



## Novel Acrylic Epoxy Hybrid System Design for Metal Protection Application

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A process for producing a novel acrylic epoxy hybrid (AEH) is demonstrated in which a small molecule epoxy diffuses from its “monomer” droplets through the aqueous phase and into an acrylic dispersion particle. The obtained hybrid can be formulated as a viscosity-stable 2K coating system by mixing pigments and fillers with waterborne polyamine hardener as Part A and the acrylic epoxy hybrid as Part B. The 2K coating formulation stability is controlled by the hybrid and hardener surface charge reflected in zeta potential values. When the zeta potentials of the hybrid and hardener were in the range at  $|\zeta_{AEH} + \zeta_{hardener}| < 30$ , the 2K coating formulation showed instability and a tendency to form gel. The 2K coating system performance relies heavily on hardener choice for metal protection properties such as salt spray corrosion resistance. It was found that water solubility of hardener is negatively correlated to salt spray resistance (SSR). With an appropriately low water solubility hydrophobic hardener, the 2K coating system can achieve  $\geq 600$  hr SSR, which is much better than a 1K acrylic and 2K acrylic/liquid epoxy resin (LER) system and similar or slightly worse than a conventional 2K waterborne epoxy system. Furthermore, the 2K coating system dries faster than a traditional 2K WB epoxy system but has much longer pot life. In addition,

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its application properties, such as dilution stability, are also excellent due to the better stability of the acrylic latex polymers.

### INTRODUCTION

The effort to replace solventborne coatings with waterborne alternatives has been ongoing for several decades. Acrylic latices are the most popular binders for waterborne coatings. Acrylics have been well accepted for architectural coatings and used for more than 60 years. However, for industrial coatings, the use of waterborne coating systems is still very limited. Compared with solvent-based systems, acrylic latex coatings have inferior barrier and resistance properties because of poor film formation and little to no crosslinking.

There are many efforts to overcome these drawbacks. In our recent work,<sup>1</sup> a novel Designed Hybridization technology was developed as a unique way to improve the performance of thermoplastic latex with a thermosetting chemistry. The model and mechanism of this novel Designed Hybridization technology is shown in *Figure 1*. A host latex particle is first made via free radical emulsion polymerization of vinyl monomers. Thermosetting or reactive small molecules are then imbibed into the latex particles by allowing the small molecules to diffuse from their “monomer” droplets through the aqueous phase into the dispersed particle.

An example of this Designed Hybridization approach is an acrylic epoxy hybrid latex composite. It is prepared by imbining liquid epoxy resin into acrylic particles. The imbibed liquid epoxy acts as a coalescent to assist the high MW acrylic polymer in film formation, and the latex acts as a controlled-release host of the liquid epoxy to enable longer pot life. As the epoxy diffuses out of the latex and cures with hardeners located in the aqueous phase, the high MW acrylic polymer regains its hardness. In the cured film, the acrylic latex polymer domains are distributed in a continuous phase of cured epoxy. This special design enables acrylic epoxy hybrid latex composite films to show barrier and resistance property performance similar to the thermosetting 2K epoxy system. At the same time, the acrylic component enables longer pot life, faster dry, better UV resistance, easier handling, and lower cost compared with traditional waterborne 2K epoxy coatings.

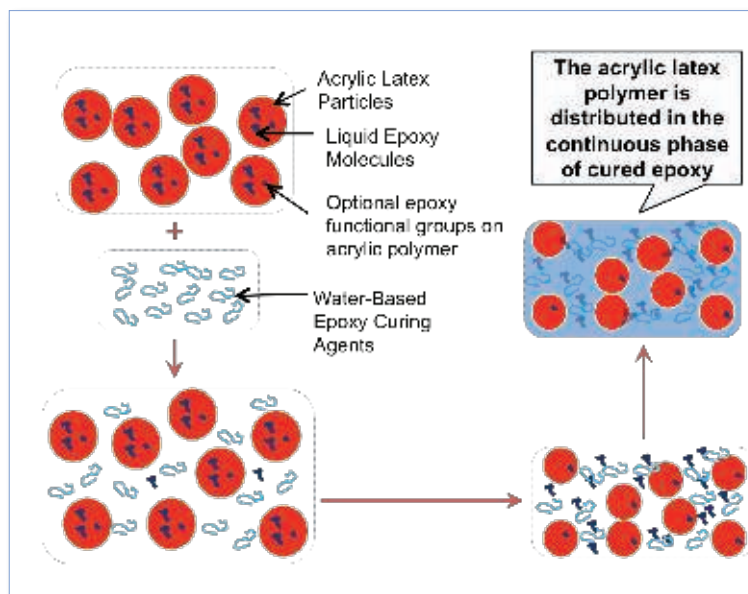
In this article, an acrylic epoxy hybrid latex composite system based on Designed Hybridization technology was studied for metal protection application. The hardener selection was found to be one of the critical factors for paint stability and final performance. The performance advantage was compared with traditional 2K epoxy and acrylic latex-based systems.

## EXPERIMENTAL

### Raw Materials and Formulations

#### Acrylic Epoxy Hybrid (AEH) Preparation

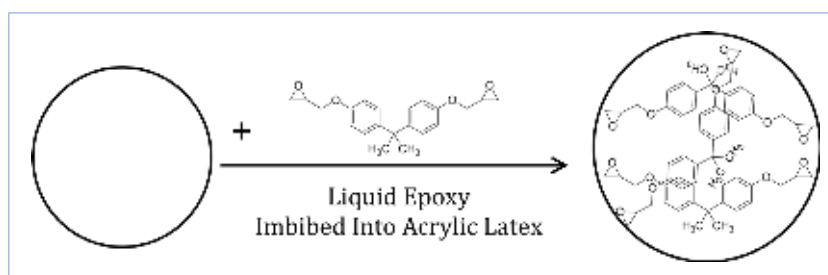
The process of making the Designed Hybridization-based acrylic epoxy hybrid (AEH) is illustrated in *Figure 2a*. A waterborne acrylic latex was produced according to a standard seeded process<sup>2-4</sup> in a 5000 mL five-necked reactor equipped with a stirrer, reflux condenser, and thermometer. An epoxy emulsion of bisphenol A diglycidyl ether,



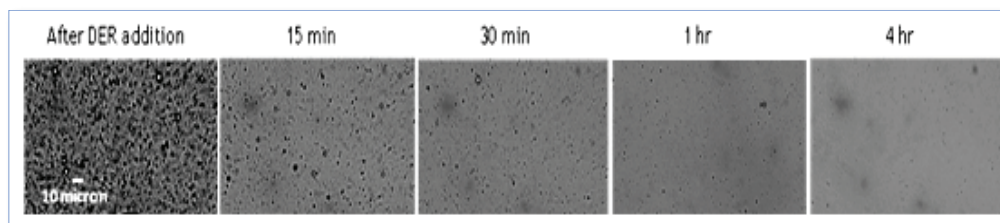
**Figure 1**—Model and mechanism for 2K application of the novel Designed Hybridization.

such as DER 331 from Dow, was made by the phase inversion method. With a temperature of 60°C, the epoxy emulsion was fed into the reactor containing the acrylic latex and held for 1 hr to allow the epoxy to transport and swell the acrylic latex. The key requirement was that the epoxy was completely imbibed in the acrylic particles.

The driving force for this transport process is the solubility of the epoxy molecule in the latex polymer, not a reaction. As a result, there is a saturation limit for the swelling and it was found that 20–40% by weight of epoxy can be fully imbibed into the acrylic latex polymer, depending on the acrylic latex polymer composition and type of epoxy. This imbibing process typically takes from 5 min to 2 hr at 60°C, depending on the size of the epoxy monomer droplets, acrylic latex composition, and particle size. In *Figure 2b*, the optical microscopy images were taken at different times after the epoxy monomer droplets were added to the latex.



**Figure 2a**—Process for making AEH dispersion.



**Figure 2b**—Optical microscopy images after epoxy addition to latex.



**Table 1**—Raw Materials for Coating Formulation

Trademark	Function	Supplier
Bentone LT	Rheology modifier	Elementis Specialties
Orotan 731A	Dispersant	Dow Chemical
Acrysol RM-8W	Rheology modifier	Dow Chemical
SN321C	Defoamer	San Nopco Limited
Ti-Pure R-902	Pigment	DuPont Titanium Technologies
Nubirox 106	Anticorrosive Pigment	Cary
Talc	Filler	Local
Blanc Fixe N	Filler	Sachtleben Chemie GmbH
Dowanol PM	Coalescence	Dow Chemical
Dowanol DPnB	Coalescence	Dow Chemical
NaNO <sub>2</sub>	Rust Inhibitor	Local
Silquest A-187	Adhesion promoter	GE Toshiba Silicones
Beckopox EH 613	Hardener	Cytec Surface Specialties
Beckopox VEH 2849	Hardener	Cytec Surface Specialties
Anquamine 401	Hardener	Air Products
OudraCure WB 8005	Hardener	Dow Chemical
OudraCure WB 8006	Hardener	Dow Chemical
OudraCure WB 8007	Hardener	Dow Chemical

**Table 2**—Parameters of Waterborne Hardeners

Trademark	Type	Solid%	AHEW as Delivered
Anquamine 401	Modified aliphatic amine	70	200
Beckopox EH 613	Aliphatic polyamine adduct	80	145
Beckopox VEH 2849	Aliphatic polyamine adduct	80	135
OudraCure WB 8005	Aqueous polyamine adduct	65	220
OudraCure WB 8006	Aqueous polyamine adduct	80	200
OudraCure WB 8007	Aqueous polyamine adduct	70	200

The particles in the images are epoxy monomer droplets and the latex particles are too small to be seen at the magnification. In about 1 hr, only a few epoxy droplets could be observed and had totally disappeared at about 4 hr.

### Raw Materials for Coating Formulation

The raw materials for coating formulation are listed in *Table 1*.

### Coating Formulation and Preparation

A starting point formulation (SPF) was developed based on AEH dispersion. To make a coating with viscosity stability, it is preferred to mix pigments and fillers with waterborne hardener as Part A, and have the AEH dispersion as Part B. Part A and Part B should be mixed well before application.

To compare anticorrosive performance, six waterborne hardeners were selected from Cytec, Air Products, and Dow (UPPC), respectively. The key parameters of these hardeners are listed in *Table 2*. The use level of each waterborne hardener was varied to account for their differing active hydrogen equivalent weights (AHEW). For better anticorrosive

performance, the stoichiometry of epoxide equivalent weight (EEW)/AHEW was selected as 1.0/0.8. The SPF developed is shown in *Table 3* with only 38 g/L VOC.

## TEST METHODS

### Zeta Potential

The zeta potential of both the AEH dispersions and waterborne hardeners was measured with a Zetasizer NANO ZS instrument from Malvern Instruments Ltd. All the samples were diluted 1:10 (w/w) with DI water. The hardener was added into AEH under stirring, and then equilibrated for 10 min; finally, the zeta potential was tested at different ratios. The zeta potential was measured in triplicate and the average value of analyses reported. All of the sample measurements were performed at 25 °C and with a cell drive voltage of 30V, using a monomodal analysis model. The count rate range was adjusted to be 200~400 for high confidence level.

### Water Solubility of Hardener

**Standard Curve Making:** A sample of 5 g commercial hardener was weighed and put in a vacuum drying oven at 70 °C for 24 hr to remove the solvent and water. A 0.500 g dry sample was dissolved in 50 mL of ethanol to get a 10,000 ppm stock solution. This stock solution was diluted to get standard solutions at a range of concentrations to make a calibration curve. UV absorption spectra were scanned between 310 and 240 nm. Transmittance was measured by Shimadzu 3100 UV-Vis spectrometer with the absorption at 276 nm used for calculation.

**Water Solubility Testing:** Exactly 0.500 g of the dried sample was diluted with 9.500 g DI water in a bottle, sealed, and shaken at room temperature for 12 hr. Each sample was then ultra-centrifuged at 80k rpm for 20 min. The supernatant was carefully separated for UV-Vis analysis. The sample concentration was measured using UV-Vis based on the external calibration.

### Coating Performance

#### Salt Spray Resistance (SSR)

The anticorrosion performance was tested according to the standard ASTM B-117. A coating film was made using a 13 mil drawdown bar. After a 2-hr flash dry, the coated panels were put into an oven at 80 °C for 1 hr. The drying and curing profile was chosen to mimic a manufacturing process in coating freight containers. After cooling down to room temperature, the edges and backs of the based panels were sealed. The sealed panels into

were put into a Q-fog cabinet, the film status regularly checked, and the time of blistering or rusting on the film recorded.

### Drying Time Test

A wet film with 150 µm thickness was drawn down on a glass strip and put on the work surface of a BYK drying time recorder for a run time of 6 hr. The result was read according to the standard ASTM D5895. An illustration of the results for this device is shown as Figure 3.

## RESULTS AND DISCUSSION

### Stability Study for Hardener Selection

Since the AEH system is based on a unique epoxy/acrylic structure, it was more sensitive to hardener selection than a general 2K WB epoxy system. To better understand the stability of AEH with different amine hardeners, zeta potential was used to monitor the stability. Generally speaking, the hardener has amine groups and is therefore positively charged, while the AEH particles are

Table 3—Starting Point Formulation of 2K AEH Coating System

	Anquamine 401	Beckopox EH 613	Beckopox VEH 2849	OudraCure WB 8005	OudraCure WB 8006	OudraCure WB 8007
<b>Part A</b>						
Grind						
Water	8.44	8.44	8.44	8.44	8.44	8.44
Bentone LT	0.26	0.26	0.26	0.26	0.26	0.26
Orotan 731A	1.3	1.3	1.3	1.3	1.3	1.3
Acrysol RM-8W	0.4	0.4	0.4	0.4	0.4	0.4
SN321C	0.5	0.5	0.5	0.5	0.5	0.5
Ti-Pure R-902	4.1	4.1	4.1	4.1	4.1	4.1
Nubirox 106	5.1	5.1	5.1	5.1	5.1	5.1
Talc	12.7	12.7	12.7	12.7	12.7	12.7
Blanc Fixe N	13.1	13.1	13.1	13.1	13.1	13.1
End of Grind						
Let Down						
Dowanol PM	0.5	0.5	0.5	0.5	0.5	0.5
Dowanol DPnB	0.7	0.7	0.7	0.7	0.7	0.7
15%NaNO <sub>2</sub>	0.4	0.4	0.4	0.4	0.4	0.4
Water	9	9	9	9	9	9
Anquamine 401	5.6					
Beckopox EH 613		4.1				
Beckopox VEH 2849			3.8			
OudraCure WB 8005				6.2		
OudraCure WB 8006					5.6	
OudraCure WB 8007						5.6
Water	4.9	6.4	6.7	4.3	4.9	4.9
<b>Total</b>	<b>67</b>	<b>67</b>	<b>67</b>	<b>67</b>	<b>67</b>	<b>67</b>
<b>Part B</b>						
Maincote AEH-20	44	44	44	44	44	44
A-187	0.5	0.5	0.5	0.5	0.5	0.5
<b>Total</b>	<b>44.5</b>	<b>44.5</b>	<b>44.5</b>	<b>44.5</b>	<b>44.5</b>	<b>44.5</b>
Mix Ratio	Part A/Part B =67 / 44.5					
Total PVC	Coating Properties (A+B)					
Volume Solids	30.12%					
Weight Solids	54.48%					

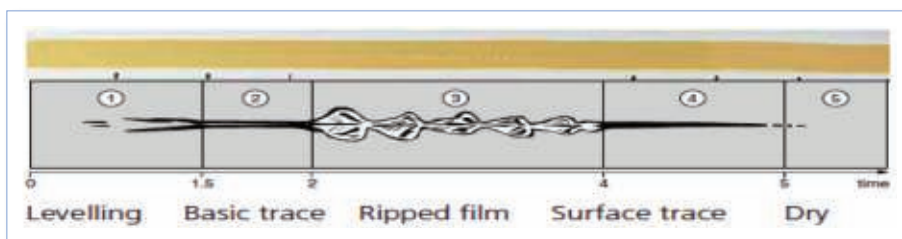


Figure 3—Drying stages on drying time recorder.



**Figure 4**—Grit formed when hardener was mixed with AEH.

negatively charged. When the AEH and hardener are combined, the interaction of opposing charges impacts the electric double layer of the colloidal particles. If the final electric double layer does not provide enough stability to the particles, grit is generated. *Figure 4* shows some grit formed when AEH was improperly mixed with hardener.

*Table 4* shows test results of zeta potential for different hardeners. All of the hardeners show a positive potential and the AEH shows a negative potential. The results of blending hardener and AEH vary with the identity of the hardener. Some hardeners form stable mixtures, while improper hardeners form grit or even gel. A rule to guide hardener selection can be drawn from the testing. All the combinations that meet the rule  $|\zeta_{\text{AEH}} + \zeta_{\text{hardener}}| > 30$  are found to be stable. Those samples that do not meet this rule form grit or gel.

Based on the zeta potential study, we choose those hardeners that form stable mixtures with AEH for further study, including: Anquamine 401, Beckopox EH 613, OudraCure WB 8005, OudraCure WB 8006, and OudraCure WB 8007.

## Formulation Design for Metal Protection

### Anticorrosion Performance

According to our previous studies, many of the unique properties of the acrylic epoxy hybrid latex composite based on Designed Hybridization technology derive from the design of liquid epoxy resin imbibed into acrylic particles and their special film-forming mechanism. The imbibed liquid epoxy acts as a coalescent to assist the high MW acrylic polymer in film formation, and the latex acts as a controlled-release host of the liquid epoxy to enable longer pot life. As the epoxy cures with hardeners located outside the polymer particles in the aqueous phase, it diffuses out of the latex and the high MW polymer regains its hardness. In the cured film, the acrylic latex polymer domains are distributed in a continuous phase of cured epoxy. This special design enables the acrylic epoxy hybrid latex composite to have barrier and resistance performance similar to a thermosetting 2K epoxy system. These properties give AEH the potential to be used for metal protection and anticorrosion.

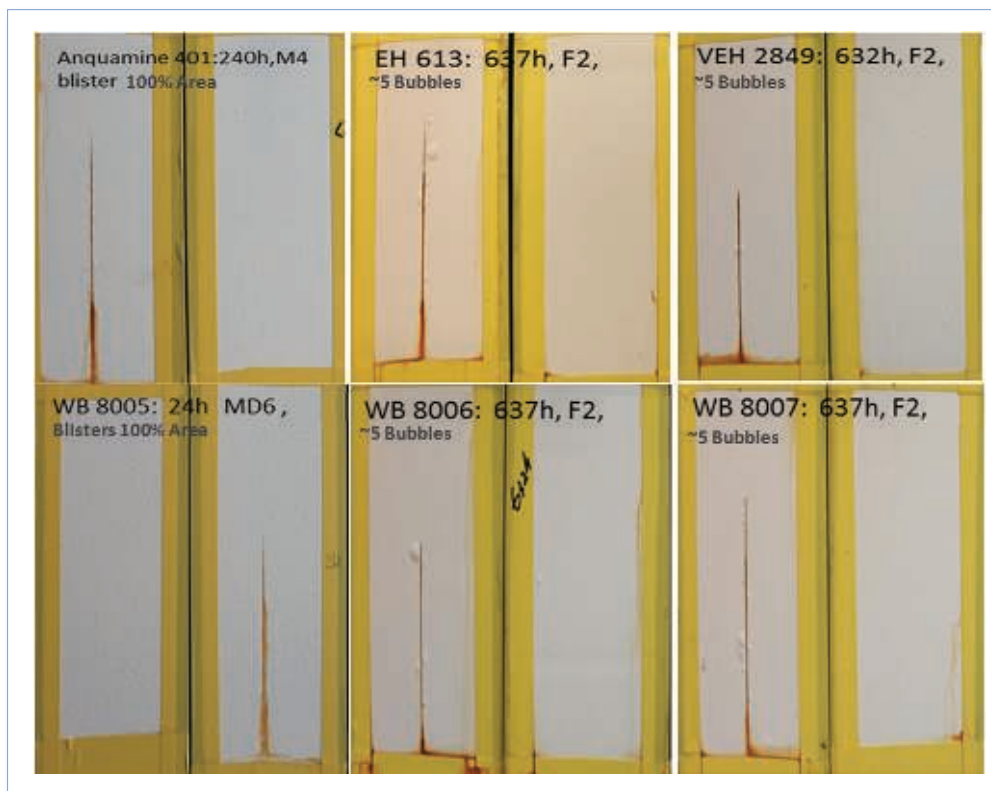
The salt spray resistance performance of acrylic epoxy hybrid latex composite with different hardeners was evaluated and is shown in *Table 5* and *Figure 5*. These results show hardener selection to be a critical factor for both paint stability and coating performance. Coatings with proper hardeners such as OudraCure WB 8006, OudraCure WB 8007, Beckopox EH 613, or Beckopox VEH 2849 can pass the 600-hr SSR test only with few bubbles. However, other hardeners, such as Anquamine 401 and OudraCure WB 8005, show very poor SSR due to blistering through the whole area.

**Table 4**—Zeta Potential of AEH, Hardener, Mixture, and Stability of Mixture

AEH/ Hardener	Zeta Potential (Component) (mV)	Zeta Potential (Mixture) (mV)	Zeta Potential (Sum of AEH with Various Hardener) (mV)	Stability
AEH	-68.2	N/A	N/A	N/A
Anquamine 419	57.4	-3.4	-10.8	Gel
Beckopox VEH 2188	63.2	-7.8	-5.0	Gel
EPICURE 8290	68.9	-5.6	0.7	Gel
EH 659	45.2	-10.2	-23.0	Gel
OudraCure WB 8001	44.2	-16.1	-24.0	Gel
Anquamine 401	14.5	-53.8	-53.7	Stable
Beckopox EH 613	10.8	-47.4	-57.4	Stable
OudraCure WB 8005	7.3	-54.6	-60.9	Stable
Anquamine 721A	2.2	-63.8	-66.0	Stable
OudraCure WB 8007	10.7	-46.1	-57.5	Stable
OudraCure WB 8006	6.8	-45.7	-61.4	Stable
EPICURE 6870	9.5	-53.9	-58.7	Stable

**Table 5**—Salt Spray Resistance Performance of Novel Acrylic/Epoxy Hybrid Latex Composite with Different Hardeners and Their Solubility in Water

Trademark	DFT ( $\mu$ )	SSR results	Solubility in Water (mg/ml)
Anquamine 401	89	<240 hr, M4, 100%area	49.5
Beckopox EH 613	89	637 hr, F2, 5-bubbles	9.5
Beckopox VEH 2849	85	632 hr, F2, 5-bubbles	17.5
OudraCure WB 8005	82	24 hr, MD6, 100% area	55.9
OudraCure WB 8006	89	632 hr, F2, 5-bubbles	11
OudraCure WB 8007	87	632 hr, F4, 5-bubbles	38.3



**Figure 5**—Pictures of SSR testing with different hardeners.

### Understanding Hardener Selection for Good Anticorrosion Performance (SSR)

To better understand why different hardeners impact SSR performance so dramatically, the hardener hydrophobicity was examined. The hypothesis is that a hydrophobic hardener may show benefits for anticorrosive performance and lower water solubility is likely indicative of a more hydrophobic hardener. Six hardeners were selected to test the water solubility to define the range which can bring good anticorrosion performance. *Table 5* compares the SSR with the water solubility of the hardener. Those hardeners with the lowest water solubility do show the best anticorrosion performance.

Based on the SSR results, we can generalize that the higher the water solubility of the hardener, the worse the anticorrosion performance. To achieve good anticorrosion performance (SSR > 300 hr), the water solubility of hardener is prefer-

ably <40 mg/mL and most preferably <20 mg/mL. We believe that more hydrophobic hardeners have improved compatibility with the epoxy resin, which enhances epoxy migration out of the latex particles during curing. This enhanced curing results in tighter films with better anticorrosion performance.

### Performance Comparison with Traditional Epoxy and Acrylic Systems

#### Anticorrosion Performance

There are three popular water-based coating systems in the market used for metal protection. Their features are listed below:

- **2K Water-based Epoxy:** provides best anticorrosion property, but is expensive and application is inconvenient due to its 2K nature.
- **2K Water-based Acrylic/Epoxy Cold Blend System:** epoxy reacts with carboxyl group on latex polymer to boost the performance.



**Table 6**—Salt Spray Resistance (SSR) Performance Comparison

Type of System	Coating System	DFT (µm)	SSR
1K WB acrylic	1K Acrylic paint	60-70	<24 hr, F4, small clusters
2K WB acrylic/epoxy cold blend system	2K Acrylic with carboxylic acid as hardener + Pre-dispersed epoxy paint	80-90	<72 hr, F6, small clusters
2K WB AEH	AEH + Beckopox VEH 2849	80-90	>600 hr, F2, 5-bubbles
2K WB Type I epoxy	Oudrasperse WB 6001 + Beckopox VEH 2849	80-90	>800 hr, F2, 5-bubbles

- *1K Acrylic Coating*: Provides moderate corrosion resistance for light to medium duty application corrosion requirements.

The novel AEH composite was compared with these three systems to better understand its performance. The results are listed in *Table 6*. The results show that both 1K WB acrylic and 2K WB acrylic/epoxy cold blend systems have SSR of less than 100 hr. The AEH shows SSR of more than 600 hr, which is quite close to the 2K WB Type I epoxy system regarded as the best anticorrosion system in the market. Again, the good corrosion resistance property is very likely due to the special system design where epoxy can migrate out of the acrylic particles and form a continuous phase during curing. This special film-forming mechanism enables acrylic epoxy hybrid latex composite to have performance barrier and resistance performance similar to a conventional 2K epoxy system.

*Figure 6* shows the panels after 600 hr salt spray testing for both AEH and 2K WB conventional epoxy systems. The AEH technology showed comparable results to conventional WB epoxy dispersion. Creep both in wet and dry states is lower than 1 mm in scribed panels. After exposure, the coatings showed good adhesion. In fact, the surface of the unscribed panels showed almost no change.

## Pot Life, Dry Speed, and Paint Stability

### Pot Life

A conventional epoxy dispersion generally has a pot life of 1–4 hr. At the same time, there is no visual

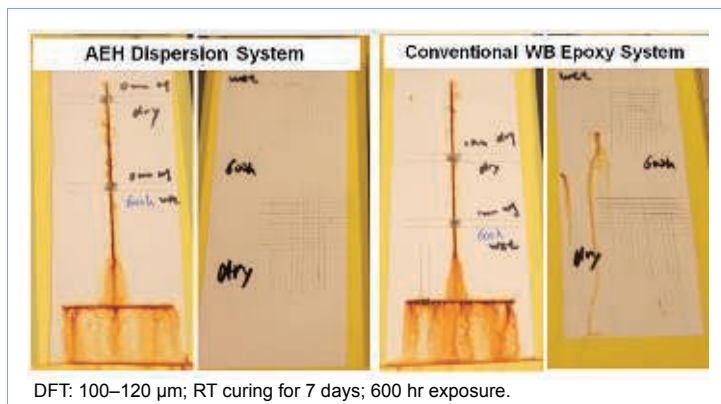
indicator of the end of useful pot life, especially for dispersions of solid epoxy resin. Without a change in viscosity or appearance, it can be hard for applicators to judge whether the paint still can be used without causing quality problems in the final coating. The design of AEH, with epoxy hosted inside acrylic particles only reacting when the epoxy migrates out to react with hardener during film formation, is expected to bring longer pot life. The pot life of AEH with Beckpox EH 613 was tracked by evaluating coating performance including gloss, adhesion, and impact resistance. As shown in *Figure 7*, the coating performance showed almost no change after 13 hr. This means that the coating performances can be guaranteed when applicators use up all of the mixture they prepare at the starting of a working day (within eight working hours). This benefits end users in both material saving and quality control.

### Dry Speed

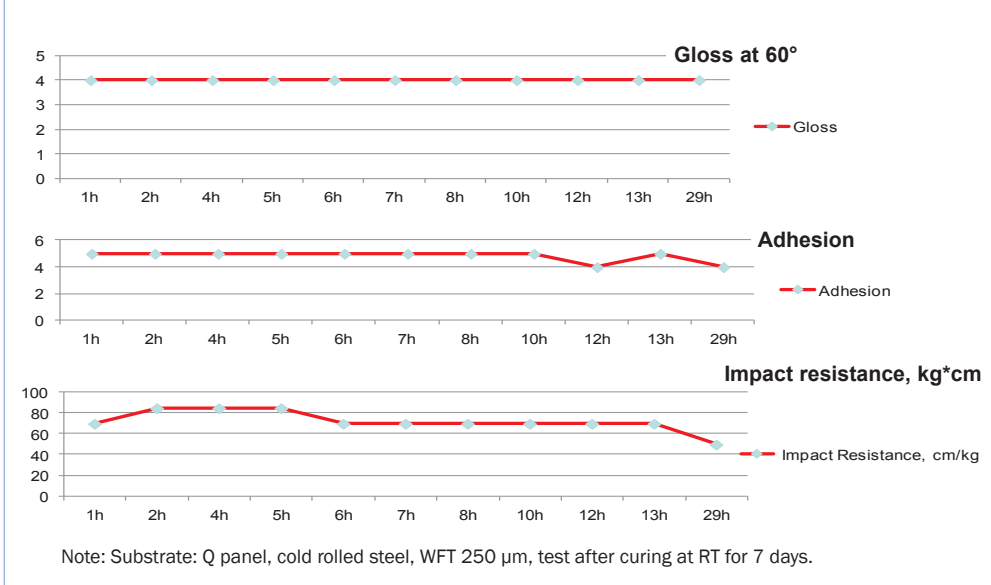
In AEH technology, the thermoplastic acrylic portion provides a quick surface drying, while hard drying is provided by the acrylic and the reaction of epoxy and polyamine curing agents. As shown in *Table 7*, AEH showed both faster surface drying (fast return-to-service time) and hard drying time (final coating performances). This dual benefit can shorten drying intervals and accelerate manufacturing speed or return-to-service time.

### Paint Dilution Stability

Traditionally, epoxy dispersions are prepared by emulsifying epoxy resin with surfactants. This yields dispersions with particle size above 500 nm, significantly larger than acrylic latices. When diluted with water, the surfactant on the surface of epoxy droplets redistributes between the droplet surface and serum phase. With greater dilution, more surfactant moves into the serum phase and no longer provides stability of epoxy droplets. This causes unstable epoxy droplets to come out of the emulsion as grit. This dilution instability causes issues when workers in the plant wash the pipes involved in paint by spraying with water, diluting the epoxy, and forming grit that sticks inside the pipes, causing trouble. *Figure 8* compares the dilution stability of AEH to a conventional epoxy dispersion. One gram emulsion was diluted with 6 g water and then drawn down on glass panels to check



**Figure 6**—SSR testing results after 600 hr exposure of AEH (left) compared to traditional Type I epoxy dispersion (right).



**Figure 7**—Pot life testing of AEH by tracking gloss, adhesion, and impact resistance after mixing hardener and AEH together.

**Table 7**—Drying Speed of AEH Compared to Traditional Epoxy Dispersion on Drying Time Recorder

Paint Code	Binder	PVC Level	Dry Time (hr) Stage 2/Stage 3/Stage 4
WBEP1	Conventional WB epoxy system	35%	0.5/1.3/5.6
WBEP2	AEH, Acrylic epoxy hybrid system	35%	0.4/0.8/1.6

Equipment: BYK drying time recorder.  
Coating films preparation: 150 μm WFT, Glass bar substrate.

the appearance. AEH showed much better dilution stability than the conventional epoxy dispersion during our testing.

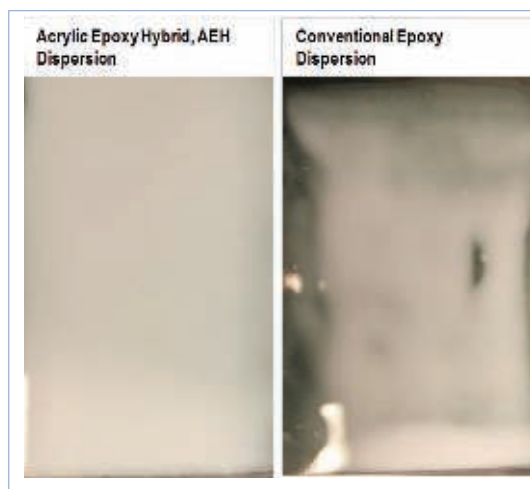
In addition to dilution stability, the AEH also has advantageous viscosity. The viscosity of commercially available epoxy dispersions is generally several thousand cps. The viscosity of AEH is less than 400 cP, much closer to acrylic emulsions and easier to handle in a plant environment.

## CONCLUSIONS

The performance of novel acrylic epoxy hybrid (AEH) was studied for metal protection. Hardener selection was found to be critically important to 2K coating formulation stability and anticorrosion performance. Zeta potential was found to be a good predictor of the stability when AEH binder and hardener are mixed. When AEH and hardener meet the rule  $|\zeta_{\text{AEH}} + \zeta_{\text{Hardener}}| > 30$ , the resulting 2K coating formulation is stable. Hardeners with low water solubility improve corrosion resistance. Compared to 1K WB acrylic or traditional 2K epoxy systems, AEH can provide a better cost/performance balance, which is beneficial to customers. In addition, the acrylic portion of the AEH gives excellent pot life, dry speed, and other application properties such as dilution stability. <sup>CT</sup>

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**Figure 8**—Water dilution stability of AEH and Type I solid epoxy dispersion.

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