Improving Corrosion Resistance, Alkaline Resistance, and Weatherability of Waterborne Coatings with Organofunctional Bipodal Silanes

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Coatings Trends & Technologies Conference 2019

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Agenda

1. Project Background
2. Silane Chemistry Introduction
3. Experimental Methods
4. Corrosion Resistance Performance
5. Electrochemical Impedance Spectroscopy Data
6. Alkaline Resistance Performance
7. Outdoor Weatherability Test Results
8. Q&A

Environmentally-friendly Corrosion Protection of Metals is an Important Topic for Evonik

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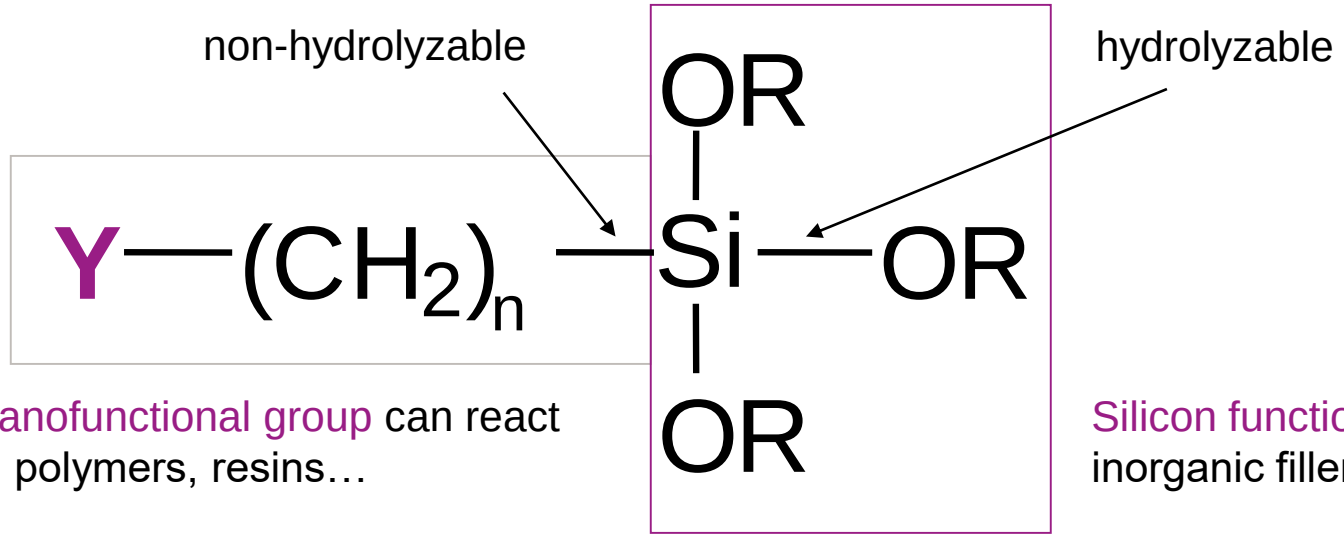
Organofunctional Silanes are Bifunctional Molecules

$$Y—(CH_2)_nSi(OR)_3$$

n = 0, 3
R = CH_3, C_2H_5, $(CH_2)_2OCH_3$
Y = Organofunctional group

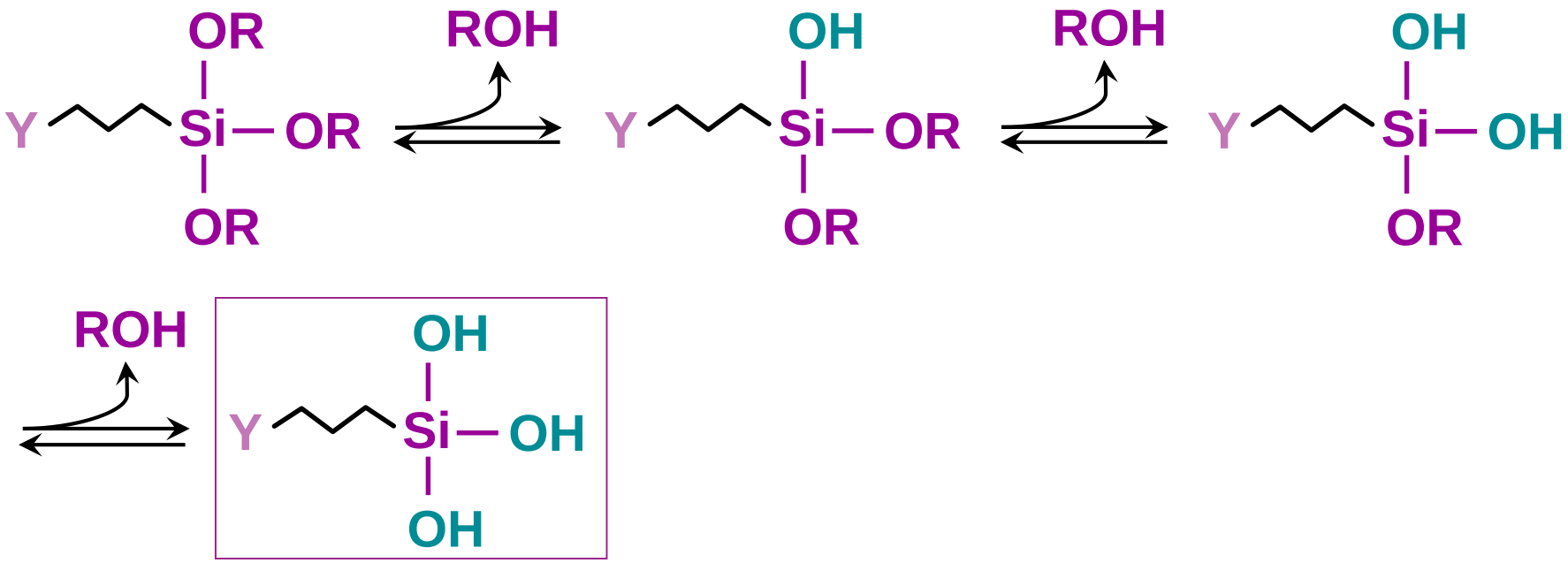


Organofunctional group can react with polymers, resins…

Silicon functional group can react with inorganic fillers, metals, glass…

Hydrolysis of Silanes

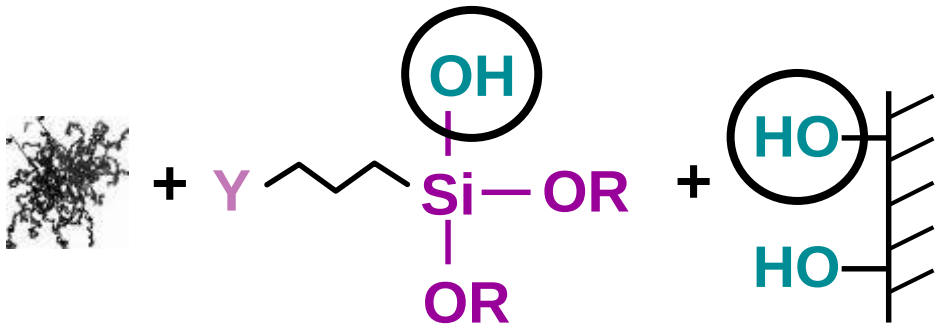
In the presence of excess water…

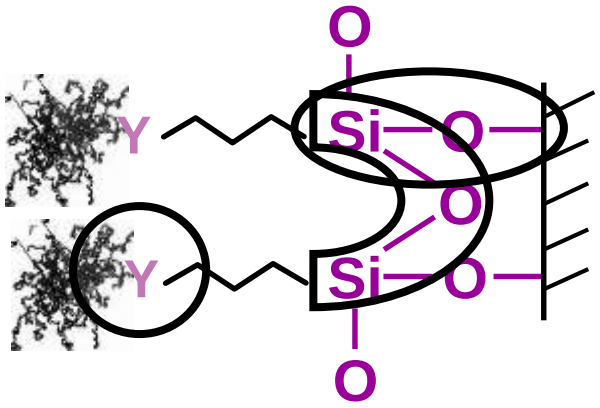


OR = “Silicon-functional“ group | Y = “Organofunctional“ group

Silanes, Hydrolysis and Condensation

Polymer + Silane + Surface → **Adhesion promotion**





OR = “Silicon-functional“ group | Y = “Organofunctional“ group

Reactivity of Metal Substrates and Organofunctional Silanes

The reactivity of a silane towards a substrate depends on the hydroxyl concentration, functionality, and polarity of the surface

Metals
▪ Copper
▪ Aluminum
▪ Silver
▪ Cold Rolled Steel
▪ Zinc
▪ Gold

good

bad

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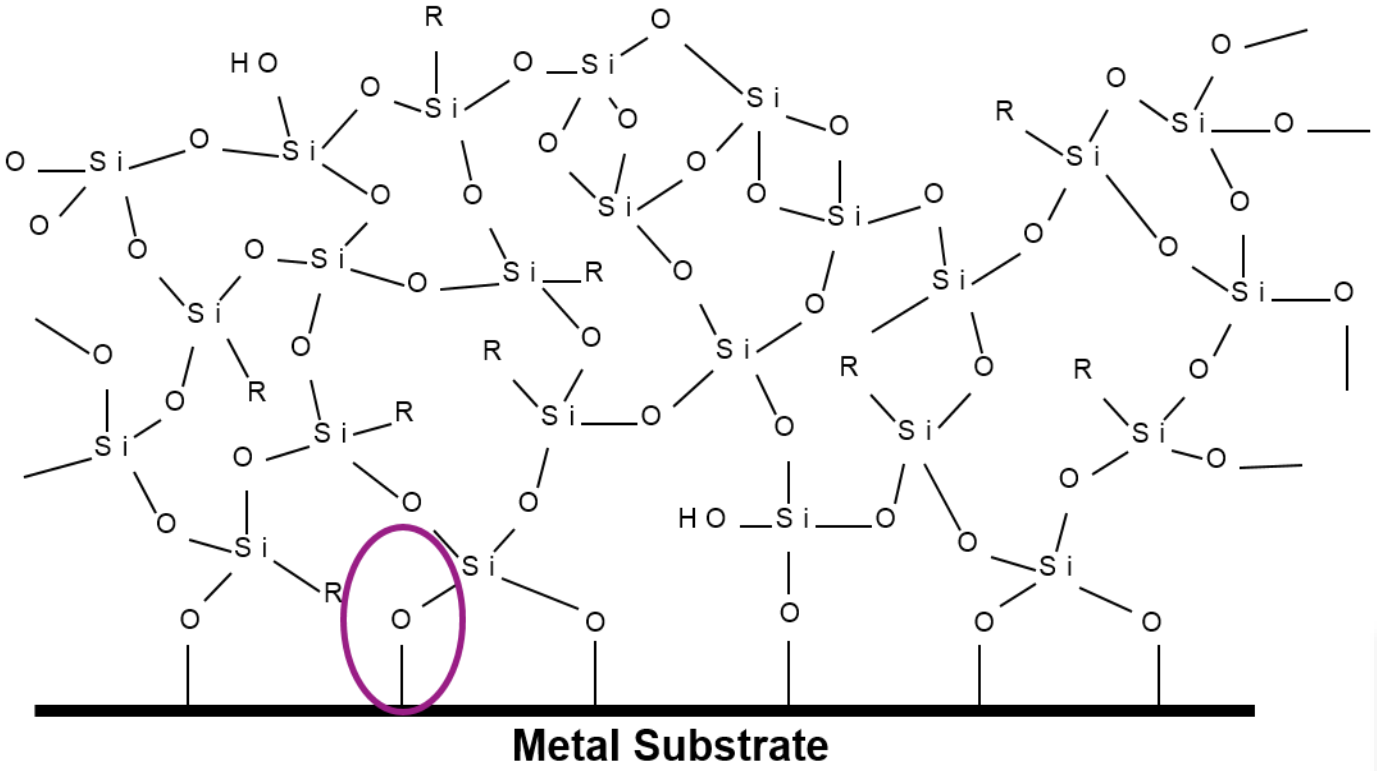
Water-borne Sol-gel Silane Systems for Metal Corrosion Protection

Water-borne Sol-gel Silane Systems – a heavy metal & VOC free alternative corrosion protection



Using a silane enables:

- Surface passivation
- Barrier effect – water and chloride
- Improved adhesion of subsequent coating

Water-borne silanes typically require high temp cure!

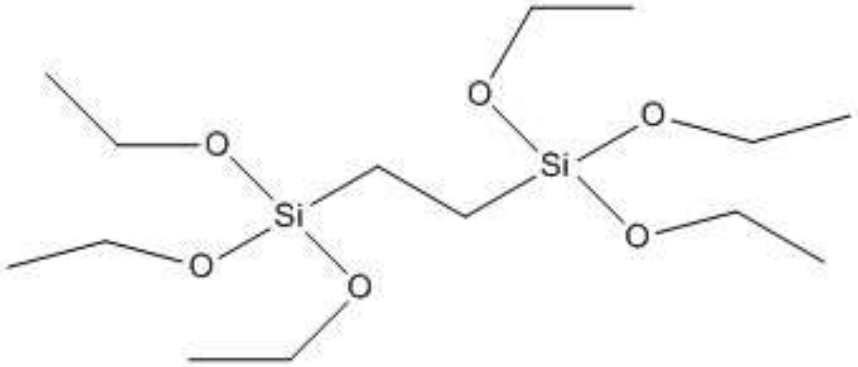
"Sol-gel Silane System"

Water-borne, Sol-gel Silane System Containing Functionalized SiO_2 Nanoparticles

Bipodal Silanes for Improving the Corrosion Resistance of Water-borne Systems

Advantages

- Hydrophobic
- High crosslinking density
- Good surface passivation
- Compatible with water-borne systems



Bis(triethoxysilyl)ethane – "Bipodal Silane #1"

More hydrophobicity and surface passivation enhances corrosion resistance!

Additional crosslinking can reduce the need for high temperature curing!

Experimental Methods

Silane Formulations

- Mix Bipodal Silane #1 in water-borne systems for 24+ hours for full hydrolysis

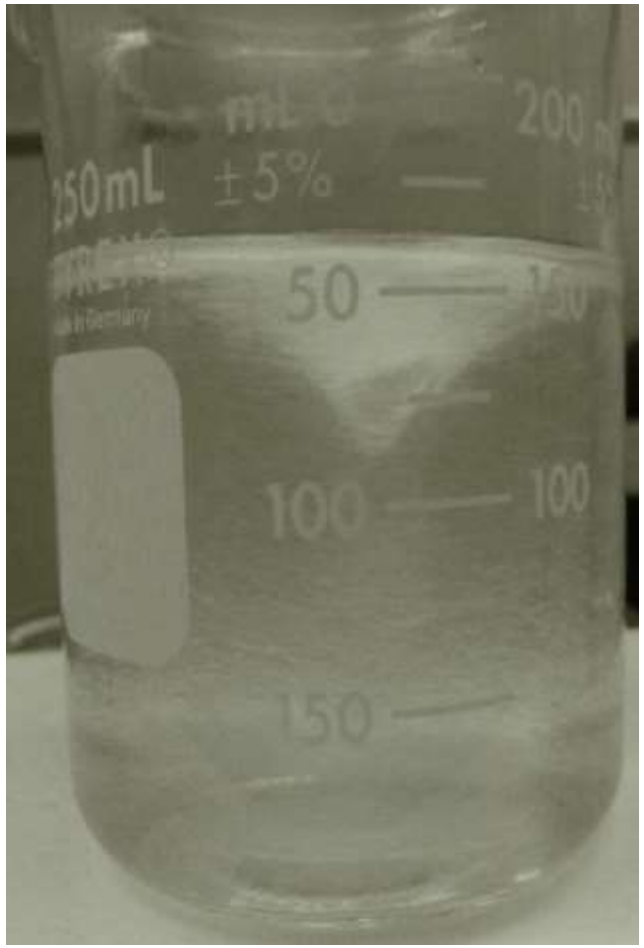
Application & Curing Procedures

- Wipe metal substrate with isopropyl alcohol
- Alkaline wash metal substrate for 3 minutes at 140°F
- Dip coat substrate for ~60 secs
- Cure in oven at desired temperature: 23°C – 180°C

Testing Procedures

- Neutral Salt Spray test (ASTM B117)
- Electrochemical Impedance Spectroscopy (performed by Matergenics Inc.)
- Alkaline Resistance test (GMW 14665 – General Motors specification)
- Outdoor weatherability testing (Piscataway, NJ location)

Mixing 2 wt.% Bipodal Silane #1 in H_2O

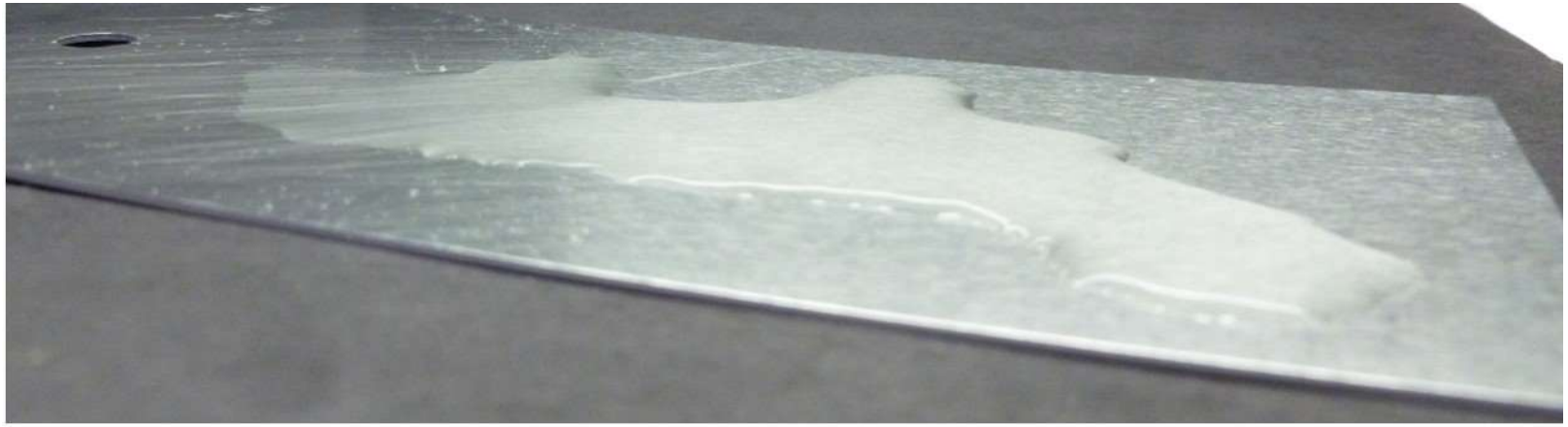


1h after mixing

24h after mixing

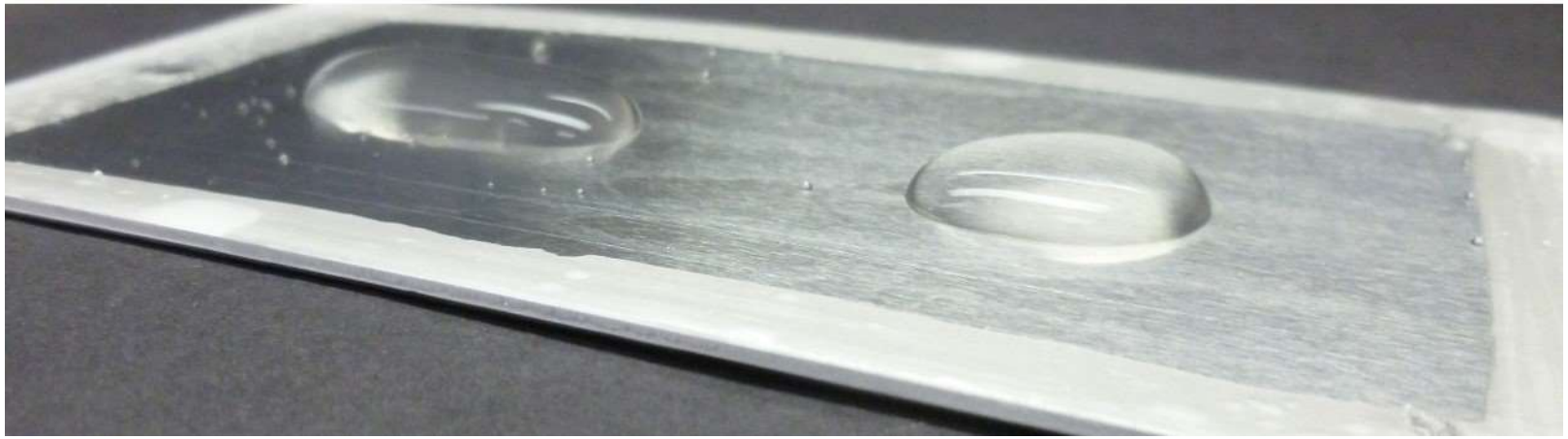
Bipodal Silane #1 Hydrophobizes Metal Surfaces

Contact Angle: ~44°



H_2O on Untreated Aluminum

Contact Angle: ~72°



H_2O on 2% Bipodal Silane #1-treated Aluminum

Bipodal Silane #1 Crosslinks Sol-gel Silane System at Lower Temperatures

2% Sol-gel Silane System + 2% Bipodal Silane #1

4% Sol-gel Silane System

23°C cure (48 hours) | **80°C cure (30 min)** | **180°C cure (30 min)**

After 250h in neutral SST

Aluminum T6061

Bipodal Silane #1 Crosslinks Sol-gel Silane System at Lower Temperatures

2% Sol-gel Silane System + 2% Bipodal Silane #1

4% Sol-gel Silane System

23°C cure (48 hours) | **80°C cure (30 min)** | **180°C cure (30 min)**

After 1000h in neutral SST

Aluminum T6061

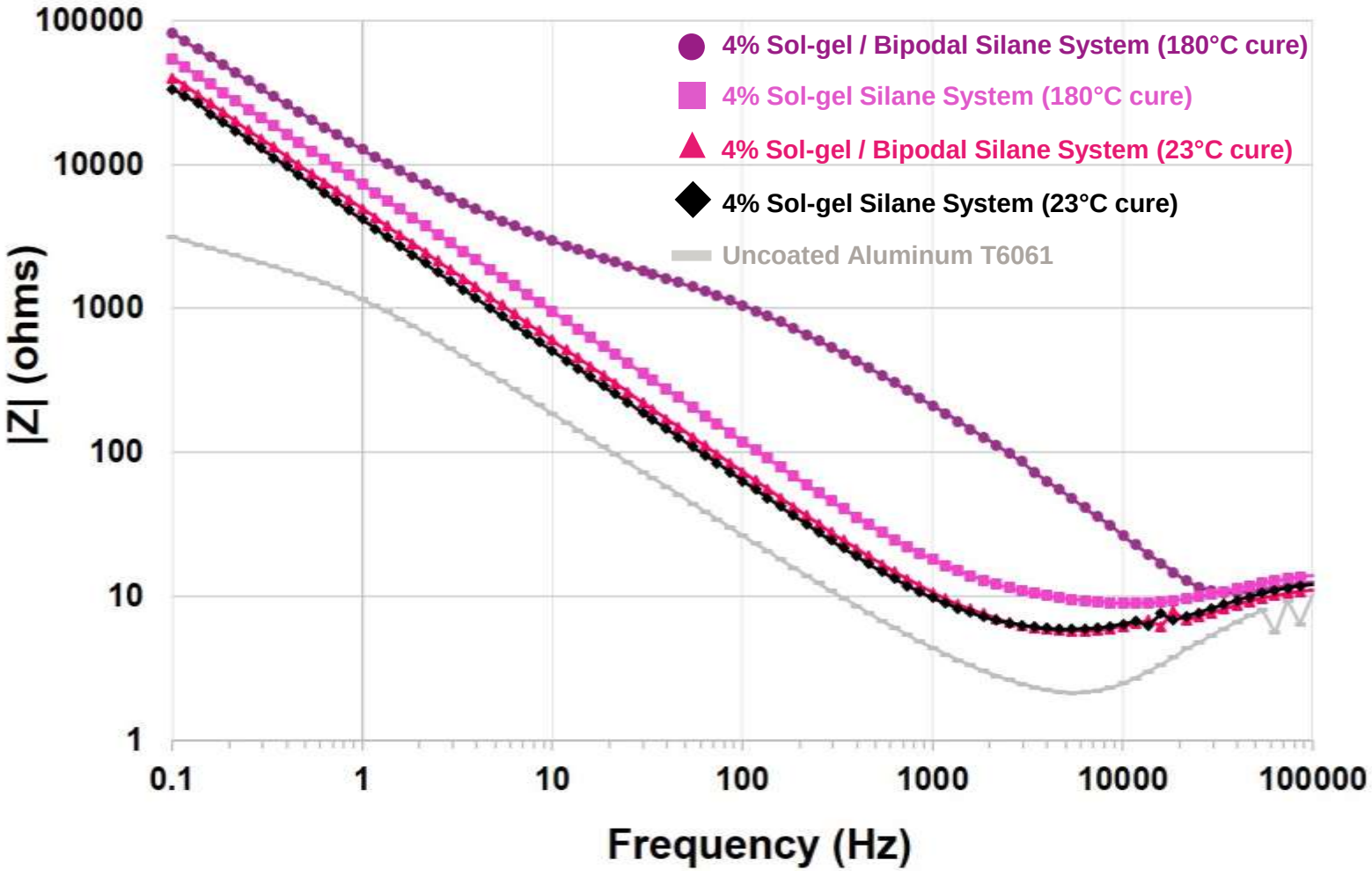
Electrochemical Impedance Spectroscopy (EIS) of Water-borne Sol-gel Systems

Background & Benefits of EIS

- Measures resistance & capacitance of coated metals in salt-based solution
- Higher impedance of coating, |Z|, indicates a more efficient barrier to surrounding water & salts

Analysis of Results

- At 0.1 Hz, Bipodal Silane #1 improved the impedance of the thermally cured system by **34%**
- Thermal curing procedure more effective at improving impedance than Bipodal Silane #1



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Improving Resistance to Alkaline Media with Bipodal Silanes

Importance of Alkaline Resistance

- Alkaline detergents commonly used for car wash applications
- Crucial performance requirement for automotive coatings industry

Testing Procedure

- Prepare 10% NaOH, 90% DI H_2O solution
- Coat Aluminum T6061 and cure at 180°C for 30m
- Immerse coated samples in alkaline solution
- Place alkaline-immersed samples in salt spray test for further evaluation
- GMW 14665 – General Motors Specification

After 6 minutes immersion in 10% NaOH solution

4% Sol-gel Silane System

2% Sol-gel Silane System + 2% Bipodal Silane #1

Improving Resistance to Alkaline Media with Bipodal Silanes

2% Sol-gel Silane System
+ 2% Bipodal Silane #1

4% Sol-gel Silane System

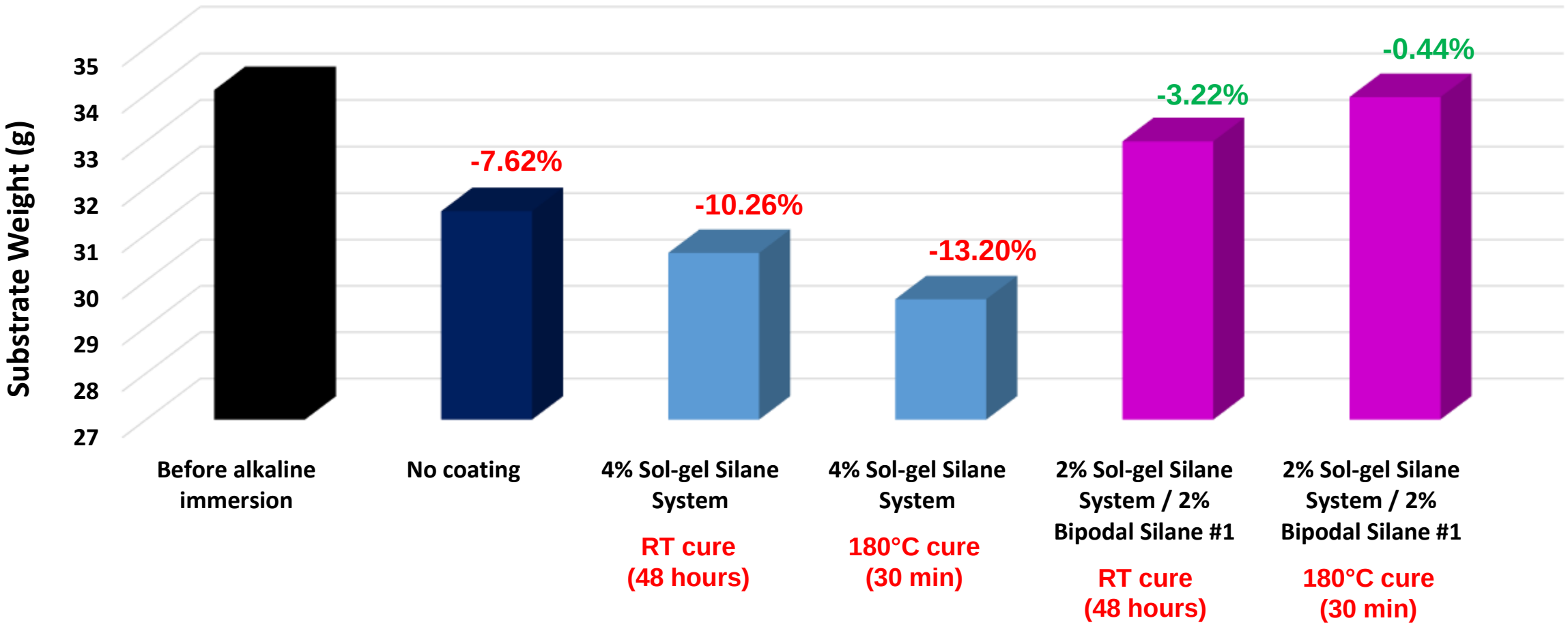
No alkaline immersion

10 min alkaline immersion

After 100h in neutral SST

Aluminum T6061

Weight Loss Comparisons of Post-Alkaline Test Aluminum 2024 Substrates



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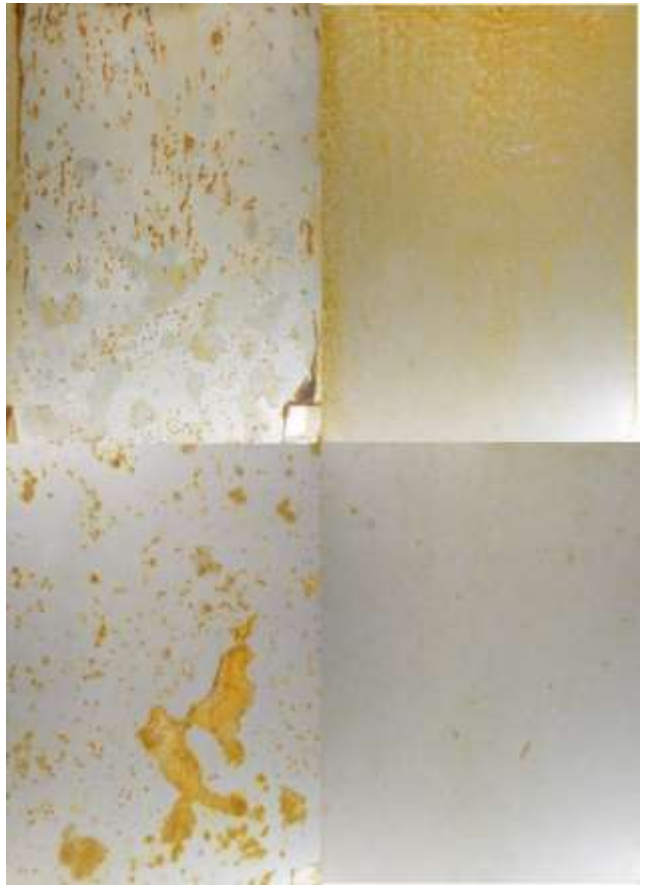
Improving Weatherability of Coated Cold Rolled Steel with Bipodal Silanes

x 4

x 3

10% Sol-gel Silane System

5% Sol-gel Silane System
5% Bipodal Silane #1



RT cure (48 hours)

180°C cure (30 min)

After 7 days left outside in NJ

Cold-rolled Steel

No coating

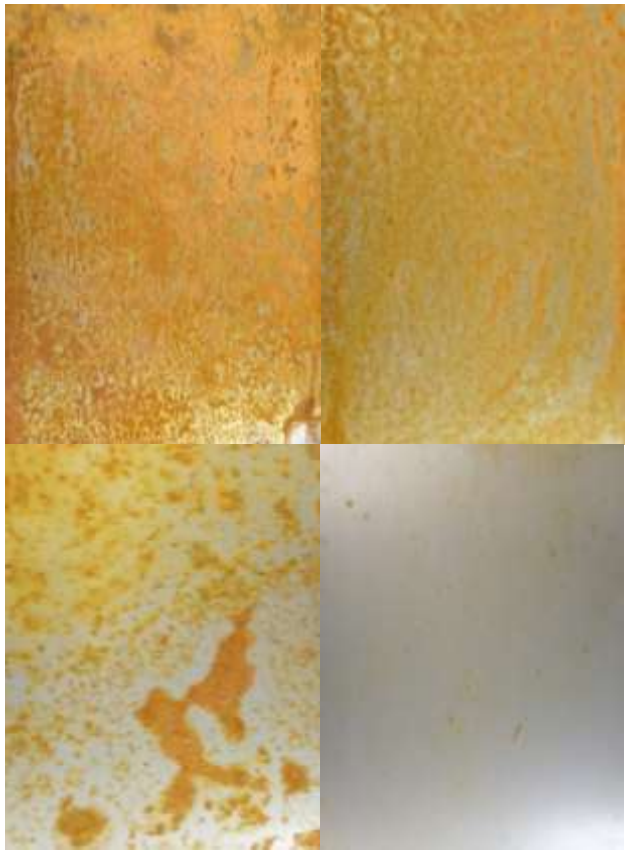
Improving Weatherability of Coated Cold Rolled Steel with Bipodal Silanes

x 9

x 5

10% Sol-gel Silane System

5% Sol-gel Silane System
5% Bipodal Silane #1



RT cure
(48 hours)

180°C cure
(30 min)

After 14 days left outside in NJ

Cold-rolled Steel

No coating

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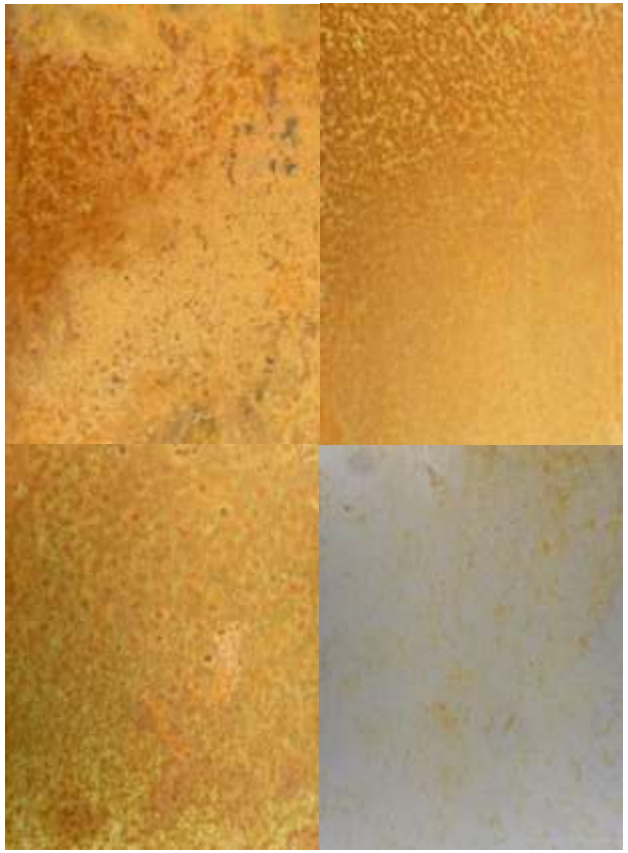
Improving Weatherability of Coated Cold Rolled Steel with Bipodal Silanes

x 21

x 9

10% Sol-gel Silane System

5% Sol-gel Silane System
5% Bipodal Silane #1



RT cure (48 hours)

180°C cure (30 min)

After 30 days left outside in NJ

Cold-rolled Steel

No coating

Conclusion & Future Work

Conclusion

- Bipodal Silanes can offer:
  - Improved hydrophobicity and corrosion resistance in water-borne coatings
  - Increased crosslinking in water-borne sol-gel silane systems at lower than required temperatures
  - Enhanced resistance to alkaline media

Future Work

- Evaluate other Organofunctional Silanes for room-temperature curing of water-borne corrosion protection technology
- Optimize corrosion protection on other metals (CRS, HDG Steel, other Aluminum alloys, etc……)
- Outdoor weatherability testing in various regional environments

For Further Assistance

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