

Construction of a novel superhydrophobic coating surface for downhole tubulars and its protection for CO₂ corrosion during CCUS application

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ABSTRACT

Injected acidic gas of CO₂ and produced acidic fluid under high temperature and high pressure are the main factors that cause corrosion of downhole tubular column in Carbon capture, utilization and storage (CCUS) application. The traditional methods, such as corrosion inhibitor mixing and regular injection are not effective method to prevent corrosion of tubular column. In this paper, as a main component, novel organic polysiliconazane polymer (OPSZ) was added to the coating formulation to form a ceramic-like layer with Si-N-Si and Si-O-Si structures on the steel surface. After coating, the non-polar surface energy of the steel decreased to 14.7 mJ/m². The hardness, thickness, friction strength, corrosion resistance and other related properties of the coating layer were evaluated in laboratory, showing its good tolerance to high temperature in corrosion resistance in the long term, which provided effective protection against corrosion and aging in the application. The novel coating material can be coated on the surface of the downhole tubular column and forming a super hydrophobic surface, which can effectively prevent the contact of acid corrosion medium and metal surface by greatly reducing surface energy, and greatly improving protection efficiency of steel due to carbon dioxide corrosion, indicating good potential in CCUS application.

Keywords: CCUS, carbon dioxide corrosion, super hydrophobic coating, low surface energy

1. INTRODUCTION

Due to its significant contribution to environmental protection, the Carbon capture, utilization and storage (CCUS) related project quickly became a research hotspot in the world. China has successfully established a CCUS project of million-ton CO₂ sequestration and oil

displacement in the Shengli Oilfield in Shandong Province. However, as CO₂ is acidic gas that could corrode downhole strings such as tubing and casing during the injection process, it will lead to a significant decrease of the life of the downhole string at the injection end. Nowadays, many CCUS injection projects in China mainly carry out anticorrosion through the injection of corrosion inhibitors. While, there are problems such as high requirements for corrosion inhibitors, uneven downhole distribution of injection dosing and high cost in the long-term addition process, which make it difficult to meet the requirements of on-site safety production. Long-term operation may lead to large downhole string corrosion at the injection end and even lead to the risk of casing rupture.

Through the specific modification of the metal surface, the wettability of the metal surface can be changed. It can effectively prevent the intrusion of CO₂ into the metal pipe, improving the corrosion resistance and other properties of the pipe, and eventually prolong the service life of downhole tools, such as high screen pipes^[1,2], thereby reducing oil production costs. While, metal surface modification technology is rarely used in this oilfield applications, and it is expected to become an important research direction to solve the above problems in the future.

Nowadays, there are three main types of hydrophobic and oleophobic coatings, organofluorine compound coatings^[3,4], organosilicon compound coatings^[5] and fluorosilicon compound coatings^[6]. Most of these coatings are topcoats, which need primers and intermediate paints to ensure good mechanical strength, wear resistance and corrosion resistance. The addition of primers and intermediate paints will lead to excessive thickness of the coating,

which is not suitable for oil pipes. Therefore, it is urgent to find a coating system with thin thickness, hydrophobicity and oilphobic property, wear resistance, corrosion resistance and high temperature resistance.

Polysilazane is a kind of polymer with Si-N as the main chain skeleton. It is named perhydropolysilazane when the side chain groups are all hydrogen atoms. While it is named organopolysilazane when the side chains are other organic groups [7,8]. The properties of polysilazane can be adjusted by changing the substituent groups on the side chain. Since the polysilazane can be directly as coating the surface without need of primer and has excellent thermal stability, it can be used as a reinforcing filler for organic polymers to enhance the heat resistance of polymerized products. In recent years, research on polysilazane coatings has become a hot spot, and has been applied in metal protection, semiconductor devices, space materials, fireproof materials and other fields [9,10]. While it has not been used in the oil exploit area. Herein, the N80 steel sheet of polymer injection screen was used as the substrate material, and the surface was modified by organopolysilazane coating. The hardness, adhesion, contact angle and corrosion resistance of the coating are tested, and the theoretical calculation of surface energy was carried out to analyze the change of surface properties and corrosion resistance of organopolysilazane used in polymer injection screen surface coating modification. The surface antifouling and corrosion resistance mechanism was explored. We believe the study of a polymer injection screen the feasibility of the surface coating of the pipe substrate provides important experimental data and technical reference for solving the problem of easy clogging of the polymer injection screen in the process of CCUS injection projects in oilfield.

2. EXPERIMENTAL SECTION

2.1 Materials

N80 steel sheet was brought from Shengxin Technology Co., Ltd., Yangxin County, Shandong Province. Organopolysilazane (Durazane 1500 rapid cure) of industrial grade was supplied from Merck. Butyl acetate, sodium chloride, calcium chloride, magnesium chloride, sodium sulfate are of A.R. grade and obtained from Sinopharm Chemical Reagent Co. Ltd.. Vacuum pump oil (LVO120) was provided by Leybold, Germany. Deionized water was used for the preparation of medicines and comparative tests in this experiment.

Preparation of 20 g/L mineralized water (CO₂ saturation): Dissolve 1.73 g of CaCl₂, 1.17 g of MgCl₂, 5.27 g of Na₂SO₄, and 11.83 g of NaCl in 1 L of deionized water, and saturate with CO₂ under closed conditions for 24 hours (2 MPa).

2.2 Preparation of coating

N80 steel sheet was used as the substrate material of the polymer injection screen. Firstly, wipe the steel sheet with absolute ethanol absorbent cotton before use to remove the anti-rust oil on the surface, and then dry it with cold air for later use. Secondly, mix and dilute polysilazane resin and butyl acetate at 40 : 60 (vol./vol.), and applied by brushing (use a soft brush to dip the paint and apply it on the steel sheet, 3 coats each time) or dip coating (apply the steel sheet is immersed in the paint, and after 1 min, the steel sheet is slowly taken out to allow the excess paint to flow back) to attach the coating, and then the steel sheet was placed in a dust-free environment and cured at room temperature.

2.3 Coating performance testing

Coating thickness test: It was done according to GB/T13452.2-2008. Measure 3 points for each steel sheet during the test, and take the average value.

Hardness test: It was done by the pencil hardness of the coating according to GB/T6739-2006.

Adhesion test: It was done according to GB/T6739-2006 T9286-2021 to test coating adhesion.

Contact angle test: Use an optical contact angle meter (Germany KRUSS, DSA100) to test 3 different points on each steel sheet and take the average value.

Soaking test [11]: Immerse the sample (the uncoated part is wrapped with adhesive tape) in deionized water, 20 g/L of mineralized water or placed in a 120 °C oven for 14 days, and take pictures to record the corrosion process of the steel sheet. After 14 days, the corrosion products were removed from the steel sheet, and the contact angle after immersion was tested. The composition of the corrosion products was analyzed by X-ray diffractometer (PXRD) (PANalytical, Netherlands, X'Pert PRO).

Electrochemical test: A three-electrode system is used for testing. The working electrode is a steel sample, and the reference electrode is a saturated calomel electrode, and the counter electrode is a platinum electrode, and the corrosion medium is 3.5% of NaCl and 20 g/L of mineralized water. The test scan range of the potentiodynamic polarization curve is -1.5~1V. Electrochemical impedance spectroscopy

measurement frequency parameter is $10^5 - 10^2$ Hz, and the external sine wave is 20 mV.

2.4 Calculation of coating surface energy

The surface energy of the coating was calculated by the three-liquid method of water-ethylene glycol-diiodomethane system [12]. By testing the contact angles of the three liquids on the coated steel sheet, the surface energy is calculated using the Lifshitz-van der Waals acid base three-parameter method. The parameters required for the calculation are shown in Table 1.

Tab. 1. Surface energy parameters of water, diiodide and ethylene glycol

Project	Water	Diiodomethane	Ethylene glycol
$\gamma_L / (\text{mJ}\cdot\text{m}^{-2})$	72.8	50.8	48.0
$\gamma_L^{LW} / (\text{mJ}\cdot\text{m}^{-2})$	21.8	50.8	29.0
$\gamma_L^+ / (\text{mJ}\cdot\text{m}^{-2})$	25.5	0	1.92
$\gamma_L^- / (\text{mJ}\cdot\text{m}^{-2})$	25.5	0	47.0

Firstly, the nonpolar surface energy component γ_s^{LW} is calculated from the contact angle of the nonpolar liquid diiodomethane [Formula (1)], and then the contact angles of the other two liquids and their surface energy parameters are substituted into the formula (2), solve the equation to obtain the Lewis acid component γ_s^+ and the Lewis base component γ_s^- , and then calculate the solid surface energy by formula (3) and formula (4).

$$\gamma_s^{LW} = \frac{\gamma_L^{LW} (1 + \cos \theta)^2}{4} \quad (1)$$

$$(\gamma_L^{LW} + 2\sqrt{\gamma_L^+ \gamma_L^-})(1 + \cos \theta) = 2(\sqrt{\gamma_s^{LW} \gamma_L^{LW}} + \sqrt{\gamma_s^+ \gamma_L^-} + \sqrt{\gamma_s^- \gamma_L^+}) \quad (2)$$

$$\gamma_s^{AB} + 2\sqrt{\gamma_s^+ \gamma_s^-} \quad (3)$$

$$\gamma_s = \gamma_s^{LW} + \gamma_s^{AB} \quad (4)$$

3. RESULTS AND DISCUSSION

3.1 Basic properties of the coating

According to the coating working environment, place, workpiece size, shape and materials of workpieces, the commonly used coating methods include dip coating, brush coating, roller coating, air spray coating and high-pressure airless spray coating. Among them, brushing and dipping are the most

commonly used and simplest coating methods for industrial anti-corrosion. Therefore, brushing and dipping are used in this paper to coat polysilazane coatings on steel sheets with different numbers. The basic performance test results are shown in Table 2.

Tab. 2. The thickness, hardness and adhesion of the coating applied by brush and dip coating

Project		Brush		Dip coating	
		steel sheet 1	steel sheet 2	steel sheet 1	steel sheet 2
Coating thickness / μm	1	7.1	8.1	6.6	5.9
	2	5.6	6.0	34.2	5.8
	3	6.3	7.2	16.2	18.2
	average value	6.3	7.1	19	10.0
Hardness		5H	5H	5H	5H
Adhesion/level		0	0	0	0

It can be seen from Table 2 that the dip coating process would cause uneven coating thickness, and the coating thickness difference at different positions of the same steel sheet can reach 10~20 μm . However, the coating thickness obtained by brush coating process is relatively average, and the average thickness is less than 8 μm , showed good thickness and uniformity. The main reason for this phenomenon is that the polysilazane coating has a high viscosity. After dipping, the coating is slow to level on the surface of the steel sheet, which is prone to poor appearance of the paint film and the formation of flow marks, which leads to inconsistent coating thickness. The brushing process applies the paint to the surface of the steel sheet more evenly through the movement of the brush, so that the coating thickness of the brushing process is more uniform. However, the brushing and dipping processes have little effect on the hardness and adhesion of the coating. The coating can reach a hardness of 5 H and an adhesion of 0, indicating that the coating has a strong ability to resist scratches and abrasions, which is not easy to be peeled off.

3.2 Changes in hydrophobic and oleophobic properties of polysilazane-coated modified steel sheets

In order to understand the changes in the hydrophobicity and oleophobicity (vacuum pump oil (LVO120)) of the steel sheet before and after coating, the water and oil contact angle tests were carried out on the steel sheet, and the results are shown in Table 3.

It can be seen from Table 3 that the uncoated steel sheet has poor hydrophobic and oleophobic properties, with an average water contact angle of 76.66° and an average oil contact angle of only 18.2°, which belongs to the lipophilic surface. After the polysilazane coating is

Tab. 3. Water and oil contact angles of brush coated, dip coated and uncoated steel sheets

Project		No paint		Polysilazane coating			
				Brush		Dip coating	
		Water contact angle	Oil contact angle	Water contact angle	Oil contact angle	Water contact angle	Oil contact angle
Contact angle/(°)	1	77.75	14.4	109.6	49.3	102.3	46.1
	2	76.47	16.6	102.2	48.5	105.4	48.3
	3	75.77	23.7	111.1	45.6	104.2	42.5
	Average value	76.66	18.2	107.6	47.8	104.0	45.6

However, whether brushing or dipping is used, the average water contact angle and oil contact angle of the coating are greatly improved, indicating that the improvement of hydrophobicity and oleophobic on the surface of the coated steel sheet. This performance improvement can reduce the adsorption of water on the surface of the oil pipe, thereby reducing the possibility of corrosion, which might be used in CCUS.

3.3 Changes in surface energy of polysilazane-coated modified steel sheets

The contact angles of water, diiodomethane and ethylene glycol on the steel sheet were measured through experiments, and then the surface energy of the steel sheet after coating modification was calculated using the Lifshitz-van der Waalsacidbase three-parameter method. The results are shown in Table 4.

Tab. 4. Contact angles of water, diiodomethane and ethylene glycol on polysilazane coating

Project	Uncoated	Polysilazane coating
Water contact angle/ °	76.66	107.6
Diiodomethane contact angle/ °	47.2	87.1
Ethylene glycol contact angle/ °	78.1	86.8
$\gamma_S^{LW}/\text{mJ}\cdot\text{m}^{-2}$	35.8	14.0
$\gamma_S^+/\text{mJ}\cdot\text{m}^{-2}$	2.3	0.3
$\gamma_S^-/\text{mJ}\cdot\text{m}^{-2}$	23.8	1.1

applied, the water and oil contact angles are increased by nearly 30°. Meanwhile, the water and oil contact angles of the steel sheet coated by the dip coating process are slightly smaller than those of the brush coating process.

$\gamma_S/\text{mJ}\cdot\text{m}^{-2}$	50.6	15.1
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After the polysilazane coating modification, the non-polar surface energy component (γ_S^{LW}), the Lewis acid component (γ_S^+), and the Lewis base component (γ_S^-) of the steel sheet were all reduced, and the surface energy γ_S was reduced from 50.6 mJ/m² to 15.1 mJ/m², which belongs to low surface energy. This shows that the modification of polysilazane coating reduces the surface energy of the steel sheet and improves the anti-adhesion performance of the coating, which can reduce the adsorption of water on the surface of the tubing, thereby reducing the possibility of corrosion.

3.4 Corrosion resistance of polysilazane-coated modified steel sheets

Since the salinity of continental oilfield water is about 5-30 g/L, the steel sheet was soaked in 20 g/L of mineralized water, compared with deionized water soaking and 120 °C baking temperature conditions, to explore the process of coating surface changes (various working fluids are saturated with carbon dioxide) for 30 days. The results are shown in Figure 1.



Fig. 1. Surface changes of coated and uncoated steel sheets after 14 days of reaction. (a) —uncoated, deionized water, (b) —uncoated, mineralized Water, (c) —uncoated, baked, (d) —coated, deionized water, (e) —coated, mineralized water, (f) —coated, baked

It can be seen from Figure 1 that the uncoated steel sheet corrodes rapidly in the deionized water and 20 g/L of mineralized water. Part of the uncoated steel sheet corrodes after 2 days, and with the prolongation of immersion time, the degree of steel sheet corrosion is intensified. After coating modification, the corrosion degree of the steel sheet is greatly reduced. Under the condition of deionized water, the polysilazane-coated modified steel sheet was soaked for 30 days without obvious corrosion; under the condition of 20 g/L of mineralized water, the polysilazane-coated modified steel sheet began to rust after 5 days of immersion, and the pitting area increases after 30 days, but the corrosion area is greatly reduced compared with the uncoated steel sheet. Under the high temperature environment of 120 °C, no matter whether the coating is applied or not, the surface of the steel sheet does not change much, and the steel sheet coated with the coating has no coating cracking or bubbling phenomenon, which shows that the coating is improving the steel sheet. Corrosion resistance also has excellent heat resistance, which can avoid the problem of the coating being deformed at high temperature and resulting in a decrease in the protection of the steel sheet.

In order to further explore the corrosion process, the corrosion products produced after soaking in deionized water and 20 of g/L mineralized water were analyzed by PXRD, and the results are shown in Figure 2.

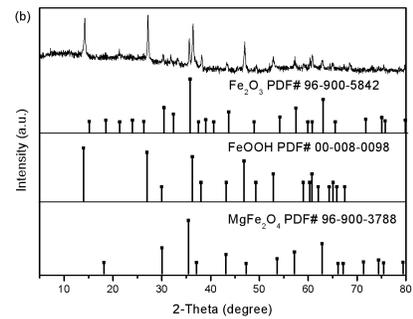
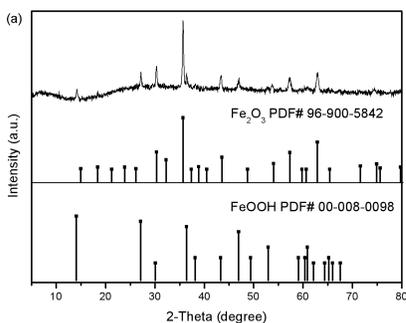


Fig. 2 PXRD patterns of corrosion products. (a) deionized water; (b) mineralized water

It can be seen from Figure 2 that the main component of the corrosion product after immersion in deionized water is Fe_2O_3 , which may contain a small amount of FeOOH . It indicates that after immersion in deionized water, Fe in the steel sheet mainly reacts with dissolved oxygen in the water to form corrosion product rust (Fe_2O_3). However, the corrosion products produced by immersing steel sheets in 20 g/L mineralized water are mainly FeOOH , which can be explained that Cl^- in mineralized water promotes the formation of green rust $\{[\text{Fe}_3(\text{II})\text{Fe}(\text{III})](\text{OH})_8\}\text{Cl}\cdot 2\text{H}_2\text{O}$. The formation of green rust can be automatically converted to FeOOH under certain conditions. Furthermore, a small amount of Fe_2O_3 and MgFe_2O_4 are also produced, which is due to the presence of Mg^{2+} ions in the mineralized water. This also indicates that ions in high mineralized water participate in the rapid oxidation of iron in the steel sheet, thereby accelerating the corrosion of the steel sheet. The coating modification can isolate the reaction between the steel sheet and ions, thereby increasing the corrosion resistance of the steel sheet.

3.5 Corrosion resistance mechanism of polysilazane-coated modified steel sheet

The potentiodynamic polarization curve is a common method to characterize the corrosion characteristics of the material surface. Generally, the lower the self-corrosion potential and the higher the self-corrosion current, the easier the surface is to be corroded [13,14]. In order to simulate the corrosion behavior of the coating in the complex environment of the oil field (high mineralized, high salinity, saturated CO_2 environment), the polysilazane-coated modified steel sheet was investigated in 20 g/L mineralized water and 3.5% of NaCl. The polarization curve, self-corrosion potential and self-corrosion current changes after soaking in the solution for 120 hours are shown in Figure 3 and Table 5.

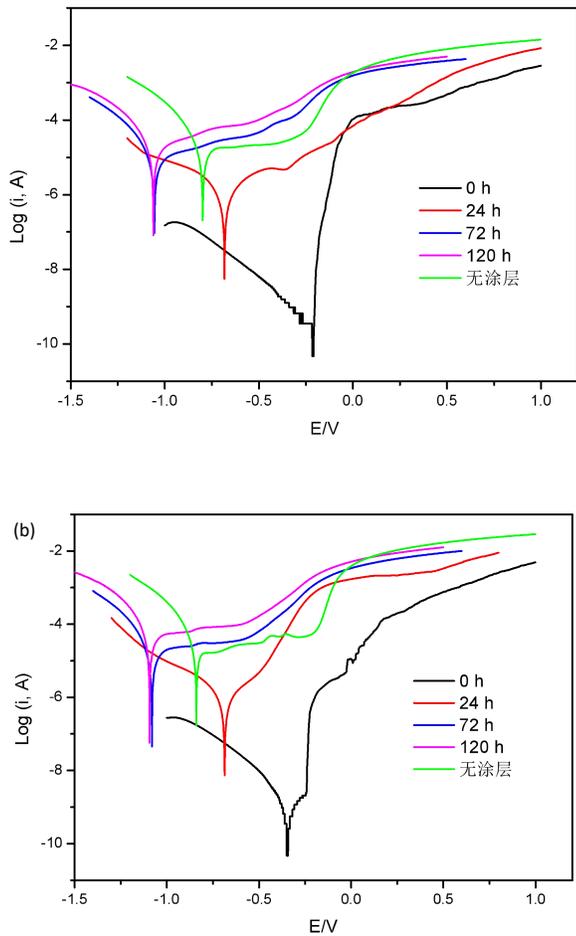


Fig. 3 Polarization curve of polysilazane coating immersed in (a) 20 g/L mineralized water, (b) 3.5% of NaCl solution

From Figure 3 (a) and Table 5, it can be seen that in 20 g/L of mineralized water, the self-corrosion potential of the uncoated steel sheet is -0.799 V, and the self-corrosion potential of the coated steel sheet is -0.216 V, compared with the uncoated steel sheet, increased by 0.583 V. The self-corrosion current decreased significantly, from 2.307×10^{-5} A to 5.407×10^{-10} A. From Figure 3 (b), in the 3.5% of NaCl solution, the self-corrosion potential of the coated modified steel sheet is -0.350 V, which is 0.491 V higher than that of the uncoated steel sheet -0.841 V. Meanwhile, the corrosion current is also reduced from 4.043×10^{-5} A to 4.932×10^{-10} A. It shows that the coating modification can significantly improve the corrosion resistance of the steel sheet. With the prolongation of immersion time, the self-corrosion potential and current of the coating first decreased rapidly and then gradually stabilized, and the corrosion resistance of the coating gradually weakened. This shows that under two different mineralization conditions, the self-corrosion potential of the steel sheet can be increased while the self-corrosion current can be reduced by coating, thereby prolonging the service life of the steel sheet.

Tab. 5. Corrosion current and potential table of polysilazane coating immersed in 20 g/L mineralized water or 3.5% NaCl solution for 120 h

Project		Uncoated	Polysilazane coating			
			0h	24h	72h	120h
Self-corrosion potential/V	20 g/L of mineralized water	-0.799	-2.016	-0.683	-1.056	-1.061
	3.5% of NaCl solution	-0.084	-0.350	-0.687	-1.081	-1.094
Self-corrosion current/A	20 g/L mineralized water	2.307×10^{-5}	5.407×10^{-10}	8.994×10^{-7}	9.954×10^{-6}	1.828×10^{-5}
	3.5% NaCl solution	4.043×10^{-5}	4.932×10^{-10}	1.065×10^{-6}	1.961×10^{-5}	6.346×10^{-5}

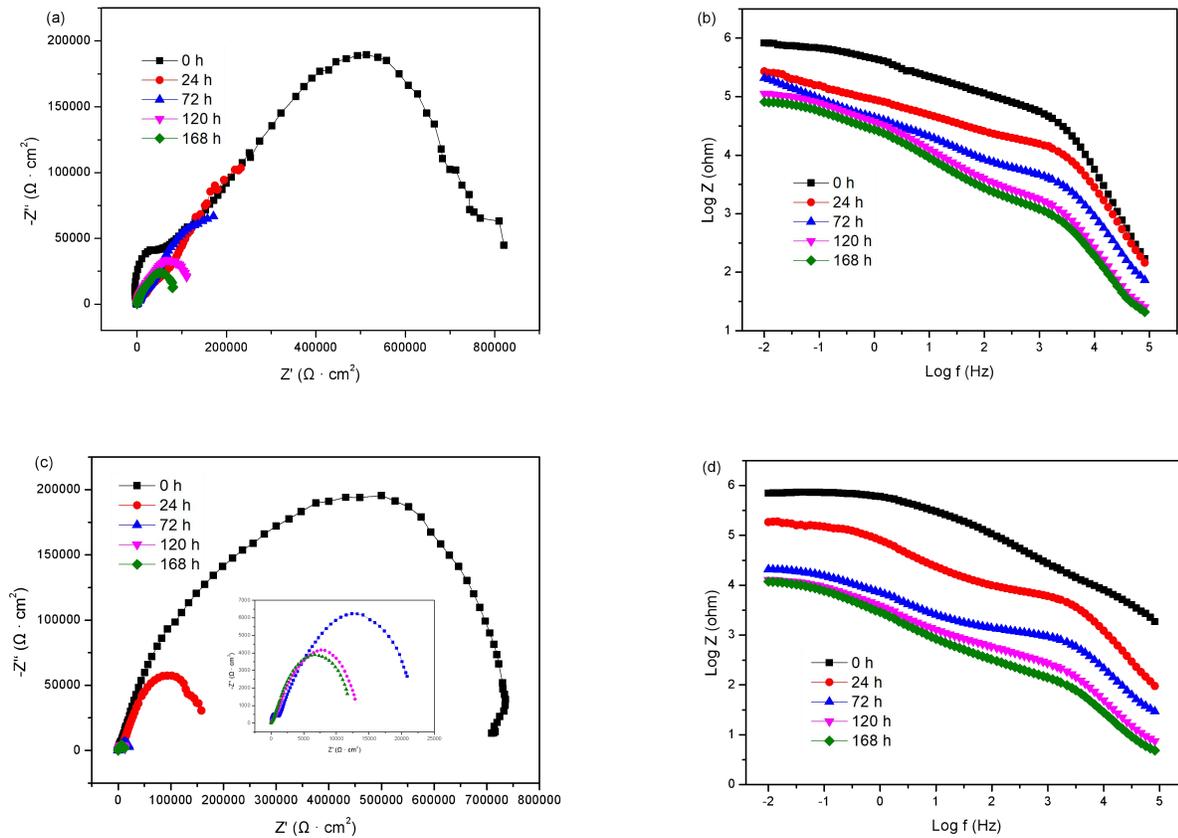
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Electrochemical AC impedance spectroscopy was used to further study the impedance change of the polysilazane coating in the 20 g/L of mineralized water and 3.5% of NaCl solution for 168 h, in order to understand the mechanism of long-term immersion corrosion. The results are shown in Figure 4.



(a)—Nyquist diagram, mineralized water; (b)—Bode modulus diagram, mineralized water; (c)—Nyquist diagram, NaCl solution; (d)—Bode modulus diagram, NaCl solution

Fig. 4 Nyquist and Bode plots of the electrochemical impedance of polysilazane coating for 168 h

It can be seen from Figure 4 that in the early stage of immersion, the surface of the coating is complete, which can be regarded as an insulating layer with high resistance and small capacitance. Therefore, the radius of the capacitive reactance arc is the largest at this time [15,16]. As the immersion time prolongs, the capacitive arc radius of the sample gradually decreases, indicating that the polarization resistance decreases with the immersion process, and the corrosion resistance decreases. The solution penetrates the coating into the surface of the material and corrodes [17,18]. The performance in the Bode diagram is that the modulus curve gradually moves down. Whether in 20 of g/L

mineralized water or 3.5% of NaCl solution, the initial anti-arc radius of the coating is not much different, indicating that the initial protection of the coating on the steel sheet in the two solutions is similar. With the prolongation of immersion time, the impedance of the coating decreased significantly between 24 and 72 hours, and the corrosion rate of the coating accelerated at this time, the corrosion rate became flat after 72 hours. The decrease rate of radius is slower than that in 3.5% of NaCl solution environment, which is consistent with the test results of potentiodynamic polarization curve. It shows that the polysilazane coating can protect the steel sheet and improve the corrosion resistance

under certain time and environmental conditions. However, it is necessary to further add additives and improve the formulation of the coating to enhance the corrosion resistance during long-term immersion.

4. CONCLUSION

(1) Comparing the changes of contact angle, hardness and adhesion of the coating under the two coating processes of brushing and dipping, brushing is easier than dipping to obtain a coating with uniform surface thickness, and the coating thickness is less than 8 μm . The coatings obtained by the two processes can reach the hardness of 5 H and the adhesion of 0.

(2) Compared with the dip coating process, the contact angle of the brush coating process is significantly improved. The average water contact angle is 107.6 $^\circ$, and the average oil contact angle is 47.8 $^\circ$. The surface energy of the polysilazane coating is obtained by calculating and the energy is 15.1 mJ/m², which belongs to low surface energy. It indicates that the improvement of hydrophobic and oleophobic properties of the coated steel sheet, which can prevent the adsorption of water during CO₂ injection, thereby reducing corrosion.

(3) Compared with the uncoated steel sheet, the polysilazane coating increases the corrosion resistance of the steel sheet by increasing the self-corrosion potential and reducing the self-corrosion current without affecting its high temperature resistance. After 30 d immersion in deionized water and baking at 120 $^\circ\text{C}$, no obvious corrosion, bubbling and cracking of the coating was found, and the degree of corrosion after immersion in 20 g/L of mineralized water for 30 days was somewhat reduced.

(4) After the coating is modified, the hydrophobicity, oil repellency, corrosion resistance and anti-clogging performance of the steel sheet are all improved, but if it is to be used in the harsh environment of the oil field for a long time, it is still necessary to add additives to the coating for further improvement, in order to achieve better and longer-lasting protective effect.

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DECLARATION OF INTEREST STATEMENT

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work

reported in this paper. All authors read and approved the final manuscript.

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