Characterization of Zinc Phosphate Coatings: Influence of the pH and temperature in morphology and corrosion resistance

Sheila Silva-Fernández, Belén Díaz and X. Ramón Nóvoa
CINTECX, Universidad de Vigo, ENCOMAT group, Campus universitario As Lagoas Marcosende, 36310 Vigo, Spain, belenchi@vigo.es, rnovoa@uvigo.es
*Correspondence: shsilva@uvigo.es

UNIVERSIDADE DE VIGO

1. INTRODUCTION

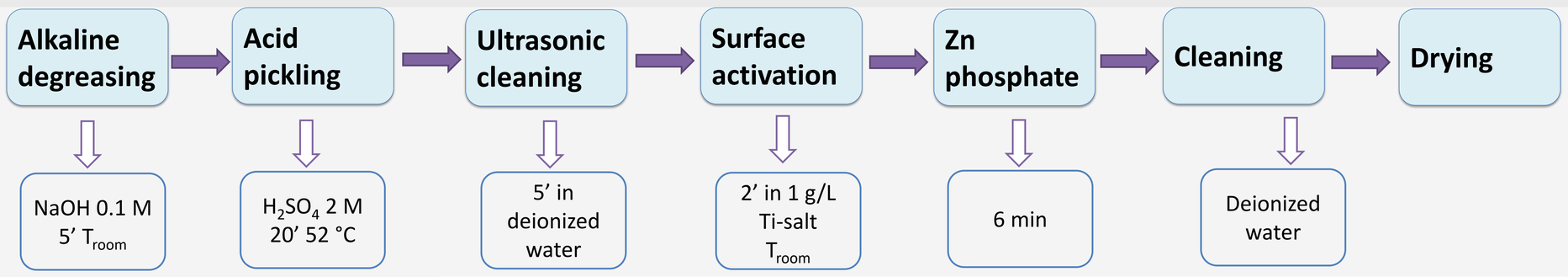
Zinc phosphate coatings are commonly used to improve the corrosion protection of steel rods. The pH value and the temperature of the bath are important parameters for improving the phosphates' properties. The large variety of bath conditions allows obtaining coatings within different properties and characteristics that can cover a large range of applications. In this work, temperatures in the range 50-75 °C and pH values between 2.4 and 3 are studied.

The coating weight was obtained by measuring the weight loss after immersion for 5 min at 70 °C in solution with Na_4EDTA 12 %, NaOH 9 %, and TEA 4 %. The surface morphology and composition were investigated via scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX). Corrosion resistance was evaluated with the electrochemical impedance spectroscopy (EIS) and voltammetry (LSV) techniques. The electrolyte was 0.1 M Na_2SO_4 solution.

1. 1 Substrate

Hot-rolled high-strength steel bars, 11 mm diameter, ≈ 20 mm long

1. 2 Experimental method



- Phosphating bath composition (for 1 L)

H_3PO_4 (85 wt. %)	ZnO	$NaNO_3$	$NaNO_2$
8.6 mL	3.5 g	3 g	0.1 g

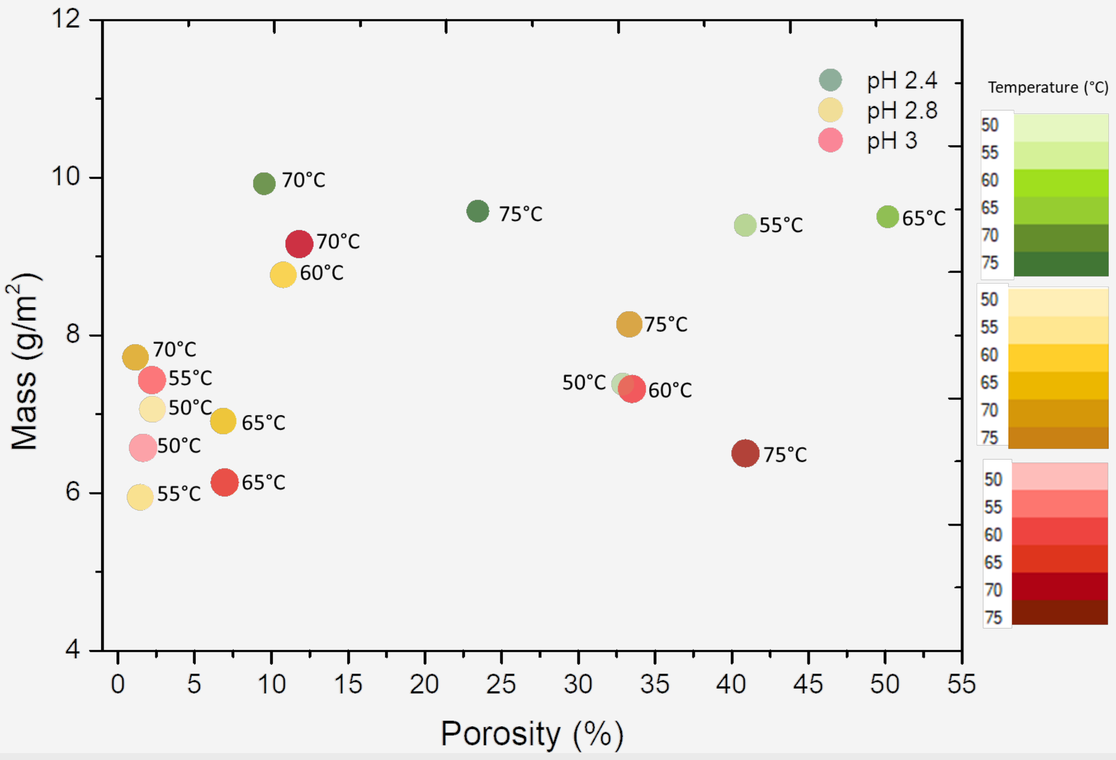
75 °C, 70 °C, 65 °C, 60 °C, 55 °C, 50 °C

pH 2.4, pH 2.8, pH 3

- pH adjusted with NaOH 1 M

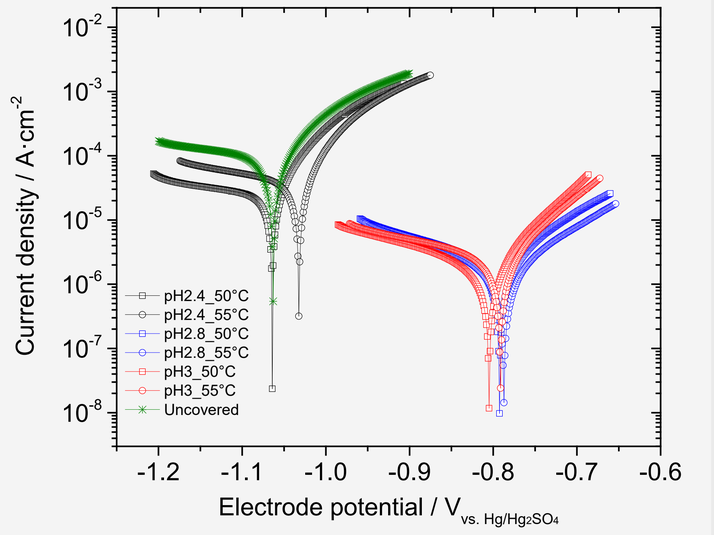
2. RESULTS

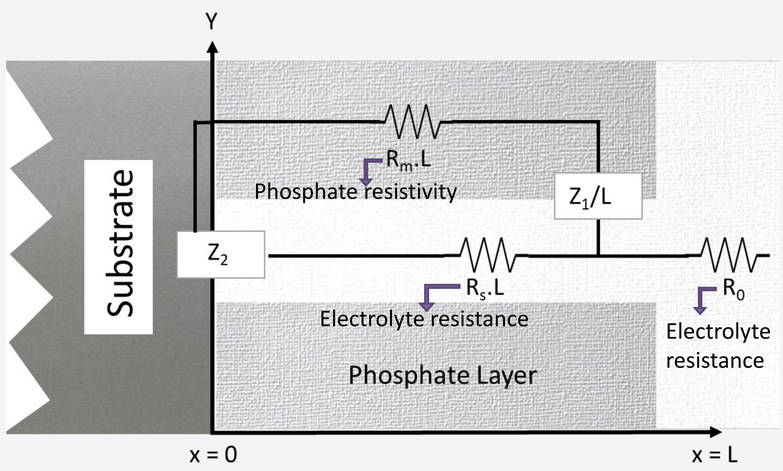
Influence of the pH and temperature on mass and film porosity



- The lower the pH, the higher the coating weight
- In general, higher pH values lead to lower porosity values

$$\% \text{ Porosity} = \frac{i_{corr\ phosphated}}{i_{corr\ uncovered}} \cdot 100$$

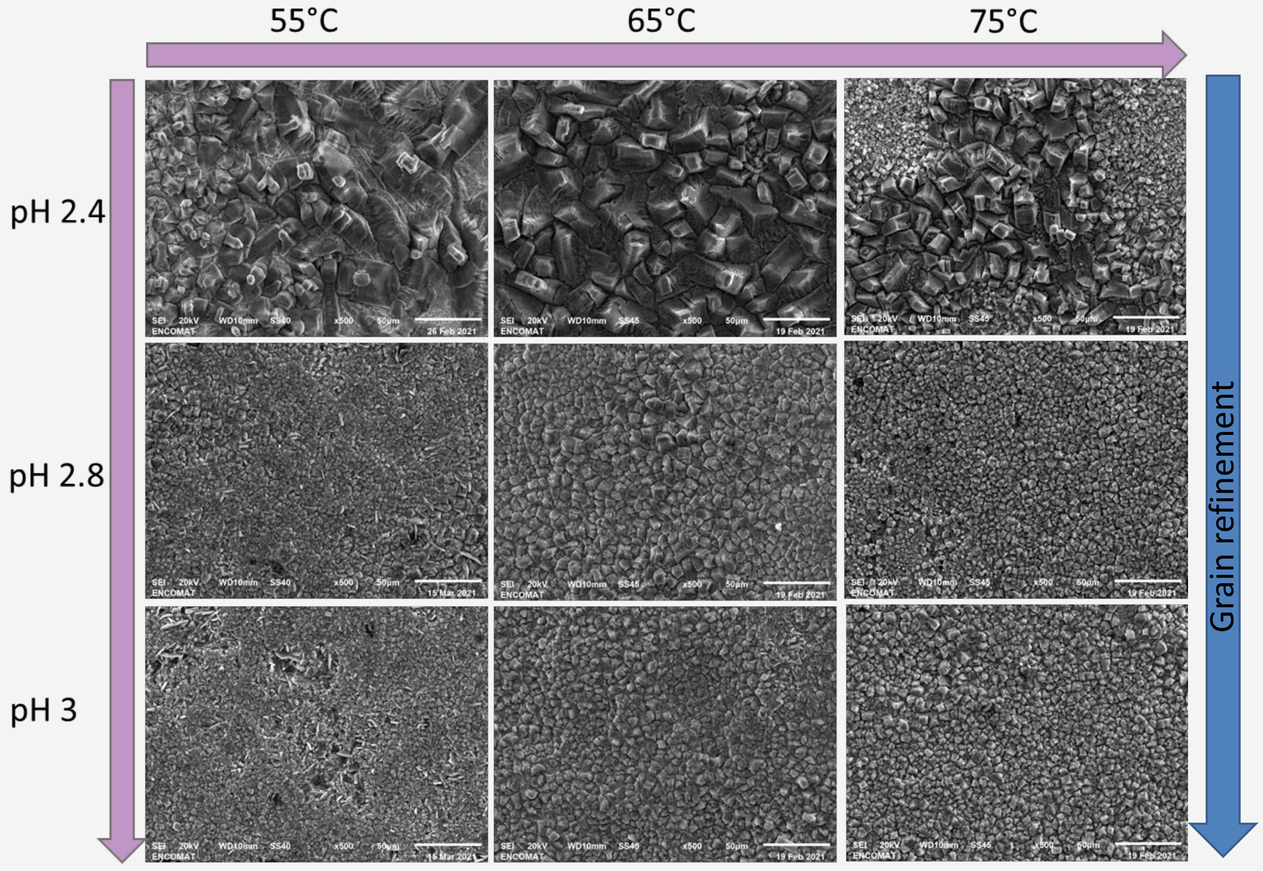




	pH 2.4	pH 3
55 °C	R_m: 67.6 kΩ.cm R_s: 82.5 kΩ.cm R_2: 440 Ω.cm²	R_m: 114 kΩ.cm R_s: 49.5 kΩ.cm R_2: 42.8 kΩ.cm²
75 °C	R_m: 316 kΩ.cm R_s: 232 kΩ.cm R_2: 1.3 kΩ.cm²	R_m: 86.6 Ω.cm R_s: 118 kΩ.cm R_2: 498 Ω.cm²

- At low temperature, when the pH increases:
↑ R_2: ↓ porosity
↑ R_m: ↑ resistivity (↓ Zn/Fe)
↑ R_s: crystal refinement

- At high temperature when the pH increases:
↓ R_2: ↑ porosity
↓ R_m: ↓ resistivity (↓ Zn/Fe but more porous)
↑ R_s: crystal refinement



Impedance spectroscopy allows obtaining the following properties:
- Porosity
- Coating resistivity
- Crystal size

Zn/Fe ratio

	50°C	55°C	60°C	65°C	70°C	75 °C
pH 2.4	0.74	0.85	0.91	1.06	0.98	0.94
pH 2.8	0.79	0.53	0.60	1.05	1.03	0.68
pH 3	0.73	0.51	0.63	0.69	0.70	0.70

The higher the pH, the lower the Zn/Fe ratio

The higher the temperature, the higher the Zn/Fe ratio (maximum at 65 °C)

3. CONCLUSIONS

The experimental methodology employed allows a complete coating characterization, including the porosity and significant parameters such as the film resistivity or the crystal size:

- The films grown at the higher pH values are less conductive and have a lower crystal size
- Thicker coatings with an increased Zn content are developed at the lowest pH
- According to the obtained data, in order to achieve a low porosity, temperature as high as 75 °C is not recommended

In a further study, the coating alkaline stability along with the wear resistance must be assessed to conclude on the above-referred findings.

4. REFERENCES

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(2) Abdalla, K.; Rahmat, A.; Azizan, A. The Effect of PH on Zinc Phosphate Coating Morphology and Its Corrosion Resistance on Mild Steel, Advanced Materials Research; 2013; Vol. 626, pp 569–574. https://doi.org/10.4028/www.scientific.net/AMR.626.569.