New Dibenzoate Plasticizer/Coalescent Blend for Low-VOC Coating Formulations

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Coatings formulators are increasingly faced with the challenge of reducing volatile organic compounds (VOCs) while providing a coating with optimal performance characteristics. To assist formulators, raw material suppliers continue to develop new, lower VOC products. As an answer to this need for compatible low-VOC coalescents, a next generation low-VOC dibenzoate plasticizer/coalescent platform has been developed. Low-VOC film forming aids allow the formulator to use conventional, well recognized emulsion systems in formulating paints and other coatings. An evaluation was conducted with three common base emulsions and formulations to demonstrate the utility of one of this new family of coalescents/plasticizers. This new film forming aid was evaluated primarily against newer low-VOC plasticizers/coalescents, including a second generation dibenzoate blend. The results of the evaluation indicated that this new low-VOC coalescent is compatible, efficient, and performs well in common emulsions used in latex coatings.

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Background

Reduction of VOC levels in coatings continues to require the attention of coatings manufacturers. Formulators are fighting to stay ahead of the curve by continually reducing their formulations' VOCs, most often by reducing or replacing the more volatile, components of the coatings. One typically volatile, but often very necessary, component is the film forming aid, which allows a coatings formulator to use conventional, well recognized latex emulsions. Also, to assist formulators in attaining their VOC goals, emulsion systems are being developed that do not require coalescents. As these polymers tend to be soft enough to allow a low minimum film forming temperature (MFFT), certain performance parameters can sometimes suffer.

Hydrophobic film forming aids such as 2,2,4-trimethyl-1,3-pentanediol monoisobutyrate (TXMB)[1] partition to the polymer in an emulsion and work by softening the dispersion particles of the emulsion during the coalescing phase of application. The coalescent will then partially or completely volatilize out of the film, allowing the film to regain its original physical properties which are reflected by such performance characteristics as scrub resistance and gloss. However, by its nature, this escaping coalescent contributes significantly to the VOC content of the film beginning with the coalescing phase and lasting for a sustained period afterwards. This can affect the air quality around the film and manifest as an unpleasant odor. Because of these issues, there has been a trend towards less volatile, more permanent film forming aids such as traditional plasticizers for the coatings industry.

Plasticizers have been used for years in coatings of various types because they are outstanding coalescents for latex paints and coatings; in some instances, they are desired as plasticizers because softening of the base polymer is required. It is also known that plasticizers can improve other paint performance characteristics.[2] However, in other instances, the permanence of plasticizers can be a detriment. For coalescents, a balance must be struck between greater permanence—and thus lower VOC—and good final film properties.

Phthalate plasticizers, such as di-n-butyl phthalate (DBP), diisobutyl phthalate (DIBP), or butyl benzyl phthalate (BBP), traditionally have been used in the coatings industry when a true plasticizer was required, as is the case when polymers with high T_g are employed in one application or another. DBP and DIBP are lower in VOC than traditional coalescents but are still somewhat volatile, while BBP is lower in VOC. However, phthalate ester use has an overriding issue other than VOC content, as DBP and BBP uses in particular are restricted due to regulatory concerns.[3]

Dibenzoate plasticizers have been recognized for various applications since about 1938. Their utility in coatings is also well known.[4] Dibenzoates by their nature are non-phthalates and do not have the restrictions or health concerns associated with phthalates. Monobenzoate ester coalescent technology has been developing since the 1980s, when isodecyl benzoate (IDB) was introduced to the coatings industry as a new, lower VOC coalescent.[5] IDB has compatibility in a wide variety of polymers and was found to be an effective, low odor coalescent in latex paint formulations versus TXMB as the coalescent.

At the beginning of the 21st Century, 2-ethylhexyl benzoate (2-EHB) was launched as a new lower-VOC film forming aid. In many formulations, 2-EHB was found to have greater efficiency than TXMB, as well as lower odor.[6,7] Isononyl benzoate (INB) was introduced in 2009 as a coalescent with a comparable formulated evaporation rate and efficiency to 2-EHB while exhibiting a more effective MFFT lowering behavior than IDB.[8]

Second generation benzoate ester coalescent technology has come about with the development of the less volatile dibenzoate esters. Newer dibenzoate products are an improvement upon existing dibenzoate plasticizer technology. Dibenzoates can satisfy reduced VOC requirements where the volatilities of monobenzoate esters are too high. This technology is based fundamentally on dipropylene glycol dibenzoate (DPGDB) and diethylene glycol dibenzoate (DEGDB) blends. Films formulated with these blends have been found to exhibit comparable performance properties to those formulated with TXMB, with improvements in scrub resistance, chemical resistance, and enhanced open times.[9,10]

Recently, a newer, next generation series blend of dibenzoate esters has been introduced.[11] The purpose of this article is to discuss the continued development of this product base by providing coatings performance data. The focus will be on one of the experimental products of the new, next generation plasticizer/coalescent platform.

Experimental

Introduction

Two common architectural latex paint emulsions and one emulsion commonly used in light industrial paint applications were selected to conduct the basic screen of the experimental blend. A vinyl acrylic emulsion (MFFT = 9 °C, T_g = 12 °C) was selected as the polymer for a flat interior formulation, and an acrylic emulsion (MFFT = 8 °C, T_g = 7 °C) was selected for an interior semigloss formulation.

A harder acrylic copolymer gloss emulsion (MFFT = 28ºC, T_g = 31 °C) was chosen to provide additional validation of performance. It is also recognized that the hard acrylic copolymer is not intended for architectural paint applications specifically, but is instead designed for light industrial applications. However, this polymer was selected to explore the compatibility of the new coalescent. The tests were the same as with the other paints to make comparisons easier when considering use of harder polymers with plasticizers as alternatives to softer polymers for low VOC. This is the first step in focusing on an alternate path to lower VOC coatings other than through the use of soft polymers.

Key formulation variables included:

- Vinyl acrylic flat paint—Volume solids = 34.8%, PVC = 58%, calculated VOC = 50 g/L
- Acrylic semigloss—Volume solids = 33.7%, PVC = 29.8%, calculated VOC = 50 g/L
- Acrylic copolymer gloss—Volume solids = 35%, PVC = 18%, calculated VOC = 160 g/L

The generic starting formulations are listed in *Appendix* 1.[12-14] Other than a determination of the proper level of coalescent required, no attempt was made to optimize the formulation for any of the coalescents.

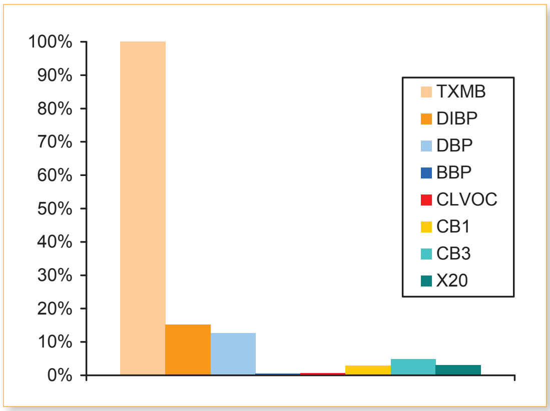

Figure 1—Determination of volatile contents of coalescents by ASTM D2369.

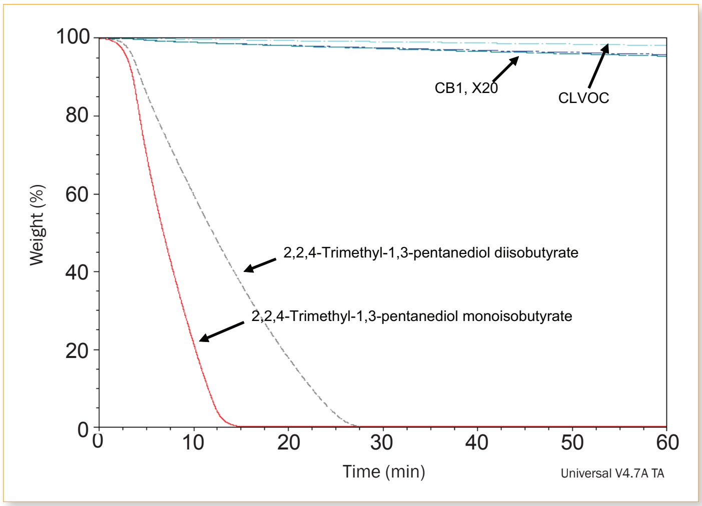

Figure 2—Determination of volatile content of coalescents via TGA.

The following plasticizers/coalescents were evaluated:

- 2,2,4–Trimethyl–1,3–pentanediol monoisobutyrate (TXMB): this is the standard in the industry and is certainly a very widely used product despite the fact that it is 100% volatile by the EPA 24 ASTM D2369 test method (only a portion of the evaluation is presented here);
- 2,2,4–Trimethyl–1,3–pentanediol diisobutyrate (TXDB): this composition can also be used as a plasticizer/coalescent. It is also 100% volatile by the EPA 24 ASTM D2369 test method (only a portion of the evaluation is presented here);
- Commercial low-VOC coalescent (CLVOC): a newer lower-VOC non-dibenzoate coalescent which is considered to be the primary control;
- A newer commercial blend of two dibenzoates (abbreviated as CB1);
- A traditional blend of two dibenzoate esters (abbreviated as CB3, only a portion of the evaluation is presented here);
- Experimental blend of three dibenzoate esters (abbreviated as X20);
- Butyl benzyl phthalate (BBP) is a common plasticizer used in some coatings. Used only in the VOC test;
- Di-n-butyl phthalate (DBP) can be used in coatings. Used only in the VOC test;
- Diisobutyl phthalate (DIBP) can also be used in coatings. Used only in the VOC test.

As this article addresses low-VOC coalescents, the first key test was the determination of the VOC of each of the coalescents tested. For this, an oven test of one hour at 110°C was used (EPA 24, ASTM D2369) as these data are used to calculate paint VOC from paint components as opposed to actually measuring the paint VOCs directly.

The paint evaluation consisted of the following:

- Wet paint and film formation—pH, viscosity, KU and ICI, freeze/thaw, flow and leveling, wet edge/open time, dry to touch, spatter, low temperature film formation, touch up (RT and low temperature), and mud cracking.
- Dry films—Scrub resistance, block resistance, contrast ratio, color and gloss.

Details of the test methods are listed in *Appendix* 2.

Plasticizer/Coalescent Volatile Content

Although the amount of coalescent used in a gallon of paint is not great, minimizing every component contributing to a coating's VOC is becoming essential. Even a raw material generally used at a relatively low level can be altered to significantly reduce the volatile content of the coating. Oven tests are a standard method to compare VOC contribution of volatile and semi-volatile components. EPA 24, ASTM D2369 testing is how the volatile contents of coatings are defined in the United States. *Figures* 1–4 illustrate the volatile content and plasticizer volatility of the coalescents of this study determined by three different methods.

The data in *Figure* 1 indicate that the low-VOC coalescents are indeed very low in volatility compared to TXMB. Of these plasticizers, DIBP is the most volatile. All of the low-VOC plasticizers/coalescents would contribute very little to the total VOC of a coating, especially at their low level of use. The data illustrated in *Figure* 2 were determined on a TA Q-500 thermogravimetric analyzer (TGA). It is quite clear that the low-VOC coalescents of the study were not only considerably low in VOC, as illustrated in *Figure* 1, but the rate of plasticizer loss of the low-VOC coalescents at 110°C is very small compared to TXMB and TXDB.

The data illustrated in *Figure* 1 were used to calculate the contribution of the coalescents of the study to the total VOC of the paints of the study. *Figures* 3 and 4 illustrate the results of these calculations. Even in the low-VOC formulations, TXMB makes a significant contribution to the VOC content of the paint. The low-VOC coalescents of the study contribute close to zero VOC. In the gloss paint, the TXMB makes a significant contribution to VOC whereas the low-VOC coalescents make almost no contribution. This gives the paint formulator increased latitude in formulating for performance such as freeze/thaw resistance or open time wet edge. The formulator may also want to formulate to improve other properties with the low-VOC coalescents as performance requirements may demand.

Coalescent Efficiency

Prior to initiating the paint evaluation, the efficiency of each coalescent was determined in the base paint formulations. Determination of the efficiency of a coalescent is often done on the binary pair and determining the MFFT of the blend of coalescent and base emulsion. As this is a preliminary study of a new plasticizer/coalescent platform, it was decided to establish each coalescent's efficiency level by determining the amount of coalescent required to obtain proper film formation at 40°F (4°C) in each of the paints. In this case, TXMB was the primary control for efficiency (as it is still a benchmark in this type of study). However, the results of this volatile coalescent were excluded from this article, as the data are discussed elsewhere.[11] The focus of this article is on low-VOC coalescents, where the best comparison is CLVOC, a commercially available low-VOC coalescent. For 100 gal, the flat paint required 6 lb, the semigloss 3.5 lb, and the glossy paint 38 lb (in addition to the Butyl Carbitol present) of each coalescent for proper coalescence.

Wet and Drying Film Characteristics

As seen in *Tables* 1–3, the performance data of the wet paint and drying of the paint were similar in all cases: pH, initial viscosity (Stormer and ICI), heat stability, flow and leveling, mud cracking (flat only, RT and low temperature), touch up (RT and low temperature), color acceptance, spatter resistance, and low temperature film formation (over sealed and unsealed substrate). Most of these parameters are very important as the coalescent choice has the potential to detract from performance in these areas.

One paint property that can suffer in lower VOC paints or coatings is freeze/thaw resistance. *Figures* 5 and 6 illustrate the results of three cycles of freeze/thaw on the vinyl acrylic flat and acrylic semigloss paints. The flat paints with dibenzoate coalescents had better freeze/thaw characteristics than those with the low-VOC CLVOC coalescent. In the semigloss formulation, all of the paints in the evaluation were superior to the CLVOC control. In the acrylic copolymer gloss formulation, all of the evaluated systems (including TXMB) failed after three cycles of freeze/thaw.

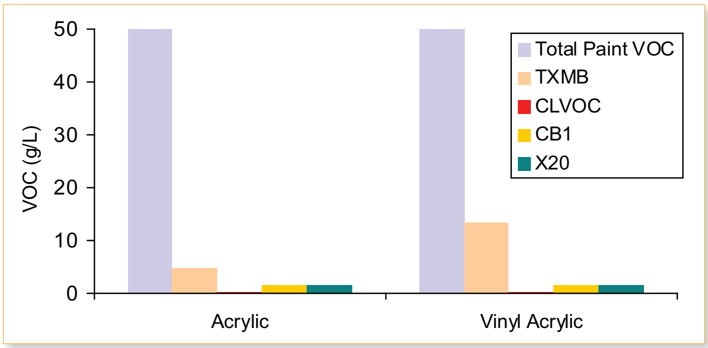

Figure 3—Total calculated VOC of acrylic and vinyl acrylic coatings and contributions to the VOC by each coalescent.

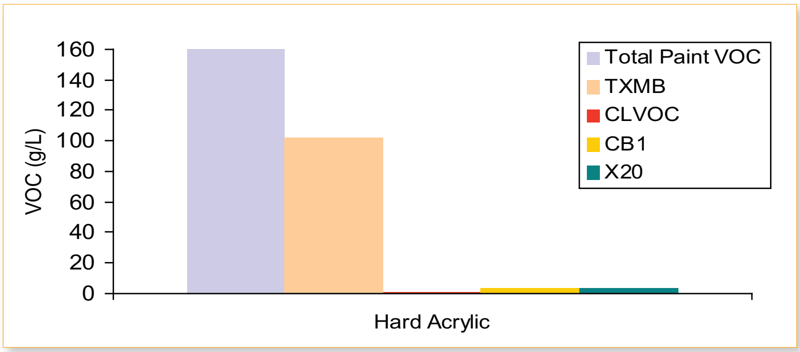

Figure 4—Total calculated VOC of hard acrylic coating and contributions to the VOC by each coalescent.

Table 1—Wet and Drying Paint Data for the Vinyl Acrylic Flat Formulation

Coalescent	**CB1**	**X20**	**CLVOC**
Viscosity, ICI, poise, 25°C	0.8	0.7	0.8
Stormer viscosity, KU, 25°C	100	100	100
Heat stability, 2 weeks at 120°F			
Viscosity, KU	97	97	98
Difference	-3	-3	-2
Spatter resistance	4	4	4
Low temperature film formation, 6 mils			
Sealed	10	10	10
Unsealed	10	10	10
Mudcracking			
Room temperature, mils with no cracking	40	40	40
Cold temperature (40°F), mils with no cracking	16	16	16
Touch up			
Room temperature	8	8	8
Cold temperature (40°F)	6	6	6
Color acceptance	10	10	10
Reflectance	90.6	90.5	90.5
Contrast ratio, 3 mils	0.960	0.958	0.960

Table 2—Wet and Drying Paint Data for the Acrylic Semigloss Formulation

Coalescent	CB1	X20	CLVOC
Viscosity, ICI, poise, 25°C	0.9	0.9	0.9
Stormer viscosity, KU, 25°C	108	109	110
Heat stability, 2 weeks at 120°F			
Viscosity, KU	109	109	111
Difference	1	0	1
Low temperature film formation, 6 mils			
Sealed	10	10	10
Unsealed	10	10	10
Reflectance	94.3	94.3	94.36
Contrast ratio, 3 mils	0.981	0.981	0.980

Table 3—Wet and Drying Paint Data for the Acrylic Copolymer Gloss Formulation

Coalescent	CB1	X20	CLVOC
Viscosity, ICI, poise, 25°C	0.5	0.4	0.4
Stormer viscosity, KU, 25°C	106	107	113
Heat stability, 2 weeks at 120°F			
Viscosity, KU	102	103	108
Difference	-4	-4	-5
Low temperature film formation, 6 mils			
Sealed	10	10	10
Unsealed	10	10	10
Reflectance	93.9	93.8	94.0
Contrast ratio, 3 mils	0.986	0.985	0.986

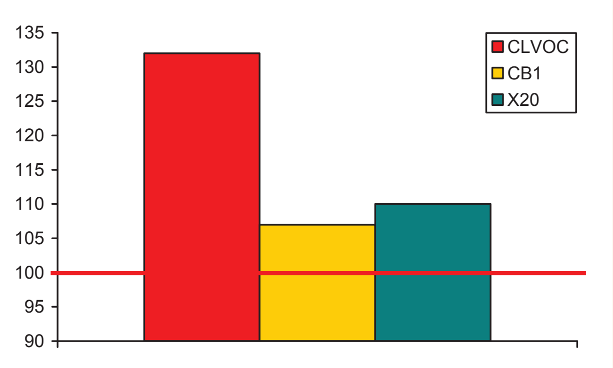

Figure 5—Vinyl acrylic flat viscosity stability after three cycles of freeze/thaw. Note: Initial viscosity was 100 KU for each formulation.

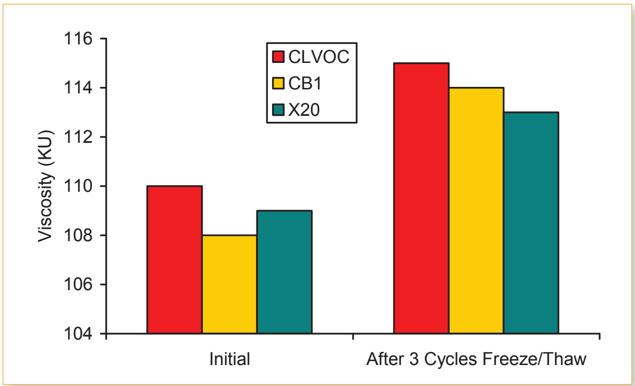

Figure 6—Acrylic semigloss viscosity stability after three cycles of freeze/thaw.

Wet edge/open time of the paint can certainly be affected by the coalescent type. It is known that plasticizers will extend wet edge better than volatile coalescents. *Figures* 7–9 present the data developed to describe this characteristic. The test used to consider wet edge or open time is somewhat different than other tests such as the one that the ASTM D01 committee is considering to describe this paint attribute. The wet edge characteristics for all three low-VOC coalescents appear to be equivalent in the acrylic semigloss and acrylic copolymer gloss coating systems, whereas the experimental X20 dibenzoate blend outperformed both CLVOC and the dibenzoate control in the vinyl acrylic flat system.

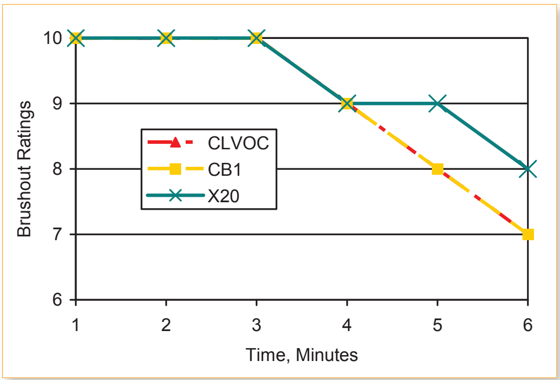

Figure 7—Vinyl acrylic flat wet edge/open time ratings.

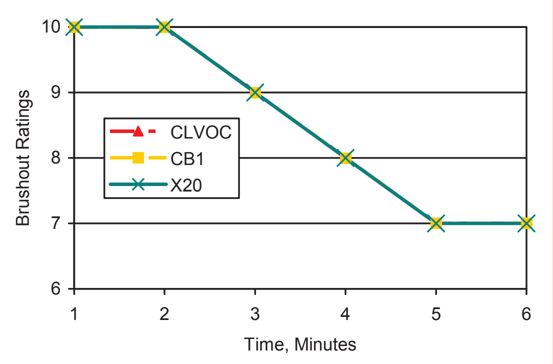

Figure 8—Acrylic semigloss wet edge/open time ratings.

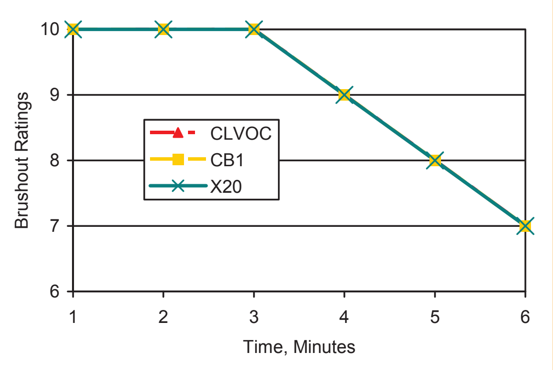

Figure 9—Acrylic copolymer gloss wet edge/open time ratings.

Dry Film Characteristics

Gloss is a good indicator of the film formation of a coating. The data in *Figures* 10 and 11 indicate that the low-VOC dibenzoate coalescents yield somewhat higher gloss in the semigloss formulation than CLVOC. In the acrylic copolymer gloss system, the X20 dibenzoate blend gave slightly higher gloss than CLVOC.

The job of a coalescent in a latex coating composition—whether it is a highly volatile organic compound or a true plasticizer for the polymer—is to assist in the process of film formation, particularly at low temperatures. The coalescent should also have a minimal impact on the coating's performance after the film is dry. One might assume that, due to their relative permanence, plasticizers may diminish film properties such as scrub; however, this is not necessarily so. *Figure* 12 illustrates how the dibenzoates had slightly better scrub resistance than the coatings formulated with CLVOC in both the flat and the semigloss paints. In *Figure* 13, the low-VOC coalescents' improvement of scrub resistance over TXMB is drastic, with the CB1 dibenzoate system showing an additional improvement over CLVOC.

Another indicator of film performance with plasticizers is block resistance. *Figure* 14 illustrates the block resistance data for the semigloss paints. Notably, all of the low-VOC benzoate plasticizers outperformed CLVOC on both room temperature and elevated temperature seven-day blocking tests. As stated earlier, there was no attempt at formulating to optimize performance. Instead, this study demonstrates the feasibility of the new generation plasticizer platform. In *Figure* 15, the acrylic copolymer gloss system formulated with X20 shows a distinct block resistance advantage at elevated temperatures over both CLVOC and the dibenzoate control after seven days of exposure, as well as an advantage over CLVOC after seven days at room temperature.

Conclusion

The data of this basic evaluation clearly indicate that the new dibenzoate triblend has a significant potential to provide the proper function as a plasticizer and low-VOC coalescent for coatings applications. In addition, the study indicates that the new dibenzoate triblend could deliver performance benefits in gloss and scrub resistance as compared to some of the current standard and low-VOC coalescent choices. X20 particularly shines in the more discerning acrylic copolymer gloss system, where

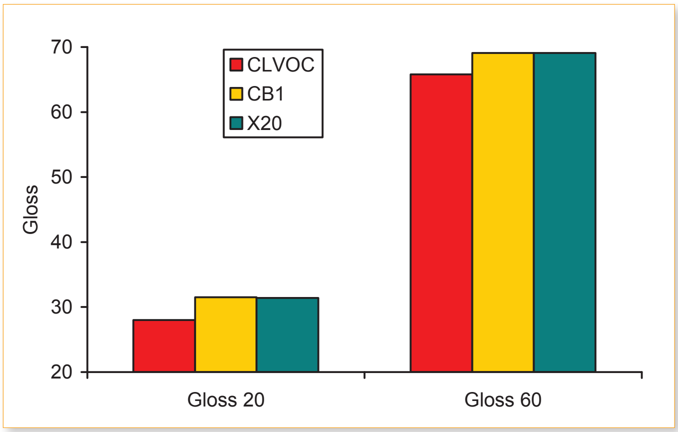

Figure 10—Gloss of the acrylic semigloss formulation.

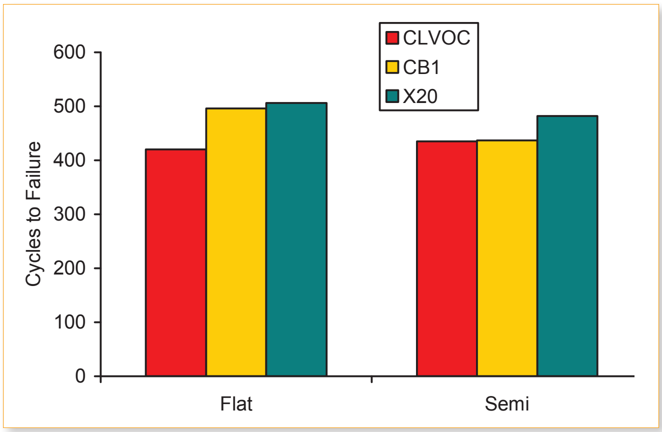

Figure 12—Scrub resistance of the acrylic semigloss and vinyl acrylic flat formulations.

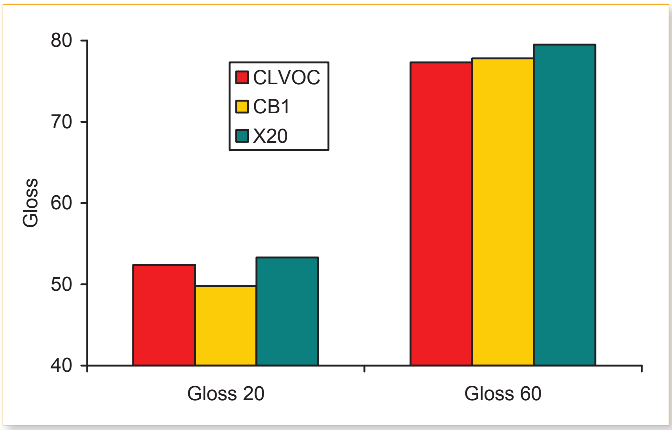

Figure 11—Gloss of the acrylic copolymer gloss formulation.

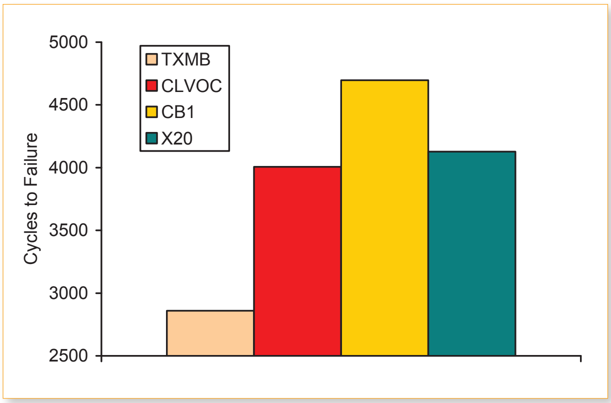

Figure 13—Scrub resistance of the acrylic copolymer gloss formulation.

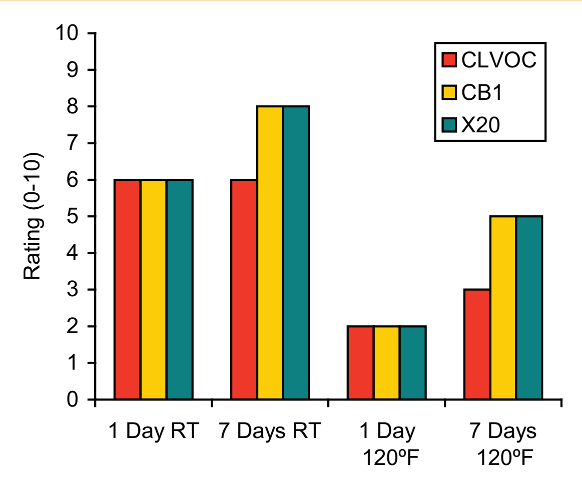

Figure 14—Block resistance of the acrylic semigloss formulation.

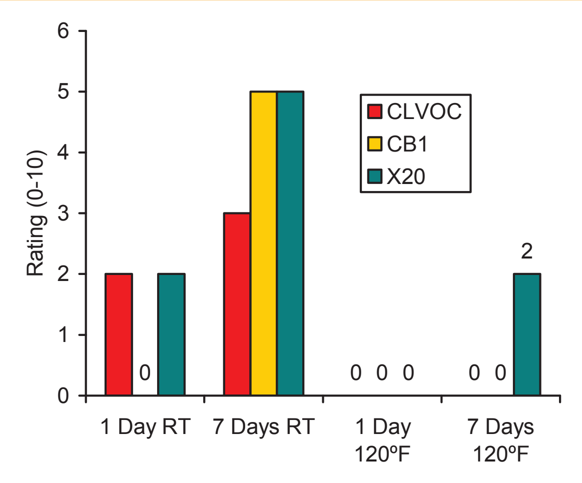

Figure 15—Block resistance of the acrylic copolymer gloss formulation.

it provides advantages to both scrub and block resistance. This product offers an opportunity to the paint formulator as an improvement upon current generation low-VOC coalescents.

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Appendix I

Table 1—Coalescent Abbreviations

Abbreviation	Coalescent Name	Coalescent Manufacturer
CB1	Blend of dipropylene glycol dibenzoate and diethylene glycol dibenzoate	Emerald Kalama Chemical
CB3	50:50 blend of dipropylene glycol dibenzoate and diethylene glycol dibenzoate	Emerald Kalama Chemical
CLVOC	Commercial non-dibenzoate type low-VOC coalescent	Several
TXMB	2,2,4-trimethyl-1,3-pentanediol monoisobutyrate	Several
X20	Triblend of dibenzoates	Emerald Kalama Chemical

Table 2—Acrylic Semigloss Paint Formulation[12]

Material Type	Amount (lb)
Water	219
Propylene glycol	7
Ammonium hydroxide (28%)	1.5
Pigment dispersant	12.5
Surfactant	2.2
Defoamer	1.5
Pigment	225
Extender pigment	10
Microbiocide	1.5
Binder	484
Polymer	43
Surfactant	1
Rheology modifier	17.4
Rheology modifier	4.3
Coalescent	3.5

Table 3—Vinyl Acrylic Flat Paint Formulation[13]

Material Type	Amount (lb)
Water	390
Propylene glycol	17.2
Thickener	5
Microbiocide	0.5
Pigment dispersant	9
Surfactant	2
Defoamer	4
Emulsifier	2
Pigment	100
Pigment	150
#1 White	190
Polymer	254
Coalescent	6

Table 4—Acrylic Copolymer Gloss Paint Formulation[14]

Material Type	Amount (lb)
Water	133.5
Ammonium hydroxide (28%)	4
Sodium nitrite, 15% aqueous	1
Coupling aid	20
Pigment	205
Pigment dispersant	9
Foam control agent	1
Surfactant	2
Resin (acrylic)	577
Rheology modifier	1.6
Rheology modifier	2.2
Coalescent	37.9
Total	1003.2

Appendix II—Summary of Test Methods Employed

Test	Reference/Method
pH	ASTM E70
Stormer Viscosity	ASTM D562
ICI Viscosity	ASTM D4287
Contrast Ratio, Reflectance, and CIE Values	ASTM D2805, E97, D2244—3 mil wet film over a Leneta 3B chart dried for five days. Contrast ratio is reflectance of black over reflectance over white.
Gloss and Sheen	ASTM D2243—3 mil wet film on Leneta 3B chart dried for five days.
Freeze/Thaw	ASTM D2243—Frozen at 0°C and thawed at ambient. Three cycles used.
Heat Stability	ASTM D1849—Tested at 120°F for two weeks. Initial and final viscosities taken.
Drying Time	ASTM D1640—3 mil wet film applied to Leneta 3B, set to touch determined at ambient.
Low Temperature Coalescence	Paint and equipment conditioned at 40°F for two hr. Paint drawn down on a Leneta Form HK to 6 mils wet. The films were dried horizontal for 24 hr and rated (see lab rating system below).
Lab Rating System	10=Excellent, 0=Very poor
Flow and Leveling	ASTM D4062—Leneta test blade used to apply paint. Dried paint rated.
Dry Adhesion	ASTM D3359B—Paint was applied to dried aged alkyd with a brush and dried for seven days before testing by cross hatch tape adhesion.
Wet Edge/Open Time	Paint applied with notched drawdown bar on Leneta WB chart. At one minute intervals, ¼ of 1" brush was dipped into the paint and brushed 10 strokes across the line. The wet edge was rated with the lab system.
Scrubbability	ASTM D2486—Paint applied at 7 mils wet to a Leneta P121-10N chart and dried at room temperature for seven days. A 10 shim was employed with abrasive media (SC-2). Failure was a continuous thin line at the shim.
Blocking Resistance	ASTM D4946—3 mil wet films applied to Leneta WB chart and the films were dried for seven days. Blocking was tested face to face at ambient and 120°F with a 1 Kg weight in place. The samples were separated and rated.
Color Acceptance	Tinted paint (with 2 oz/gal black) drawn down at 3 mils. After one minute, the paint is rubbed up in the unsealed area. The color acceptance is then rated.
Touch Up	Touch up was tested with the paint prepared for the color acceptance. Self primed Upsom was used and applied with a Linzer 2" Bristle and polyester brush at RT and 40°F and allowed to dry overnight. The test paint was applied and rated for sheen uniformity and color difference.
Roller Splatter	ASTM D4707—Black catch panel placed under an Upsom panel from the touch up test. After applying the test panel was evaluated via D4707.
Mud Cracking	Paint was applied with a Leneta Antisag meter (14–60 mils) on an HK chart at ambient and 40°F. After 24 hr dry, the greatest mils without cracking noted.
Oven Volatility	ASTM D-2369 using 1 mL toluene.
TGA Volatility	~10 mg samples were ramped at 20°C/min to 110°C, then held isothermally at 110°C for one hour under 160 mL/min air flow.

AUTHORS

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