AGC Chemicals Americas, Inc.

"FEVE Hybrid Fluoropolymer Technology – Still the New Face in High Performance Coatings"

Presented by:

Bob Parker

FEVE Resins – Still the New Face in Coatings

OUTLINE OF WEBINAR

- *Overview of "FEVE" resin technology*
- *Introduction of FEVE resin family*
- *Commercial History of FEVE resins*
- *Low VOC Potential for FEVE resin –based coatings*
- *15 year exposure test and aged film analysis*
- *20 year exposure test of FEVE coatings and PVdF coatings and SEM photographs*
- *Exposure results of FEVE emulsion-containing coatings – QUV and Xenon Arc Weatherometer*
- Achieving 50 g/l VOC with low-VOC coalescents

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FEVE Resins – Still the New Face in Coatings

FEVE Fluoropolymer Resins

- **"FEVE" is an abbreviation for "Fluoroethylene Vinyl Ether" resins for coatings**

- **Known for their high performance properties:**

1) Exceptional resistance to UV degradation
2) Superior Chemical Resistance
3) Excellent Thermal Resistance

- **Known for their unique formulation properties:**
  1) Solvent soluble; unique among fluorinated polymers
  2) Can be cured at ambient temperature with isocyanates
  3) Can achieve high gloss formulations

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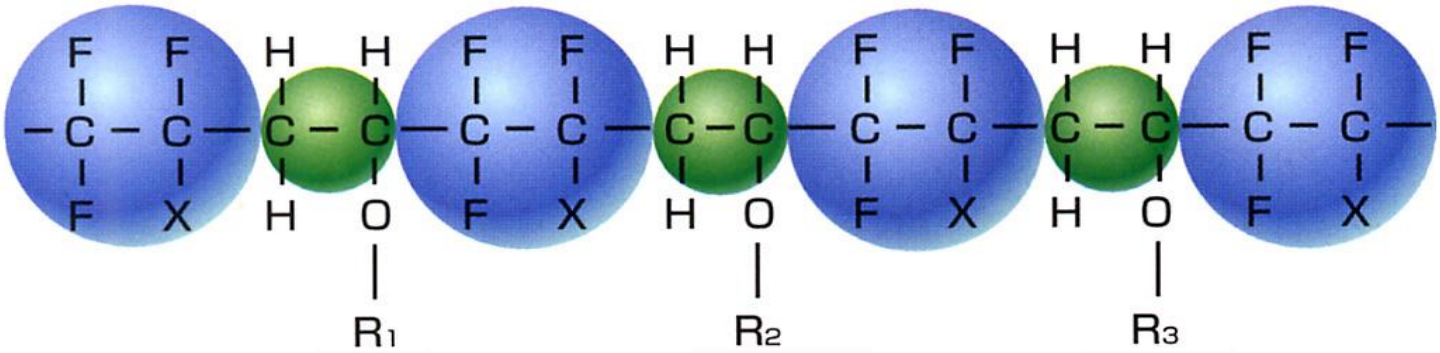
Fluoroethylene Vinyl Ether (FEVE) Resins

Fluoro Ethylene

Vinyl Ether



FLUORINATED SEGMENTS: Weatherability, durability, chemical resistance

VINYL ETHER SEGMENTS: Gloss, solubility, crosslinking

FEVE Resins – Still the New Face in Coatings

Commercial Types of "FEVE" Resins:

1) Solvent soluble resins –

uses organic solvents for coatings formulation;
air dry cure with isocyanates; high temperature cure
with blocked isocyanates and melamines;
available as resin solutions or as 100% solid resins
(for low VOC formulations or powder coatings)

2) Water-based emulsions –

use vinyl ether macromonomers containing
polyoxyethylene (EO) units to create stable emulsions

3) Water-based dispersion –

addition of carboxyl groups for amine neutralization

FEVE Resins – Still the New Face in Coatings

1982: FEVE resin commercialized

1991: Powder-grade introduced

1992: 1K solvent resin and emulsions introduced

2002: High Tg Powder-grade introduced

2008: Higher OH-functionality resins introduced

2012: Waterborne dispersion introduced

FEVE Resin Timeline

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FEVE Resins – Still the New Face in Coatings

Solvent Soluble FEVE resins – Properties

- Hydroxyl Equivalent weights range from 330 to 1800
- Tg ranges from 20°C. to 51°C.
- VOC compliant formulations are attainable
- Can be blended with many acrylic and polyester polyols
- Can be blended with reactive diluents
- Solid resins can be dissolved in a wide variety of solvents, including exempt solvents

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Higher MW Solid FEVE Resin – Solubility in Exempt Solvents

Solvent	Resin Solids by Weight	Brookfield Viscosity
T-Butyl Acetate	50	869
Acetone	60	975
Oxsol 100	50	6632
Dimethyl Carbonate	50	825
Propylene Carbonate	Insoluble	

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FEVE Resins – Still the New Face in Coatings

Lower MW Solid FEVE Resin – Solubility in Exempt Solvents

Solvent	Resin Solids by Weight	Brookfield Viscosity
T-Butyl Acetate	65	2074
Acetone	65	240
Oxsol 100	50	1748
Dimethyl Carbonate	50	70
Propylene Carbonate	Insoluble	

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Water-based OH-functional FEVE Emulsion – Properties

- Equivalent Weight of emulsion solution is 2290
  (50% weight solids in H_2O)
- MFFT = 55°C
- Particle Diameter range is 0.1-0.2 µm
- Appearance is milky-white
- Needs coalescing solvents to form ambient-cured film
- Does not have shear stability for pigment dispersion
- Can be crosslinked with isocyanates or amino resins
- Molecular weight matches typical acrylic emulsions used in 1K coatings

FEVE Resins – Still the New Face in Coatings

Water-based 1K FEVE Emulsion – Properties

- 50% weight solids in H_2O
- MFFT = 35°C
- Particle Diameter range is 0.1-0.2 µm
- Appearance is milky-white
- Needs coalescing solvents to form ambient-cured film
- Does not have shear stability for pigment dispersion
- Can only be used for 1K coatings
- Shows excellent compatibility with a wide variety of acrylic emulsions
- Molecular weight matches typical acrylic emulsions used in 1K coatings

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Water-based Dispersion – Properties

- Hydroxyl Equivalent Weight of dispersion solution is 1650 (40% weight solids in H_2O)
- MFFT = 27°C.
- Particle Diameter range is 0.05 – 0.3 µm
- Appearance is translucent liquid
- Can achieve “0” VOC formulations
- Does not have shear stability for pigment dispersion
- Molecular weight is too low for 1K coatings

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FEVE powder resin:

- Appearance is light yellow flake
- Tg = 50-55^{o} C
- Softening Point: c.a. 90^{o} C -136^{o}C
- Hydroxyl Equivalent Weight is 1058

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15 YEAR EXPOSURE TEST IN JAPAN

Sealed Portion of Test panel

Test Panels used for SEM Scans and IR Imaging

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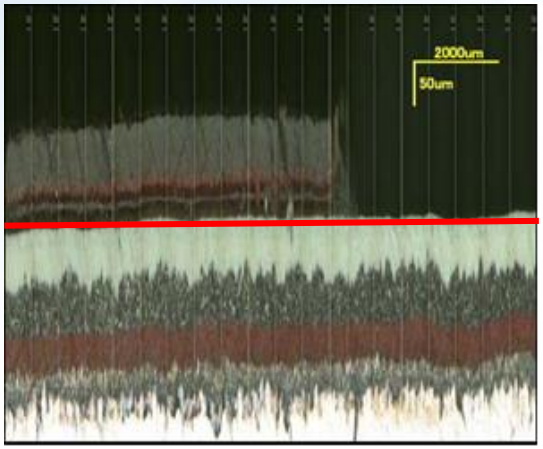
FEVE Resins – Still the New Face in Coatings

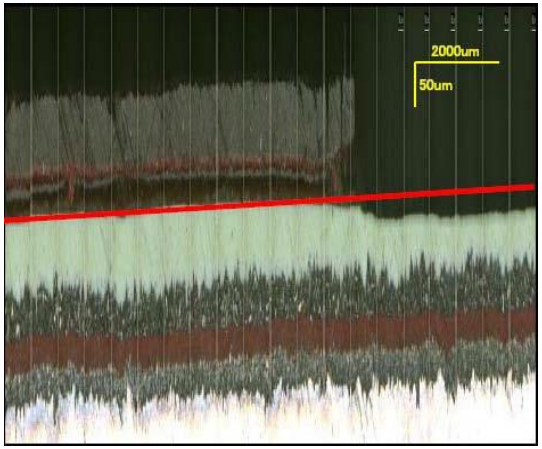
SEM Scans of Exposure Panels

FEVE White Coating



0-1.1 microns of erosion
after 15 years of exposure

Standard Polyurethane White Coating

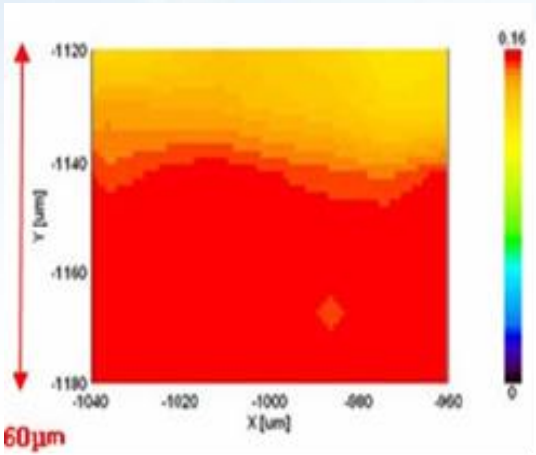


22-28 microns of erosion
after 15 years of exposure

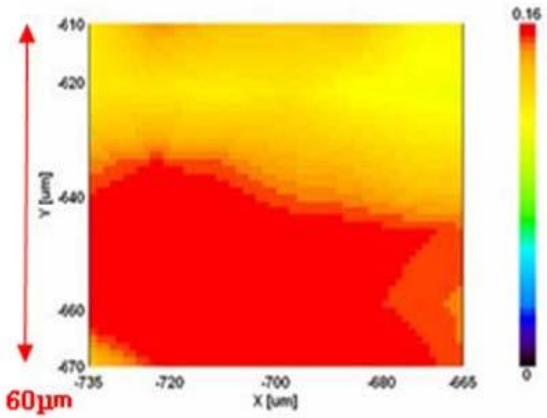
FEVE Resins – Still the New Face in Coatings

Measurement of Urethane Bond Retention – Infrared Analysis

Fluorourethane – Sealed Surface



Fluorourethane – Exposed Surface

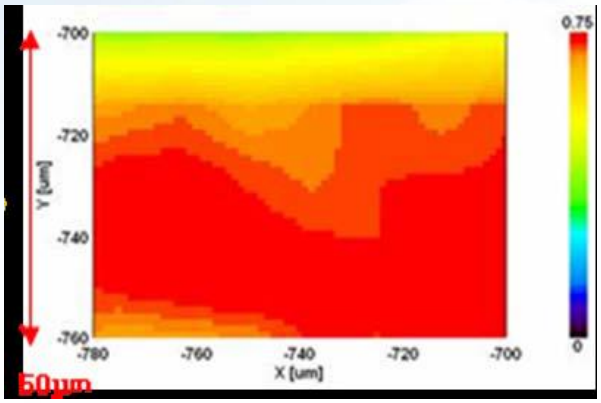


Deep Red Color is indication of >80% Retention of Urethane Bond

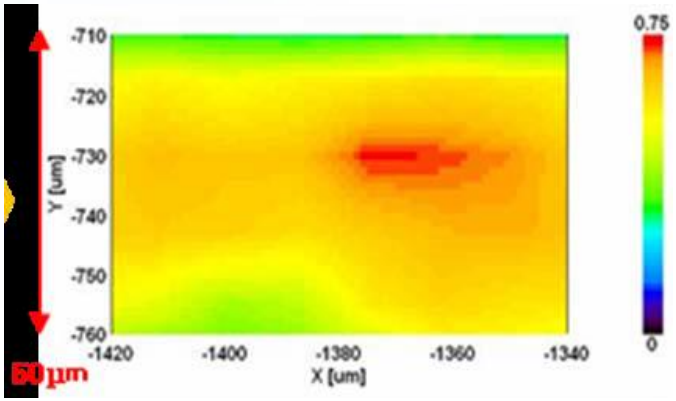
FEVE Resins – Still the New Face in Coatings

Measurement of Urethane Bond Retention – Infrared Analysis

Conventional Urethane – Sealed Surface



Conventional Urethane – Exposed Surface



Yellow Color is indication of < 60% Retention of Urethane Bond

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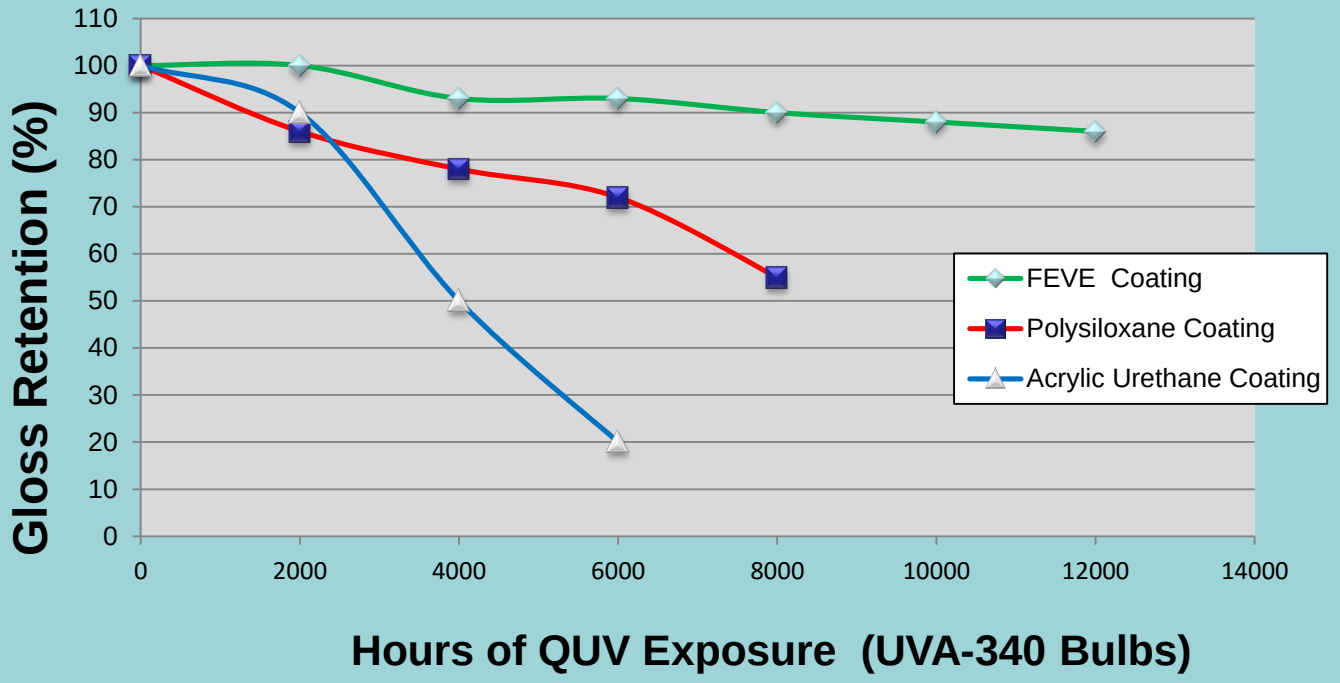
QUV Weatherometer Exposure Testing



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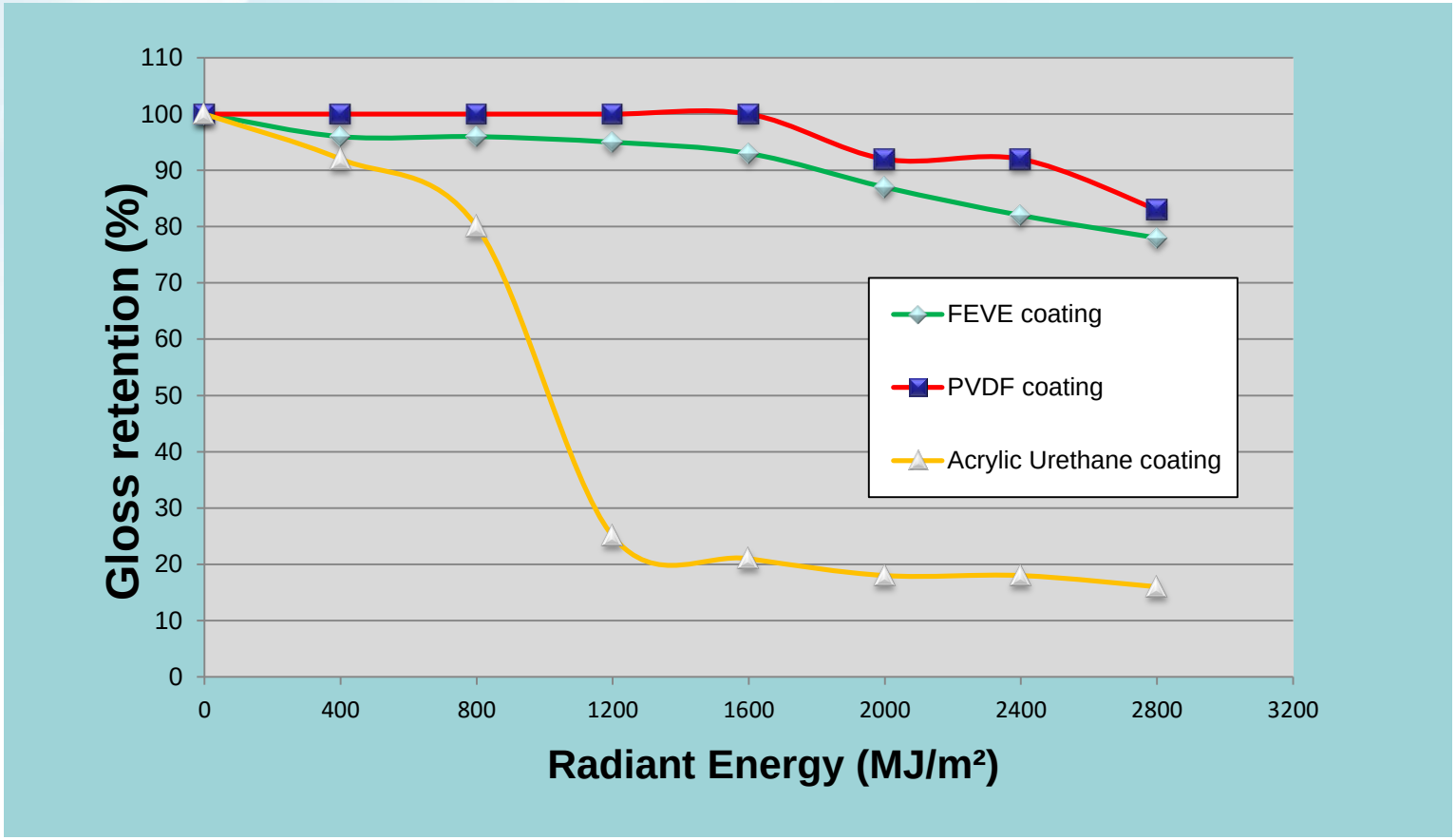
EMMAQUA Testing (Outside Test Fence – Arizona)



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Twenty Year Exposure of 2 Commercial Fluorinated Coatings

Sample ID	Fluorinated Resin Type	Paint System	Color	Applicator
Sample A	PVdF	2 coat system; epoxy primer and PVdF topcoat	Brown	Taiyo Steel
Sample B	FEVE	1 coat system	Brown	Nippon Steel

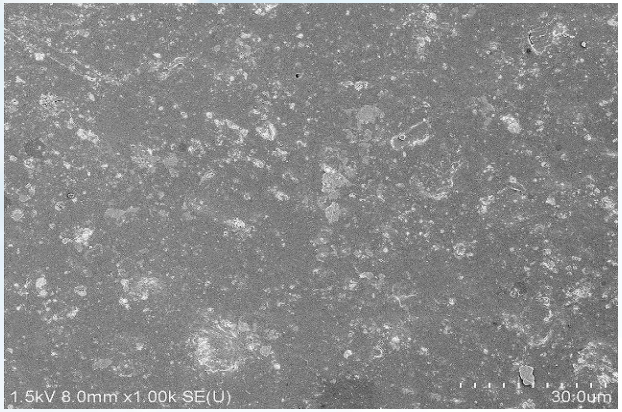
FEVE Resins – Still the New Face in Coatings

Twenty Year Exposure of 2 Commercial Fluorinated Coatings

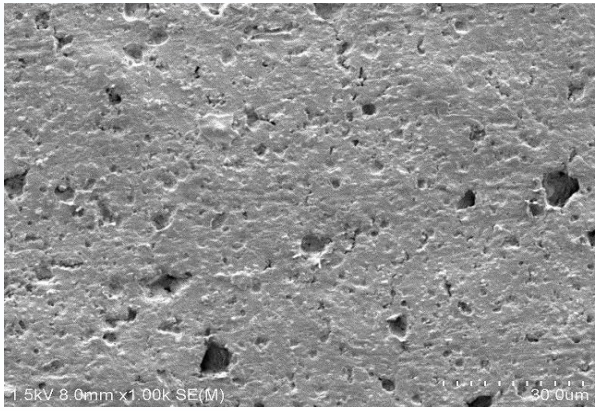
Sample ID	Sample A (PVdF)		Sample B (FEVE)	
Length of Exposure	Initial Measurement	After 20 year exposure	Initial Measurement	After 20 year exposure
60^{o} Gloss	24	12	74	41
Gloss Retention (%)	100	52	100	49

FEVE Resins – Still the New Face in Coatings

SEM Imaging of Surface

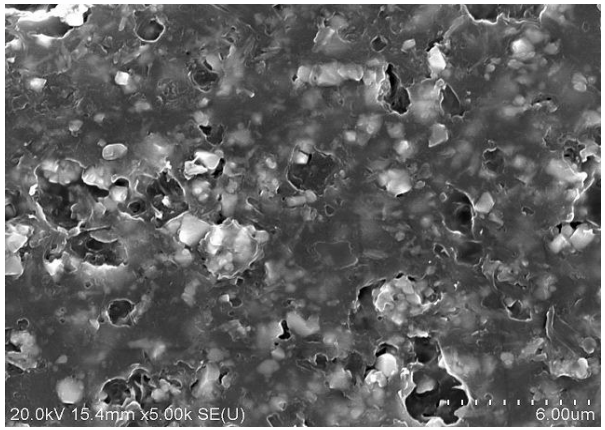


Initial Scan
(PVdF) -1000X



PVdF Scan after 20
years exposure – 1000X

PVdF Scan after 20
years exposure
5000X



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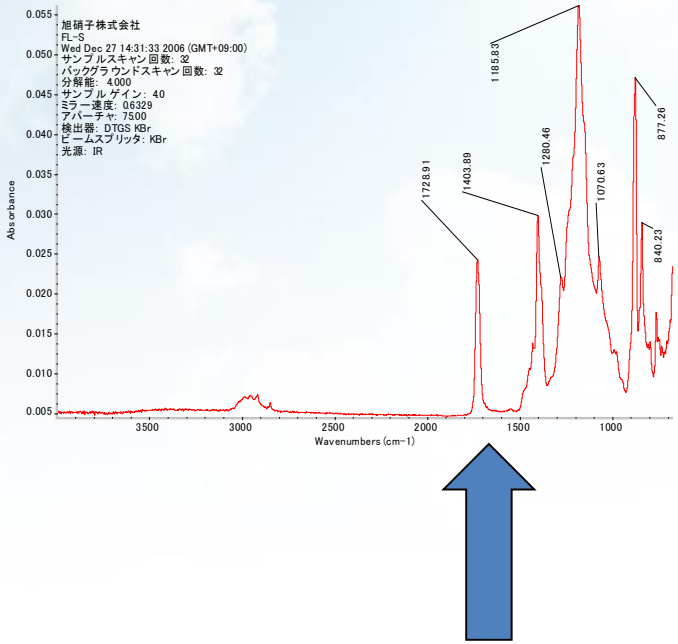
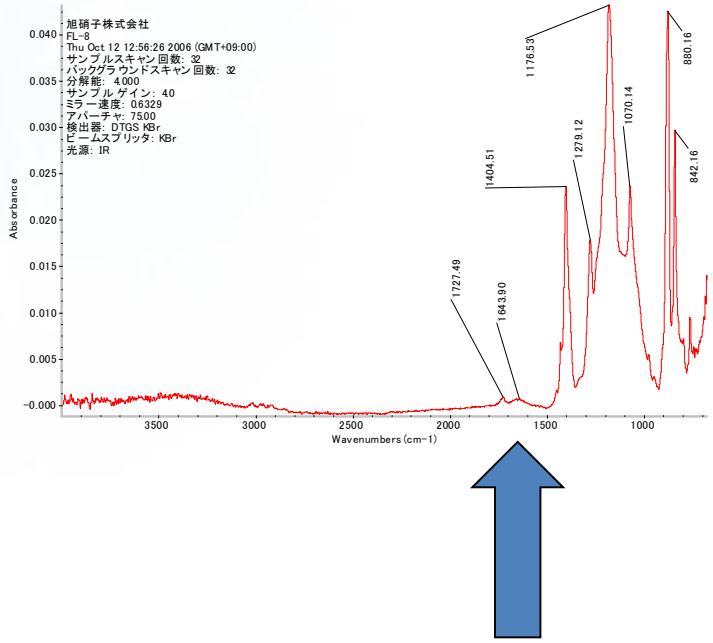
FTIR Scans of PVdF Coatings

Scan before exposure



C=O Bond Peak
is indication of Acrylic Resin

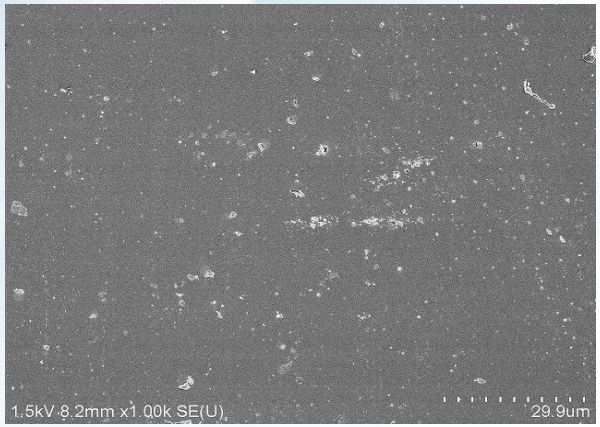
Scan After Exposure



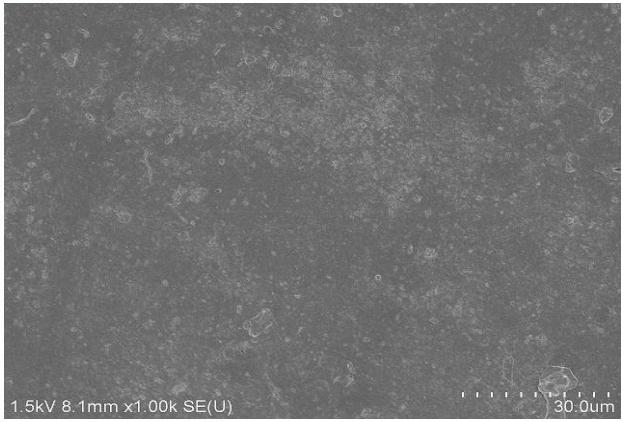
Absence of peak after
20 years exposure

FEVE Resins – Still the New Face in Coatings

SEM Imaging of LUMIFLON Surface

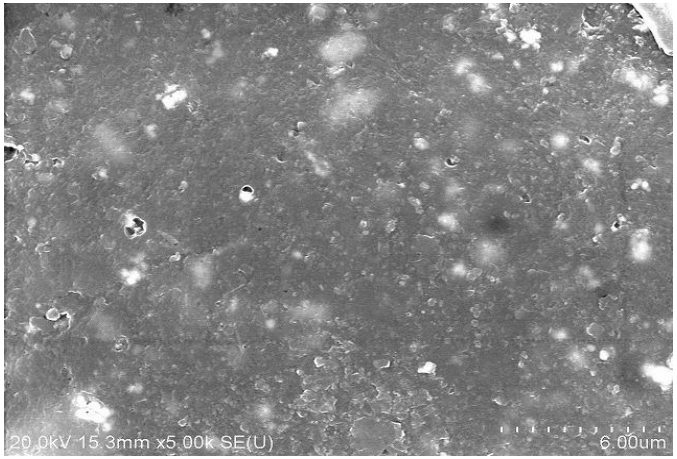


Initial Scan (FEVE) -1000X



FEVE Scan after 20 years exposure – 1000X

FEVE Scan after 20 years exposure 5000X



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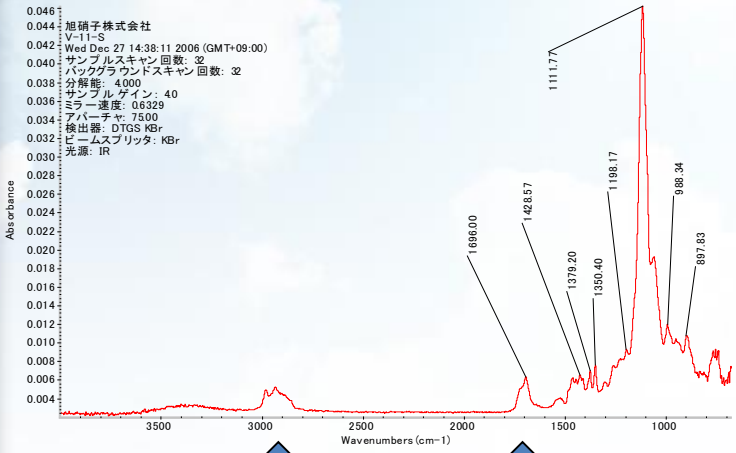
FTIR Scans of FEVE Coatings

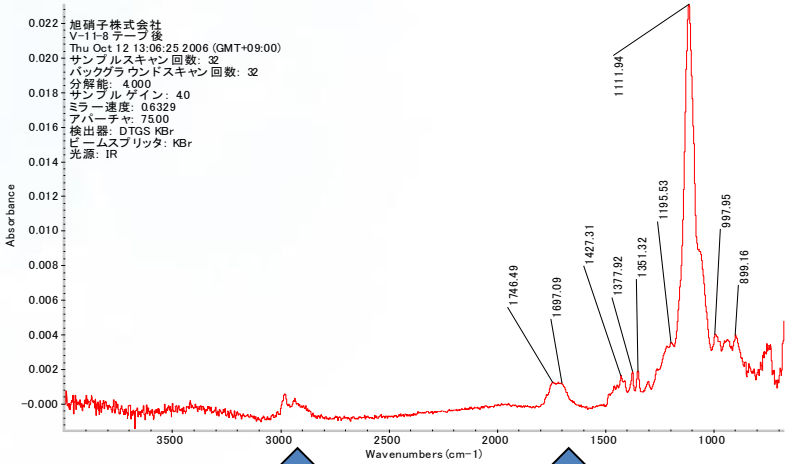
Scan before exposure



Aliphatic CH & CH_2 peak

C=O Bond Peak for urethane

Scan After Exposure



Both Peaks still present after 20 years exposure

FEVE Resins for Long-Life Coatings

Medium Green Finish Coat for Exterior Steel - Formulation Properties	
Solids (Volume)	44.2%
FEVE Emulsion	FE-4300
Acrylic Emulsion Source	Hexion
pH	8.3-8.7
Color Standard	FED. 595B #14223
TiO_2 Choice	Ishihara PFC-105
PVC	14.6-15.5
Thickeners	associative-type
60° Gloss Range	29-33
VOC	100 g/liter

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Testing procedure :

1) Use Customer's Formulation as the template to manufacture test coatings.

2) Manufacture 3 ***Medium Green Finish Coat*** formulations:

   a. Binder = *100% Acrylic Emulsion*

   b. Binder = ***70%*** *Acrylic Emulsion + **30%** FE-4300 (FEVE Emulsion)*

   c. Binder = ***55%*** *Acrylic Emulsion + **45%** FE-4300 (FEVE Emulsion)*

3) Preparation of test panels (Al panels coated with 8 wet mils of coating)

4) QUV Weatherometer Exposure (UVA 340 Bulbs used)

   a. Test Cycle = 8 hours UV light @ 60°C. + 4 hours condensation @ 50°C.

5) Xenon Arc Weatherometer Exposure

   a. Test Cycle = modified ASTM G155 - Cycle 1 with 0.55 W/(m²·nm)

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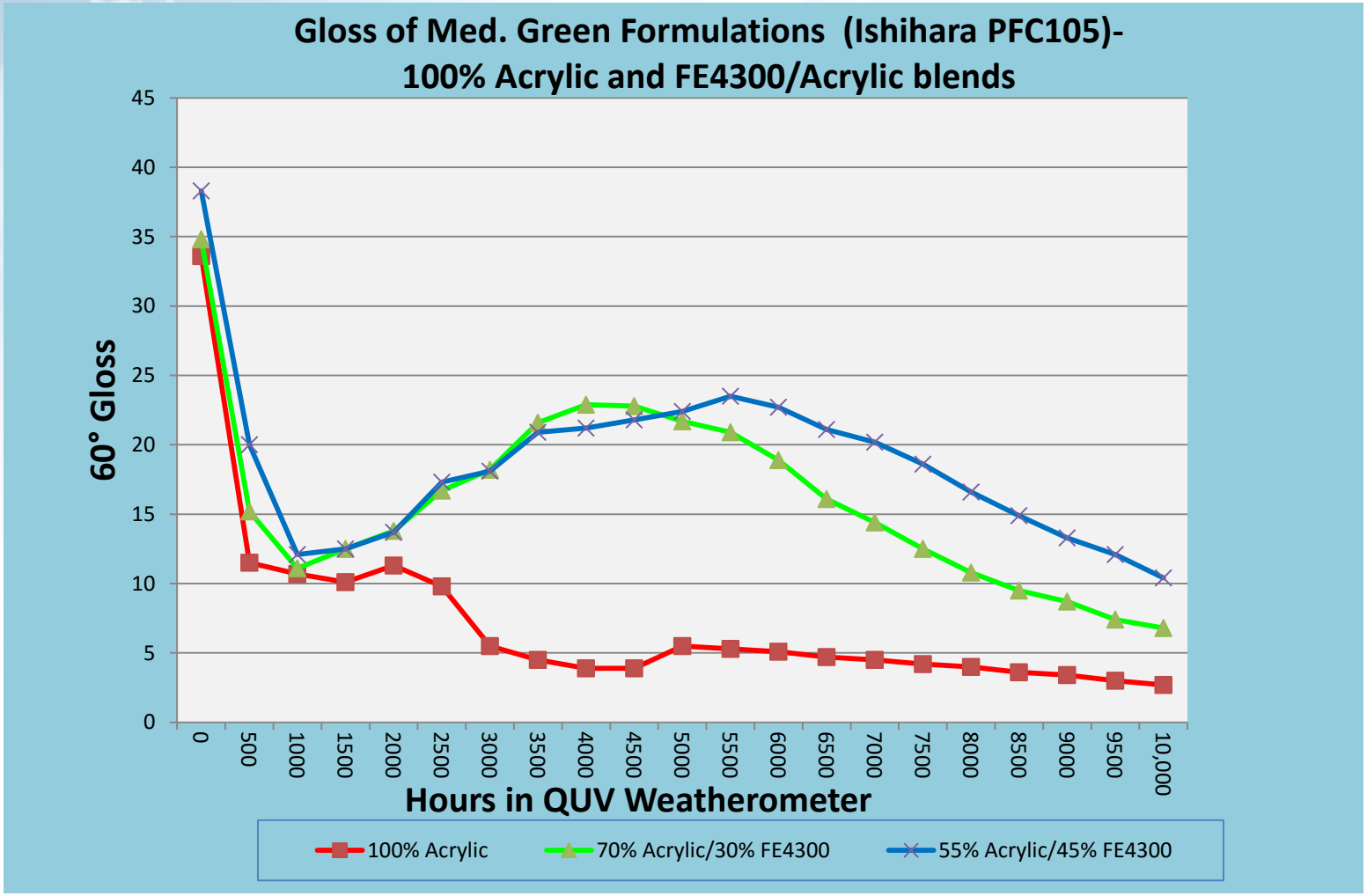

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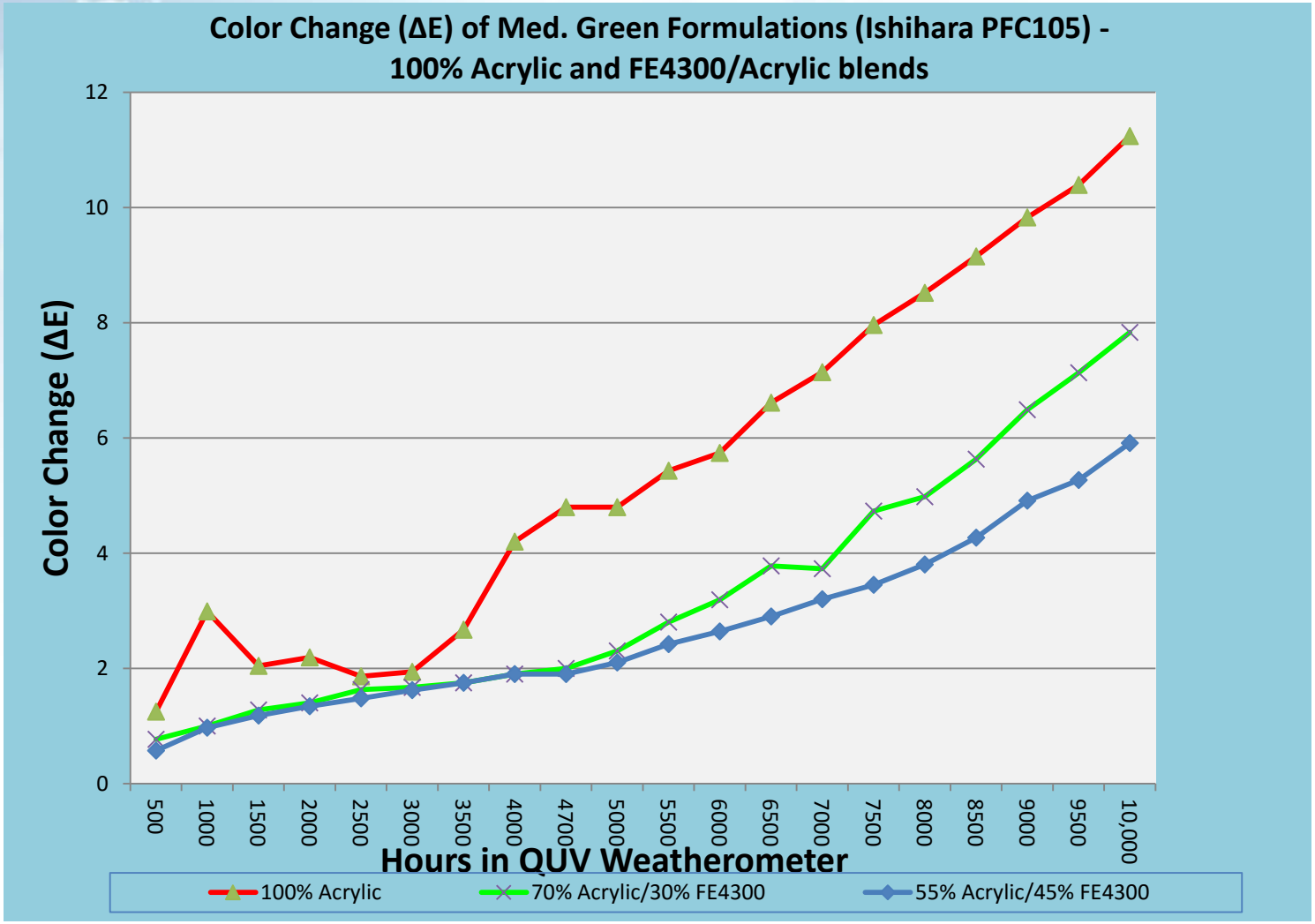

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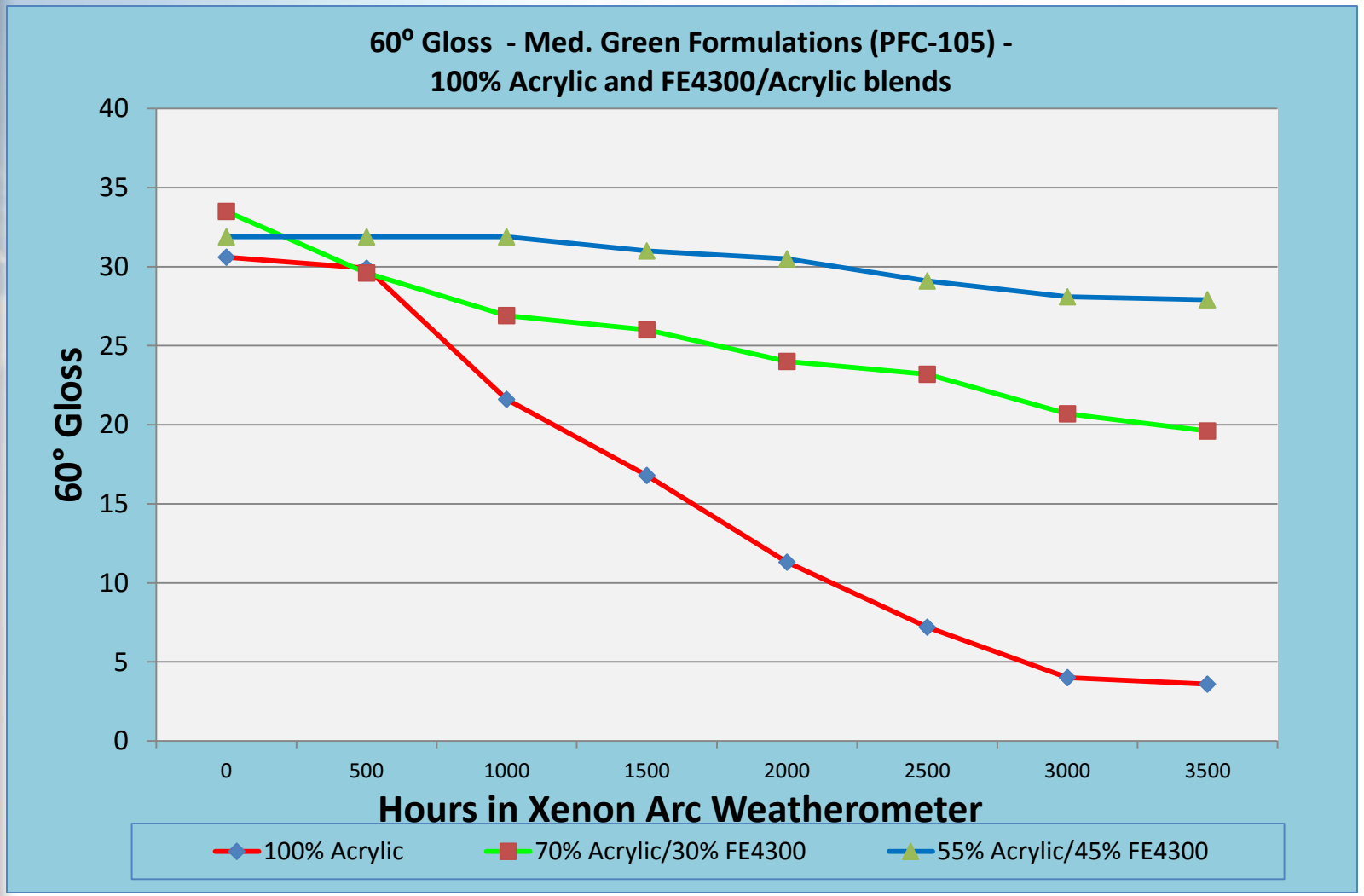

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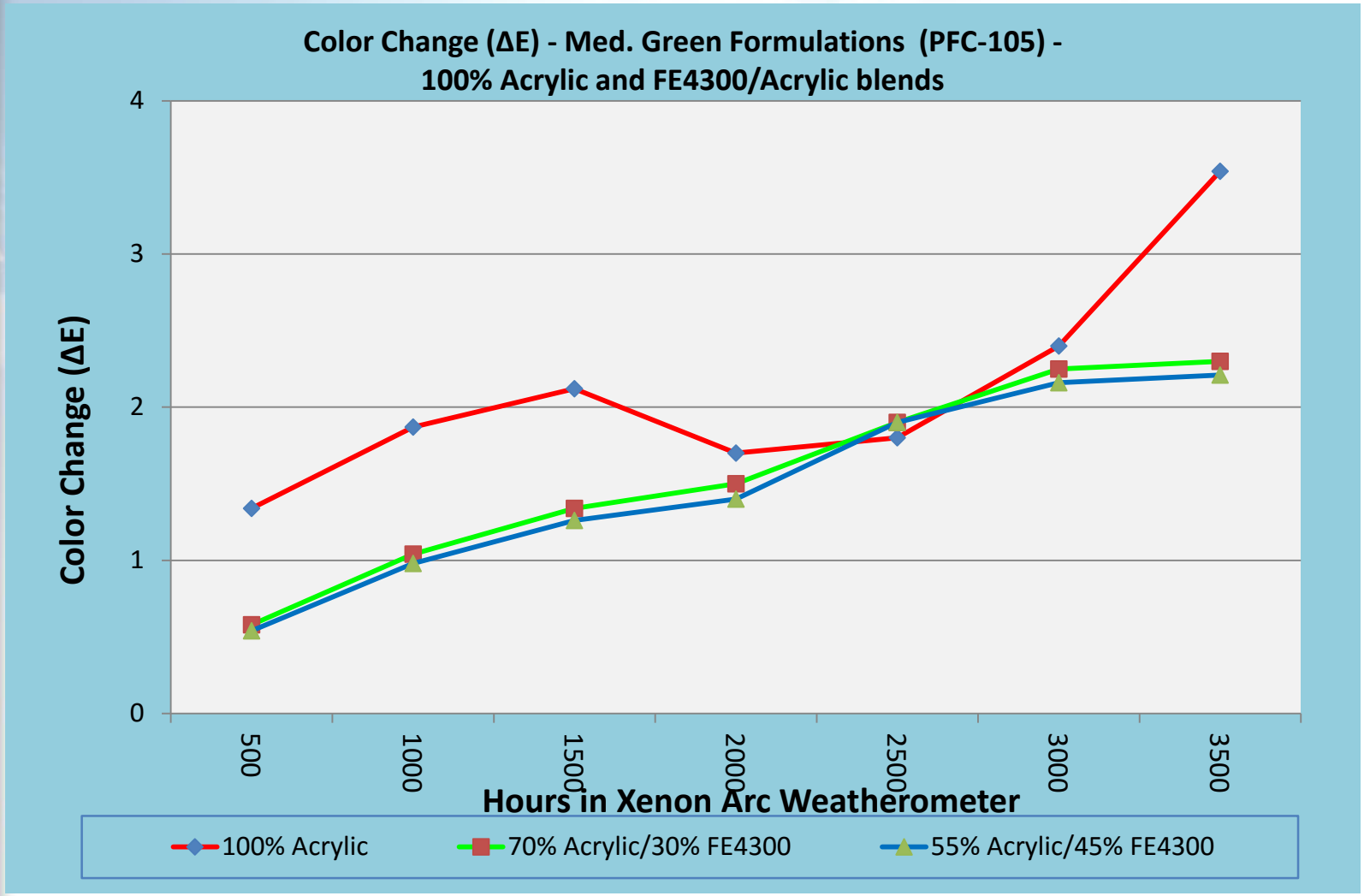

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Summary of Testing

- Testing Clearcoat formulations using 100% FE4300
- Testing film forming properties at 77°F. (Ambient) and at 35°F.
- Blending standard coalescing solvents with low-VOC coalescing solvents to achieve a solvent level of 10% based on resin solids
- Maintaining a maximum VOC of 50 grams/liter for the formulation
- "PASS" in the "Film Formation" columns signifies a complete film formation without the presence of any cracking in the film
- "FAIL" in the "Film Formation" column signifies the presence of any cracking in the film

FEVE Resins – Still the New Face in Coatings

FE4300 Clearcoat Formulations (Total solvent Level is 10% on resin solids)					
Formula #	***VOC (grams/liter)***	***Standard Coalescent***	***Low-VOC Coalescent***	***Film Formation at 77°F. (Ambient)***	***Film Formation at 35°F.***
510/1-3	50	Texanol	Eastman Optifilm 400	PASS	FAIL
510/1-4	50	Dowanol DPM	Eastman Optifilm 400	PASS	FAIL
510/1-5	50	Dowanol DPnP	Eastman Optifilm 400	PASS	PASS
510-1-6	50	Dowanol DPnB	Eastman Optifilm 400	PASS	PASS

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FE4300 Clearcoat Formulations (Total solvent Level is 10% on resin solids)					
Formula #	***VOC (grams/liter)***	***Standard Coalescent***	***Low-VOC Coalescent***	***Film Formation at 77°F. (Ambient)***	***Film Formation at 35°F.***
513/1-1	50	Dowanol DPnB	Eastman TEG-EH Plasticizer	PASS	PASS
513/1-2	50	Dowanol DPnP	Eastman TEG-EH Plasticizer	PASS	PASS
513/1-3	50	Dowanol DPM	Eastman TEG-EH Plasticizer	PASS	FAIL
513/1-4	50	Texanol	Eastman TEG-EH Plasticizer	PASS	FAIL

FEVE Resins – Still the New Face in Coatings

FE4300 Clearcoat Formulations (Total solvent Level is 10% on resin solids)					
Formula #	***VOC (grams/liter)***	***Standard Coalescent***	***Low-VOC Coalescent***	***Film Formation at 77°F. (Ambient)***	***Film Formation at 35°F.***
516/1-1	50	Dowanol DPnB	Eastman Velate 375	PASS	FAIL
516/1-2	50	Dowanol DPnP	Eastman Velate 375	PASS	FAIL
516/1-3	50	Dowanol DPM	Eastman Velate 375	PASS	FAIL
516/1-4	50	Texanol	Eastman Velate 375	PASS	FAIL

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FE4300 Clearcoat Formulations (Total solvent Level is 10% or 15% on resin solids)					
Formula #	**VOC (grams/liter)**	**Standard Coalescent**	**low-VOC Coalescent**	**Film Formation at 77°F. (Ambient)**	**Film Formation at 35°F.**
572/1-7	50	Dowanol DPnB	EPS 9147	PASS	PASS
572/1-8	50	Texanol	EPS 9147	PASS	FAIL
572/1-9 *	50	Dowanol DPnB	EPS 9147	PASS	PASS
572/1-10 *	50	Texanol	EPS 9147	PASS	PASS
* These formulations have a total of 15% solvent on resin solids					

FEVE Resins – Still the New Face in Coatings

Summary:

Inclusion of FEVE resins in architectural and industrial coating formulations can increase gloss retention, color retention, and the overall durability of the dry film. These characteristics will prolong both the aesthetic properties and the protection properties of these coatings, thereby increasing the time frame between recoats.