LUMIFLON® FLUOROPOLYMER RESINS FOR ULTRA-WEATHERABLE COATINGS

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INTRODUCTION

Fluoropolymer Resins

Fluoropolymers are known for their excellent thermal, chemical, and weather resistance, along with surface properties like water and oil resistance, and optical properties. Because of these characteristics, fluoropolymers are widely used in the chemical, oil and gas, textile, paper, and plastics industries in a range of applications.

Since their introduction in the 1930's, fluoropolymers have been used as coatings on a variety of substrates, imparting their physical characteristics to the coatings. Examples of coatings raw materials include aqueous dispersions of polytetrafluoroethylene (PTFE), tetrafluoroethylene/hexafluoroethylene copolymers (FEP), and TFE/perfluoroalkyl vinyl ether copolymers (FEP). These materials are used primarily as non-stick and anti-corrosion coatings.

Unfortunately, use of fluoropolymers in coatings is limited due to their physical properties. Fluoropolymers have poor solubility in traditional solvents used in the coating industry. Usually, fluoropolymer resins must be heated to temperatures greater than 200° C to form a coating, making them unsuitable for field application or for use on temperature sensitive substrates. Finally, the low surface energy of the resins inhibits acceptable adhesion to metals and other substrates.

Among traditional fluoropolymers, only polyvinylidene fluoride (PVDF) is widely used in coatings. This resin is usually supplied as a dispersion in a high boiling solvent blend, and is used mainly in coil coatings, requiring exposure to a high temperature to form a coating. PVDF is employed primarily in architectural markets due to its exceptional weatherability.

Fluoroethylene Vinyl Ether (FEVE) Resins

A series of unique fluoropolymer resins was developed in the 1980's in an attempt to overcome the difficulties associated with using traditional fluoropolymer resins in coatings, while still maintaining their positive properties. These resins are known generically as fluoroethylene vinyl ether (FEVE) resins and are trade named LUMIFLON®. FEVE resins have a unique chemical structure, shown below in Figure 1. It is this structure, consisting of alternating fluoropolymer and vinyl ether segments, that imparts desirable physical properties to FEVE resins.

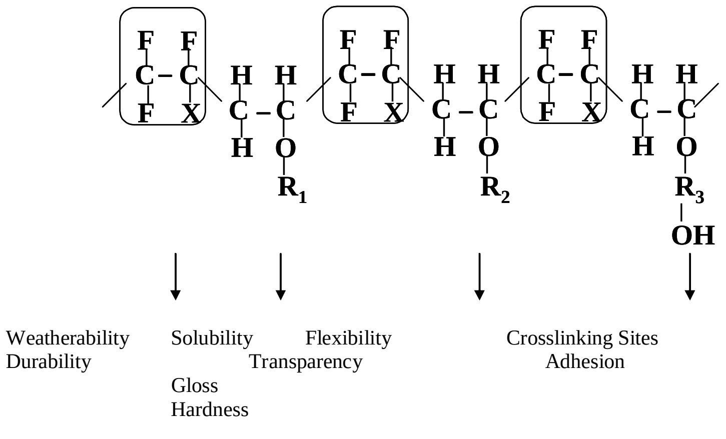


Figure 1: Chemical Structure of LUMIFLON Resins

By making changes in the vinyl ether portion of the polymer, coating properties like solubility, transparency, hardness, flexibility, pigment compatibility, adhesion, and gloss can be varied. In addition, the vinyl ether segments can be functionalized with hydroxyl groups to allow them to be reacted with aliphatic isocyanates to form fluorourethanes. The fluorinated portion of the polymer protects the vinyl ether and urethane parts of the crosslinked polymer from degradation by UV light and chemicals. These characteristics allow FEVE resins to be used in a wide variety of coating types, all of which are notable for their exceptional weatherability.

PROPERTIES OF LUMIFLON FEVE RESINS

Fluorourethanes made from FEVE resins offer the same outstanding weatherability as traditional fluoropolymers, but offer a number of advantages. FEVE resins are soluble in common solvents, making them easy to handle and apply. Fluorourethanes can be cured at either room temperature or elevated temperatures. This means they can be used as maintenance coatings, which are applied in the field, or in shop applied coatings. LUMIFLON based coatings can be manufactured in a wide range of gloss, up to 90 at 60^o or to a completely flat finish of <5, unlike other fluoropolymers used for coatings. Since they are solution polymers, FEVE resins have better compatibility with a wide range of pigments, enabling a broader color palette. Because fluorourethanes are crosslinked polymers, they tend to offer higher hardness and better corrosion resistance than other fluoropolymers commonly used in coatings. Yet, fluorourethanes retain enough flexibility and toughness for use as topcoats for military aircraft, where good flexibility and adhesion are required at -40^o C.

Weatherability of LUMIFLON FEVE Resins

FEVE resin based coatings, when exposed in both accelerated and natural weathering tests, offer durability that far exceeds that of most competitive coatings. In many cases, FEVE coatings applied to structures such as bridges are meant to last for almost 60 years.

The weatherability of FEVE resins is due to the nature of the chemical bonds that fluorine forms with other elements, and to the unique alternating structure of the FEVE resin. Fluorine forms extremely strong bonds with other elements. In addition, fluorine also increases the bond strength of chemical bonds adjacent to it. In all cases, the bond strengths exceed the maximum energy available in UV light, 411 kJ/mol. Table 1 below illustrates this point.

Resin Type	C-C Bond Type	C-C Bond Strength, kJ/mol	C-F, C-H Bond Types	C-F, C-H Bond Strength, kJ/mol
Fluoropolymer	CF_3-CF_3	414	F-CF_2-CH_3	523
Fluoropolymer	CF_3-CH_3 (1)	424	CF_3-CH_2-H (3)	447
Hydrocarbon	CH_3-CH_3 (2)	379	CH_3-CH_2-H (4)	411

Table 1: Chemical Bond Strength in FEVE Resins

Data from Table 1 show that in all cases, the fluorine bond strength exceeds the maximum UV energy of 411 kJ/mol. However, it also shows that the bond strength of C-C bond (1) is much higher than that of C-C bond (2). The C-H (3) bond strength is also higher than that of the C-H bond (4). The higher strength bonds are found adjacent to fluorinated units. In the FEVE polymer since each fluorinated unit alternates with a vinyl ether unit, there is a fluorinated unit adjacent to each vinyl ether unit. This serves to increase the relatively weaker bond strengths in the vinyl ether, reducing degradation of the polymer by UV radiation.

Fluorourethanes made with LUMIFLON resins have been tested in a number of accelerated and natural weathering tests. Figure 2 below shows results from ASTM D-4587 test where the coating has been exposed to a fluorescent UVA 340 nm light source. The exposure cycle was 4 hours UV exposure at 60^o C followed by 4 hours of condensation at 60^o C.

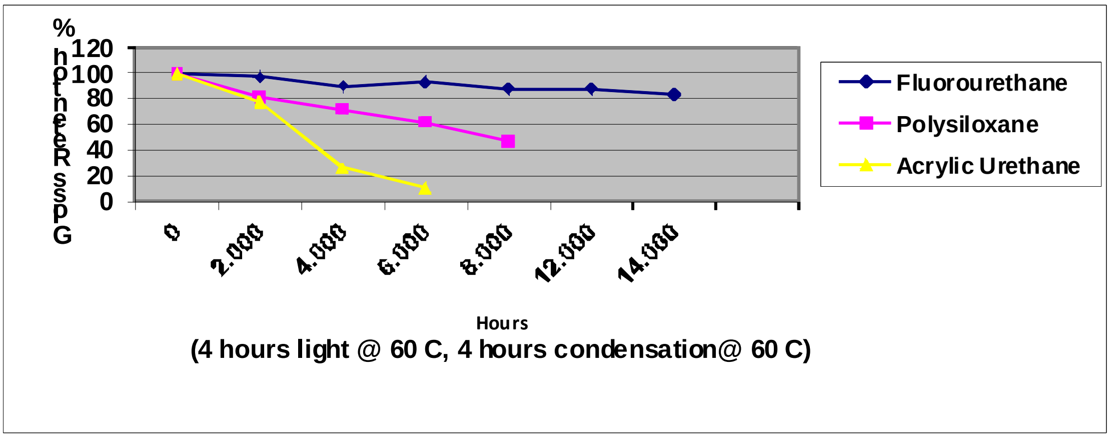


Figure 2: QUV-A Accelerated Weathering of Fluorourethane Coating

Results in Figure 2 show that the fluorourethane coating shows excellent gloss retention even after exposure exceeding 15,000 hours. The LUMIFLON based coating outperforms even the polysiloxane, a coating known for good UV stability.

FEVE based coatings have also been tested in the Equatorial Mount with Mirrors for Acceleration with Water (EMMAQUA) apparatus. In this test, 10 mirrors focus and concentrate sunlight on the sample panels. The coatings are exposed to the entire range of wavelengths of radiation found in natural light. They are periodically exposed to deionized water to simulate exposure to rain. Results are reported as energy exposure per unit area (megaJoules/m^2) rather than in time of exposure. Figure 3 below shows test results using ASTM G-90 for a fluorourethane, an acrylic urethane, and a PVDF coating.

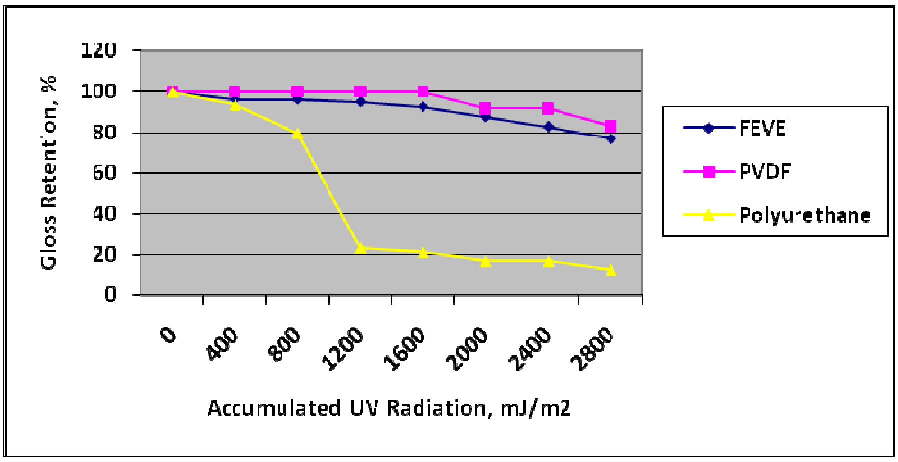


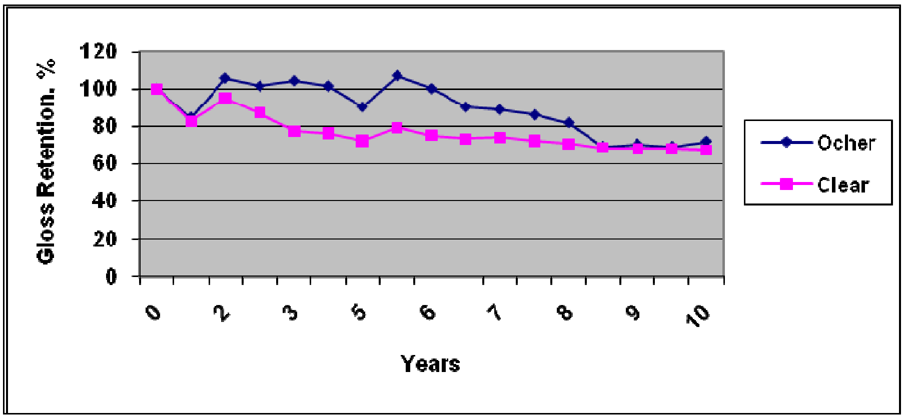
Figure 3: EMMAQUA Accelerated Weathering of Fluorourethane Coating

Results from the EMMAQUA test show that the fluoropolymers easily outperform the acrylic urethane coating.

All accelerated weathering tests suffer disadvantages compared to natural weathering. In many cases, the coatings are exposed to only one wavelength of light, while sunlight consists of many wavelengths. They are most useful in comparing the weatherability of competitive coatings rather than in predicting the life of a coating. For this, natural weathering tests must be performed.

For extremely durable coatings like fluorourethanes, natural weathering takes many years to develop useful data. Natural weathering tests are usually run in tropical, subtropical, or desert environments to maximize exposure to the sun's UV radiation. Many are run near the ocean due to the corrosive nature of water and mist at such locations. In the U. S., the best known site for natural weathering testing is south Florida.

Two LUMIFLON based fluorourethane coatings, a yellow pigmented and a clear, were placed in a south Florida test site for 10 years. Results of the weathering test are shown below in Figure 4.



Location: Miami, FL

Exposure: Direct, 30 degrees South, Open Back

Figure 4: South Florida Weathering of Fluorourethane Coatings

Results from Figure 4 show that the gloss retention of both the pigmented and clear FEVE coatings was greater than 65% even after ten years of weathering in south Florida. The color change of the pigmented coating was less than 1.5ΔE over the same exposure period.

In Japan, natural weather exposure of coatings is often done on Okinawa, which lies at the same latitude as Jacksonville, FL in the U. S. Because Okinawa is an island, it is believed that coating exposure there is more severe than that found in mainland exposure sites. Figure 5 below shows test results comparing exposure of a fluorourethane coating with a PVDF coating.

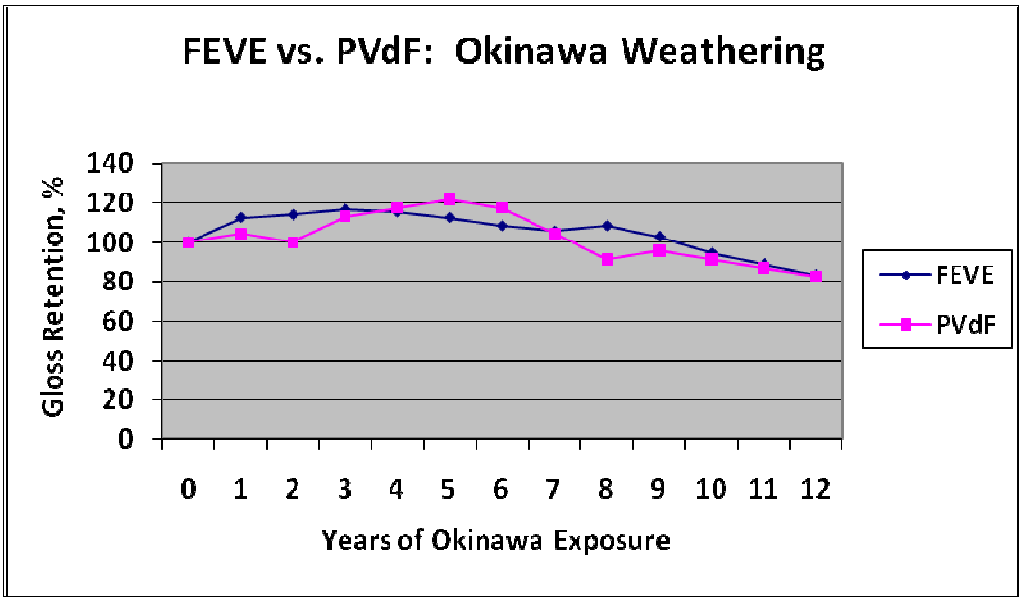


Figure 5: Okinawa Weathering of Fluoropolymer Coatings

Test results show that both fluoropolymer coatings offer excellent gloss retention in the harsh marine environment, exceeding 80% after 12 years.

The true test of any coating is how it performs over time when applied to structures. Over the last 25 years, LUMIFLON based coatings have been used on thousands of buildings and other structures, aircraft, and automobiles. When the resins were first developed in the early 1980's, the coatings were applied to a number of bridges in Japan in cooperation with the Japanese government. In most cases, half of the bridge was coated with a LUMIFLON resin, while the other half was coated with a coating commonly used on bridges at that time. These structures were monitored over the next 20-25 years.

One such bridge was the Tokiwa Bridge. This bridge, located in a mountainous area of Japan near Hiroshima, was topcoated with a fluorourethane in 1986. Color and gloss retention were measured periodically. Photos of the bridge are shown below in Figure 6.

October 1988 April 1993 July 2007

Figure 6: Photos of the Tokiwa Bridge Over 21 Years

The photographs show that the color and gloss of the fluorourethane topcoat were virtually unchanged after 21 years.

After the last photograph was taken, gloss and color of the coating were measured and compared to that obtained immediately after coating application in 1986. Results are shown below in Table 2.

(Insert Table 2)
Table 2: Color and Gloss Change of Fluorourethane Coating after 21 Years

Results from Table 2 indicate that the color and gloss of the FEVE coating are virtually unchanged after 21 years.

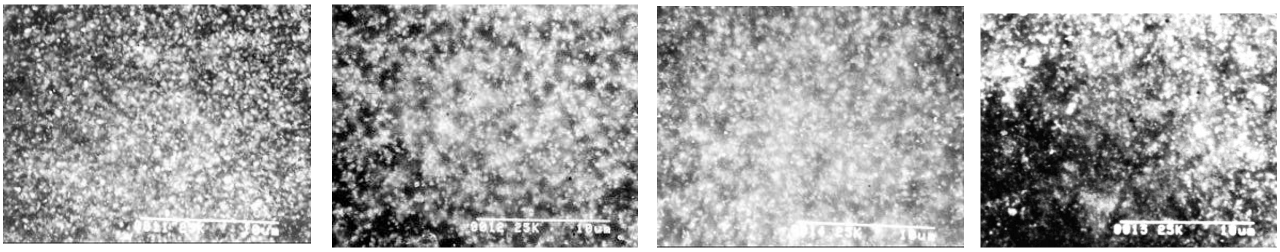
Corrosion Resistance of LUMIFLON FEVE Coatings

It has been demonstrated that FEVE coatings offer substantial improvements in appearance over extended period of time compared to standard coatings. However, the main reason for using coatings on structures and other assets is to prevent corrosion. Unchecked corrosion can lead to rapid degradation of structures like bridges and offshore oil and gas rigs. A 3-part coating is commonly used for structures, especially those in marine environments: a zinc rich primer, an epoxy or polyurethane middle coat, and a topcoat, increasingly of polyurethane. The zinc rich primer is designed to corrode preferentially to steel if the coating is damaged and corrosion initiators like chloride, oxygen, and water reach the metal surface. The topcoat, in addition to maintaining appearance, also prevents movement of corrosion initiators to the midcoat and topcoat.

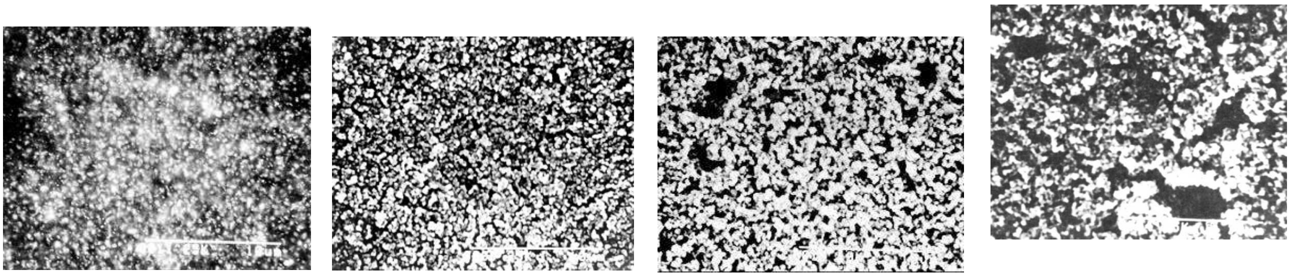
Coatings are degraded when chemical bonds in their constituent polymers are broken by UV light or chemical exposure. The coating polymers are reduced into lower molecular weight components that are more easily removed by wind, rain, and other environmental factors. Over time, the coating loses thickness which reduces its ability to act as a barrier to corrosion initiators. LUMIFLON coatings do degrade, but only very slowly, due to the high bond strength imparted to the polymer by its fluorinated segments. This means that the topcoat continues to protect against corrosion over extremely long periods of time. By using multiple topcoats, the topcoat life on some bridges in Japan is estimated at 50-60 years, even in marine environments.

Figure 7 below illustrates the comparative degradation of different coating types in an accelerated weathering test, the Sunshine Weatherometer Test (SWOM). Surfaces of each coating were examined before and after exposure using scanning electron microscopy.

Unexposed Coatings



Coatings Exposed 2,000 in SWOM Test



Fluorourethane Polyurethane Chlorinated Rubber Alkyd

Figure 7: Comparative Degradation of Coatings in SWOM

In the scans, the constituent polymers of each topcoat are seen as fuzzy white areas. After exposure, the fluorourethane coating shows a small amount of change at the surface. In contrast, the scan of the polyurethane topcoat shows virtually no polymer remaining at the surface. The white seen in the scan is the TiO_2 pigment unsupported by any polymer. The chlorinated rubber and alkyd are even more degraded. Cracks and crevices can be seen in the surfaces of these topcoats. Topcoat degradation leads to increased penetration of corrosion initiators like oxygen, water, and chloride ion, reducing the ability of the coating to prevent corrosion. Because the LUMIFLON coating suffers less degradation than competitive materials, they are much more effective in acting as a barrier to corrosion initiators.

Comparative corrosion resistance of coatings can be obtained by testing in the Electrochemical Impedance Spectroscopy (EIS) test. EIS involves sending an alternating current between two electrodes, where the main electrode is in a 3% salt water solution and the counter electrode on the metal coupon substrate. The change in impedance at a constant frequency of 1 kHz is then measured. The smaller the change in impedance during the test, the better the corrosion protection offered by the coating system. The configuration of the tested coating systems was zinc rich primer/epoxy/topcoat. Since the only difference between each coating system was the topcoat, the EIS test yields data that can be used to compare the relative performance of each topcoat. Relative coating performance is usually measured by determining the tangent of the angle θ, which is the acute angle each line makes with the vertical.

In the version of the test used in this case, the initial impedance of each coating system was measured. The coatings were exposed for 1,000 hours in the Sunshine Weatherometer, and then impedance measured again. Finally, each coating system was exposed to 500 and 1,000 hours in the ASTM B-117 salt fog test, and impedance measured after each exposure. Results of the test are shown below in Figure 8.

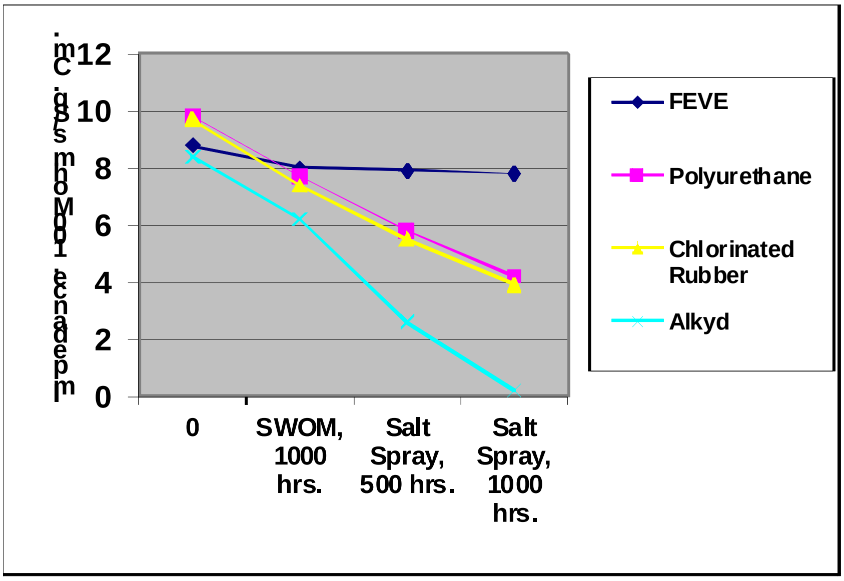


Figure 8: Electrochemical Impedance Spectroscopy Test Results

EIS test results indicate that the fluorourethane coating outperforms all of the other coating types. In fact, the alkyd topcoat had degraded completely as indicated by its impedance of zero at the end of the test. Both the polyurethane and chlorinated rubber topcoats also show significant changes in impedance during the test.

When the tangent of the angle to the vertical of each coating system is measured, it yields the data shown below in Table 3. Relative coating life can be estimated from the results.

Topcoat Type	Tangent θ	Relative Life (Polyurethane=1)
Fluorourethane	5.7	2.2
Polyurethane	2.6	1.0
Chlorinated Rubber	1.8	0.7
Alkyd	1.3	0.5

Table 3: Relative Coating Life from EIS Results

Based on the EIS results, it can be estimated that the relative life of the FEVE coating is more than twice that of the polyurethane. The difference in performance in this test is believed to be due to the difference in degradation of each topcoat. A degraded topcoat allows corrosion initiators like chloride and water to penetrate the topcoat, accelerating the corrosion process.

TYPES OF LUMIFLON FEVE RESINS

Because the structure of the FEVE resin can be varied so easily, a wide variety of resin types can be manufactured.

Solvent Grade FEVE Resins

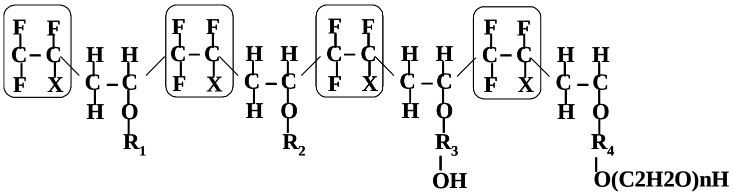
Traditional LUMIFLON resins are supplied in xylene. Solvent grade resins differ in molecular weight and crosslink density, allowing for changes in properties like chemical resistance, flexibility, hardness, and adhesion. Most are used in exterior site applied coatings. One solvent grade substitutes an Aromatic 150/cyclohexanone blend as solvents; this resin is used in factory applied coil coatings. Because coated coil components are usually fabricated at the building site, excellent flexibility is required.

Solid LUMIFLON Resins

In an effort to enable coating manufacturers to meet environmental standards in the U. S., solid resins are now offered. These resins are manufactured in the same manner as solvent based resins, and have the same performance as solvent grade resins. Several additional processing steps are involved, including distillation to remove solvent, chilling the resulting resin mass, and chopping the resin into flakes. Solid resins are soluble in so-called volatile organic compound (VOC) exempt solvents, including para-chlorobenzotrifluoride (Oxsol® 100), acetone, t-butyl acetate, and dimethyl carbonate. When dissolved in exempt solvents or a combination of such solvents, coatings meeting the 100 g/l VOC standard for southern California can be manufactured. These resins are also soluble in hazardous air pollutant (HAPS)-free solvents like MEK, MAK, and glycol ethers and esters.

Water Emulsion FEVE Resins

Water-based LUMIFLON resins have also been developed. The first waterborne FEVE resins products were water emulsions. To manufacture these resins, vinyl ether monomers substituted with polyoxyethylene (EO) units are copolymerized with a fluorinated monomer and other vinyl ethers, maintaining the conventional FEVE structure. The resulting polymers are high in molecular weight, so they can be used in either in single component coatings or in formulations crosslinked with aliphatic isocyanate dispersions. Surfactants are used to improve the water compatibility of the fluorinated polymers. The structure of a typical FEVE water emulsion is shown in Figure 9.



Emulsion stability

Figure 9: Polymer Structure of FEVE Emulsion

Coating properties obtained from single and two component FEVE emulsions, while exceeding those of many competitive coatings, are not as good as those obtained from solvent and solid grades of the resins. This is likely due to the presence of surfactant in the coating, as well as to the presence of the ethylene oxide units in the polymer. In general, the water resistance of the emulsions is lower than that of solvent grade coatings, and weatherability, while still good, does not attain the levels of the solvent resins.

Research has shown that the LUMIFLON emulsion resins are effective when used in blends with standard water based resins to improve the weathering of these systems. Results from one test are shown below in Figure 10.

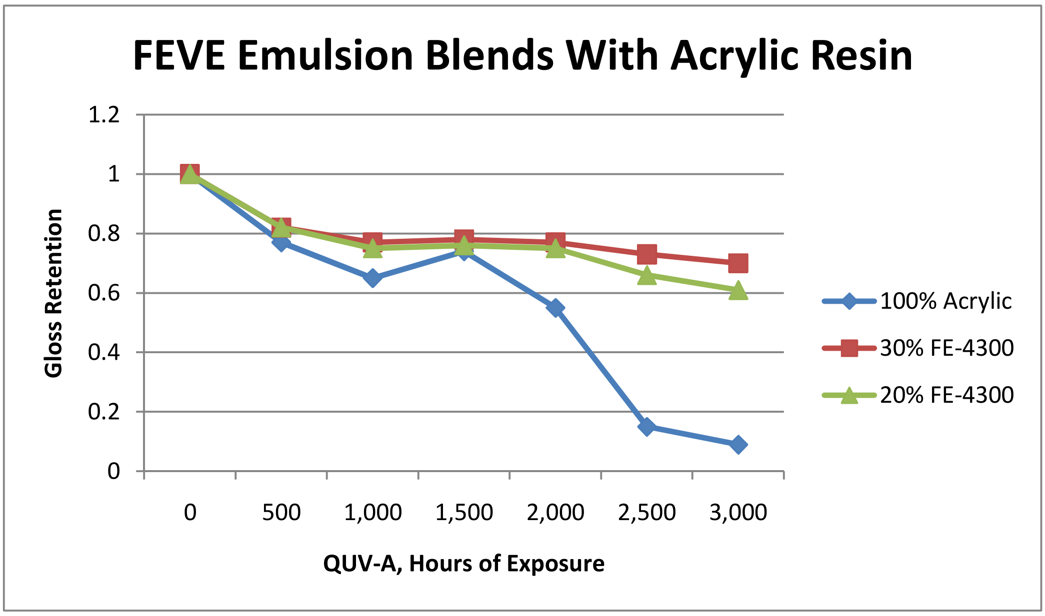


Figure 10: Accelerated Weathering of FEVE Emulsion Blends with Acrylic Resin

Test results show that the replacement of 20% or 30% of the water based acrylic resin with the FEVE emulsion leads to a significant improvement in weatherability of the resulting coating. Traditionally, LUMIFLON resins have been used at 100% of the coating resin component to manufacture coatings with weatherability exceeding 30 years. In many cases, this level of weathering is not required. For example, FEVE resins are being used in blends in the automotive industry to improve the weathering of conventional protective coatings. In this case, excellent gloss retention is desired for 7-10 years, while existing coating systems deliver only 3-6 years.

FEVE Water Dispersion Resins

FEVE water dispersions were developed to overcome the problems inherent in FEVE water emulsions. These dispersions are made through a multi-step process using solid FEVE resins as the raw material. First, FEVE solid resins are synthesized in a solvent, which is then removed by vacuum distillation. The resulting resin is chilled, and then processed into flakes. These flakes are then dissolved in a hydrophilic solvent. To make the dispersion, a portion of the hydroxyl functional vinyl ether groups are denatured with an acid anhydride to form carboxylic acid groups (I). These acid groups are neutralized with an amine (II), and the resulting polymeric carboxylic acid salt dispersed in deionized water. Finally, the solvent is evaporated, yielding an FEVE dispersion containing no solvents or emulsifiers (III). The preparation of these dispersions is shown schematically in Figure 11.

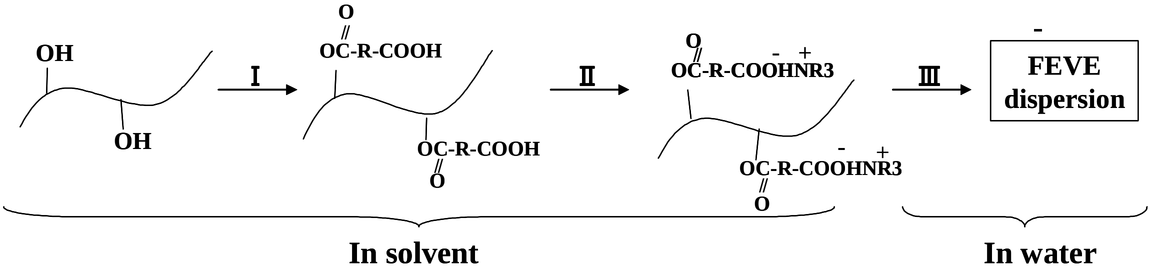


Figure 11: Manufacture of FEVE Water Dispersion Resin

After synthesis of a number of dispersions, it was found that dispersion stability was influenced by several factors, including molecular weight of the polymer, particle size, and acid value. The most stable dispersions were derived from lower molecular weight, moderate particle size, moderate acid value polymers.

To test the performance of FEVE water dispersions, coatings were prepared from the FEVE water dispersion, a water emulsion FEVE resin, and a solvent-based FEVE resin. Chromate treated steel panels were coated with the coatings, which were allowed to cure for 14 days.

The resulting fluorourethane coatings, along with a single component FEVE emulsion resin, were subjected to several standard tests for coatings. The results are shown below in Table 4.

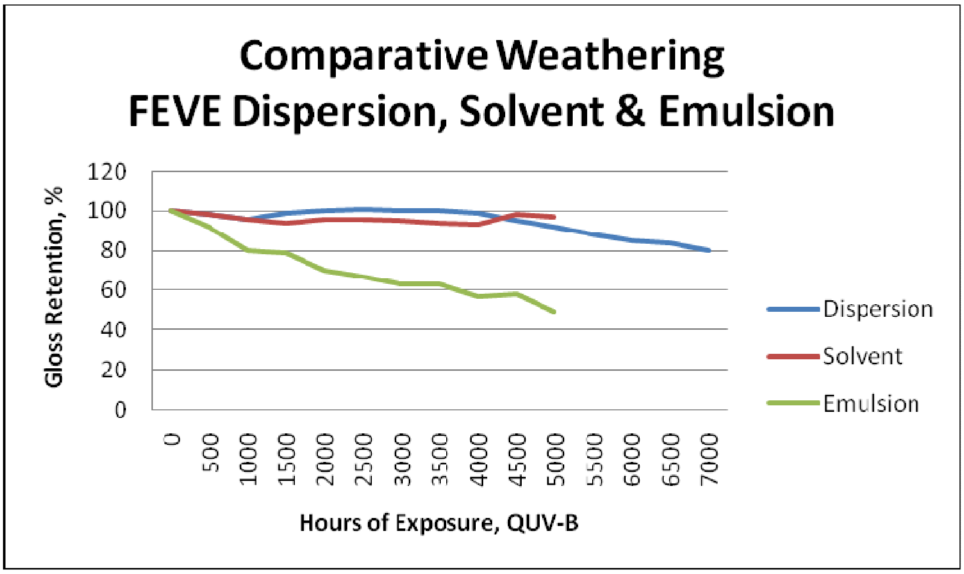
		FEVE Dispersion (OHV=85)	FEVE Solvent-Based (OHV=52)	FEVE Emulsion (OHV=55)	
Crosslinker (NCO/OH=1)		Water Dispersible Isocyanate	HDI Based Polyisocyanate	Water Dispersible Isocyanate	None
Gloss, 60^{o}	ISO 2813	88	90	78	78
Pendulum Hardness	ASTM D 4366	79	80	75	19
DuPont Impact	ASTM D 2794 (D=0.5”)	>1.0 m	>1.0 m	1.0 m	0.3 m
Cross Cut Adhesion*	ASTM D 3359	5B	5B	5B	0B
Water Resistance ISO 2812 40^{o} C, 24 hrs	. Adhesion, ASTM D 3359	4B	5B	3B	0B
	Blistering, ASTM D 714	No Blistering	No Blistering	<8, Medium	2 Dense

*Cross cut adhesion test performed after soaking in hot water for 24 hours.

Table 4: Comparative Performance of FEVE Coatings

Test results in Table 4 show that the initial gloss of the dispersion-based fluorourethane is close to that of the solvent-grade coating, and higher than that from the emulsion. Hardness and adhesion of the three crosslinked coatings are about the same, although the reacted emulsion has slightly lower impact resistance. The single component FEVE emulsion has far lower hardness and impact resistance, and poor adhesion. The emulsion alone is high enough in molecular weight to form a film using a coalescing solvent, but without the isocyanate crosslinker its properties are poor. The biggest difference in performance is the water resistance of the three fluorourethane coatings. The water dispersion and solvent grade fluorourethanes show excellent water resistance, while the emulsion develops blisters during the test. In this battery of tests, the FEVE dispersion offered performance equivalent to that of the solvent-based coating. This means that zero VOC fluorourethane coatings with excellent properties can be formulated using the FEVE dispersions.

The crosslinked coatings described above were examined by the ASTM G 53 accelerated weathering test. In this case, the coatings were tested with the QUV-B bulb. The wavelength is believed to be more aggressive than the QUV-A bulbs used in the test in Figure 2. Results are shown below in Figure 12.



UV / Condensation Cabinet Cycle

8 hours UV at 70 deg.C and 4 hours Condensation at 50 deg.C

Figure 12: QUV-B Weatherometer Testing of FEVE-Based Coatings

Accelerated weathering tests show that the dispersion-based fluorourethane weathers as well as the solvent-based coating, and outperform the water emulsion.

Based on the test results, it should be possible to use FEVE dispersions for all applications where solvent-borne products are used today. Because FEVE dispersions can be used without coalescing solvents which may be considered VOCs, they can be used as industrial maintenance coatings even in California which has the strictest VOC regulations in the U. S. In addition, the dispersions can displace solvent-based coatings in applications where solvent odor can affect occupants of a structure, such as office buildings or hospitals.

APPLICATIONS FOR LUMIFLON COATINGS

FEVE resins are usually formulated into fluorourethane coatings. In some cases, LUMIFLON resins are used in blends to improve weathering. Markets for LUMIFLON coatings include architectural, industrial maintenance, aerospace, automotive, and alternative energy. Typical applications are summarized below.

Architectural Markets

FEVE resins are used to make coil coatings that are applied to metal composite panels for commercial and industrial buildings. These are shop applied coatings. FEVE resins can also be used for field applied air dry fluorourethane coatings useful for repainting existing buildings and other structures. FEVE powder coatings are used to replace liquid fluoropolymer coatings for aluminum extrusions, used mainly for window frames. Fluoropolymer architectural coatings are usually formulated to meet the American Architectural Manufacturer’s Association (AAMA) specification AAMA 2605. The coating portion of this specification requires at least 10 years of South Florida weathering with >50% gloss retention, and a maximum color change of 5ΔE.

Industrial Maintenance Markets

FEVE resins are widely used on industrial structures, including water towers, bridges, and offshore platforms. For these applications, corrosion protection in addition to coating weatherability is critical. Coatings used on industrial structures are usually 3-coat systems, with a zinc rich primer, an epoxy or urethane mid-coat, and a fluorourethane topcoat. In most cases, these are field applied coatings, although new bridge construction involves shop coating a significant portion of the components.

Aerospace Coatings Markets

FEVE resins are used in the U. S. Air Force's Advanced Performance Coating system. The Air Force made the choice of a fluoropolymer coating because of its improved weathering, low color change, and its ease of cleaning. Because military aircraft are repainted on a regular basis, 30 years of durability are not required. Therefore for this application, FEVE resins are blended with standard aerospace coating resins. FEVE resins are beginning to find a place in commercial aerospace as well. Fluorourethanes have been approved for use on the Boeing 787, which is manufactured using large amounts of composite materials.

Automotive Markets

Due to their high cost, LUMIFLON resins find only limited applicability in the automotive market. FEVE resin blends are used as an appearance coating for components used on the Chevrolet Cruze, General Motors' new small car platform. In addition, the resins are used in a coil coating for exterior metal components on several different automobiles.

Alternative Energy Markets

FEVE coatings are being used extensively in alternative energy markets, especially in solar. Coatings for wind towers and blades are also being tested. These markets are ideal for fluorinated coatings, since the expected life of solar panels and wind towers are 30+ years. In many cases, wind towers and solar panels are in remote locations, making recoating expensive and time consuming. LUMIFLON coatings are used on solar panel back sheets, used to protect the solar cell from moisture and UV degradation. Fluoropolymer laminates were and are still used in this application; however, FEVE coatings have proven themselves to be less expensive and have therefore substantially increased market share over the last 4-5 years.

CONCLUSIONS

FEVE resins continue to gain market share in areas where ultra-weatherability is required. Traditional solvent-based FEVE resins are being supplemented by other types of resins that can be formulated to meet the latest environmental regulations. Fluorourethanes are gaining wide acceptance for their life cycle cost advantages in architectural, aerospace, automotive, alternative energy, and industrial maintenance markets. FEVE blends can be used to improve the weathering of conventional water based resin systems for similar applications.

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