AIR PRODUCTS

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Formulating novel aqueous epoxy resin systems for metal primer applications

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Formulating Novel Aqueous Epoxy Resin Systems for Metal Primer Applications

Abstract

Two component, waterbased epoxy coatings can be classified by the type of epoxy resin employed. Low molecular weight, liquid epoxy systems are commonly referred to as *Type I*, whereas systems based on higher molecular weight, solid epoxy dispersions are referred to as Type II. This paper will focus on coatings based on both types of resins.

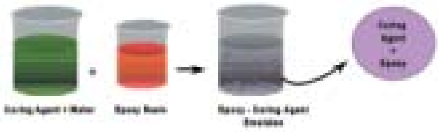
In the first case, novel curing agent technology for use with liquid epoxy resins will be discussed. Benefits of this technology include formation of high performance, zero VOC coatings, with a visible end to the pot life and excellent anti-corrosive properties. More recently a new *Type II* epoxy technology has been developed, this technology offers the unique combination of good film formation while also providing the option of zero VOC formulations.

Coatings formulated with this new resin exhibit properties on metal substrates that are equal in performance to conventional solvent based systems. Advantages include rapid lacquer dry, fast development of early water resistance and a long pot life. Performance properties of both technologies will be reviewed.

Introduction

Waterborne epoxy coatings have evolved into two fundamentally different technologies commonly identified as Type I and Type II systems[1]. Type I systems are based on liquid bisphenol A/F epoxy resins, with an EEW <250. The amine curing agent serves as the emulsifier for the epoxy resin and the emulsion, which contains both epoxy resin and amine is readily formed when the two components of the paint are mixed together.

Type I Waterbased Epoxy System



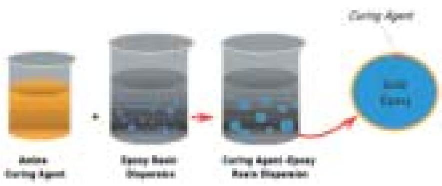
In Type I systems, the emulsion particles contain both curing agent and epoxy resin. After application of the coating, water slowly evaporates and the particles coalesce, if good coalescence occurs coatings will form that posses a relatively uniform film morphology develops.

Type I Waterbased Epoxy Film Formation

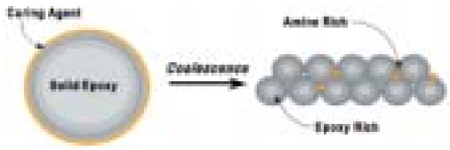
Curing agent and epoxy resin are both present in emulsion particle

Type II technology utilizes solid epoxy resin pre-dispersed in water and co-solvent. With these systems the dispersed resin particles contain only solid epoxy resin, therefore the curing agent must migrate from the aqueous phase into the dispersed epoxy particles for the crosslinking reaction to occur. As the coating coalesces, there is a greater tendency for films with a heterogeneous film morphology developing. Cross sectional analysis of resultant coatings has highlighted areas within the coating that are rich in both un-reacted epoxy resin and un-reacted amines[2].

Type II Waterbased Epoxy System



Type II Waterbased Epoxy System Film Formation



Curing agent reacts at surface and slows diffusion resulting in un-reacted epoxy at core

Type I systems can often be formulated to zero VOC coatings, primarily because liquid epoxy resins exhibit a good combination of handling, flow and film formation properties without the aid of co-solvents. A typical liquid epoxy resin (epoxide equivalent weight 190), consists of low molecular weight species plus a high concentration of epoxide groups. The high epoxide functionality and the close proximity the active amine-hydrogens within the same dispersed particle leads to rapid reaction within the particle.

As a result, the working life (pot life) and such systems are usually very short, typically 2 hrs. Liquid epoxy with its short rigid backbone yields cured coatings with high hardness but low flexibility and low impact resistance. While suitable for cementitious applications, liquid epoxy coatings can sometimes be too brittle for use on metal substrates, unless flexibility is introduced into the cured matrix from the amine curing agent.

To overcome the limitations of the Type I approach, waterborne systems based on high molecular weight solid epoxy resins were developed. Solid epoxies are supplied pre-dispersed at 50-55% solids in water and co-solvent. To aid in processing and to overcome the poor flow and coalescence of solid epoxies, 5-10% glycol ether is added to the dispersions thereby eliminating any possibility for zero VOC formulations.

Type II systems offer the handling and performance benefits associated with conventional solvent based solid epoxy resins. Dry times are very fast due to the lacquer dry of the high molecular weight resin and impact resistance is improved due to the flexible nature of the resin backbone. Pot life is longer due to the lower concentration of epoxide groups and also because the migratory effect of the amine curing agent, as previously discussed.

The principal weakness of Type II systems is their tendency to form heterogeneous films with epoxy rich and amine rich domains due to incomplete coalescence. This can result in films with inferior chemical resistance and poor barrier properties. Co-solvents and plasticizers are added at levels of 100-150g/l to improve film formation and to extend pot life. During the pot life, as amine reacts with epoxy, the minimum film formation temperature (MFFT)[3], of the polymer keeps increasing until the film can no longer coalesce. The end of pot life is not signaled by a viscosity increase but rather by decrease in performance properties such as a loss of gloss or a loss of corrosion protection. Typical properties of coatings based on these technologies are summarized in Table 1 below:

Table 1: Comparison of Type I and Type II Systems

Property	Type I Liquid Epoxy	Type II Solid Epoxy
Epoxy Equivalent Weight (EEW)	175-240	450-750
VOC (g/l)	0	100-300
Typical Pot Life	1-2 hours	> 4 hours
Typical Drying Speed	Slow	Fast
Key Drying Mechanism	Chemical Reaction	Lacquer Dry

The contents of the paper will demonstrate how recent changes in both the curing agent and epoxy resin chemistry have lead to the successful development of coatings for use in metal applications.

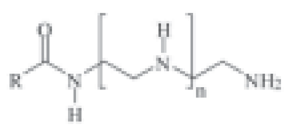
Curing Agent Chemistry for Type I Technology

The nature of the amine hardener for Type I systems serves a dual purpose. Initially it acts as an emulsifying agent for the liquid epoxy resin in the aqueous environment, and secondly it acts as a cross-linker. The degree and nature of the cross-linking thus giving rise to the ultimate performance properties of the coating. There are three generic types of curing agents, which have the chemical structure required to carry out these roles. The products concerned are (a) amidoamines **1**: condensation reaction products between monomeric, C_{18} fatty acids and polyalkyleneamines, (b) polyamides **2**: a condensation reaction between C_{36} dimer fatty acids and polyalkyleneamines and (c) amine adducts **3**: reaction products between an amine and an epoxy resin.

The base performance of products **1-3**, suffer from some inherent weaknesses, most common being poor resin compatibility leading to short pot lives, coupled with coatings that exhibit inferior water resistance when compared to their solvent borne counterparts. Modifications to the above structures and the techniques adopted during manufacture, has proved to be a major factor in the development of new products that address these deficiencies.

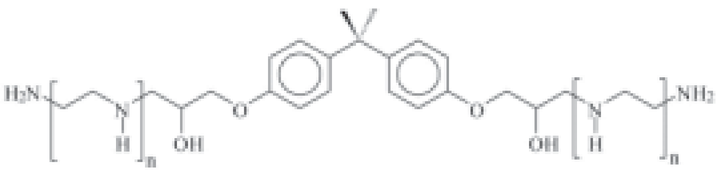
The most common method of modification involves reducing the primary amine content. This approach also serves a dual role. First, the overall reactivity of the curing agent is reduced, which is beneficial for extending the pot life of the system and second, is to dramatically improve the compatibility of the curing agent with the epoxy resin. Typical approaches at reducing the primary amine content involve reacting **1-3** with mono glycidyl ethers[4], formaldehyde or with unsaturated compounds such as acrylonitrile,[5] which are capable of undergoing Michael addition reactions.

Although modifications improve the compatibility of the amine hardener with the epoxy resin, the water solubility of the curing agent decreases and quite often the addition of an organic acid such as acetic acid is required to maintain a stable aqueous solution. Depending upon the level and nature of the acid employed, it can remain entrapped within the cured film and therefore result in a decrease in the water resistance and corrosive properties of the cured coating. The presence of acidic salting agents is also known to contribute to the phenomena of flash rusting. For these reasons, any modification employing organic acids must be kept to an absolute minimum.



1
R = $C_{17}H_{29-35}$
n = 1 - 4

2
n = 1-4



3
n = 2 - 4

Novel Curing Agent Technology

A new class of waterbased curing agent (Epilink® 701) has been developed.[6] The product is a high molecular weight aliphatic amine emulsion supplied at 55% solids in water. The product readily emulsifies liquid epoxy resins and in addition, the high molecular weight of the curing agent produces very fast dry times which are comparable to those observed with high VOC solvent borne coatings. By combining the drying properties of the curing agent with the good film formation properties of liquid epoxies, it is now possible to obtain good water resistance and good wet adhesion in a lacquer dry, zero VOC formulation. A key feature of the emulsified curing agent is the fact that it has been specifically developed to eliminate the effect of flash rusting, a feature all too common with existing waterbased epoxy technology. The ability of formulated coatings not to exhibit this phenomena, thus makes the curing agent an ideal product for use in 2 pack waterbased metal primers.

Formulations based on Novel Curing Agent

Anti-corrosion resistance primers and high gloss metal enamels have been developed using Epilink® 701. (see Appendix I & II for start point formulations). Formulation attributes for the two coatings are presented in Table 2.

Table 2: Formulation Attributes

Property	Anti-Corrosive Metal Primer (ACP01)	High-Gloss Metal Enamel (HGE01)
VOC (g/l)	0	0
Mix Viscosity (mPa.s)	500	1000
Weight Solids (%)	63.0	59.4
Volume Solids (%)	47.5	47.3
PVC (%)	30.0	15.6
Resin Stoichiometry (Epoxy:Amine)	1.0:1	1.0:1

For highest performance it is extremely important to ensure a high level of compatibility is achieved between the amine curing agent and epoxy resin. Optimum performance properties are obtained using either a liquid Bisphenol A epoxy resin or a blend of Bisphenol A/F resins diluted with an aromatic glycidyl ether.

The preferred diluents are cresyl glycidyl ether and p-t-butylphenyl glycidyl ether. End of pot life is determined by an increase in viscosity, which is clearly visible to the applicator. Residual paint forms a solid gel overnight for easy waste disposal.

For metal primer applications, optimum results for coatings based on Epilink® 701 are obtained for primers with a PVC in the range of 30-35%. Preferred anti-corrosive pigments include Halox SZP 391, Halox SW-111 and Heucophos ZMP, which can be added to paint formulations at 60-100g/l. Extender pigments are selected to give a variety of particle shapes and good filler packing. Epilink® 701 is an excellent pigment wetter so pigments can be ground into the curing agent side, without the need for additional pigment dispersing aids.

Novel Epoxy Resin Technology

An epoxy resin dispersion, (Ancarez® AR550) has been developed which incorporates the best characteristics of both Type I and Type II systems[7]. The unique chemistry of this epoxy resin enables the formulation of a Type II system that retains the advantages of such systems including high epoxy equivalent weight (EEW), long pot life and fast lacquer dry. Coatings based on this new resin also exhibit desirable performance attributes previously only found with Type I waterborne epoxy systems including zero VOC and lower cost-in-use.

Important aspects of the epoxy resin's properties include its volume average diameter (D_v) of 0.5 microns, consistent with other high-quality Type II dispersions. Particle size in Type II systems is important for two reasons. First, overall particle size distribution impacts film formation, emulsion stability and emulsion viscosity. Second, because the curing agent must penetrate and react with the epoxy resin in Type II systems, the smaller the particle size the greater the overall surface area of all the particles, and hence, the greater the opportunity for reaction between the curing agent and the epoxy resin. Typical properties for the new resin dispersion are given in Table 3.

Table 3: Resin Dispersion Properties

Appearance, liquid	Milky white
Appearance, film	Clear, Glossy
Solids content (wt. %)	55
Solvent	water
Viscosity @ 25 °C (mPa.s)[a]	100
Specific Gravity @ 25 °C	1.09
Epoxy Equivalent Weight (EEW, g/eq)[b]	712 on solids
Volume Average Diameter (D_v, microns)	0.5

a Brookfield viscosity, Spindle #3, 12 rpm, b Empirical calculation

Another important property of the Ancarez® AR550 resin is its molecular weight distribution. Molecular weight distribution impacts film formation, intra-particle viscosity and cure kinetics in waterborne epoxy systems. The unique manufacturing process of Ancarez® AR550 resin leads to a significantly larger percentage of lower molecular weight material than other established Type II solid dispersion resins. This allows Ancarez® AR550 resin to coalesce without the aid of co-solvents. Thus Ancarez® AR550 resin can be readily formulated to zero or low VOC coatings while retaining the film formation characteristics of Type II systems.

Formulations based on Novel Resin Technology

Coatings were formulated to evaluate the performance of the Ancarez® AR550 resin in two component ambient cure epoxy systems. The systems evaluated include a low-VOC, anticorrosive metal primer and an ultra-low VOC, high gloss metal enamel[8], (Appendix I & II). Formulation attributes for the two coatings are presented in Table 4.

Table 4: Formulation Attributes

Property	Anti-Corrosive Metal Primer (ACP02)	High-Gloss Metal Enamel (HGE02)
VOC (g/l)	76	6
Mix Viscosity (mPa.s)	500	1000
Weight Solids (%)	61.0	58.7
Volume Solids (%)	47.1	47.8
PVC (%)	30.0	15.5
Resin Stoichiometry (Epoxy:Amine)	0.8:1	1.15:1

Three important factors need to be considered when formulating Type II waterborne epoxies, these include: (a) coalescence, (b) stoichiometry and (c) stability. As previously mentioned, good coalescence is critical for uniform film formation and for good barrier properties. Ancarez® AR550 resin exhibits very good coalescence when used with either Anquamine® 401 or Anquamine® 419 curing agents. To further improve properties such as gloss or pot life, aromatic epoxy diluents such as Epodil® 742 (cresyl glycidyl ether) can be added to the Ancarez® AR550 resin at approximately 5% (w/w).

Changing the curing agent to epoxy resin stoichiometry is a valuable formulation tool. For optimum corrosion protection, 20-40% excess epoxy resin is desirable. Faster property development, in terms of dry speed and hardness build up are obtained when 10-20% excess curing agent is employed, whereas a good balance of properties is obtained at 1:1 stoichiometry.

Pigments or high levels of dilution can de-stabilize either the epoxy side of the formulation or the curing agent side of the formulation. When pigmenting the epoxy dispersion, a suitable dispersing agent dispersant should be added to avoid agglomeration of the epoxy particles. When pigmenting the curing agent side of the formulation, small amounts of acetic acid (0.2-0.5% based on curing agent weight) can assist in improving water solubility and avoid separation. This is especially true when formulating with hydrophobic curing agents.

Coating and Panel Preparation

The two components of the paint formulation are best mixed at high viscosity and then let down with water to the desired application viscosity. Salt spray and prohesion tests were conducted on coatings spray applied on to shot blasted steel (50 microns profile). The coatings (75-100 microns DFT), were allowed to cure seven days at ambient temperature before testing. Bonderite 1000 panels were used for humidity and Electrochemical (EIS) testing.

Handling and Performance Results of Waterbased Epoxy Coatings

Pot Life Characteristics: Pot life profiles are very much dependent upon the type of resin technology utilized. Formulations based on Epilink® 701 exhibit the typical Type I viscosity profile, moderate rise during the working life, followed by a more rapid rise leading to gelation. This usually occurs after 3-4hrs from the initial point of mixing.

The two coating formulations based on Ancarez® AR550 exhibit longer pot lives (4-6hrs). Viscosity changes over the pot life are minimal, with no sign of gelation occurring even after 24hrs. Pot life of coatings based on Ancarez® AR550 is viewed as the point where significant changes in gloss start to occur, resulting in its decreasing ability to coalesce. Pot life profiles via viscosity and gloss changes are given in Figures 1 & 2

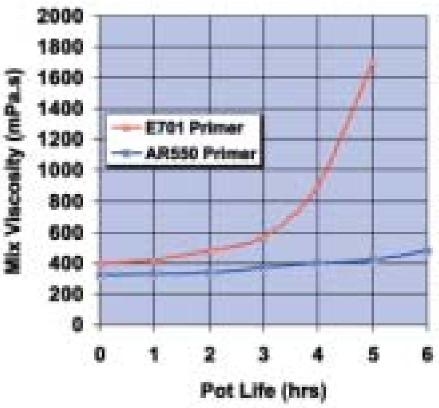


Figure 1: Pot Life via Viscosity

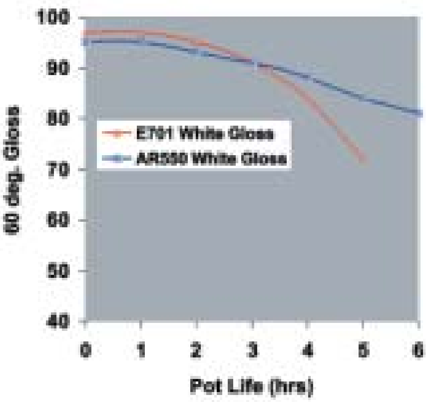


Figure 2: Pot life via Gloss

Base Performance Properties: Standard performance properties including hardness development, cross hatch adhesion, and the direct and reverse impact resistance were also conducted on the coating formulations. Results from these tests are summarized in Table 5.

Table 5: Waterbased Coatings Properties

Coating Formulation	Dry Times [a]	Persoz Hardness	Dry Adhesion	Wet Adhesion [b]	Direct Impact cm.kg	Reverse Impact cm.kg
AR550 Metal Primer *(ACP02)*	30 mins	140	5B	5B	30	6
AR550 Metal Enamel *(HGE02)*	30 mins	190	5B	4B	65	6
E701 Metal Primer *(ACP01)*	85 mins	220	5B	5B	25	4
E701 Metal Enamel *(HGE01)*	95 mins	300	5B	5B	50	4

a: Set to touch, b: 14 day cure; 1 week Cleveland Humidity exposure, ASTM D3359

Table 5 clearly highlights some of the fundamental differences between the liquid resin and solid resin dispersion technology. Coatings based on the novel resin dispersion exhibit rapid dry times (< 30 mins), whereas tack free times for coatings based on liquid resins cured with the new curing agent emulsion are slightly longer, taking ~ 90 mins to reach the same physical state. Ancarez® AR550 also gives rise to softer films compared to Epilink® 701; however, coatings based on the solid resin possess a higher level of flexibility. This is demonstrated by an improvement in both the direct and reverse impact resistance values. All coatings exhibited excellent adhesive properties over phosphate treated steel; with no significant loss of adhesion being observed after panels had undergone 24 hrs exposure to constant humidity.

Corrosion Resistance Properties: Anti-corrosive primer formulations were also subjected to accelerated weather testing. Tests included long term exposure to salt spray, condensing humidity and prohesion. Results of the weathering study are given in Table 6.

Table 6: Accelerated Weather Testing (1000 hrs exposure)

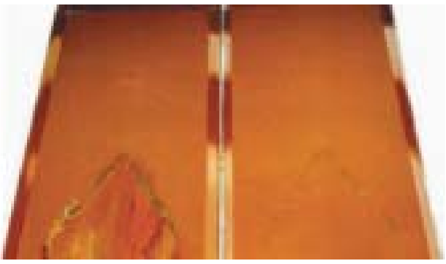
Formulated Coating	Salt Fog[a] Scribe/ Field	Prohesion[b] Scribe/ Field	Humidity Field
ACP01 (Liquid resin system)	10/10	7/10	10
ACP02 (Solid resin system)	9/10	7/10	10
Conventional Solvent Based Primer [c]	10/10	9/10	10

a. ASTM D117-90, b. ASTM B85-94, ASTM D-1654 ratings, c. Ancamide 260A/liquid epoxy resin

Field Blisters & Scribe : 10 - best, 0 – worst

The data presented in the table above clearly indicates that the anti-corrosive primers based on both types of waterbased systems can deliver exceptional, long term corrosion protection. After 1000hrs exposure, no signs of field blistering were observed on the panels under test. Salt spray and humidity resistance are excellent. Figure 3 depicts photographs of the panels based on ACP01 following 1000hrs salt spray & prohesion exposure.

Figure 3: Epilink® 701 Metal Primer (ACP01) Panels Following 1000hrs Accelerated Weather Tests



Salt Spray *Prohesion*

Data obtained during the prohesion study tends to indicate waterbased coating technology is more prone to scribe damage when subjected to this aggressive, cyclic test programme. Overall, the anti-corrosive properties of the waterbased coatings are comparable in performance to the results obtained for conventional solvent based polyamide primers.

Early Rain Evaluation: The anticorrosive metal primer and high-gloss metal enamel based on the Ancarez® AR550 resin were evaluated for early rain resistance by periodically applying de-ionized water to sections of a freshly applied coating. Coatings were judged to be rain resistant when they exhibited complete resistance to blistering and softening and only slight decrease in gloss was observed.

This evaluation revealed that after a cure of only 2-3 hours (see Table 7 below) the coatings were resistant to rain. Competitive Type II waterborne epoxy systems where high levels of co-solvent are required for good film formation need over 6 hours of cure time to achieve an equal degree of rain resistance.

Table 7: Summary of early rain evaluation of AR550 coatings

Formulation	Early Rain Resistance
Anticorrosive Metal Primer (ACP02)	3 hours
High-Gloss Metal Enamel (HGE02)	3 hours
Competitive Type II Waterbased Resin Primer	6 hours

Performance Evaluation using Electrochemical Impedance Spectroscopy

Electrochemical Impedance Spectroscopy (EIS) is a relatively new technique for measuring the barrier properties of coatings. EIS experiments are typically 24 hours in duration and correlate well with long term tests such as salt fog and prohesion. As shown in Figure 4, EIS involves exposing a coated steel panel to a salt water solution. A low AC voltage is applied to the steel panel. The electrical current that passes through the coating into the salt water is measured.

By varying the frequency of the AC voltage, coating performance can be measured. Key properties include the water uptake of the coating (capacitance) and the resistance of the coating to ion penetration (pore resistance).

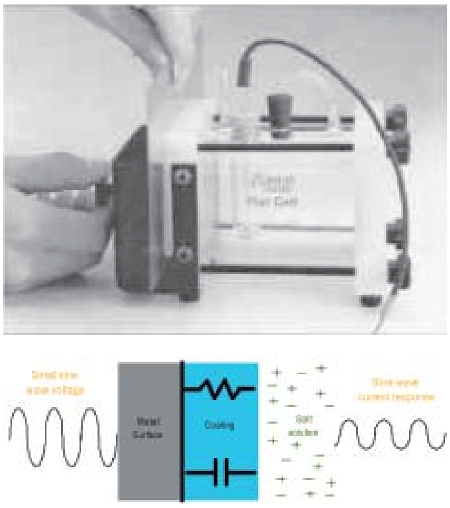


Figure 4: Electrochemical impedance spectroscopy cell

Resistance (Log Modulus [ohm]) is graphed vs frequency which provides all the appropriate information. A good barrier coating will exhibit low water uptake and high resistance to ion penetration, whereas a poor coating will exhibit high water uptake and very low pore resistance. Illustrations of the various types of EIS plots are given in Figure 5.

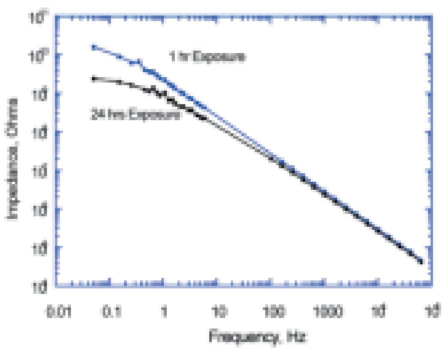


Figure 5: Sample EIS Plots of Barrier Coatings

EIS Plots for Waterbased Primers

The anticorrosive metal primers using Epilink® 701 and Ancarez® AR550 were each spray applied (75-100 microns DFT) to shot blasted steel panels (50 microns profile). Using a Gamry FAS1 Potentiostat with EIS 900 software, measurements were taken after 1hr and 24 hrs of immersion in 1 M NaCl solution for both formulations.

Figure 6: EIS Plot for ACP01

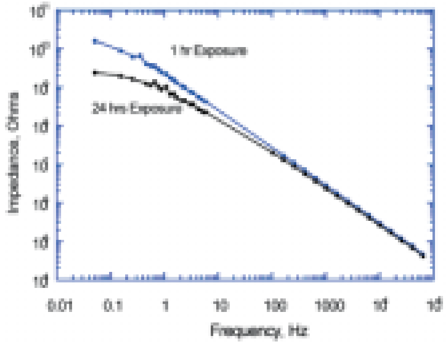
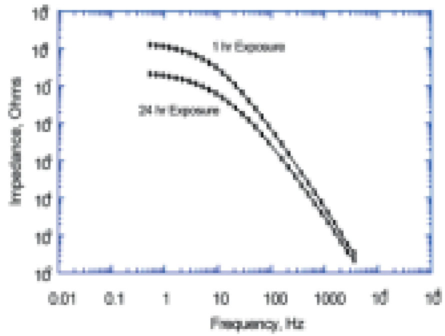


Figure 7: EIS Plot for ACP02



Electrochemical impedance data for the Epilink® 701 metal primer (Figure 6), highlights the excellent electrochemical resistance properties that can be obtained with Epilink® 701 in a Type I waterbased epoxy coating. The data shows that the coatings are highly resistant to ion penetration with a very low water uptake. Pore resistance values of 10^{9}Ohms and capacitance values of <0.5nF are obtained following 24hrs immersion in the salt solution. Primers based on Ancarez® AR550, exhibit slightly lower barrier properties as measured by EIS. For the cured film, pore resistance equivalent to 10^{7}Ohms was obtained following 24hrs immersion.

Capacitance values were comparable to those obtained for Epilink® 701 (~ 0.5nF). Despite the lower barrier properties, the values obtained for the above primer still indicates that it has the potential to offer good anti-corrosive properties. Test results obtained following the salt spray and humidity study clearly confirm our findings.

Summary

Innovative Type I and Type II waterbased technology has been developed that leads to high performance, ambient cure epoxy coatings. Typical industrial maintenance coatings developed from either the novel solid resin dispersion or from a liquid resin plus the new curing agent emulsion technology, were shown to deliver exceptional performance characteristics, compared to existing waterbased and solvent based systems. Based on the performance evaluations that have been conducted to date, the above products offer anti-corrosive properties, which make them ideal for a wide range of industrial metal protection applications.

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Appendix I

New Waterborne Systems – Anti-corrosive Primer Formulations

Waterborne Epoxy Primer based on Epilink® 701 Curing Agent (ACP01)

A Side	g
Dow DER 331	154.4
Cresyl glycidylether	38.6
Total	193.0

B Side	
Epilink® 701	300.6
Surfynol DF62	2.6
Disperbyk 190	13.1
Deionized water	204.3
Red Iron Oxide	77.7
Barium sulphate	177.6
10ES Wollastocoat	111.0
Talc (LVT400)	99.9
Halox SZP391	55.5
Total	1043.0

Properties:	
Volume solids	60.5%
Weight solids	72.5%
PVC	33.5%
VOC	0 g/litre
Mixing ratio (A:B)	1:4 (vol)
Pot life	3 hours

Waterborne Epoxy Primer based on Ancarez® AR550 Epoxy Resin (ACP02)

A Side	g
Deionized water	126.8
Disperbyk 190	12.5
Surfynol DF110D	3.0
Mica	10.0
10ES Wollastocoat	65.0
Halox SW-111	100.0
Red Iron Oxide	75.0
Barium Sulphate	65.0
Zeospheres 400	65.0
grind to Hegman 5 then add:	
Ancarez® AR550	430.0
Dowanol PM	3.0
Acrysol RM-8W	11.0
Total	966.3

B Side	
Anquamine 419	117.5
Deionised water	58.9
Total	176.4

Properties:	
Volume solids	47.1%
Weight solids	61.0%
PVC	30.0%
VOC	76 g/litre
Mixing ratio (A:B)	4:1 (vol)
Pot life	4 hours

Appendix II

New Waterborne Systems - White Gloss Enamel Formulations

Waterborne Epoxy Enamel based on Epilink® 701 Curing Agent (HGE01)

A Side	g
Epires ER8	25.0
Total	25.0

B Side	
Epilink® 701	40.0
Bentone EW 3% in water	1.0
Borchigol VL73S	0.4
Surfynol 420	0.1
Foamex 815	0.2
Kronos 2160	25.0
Blanc Fix Micro	15.0
Talc 10M2	2.0
DI water	16.4
Total	100.0

Properties:	
Volume solids	50.5%
Weight solids	71.4%
PVC	20.5%
VOC	0 g/litre
Mixing ratio (A:B)	1:4 (w/w)
Pot life	2 hours

Waterborne Epoxy Enamel based on Ancarez® AR550 Epoxy Resin (HGE02)

A Side	g
Ancarez® AR550	595.0
Acrysol RM-8W	2.1
DI Water	8.8
Total	605.9

B Side	
DI Water	70.0
Surfynol DF62	3.5
Anquamine 401	80.1
Glacial acetic acid	0.5
TiPure R706	250.0
Surfynol 420	1.5
Dowanol PM	3.0
Acrysol RM2020	2.0
Acrysol RM-8W	12.0
Total	472.6

Properties:	
Volume solids	47.8%
Weight solids	58.7%
PVC	15.4%
VOC	6 g/litre
Mixing ratio (A:B)	2:1 (vol)
Pot life	4 hours

For more information
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