Optimization and characterisation of commercial water-based volatile corrosion inhibitor

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Abstract

Volatile corrosion inhibitor (VCI) provides protection for metal surfaces. VCI coating and molecules attach themselves to metal surfaces to form both a physical film when contacted and an invisible thin film for indirect contact through Vapour (only a few molecules thick), thus inhibiting metals atmospheric corrosion. Optimization and characterisation of commercial water-based volatile corrosion inhibitor VCI (Vapour-Phase-Protection, VAPPRO 837C diluted commercial solution from CORPPRO) was prepared to determine their characteristics and effectiveness against corrosion of carbon steel. The main scope of this work is to characterise the rheological and corrosion inhibition properties of the VAPPRO 837C with varying formulations and processing parameters (coating and drying times). Different tests were performed to determine the corrosion behavior of inhibitor. The application of VCI on the metal surface was done by dip-coating process. An Electrochemical Workstation from HCH Instruments has been used to evaluate the corrosion inhibition efficiency of the VCI and to determine the corrosion rate of the uncoated and coated samples. In addition, viscosity tests were carried out in order to determine the rheological properties of the formulation, as well as Freeze–Thaw resistance of water-borne coatings and pH tests were done. A FTIR spectrometer has been used to determine the functional groups present at a specific concentration. Results reveal that the most effective VCI film was obtained from a 0.25 CORPPRO (Concentrated VCI) (vol%) formulation using a coating time of 10 min and a drying time of 24 h. Therefore, 0.25 CORPPRO would be the optimum concentration to be used because it is able to achieve the highest corrosion inhibition efficiency with the optimum coating drying time.

Key words: corrosion inhibitor, vapour phase protection, dip-coating.

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Introduction

Metallic corrosion is a world-wide problem caused by the interaction of the metal surface with the surrounding environment. It has always been problematic to metal constructs and tools as degradation or rusting of metals due to the tendency to return to their natural state

(oxides or other corrosion leading to a weakening of mechanical properties and failure) [1]. Replacing them outright is expensive and, with resources on Earth becoming more limited, problematic in the long run [2]. Thus, corrosion protection of metallic infrastructures is preferable by their cost over part replacement and prolongs the working life of the product in the field, with many methods to do so currently available such as corrosion inhibitors, coatings and cathodic protection [3].

Volatile corrosion inhibitors are widely used as corrosion control and prevention method. They are a part of a class of corrosion inhibiting compounds with a finite vapour pressure where the inhibitors are transported to the target metal through space to condense on the metal surface forming a protective film, lowering the corrosion rate of the metal itself [4]. Factors that determine the efficiency of VCIs are the concentration of VCI compounds, the period of exposure and vapour pressure [5]. The main advantages of these inhibitors are their user-friendly and application VCI onto the metal. Other benefits include to reach crevices, blind holes and other “difficult to reach” areas. Limitations include being temporary films as they can be removed easily, are used at fairly low concentrations and a higher concentration is needed for the self-healing effect which could increase the cost of using it and corrosion protection may not be the only requirement, with others such as colour limitation and film hardness needed. It is due to these factors that they are largely ignored in industrial maintenance coatings [6]. However, a long side corrosion-resistant materials and corrosion protection coatings, corrosion inhibitors are still researched and developed to further lower corrosion rates and to reduce the costs of corrosion [7].

An example of these inhibitors is the ammonia. This compound has been used as a VCI to protect immersed and exposed parts of steam boiler circuits at the beginning of the 20th century [8]. In the 1940s, less odorous, safer and more effective substances were used for protection. With more than 1000 types of VCI compounds known today, only a few are used as these are acceptably efficient, cost effective and environmentally friendly [9]. One type of inhibitor used in VCIs is salts of dicyclohexylamine such as dicyclohexyl ammonium nitrite (DCHN) and cyclohexylamine carbonate (CHC) [10]. The VCI developed by Magna International uses amine carboxylate as the inhibitor. It is a salt synthesized by neutralizing carboxylic acid with a blend of amines, with the number of carbon atoms ranging from 1 to 26, with corrosion inhibition increasing with the number of carbon. Other than corrosion inhibition, it is also used for boundary lubrication, emulsification and detergency [11].

Recently, there has been a shift in using water-based VCIs, which are less hazardous to human health and are environmentally friendly, as opposed to oil-based VCIs. By substituting the organic solvent with water as the transport medium, it would lower the levels of volatile organic compounds (VOCs) emitted by VCIs [12]. This study aims to deduce the concentration of the VCI developed by Magna International, Vappro 837C, that can offer the best corrosion protection and under what conditions can this be achieved. The

effects of the concentration of inhibitors, coating time, drying time and temperature on its performance are studied and observed.

Experimental

Preparation of metal samples

Carbon steel UNS G10100 disks with 0.7853 cm^2 surface area were prepared and de-rusted by hand using 600 grit abrasive paper. Lastly, the samples were raised with deionised water and isopropyl alcohol to remove debris from the de-rusting procedure [13].

Determination of corrosion rate and corrosion inhibitor efficiency

Electrochemical corrosion tests were performed using a HCH Instruments Electrochemical Workstation and a three electrode cell array. The electrochemical method was DC Polarization using the coated and uncoted samples as working electrode, and a silver/silver chloride (Ag/AgCl) as reference electrode, while a platinum mesh was used as counter electrode. A schematic diagram of the electrochemical corrosion cell is given in Figure 1.

Figure 1. Schematic diagram of three-electrodes cell: (a) reference electrode (Ag/AgCl), (b) working electrode (sample), (c) counter-electrode (platinum Pt), (d) electrolyte container, (e) electrolyte, (f) potentiostat.

The parameters established for the electrochemical test were 10 mV s^{-1} scan rate and the polarization range from -100 to -600 mV vs corrosion potential. Final results were adjusted by Tafel analysis to calculate corrosion rates and the percentage of corrosion inhibition efficiency. The corrosive media was Singapore tap water with the typical values of microbiological and physicochemical parameters shown in Table 1 [14].

The corrosion rate was calculated using the following formula:

$$\text{Corrosion rate} = k \left[\frac{(a \cdot i_{\text{corr}})}{(n \cdot D)} \right] \quad (1)$$

where D is density of metal specimen, a is atomic weight, i_{corr} is corrosion current density, n is the number of electrons lost and k is the constant, which depends on unit of corrosion rate. For this work, the k value applied was 0.00327 mm/(A cm year).

Table 1. Microbiological and physical chemical parameters typical values.

Parameter	Unit	Typical value
Microbiological		
<i>Escherichia coli</i> (<i>E. coli</i>)	cfu/100 mL	< 1
Physical		
Colour	Hazen	< 5
Conductivity	μS/cm	< 250
Chlorine	mg/L	< 2.0
pH	Units	7.0 – 8.5
Total Dissolved solids (TDS)	mg/L	< 150.0
Turbidity	NTU	< 5
Chemical		
Ammonia (as N)	mg/L	< 1.0
Calcium	mg/L	4.0 – 20.0
Chloride	mg/L	< 20.0
Copper	mg/L	< 0.05
Fluoride	mg/L	< 0.50
Iron	mg/L	< 0.04
Manganese	mg/L	< 0.05
Nitrite (as N)	mg/L	< 11.0
Sodium	mg/L	< 20.0
Sulphate	mg/L	< 5.0
Silica (as SiO ₂)	mg/L	< 3.0
Total Organic Carbon (TOC)	mg/L	< 0.50
Total Hardness (as CaCO ₃)	mg/L	< 50.0
Zinc	mg/L	< 0.10

Based on the corrosion rates of the metal sample before and after coating, the corrosion inhibition efficiency was calculated based on the formula below [15]:

$$\text{Corrosion inhibition efficiency(\%)} = \frac{(CR_{\text{uninhibited}} - CR_{\text{inhibited}})}{CR_{\text{uninhibited}}} \times 100 \quad (2)$$

where $CR_{\text{uninhibited}}$ is the corrosion rate of uninhibited sample and $CR_{\text{inhibited}}$ is the corrosion rate of inhibited sample.

Formulation of VAPPRO 837C

A concentrated corrosion inhibitor solution based on amine carboxylates was used to prepare formulations of commercial VAPPRO 837C at concentrations of 0.25, 0.50, 0.75 and 1.0 ml/100 ml (v/v) solution. The concentrated corrosion inhibitor CORPPRO was diluted in deionized water to reach the concentrations mentioned above for testing. In order to ensure the stability of the mixtures, the solutions left stand for one day before being used for testing.

Coating of metal samples

VAPPRO 837C was applied on clean metal samples of carbon steel by immersion at three different times using a clock glass as a coating reservoir. Different immersion times were selected for the coating process (10 min, 20 min, 30 min, 8 h and 24 h). After the coating, the samples were removed using tweezers and left to dry. Immediately after drying, the metal samples were tested using potentiostat to calculate their corrosion rates. Eventually the immersion coating and drying time were also varied in order to establish the best condition for an optimum protection efficiency.

Elevated temperature drying

Figure 2 shows the set-up used for the elevated drying temperature test. The light bulb was first switched on to heat up the interior of the box. When the temperature inside the box stops fluctuating, metal samples that have just completed coating were clipped onto the string for drying. After drying, the samples were immediately tested using the potentiostat to determine their corrosion rates.

Figure 2. Elevated temperature drying set-up: (a) light bulb, (b) cal thermometer, (c) sample and (d) container.

Freeze–Thaw Test

The freeze thaw test was conducted following the ASTM D2243-95(2008) standard [16]. The formulations were first tested for their initial viscosity (before freezing). After the first testing, the formulations were stored at -18°C in a freezer for 17 hours. Subsequently, the formulations were removed and thawed at room temperature for 7 hours. Posterior to thawing, the formulations were placed back in the freezer at -18°C and one more cycle of freeze-thaw was carried out. After the final thawing step was completed, the formulations were tested for their viscosity. The viscosity before and after freeze-thawing were compared to determine the freeze-thaw stability of the formulations.

FTIR Analysis

A Fourier Transform Infrared analysis was conducted on a 0.25 CORPPRO formulation using the Attenuated Total Reflectance (ATR) method. A background scan was first carried out. After which, 2–3 drops of the formulation was added onto the zinc selenide crystal surface using a disposable dropper. The formulation was then scanned, and the resulting spectrum was analysed.

Results and Discussion

Polarisation Curves

The Annex 1 shows the potentiodynamic polarisation curves (Figures 4 to 7) and Annex 2 presents the data from these curves. The results were grouped according to the drying time and compared with their concentrations. The behavior of the polarization curves depends of the inhibitor such as cathodic, anodic and organic inhibitors [18]. For the organic inhibitors, the effect of the solution in the metal sample can be presented by a mixed behavior [19]. The results in this work suggest that the behavior of the polarization curves could change due to different factors such as the concentration, the coating time and the drying time. But it is possible to follow a tendency.

For all curves, the cathodic and anodic slopes showed the same behavior but the position of potential and current changed regarding the blank. With a drying time of 1 h, results showed that for any concentration, the polarization curve had a nobler position when the coating time was below 30 min. As well, the value of the current decrease when a coating time was below 30 min. This behavior is repeated on 2 h of drying time, but this changed when a 5 h or 24 h of drying time was applied. In this case, the nobler positions were obtained when a coating time was over 30 min.

As a result of this way of acting, samples with inhibitor presented an effect of corrosion inhibition. Also observed in the values of corrosion efficiency and corrosion rate because all samples with inhibitor had a better performance in the environment. This may be due to the formation of a protective film of molecules on the metal surface and a mixed-effect of inhibition. Different articles have suggested this mechanism of inhibition [20, 21]. Most VCIs form a film by surface adsorption upon hydrolysis or dissociation. The presence of amino groups, carboxylate anions and the π -bond between carbon atoms enhance the corrosion-inhibiting [22].

Corrosion Reduction Efficiency

The effect of drying time on corrosion reduction efficiency is shown in Tables 6 to 9 (Annex 2). The data gathered suggests that increasing the drying time, the corrosion reduction efficiency becomes stable for any time of immersion or inhibitor concentration. It can be seen with a drying time of 5 h and 24 h where is presented with a small variation of efficiency for 10, 20, and 30 min for any concentration. However the highest corrosion efficiency was provided with 1 h of drying time (91.29%), but it is not uniform regard the concentration.

This could be because while the coating appears to be dry visually after 20 min of drying, there could still be some water content present in the film which promotes corrosion. It is also possible that the protective film of inhibitor molecules have not been properly organized for drying times before 5 h. This suggests that the optimum drying time should be around 24 h.

This optimum drying time of 24 h is also conducive for practical consideration of working hours in the industry. It would be easy to keep track of the end of the drying time and ensure that it ends within working hours so that the metals can be stored immediately after drying.

Similarly, the effect of concentration of CORPPRO can be interpreted. The highest corrosion reduction efficiency was provided with a CORPPRO concentration of 0.5 when the drying time was 1 h, 2 h, and 5 h. For 24 h the highest corrosion reduction efficiency was obtained with a concentration of 0.25 (85.07%), nevertheless the concentration of 0.5 is close to this efficiency (82.27%).

Through varying the coating time of CORPPRO, it was observed that the corrosion reduction efficiency of the formulations is different according to drying time. For 1 and 2 h

of drying the efficiency does not show a stable trend varying coating time. On the other hand for 5 h and 24 h of drying the corrosion reduction efficiency is stable from 10 min to 8 h of coating time. This suggests that increasing the coating time beyond 30 min has no significant effect on the corrosion reduction efficiency compared to a coating time of 30 min.

This could be because at 30 min the surface of the metal substrate is fully saturated with inhibitor molecules and the metal surface-inhibitor interactions have stabilised. Therefore any further coating time would not cause a decrease in corrosion, while a lower coating time would result in the substrate's surface not being saturated with inhibitor molecules which correlates to a higher corrosion rate.

Considering the cost of time for a corporation, using a coating time of 10 min would be optimum as this would allow a fast processing time without any loss in corrosion inhibition.

Effect of drying at elevated temperatures

Through varying the temperature of drying, it was observed that drying at an elevated temperature of about 30°C obtains higher corrosion reduction efficiency. This can be seen from the 30°C drying obtaining a corrosion reduction efficiency of 58.6% while the 20°C drying obtained a corrosion reduction efficiency of 38%, as shown in Table 2 below.

Table 2. Effect of temperature during drying on corrosion reduction efficiency of 0.25 CORPPRO for 30 min coating time.

Temperature (°C)	Corrosion Reduction Efficiency (%)
	0.25 CORPPRO 30 min coating 2 h room temperature drying
20	38
33	58.6

This could be because at elevated temperatures, the inhibitor molecules have more kinetic energy and are more mobile and thus more likely to orient themselves such that the polar functional groups faces the metal surface to preferentially form a protective film over the surface of the metal substrate, therefore resulting in an increase in corrosion reduction efficiency for the metal sample that was dried at elevated temperatures [17].

After 2 h drying at elevated temperatures, the surface of the metal sample still appeared wet on visual inspection. This is likely because the box used to contain the samples at elevated temperatures also limited the ventilation to a small area. This would reduce the rate of evaporation of the wet coating layer. Therefore for future testing of drying at elevated temperatures, it would be suggested to carry out the drying in a well-ventilated area.

Compared to samples that were dried at room temperature and not contained in a box but left in a well-ventilated area and had a dry surface on visual inspection, the samples dried at elevated temperatures showed higher corrosion reduction efficiency. This suggests that the corrosion reduction efficiency is not directly related to the water content present in the coated film, but rather on the complete formation of the protective film of inhibitor molecules.

Freeze–thaw tests

By varying the concentration of anti-freeze (Ethylene glycol) in the formulations, it was observed that all the formulations froze at -18°C . It was also observed that the viscosities of all the formulations remained around 29 cP after undergoing the freeze-thaw test, as shown in Table 3. For all the formulations, there was no significant change in the viscosity after two cycles of freeze-thawing. Further testing is required in order to determine if the viscosity of the formulations will remain consistent after going through more freeze-thaw cycles.

The 0.25 CORPPRO formulations froze even after the addition of 1% ethylene glycol anti-freeze agent. To prevent the freezing of the formulations at -18°C , a higher concentration of anti-freeze agent would have to be used.

Table 3. Effects of freeze-thawing on the viscosity of 0.25 CORPPRO formulations with and without 1% antifreeze.

% Antifreeze	Viscosity (cP)		
	Before freeze	After freeze	Freeze
Freeze Thaw 1 with 1% antifreeze	28.2	30.9	Yes
Freeze Thaw 2 with 1% antifreeze	29.3	28.3	Yes
Freeze Thaw 1 w/o antifreeze	31.2	30.0	Yes
Freeze Thaw 2 w/o antifreeze	28.0	30.6	Yes

pH test for varying concentrations of CORPPRO

Through varying the concentration of CORPPRO, it was observed that the pH is above 9 for all concentrations, as shown in Table 4. The MSDS of CORPPRO states that it has a pH of 9, therefore it can be seen that diluting CORPPRO with distilled water does not affect its pH. For all formulations, the pH is below 11 and thus, they will not cause skin irritation.

Table 4. Effect of concentration of CORPPRO on pH of formulations with different concentrations.

CORPPRO Concentration (vol%)	Average pH
0.25	9.60
0.50	9.60
0.75	10.60
1.0	10.7

Since the pH of the formulations does not vary much as the concentration of CORPPRO varies, it would not be viable to use pH as a measure to determine the concentration of CORPPRO formulations during manufacturing. It would be advised to use viscosity instead of pH to determine the concentration of formulations produced during manufacturing.

Functional Group Determination by FTIR

Figure 3 below shows the FTIR spectrum of 0.25 CORPPRO. The significant peaks were identified and their correlated functional groups peaks are shown in Table 5.

Figure 31. 0.25 CORPPRO FTIR spectrum.

Table 5. Correlated functional groups of significant peaks in 0.25 CORPPRO FTIR spectrum.

Wavenumber (cm ⁻¹)	Functional group	Transition
≈3300	Amine	–N–H stretch
3000–2850	Alkanes	C–H stretch
1640–1550	Primary and secondary amines	N–H bend
1599	Carboxylate	COO ⁻ asym stretch
1390–1410	Carboxylate	COO ⁻ sym stretch
1300–1450	Aromatic	C–C stretch

The spectrum shown in Figure 3 has a peaks at around 2927 cm⁻¹, this could be due to carboxylate O–H stretching or alkane C–H stretching. The peaks at 1595 cm⁻¹ and 1547 are due to COO⁻ asymmetric stretch or primary/secondary amine N–H bending. The peak at 1401 cm⁻¹ corresponds to carboxylate COO⁻ symmetric stretching. The wavenumber, functional groups and transition are presented in Table 5.

Based on the absorption peaks present in the spectrum, it is likely CORPPRO contains an organic compound amine carboxylate aromatic type. This matches the description of CORPPRO in its MSDS which states that CORPPRO contains “amine carboxylate”.

Conclusions

The following conclusions can be derived based on the results obtained:

1. The optimum concentration for VAPPRO 837C formulations to achieve the highest corrosion reduction efficiency is 0.25 CORPPRO according to the optimum drying time.
2. The optimum processing parameters to achieve a short processing time without sacrificing corrosion reduction efficiency for coating carbon steel samples are 10 min coating and 24 h drying, as seen in effect of varying coating time on corrosion reduction efficiency and effect of varying drying time on corrosion reduction efficiency.
3. The formulations were found to have stable viscosity over a 5 week period. The viscosities were also stable after freeze thawing for two cycles. This would suggest that there is no degradation of the VCI over the 5 week period and from freezing. But further testing is required to determine the amount of anti-freeze required to prevent freezing since the formulations with 1% antifreeze still froze at –18°C. This can be seen in Viscosity Stability Tests and Freeze–thaw Tests.

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Annex 1

Figure 4. 1 h of drying. 0.25, 0.5, 0.75, 1.0 CORPPRO.

Figure 5. 2 h of drying. 0.25, 0.5, 0.75, 1.0 CORPPRO.

Figure 6. 5 h of drying. 0.25, 0.5, 0.75, 1.0 CORPPRO.

Figure 7. 24 h of drying. 0.25, 0.5, 0.75, 1.0 CORPPRO.

Annex 2

Table 6. Results obtained from polarization curves for carbon steel with different concentrations of VAPPRO CORPPRO (Drying time 1 h).

Concentration (vol %)	Coating time	E_{corr} (V)	I_{corr} ($\mu\text{A cm}^{-2}$)	Corrosion Rate (mm/year)	Efficiency (%)
0.25	Blank	−0.518	0.4576	0.643	
	10 min	−0.527	0.2893	0.501	22.08
	20 min	−0.49	0.2613	0.476	25.97
	30 min	−0.479	0.2066	0.377	41.37
	8 h	−0.503	0.08183	0.12	81.34
	24 h	−0.429	0.2065	0.3815	40.67
0.50	10 min	−0.545	0.1853	0.345	46.35
	20 min	−0.556	0.3094	0.571	11.20
	30 min	−0.517	0.1971	0.364	43.39
	8 h	−0.525	0.03852	0.056	91.29
	24 h	−0.488	0.4198	0.463	27.99
0.75	10 min	−0.449	0.1094	0.202	68.58
	20 min	−0.501	0.1115	0.426	33.75
	30 min	−0.418	0.1831	0.338	47.43
	8 h	−0.443	0.3177	0.469	27.06
	24 h	−0.485	0.1445	0.266	58.63
1.0	10 min	−0.492	0.1007	0.186	71.07
	20 min	−0.476	0.2449	0.453	29.55
	30 min	−0.51	0.0347	0.064	90.05
	8 h	−0.422	0.1845	0.34	47.12
	24 h	−0.473	0.06921	0.102	84.14

Table 7. Results obtained from polarization curves for carbon steel with different concentrations of VAPPRO CORPPRO (Drying time 2 h).

Concentration (vol%)	Coating time	E_{corr} (V)	I_{corr} ($\mu\text{A cm}^{-2}$)	Corrosion Rate (mm/year)	Efficiency (%)
0.25	Blank	−0.518	0.4576	0.643	
	10 min	−0.446	0.1655	0.305	54.25
	20 min	−0.442	0.1755	0.324	51.20
	30 min	−0.424	0.2111	0.39	40.61
	8 h	−0.432	0.08577	0.126	82.99
	24 h	−0.423	0.2035	0.3	55.06
0.50	10 min	−0.533	0.08052	0.148	79.45
	20 min	−0.466	0.2377	0.439	32.74
	30 min	−0.483	0.3701	0.583	9.63
	8 h	−0.497	0.08001	0.118	84.27
	24 h	−0.517	0.05817	0.107	86.04
0.75	10 min	−0.548	0.1441	0.212	69.18
	20 min	−0.471	0.2774	0.512	21.03
	30 min	−0.473	0.2236	0.413	36.92
	8 h	−0.485	0.09218	0.136	81.38
	24 h	−0.475	0.1726	0.319	52.01
1.0	10 min	−0.446	0.3358	0.618	4.01
	20 min	−0.434	0.1722	0.318	52.17%
	30 min	−0.43	0.2025	0.374	43.18%
	8 h	−0.453	0.3664	0.616	4.33%
	24 h	−0.477	0.07589	0.112	85.23%

Table 8. Results obtained from polarization curves for carbon steel with different concentrations of VAPPRO CORPPRO (Drying time 5 h).

Concentration (vol%)	Coating time	E_{corr} (V)	I_{corr} ($\mu\text{A cm}^{-2}$)	Corrosion Rate (mm/year)	Efficiency (%)
0.25	Blanc	−0.518	0.4576	0.643	
	10 min	−0.386	0.1999	0.37	42.46
	20 min	−0.404	0.1385	0.255	60.34
	30 min	−0.463	0.07362	0.136	78.85
	8 h	−0.466	0.1022	0.15	76.67
	24 h	−0.546	0.2198	0.324	49.61
0.50	10 min	−0.514	0.1928	0.356	44.63
	20 min	−0.487	0.07031	0.123	80.87
	30 min	−0.458	0.04859	0.089	86.16
	8 h	−0.484	0.1034	0.152	76.36
	24 h	−0.405	0.1473	0.286	55.52
0.75	10 min	−0.503	0.1051	0.194	69.83
	20 min	−0.492	0.3276	0.5651	12.12
	30 min	−0.455	0.2146	0.396	38.41
	8 h	−0.462	0.1088	0.16	75.12
	24 h	−0.566	0.2143	0.316	50.86
1.0	10 min	−0.477	0.2384	0.44	31.57
	20 min	−0.42	0.1613	0.298	53.65
	30 min	−0.465	0.0885	0.164	74.49
	8 h	−0.473	0.0971	0.143	77.76
	24 h	−0.435	0.08684	0.128	80.09

Table 9. Results obtained from polarization curves for carbon steel with different concentrations of VAPPRO CORPPRO (Drying time 24 h).

Concentration (vol%)	Coating time	E_{corr} (V)	I_{corr} ($\mu\text{A cm}^{-2}$)	Corrosion Rate (mm/year)	Efficiency (%)
0.25	Blanc	−0.518	0.4576	0.643	
	10 min	−0.504	0.06517	0.096	85.07
	20 min	−0.507	0.1209	0.178	72.32
	30 min	−0.445	0.1174	0.173	73.09
	8 h	−0.547	0.2731	0.504	21.62
	24 h	−0.492	0.2689	0.397	38.26
0.5	10 min	−0.425	0.1191	0.175	72.78
	20 min	−0.465	0.1088	0.16	75.12
	30 min	−0.462	0.08547	0.126	80.40
	8 h	−0.44	0.0777	0.114	82.27
	24 h	−0.49	0.3139	0.463	27.99
0.75	10 min	−0.559	0.1274	0.188	70.76
	20 min	−0.544	0.09266	0.136	78.85
	30 min	−0.461	0.09375	0.138	78.54
	8 h	−0.447	0.1013	0.149	76.83
	24 h	−0.584	0.2314	0.341	46.97
1.0	10 min	−0.546	0.1061	0.156	75.74
	20 min	−0.527	0.0883	0.13	79.78
	30 min	−0.533	0.06786	0.1	84.45
	8 h	−0.415	0.05728	0.105	83.67
	24 h	−0.471	0.2035	0.3	53.34