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A Study on the Performance Evaluation of Developed Epoxy-Based Coatings on Metallic Surfaces

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Presentation Outline:

1) Purpose
2) Originality & Value
3) Introduction
4) Materials and Methods
5) Results
6) Conclusion
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Purpose of the Study

•To delve into the impact of varying solid content ratios on the corrosion resistance of epoxy-based coatings.

•Going over tensiometric and rheological behaviors, while carrying out electrochemical impedance spectroscopy (EIS), neutral salt spray (NSS) and humidity tests within the framework of the study

Originality & Value

•Highlighting the impact of solid content percentage on key coating properties constitutes a novel aspect of this study.

•Supporting the formulation of advanced, high-performance epoxy coatings specifically designed for industrial use.

Introduction

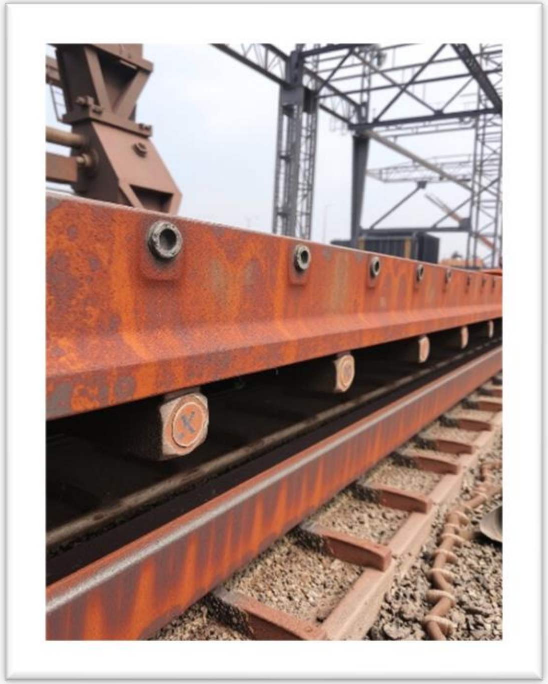
Epoxy Resins in Protective Coatings

•Significant advancements in epoxy resin syntheses

•Widely used due to cost efficiently, strong adhesion, chemical and corrosion resistance

•Applications: coatings, adhesives, electronics, composites

•Epoxy thermosets provide effective protection against corrosion



Corrosion of Carbon Steel (CS)

•CS is extensively used in infrastructure and industry

•Prone to corrosion in aggressive environments

•Coatings are the most efficient protection method

- Qian, M., Soutar, A.M., Tan, X.H. Zeng, X.T., and Wijesinghe, S.L. (2009), "Two-part epoxy-siloxane hybrid corrosion protection coatings for carbon steel", Thin *Solid Films*, Vol. 517, pp. 5237-5242.
- Hsissou, R., Hilali, M., Dagdag, O., Adder, F., Elbachiri, A., and Rafik, M. (2022), "Rheological behavior models of polymers", *Biointerface Research in Applied Chemistry*, Vol. 12, pp. 1263-1272.

Role of Zinc-Containing Epoxy Systems

- Provide cathodic protection & self-healing effects
- Zinc phosphate acts as eco-friendly inhibitive pigment
- Passivation mechanism by Zn^{2+} and PO_4^{3-} ions

Solvent-Free Organic Coatings

- Reduced VOC emissions; environmentally safer

This Study Focuses On

- Evaluating **corrosion resistance** of epoxy coatings with different solid contents
- Using **EIS**, **tensiometry**, and **rheology** to characterize formulations
- Performance is assessed before and after methanol immersion, with methanol serving as an aggressive medium to challenge the chemical resistance of the coating

❑ Kalendova, A., Vesely, D., Kohl, M., and Stejskal, J. (2015), "Anticorrosion efficiency of zinc-filled epoxy coatings containing conducting polymers and pigments", *Progress in Organic Coatings*, Vol. 78, pp. 1-20

Materials and Methods

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Materials Used in the Formulations

Epoxy Resins

- *EPOKUKDO YD-128*: Liquid, Bisphenol-A based (EEW: 184–190 g/eq; Viscosity: 11,500–13,500 cps at 25°C)
- *YDPN 638*: Semi-solid, phenol novolac multifunctional (EEW: 175–182 g/eq; Viscosity: 20,000–50,000 cps at 25°C)

Solvents

- n-Butanol (Ineos Oxide), IPA (LG Chemical, Merck)
- Xylene, Toluene (Gadiv)
- 1,4-Butanediol Diglycidyl Ether (Anhui Xinyuan), Benzyl Alcohol, Butyl Acetate (BASF), Butyl Glycol (Dow)

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Additives & Agents

- Rheology agents (HS Chem, Arkema)
- Wetting agents (Lubrizol), Surface additives (BYK)

Fillers

- Talc (Imerys), Calcium Carbonate (Omya), Barium Sulfate (Baser), Zinc Phosphate (Akdeniz Chemson)

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Coating of carbon steel panels

•**Three formulations (A, B, C)** with different **filler and solvent ratios**; resin content constant.
•**Solid contents:** A – 55%, B – 80%, C – 100%
•Applied on **carbon steel (CS) panels** cleaned with acetone & IPA.
•**Application:** Air spray (30 s DIN4/20°C), cured 1h in oven
•**Dry film thickness:** 140–150 µm
•**All tests performed in triplicate** under identical conditions.

Protective features of coatings

•**Neutral Salt Spray (NSS) Test**
* According to **ISO 9227** and **ASTM B117-11**
*Panels scratched (1 mm × 100 mm) and exposed to **NaCl (50 g/L)** for **720 hours**
*Evaluated for **rusting** (ISO 4628-3) and **blistering** (ASTM D 714-02)

•**Humidity Resistance Test**
*Based on ASTM D714-02, ASTM 3359, ISO 4628
*Panels placed in a **climate chamber at 40°C** (opposite side at 25°C)
*Created **~100% condensation** on the test surface

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Electrochemical Impedance Spectroscopy (EIS)

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🔬 **System:** Gamry 600+ Potentiostat
Setup:
3-electrode system

- **Working:** Coated panel
- **Reference:** Ag/AgCl
- **Counter:** Carbon rod

💧 **Test Conditions:**
•Panels immersed in **methanol** for **1 week**
•Electrolyte: **5% NaCl** (pH 7)
•Exposed area: **16 cm²**

📈 **Measurements:**
•**OCP:** Measured for 40 min
•**Linear Polarization:** ±100 mV at 1 mV/s
•**EIS (Nyquist & Bode):** 10^{-2} to 10^{5} Hz, 20 mV AC

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Rheological Measurements

Device: Anton Paar MCR 102 Rheometer (PP25, 1 mm gap)
🌡️ **Temperatures:** 25°C and 40°C

🔬 **Test Methods**
Flow Curve (Shear Rate: 0.1–1000 s^{-1})
➤ Evaluates viscosity vs. shear rate over 10 min

Oscillatory Amplitude Sweep (0.01–100% strain at 10 Hz)

3-Interval Thixotropy Test (3ITT): (fotoalrı koy)
* 1 s^{-1} for 100 s → paint loading
* 100 s^{-1} for 100 s → brushing/rolling
* 1 s^{-1} for 100 s → drying phase

Tensiometry Measurements

Device: Attension Theta (Biolin Scientific)

📏 **Techniques Used:**

•**Sessile drop method** (for contact angle measurement)
•**Pendant drop method** (for surface & interfacial tension)

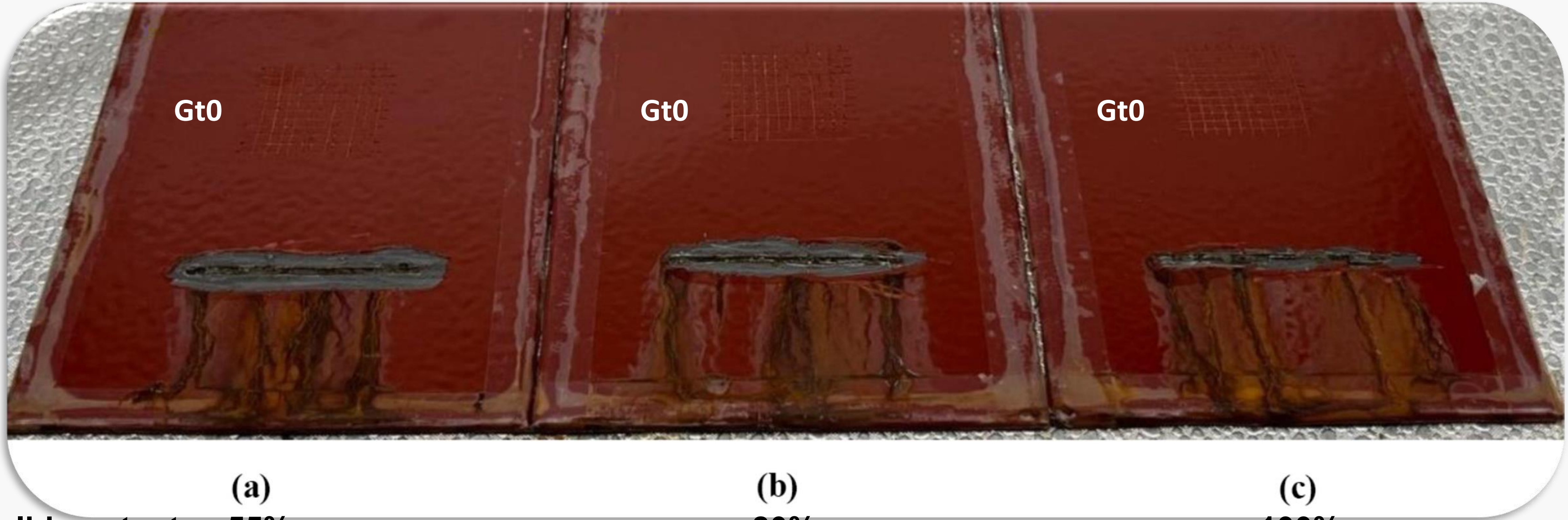
➡️ Calculations done using **Theta Software**

🔁 **Each test repeated 3 times** for reproducibility

Result and Discussion

Neutral salt spray and humidity test

Figure 1: The images of a) panel A, b) panel B and c) panel C after 720 h of exposure to NSS



Solid contents: 55% 80% 100%

- Yan, H., Zeng, D., Shi, L., and Zhang, L. (2024), "Multifunctional Ti3C2Tx-based epoxy composite coating towards intelligent response and corrosion/wear resistance", *Progress in Organic Coatings*, Vol. 196, pp. 108746.

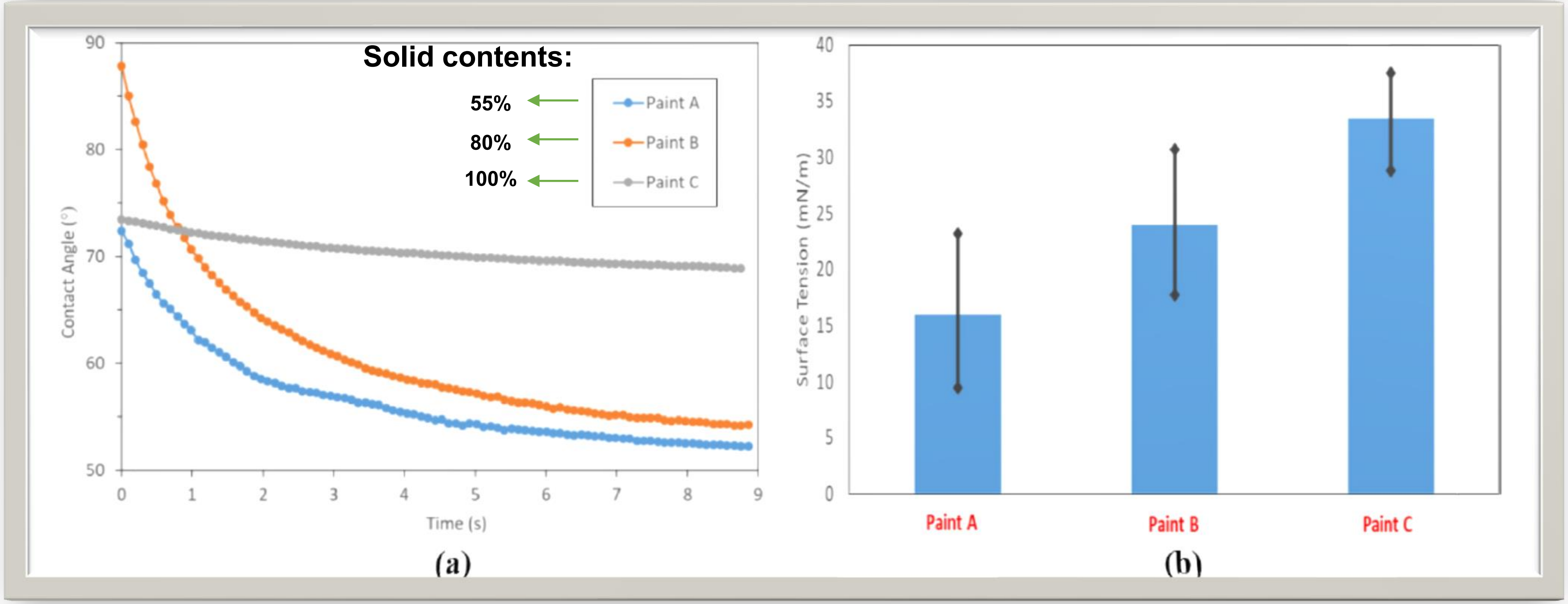
Table 1: Results of NSS and humidity tests according to ISO 12944-6

Evaluation methods (ISO 12944-6)	Accept conditions	Evaluation Panel A	Panel B	Panel C
Corrosion test (neutral salt spray), ISO 12944-6, 720 h				
ISO 4628-2 blistering	0(S0)	0(S0)	0(S0)	0(S0)
ISO 4628-3 Corrosion	Ri 0	Ri 0	Ri 0	Ri 0
ISO 4628-4 Cracking	0(S0)	0(S0)	0(S0)	0(S0)
ISO 4628-5 flaking	0(S0)	0(S0)	0(S0)	0(S0)
Line edge corrosion (mm)	$<$ 1.5 mm	0.5	0.5	0.5
Humidity test (ISO 6270-1) 480 h				
ISO 4628-2 blistering	0(S0)	0(S0)	0(S0)	0(S0)
ISO 4628-3 Corrosion	Ri 0	Ri 0	Ri 0	Ri 0
ISO 4628-4 Cracking	0(S0)	0(S0)	0(S0)	0(S0)
ISO 4628-5 flaking	0(S0)	0(S0)	0(S0)	0(S0)
ISO 2409- Cross Cut		Gt0	Gt0	Gt0

- Yan, H., Zeng, D., Shi, L., and Zhang, L. (2024), "Multifunctional Ti3C2Tx-based epoxy composite coating towards intelligent response and corrosion/wear resistance", *Progress in Organic Coatings*, Vol. 196, pp. 108746

Tensiometry

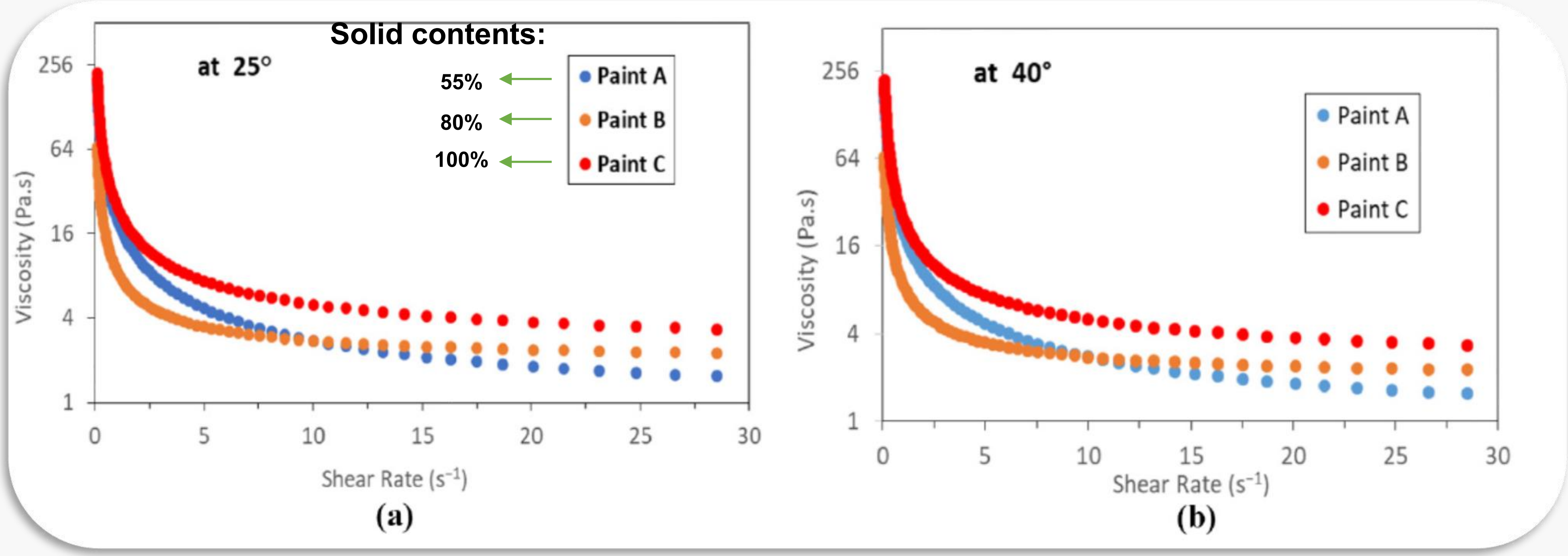
Figure 2: Contact angle (a) and surface tensions (b) results of paint formulations



❑ Crowley, S.V., Burlot, E., Silva, J.V.C., McCarthy, N.A., Heni, B., Wijayanti, A.M., Alan, L.F. and Kelly, A.O. (2018), "Rehydration behaviour of spray-dried micellar casein concentrates produced using microfiltration of skim milk at cold or warm tem

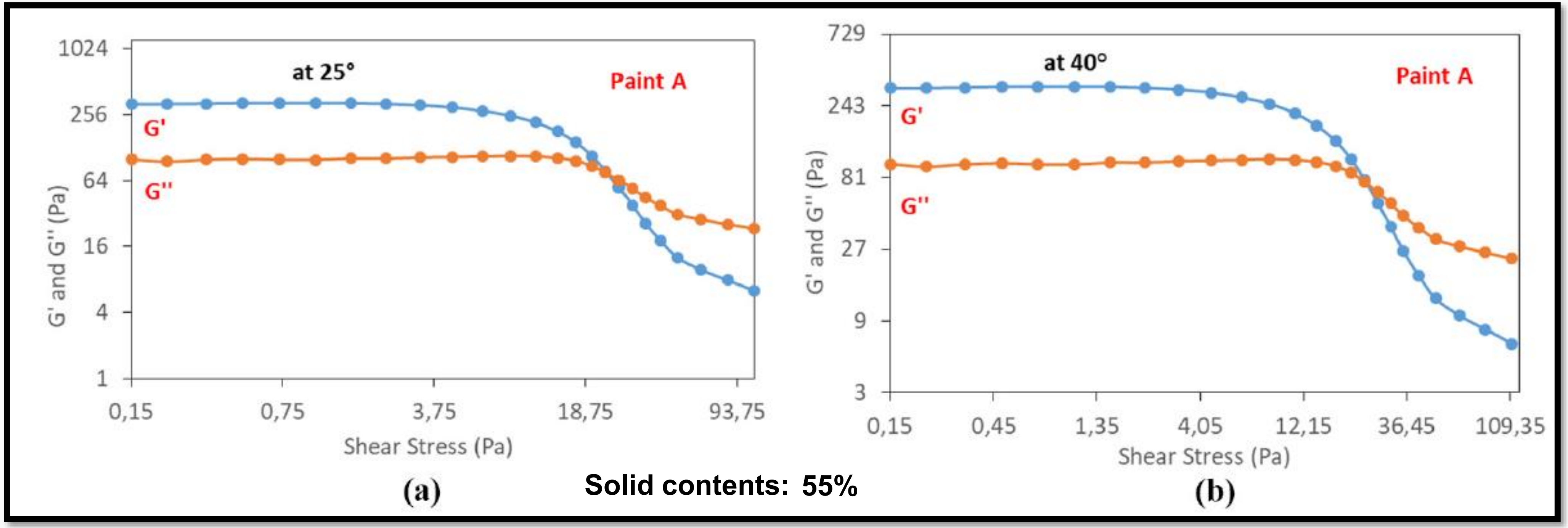
Rheometter

Figure 3: Viscosity of paints with varying solid ratios as a function of shear rate at 25°C and 40°C

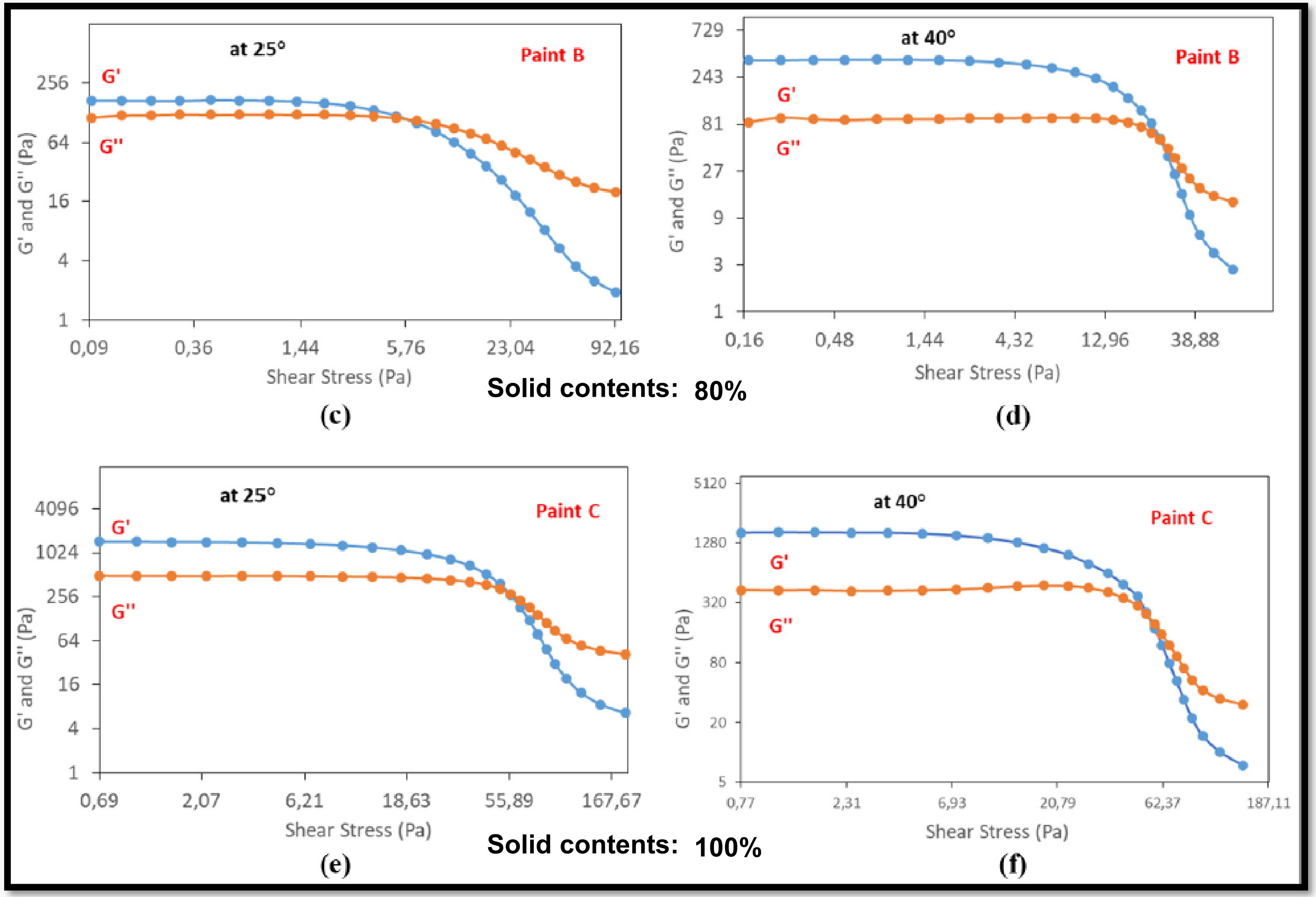


❑ Wang, C.S., Chapelle, G., Carreau, P. and Heuzey, M.C. (2021), "Prediction of sag resistance in paints using rheological measurements", Progress in Organic Coatings, Vol. 153, p. 106139.

Figure 4: The results of the oscillatory amplitude sweep test for the paint formulations were obtained at 25°C and 40°C

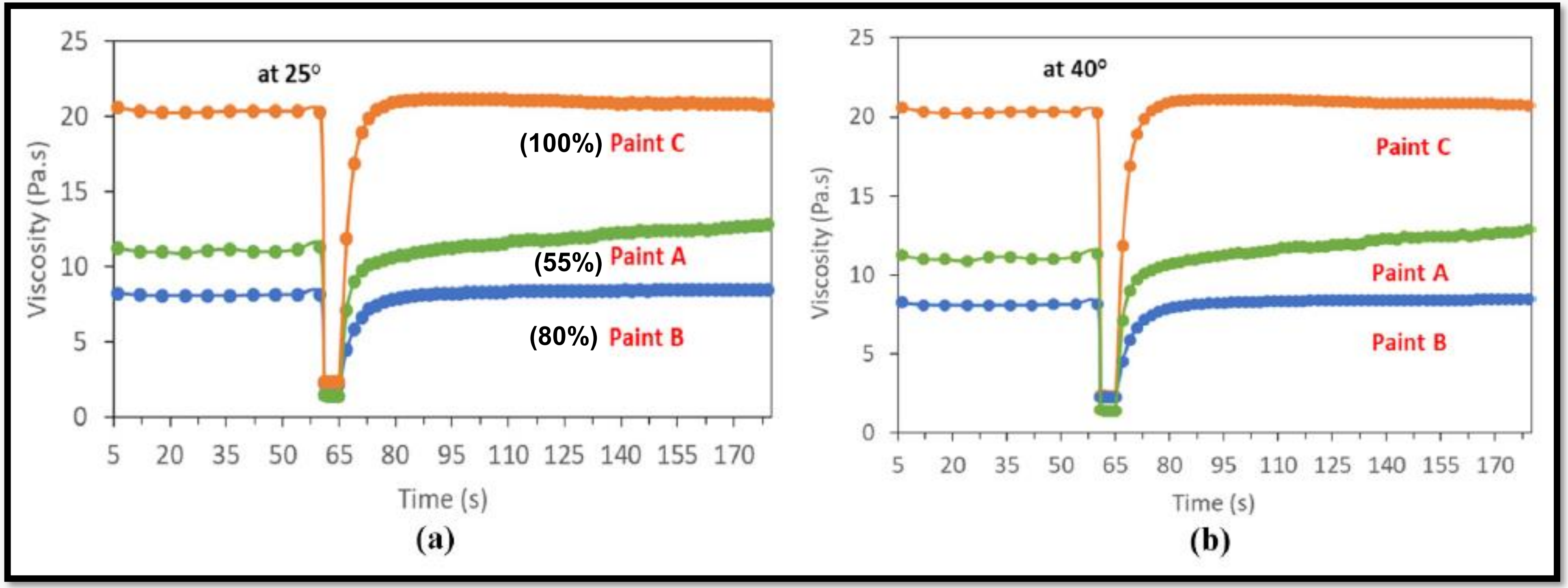


- Haramagatti, C.R., Nikam, P., Bhavsar, R., Kamath, V. And Sawant, V.S. (2020), "Stability assessment of iron oxide yellow pigment dispersions and temperature dependent implications of rheological measurements", Progress in Organic Coatings, Vol. 144, p. 105669.



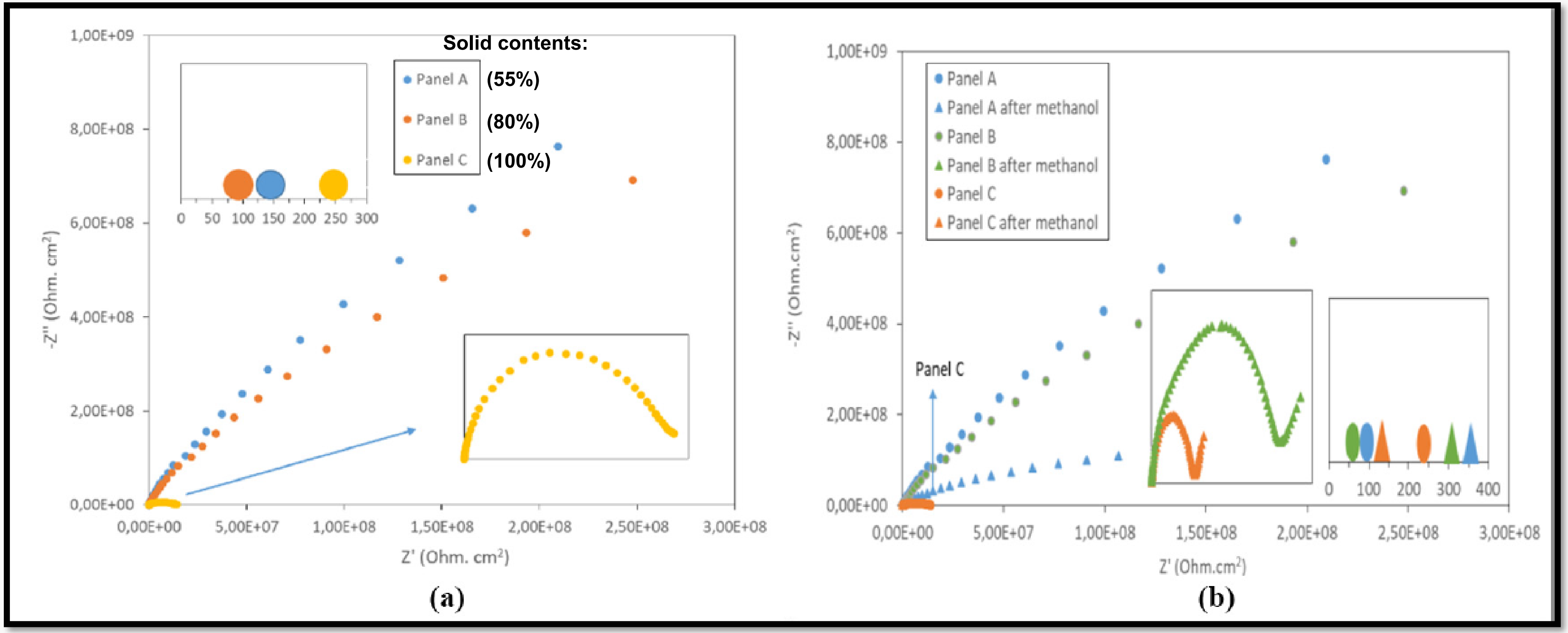
- Franchini, E., Galy, J. and Gerard, J.F. (2009), "Sepiolitebased epoxy nanocomposites: relation between processing, rheology, and morphology", Journal of Colloid and Interface Science, Vol. 329 No. 1, pp. 38-47.

Figure 5: The 3ITT curve for the paints at 25°C and 40°C

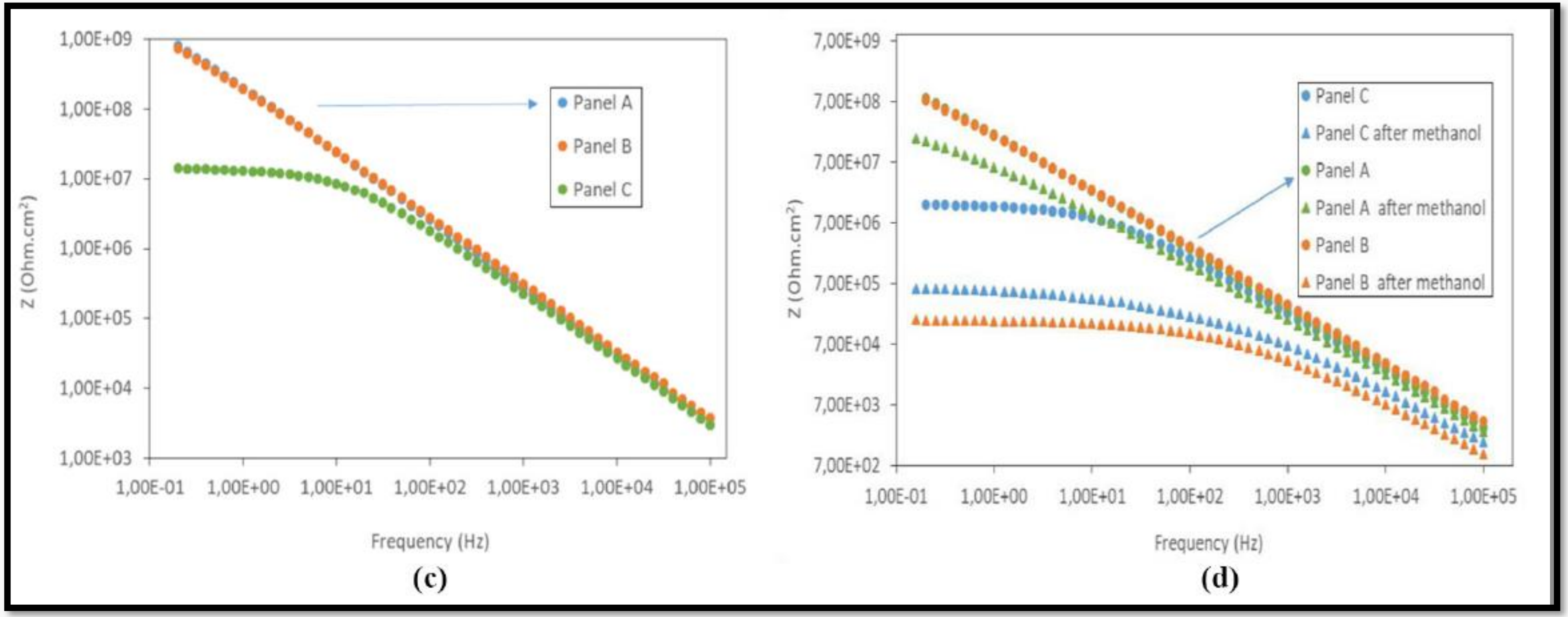


- Franchini, E., Galy, J. and Gerard, J.F. (2009), "Sepiolitebased epoxy nanocomposites: relation between processing, rheology, and morphology", Journal of Colloid and Interface Science, Vol. 329 No. 1, pp. 38-47.

Impedance



- Li, S., Wei, H., Zhao, X., Tang, Y. and Zuo, Y. (2024), "Failure behavior and life prediction of solvent-free epoxy coatings based on impedance spectroscopy", Electrochimica Acta, Vol. 492, p. 144349.



- Li, Y., Yang, Z., Qiu, H., Dai, Y., Zheng, Q., Li, J. and Yang, Y. (2014), "Self-aligned graphene as anticorrosive barrier in waterborne polyurethane composite coatings", Journal of Materials Chemistry, Vol. 2 No. 34, pp. 14139-14145.

The corrosion prevention mechanism

Figure 8: Schematic procedure of corrosion prevention mechanism of zinc-containing epoxy materials

Cathodic Zone

$$O_2 + 2H_2O + 4e \longrightarrow 4OH^-$$

$$Zn^{2+} + 2OH^- \longrightarrow Zn(OH)_2$$

Anodic Zone

$$2Fe \longrightarrow 2Fe^{2+} \longrightarrow 2Fe^{3+} \longrightarrow Fe_2O_3 + 3H_2O$$

2e

$$1/2O_2 + 2H_2O \longrightarrow 2OH^-$$

$$Fe^{2+} \text{ or } Fe^{3+} + PO_4^{3-} \longrightarrow Fe_3(PO_4)_2 \text{ or } Fe\,(PO_4)$$

- Ramezanzadeh, M., Ramezanzadeh, B. and Mahdavian, M. (2022), “Epoxy-zinc phosphate coating dual barrier/active corrosion prevention properties improvement via polyaniline modified lamellar kaolinite (ka@PAni) hybridpigment”, Progress in Organic Coatings, Vol. 172, p. 107132.
- Chen, C., Xiao, G., He, Y., Zhong, F., Li, H., Wu, Y. And Chen, J. (2020), “Bio-inspired superior barrier self-healing coating: self-assembly of graphene oxide and polydopaminecoated halloysite nanotubes for enhancing corrosion resistance of waterborne epoxy coating”, Progress in Organic Coatings, Vol. 139, p. 105402.

Conclusions

- ✓ Epoxy-based coatings, cured with amine compounds, show excellent corrosion resistance with no blistering, cracking, or flaking and minimal edge corrosion after exposure to humidity and a corrosive atmosphere.
- ✓ The anticorrosive performance is consistent in salt spray and humidity environments.
- ✓ Paint formulation C should have the best sag resistance and is more stable against paint separation and sedimentation compared to other formulations.
- ✓ EIS measurements show that the coating has low porosity and defects, limiting electrolyte diffusion and providing a strong physical barrier against corrosion.
- ✓ After one week in methanol, the anticorrosive performance slightly decreases.
- ✓ The developed paint formulations have effective anti-corrosion performance and serve as an important reference for industrial metal protection.

Thank you all for your kind attention and interest.

I would like to sincerely thank **Dr. Merve Demirkurt** and **Zeynep Yılmazer** for their valuable contributions and support throughout this study.

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Figure 7: Schematic of proposed electrical equivalent circuit (EEC) designs

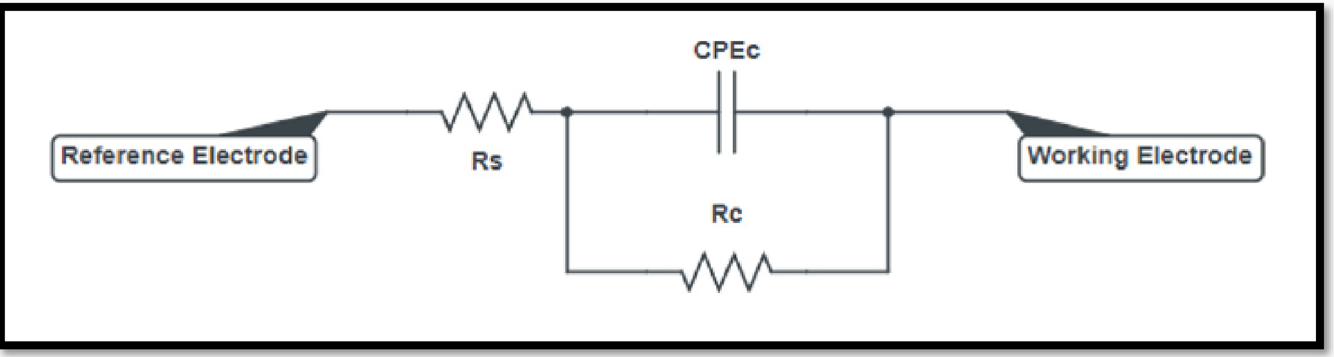

Table 2: Data for all elements in the models

	Rs (Ohm cm^2)	CPEc (S.sec^n/cm^2)	Rc (Ohm cm^2)
Panel A	5.27×10^{9}	744.8×10^{-12}	276.2×10^{3}
Panel B	1.190×10^{9}	628.4×10^{-12}	387.2×10^{3}
Panel C	12.62×10^{6}	758×10^{-12}	27.3×10^{3}
Panel A after methanol	166.1×10^{6}	1.453×10^{-9}	33.68×10^{3}
Panel B after methanol	571.2	3.185×10^{-9}	129.7×10^{3}
Panel C after methanol	501.9	2.185×10^{-9}	245.5×10^{3}

*The EIS data were fitted using a simplified Randles circuit model including Rs, Rc, and CPEc components. After one week of methanol immersion, a significant decrease in **Rc values** was observed, especially in panels B and C, indicating **electrolyte penetration**.

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