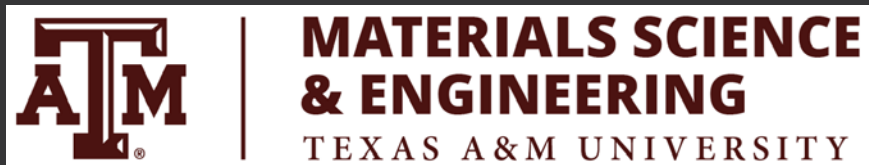


Development of Accelerated Corrosion Test Methods Using Wet-Dry Cycling of Varying Salt Solutions and the Study of Coating Damage Evolution via Electrochemical Test Methods

Victor Ponce, Shaik Merkatur Hakim Marjuban, and Homero Castaneda (Texas A&M University)

Heather Eich, Allison Mahood, and Rishi Gupta (Toyota Motor North America)



TOYOTA MOTOR
NORTH AMERICA

Agenda

- Background
- Experimental Setup
- Coating Degradation Model & Results
 - Barrier Coating
 - Sacrificial Coating
 - Hybrid Coating
- Corrosion Prediction Modelling Example
- Conclusion & Future study
- Acknowledgements

Background

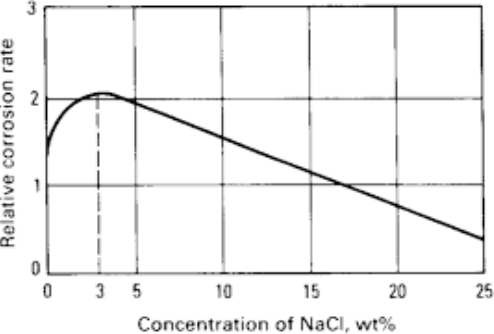
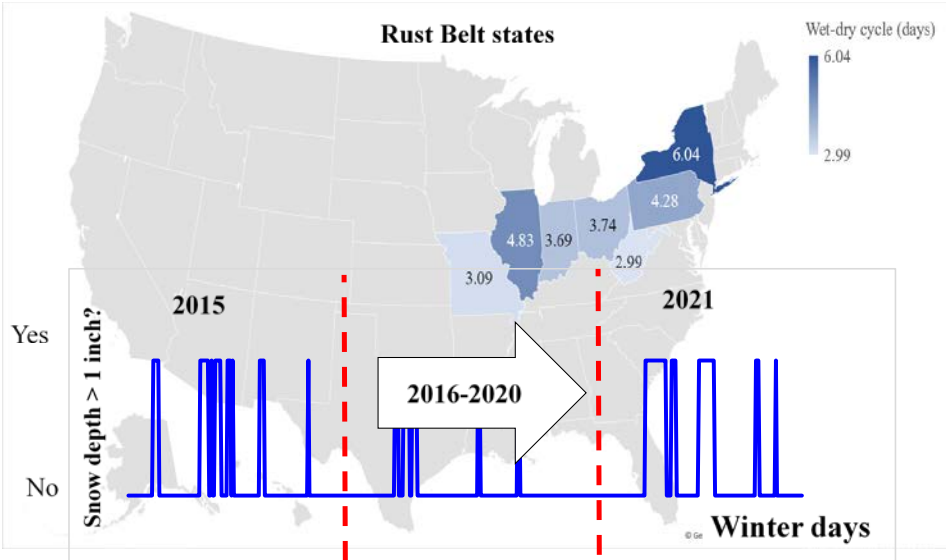


Objectives

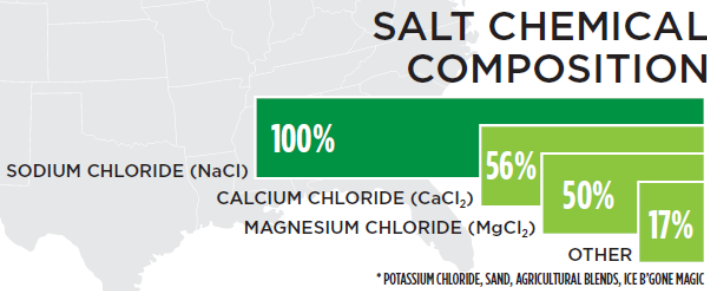
- Develop innovative and reliable accelerated corrosion testing methodologies with reduced lead time for automotive applications.
- Accurately replicate real-world corrosive environments.
- Incorporate electrochemical testing methods to further study corrosion mechanisms.
- Study the impact of varying salt solutions.

Wet-Dry Cycle Conditions

- Anti-Icing Process
 - In North America, salts are strategically applied to prevent the accumulation of snow on roads.
 - Salt chemical composition varies by region/agency.
- Cyclic Testing Development
 - North America climate data was studied to determine critical thresholds for wet conditions in Salt Belt Region.



Effect of NaCl concentration on corrosion of iron



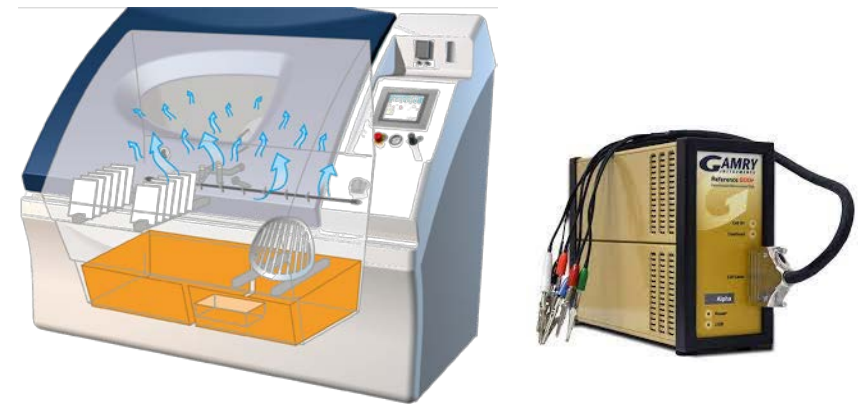
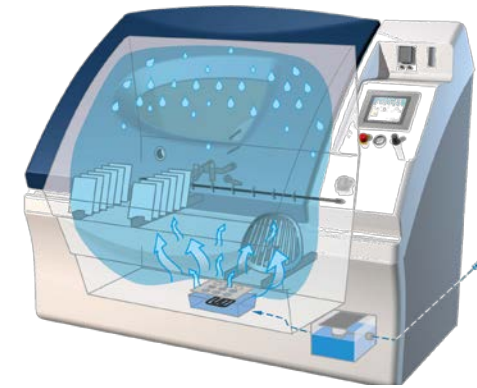
Results of a 2017 Salt Survey in North America

Table 1. Duration of wet condition along Rust Belt States

States	Duration of wet condition (days)
Illinois	4.83
Indiana	3.69
Missouri	3.09
New York	6.04
Ohio	3.74
Pennsylvania	4.28
West Virginia	2.99

Accelerated Corrosion Testing Methods

- Accelerated corrosion testing simulates long-term corrosion behavior in shorter time frames.
- Cyclic Corrosion Testing (CCT) chambers are effective tools for accelerated corrosion testing.
- Precisely regulated variables include temperature, humidity, and exposure to corrosive agents.
- In this study, Electrochemical Impedance Spectroscopy (EIS) methods are used to study phase angle results mid-CCT cycle.



Experimental Setup

Test methodology

➤ Testing Environment

- Temperature: 30 °C
- Electrolyte: Various
- Cycling:
 - 96 hours wet condition
 - 72 hours dry condition

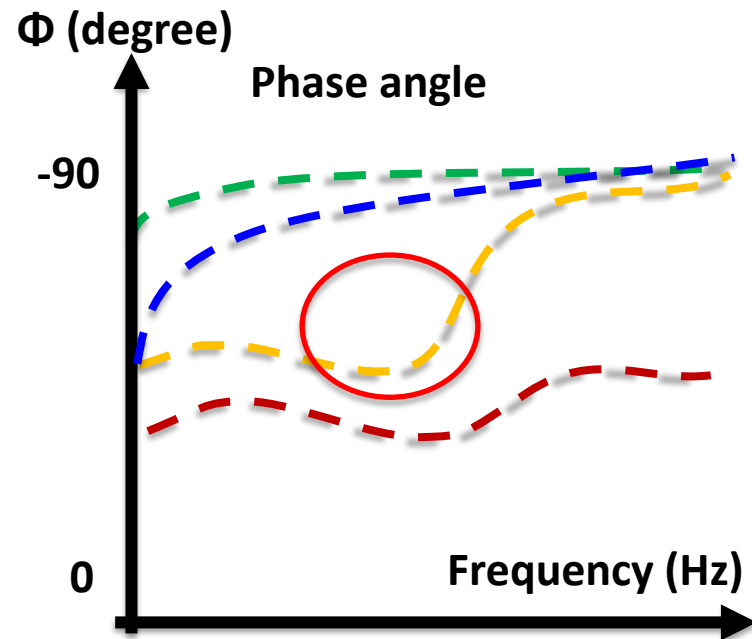
➤ Electrochemical Test Parameters

- AC Voltage: 10 mV
- Frequency Range: 0.01 to 100000 Hz
- Dry Test Cell using Clamp
- Measurements taken during dry cycle

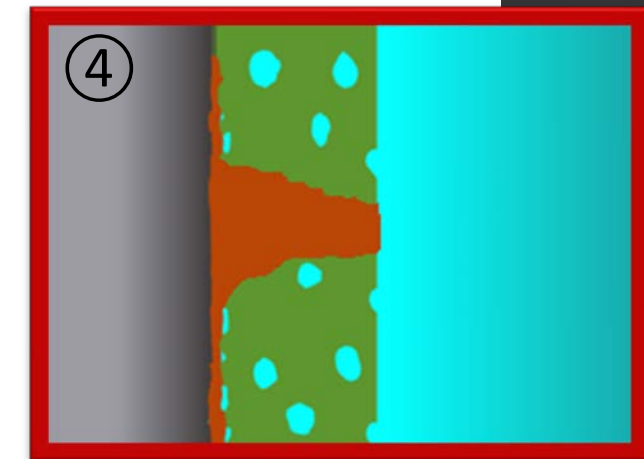
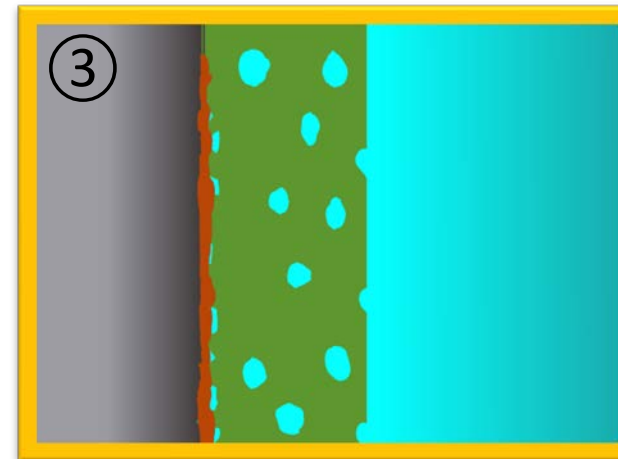
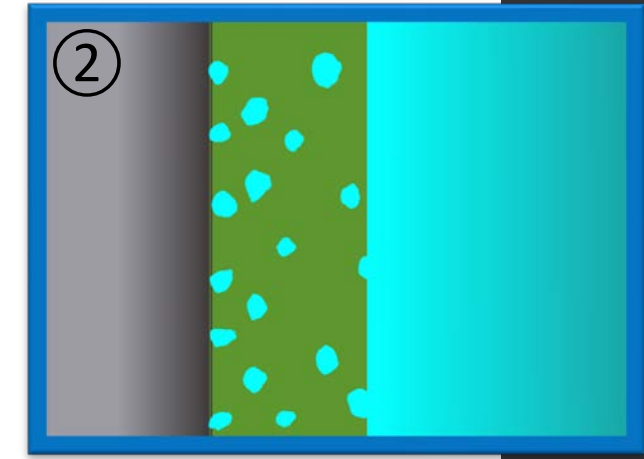
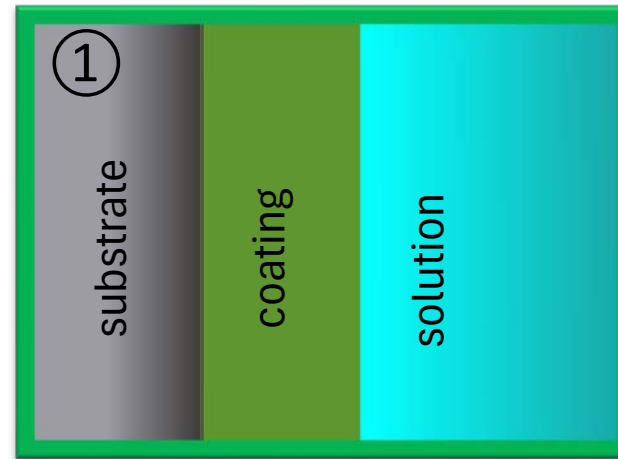


Coating Degradation Model & Results

Phase Angle Model: Barrier Coating

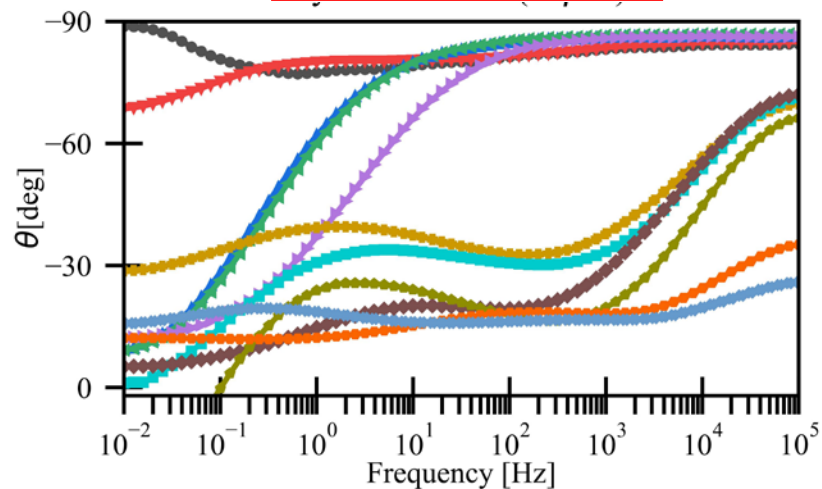


- ① Capacitor Behavior
- ② Water Uptake
- ③ Substrate Reaction
- ④ Corrosion Propagation

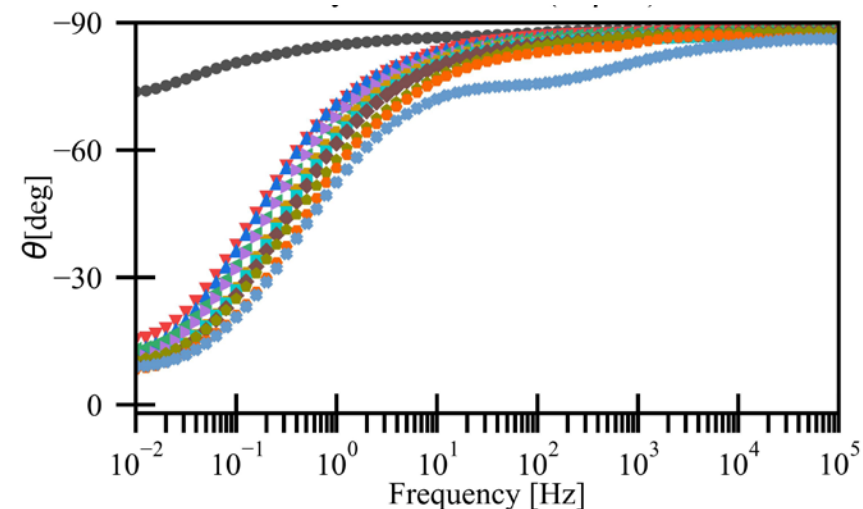


EIS Results: 10 μ m Barrier Coating

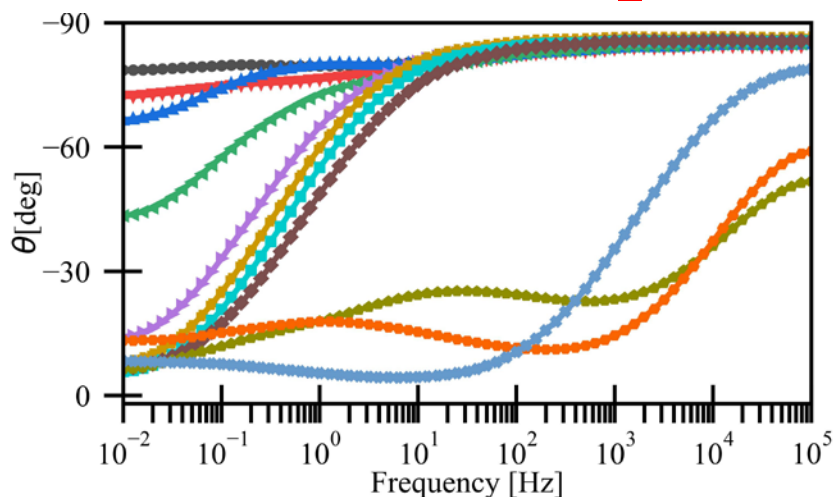
5wt% NaCl Solution



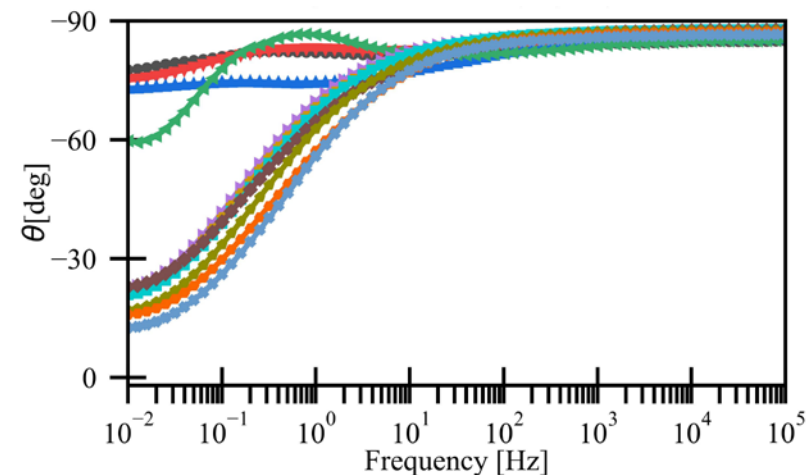
2wt% NaCl Solution



2wt% NaCl + 0.04wt% CaCl₂ Solution



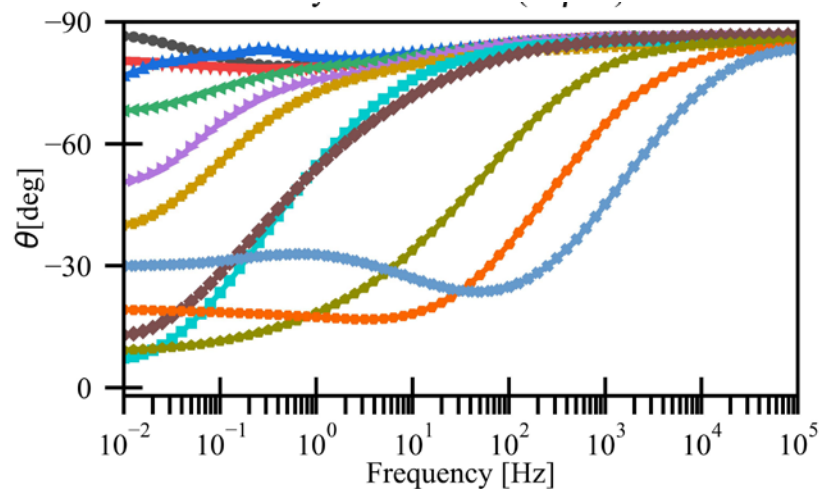
2wt% NaCl + 0.04wt% MgCl₂ Solution



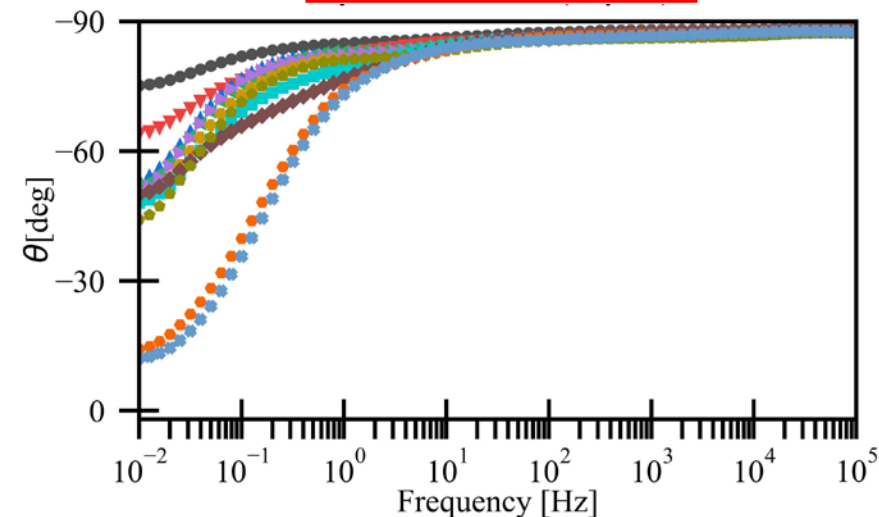
- Day 0
- ▼ Day 4
- ▲ Day 7
- ◀ Day 11
- ▶ Day 14
- ✦ Day 18
- ◼ Day 21
- ◆ Day 25
- ◊ Day 28
- ◇ Day 32
- ⬠ Day 35

EIS Results: 25 μ m Barrier Coating

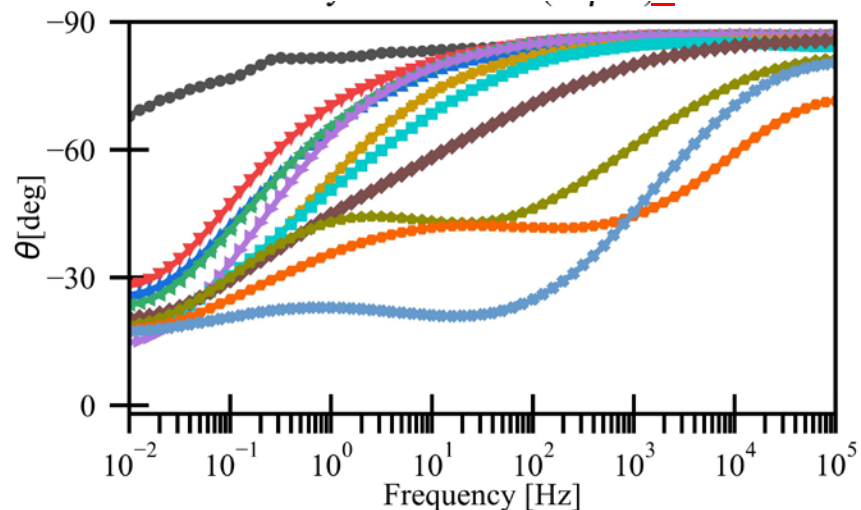
5wt% NaCl Solution



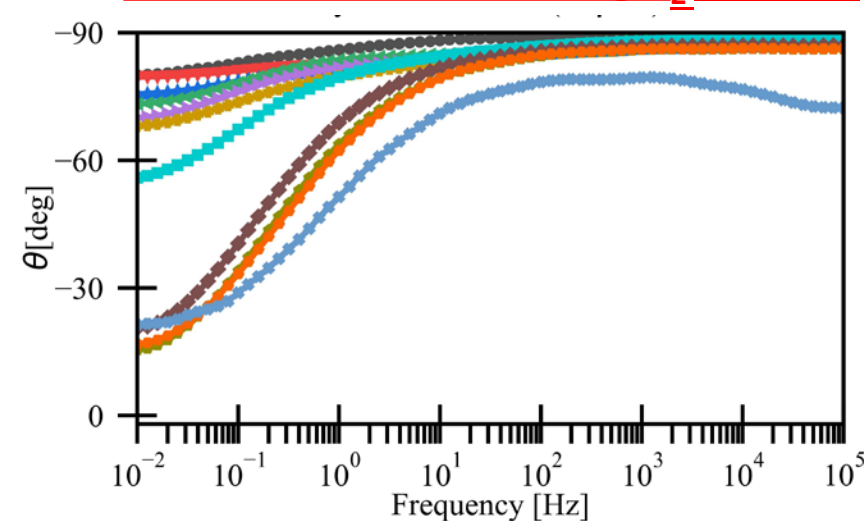
2wt% NaCl Solution



2wt% NaCl + 0.04wt% CaCl_2 Solution

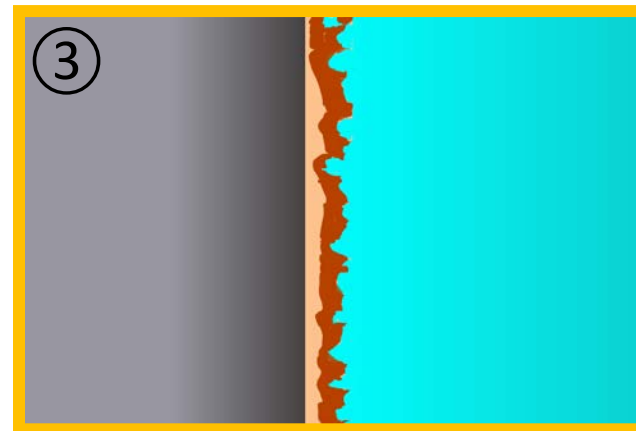
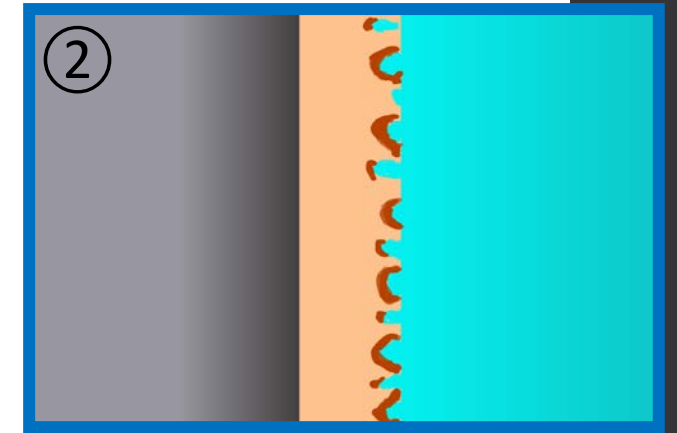
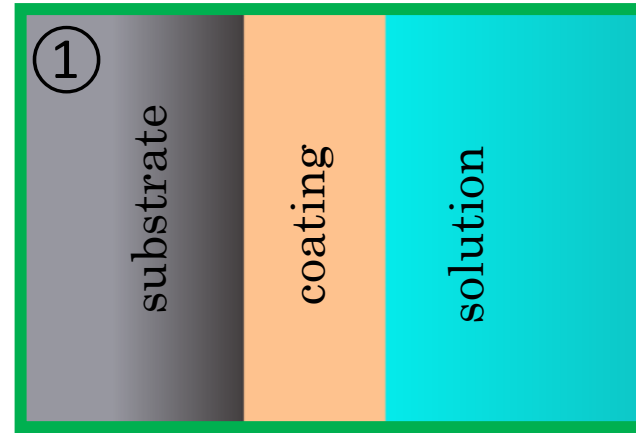
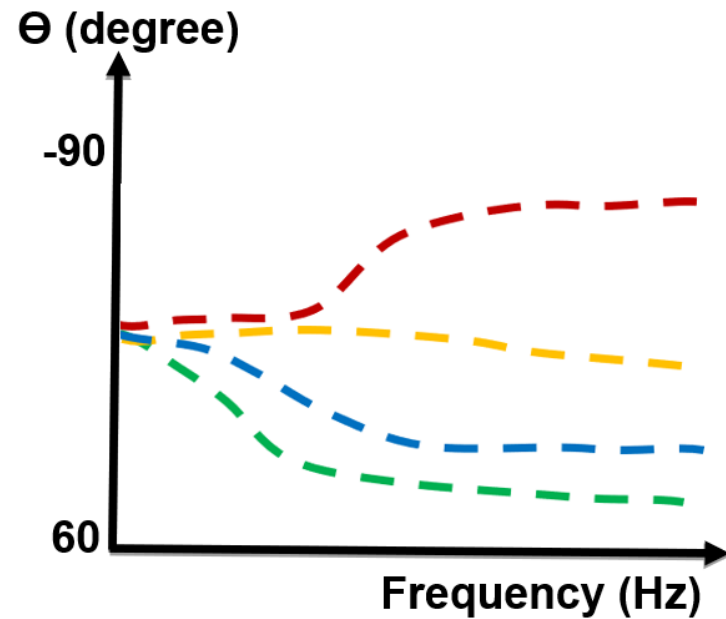


2wt% NaCl + 0.04wt% MgCl_2 Solution



- Day 0
- ▼ Day 4
- ▲ Day 7
- ◀ Day 11
- ▶ Day 14
- ✦ Day 18
- Day 21
- ◆ Day 25
- ◇ Day 28
- ◈ Day 32
- ✧ Day 35

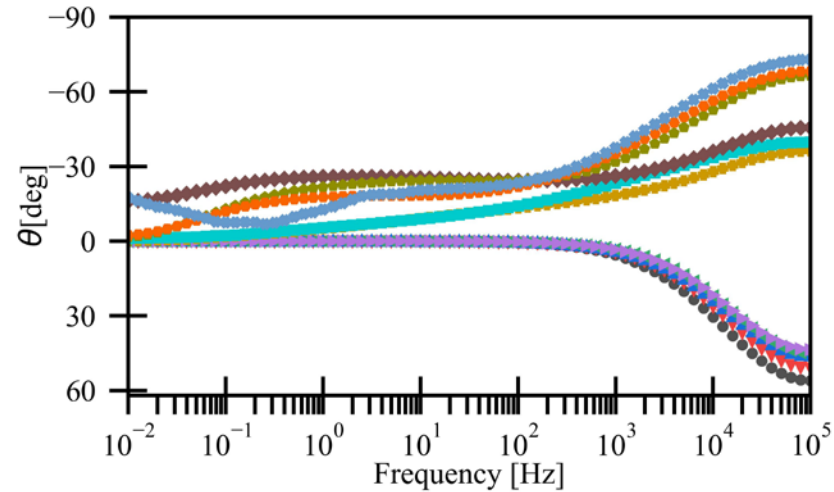
Phase Angle Model: Sacrificial Coating



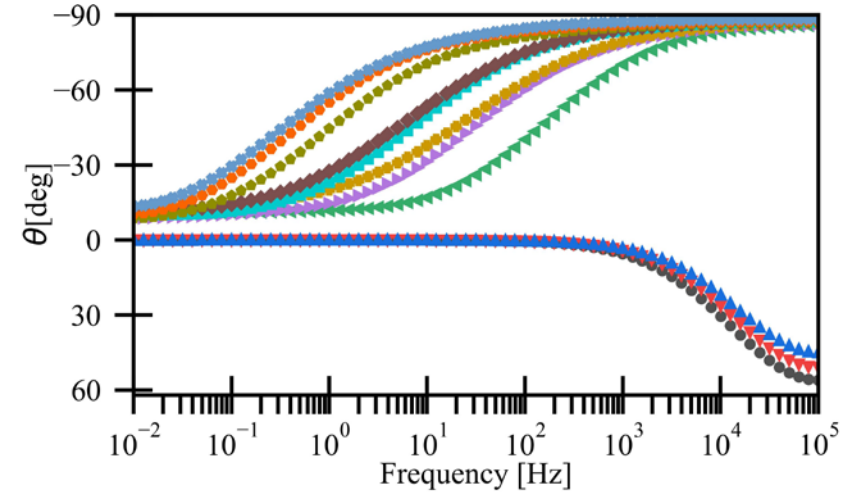
- ① Sacrificial Metallic Layer
- ② Sacrificial Metal Reaction
- ③ Sacrificial Reaction & Passive Layer Formation
- ④ Passive Layer Formation

EIS Results: Sacrificial Coating

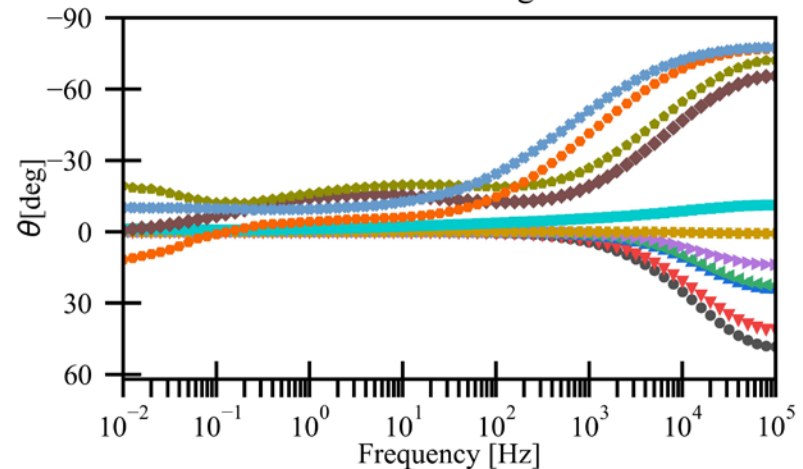
5wt% NaCl Solution



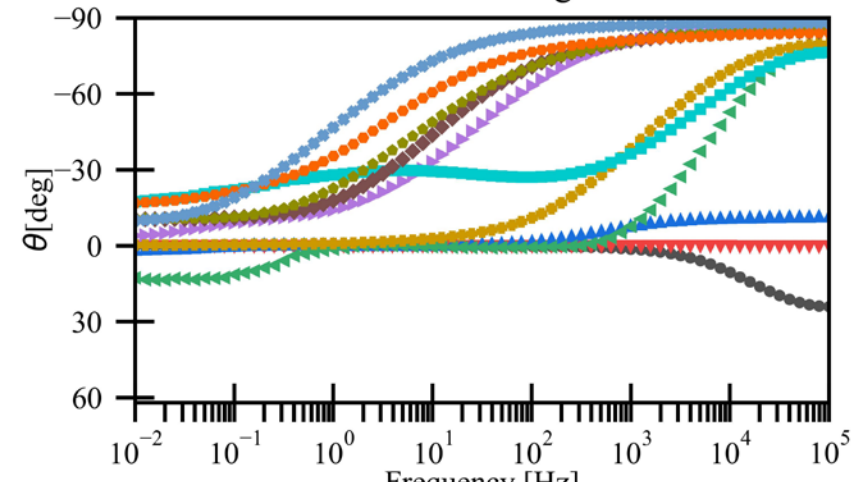
2wt% NaCl Solution



2wt% NaCl + 0.04wt% CaCl₂ Solution

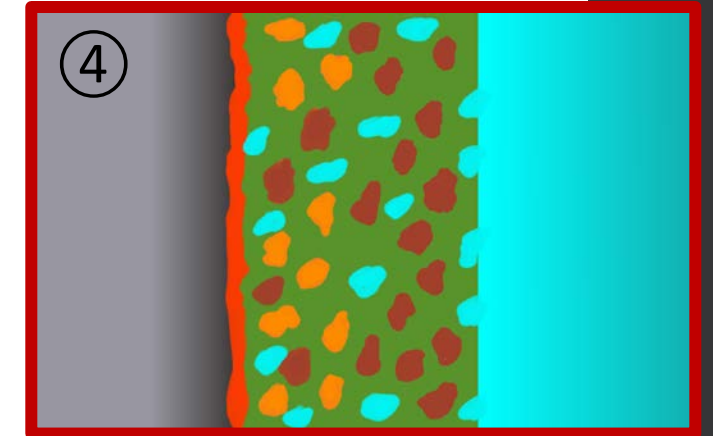
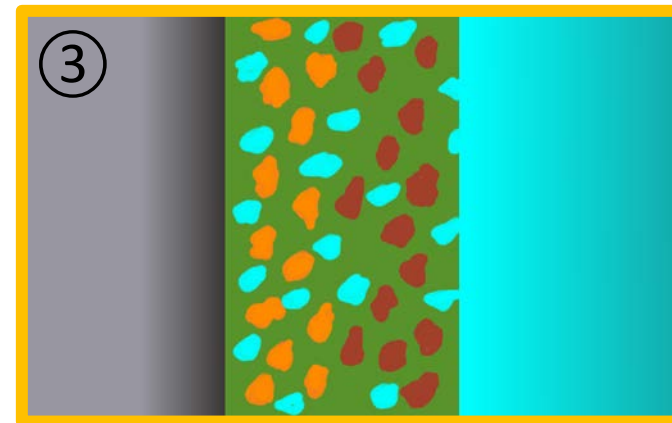
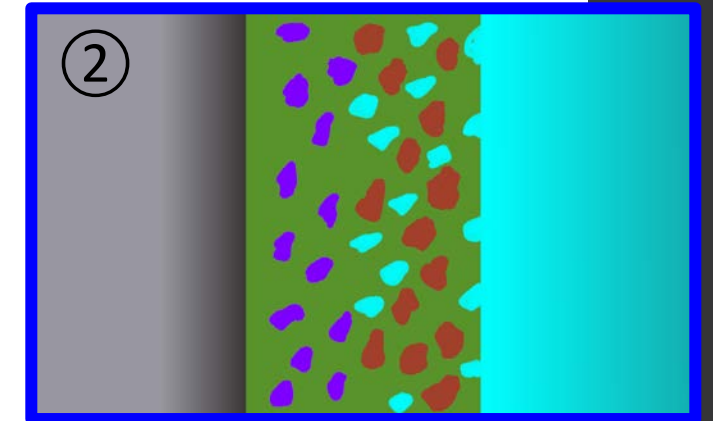
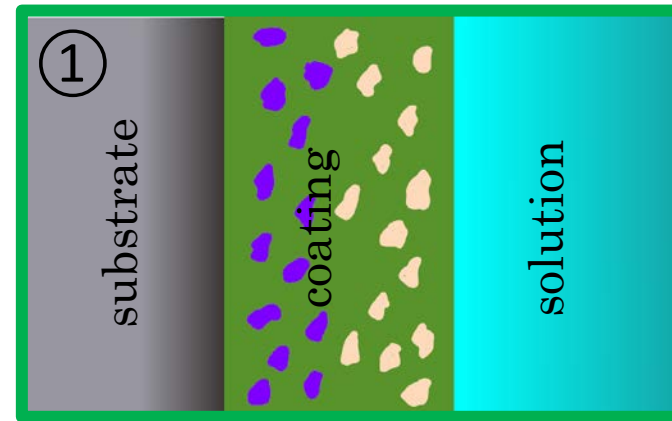
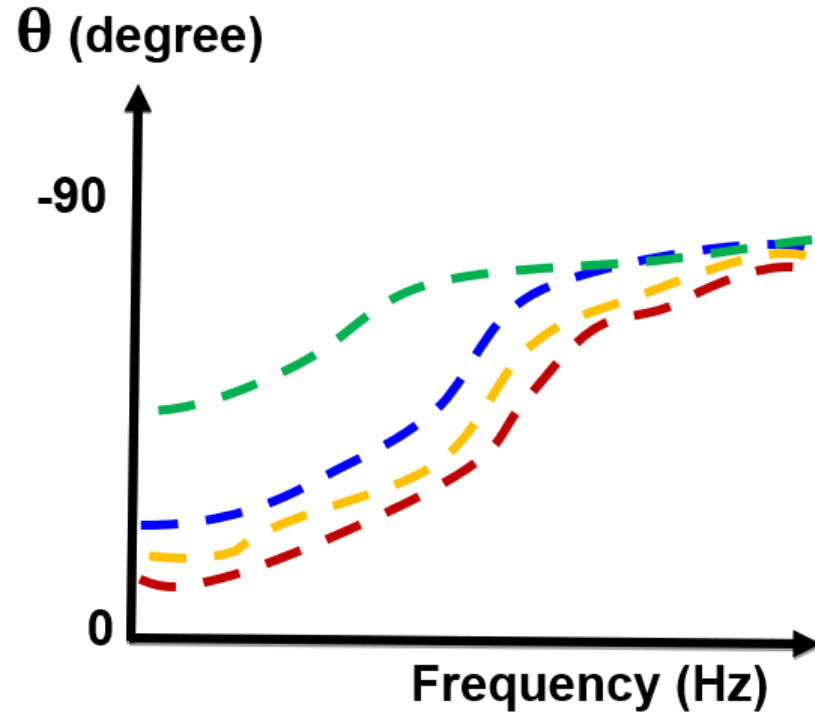


2wt% NaCl + 0.04wt% MgCl₂ Solution



- Day 0
- ▼ Day 4
- ▲ Day 7
- ◀ Day 11
- ▶ Day 14
- ✦ Day 18
- ◼ Day 21
- ◆ Day 25
- ◇ Day 28
- ◈ Day 32
- ✧ Day 35

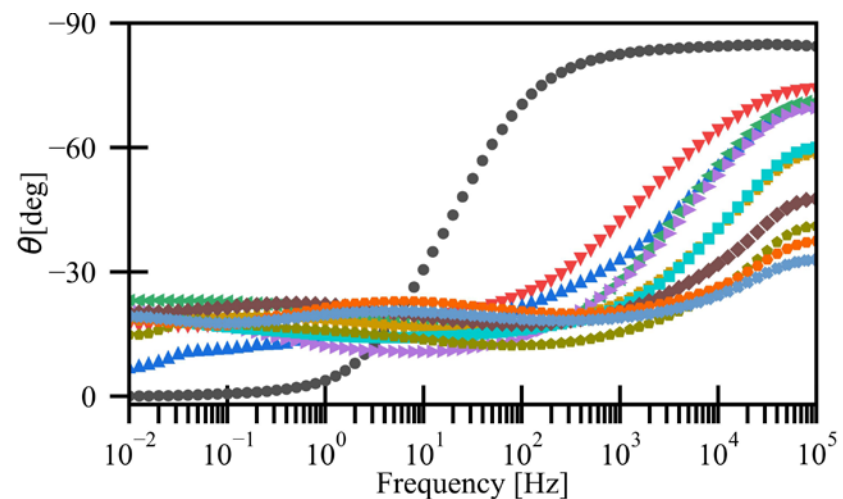
Phase Angle Model: Hybrid Coating



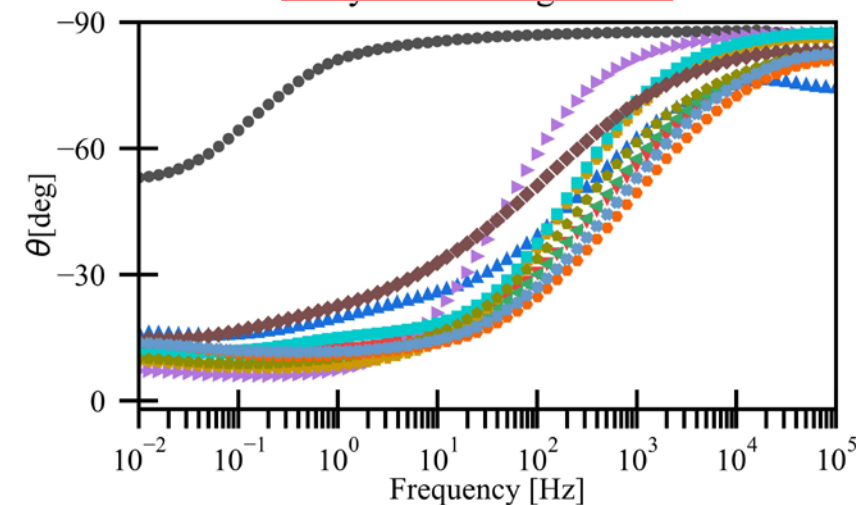
- ① Hybrid Coating Layer
- ② Water Uptake & Sacrificial Metal Reaction
- ③ Water Uptake & Sacrificial Metal Reaction
- ④ Water Uptake & Surface Reaction

EIS Results: Hybrid Coating

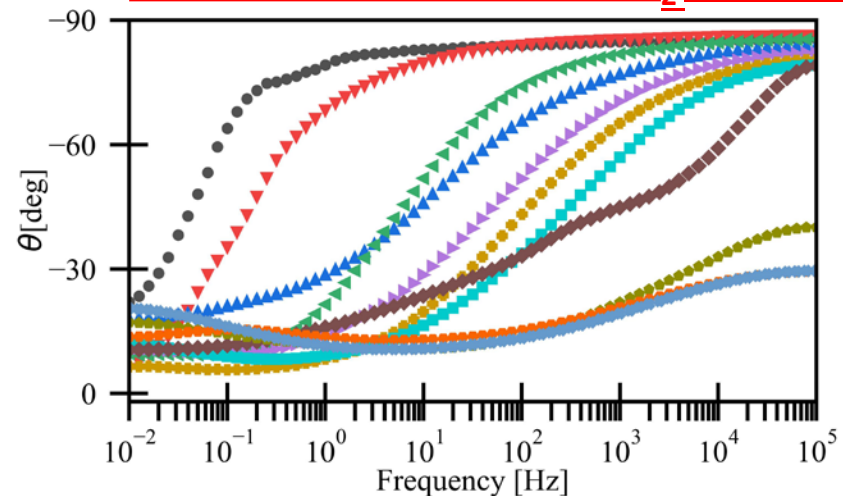
5wt% NaCl Solution



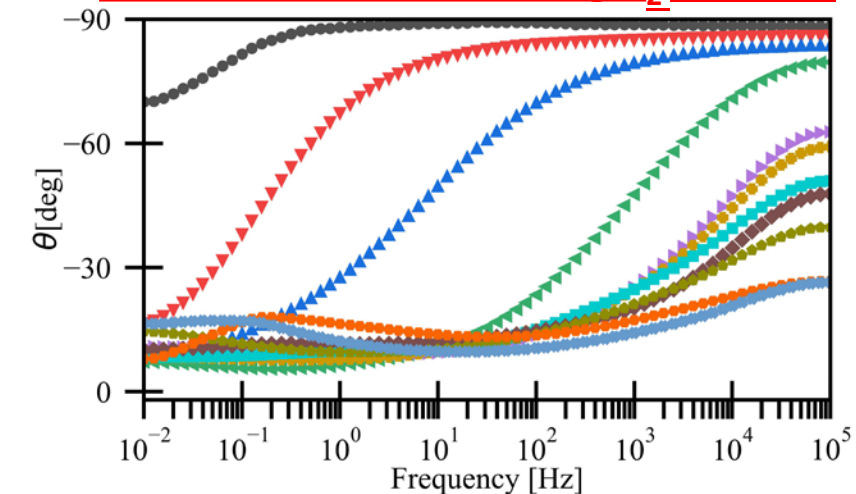
2wt% NaCl Solution



2wt% NaCl + 0.04wt% CaCl_2 Solution



2wt% NaCl + 0.04wt% MgCl_2 Solution



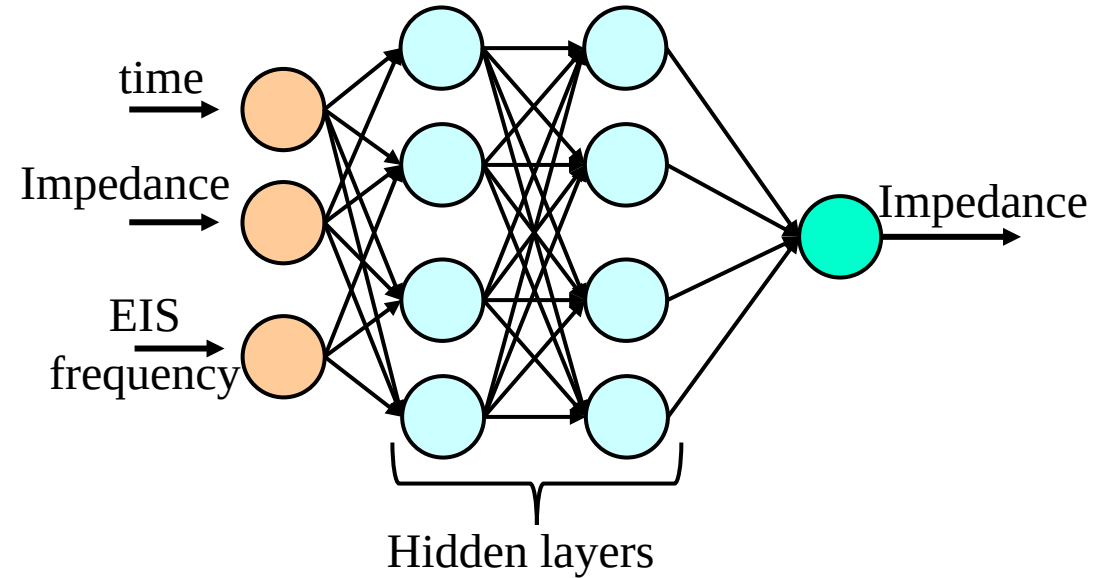
- Day 0
- Day 4
- Day 7
- Day 11
- Day 14
- Day 18
- Day 21
- Day 25
- Day 28
- Day 32
- Day 35

Corrosion Prediction Modelling Using ANN

Preprocessing

		Frequency					
Days		100078.1	79453.13	63140.62	50203.12		0.01
1	27.54086	33.2942	40.34059	49.74229		5.49E+08	
2	40.2433	45.49031	53.75675	64.45002		5.39E+08	
3	35.73117	41.60759	49.97059	59.27808		5.31E+08	
4	36.31976	42.09015	50.32156	61.91766		5.25E+08	
5	36.70223	42.92453	50.60925	60.40915		5.24E+08	
6	36.55861	42.6273	49.76031	60.2007		5.23E+08	
7	36.53695	42.16451	49.99495	61.14406		5.13E+08	
8	37.24772	42.76405	50.8639	61.51939		5.13E+08	
.		Real and Imaginary components of Impedance					
.							
.							

From high to low frequency



Time Series of Impedance

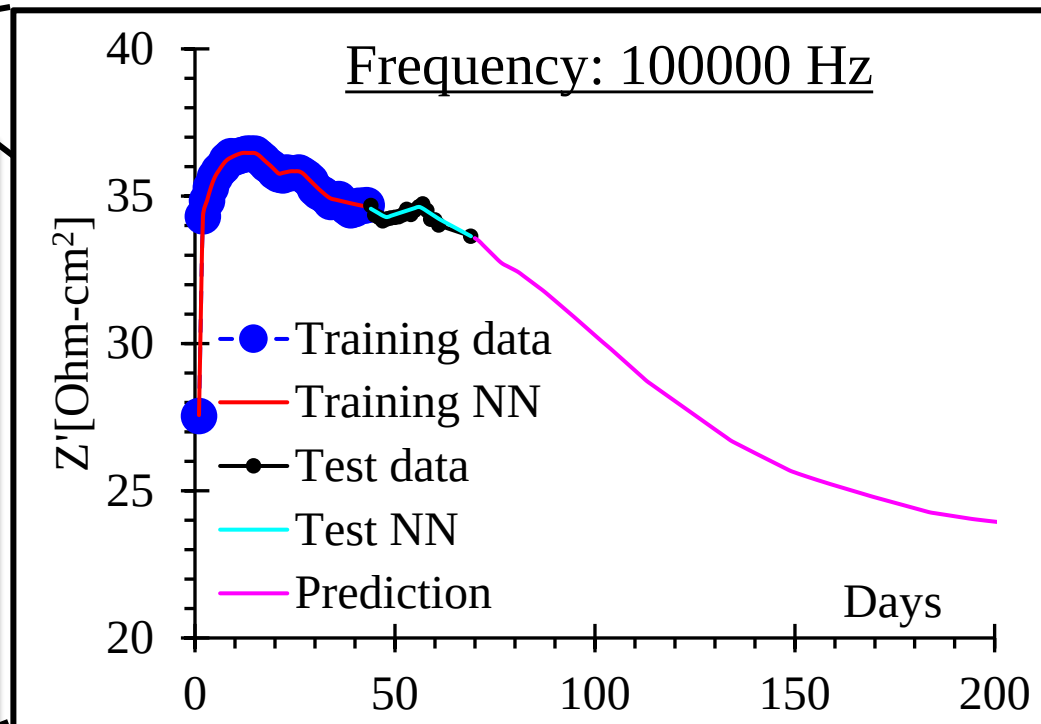
Days

Frequency

	100078.1	79453.13	63140.62	50203.12		0.01
1	27.54086	33.2942	40.34059	49.74229		5.49E+08
2	40.2433	45.49031	53.75675	64.45002		5.39E+08
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8	37.24772	42.76405	50.8639	61.51939		5.13E+08
.						
.						
.						

Real and
Imaginary
components of
Impedance

From high to low frequency



Time Series of Impedance

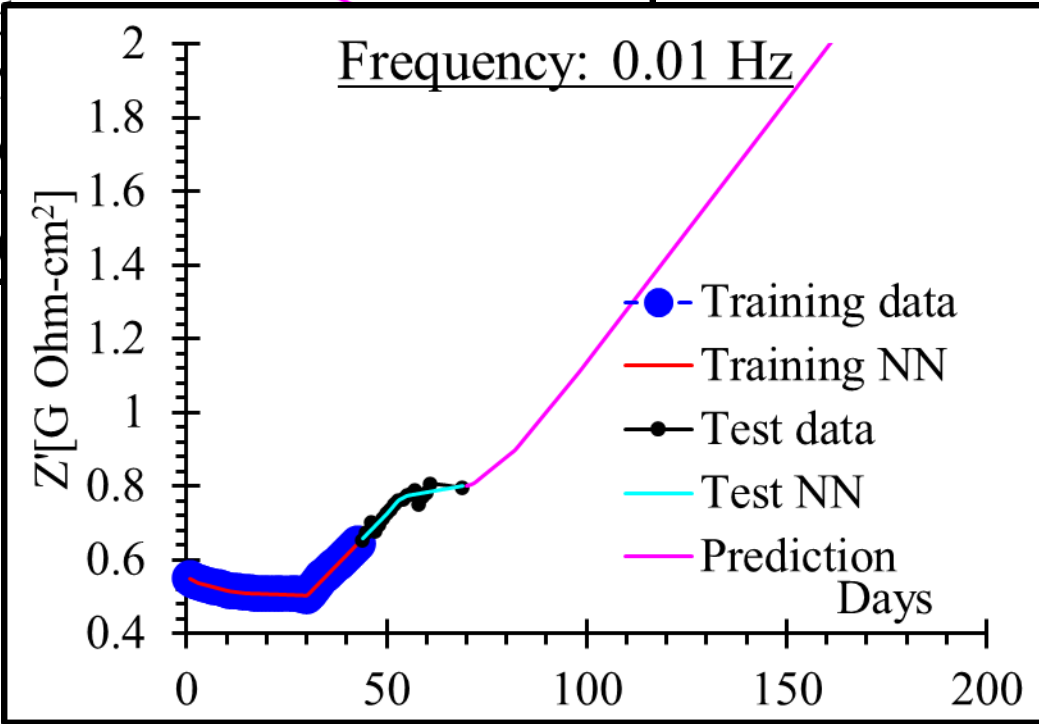
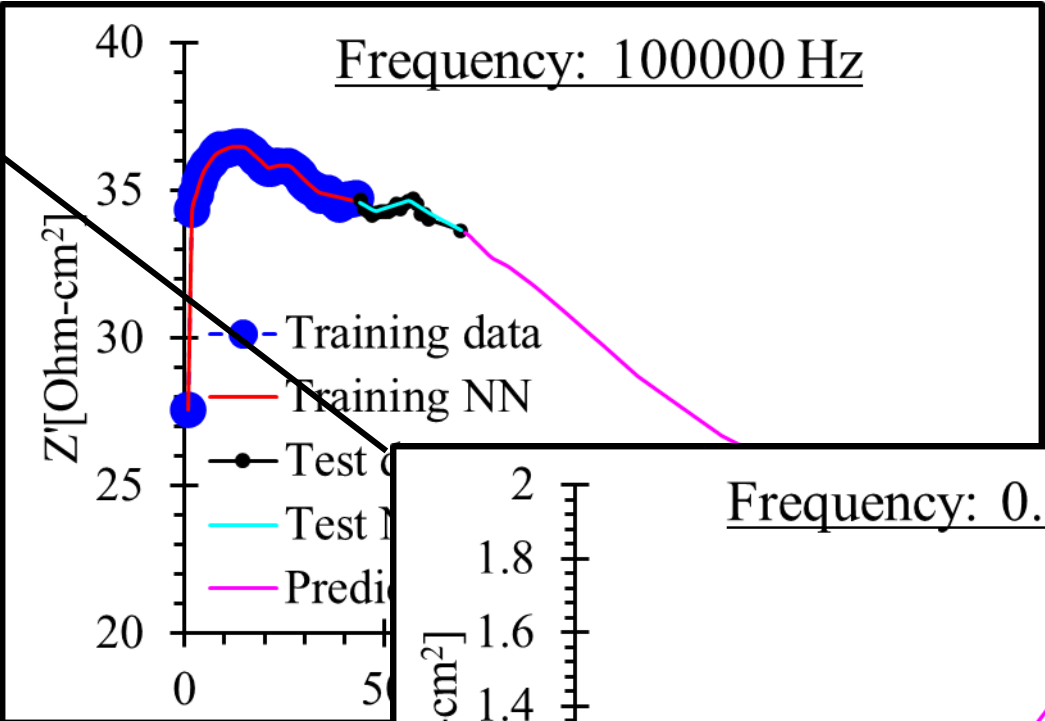
Days

Frequency

	100078.1	79453.13	63140.62	50203.12		0.01
1	27.54086	33.2942	40.34059	49.74229		5.49E+08
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·						
·						
·						

Real and
Imaginary
components of
Impedance

From high to low frequency



Validation

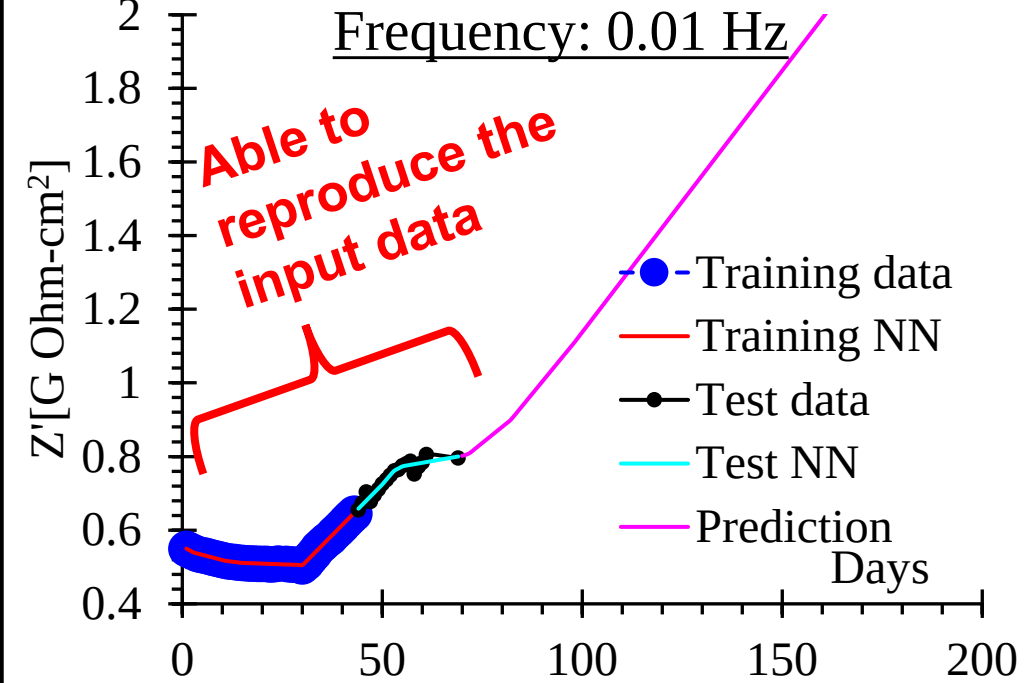
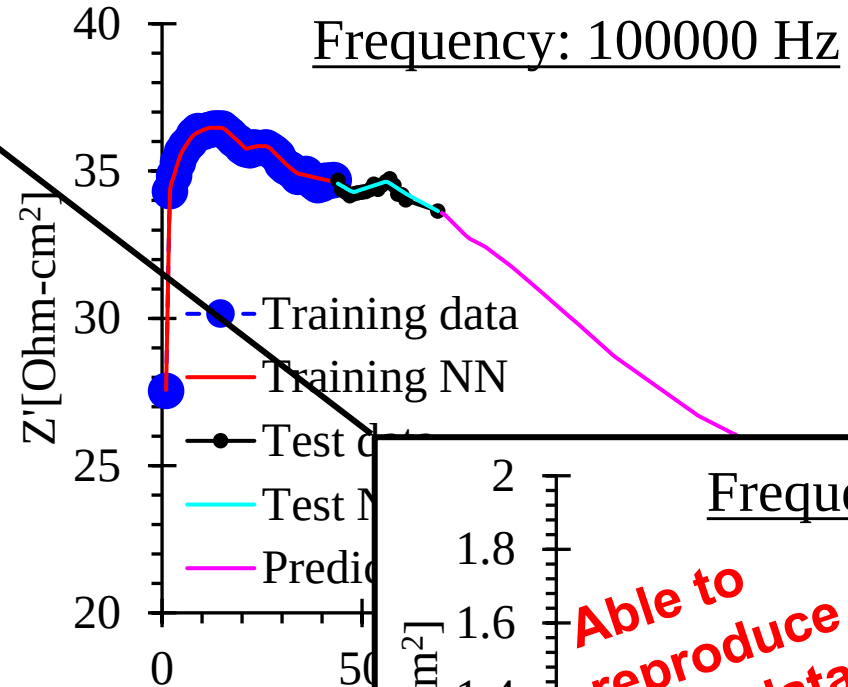
Days

Frequency

	100078.1	79453.13	63140.62	50203.12		0.01
1	27.54086	33.2942	40.34059	49.74229		5.49E+08
2	40.2433	45.49031	53.75675	64.45002		5.39E+08
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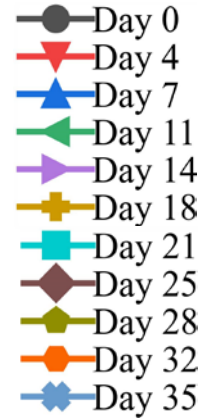
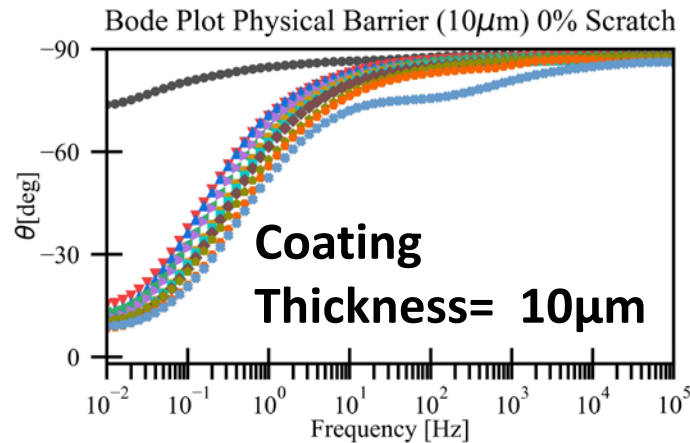
Real and
Imaginary
components of
Impedance

From high to low frequency

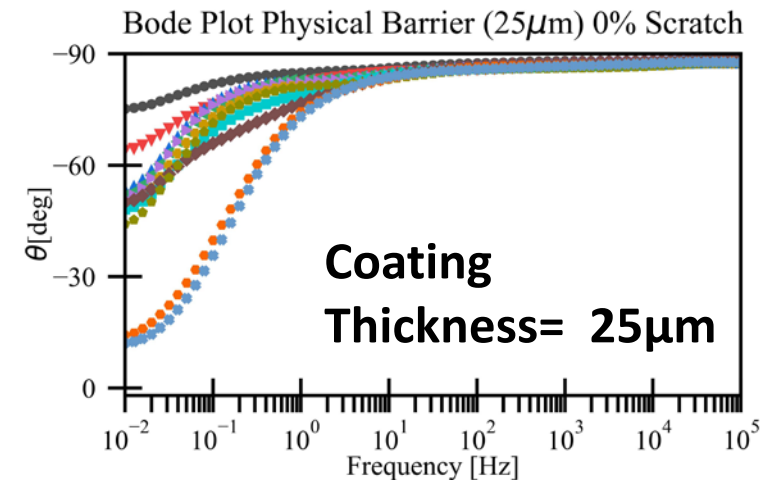


Results: Corrosion Prediction for Barrier Coating in 2wt% NaCl Solution

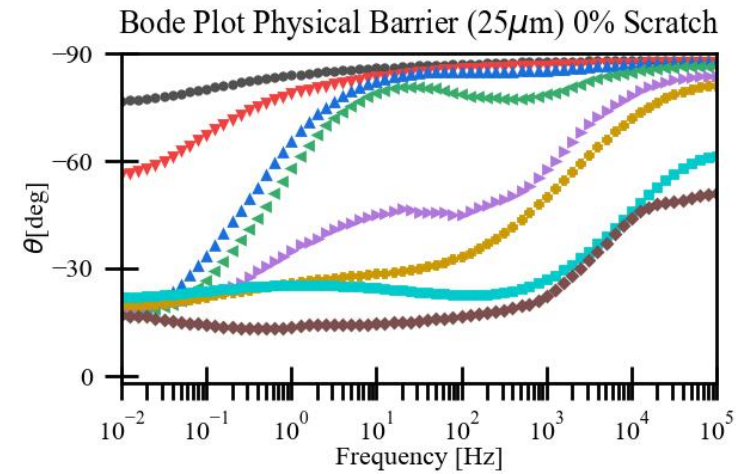
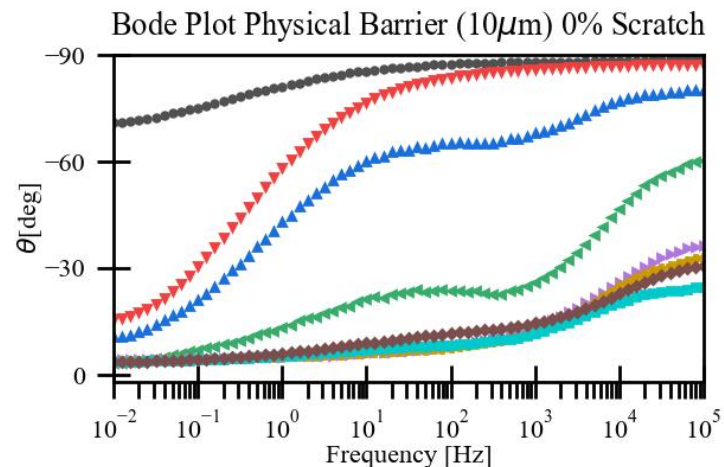
Experimental Data (Input)



Experimental Data (Input)



**Prediction
(Output)**

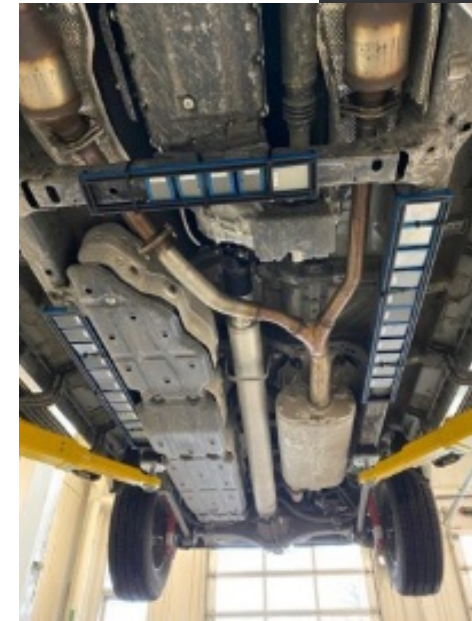


Conclusion

- With accelerated corrosion + electrochemical methods we observe corrosion mechanisms in coatings in less than 30 days. Results can be confirmed by SEM.
- ANN Modelling can be used to support long term corrosion prediction.
- The impact of salt type varies by coating type (barrier vs sacrificial vs hybrid).

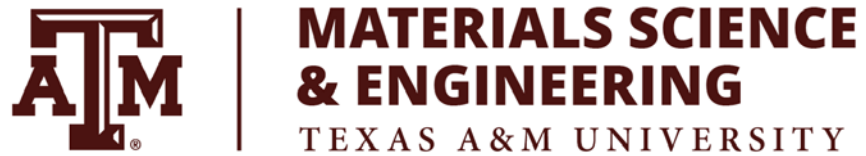
Future Study

- Continue development and validation of the corrosion prediction model.
- Study the impact of different CCT cycle lengths and salt combinations to corrosion rate.
- Compare results to real world corrosion monitoring currently in progress in North America.



Field monitoring activity setup in NA

Acknowledgments



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NORTH AMERICA