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Influence of Reaction Temperature on Corrosion-Resistant Characteristics of Poly(Aniline-Co-2-Methylaniline) Coatings

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Abstract

The poly(aniline-co-2-methylaniline) PAMA coatings were synthesized by keeping the feed ratio 1:1 of aniline and 2-methylaniline, respectively. These PAMA coatings were deposited at different reaction temperatures, 0˚C, 15˚C, 27˚C (ambient temperature (AT)), 40˚C, and 60˚C on the low carbon steel (LCS) surface by electrocopolymerization of aniline with 2-methylaniline. This electro polymerization was carried out in an aqueous solution containing 0.2 M sodium tartrate as the supporting electrolyte. *In situ* characterization of these coatings was performed by cyclic voltammetry, while the corrosion-resistant characteristics of coatings against corrosion of LCS were investigated in aqueous 3% NaCl by Tafel plot. The results of the Tafel plot measurements show that the PAMA coatings synthesized at AT have the lowest Corrosion Rate, 50 times lower than uncoated LCS, and also a higher positive shift in E_{corr} of 381 mV, so it provides more effective corrosion-resistant characteristics than that of 0˚C, 15˚C, 40˚C, and 60˚C against the corrosion of LCS.

Keywords

Reaction Temperature, Poly(Aniline-Co-2-Methylaniline), Corrosion, Cyclic Voltammetry, Low Carbon Steel

1. Introduction

Conducting polymers (CPs) are widely investigated because of their remarkable features, such as electrical conductivity, electrochemical activity, environmental stability, processability, optical, and promising thermoelectric properties, exhibit high biocompatibility, and can act as a barrier against corrosion of metals [1]-[12].

In order to further increase the versatility and functionality for enhanced performance of conducting polymers in applications, copolymers are synthesized by polymerizing two or more different monomers to combine the desirable properties of each component [13]-[19].

The extensive study was conducted to find how synthesis temperature significantly influences the properties of conducting polymers (CPs) [20]-[27]. Koinker *et al.* [20] represent that synthesis temperature significantly affects the ECP of CPs. Liu *et al.* investigated the impact of temperature on the corrosion and cathodic protection of X65 pipeline steel in a 3.5% sodium chloride solution, which is crucial for the longevity and safety of oil and gas pipelines. The result of this study indicates that the corrosion resistance of X65 steel decreased with the increase in temperature. In order to protect material from corrosion, the temperature must be controlled at a low level [21].

In the present work, the poly(aniline-co-2-methylaniline) 1:1 copolymer (PAMA), polyaniline (PANI), and poly-2-methylaniline (PMA) coatings have been deposited on LCS in an aqueous tartrate solution at various reaction temperatures, such as 60˚C, 40˚C, 27˚C, 15˚C, and 0˚C [20] [22]. The selected temperature range of 0˚C to 60˚C was chosen to encompass typical environmental and industrial conditions relevant to the synthesis and application of polymer coatings. This range allows us to observe the effects of both sub-ambient and elevated temperatures on the electro polymerization process and study the influence of reaction temperature on the corrosion performance of the resulting coatings.

2. Materials and Methods

2.1. Materials

Aniline and 2-methylaniline are monomers, and an aqueous sodium tartrate solution was used as the supporting electrolyte. The LCS substrate was polished with polishing paper, cleaned, and washed with acetone and deionised water.

2.2. Synthesis of Coatings

Initially, the polyaniline (PANI), poly-2-methylaniline (PMA), and poly(aniline-co-2-methylaniline) 1:1 copolymer (PAMA) coatings were synthesized on low-carbon steel (LCS) in an aqueous tartrate solution. During the overall synthesis process, the concentration of each monomer and tartrate solution was kept constant at 0.2 M. The electrochemical polymerization (ECP) was accomplished by using a three-electrode configuration, working electrode (LCS), counter electrode (platinum), and reference electrode (saturated calomel electrode (SCE)).

The synthesis was carried out under cyclic voltammetry by scanning the electrode potential in the range −0.5 to 1.5 V at a scan rate of 20×10^{-3} V/Sec. The CV was recorded using a PARSTAT 2363-1, EG and G, Princeton Applied Research (U.S.A.) in triplicate to ensure reproducibility.

Afterwards, the PAMA copolymer coatings were synthesized at various reaction temperatures by keeping the same experimental conditions as explained above, ex-

cept for the temperature. The higher temperatures, *i.e.*, 60˚C and 40˚C, were kept constant by a hot plate on a magnetic stirrer. While the lower temperature of 15˚C was maintained by an ice bath, 0˚C was retained by adding salt to the ice bath.

2.3. Characterizations

The coatings were characterized by cyclic voltammetry (CV), scanning electron microscopy (SEM) with a Leica (United Kingdom) Cambridge 440 microscope., and corrosion resistant characteristics are characterized by Tafel plots by scanning the electrode voltage in range −0.25 to 0.25 V at the scan rate of 2×10^{-3} V/s by PARSTAT 2363-1 in triplicate to ensure reproducibility.

3. Results and Discussion

3.1. Discussion on the Synthesis of Coatings

The polyaniline (PANI), poly-2-methylaniline (PMA), and poly(aniline-co-2-methylaniline) 1:1 copolymer (PAMA) coatings were synthesized on LCS in an aqueous tartrate solution, showing the same cyclic voltammetry results as reported in Pawar *et al.* [19]. The electroanalysis of the first scans of PAMA copolymer coatings at different reaction temperatures is shown in Figure 1. These cyclic voltammograms are characterized by three anodic peaks: A, B, and C. The anodic peak A represents the dissolution of the reactive LCS electrode surface, which produces Fe2+ ions in its vicinity. These ions interact with the tartrate counter-ions of the electrolyte to form insoluble iron (II) tartrate ($FeC_4H_4O_6$), which adheres to the electrode surface, thereby forming an iron tartrate film. Oxidation peak B represents the oxidation of the monomer(s) and formation of radical cations, which are rapidly consumed in subsequent reactions to yield dimers, trimers, tetramers, and so forth. Anodic peak C is assigned to the oxidation of the tartrate electrolyte [19].

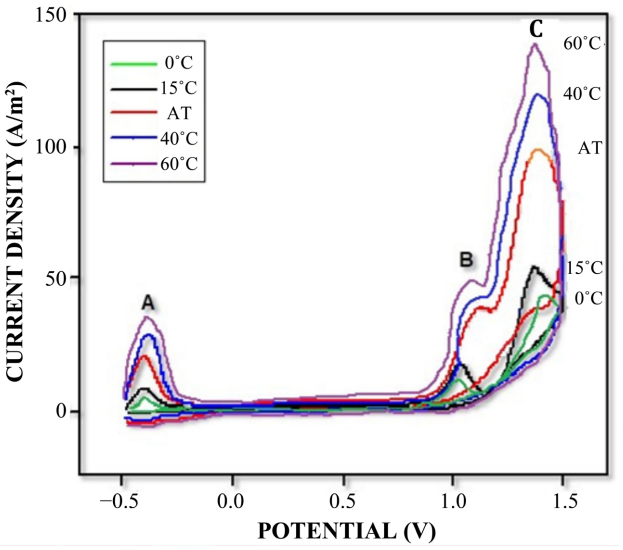


Figure 1. First scan of CV recorded during the synthesis of PAMA copolymer coatings at different reaction temperatures.

Nevertheless, a critical observation of 1st scan of the CV represents that the area of the anodic peak A of PAMA copolymer diverges substantially depending on the reaction temperature, shown in Figure 2 and it obeys the sequence 60˚C > 40˚C > 27˚C (AT) > 15˚C > 0˚C so highest for 60˚C and lowest for 0˚C while intermediate for AT. In the second scan of cyclic voltammograms of PAMA shown in Figure 3, recorded at different reaction temperatures, the anodic peak A which represents dissolution of LCS, is not observed. The growth of PAMA copolymer occurs in the second scan as current densities of oxidation peaks are increased, and it follows the order 60˚C > 40˚C > 27˚C > 15˚C > 0˚C as shown in Figure 4. After the second scan, the current density corresponding to the anodic peaks decreased gradually with the number of scans.

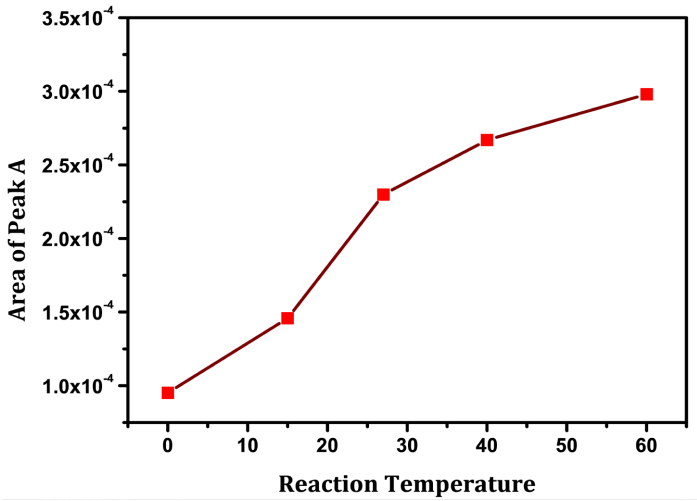


Figure 2. Variation area of the anodic peak A of the PAMA copolymer with reaction temperature.

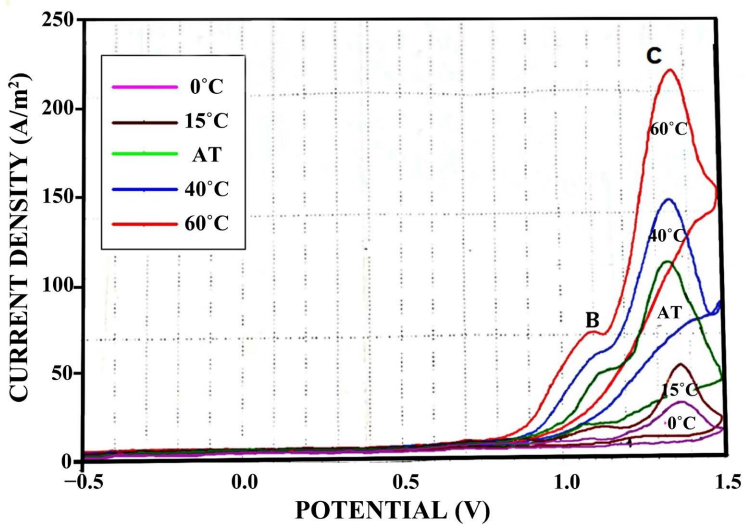


Figure 3. A second scan of CV was recorded during the synthesis of PAMA copolymer coatings at different reaction temperatures.

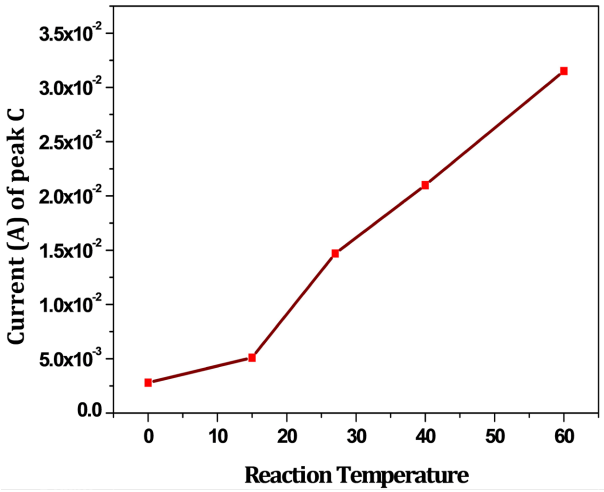
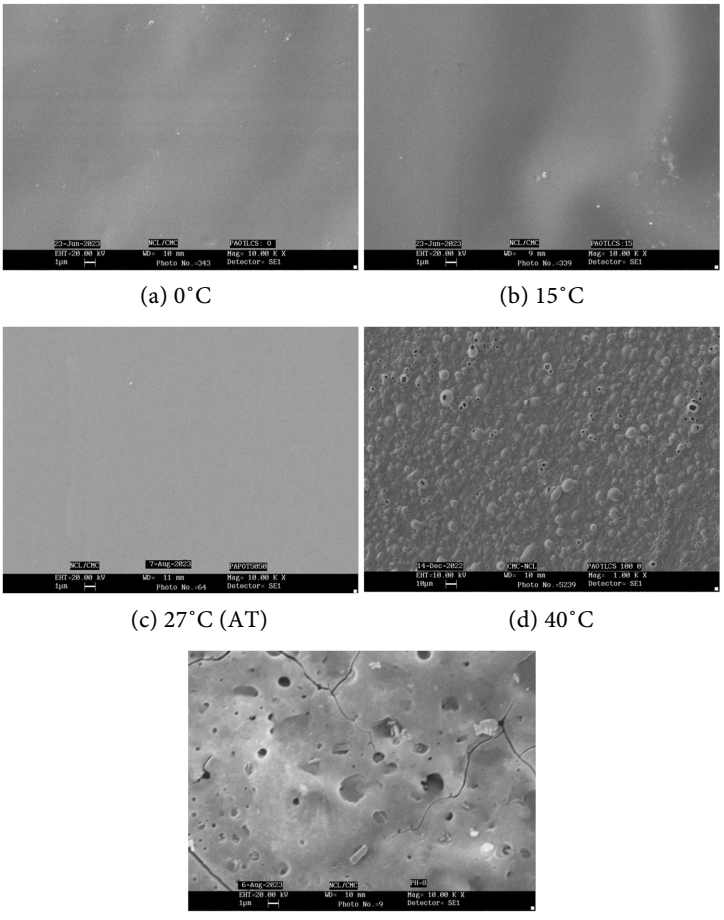


Figure 4. Variation of anodic peak C of PAMA copolymer coatings with reaction temperature during the 2nd scan of CV.

3.2. SEM Images of PAMA Copolymer at Different Reaction Temperature



(a) 0˚C (b) 15˚C

(c) 27˚C (AT) (d) 40˚C

(e) 60˚C

Figure 5. The SEM images of PAMA coatings synthesized at reaction temperatures: (a) 0˚C, (b) 15˚C, (c) 27˚C (AT), (d) 40˚C, (e) 60˚C.

The SEM images of PAMA coatings synthesized at different reaction temperatures after completion of the 10th scan shows that (Figure 5) at higher temperature such as 60˚C and 40˚C thick black colored porous nonuniform surface morphology, *i.e.*, degradation in quality of film is observed it may result due to very high electric current densities of oxidation peaks outcome the hyper-oxidation of monomer. However, at AT, the surface morphology is uniform and featureless as the current densities of the peaks are intermediate. But at lower temperatures, such as 15˚C and 0˚C, the surface morphology is uniform and featureless, but visual observation shows that films are very thin and non-adhesive due to small current densities and do not show complete oxidation or reduction performance.

3.3. Corrosion Protection Performance

3.3.1. Corrosion Protection Performance of PANI, PMA, and PAMA Coatings

The Tafel plots recorded in aqueous 3% NaCl of PANI, PMA, and PAMA copolymer and uncoated LCS can be observed in Figure 6. The Corrosion Rate (CR) measured in mm/year for PANI (0.07), PMA (0.01), and PAMA (0.004), and uncoated LCS (0.20), and those are 3, 20, and 50 times lower than that observed for uncoated LCS. The considerable shift in E_{corr} is observed as compared to uncoated LCS, showing the order PAMA (−329 mV) > POT (−375 mV) > PANI (−506 mV) > uncoated LCS (−710 mV). Thus, this analysis indicates that the copolymers are more effective in protecting the LCS than the corresponding homopolymers, *i.e.*, Polyaniline and Poly-2-methylaniline.

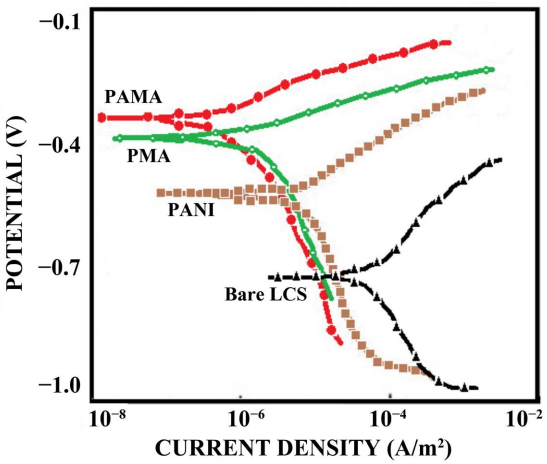


Figure 6. The Tafel plots of PANI, PMA, PAMA copolymer, and Bare LCS were recorded in aqueous 3% NaCl.

3.3.2. Corrosion Protection Performance of PAMA Coatings of Different Reaction Temperature

By taking previous results as a reference [19] and moving forward in it, PAMA copolymer coatings, which effectively protect the LCS, are synthesized at various reaction temperatures and evaluated for their corrosion-resistant characteristics in aqueous 3% NaCl solution by recording the Tafel plots as shown in Figure 7. The analysed values of E_{corr}, I_{corr}, and CR derived from the Tafel plot fitting are given in Table 1.

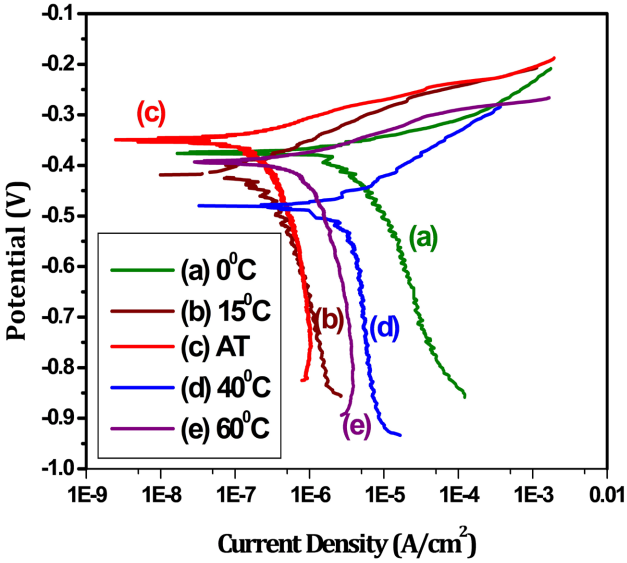


Figure 7. Tafel plots of PAMA copolymer coatings synthesized at different reaction temperatures recorded in aqueous 3% NaCl solution.

The Tafel plot analysis shows a positive shift in corrosion potential (Ecorr) and a significant decrease in corrosion current (Icorr) and CR of LCS due to these PAMA coatings at various temperatures. The corrosion potential (Ecorr) is function of reaction temperature and shows sequence 27˚C (AT) (−329 mV) > 0˚C (−369 mV) > 40˚C (−383 mV) > 15˚C (−390mV) > 60˚C (−413 mV) (with positive shift in Ecorr of (381, 341, 327, 320, 297) mV resp. as compared to uncoated LCS). In contrast, the corrosion rate of these coatings follows the order AT (0.004) > 40˚C (0.009) > 0˚C (0.0136) > 60˚C (0.014) > 15˚C (0.017), and a substantial reduction in CR 50, 22, 15, 14, 12 times, respectively. Compared to uncoated LCS. The hyper-oxidation at higher temperatures, 60˚C and 40˚C, disrupts the polymer's conjugated structure, reducing its electrical conductivity, which is essential for effective corrosion protection. The polymer's morphology becomes less ordered, leading to increased porosity and cracks and reduced barrier properties against corrosion. In comparison, the under-oxidation (at 15˚C and 0˚C) of conducting polymers during synthesis leads to incomplete doping and poor conductivity, which compromises the polymer's ability to protect substrates from corrosion [28].

Thus, these corrosion performance results reveal that the PAMA copolymer coatings synthesized at ambient temperature show better corrosion protection characteristics than other reaction temperatures, with a maximum positive corrosion potential (E_{corr}) shift of 381 mV and also a maximum reduction in CR of 50 times as compared to uncoated LCS.

The detailed comparison of our findings with those reported in existing literature is represented in **Table 2**, which highlights the novelty and significance of our work, especially the identification of 27˚C as an optimal synthesis temperature for achieving enhanced corrosion protection.

Table 1. Analysis of corrosion protection performance results of PAMA copolymer synthesized at various reaction temperature in 3% NaCl.

Synthesis Temperature	E_{corr} (mV)	Positive shift in E_{corr} compared to uncoated LCS (mV)	I_{corr} (A/cm^2)	Corrosion rate (CR) (mm/yr)	Reduction in CR compared to uncoated LCS
Uncoated LCS	−710	--	17.79×10^{-6}	0.20	--
0˚C	−369	341	1.17×10^{-6}	0.0136	15 times
15˚C	−390	320	1.38×10^{-6}	0.017	12 times
AT (27˚C)	−329	381	4.18×10^{-7}	0.004	50 times
40˚C	−383	327	8.59×10^{-7}	0.009	22 times
60˚C	−413	297	9.84×10^{-7}	0.014	14 times

Table 2. Comparison of the influence of synthesis temperature on corrosion resistance of conducting polymers with existing literature.

Ref. No.	Citation	Polymer/System	Temperature Range Studied	Observed Effect on Corrosion Resistance	Key Findings
--	**Current study**	Poly(aniline-co-2-methylaniline)	0˚C to 60˚C	Best corrosion resistance at ambient temperature	PAMA coatings deposited at 27˚C (AT) effectively protect LCS against corrosion than the other reaction temperatures in 3% NaCl.
20	Koinkar *et al.* (2002)	Poly(o-anisidine) on steel	0˚C to 40˚C	------	The surface morphology of coating depend on the synthesis temperature.
21	Liu *et al.* (2019)	Steel in NaCl (non-CP)	25˚C to 60˚C	Higher temp increased corrosion rate	Used for cathodic protection comparison; not a polymer
22	Raotole *et al.* (2017)	Polyaniline	0˚C to 60˚C	Best resistance at ~27˚C (RT)	Higher and lower synthesis temperatures are not suitable for better corrosion performance
23	Zhao & Xu (2017)	Generic CP coatings	Not specified, variable	Higher temp reduced dopant stability and film integrity	Stressed dopant role alongside temperature
24	Khan & Ahmed (2016)	Polythiophene	10˚C to 40˚C	Resistance dropped at ≥30˚C	Higher temp led to overoxidation and porosity
25	Zhang & Wang (2019)	CP films	10˚C to 50˚C	Optimal at 10˚C - 20˚C; degraded at 50˚C	Surface cracks and decreased conductivity at high temperatures
26	Li & Wang (2018)	Polyaniline on mild steel	5˚C to 40˚C	Best corrosion resistance at 5˚C - 15˚C	High-temp films are less compact and more permeable
27	Tombácz & Szabó (2014)	Various CPs	5˚C to 35˚C	Lower temperatures yielded better corrosion resistance	Synthesis temp affected morphology and barrier efficiency
28	Holze (2022)	General CPs	Review	Overoxidation accelerated by high temp	Emphasized temperature control to avoid structural damage

4. Highlights

i) PAMA coatings were deposited at different reaction temperatures on the LCS surface by electro-copolymerization in an aqueous sodium tartrate solution as the supporting electrolyte.

ii) Tafel plots show that corrosion potential (E_{corr}) and corrosion rate (CR) effectively depend on reaction temperature.

iii) At lower temperatures (15˚C and 0˚C), CV shows complete oxidation, reduction performance with uniform and featureless surface morphology, but very thin and non-adhesive films, so compromising the polymer's ability to protect substrates from corrosion.

iv) At higher temperatures (40˚C and 60˚C), very high electric current densities of oxidation peaks result in the hyper-oxidation of monomer with porous nonuniform surface morphology, thus reducing its electrical conductivity and corrosion protection performance.

v) However, at AT (27˚C), the surface morphology is uniform and featureless, as current densities of the peaks are intermediate, showing better corrosion protection characteristics than other reaction temperatures with a positive corrosion potential (E_{corr}) shift of 381 mV and also a reduction in CR of 50 times as compared to uncoated LCS.

5. Conclusions

The sodium tartrate is a convenient supporting electrolyte for the ECP of aniline with 2-methylaniline.

The Tafel plot measurements reveal that the PAMA copolymer coatings effectively protect the LCS compared to the corresponding homopolymers PANI and PMA.

The PAMA coatings were successfully synthesized by cyclic voltammetry at different reaction temperatures.

The CV and SEM results show that at higher temperatures (40˚C & 60˚C), the quality of PAMA coatings degrades because of hyperoxidation of monomer.

Nevertheless, at lower temperatures, it doesn't show any oxidation or reduction performance, and very thin and non-adhesive films.

The corrosion-resistant characteristics of the resulting coating were evaluated by Tafel plot in 3% NaCl.

The Tafel plot results unveil that the PAMA coatings deposited at 27˚C (ambient temperature) effectively protect LCS against corrosion than the other reaction temperatures in 3% NaCl.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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