



REGULAR ARTICLE

Impact of Bath Temperature on the Morphology, Composition, and Corrosion Resistance of Cerium Oxide Coatings Electrodeposited on Zinc

Farida Boucetta^{1,2,*}✉, Kamel Belmokre¹, Faïçal Djani³,
Mohammed-Amin Boumechraz⁴, Kamel Goudjil⁵

¹ Laboratory of Physico-Chemistry Research on Surfaces and Interfaces, University of 20 August 1955, Skikda 21000, Algeria

² Department of Chemistry, Faculty of Exact Sciences and Computer Science, University of Mohammed Seddik Benyahia, Jijel 18000, Algeria

³ Molecular Chemistry and Environment Laboratory, Mohammed Khider University Biskra, 07000, Algeria

⁴ Laboratory of Research in Civil Engineering, Mohammed Khider University of Biskra, 07000, Algeria

⁵ Department of Civil Engineering, Mohamed-Cherif Messaadia University of Souk Ahras, 41000, Algeria

(Received 12 November 2024; revised manuscript received 18 February 2025; published online 27 February 2025)

This study investigates the influence of bath temperature on the electrodeposition of cerium oxide (CeO₂) coatings on zinc substrates. The research focuses on how temperature affects the formation, morphology, composition, and corrosion resistance of the deposited films. To analyze these effects, a combination of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), electrochemical impedance spectroscopy (EIS), and potentiodynamic polarization was employed. The findings reveal that the most corrosion-resistant coatings were obtained when the electrodeposition process was conducted at temperatures between 20 °C and 30 °C. Increasing the bath temperature led to significant modifications in coating adhesion and surface morphology, with higher temperatures promoting irregular growth patterns. EDS analysis indicated that as the temperature increased, the deposited layers exhibited a higher Zn content and a lower Ce content, suggesting a temperature-dependent variation in the deposition mechanism. Electrochemical tests confirmed that coatings formed at room temperature provided optimal protection against corrosion, demonstrating improved barrier properties. These results highlight the importance of temperature control in the electrodeposition process to enhance the structural integrity and protective performance of CeO₂ coatings, making them suitable for corrosion-resistant applications in various industries.

Keywords: CeO₂ thin film, Electrodeposition, EIS, Corrosion, Temperature.

DOI: [10.21272/jnep.17\(1\).01018](https://doi.org/10.21272/jnep.17(1).01018)

PACS numbers: 81.15.Pg, 81.65.Kn,
81.05.Rm, 82.45.Bb

1. INTRODUCTION

Cerium oxides have recently gained significant attention due to their exceptional ability to inhibit various metals [1], their applications in solid oxide fuel cells [2], and their use as high-temperature anticorrosion and thermal barriers [3]. CeO₂, a key N-type semiconductor, is notable for its high electrical conductivity, its remarkable capacity to absorb and release oxygen, and its robust redox properties [4]. Additionally, CeO₂-based materials are essential in three-way catalysts for vehicle exhaust purification [5], and are also involved in processes such as the water-gas shift reaction [6], and hydrocarbon reforming [7]. This feature makes CeO₂ based materials crucial for enhancing the performance and longevity of catalysts [8]. In particular, during heterogeneous catalytic reactions, they are effective in managing variations in oxygen concentration, thereby ensuring optimal catalytic efficiency [9].

There are various methods for synthesizing CeO₂

films, including chemical vapor deposition (CVD) [10], thermal evaporation [11], and electrochemical deposition [12]. Among these techniques, electrodeposition has recently attracted significant interest due to its affordability and effectiveness [13]. This approach typically employs two main modes: potentiostatic and galvanostatic [14]. The morphology of CeO₂ films can be tailored by modifying various experimental parameters, including electrolyte concentration, deposition potential, current density, deposition time, and bath temperature [12]. Of all these parameters, temperature plays a particularly critical role in determining the structure and performance of the resulting CeO₂ nanofilms [15].

In this study, we deposited high corrosion-resistant cerium oxide coatings on a zinc plate using a cerium nitrate electrolyte. The aim was to investigate the effect of temperature on the electroplating process, as well as to analyze the morphology, composition, and electrochemical behavior of the CeO₂ coatings.

* Correspondence e-mail: farida.boucetta@univ-jijel.dz



2. EXPERIMENTAL PROCEDURE

Cerium oxide films were developed on a zinc substrate using the electrodeposition technique in galvanostatic mode. Initially, the zinc substrates were masked with an acid- and alkali-resistant tape, leaving only a 10 mm × 10 mm exposed area. The electrolyte used for cerium film deposition was a 0.01 M solution of Ce(NO₃)₆H₂O. A three-electrode system was employed for the electrodeposition, and the experiments were conducted using a Potentiostat/Galvanostat electrochemical workstation, Solartron SI 1287 model. The zinc plate served as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum (Pt) wire as the counter-electrode. Electrodeposition was carried out at temperatures of 20, 30, 40, and 50 °C, with a deposition duration of 20 minutes. The applied current density was set at -2 A.dm^{-2} . After deposition, the samples were rinsed with ethanol and stored in desiccators at a temperature of $22 \text{ °C} \pm \text{ °C}$ until further analysis.

Electrochemical tests, including open circuit potential (OCP) measurement, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS), were performed using a potentiostat/galvanostat (OrigaFlex OGF 500) in a 3.5 wt. % NaCl solution. Before conducting the polarization curves and EIS tests, the samples were immersed in the corrosive solution for 30 minutes to stabilize the system. EIS was performed by applying a sinusoidal signal of $\pm 10 \text{ mV}$ around the OCP, with a frequency range from 0.01 to 100.000 Hz. The scan rate for the potentiodynamic polarization curve was 1 mV/s, and the polarization range covered $\pm 250 \text{ mV}$ relative to the OCP.

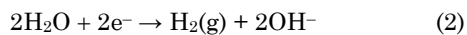
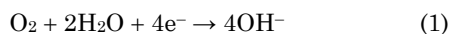
The morphology of the CeO₂ films was examined using a scanning electron microscope (SEM, TESCAN-VEGA3) equipped with an energy-dispersive X-ray spectroscope (EDS).

3. RESULTS AND DISCUSSION

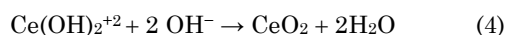
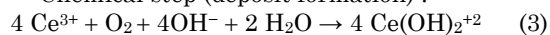
3.1 Elaboration of Cerium Oxide Films

The deposition of cerium oxide on the cathode surface from a nitrate medium can be explained by a two-step electrochemical mechanism as follows [1, 2]:

- Electrochemical step (base electrogeneration at the cathode surface):



- Chemical step (deposit formation) :



The electrochemical reactions (Eqs. 1 and 2) lead to a local increase in pH at the cathode surface, a process known as electrogeneration of base [3]. As the OH⁻ concentration rises to the required level for the chemical reactions (Eqs. 3 and 4), cerium oxide forms and deposits on the cathode. The evolution of hydrogen during this process can lead to a unique porous structure and preferential crystal growth in a specific direction [4].

3.2 Characterisation of Cerium Oxide Films

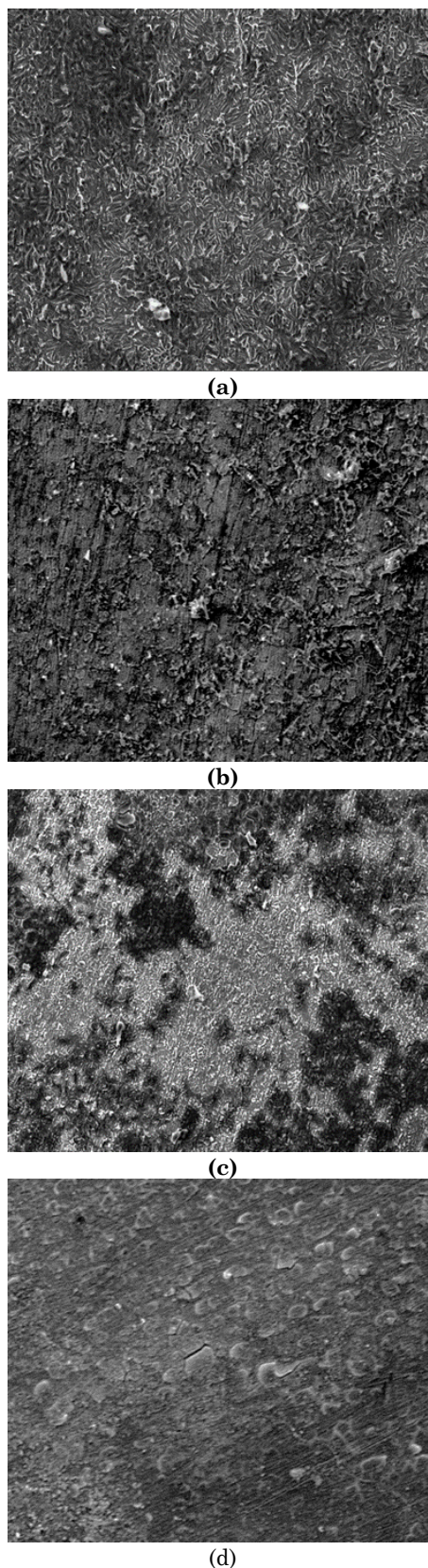


Fig. 1 – SEM micrographs (50 μm) of zinc samples coated at different temperature in 0.01 M Cerium (III) solution: (a) 20 °C, (b) 30 °C, (c) 40 °C, and (d) 50 °C.

Table 1 – EDS analysis of cerium conversion coatings obtained after different immersion temperatures

Temperature (°C)	Weight % of Zn	Weight% of Ce	Weight% of O
20	15.06	54.57	30.37
30	24.10	51.67	24.24
40	31.41	32.62	35.96
50	51.54	12.97	35.48

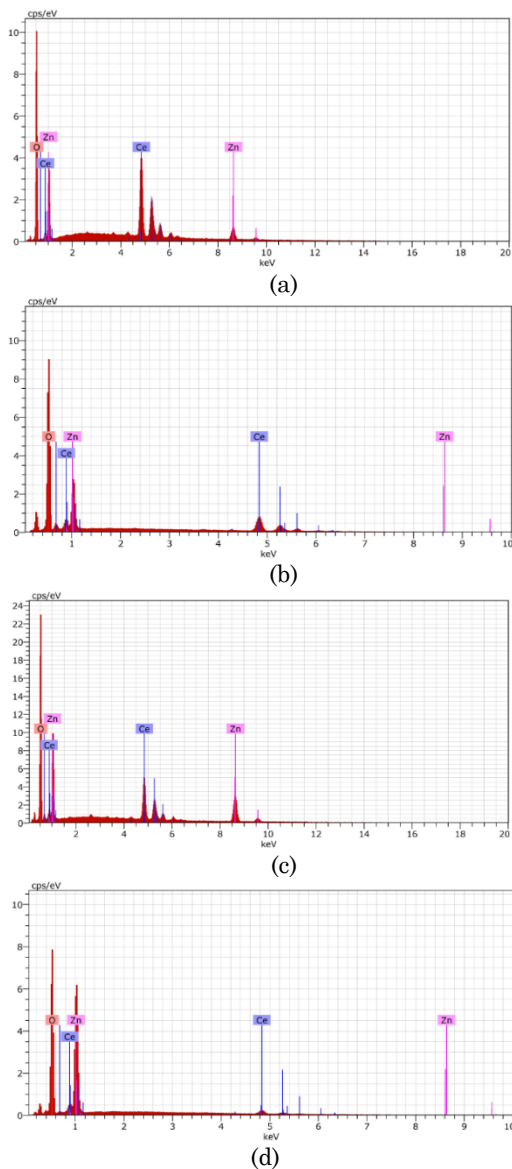


Fig. 2 – EDS analysis of zinc samples coated at different immersion temperature in 0.01 M Ce(III) solution: (a) 20 °C, (b) 30 °C, (c) 40 °C, and (d) 50 °C

The surface characterization of cerium coatings on zinc revealed a significant influence of deposition temperature on morphology and composition. SEM analysis (Fig. 1) showed that at 20 °C and 30 °C, the surface appeared uniform with small dispersed particles, promoting better coverage and adhesion, while at 40 °C and 50 °C, coarser structures and defects emerged, likely due to excessive growth or partial dissolution. Higher temperatures accelerated secondary reactions, compromising coating quality. EDX analysis (Fig. 2, Table 1) indicated that cerium content was highest at 20 °C (54.57 wt %) and

decreased with increasing temperature (12.97 wt % at 50 °C), while zinc exposure increased, suggesting a decline in coating density. Oxygen levels remained relatively stable (24.24 %-35.96 %), likely due to oxidation or intrinsic oxide content. Structurally, low-temperature coatings (20 °C) were thicker and more uniform, providing better surface coverage, whereas higher temperatures led to more porous, thinner deposits, potentially altering electrodeposition or precipitation mechanisms. In terms of corrosion protection, lower temperatures favored dense and homogeneous coatings, while higher temperatures (50 °C) resulted in increased zinc exposure, reducing the protective barrier’s effectiveness. Overall, 20 °C appears optimal for achieving thick, dense cerium oxide coatings essential for corrosion protection applications.

3.3 Corrosion Behaviour of Cerium Oxide Films Deposited

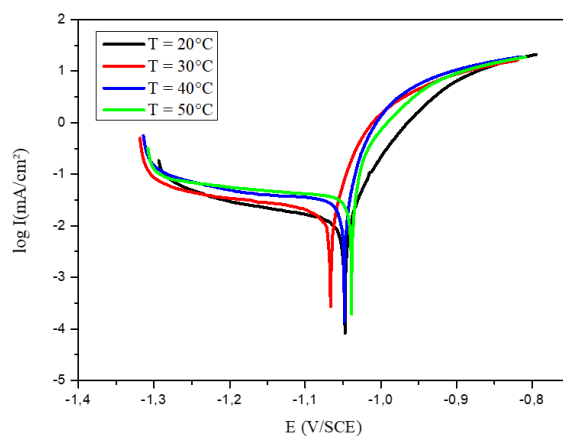


Fig. 3 – Potentiodynamic polarization curves of electrodeposited coatings in 3,5 % NaCl solution. The coatings were prepared from 0.01 M Ce(III) solution at different temperatures

Table 2 – Polarization parameters of electrodeposited coatings in 3.5 % NaCl solution

Temperature (°C)	E_{corr} (mV)	R_p ($\Omega \cdot cm^2$)	I_{corr} ($mA \cdot cm^{-2}$)
20	-1047.7	953.33	0.0058
30	-1066.5	513.99	0.0131
40	-1048.2	379.63	0.0169
50	-1038.7	261.05	0.0361

Fig. 3 displays the polarization curves for zinc electrodes featuring cerium oxide coatings immersed for 20 minutes in a cerium bath at various temperatures and the corresponding corrosion parameters, which can be found in Table 2. Notably, the results indicate that as the temperature is raised from 20 to 50 °C, both I_{corr} (corrosion current) and the corrosion rate of the coated samples increase. The cerium oxide coatings prepared at 20 °C and 30 °C exhibit the highest polarization resistances and the lowest corrosion current densities. This can be attributed to the compact structure of the coating at these temperatures.

3.4 EIS Measurement

Fig. 4 presents Nyquist plots at open circuit potential (OCP) for cerium conversion coatings obtained by

applying a current density of -2 A/dm^2 in a cerium bath at different temperatures. The actual capacitances were then calculated from the respective CPE parameters. In the equivalent circuit shown in Fig. 5, R_s represents the electrolyte resistance; CPE_c and R_c correspond to the coating capacitance and resistance, respectively; CPE_{dl} represents the electrochemical double-layer capacitance at the metal/electrolyte interface; and R_{ct} is the metal's charge transfer resistance [5]. The experimental results are presented in Table 3 for zinc coated with cerium at various operating temperatures. It is evident that higher temperatures lead to a reduction in corrosion resistance. This decrease is linked to the increased dissolution rate of zinc at elevated temperatures in the cerium bath. As the corrosion rate rises, fewer active sites remain available on the zinc surface for cerium oxide deposition, which confirms the result of polarization.

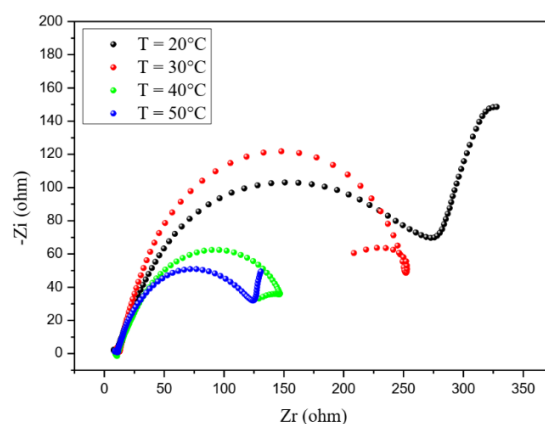


Fig. 4 – Nyquist plots curves of electrodeposited coatings in 3.5 % NaCl solution. The coatings were prepared from 0.01 M Ce(III) solution at different temperatures

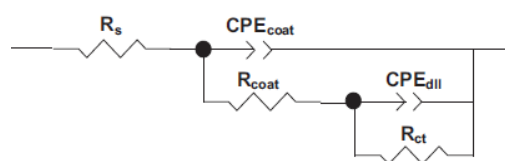


Fig. 5 – Equivalent circuits compatible with the experimental impedance data in Figure 4

Table 3 – Fitting results of EIS data for coated substrate in 0.5 M NaCl solution

Temperature (°C)	R_s ($\omega \cdot \text{cm}^2$)	$CPE_c \cdot 10^{-3}$ ($\omega^{-1} \cdot \text{cm}^2 \cdot \text{s}^n$)	n_1	R_c ($\omega \cdot \text{cm}^2$)	$CPE_{dl} \cdot 10^{-6}$ ($\omega^{-1} \cdot \text{cm}^2 \cdot \text{s}^n$)	n_2	R_{ct} ($\omega \cdot \text{cm}^2$)
20	11.90	0.0194	0.76	346.6	0.104	0.87	407.12
30	11.77	0.0819	0.87	208.3	7.779	0.89	263.60
40	10.65	0.1304	0.80	132.7	8.959	0.41	162.3
50	9.319	0.1723	0.88	115.3	24.295	0.76	159.4

4. CONCLUSION

The electrodeposition of cerium oxide thin films at varying bath temperatures demonstrated a significant impact on the structural and electrochemical properties of the coatings. The study revealed that room-temperature deposition yielded a uniform, dense, and crack-free CeO_2 layer with a characteristic matte yellow appearance. Notably, the cerium content in the coatings decreased with increasing bath temperature, affecting their composition and protective properties.

Electrochemical evaluations confirmed that the coating deposited at room temperature exhibited the lowest i_{corr} and highest R_p , highlighting its superior corrosion resistance. These findings emphasize the critical role of temperature control in optimizing coating performance. The enhanced stability and protective capabilities of these cerium oxide films suggest their potential application in corrosion-prone environments, particularly in marine and industrial sectors, where prolonged material durability is essential.

REFERENCES

- E. González, R. Stuhr, J.M. Vega, E. García-Lecina, H.-J. Grande, J.R. Leiza, M. Paulis, *Polymers* **13** No 6, 848 (2021).
- M. Zhao, S. Geng, G. Chen, F. Wang, *Int. J. Hydrogen Energy* **50**, 1087 (2024).
- P. Rajendran, A. Muthuraj, N.E. Rajagounder, *Trans. Indian Ceram. Soc.* **81** No 4, 158 (2022).
- X. Meng, C. Wan, X. Jiang, X. Ju, *J. Mater. Sci.* **53**, 13966 (2018).
- B. Asif, M. Zeeshan, S. Iftexhar, M. Sillanpää, *Atmospheric Pollution Res.* **12** No 7, 101088 (2021).
- T. Shido, Y. Iwasawa, *J. Catal.* **136** No 2, 493 (1992).
- S. Sepahri, M. Rezaei, Y. Wang, A. Younesi, H. Arandian, *Int. J. Hydrogen Energy* **43** No 49, 22340 (2018).
- J. Wang, X. Xiao, Y. Liu, K. Pan, H. Pang, S. Wei, *J. Mater. Chem. A* **7** No 30, 17675 (2019).
- X.Z. Song, W.Y. Zhu, X.F. Wang, Z. Tan, *ChemElectroChem* **8** No 6, 996 (2021).
- J. Schlafer, D. Graf, G. Fornalczyk, A. Mettenborger, S. Mathur, *Inorgan. Chem.* **55** No 11, 5422 (2016).
- T. Brezesinski, M. Antonietti, M. Groenewolt, N. Pinna, B. Smarsly, *New J. Chem.* **29** No 1, 237 (2005).
- B. Assaker, Z. Landolsi, I. Chérif, R. Marzouki, S.M. Keshk, R. Chtourou, *Chem. Africa* **7**, 2195 (2024).
- L. Yang, X. Pang, G. Fox-Rabinovich, S. Veldhuis, I. Zhitomirsky, *Surf. Coat. Technol.* **206** No 1, 1 (2011).
- S.F.U. Farhad, M.M. Hossain, M.S. Bashar, N.I. Tanvir, S. Islam, *ECS Adv.* **3** No 1, 013001 (2024).
- N. Ahmadizadeh, P. Najafisayar, *Ceram. Int.* **46** No 11, 19583 (2020).
- A. Aldykiewicz, A. Davenport, H. Isaacs, *J. Electrochem. Soc.* **143** No 1, 147 (1996).

16. F.B. Li, G. Thompson, *J. Electrochem. Soc.* **146** No 5, 1809 (1999).
17. Y. Hamlaoui, L. Tifouti, C. Remazeilles, F. Pedraza, *Mater. Chem. Phys.* **120** No 1, 172 (2010).
18. L. Zu, J. He, X. Liu, L. Zhang, K. Zhou, *Int. J. Hydrogen Energy* **44** No 10, 4650 (2019).
19. O. Gharbi, M.T. Tran, B. Tribollet, M. Turmine, V. Vivier, *Electrochimica Acta* **343**, 136109 (2020).

Вплив температури ванни на морфологію, склад і корозійну стійкість покриттів з оксиду церію, електроосаджених на цинк

Farida Boucetta^{1,2}, Kamel Belmokre¹, Faïçal Djani³, Mohammed-Amin Boumehraz⁴, Kamel Goudjil⁵

¹ *Laboratory of Physico-Chemistry Research on Surfaces and Interfaces, University of 20 August 1955, Skikda 21000, Algeria*

² *Department of Chemistry, Faculty of Exact Sciences and Computer Science, University of Mohammed Seddik Benyahia, Jijel 18000, Algeria*

³ *Molecular Chemistry and Environment Laboratory, Mohammed Khider, University Biskra, 07000, Algeria*

⁴ *Laboratory of Research in Civil Engineering, Mohammed Khider, University of Souk Ahras, 41000, Algeria*

У цьому дослідженні досліджується вплив температури ванни на електроосадження покриттів з оксиду церію (CeO_2) на цинкових підкладках. Дослідження зосереджено на тому, як температура впливає на формування, морфологію, склад і стійкість до корозії нанесених плівок. Для аналізу цих ефектів була застосована комбінація скануючої електронної мікроскопії (SEM), енергодисперсійної спектроскопії (EDS), електрохімічної імпедансної спектроскопії (EIS) і потенціодинамічної поляризації. Результати показують, що найбільш стійкі до корозії покриття були отримані, коли процес електроосадження проводився при температурах від 20 °C до 30 °C. Підвищення температури ванни призвело до значних змін в адгезії покриття та морфології поверхні, при цьому більш високі температури сприяли нерівномірним моделям росту. Аналіз EDS показав, що зі збільшенням температури нанесені шари демонструють вищий вміст Zn і нижчий вміст Ce, що свідчить про залежну від температури зміну механізму осадження. Електрохімічні випробування підтвердили, що покриття, сформовані при кімнатній температурі, забезпечують оптимальний захист від корозії, демонструючи покращені бар'єрні властивості. Ці результати підкреслюють важливість контролю температури в процесі електроосадження для підвищення структурної цілісності та захисних характеристик покриттів CeO_2 , що робить їх придатними для корозійностійких застосувань у різних галузях промисловості.

Ключові слова: Тонка плівка CeO_2 , Електроосадження, EIS, Корозія, Температура.