

IMPROVING CORROSION RESISTANCE OF

# Powder Coating Systems for ACE Applications



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*A new generation of modified superdurable polyesters for powder coatings that demonstrates improved corrosion resistance for ACE (agriculture and construction equipment) applications has been developed. The  $\beta$ -hydroxy-alkyl-amide hardened binders based on these polyesters are expected to close the observed performance gaps of novel anticorrosion pretreatment solutions, like nanoceramics or titanium/zirconium systems, relative to current systems such as zinc or iron phosphate on steel and yellow chromate on aluminum surfaces.*

*The objective of this article is to present findings that show improved corrosion resistance after*

*salt spray tests on both pretreated and untreated shot-blasted substrates for ACE applications. The article also covers their potential use in more general industry applications and introduces preliminary results in extending the program to binders based on triglycidyl-isocyanurate.*

## INTRODUCTION

Powder coating is an advanced, fast-growing technology, widely recognized for its durability; gloss retention; weatherability; ability to apply up to 200 microns high thickness; and unlimited range of colors, finishes, glosses, and textures. This, combined with its ability to prevent corrosion on metallic substrates (considering that corrosion has a global estimated annual cost of ca. \$2.2 billion,

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which accounts for over 3% of the world's GDP<sup>1</sup>), makes powder coatings an important technology in the coatings world. In addition, powder coatings provide solvent-free finishing and almost 100% recyclable processes, making them an important part of a sustainable or green building project that incorporates low-VOC-emitting products.

Driven by regulations and opinion leaders, further improvements in the environmental friendliness achieved by powder coating producers and end users are required, including the processes and materials used in the different pretreatment steps of metal substrates. However, at the same time, there is a need to comply with the guarantee and warranty levels of corrosion protection as stipulated by existing accreditation systems.

It is well known that surface pretreatment for corrosion protection of metal substrates has a strong impact from an environmental perspective,<sup>2</sup> as illustrated by its current widespread use on steel (such as iron and especially zinc phosphate) and on aluminum (yellow hexavalent chromium, a specie associated with DNA damage and cancer, which is a primary reason for legislation against anticorrosion coatings<sup>3</sup>).

The recent introduction into the market of more environmentally friendly pretreatments, like nano-ceramics and titanium- or zirconium-based systems, has occurred in parallel with the need for better corrosion-resistant coatings that balance their lower efficiency when used at the same level or cost.<sup>2</sup> While suppliers are continuing to improve the performance of their pretreatments, a new generation of cost-competitive resins is being developed to target the improved corrosion resistance of powder coatings for ACE applications.

In this article, we begin with a short reminder of the mechanisms of corrosion and typical prevention and analysis methods and then provide a detailed overview of the experimental work which has led to the development of such materials as:

- A new superdurable polyester for ACE applications in combination with  $\beta$ -hydroxy-alkyl-amide ( $\beta$ -HAA), referred to as NEW-SD-PES1;
- A new polyester for industrial applications in combination with  $\beta$ -HAA or triglycidyl-isocyanurate (TGIC), identified as NEW-IND-PES2; and
- A new polyester for industrial applications in combination with TGIC, referred to as NEW-IND-PES3.

The article concludes with more general findings and a preview of our company's next research and development steps, which will include and extend the work on binders for ACE application based on TGIC.

## BASICS OF THE MECHANISMS OF CORROSION

Corrosion is defined as an electrochemical deterioration of a metal due to the reaction with its environment involving the oxidation of a metal and the reduction of another material,<sup>4</sup> resulting in the breakdown of the metal (corrosion).

Factors influencing corrosion are:

- The presence of water, oxygen, and ions which reduce the ohmic resistance provided from the presence of a coating between the metallic substrate and the corrosive environment<sup>5-6</sup>;
- The adhesion to the substrate;
- The porosity and thickness of coating film; and
- The cleanliness of the substrate from grease and rust.

Corrosion resistance of coatings is dependent on their barrier properties and generally varies with the nature of the film-forming polymers and paint formulation used. It has been found that permeation of moisture and corrosive gases negatively affects adhesion and, in many cases, results in corrosion of the substrate. Failures in coating adhesion also deteriorate the protective properties of coatings.

The barrier properties of polymer films towards water have been shown to be dependent on the sorption and the diffusion behavior of water. Sorption-desorption studies indeed show that moisture readily passes into the paint films, but that the rate of desorption varies according to the type of binder and pigmentation used.<sup>7,8</sup>

Permeation of gases through the film has been shown to involve three main processes: the dissolution of permeating species in the films at the high-pressure surface; diffusion of dissolved gases through the film along a concentration gradient; and re-evaporation of gases from the low-pressure surface of the film.

For corrosion protection by the application of a surface coating to the substrates, four strategies normally are applied:

1. Barrier coatings to separate the electrodes from the electrolytes;
2. Addition of inhibitors/passivating partially soluble inhibitive pigments and/or additives;
3. Addition of cathodic/sacrificial protection (for example, zinc, phosphates, borates, molybdates, or zirconate-rich coatings); or
4. Improvement of adhesion to reduce water and oxygen permeation.

Table 1—Research Targets

| Target Performances                               |                                  |                                   |
|---|----------------------------------|-----------------------------------|
| Product Description, Key Specification Properties | ACE Applications (Superdurable)  | Industrial Applications (Durable) |
| Substrate   | Solely shot-blasted steel        |                                   |
| Salt spray test [hr]                              | Desired: 1000 (at least 240)     |                                   |
| Humidity [hr]                                     | Desired: 1000 (at least 240)     |                                   |
| Weathering  | 3 years Florida and 600 hr QUV-B | 250 hr QUV-B                      |
| Impact resistance [in/lb]                         | 40                               | 160                               |
| Gloss at 60° [%]                                  | 80 to 90                         |                                   |
| Thickness [µm]                                    | 75–150, one coat                 |                                   |
| Solvent (acetone) resistance <sup>a</sup>         | good                             |                                   |
| Edge coverage                                     | good                             |                                   |

(a) Acetone resistance: The method consists in evaluating the variation of appearance and the softening of the coating film subjected to contact with a cotton pad soaked in acetone. The cotton pad soaked in acetone is left over the paint for 30 min. After this time, it is removed and the acetone is left to evaporate before evaluating, with 0=best (no coating variation) and 5=worst (coating fully removed).

### Methods for Improving Corrosion Resistance

The typical methods for improving corrosion resistance are<sup>9</sup>:

- **Substrate Pretreatments:**
  - ◊ *Conversion Coatings*—Conversion coatings act as a base for paints and at the same time provide corrosion protection. They are a slightly acidic aqueous solution (water-based) of chemicals. Iron or zinc phosphates are the most common chemicals in the formulation, although other chemical salts are also added to perform various functions. The metal is immersed in a tank containing the solution and, while immersed, the metal dissolves very slightly and the phosphate actually plates out onto the clean metal. The coating thickness is normally less than 100 nm. Both zinc and phosphate are considered to be environmentally unfriendly.
  - ◊ *Wash Primers*—Wash primers passivate the surface and temporarily provide corrosion resistance, providing an adhesive base for the next coating. They are water-based formulations of phosphoric acid in solutions of vinylbutyral resin, alcohol, and other ingredients such as zinc chromate and have a dry film thickness of 8–13 microns.
- **Electrocoating (in OEM and ACE Processes):**
  - ◊ This method employs an electric current to deposit an organic finish and is a process that uniformly applies thin-film primers and one-coat finishes to metallic substrates. Four steps are involved in this process: (1) substrate cleaning, (2) conversion coating, (3) sealing, (4) drying-curing, and cooling.

The coating thickness is dependent on the immersion time, the bath temperature, the process voltage, and the bath chemistry.

- **Use of Main Primers:**
  - ◊ Main primers are used to protect the substrate from corrosion for at least 5 to 15 years. They “seal” the surface so that the solvents or water of topcoats will be able to evaporate away as designed, and will create a barrier to oxygen, moisture, and corrosive compounds at the metal surface. The most common binder chemistries are polyurethanes, epoxy, moisture-cured urethanes, and fluor polymers (mainly used in coil coatings).<sup>10</sup> Additives that can be incorporated into primer coatings are: anticorrosion pigments/corrosion inhibitors, active pigment corrosion inhibitors, or passive pigment corrosion inhibitors.

It is important to remember that in ACE applications, coatings are required to keep both protective and aesthetic performances for a long time. However, degradation of aesthetics, like blistering and dripping of rust, happens earlier than the serious degradation of the structural properties of the metallic substrate.<sup>11</sup>

### Analysis Methods

Typical analysis methods performed to check corrosion resistance are:

- *Salt Spray Test (ASTM B-117)*: A 5% sodium chloride solution is sprayed by means of a nozzle into a closed chamber to produce a static fog. The panels with an Andreas cross (or vertical scribe) are suspended in this for a prescribed period of time, while the temperature is kept constant at 35 °C. The scribe is needed to verify the behavior of the damaged coating and substrate in the corrosive environment.
- *Controlled Humidity Test (ASTM D2247)*: This test estimates the influence of moisture on corrosion. The samples are exposed to 100% relative humidity (RH) at 40 °C.
- *Filiform Corrosion Test (ASTM D2803)*: Scribed panels are placed in a corrosive atmosphere (salt spray for 4–24 hr) or immersed in a salt solution and are exposed to humidity (25 °C and 85% RH).
- *Prohesion ASTM G 85-A5*: Scribed panels are placed in a weaker corrosive atmosphere with alternating cycles at room temperature and at 35 °C.
- *Electrochemical Tests*: Electrochemical tests like electrochemical impedance spectroscopy (EIS) on intact or scribed panels have the advantage of continuously recording the variation of resistance and capacity, and are able to relate with the corrosion mechanism. However, they need a skilled person in order to fully understand the results. Additional electrochemical test methods include potentiodynamic polarization, as well as others.

**Table 2**—Powder Coating Formulation Based on  $\beta$ -Hydroxy-alkyl-amide

| Raw Material                 | Charge (g) |
|------------------------------|------------|
| Carboxylated polyester       | 570        |
| $\beta$ -Hydroxy-alkyl-amide | 30         |
| Benzoin                      | 5          |
| Flow promoter Masterbatch    | 50         |
| Titanium dioxide             | 345        |
| Total                        | 1000       |

Our objective was to develop a solution to answer the critical market need to introduce a more environmentally friendly powder coating solution that provides outstanding corrosion resistance even when applied on less pretreated substrates. Although there will be intermediate offerings, the project's ultimate goal remains to provide powder coating resins able to substitute in terms of corrosion resistance performance, even when applied on solely shot-blasted panels, epoxy powder coatings, or the actual benchmark powder coatings applied on standard pretreated substrates (initially, iron phosphate and, in the future, zinc phosphate).

Moreover, the study will demonstrate that the improvement of corrosion resistance performance on solely shot-blasted steel is clearly replicated when the same powder coatings are applied on iron or zinc phosphate pretreated panels.

## EXPERIMENTAL RESULTS

Table 1 summarizes the targets of this project. With the exception of salt spray resistance, the other goals are typical application properties for superdurable offerings for ACE application and for industrial powder coating systems, which are also explored.

Solely shot-blasted steel substrates have been selected as the ultimate target, since they are more commonly available and are the most stringent from a corrosion perspective.

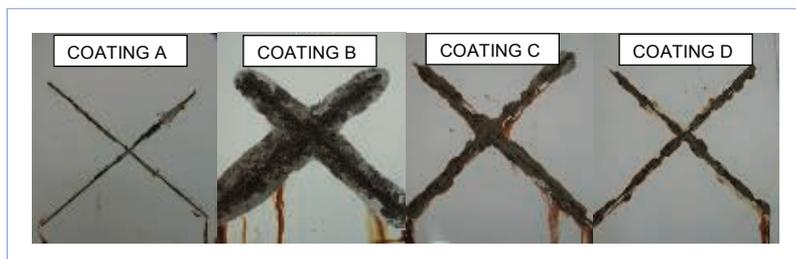
The powder coatings are applied by electrostatic spray deposition, using a corona charging system, which imparts the electrostatic charge to the powder coating. A voltage source generates an electrical field between the application gun and the substrate. The powder coating is fed through the gun, and sprayed onto the grounded metal substrate using compressed air. As the powder coating passes through the electrostatic field at the gun tip, it is charged and is attracted to the grounded metal substrate. Once applied, the coated substrate is put in an oven at the reported temperature and time. There, the powder coating initially melts and later cures.

**Table 3**—Salt Spray Resistance After 500 Hours for Superdurable Powder Coatings Based on  $\beta$ -HAA

| Superdurable Polyester | ACE Coating A based on REF-SD-PES | ACE Coating B based on REF-SD-PES | ACE Coating C based on NEW-SD-PES1 (version 1) | ACE Coating D based on NEW-SD-PES1 (version 2) |
|------------------------|-----------------------------------|-----------------------------------|--|--|
| Substrate pretreatment | Iron phosphate                    | Sand blasted                      | Sand blasted                                   | Sand blasted                                   |
| Creep [mm]             | 0–3                               | 10–15                             | 4–6  | 3–5  |

(a) Iron phosphate: with iron phosphating as passivation agent

(b) Sand blasted: without chemical treatment—steel, rolling mill surface Ra 0.9–1.8  $\mu$ m.



**Figure 1**—Salt spray results after 500 hr for REF-SD-PES on pretreated (Coating A) and on untreated (Coating B) and NEW-SD-PES1v1 (Coating C) and v2 (Coating D).

## SUPERDURABLE POWDER COATINGS BASED ON $\beta$ -HYDROXY-ALKYL-AMIDE FOR ACE APPLICATION

The powder coating formulation used for all the tests is reported in Table 2. It contains 65% of binder and is based on a polyester and  $\beta$ -hydroxy-alkyl-amide hardener, with titanium dioxide only in the pigment-filler part. It is exempt from any anticorrosive pigments or additives.

The reference grade used in this part of the study is a superdurable polyester (REF-SD-PES, supplied by Allnex), used in many ACE applications. The new generation of polyester resins developed and presented in this article is geared toward:

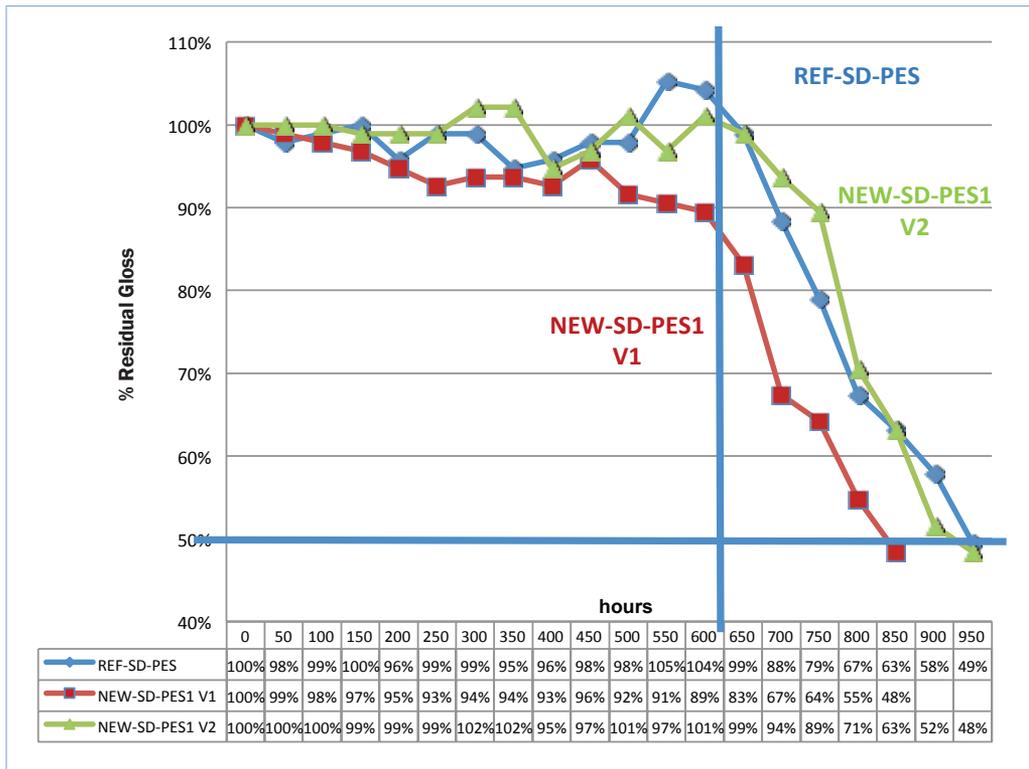
- Reducing the permeation of humidity and oxygen with formation of ions at the interface coating-substrate through:
  - ◇ The improvement of barrier performances;
  - ◇ The increase of adhesion of the coating to the substrate: providing good wetting of the substrate by the coating system and reinforcing the wet adhesion of the polymer; and
- The increase of stability of the coating to its environment, hydrolysis, UV radiation, and to thermal stress.
- Inhibiting the corrosion through the increase of inhibition/passivating performances, mainly with the help of additives.

Different polymeric structures have been tested initially for their salt spray resistance and subsequently for all the other typical application performances.

**Table 4**—Application Performances of Superdurable Powder Coatings

| Superdurable Polyester          | REF-SD-PES | NEW-SD-PES1(v1) | NEW-SD-PES1(v2) |
|---------------------------------|------------|-----------------|-----------------|
| Acid value [mg KOH/g]           | 31         | 35              | 35              |
| Viscosity [mPa.s at 200°C]      | 3500       | 1580            | 2500            |
| Glass transition [°C]           | 59         | 58              | 62              |
| Applicative test curing         | 200°C *10' | 200°C *10'      | 200°C *10'      |
| Gel time at 200°C [sec]         | 97         | 210             | 120             |
| Gloss at 20°-60° [%]            | 88- 96     | 88- 96          | 87- 97          |
| Impact [in/lb]                  | 0- 0       | 0-0             | 0-0             |
| Cross-cut adhesion <sup>a</sup> | G0T0       | G0T0            | G0T0            |
| Flow [mm]                       | 44         | 82              | 57              |
| Acetone resistance              | 3          | 4               | 4               |
| Thickness [µm]                  | 70-80      | 70-80           | 70-80           |

(a) Cross-cut adhesion is based on EN ISO 2409: The basic principle of cross-cut adhesion is to cut through the coating with a series of several cuts at right angles. On the square pattern obtained, Scotch® tape is well adhered. After 60-120 sec, the tape is removed and the coating is evaluated visually by examining the way in which the coating has broken away from the substrate and comparing it with the standard references. The result is reported as a judgment scale, with G0T0=best, completely smooth blades of the cuts without any loss of coating material, and G5T5=worst, with the coating material's loss of distinctly more than 65%.



**Figure 2**—Accelerated QUV-B test results of superdurable powder coatings based on β-hydroxy-alkyl-amide for ACE applications.

This work finally led to the development of a new ACE polyester for  $\beta$ -hydroxy-alkyl-amide (NEW-SD-PES1).

Table 3 and Figure 1 report the experimental results in terms of salt spray resistance after 500 hr for the reference product and two versions of the new development grade, where the second still requires future, full evaluation.

The improved creep performance of the NEW-SD-PES1v1 (Coating C and Coating D) in comparison to the REF-SD-PES is evident, when applied on the same untreated substrate (Coating B). It is still slightly worse when compared with the coating based on the reference applied on iron phosphate pretreated panels (Coating A). Similar promising results have been found with tests in a humidity chamber.

When looking at the other typical properties (as presented in Table 4), and again in comparison to REF-SD-PES, the viscosity of the NEW-SD-PES1(v1) and (v2) is reduced, while, when comparing the powder coatings, gel-time and flow are much longer. The lower reactivity does not impact the chemical resistance, which is demonstrated by the good solvent resistance (acetone test) result.

Recently, during the study of new methods of adhesion improvement, the second, alternative, version of NEW-SD-PES1, (v2), was developed, demonstrating even better salt spray resistance test results with a thickness of creep of 3 to 5 mm in comparison to 4 to 6 mm with the first version. However, as mentioned previously, further general applicative performance evaluations still need to be concluded for this resin.

Preliminary outdoor durability results of both NEW-SD-PES1 (v1 and v2) are in line with expectations, and are similar to the reference reported in Figure 2.

## INDUSTRIAL POWDER COATINGS BASED ON $\beta$ -HYDROXY-ALKYL-AMIDE

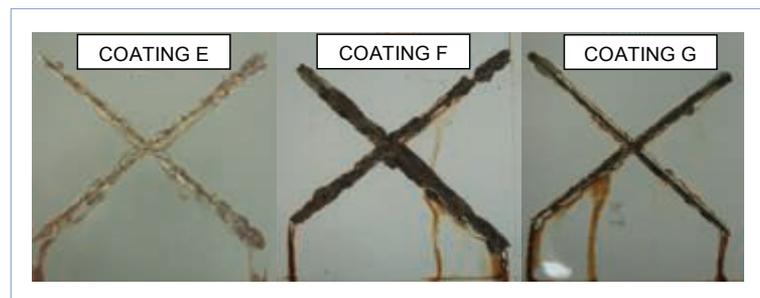
In a second area of interest, powder coating systems used in more general industrial applications were investigated, first in combination with  $\beta$ -hydroxy-alkyl-amide hardener. The powder coating formulation studied is the same as reported in Table 2, with 65% of the binder content based on a polyester resin and  $\beta$ -hydroxy-alkyl-amide hardener.

The reference grade is a well-known polyester resin for industrial applications (REF-IND-PES1). Additionally, and as a reference for the salt spray resistance test, a powder coating based on a pure epoxy (REF-EPOXY) was included, since this is the most resistant chemistry known today in terms of powder coating solutions.

Similar to the research work for ACE applications, several modifications of the resins' polymeric structures were developed and tested for corrosion resis-

**Table 5**—Salt Spray Resistance After 500 Hours for Industrial Powder Coating Systems Based on  $\beta$ -Hydroxy-alkyl-amide

| Industrial Polyester   | Coating E based on REF-EPOXY (Customer Coating) | Coating F based on REF-IND-PES1 | Coating G based on NEW-IND-PES2 |
|------------------------|---|---------------------------------|---------------------------------|
| Substrate pretreatment | Sand blasted                                    | Sand blasted                    | Sand blasted                    |
| Creep [mm]             | 2–4   | 4–6                             | 1–3                             |



**Figure 3**—Salt spray results after 500 hr for pure epoxy powder coating (Coating E), the reference (Coating F), and the new industrial polyester (Coating G).

**Table 6**—Application Performances of Industrial Powder Coating Systems Based on  $\beta$ -HAA

| Industrial Polyester       | REF-IND-PES1 | NEW-IND-PES2 |
|----------------------------|--------------|--------------|
| Acid value [mg KOH/g]      | 32           | 33           |
| Viscosity [mPa.s at 200°C] | 4500         | 4000         |
| Glass transition [°C]      | 66           | 65           |
| Applicative test curing    | 180°C *10'   | 180°C *10'   |
| Gel time [sec]             | 165 at 180°C | 140 at 200°C |
| Gloss at 20°-60° [%]       | 88–97        | 84–95        |
| Impact [in/lb]             | 160–160      | 160–160      |
| Cross-cut adhesion         | G0T0         | G0T0         |
| Flow [mm]                  | 35           | 36           |
| Acetone resistance         | 3            | 3            |
| Thickness [ $\mu$ m]       | 70–80        | 70–80        |

(a) Adhesion: G0T0=best and G5T5=worst  
(b) Acetone: 0=best and 5=worst

tance and other typical properties, and this led to the development of a new industrial polyester for  $\beta$ -HAA (NEW-IND-PES2).

Table 5 and Figure 3 report on the experimental results in terms of salt spray resistance after 500 hr for the pure epoxy (Coating E), the polyester reference product developed by Allnex (Coating F), and the new development grade (Coating G).

Table 5 and Figure 3 clearly show that the NEW-IND-PES2 (Coating G) represents a significant improvement in comparison to the REF-IND-PES1 (Coating F) in terms of salt spray resistance and

performs similarly to the REF-EPOXY powder coating solution (Coating E).

The new industrial polyester for  $\beta$ -hydroxy-alkyl-amide also demonstrated appropriate application performance, as reported in *Table 6*.

## INDUSTRIAL POWDER COATINGS BASED ON TGIC

These results, and the interest in the different markets like Asia and the United States where  $\beta$ -hydroxy-alkyl-amide is not the reference hardener, encouraged us to extend the investigation to the combination with triglycidyl-isocyanurate (TGIC).

The powder coating formulation used for all the tests is based on a ratio of 93 to 7 between polyester and TGIC with a 70% binder content in the presence of titanium dioxide as the only pigment (*Table 7*).

The reference grade used is a carboxylated polyester resin (REF-IND-PES2 used in Coating H) developed by our company for industrial applications. In addition to the same pure epoxy powder coating (Coating E), due to some similarities between the chemistries based on TGIC and  $\beta$ -HAA, the initial references also included Allnex new industrial polyester for  $\beta$ -HAA (NEW-IND-PES2, used in Coating I).

This research led to two polyesters, based on different modifications of the structure and additives, which provide similar improvements: the new

industrial polyester for  $\beta$ -HAA and a new industrial polyester for TGIC (NEW-IND-PES3, used in Coating J). Although these are still not at the same level as the pure epoxy powder coating, they are much better in comparison to the reference (Coating H).

*Table 8* and *Figure 4* report on the experimental results in terms of salt spray resistance after 500 hr for Coating H, the new development grades (Coating I and J), and the pure epoxy system (Coating E).

Coating I and J, in spite of the longer gel time, demonstrated good mechanical properties and, in general, typical application performance, as reported in *Table 9*.

## APPLICATION ON IRON AND ZINC PHOSPHATE PRETREATED SUBSTRATE BASED ON $\beta$ -HYDROXY-ALKYLAMIDE

In the case of powder coatings based on  $\beta$ -HAA, our investigation was extended to pretreated substrates. For this reason, salt spray resistance test times were increased to 1000–1250 in the case of iron phosphate and 2000 hr in the case of the zinc phosphate pretreatment.

The observed results shown in *Tables 10–13* confirm the improvement for both ACE and general industry applications when compared with their respective references. This led us to predict a similar improvement in the case of the developmental polyester for TGIC; these results are pending.

The same polyesters were evaluated for acetic salt spray resistance on nonchromate aluminum substrates. In this case also, the results were good, with no corrosion initially observed for all the involved coatings. However, at this time we are unable to better evaluate these results.

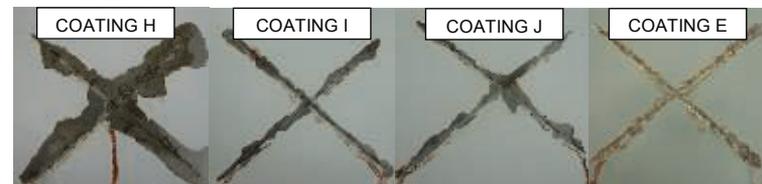
**Table 7**—Powder Coating Formulation for TGIC

| Raw Material           | Charge (g) |
|------------------------|------------|
| Carboxylated polyester | 638        |
| TGIC                   | 48         |
| Benzoin                | 4          |
| Flow promoter          | 10         |
| Titanium dioxide       | 300        |
| Total                  | 1000       |

**Table 8**—Salt Spray Resistance After 500 Hours for Industrial Powder Coating Systems Based on TGIC

| Industrial Polyester TGIC | Coating H based on REF-IND-PES2 | Coating I based on NEW-IND-PES2 | Coating J based on NEW-IND-PES3 | Coating E based on REF-EPOXY |
|---------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------------|
| Substrate pretreatment    | Sand blasted                    | Sand blasted                    | Sand blasted                    | Sand blasted                 |
| Creep [mm]                | 8–20                            | 2–6                             | 2–8                             | 2–4                          |

**Figure 4**—Salt spray results after 500 hr for the reference (Coating H), the new industrial polyester for  $\beta$ -HAA (Coating I), the new industrial polyester for TGIC (Coating J), and the pure epoxy powder coating (Coating E).



**Table 9**—Application Performances of Industrial Powder Coating Systems Based on TGIC

| Industrial Polyester TGIC       | REF-IND-PES2      | NEW-IND-PES2 | NEW-IND-PES3 |
|---------------------------------|-------------------|--------------|--------------|
| Acid value [mg KOH/g]           | 32                | 33           | 28           |
| Viscosity [mPa.s at 200°C]      | 5000              | 4000         | 4900         |
| Glass transition [°C]           | 68                | 65           | 61           |
| Applicative test curing         | 200°C *10'        | 200°C *10'   | 200°C *10'   |
| Gel time [sec]                  | 240 at 200°C      | 345 at 200°C | 480 at 200°C |
| Gloss at 20°–60° [%]            | 91–96             | 90–98        | 89–97        |
| Impact [in/lb]                  | 160–160           | 160–160      | 160–160      |
| Cross-cut adhesion <sup>a</sup> | G0T0 <sup>a</sup> | G0T0         | G0T0         |
| Flow [mm]                       | 51                | 70           | 54           |
| Acetone resistance <sup>b</sup> | 3                 | 3            | 3            |
| Thickness [µm]                  | 75-85             | 70-80        | 70-80        |

(a) Adhesion: G0T0=best and G5T5=worst  
 (b) Acetone: 0=best and 5=worst

**Table 10**—Salt Spray Resistance After 1250 Hours for Superdurable Powder Coatings Based on β-HAA for ACE Application Applied on Iron Phosphate Pretreated Substrate

| Superdurable Polyester | ACE REF-SD-PES | ACE NEW-SD-PES1(v1) |
|------------------------|----------------|---------------------|
| Substrate pretreatment | Iron phosphate | Iron phosphate      |
| Salt spray test [hr]   | 1250           | 1250                |
| Creep [mm]             | 10–20          | 4–9                 |

**Table 11**—Salt Spray Resistance After 1000 Hours for Powder Coatings for Industrial Application Based on β-Hydroxy-alkyl-amide Applied on Iron Phosphate Pretreated Substrate

| Industrial Polyester β-HAA | REF-IND-PES1   | NEW-IND-PES2   |
|----------------------------|----------------|----------------|
| Substrate pretreatment     | Iron phosphate | Iron phosphate |
| Salt spray test [hr]       | 1000           | 1000           |
| Creep [mm]                 | 25–35          | 15–25          |

**Table 12**—Salt Spray Resistance After 2000 Hours for Superdurable Powder Coatings Based on β-Hydroxy-alkyl-amide for ACE Application Applied on Zinc Phosphate Pretreated Substrate<sup>a</sup>

| Superdurable Polyester | ACE REF-SD-PES | ACE NEW-SD-PES1(v1) |
|------------------------|----------------|---------------------|
| Substrate pretreatment | Zinc phosphate | Zinc phosphate      |
| Salt spray test [hr]   | 2000           | 2000                |
| Creep [mm]             | 5–10           | 2–4                 |

(a) Zinc phosphate: with zinc-phosphating as passivation agent.

**Table 13**—Salt Spray Resistance After 2000 Hours for Powder Coatings for Industrial Application Based on β-Hydroxy-Alkyl-Amide Applied on Zinc Phosphate Pretreated Substrate

| Industrial Polyester β-HAA | REF-IND-PES1   | NEW-IND-PES2   |
|----------------------------|----------------|----------------|
| Substrate pretreatment     | Zinc phosphate | Zinc phosphate |
| Salt spray test [hr]       | 2000           | 2000           |
| Creep [mm]                 | 5–11           | 2–10           |

## FURTHER FINDINGS

Another interesting finding was related to the effect of the curing temperature, as reported in *Table 14*. We found that the higher the curing temperature, the better was the salt spray resistance. This is in line with the results of other studies that state that the crosslinking density appears to affect ionic conductivity of the film and reduces the permeability of water and ions through the coating film, leading to a structure that is more resistant to corrosion.<sup>5,6</sup>

New from the study results is that this is valid even on powder coatings expected to be already fully cured, like a binder based on polyester and β-hydroxy-alkyl-amide, and cured at 160 °C for 10' object temperature.

## ONGOING AND FUTURE WORK

While salt spray tests are running for TGIC-based industrial powder coatings on panels pretreated with the more effective iron phosphate, and because we are aware that salt spray resistance is a method with potential issues of reproducibility and repeatability, more care has been continuously paid by increasing the number of trials on identical coatings and confirmation of the salt spray results on the most promising systems in different laboratories. Moreover, other corrosion measurement techniques are being explored, such as electrochemical impedance spectroscopy.

**Table 14**—Salt Spray Resistance After 500 Hours for a Low-Curing Binder at Different Curing Temperatures

| Low-Curing Polyester/ B-HAA Ratio 93/7 | Curing 160°C *10' | Curing 180°C *10' | Curing 200°C *10' |
|--|-------------------|-------------------|-------------------|
| Substrate pretreatment                 | Sand blasted      | Sand blasted      | Sand blasted      |
| Creep [mm]                             | > 25              | 8–10              | 6–8               |

In parallel with further improvements in the presented grades, investigations are now under way, involving the evaluation in combination with TGIC for ACE applications.

## CONCLUSIONS

Salt spray corrosion resistance provided by powder coatings for general industry and ACE applications can be improved through use of the new standard and superdurable polyesters developed and reported here. The improvements are evidenced both on standard pretreated and untreated substrates.

Those new developments are targeting better adhesion and barrier properties, considered to be the best manner by which polyester can reduce the corrosion speed. The ultimate target being represented here from the development of a resin technology and related coatings is the ability to provide corrosion protection on untreated steel that has the same performance as the current pretreated steel. Additional studies and tests have been deployed in that respect. <sup>CT</sup>

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