High Performance Waterborne Coatings Based on Dispersions of a Solid Epoxy Resin and an Amine-Functional Curing Agent

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INTRODUCTION

Taterborne epoxy resin systems for ambientcured coatings have attracted much attention over the last several years. Compared to their solvent-based counterparts, waterbornes offer the advantages of lower levels of volatile organic compounds (VOCs), reduced odor, decreased flammability, improved safety, and easier clean-up with water. Despite the typically higher cost of the waterborne materials, these beneficial attributes have contributed to their increasing popularity for use in a variety of commercial and industrial applications.

Most commercially available waterborne epoxy coating systems are composed of a hydrophobic epoxy resin component and a hydrophilic, amine-functional curing agent. Classification of the various types of systems can be made on the basis of the physical form of the components in a given paint formulation.1-4 As shown in Table 1, five categories of systems have been described using that scheme.⁵ All of these waterborne epoxy systems share one characteristic—the epoxy resin is in the form of a dispersed, hydrophobic phase in water at the time of application of the coating. The most important commercial types, Types 1 and 2, differ in that the resin is either a liquid or a solid, respectively. Both types share the commonality that the curing agent is a relatively hydrophilic, water-soluble amine compound.

For the Type 1 and Type 2 systems, the significant difference in the hydrophobic/hydrophilic characters of the resin and the curing agent translates into a mismatch in their solubility parameters. This mismatch profoundly influences the nature of film formation in these materials. The film formation process involves the coalescence of the hydrophobic resin particles and the eventual diffusion/solubilization of the hydrophilic, amine curing agent into the resin phase. Although a number of factors, which include the phase viscosities, solvents, and the form of the components, can influence the process, the solubility parameters of the resin and curing agent should significantly affect the degree to which they mix at the molecular level. Simply by matching the solubility

vents, as judged by gloss and gloss potlife, were found to have low solubility parameters and high boiling points. parameters of the resin and the curing agent, the homogeneity and the resultant morphology of the films will be enhanced. Furthermore, it was postulated that one result of the improvements in film ĥomogeneity would be more robust performance over a wider range of amineto-epoxy stoichiometries.

A novel, waterborne epoxy system comprised of

non-ionic stabilized dispersions of a solid epoxy

resin and an amine curing agent has been de-

signed for ambient-cure coatings. The performance

characteristics of coatings formulated from the

new system have been compared to a standard

system. The new system produced formulated

coatings with robust performance over a wide

range of amine-to-epoxy stoichiometries (65 to

130%). At 100% stoichiometry, the new system

was found to have lower VOC, faster hardness

development, better gloss, and higher impact re-

sistance than the standard system. The new for-

mulations also exhibited superior water and salt

spray resistance, and a definitive viscosity rise at

the end of potlife. Unlike the standard system,

coating morphology of the new waterborne sys-

tem was found to be similar to a solvent-based

coating. For the new system, good coalescing sol-

Based on this hypothesis, in order to improve upon the performance of the Type 1 and Type 2 systems, the solubility parameters of the resin and the curing agent

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Table 1—Descriptions of the Types of Waterborne Epoxy Resin Systems for Ambient-Cure Coatings

Туре	Resin Form	Curing Agent Form
	. Liquid or liquid emulsion . Solid dispersion	Water soluble amine Water soluble amine
	. Liquid/solid emulsion	Acid- or amine-functional acrylic dispersion
	. Liquid or liquid emulsion . Solid dispersion	Amine dispersion Amine dispersion

need to be more closely matched. The most common and cost-effective epoxy resins are those based on bisphenol A, which makes them inherently hydrophobic and necessitates their supply in dispersed form in water. A more compatible curing agent, therefore, should be somewhat hydrophobic, which means that it will have low solubility in water. Similar to the case for the resin described earlier, a convenient way to supply such a curing agent would be in dispersed form in water, since a certain level of hydrophobicity is generally required for emulsification in water. Thus, a system comprised of two dispersions—one of a solid-type epoxy resin and the other of an amine-functional curing agent—could satisfy the requirement for similar resin and curing agent solubility parameters. Therefore, to fulfill that requirement, a new Type 5 waterborne epoxy system was developed. A schematic comparison of the Type 2 and Type 5 systems is shown in *Figure* 1.

Since the resin and curing agent components are distributed in different manners for the Type 2 and Type 5 systems, it was envisioned that their film formation processes might differ to some extent. Type 2 (and Type 1) systems, for example, have a water-soluble curing agent component that must diffuse into the resin particles from the aqueous phase to effect cure. Therefore, at any given time after mixing, relatively high concentrations of curing agent are present at the resin particle surfaces, which can become relatively hard. The hard shells of the resin particles retard complete coalescence, and that leads to a film morphology which consists of areas that are rich in either resin or curing agent. Indeed, it has been shown that in several of these systems, the film morphologies showed vestiges of the original resin particles, and that was attributed to partial coalescence.^{3,6} In contrast, for

Type 5 systems, the two phases remain separate until coalescence which, in this case, can simply be considered de-emulsification. This process can occur very rapidly and, if the viscosities of the dispersed phases are low enough, should produce some turbulent mixing at the particle level. If the two phases are relatively compatible, they should mix better than in the case of the Type 2 system and thereby produce films with a more homogeneous morphology.

Besides possible effects on the film morphology, there are other performance issues that are related to the degree of water solubility of the Type 2 curing agents. Many of the Type 2 curing agents have finite water solubilities, and over-dilution of the less water-soluble versions can lead to phase separation that may produce incompatibility with the resin. This phase separation can usually be overcome by the addition of volatile organic acids, such as acetic acid, which neutralize the amine groups and render the curing agent more water soluble. Although acetic acid is, in many cases, already present in the curing agent, additional acetic acid can lead to undesired loss of chemical resistance and detrimentally affect other cured-coating properties as well. Therefore, the extent of water solubility of the Type 2 curing agents can limit their utility in high PVC primer formulations to which relatively large amounts of water need to be added to reduce viscosities. For primers, minimizing or eliminating the acetic acid addition is desired to maximize cured-coating performance.

In comparison, Type 5 curing agents are in the form of aqueous dispersions and, therefore, viscosity reduction without any phase separation is readily accomplished with only water and without the need for acetic acid. Furthermore, the more water-soluble curing agents used in Type 2 systems tend to produce films with less aqueous resistance, while those that produce films with better water resistance usually have a somewhat lower water solubility. Since both the resin and the curing agent for the Type 5 system employed in this study were somewhat hydrophobic, improved aqueous resistance over a wider range of amine-to-epoxy stoichiometries was expected compared to the Type 2 materials.

A further improvement of the Type 5 system was anticipated to come from the use of novel, non-ionic surfactant technology to produce both the resin and

curing agent dispersions (emulsions). This new technology allows for the use of less surfactant relative to the standard Type 2 systems, and eliminates the use of acetic acid neutralization to render the curing agent water soluble. In addition, the surfactant was designed to reduce the sensitivity of the coatings to aqueous chemicals. The net result should be coatings with less sensitivity to aqueous chemicals.

Coatings formulated from the Type 5 waterborne epoxy systems were prepared and tested. This paper describes the results of those tests in the context of the discussion. Further-

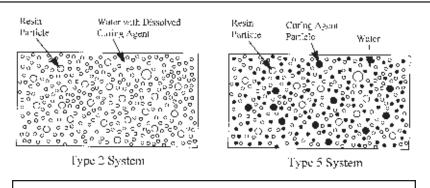


Figure 1—Schematic representation comparing the fundamental characteristics of a Type 2 system to a Type 5 system.

more, factors which affect the performance of coatings formulated with the Type 5 system are presented.

EXPERIMENTAL

Material Description

Properties of the resin and curing agent dispersions are provided in *Table* 2. The epoxy resin was based on a standard bisphenol A resin, while the curing agent was a bisphenol A epoxy-amine adduct type. Both the resin and curing agent were modified with a proprietary, specially designed, nonionic surfactant in order to render them self-emulsifiable. This surfactant differed from standard non-ionic surfactants in that it was incorporated into the molecular structure of the resin and curing agent and, consequently, virtually no free surfactant was present. Dispersions of the resin and curing agent were prepared via an inversion process, where water was added to the materials under agitation until an oil-in-water emulsion forms.

Coatings Formulation and Testing

Examples of the gloss white enamel and primer formulations have been described previously.⁵ The white enamel formulations were tested at stoichiometries of 65, 85, 100, 115, and 130%, while the primer formulations were evaluated at 85, 100, and 115% stoichiometries. For comparison purposes, standard formulations^{7,8} based on Type 2 system were prepared and tested. These formulations were chosen because of their relevance to systems in widespread commercial application. The standard formulations were

based on a polyfunctional epoxy resin dispersion and a polyfunctional, water-soluble amine adduct solution. The standard formulations were at 70 and 50% stoichiometry for the enamel and the primer formulations, respectively. Pigmentation for the Type 5 formulations and the standard primer was done in the resin component using a high-speed disperser, while the standard enamel was pigmented in a similar fashion in the curing agent component. Formulation properties are provided in *Table 3*.

Table 2—Properties of the Experimental Resin and Curing Agent Dispersions for the New Waterborne Epoxy Resin System

	Resin Dis	Curing Agent ^b		
Property	Used in Enamel	Used in Primer	Dispersion	
Equivalent weight, g/eqc	543	553	216	
% Solids by weight	56.3	57.0	43.8	
Density, g/cc, 25°C	1.1	1.1	1.1	
% VOC by weight		1.6	0.0	

Table 3—Formulation Constants for the New System at 100% Stoichiometry and the Standard Formulations. The Constants Were Calculated with the Exception of the Viscosity

	Type 5	System	Type 2 Standard	
Property	Enamel	Primer	Enamel ⁷	Primer ⁸
VOC, g/l (lb/gal)	120 (1.0) 15.9 54.7 43.1 ~3:1 68	34 (0.28) 30.5 63.9 49.8 3:1 68	240 (2.0) 18.8 54.1 40.7 2:1 64°	168 (1.4) 34.9 62.3 46.5 4:1 67 ^b
(a) After letdown with water. 80 KU (b) After letdown with water. 77 KU (c)	_			

Table 4—Enamel Formulation at 100% Stoichiometry Used for Testing of Formulation Variables. For the Various Formulation Modifications Tested, the Amount of Water Was Adjusted to Compensate for Mix Viscosity and Mix Ratio

Material	Trade Name/Supplier		Weight %
Part A Resin dispersion	Heloxy® Modifier 10/Shell Chemical Fluorad® FC-430/3M		36.5 0.3 0.2 5.1 1.9
Part B Curing agent dispersion	Disperbyk®-190/BYK-Chemie Dee-Fo® PI-4/Ultra Additives Ti-Pure® R-960/DuPont		4.9 0.2 0.2 18.3 4.8
—High spe	eed dispersion to Hegman 8—		
Acetic acid(Curing agent dispersion		Total	9.5 13.2 4.9 100.0

To prepare the coatings, the resin and curing agent components were blended with mild agitation until completely mixed. The mixed systems were allowed to induct for 30 min before being applied to solvent-washed (rinsed with isopropanol followed by methyl ethyl ketone and then air dried) cold-rolled steel panels (Q-Panel Co.–Type S, ground finish, 0.8 mm thick). The coatings were spray applied using a suction-fed spray gun (DeVilbiss). The average film thicknesses for the enamel coatings were in the range of 58-66 µm (2.3-2.6

Table 5—Formulation Constants for the Type 5 Enamel Formulation Given in Table 4 and the Type 2 Standard at 100% Stoichiometry. The Constants Were Calculated with the Exception of the Viscosity

Property	Type 5	Type 2
VOC, g/l (lb/gal) PVC, % Stoichiometry, % A:B mix ratio by volume Mix viscosity, KU, 25°C	16.0 100 1:1	252 (2.1) 18.1 100 1:8:1 65

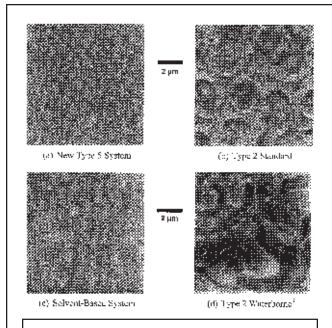


Figure 2—AFM micrographs of the surface morphologies for the Type 5 and Type 2 waterborne coatings and a solvent-based system.

Table 6—Compositions of the Clear Coating Formulations for AFM Testing

Material	Trade Name	1	2
Part A Resin dispersion Resin dispersion Solvent Solvent Deionized water	EPI-REZ® 5522-WY-55 ^b Dowanol® PPh ^c Shell Sol® Aromatic 150 ^b	54.4 5.0 12.9	66.4 — 3.0 5.3
Part B Curing agent dispersion. Curing agent solution Deionized water	Epi-Cure® 8290-Y-60 ^b	27.7 — — — 100.0	10.7 14.6 100.0

⁽¹⁾ Type 5 system. VOC = 140 g/(1.2 lb/gal).

mils), while the primer coatings averaged thicknesses of 61-84 μm (2.4-3.3 mils). Application, drying, and curing of the coatings were done at $25\pm1^{\circ}$ C ($77\pm2^{\circ}$ F) and $50\pm5^{\circ}$ relative humidity. After 14 days cure, the final property sets were determined, and the long-term testing was initiated. Testing was performed according to standard methods as described in reference (9).

In addition to the enamels pigmented in the epoxy dispersion component for the Type 5 system, an enamel formulation that was pigmented in the curing agent dispersion was developed. This formulation is shown in Table 4 at a stoichiometry of 100%. For comparison, a Type 2 standard formulation, which was formulated similar to the one described but at 100% stoichiometry, was used. Properties of these formulations are given in Table 5. Coatings were prepared and tested as described previously.

Atomic Force Microscopy

The atomic force microscopy (AFM) techniques used for these experiments have been reported previously.^{3,10} Standard surface scans covered an area of 10 µm by 10 μm at a scan frequency of 0.5 Hz. Clear coatings (*Table* 6) of the waterborne systems were prepared immediately (no induction period) after mixing and conditioned at 25 ± 1 °C (77 ±2 °F) and 50 ± 5 % relative humidity for at least four days prior to testing. A solvent-based formulation as reported in reference (3) was used for comparison; this formulation was allowed to induct for 30 min prior to coating.

Coalescing Solvents

In order to prepare formulations containing little or no hazardous air pollutants (HAPs), only solvents from the propylene glycol ether series (P-series) were utilized. A sample formulation is provided in *Table 4*. Pigmentation was done in the curing agent component using a high-speed disperser. Coalescing solvents were only added to and varied in the resin component. Effects of the solvents were determined by monitoring gloss changes through the pot life.

> The coatings were prepared according to the procedures described with the following exceptions. Coating of the mixed systems was initiated immediately after mixing. The formulations were applied to the steel panels by the drawdown method using a wire-wound drawdown bar (#70). Gloss was measured after the coatings were dried for 24 hr.

RESULTS AND DISCUSSION

Coatings Performance

The results of the performance of the new system coatings, which were pigmented only in the resin component, have been discussed elsewhere.⁵ As described earlier, a wide range of amine-to-epoxy stoichiometries were evaluated for the new system, and the results were

⁽²⁾ Commercial Type 2 standard. VOC = 270 g/l (2.3 lb/gal).
(a) Experimental, Shell Chemical.
(b) Water-soluble type, Shell Chemical.

⁽c) Dow Chemical.

Table 7—Comparison of the Properties for the Resin-Pigmented, Type 5 Enamel Coatings and the Curing Agent-Pigmented Type 2 Enamel Coating at the Formulated Stoichiometries

	Type 5 Systems by Stoichiometry					Type 2
Property	65%	85%	100%	115%	130%	System
Calculated VOC, g/I Dry time, hr	128	123	119	116	112	252
Hard gel (tack free)	5.75	4.0	3.0	2.0	2.5	10.5
Mar resistance (through)	8.5	8.0	8.0	4.0	8.0	16.5
Pencil hardness						
24 hr/14 days Impact resistance, inlb	5B/F	2B/H	HB/H	HB/H	HB/H	6B/H
Direct/reverse	24/4	36/4	36/12	52/36	56/16	16/<4
Adhesion	5A	5A	5A	5A	5A	5A
MEK resistance, double rubs	149	308	165	337	365	111
MIBK resistance, min	15	35	45	>60	>60	>60
25°C water immersion, daysa	>267	>267	>267	>267	113 ^b	>267
Salt spray, hr	960	960	960	432	264	960
ASTM field blister rating	8F	7F	6F-M	4M	2M	6F

compared to standard formulations, which were chosen for their relevance to commercially available coatings. Because of the low solvent content in the Type 5 resin and curing agent and the need for relatively low levels of coalescing solvents, very low VOC coatings were developed. Compared to the standard formulations, the new enamel and primer coatings showed robust performance over a wide range (65 to 130%) of amine-to-epoxy stoichiometries. As the data in Tables 7 and 8 show, the new system provided lower and non-HAP VOC, faster dry, more rapid hardness development, and good chemical and aqueous resistance. Notably, the aqueous resistance was found to be excellent up to an amine level of 115% stoichiometry; this was hypothesized to be the result of the hydrophobic nature of the resin and curing agents employed.

Because of the desire to use standards of commercial applicability, all of the comparisons noted were made using standard formulations at relatively low stoichiometries (<70%). Since the properties of the standard for-

mulations are known¹¹⁻¹³ to change significantly with the amine-to-epoxy ratio, a direct comparison of enamel coatings at 100% stoichiometry with pigmentation in the curing agent was performed. The properties of those coatings are provided in *Table* 9. In agreement with that discussed previously, overall performance of the new system was found to be better than the standard. The formulations based on the new system had lower VOC, faster hardness development, better gloss, higher impact resistance, and superior water and salt spray resistance. The better performance of the new system can be attributed to the improved compatibility of the resin and curing agent, the increased hydrophobicity of the curing agent, and the specially designed surfactant used for emulsification.

Coating Morphology

AFM micrographs of the surface morphologies are shown in *Figure* 2. Almost identical to that reported

Table 8—Comparison of the Properties for the Resin-Pigmented Primer Coatings at the Formulated Stoichiometries

	Type 5 Systems by Stoichiometry			Type 2
Property	85%	100%	115%	System
Calculated VOC, g/I	35	34	33	156
Hard gel (tack free)	30	30	30	210
Mar resistance (through) Pencil hardness	30	30	45	270
24 hr/14 days Impact resistance, inlb	H/H	H/H	H/H	В/Н
Direct/reverse	16/<4	12/<4	16/<4	16/<4
Adhesion	4A	4A	4A	5A
MEK resistance, double rubs	27	25	29	28
MIBK resistance, min	<5	<5	<5	<5
25°C water immersion, daysa	>387	>387	>387	>387
Salt spray, hr	528	528	528	528
Scribe undercutting, mm	0.5	0.0	0.0	2.0
ASTM field blister rating	8M	10/4M ^b	10	10

Table 9—Gloss White Enamel Coating Performance Comparison of New Type 5 System and the Type 2 Standard at 100% Stoichiometry

Property	Type 5 System	Type 2 System
Pencil hardness		
24 hr	5B	<6B
14 days	F	F
Gloss value		
60°	102	70
20°	90	24
Impact resistance, inlb		
Direct	>160	68
Reverse	>160	40
Adhesion	5A	5A
MEK resistance, double rubs	>300	>300
MIBK resistance, min	<5	30
Water immersion, daysa	65 (8F)	<23 (8D)
Coatings appearance	` '	Yellow
Salt spray, hr	1152	1152
ASTM field blister rating	10 (none)	4F
(a) Time to blister. ASTM blister rating in po	arentheses.	

previously, the standard waterborne system showed a series of interconnected ridge-like structures. In stark contrast, the surface of the new system showed a smooth, almost featureless topography. Despite no induction period, the surface morphology of the new system was observed to be almost identical to that of the solvent-based coating, which was allowed to induct for 30 min. With further analysis of the AFM data, an average (root-mean-square) surface roughness could be calculated over the entire scan area (10 μ m \times 10 μ m). This analysis showed that the average surface roughness (1.1 nm) for the Type 5 waterborne coating compared closely with that (0.6 nm) of the solvent-based coating, while the surface of the standard system was about 15 times rougher (15.3 nm) on average.

The ridge-like morphology of the standard system has been attributed to partial coalescence of the dis-

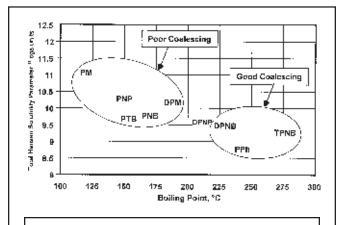


Figure 3—Results of non-HAP (P-series glycol ethers) coalescing solvent study. Good coalescing solvents were found to be those that had a relatively high boiling point and a low solubility parameter. Data labels are abbreviations for the solvents evaluated. See reference (14) for data and explanation of the solvent abbreviations.

persed resin particles. In comparison, the smooth surface morphology of the new system would appear to be indicative of virtually complete coalescence. This result supported the notion that the designed compatibility of the resin and curing agent for the new system leads to improved coalescence. Furthermore, the improved compatibility also eliminates the need for an induction period and, thereby, delivers the benefit of increased productivity.

Coalescing Solvents

As mentioned earlier, the effects of a number of formulating variables on the performance properties of the new system have been studied. Particularly, the choice of coalescing solvent was found to have a profound influence upon performance. *Figure* 3 shows the results obtained from evaluations of the individual solvents and their combinations. Based on gloss and gloss-potlife, it was observed that the best coalescing solvents were those that had both relatively low solubility parameters and high boiling points. This outcome can be rationalized as follows. Because the resin and curing agent are somewhat hydrophobic, it would be expected that their solubility parameters would be relatively low. Therefore, solvents which have comparable solubility parameters should be more soluble in the resin and curing agent mixture, where those solvents can act as plasticizers to lower the minimum film formation temperature. Since the processes involved in the formation of a smooth film are time dependent, a good coalescing solvent should remain in the film long enough for those processes to be adequately completed. In this case, higher boiling solvents with low solubility parameters were required to achieve the appropriate level of film aesthetics.

Figure 4 illustrates the exceptional gloss and long potlife that can be achieved when formulating with good coalescing solvents. At a low VOC level of about 200 g/l, DPnB (dipropylene glycol n-butyl ether), TPnB (tripropylene glycol n-butyl ether), and PPh (propylene phenyl ether) provided coatings with high 20° gloss values of about 90 for at least nine hours. The 60° gloss values were approximately 100 for all the solvents. Be-

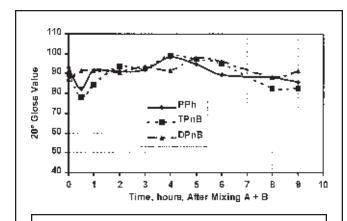


Figure 4—Gloss during the potlife for Type 5 coatings formulated with good coalescing solvents. Gloss was measured 24 hr after coating application. The coatings were formulated at a VOC of 204 g/l (1.76 lb/gal).

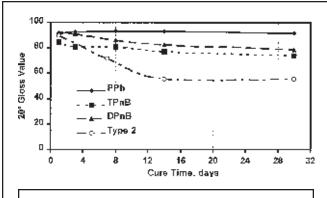


Figure 5—Effect of coating age on gloss for Type 5 system with good coalescing solvents versus the Type 2 standard coating. The Type 5 coatings were formulated at a VOC of 204 g/l (1.76 lb/gal) with 2% acetic acid on the basis of the total curing agent (RSW-2819). The Type 2 standard coating was at a VOC of 252 g/l (2.10 lb/gal). Induction time was one hour.

cause of the excellent compatibility of the new system, high gloss values are observed immediately after mixing of the components.

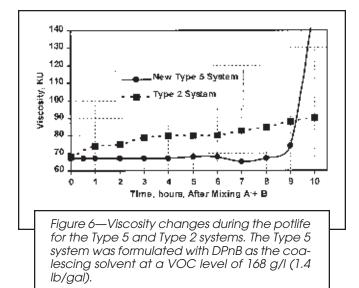
As shown in *Figure* 5, another aspect of the new system is that the gloss remained relatively stable as the coatings aged. In contrast, the gloss of the standard system decreased somewhat over the first several days of curing and after about 14 days reached a constant value. Presumably, the stable gloss of the new system was related to the improved resin and curing agent compatibility.

Potlife Characterization

During the investigation of the coalescing solvents for the new system, an unanticipated feature of the system was discovered. The end of the potlife was signaled by a relatively rapid, dramatic rise in the viscosity and subsequent gelation. This observation is shown in *Figure* 6. After mixing the components, the new system maintained a relatively stable viscosity for eight hours and, by the ninth hour, a slight increase in viscosity was seen. However, the material had formed a soft gel by 10.5 hr after mixing. The standard system, on the other hand, increased linearly in viscosity by 20 KU over the same time frame. Desirably, the viscosity changes clearly characterized the end of potlife for the new system, whereas the end of potlife for the standard system was less well defined. The reason for this behavior is not known, but it suggests that some inter-mixing of the resin and curing agent phases may occur. Further work is planned to help elucidate the mechanism responsible for this phenomenon.

SUMMARY AND CONCLUSIONS

A new waterborne epoxy resin system consisting of dispersions of a solid-type resin and curing agent (Type 5)



has been developed for ambient-cure coatings applications. This new system offers robust performance over a wide range of amine-to-resin stoichiometries. Performance advantages over the Type 2 system included lower and non-HAP VOC, faster dry, more rapid hardness development, and good chemical and aqueous resistance. Without an induction period, the surface morphology of the new system was observed to be more like that of a solvent-based epoxy rather than that of the standard system. This finding supported the conception that the designed compatibility of the resin and curing agent for the new system would lead to improved film coalescence.

In a study investigating the effects of non-HAP, P-series solvents, it was found that the best coalescing solvents were those that had relatively low solubility parameters and high boiling points. Appropriate selection of coalescing solvents led to the development of formulations with exceptional gloss and long potlives. The end of potlife was characterized by a distinct rise in the viscosity and subsequent gelation.

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