morphology before (CTs-1 and Xs-1 substrates) and after electrodeposition for 5 minutes (CTs-5 and Xs-2 substrates), and for 15 minutes (CTs-6 and Xs-3 substrates).

From Figure 3, after polishing by sandpaper (100, 200, and 600 grit), the morphology surfaces of two types of steel substrate, X65 and CT3, are not similar. Some minor scratches appear on the CT3 steel substrate (CTs-1) while small holes show on the X65 steel substrate (Xs-1). Obviously, the Xs-1 surface is rougher than the CTs-1; i.e. a higher amount of air can be trapped into the structure of the Xs-1 surface compared to the CTs-1 surface. It results in a higher hydrophobicity of the Xs-1 surface (CA =  $126 \pm 2^\circ$ ) than that of the CTs-1 (CA =  $93 \pm 2^\circ$ ) after the chemical modification with silane. Increasing time of electrochemical deposition leads to a larger thickness and a higher roughness of micro and nano ZnO cover:

+ After 5 minutes of electrochemical deposition, pores appear on the X65 substrate with higher density and size of around  $0.25 \mu\text{m}$  (Xs-2 substrate's SEM image). Meanwhile, a high number of ZnO sphere particles grows on the CT3 surface (CTs-5 substrate's SEM image) with diameters of about  $0.5 \mu\text{m}$ . The Xs-2 and CTs-5 substrates,



**Figure 3.** SEM morphology of (a1) Xs-1; (a2) Xs-2; (a3) Xs-3; (b1) CTs-1; (b2) CTs-5; (b3) CTs-6.



**Figure 4.** EDS analysis of the porous ZnO-coated steel surface (a) before silanisation and (b) ZnO coated steel surface with silanisation.

**Table 1.** Contact angle on different substrate: Xs-0; Xs-1; Xs-2; Xs-3; CTs-0; CTs-1; CTs-5; CTs-6

X65 steel substrate	CT3 steel substrate
54° ± 2° (Xs-0)	63 ± 2° (CTs-0)
126 ± 2° (Xs-1)	93 ± 2° (CTs-1)
130 ± 2° (Xs-3)	110 ± 2° (CTs-5)
152 ± 2° (Xs-2)	142 ± 2° (CTs-6)

with dramatically increasing hydrophobic properties, have a contact angle of 132 ± 2° and 112 ± 2°, respectively.

+ After 15 minutes of electrochemical deposition, the ZnO micro and nanostructure on the X65 substrate appears with a larger diameter and consists of double structure, including a small open circle structure and a small porous structure inside (Xs-3 SEM image). Meanwhile, in the case of the CT3 substrate, the appeared structure looks like a flower with a sphere particle (CTs-6 SEM image). In both substrates, the rough surface is upgraded, resulting in a significant rise in hydrophobicity on these substrates. Therefore, the CTs-6 surface is highly hydrophobic with a contact angle of 142 ± 2° and the Xs-3 surface becomes superhydrophobic with a contact angle of 152 ± 2°.

### 3.3. Chemical surface

The composition of ZnO thinly coated on steel substrate is analysed by EDS as shown in Figure 4a. EDS spectra show the presence of elements such as zinc (8.6 keV and 1.0 keV), oxygen (0.53 keV). From the data, it is apparent that thin ZnO layers have been successfully introduced through a simple deposition method, in agreement with the published data [12].

After silanisation, the composition of the ZnO-deposited steel interface is presented in Figure 4b. Compared to Figure 4a, it is important to note the appearance of Si atoms (1.7 keV) and the increase of C composition from 6.2% to 19.5% on the interface. These results are suitable for the silanisation process to form the CH<sub>3</sub>-Si group on the ZnO-coated steel surface. In addition, the surface's wettability is changed after chemical modification. These results confirm that the silanisation of the ZnO-coated steel surface is successful.

### 3.4. Anticorrosion properties

Figure 5 presents the potentiodynamic polarisation curves of steel surface and ZnO-functionalised steel substrate. The substrate with coating shows a lower corrosion current density, resulting in improved corrosion resistance. In addition, it also shows a protected range in the anodic polarisation and there was no pitting observed up to 200 mV Ag/AgCl. The

rapid increase in current density in the anode can be attributed to the anodic current density associated with oxygen evolution.

In this figure, the cathode branch is divided into two regions at slightly variable current densities. Zone I corresponds to the area near the corrosive voltage. The shape of this region depends on the depth of the solution layer. For the depth of 1.0 - 10 cm of the solution, near E<sub>corr</sub>, it is possible to observe the presence of an interference, which may relate to the reduction of corrosive products to be formed in a stable time. Zone II - in addition to 50 mV of corrosive voltage, the shape and relationship of this region are due to the reduction of oxygen and controlled by mass transfer. In this region, the process of reducing oxygen and corrosive products takes place and is controlled by the rate of oxygen diffusion. The length of this region increases gradually as the depth of the solution layer increases, which may indicate that the amount of oxygen removed is proportional to the volume of the solution.

Table 2 indicates the corrosion properties of steel substrate and the ZnO-functionalised steel substrate obtained from the polarisation test. The protective efficiency (P<sub>i</sub>) of the coatings is determined by Equation (1):

$$P_i = 100 \times \left( 1 - \frac{i_{corr}}{i_{corr}^0} \right) \tag{1}$$

where i<sub>corr</sub> and i<sub>corr</sub><sup>0</sup> are the corrosion current densities in the presence and absence of a coating layer, respectively. The electrochemical characteristics of the surface with ZnO-functionalised coating show a lower protective current density in comparison to the results of the steel substrate without coating. Table 2 indicates that the protective efficiency of surface with coating is quite high, 93.12%, indicating good corrosion protection.

Figure 6a indicates the Nyquist plots of steel surface without coating (black line) and ZnO-functionalised steel substrate (red line) in 3.5 wt%

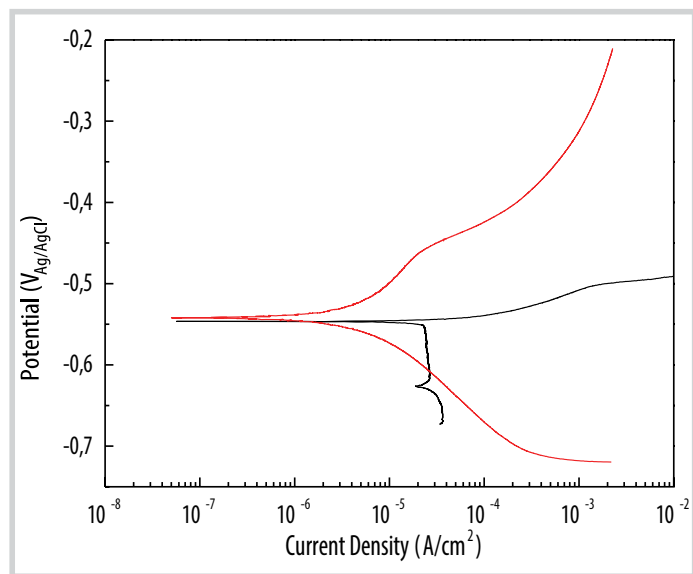


Figure 5. Potentiodynamic polarisation curves of steel surface without coating (black line) and ZnO functionalised steel substrate (red line).

Table 2. Corrosion properties of steel surface without coating and ZnO functionalised steel substrate

Specimen	E <sub>corr</sub> (mV <sub>Ag/AgCl</sub> )	i <sub>corr</sub> (×10 <sup>-6</sup> A/cm <sup>2</sup> )	β <sub>a</sub> (mV/decade)	-β <sub>c</sub> (mV/decade)	η (%)
Steel substrate	-547	24.7	10.8	253.2	-
Coated steel	-542	1.7	86.7	68.4	93.12

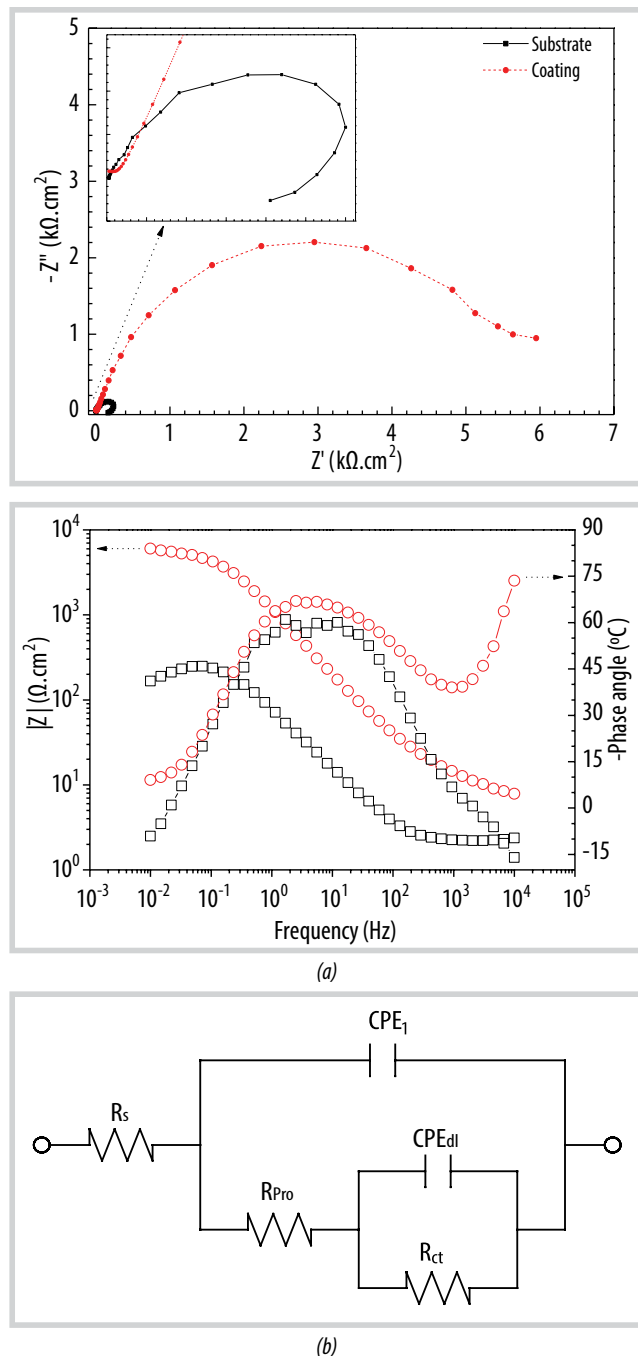
NaCl solution after 1 hour immersion. The impedance diagrams of the coated substrate present a much larger impedance than the bare steel, indicating an effective barrier behaviour on the corrosion process. It is related to

the treatment process controlled by ZnO functionalisation with the  $-CH_3$  group on the steel surface. In this study, the semicircle depression in the Nyquist diagram of the steel substrate without coating can be attributed to the heterogeneity of the steel surface, which is caused by the difference in droplet distribution and surface roughness. This effect is omitted due to a protective film formation, which acts as a physical barrier layer formation on the steel surface. The data typically shows a two-phase constant. Therefore, the corresponding equivalent circuit for fitting EIS data is proposed as shown in Figure 6b, where  $R_s$  represents the solution resistance between the reference and working electrodes; CPE1 is the coating constant phase element;  $R_{coat}$  is the coating resistance;  $R_{ct}$  is the charge transfer resistance; and CPE2 is the double layer constant phase element ( $R_{coat}$  is replaced by  $R_{rust}$  for the steel substrate). The ZSimpWin software is used to fit the EIS data to determine the optimised values for the protective film and charge transfer resistance, as well as protective layer and double layer capacitances, which are given in Table 3. The results show that the coating exhibits a high charge transfer resistance, indicating a low corrosion rate, as shown in Tables 2 and 3. Overall, a coating provides a good corrosion protection by improving the corrosion resistance of a steel substrate in 3.5 wt% NaCl solution.

**4. Conclusion**

We have successfully achieved the superhydrophobic steel surface with micro/nanostructured ZnO coating by different methods. The obtained superhydrophobic surface presents a water contact angle of  $152 \pm 2^\circ$  with low hysteresis. Furthermore, the steel surface with ZnO-functionalisation also provides a good anti-corrosion property with a protective efficiency of 93.12%. Hence, through a simple process of ZnO deposition and silanisation, the steel substrate shows both superhydrophobic properties with low hysteresis and a good corrosion resistance.

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**Figure 6.** Nyquist and Bode plots of steel surface without coating (black line) and ZnO functionalised steel substrate (red line) (a); Proposed equivalent circuit for fitting EIS data (b).

**Table 3.** The fitted results of EIS data

Specimen	$R_s$ ( $\Omega \cdot \text{cm}^2$ )	$CPE_{pro}$		$R_{pro}$ ( $\Omega \cdot \text{cm}^2$ )	$CPE_{dl}$		$R_{ct}$ ( $\Omega \cdot \text{cm}^2$ )
		$C$ ( $\mu\text{F}/\text{cm}^2$ )	$n$ (0~1)		$C$ ( $\mu\text{F}/\text{cm}^2$ )	$n$ (0~1)	
Steel substrate	2.25	1536	0.8727	61.7	1266	0.8001	190
Coated-steel	3.45	159	0.8901	1039.4	41	0.9544	6964



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