

e often think of technology in terms of software, computers, robotics or other tangible machinery that automates some part of our daily lives.

However, advancements often occur at the atomic or chemical level, bringing us new or improved technologies. Chemists in the powder coating industry continually seek ways to improve the durability and fade resistance of powder coatings. Fluoroethylene Vinyl Ether copolymer (abbreviated helps with corrosion, chemical and UV resistance. The as FEVE copolymer) was developed in 1982 by Asahi Glass. FEVE is composed of thermoset polymers, which can be easily processed through the typical thermoset powder coating equipment. Different than polyvinylidene difluoride (PVDF) powder coatings, no liquid nitrogen is needed for obtaining FEVE powders. Thermoplastic PVDF is tricky to process when used as a polymer for exterior durable powder coatings. As a thermoplastic polymer, crystallinity of PVDF changes in the extruder and a managed cooling period is required after compounding. For some applications, such as architectural, this can make FEVE a better choice for powder coatings than other fluoropolymers. FEVE powder coating can be cured at 200 degrees C (392 degrees F) or lower. This is critical for American Architectural Manufacturers

Association (AAMA) coatings since aluminum substrates cannot sustain high heat needed by other fluoropolymers, e.g., PTFE and ETFE, for film formation.

FEVE resins have a unique chemical structure, which vinyl ether groups make FEVE polymers useable as resins for powder coatings by providing high gloss, hardness, transparency, and flexibility, as well as pigment compatibility and adhesion. The vinyl ether groups also allow for functional groups, like hydroxyl groups, to be incorporated into the structure for crosslinkability. Powder coatings based on FEVE have been used in the AAMA 2605 applications.

Over 10 years ago, panels coated with FEVE and superdurable polyester blends were sent to South Florida for outdoor exposure tests (courtesy of Royal DSM N.V.). Gloss and color retention were measured over the duration of the tests. The results are promising for those advocating greater expansion of super-durable polyester powder use in the AAMA 2605 architectural coatings market.

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FEVE and Polyester Blend Formulations and the Coating Process

Formulations of typical FEVE white powder coating are shown in Figure 1. In the FEVE/polyester blend formulation, FEVE is substituted with up to 50% by weight of super-durable polyester. The same process conditions for pure FEVE powder coating can be applied to the blends. FEVE and polyester resins are pre-blended together with other ingredients. Extrusion can be done on a twin-screw extruder. Extrusion conditions are: 120 degrees C, 250 rpm, and 50% shear. After milling and

Ingredients	Description	Parts by Weight
FEVE	resin	56
B1530	cross linker	13
BYK360P	flow agent	2
Benzoin	degassing agent	0.4
R960	TiO ₂	35
Tinuvin 144	UV stablizer	0.52
Tinuvin 405	HALS	1.04
DBTDL 1/100	catalyst	0.21
Total		108

Figure 1. FEVE white powder coating formulations.

sieving, formulated powders are electrostatically coated on the substrate and the coated panels are cured at 200 degrees C for 20 min.

In high durable exterior coating applications, it is a challenge to formulate coatings containing high levels of titanium dioxide (TiO₂). In this case, the detrimental effect of TiO₂ in resin degradation through photocatalytic reaction outweighs the positive role of TiO₂ as a UV absorber. In general, it is even more challenging to formulate powder coatings with high TiO₂ loading than liquid coatings for high durable exterior applications due to the process difference.

Coating Gloss and Accelerated HE-XWM Weathering Test

Gloss is a measure of coating smoothness on the optical scale of 0.5 micron. In AAMA 2605 standards, gloss retention needs to remain 50% or more after exposure to the South Florida weathering test for ten years. To speed up the weathering process, accelerated weathering tests like QUV and Xenon Arc, are normally used for product development. Correlating the results of accelerated tests to natural exposure is challenging.

Coating durability is interpreted by appearance (gloss and color) retention, not degradation (weight loss) even though the latter influences the former. The degree of



influence can vary depending on the grades of TiO_2 . As resin degrades on the coating surface, it erodes from the paint and is washed out through cleaning or rain. As a result, Pigment Volume Concentration (PVC) increases and gloss drops. The gloss decrease is especially severe as PVC passes CPVC (Critical Pigment Volume Concentration). Any property of TiO_2 or coating film which affects CPVC will also affect the relationship of resin degradation and gloss reduction.

Accelerated exposure tests were done through a Hybrid Exposure Xenon Weather Meter (HE-XWM), a method developed by Toyota Central R&D Labs., Inc. Testing results from the hybrid system have a very good correlation to the results of South Florida exposure. This is especially true for coatings with high TiO2 content. Unlike the traditional QUV and Xenon Arc accelerated tests, coating under the hybrid test is also subject to hydrogen peroxide spray. H₂O₂ reacts with electrons being generated through a TiO₂ photocatalytic reaction on the coating surface, producing HO• radicals. HO• radicals contribute to resin degradation. As a result, the radical level is much higher under the hybrid testing conditions than the traditional exposure tests without H₂O₂. The extra free radicals accelerate the degradation rate of the coatings. The total testing time of the hybrid system is only two hundred hours. Unlike QUV or other traditional accelerated weathering tests, HE-XWM test measures coating degradation through TiO2 photocatalytic reaction.

Correlation of HE-XWM Gloss Retention to Coating Morphology

Figure 2 shows the results of gloss retention at 60 degrees of FEVE/polyester blend powder coatings after being exposed to accelerated HE-XWM and Okinawa natural exposure. Weathering conditions in Okinawa are

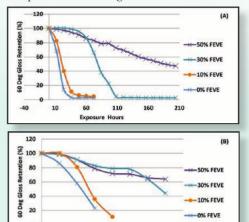


Figure 2. Gloss retention of FEVE/super-durable polyester formulations with different blend ratio (A) HE-XWM and (B) Okinawa.



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very similar to South Florida. Results from HE-XWM and Okinawa natural exposure show a good correlation. Super-durable polyester is used in the FEVE/polyester powder blends based on the formulations from Figure 1. FEVE is substituted by polyester up to 50% by weight, which improves the gloss retention of the polyester. Formulations with 50% super-durable polyester result in the best gloss retention. After 200 hours of HE-XWM exposure, the gloss retention remains at 50%. Gloss retention of the same formulation is 64% after being exposed in Okinawa for 7 years.

Performance of the FEVE/polyester blends depends on the choice of the super-durable polyester. Not all super-durable polyesters can be modified by FEVE for an improved performance. The solubility parameter of polyester must be different than that of FEVE. The bigger the difference, the better the chance to achieve the desired structure. Qualified polyester candidates include Uralac® P1550, P1651, P1680 and CRYLCOAT® 4890-0.

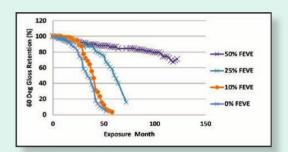


Figure 3. South Florida gloss retention of FEVE/ Uralac® P1550 formulations with different blend ratio.

Fifty percent Uralac® P1550 blending with FEVE passed the ten-year South Florida exposure test with gloss retention above 50% (Figure 3).

Coating morphology analysis was performed using scanning electron microscopy coupled with energy dispersive x-ray spectroscopy (SEM/EDX). Elemental mapping mode is used to test the coating cross section to detect phase separation, and spot mode in EDX is used to detect fluorine (F) and titanium (Ti) on the coating surface. The ratio of normalized atomic percent of F and Ti is used for data analysis.

As seen from Figure 3, when different amounts of FEVE are blended into polyester, cross section coating morphology changes. As learned from Figure 2, formulation with 50% of FEVE shows the best gloss retention. The morphology difference of this formulation, as observed from Figure 4, is a F-rich clear layer formed on the surface of the coating.

In Figure 5, both Formulation A and B contain 50% FEVE. The F-rich clear layer in Formulation A is thicker than that of Formulation B. The F to Ti ratio (F/Ti) of

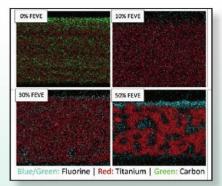


Figure 4. SEM/EDX of coating cross section.

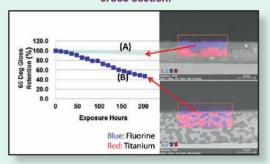


Figure 5. Correlation of HE-XWM gloss retention to coating morphology.

the two formulations detected by EDX using spot mode is different. Surface F/Ti is 32.22 for Formulation A and 9.34 for Formulation B. The thicker the clear FEVE layer, the bigger the F/Ti surface ratio, the better HEXWM gloss retention.

Possible reasons for the contributions of the surface FEVE layer to gloss retention are:

- TiO₂ photocatalytic reaction is a statistical event. Since little Ti was detected in the top F-rich layer, the probability of indirect resin degradation through TiO₂ photocatalytic reaction on the coating surface is reduced. Indirect resin degradation through TiO₂ photocatalytic reaction is considered as the primary reason for degradation of coatings with resins which are UV stable inherently.
- \bullet FEVE is transparent to UV light. But the intensity of the light declines logarithmically due to absorption by resin. In the stratification structure of FEVE and polyester, even though UV can reach to TiO2 below the F-rich clear layer, its intensity drops.
- The F-rich top layer serves as a barrier for moisture and oxygen, which are the necessary elements for a photocatalytic reaction to happen.
- Even if a photocatalytic reaction happens below the coating surface, it will not impact the surface CPVC, thus limiting the impact on gloss.

When there is stratification structure achieved in the FEVE/polyester blends, dependence of coating durability on the type of TiO_2 is reduced. Most commercially available durable TiO_2 are qualified (Figure 6).

AAMA has three specifications for superior performing organic coatings applied to aluminum extrusions and panels for architectural products: AAMA 2603, AAMA 2604 and AAMA 2605, which is the highest standard. These three specifications apply to progressively stronger levels as indicated by South Florida outdoor exposure and laboratory accelerated testing results. Super-durable polyester powder is widely used in applications which qualify AAMA 2604. FEVE resin is currently the only crosslinking and powder coating process-friendly fluoropolymer which can pass AAMA 2605. Testing shows coatings based on FEVE resin can provide excellent weatherability, corrosion and chemical resistance due to the unique chemistry of the resin. FEVE can form a cost and performance synergy with super-durable polyester through a simple blending process. Coatings based on the FEVE/polyester blends pass the gloss and color retention requirements after exposure to the South Florida weathering test for ten years. This technology makes it possible for a superdurable polyester powder formulation to be modified

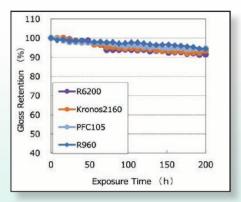


Figure 6. HE-XWM gloss retention of FEVE/ super-durable polyester blends with 50% super-durable polyester using different TiO₂.

to pass AAMA 2605 specifications, which is an exciting opportunity for the powder coating market.

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Special thanks to Royal DSM N.V. for their assistance with this article.





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