A Biobased, Zero-VOC Coalescent for Architectural and Industrial Coatings

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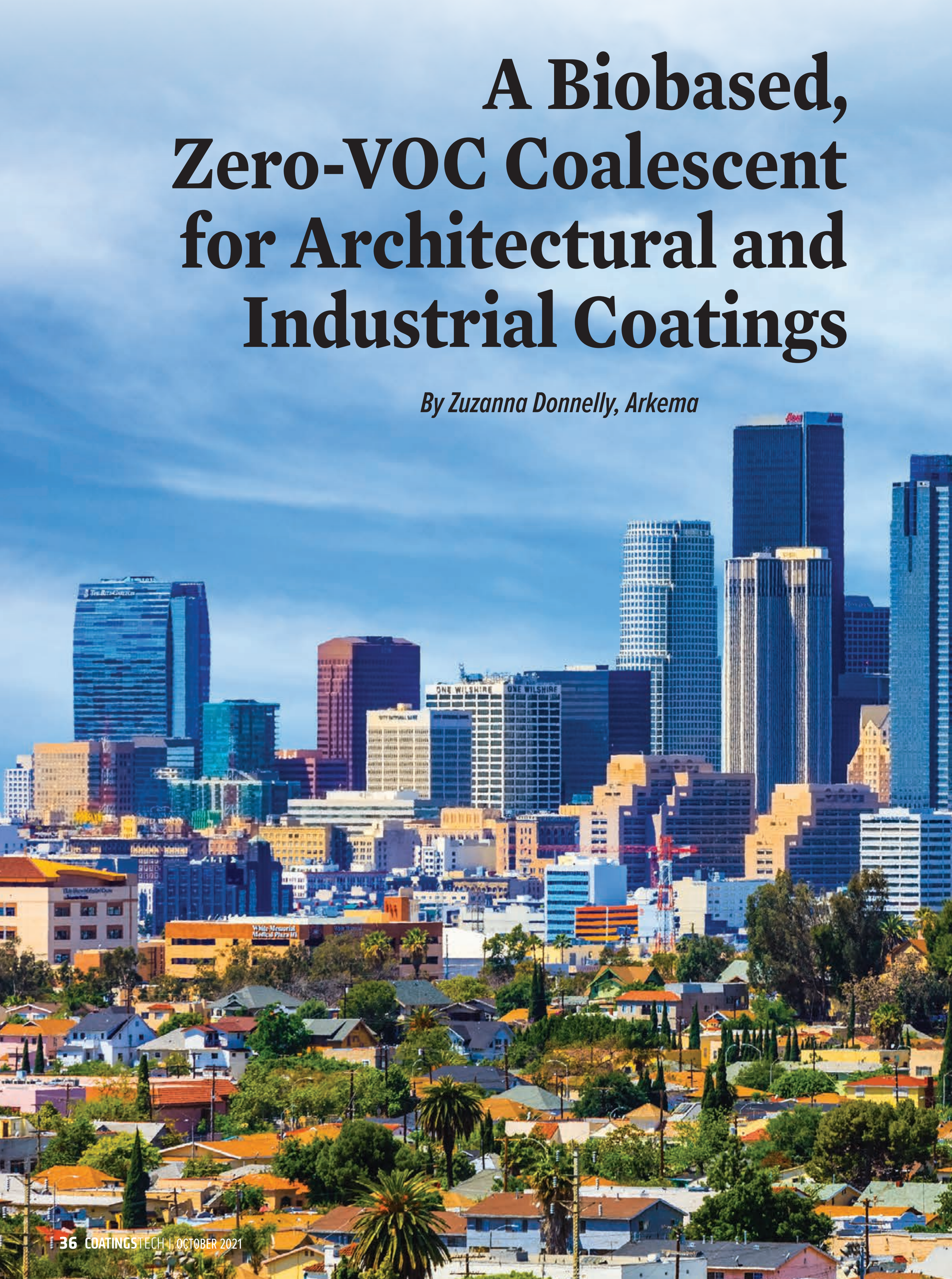


In the coatings industry, the concept of sustainably is becoming increasingly important, whether to meet regulatory requirements or growing customer and consumer expectations.[1,2] Sustainability is also a significant driver for coatings innovations.[3,4,5]

The United Nations has adopted a list of 17 "Sustainable Development Goals"[6] that seek to define a blueprint for sustainability on a global scale. Many of these relate to sustainability in coatings, such as reducing volatile organic compounds (VOCs) and greenhouse gases and replacing fossil fuel-based products with biosourced materials.[7,5]

With these UN Sustainable Development Goals in mind, a new coalescent technology for waterborne paints that meets the zero-VOC requirement as measured by ASTM D6886 and has a high biobased carbon content of 96% as measured by ASTM D6866 has been developed.

This goal of the study described in this article is to evaluate the performance of this product in several representative architectural and industrial waterborne paint formulations and benchmark performance against other VOC and zero-VOC coalescents available on the market.

Performance of this new coalescent was evaluated in a range of waterborne binder chemistries, including all acrylic, vinyl acrylic, styrene acrylic hybrid, and self-crosslinking acrylic systems in several architectural and industrial maintenance paint formulations.

This new coalescent technology was found to compare favorably with commercial zero-VOC coalescents in architectural coatings formulations. In industrial coatings formulations, substitution of VOC solvents, in whole or in part, with this new technology allows for significant lowering of overall formulation VOC while maintaining good coating performance. Improved metal adhesion and impact properties were observed with retention of high gloss and good corrosion resistance.

EXPERIMENTAL DETAILS

Test Methods

Viscosity and Heat Age Stability: The initial Stormer viscosity—measured with a Krebs Stormer viscometer according to ASTM D562 and reported as Krebs Unit (KU)—was measured at room temperature prior to placing the paint can into a 120 °F oven. The can was removed after 2 weeks and allowed to return to room temperature. The Stormer viscosity in KU was measured again.

Gloss measurements and yellowness index: Gloss and yellowness of paint films were measured using micro-TRI-gloss and color-guide 6805 (both meters from BYK-Gardner), respectively.

FORMULATIONS

Several representative architectural and industrial waterborne paint formulations were developed for this study. Their performance was evaluated and benchmarked against other VOC and zero- VOC coalescents available on the market.

Semigloss Architectural Paint Formulation

MATERIALS	POUNDS	GALLONS
Grind		
Water	185.0	22.2
Natrosol™ Plus 300	2.0	0.2
COADIS™ BR40 (40%)	6.5	0.7
CARBOWET® 109	2.2	0.3
Drewplus™ L-475	8.0	1.0
Ti-Pure™ R706	225.0	6.8
MINEX® 10	50.0	2.2
Ammonium Hydroxide (28%)	2.0	0.3
PROXEL® GXL	0.5	0.1
Letdown		
ENCOR® 309 vinyl acrylic binder	450.0	49.9
Water	53.0	6.4
Coalsecent	14.9	1.7
COAPUR™ 830W	11.0	1.3
Water	56.5	6.8
Drewplus™ L-475	2.5	0.3
Total	**1069.1**	**100.2**

Low-VOC DTM Industrial Paint Formulation

MATERIALS	POUNDS	GALLONS
Grind		
Water	61.0	7.3
TAMOL™ 681	9.1	1.0
TRITON™ HW-1000	2.0	0.2
DOWSIL™ 8590	1.0	0.1
Ammonia (28%)	2.0	0.3
Ti-Pure™ R706	213.7	6.4
Water	21.1	2.5
Letdown		
Acrylic binder	571.8	65.5
Water	50.9	6.1
Coalescent	7.7	1.7
Sodium Nitrite (15%)	9.2	1.0
Water	63.0	7.6
ACROSYL™ RM 8W	2.8	0.3
Total	**1007.6**	**100.0**

Flat Architectural Paint Formulation

MATERIALS	POUNDS	GALLONS
Grind		
Water	280.0	33.6
Natrosol™ 250 HBR	2.0	0.2
ECODIS™ P50 (40%)	12.6	1.4
FOAMSTAR® ST 2436	1.0	0.1
Ti-Pure™ R706	210.0	6.3
MINEX® 4	295.0	13.2
AMP-95TM	1.0	0.2
Proxel® GXL	3.0	0.3
Letdown		
ENCOR® 626 acrylic binder	250.0	29.1
Coalesecent	11.3	1.3
Rheotech™ 3800	6.0	0.7
Water	93.0	11.2
FOAMSTAR® ST 2436	1.0	0.1
COAPUR® 2020	21.0	2.4
Total	**1186.9**	**100.0**

Gloss DTM Industrial Paint Formulation

MATERIALS	POUNDS	GALLONS
Grind		
Water	40.0	4.8
Ammonium Benzoate	1.0	0.1
40% Sodium Nitrite	1.0	0.1
28% Ammonia	1.0	0.1
TAMOLTM 681	10.0	1.1
SURFYNOL® 104 DPM	1.5	0.2
BYK® 024	1.0	0.1
Ti-PureTM R706	200.0	6.0
Letdown		
Water	30.0	3.6
Styrene acrylic modified binder	590.7	68.1
Water	96.0	11.5
Coalescent	30.0	4.0
Premix the next 2		
TAFIGEL® PUR 41	2.0	0.2
Butyl Carbitol	0.8	0.1
Total	**1005.0**	**100.0**

Minimum Temperature Film Forming (MFFT) testing: MFFT testing was carried out on a Rhopoint instrument equipped with a variable temperature MFFT bar. A temperature range of -4.5 °C to 13 °C was used. Latex films were drawn down at 75 microns thickness and allowed to dry under flowing N$_2$ gas for 30 minutes. MFFT temperature readings were taken at the point where the latex film transitioned from clear cohesive film to white powder.

Tint strength: Five grams of Colortrend Phthalo Blue was weighed into a half-pint can containing 250 grams of test paint. After the colorant was added, the paint can was shaken on a Red Devil shaker for 3 to 5 minutes. Paint drawdowns using the tinted paint compositions were then prepared on Leneta B charts using a 3 mil bird bar. These were allowed to dry for 1 day in a controlled temperature and humidity chamber at 25 °C and 50% relative humidity. The Y% brightness value was measured on a colorimeter and the percent tint strength was calculated by the Kubelka-Munk (KM) formula.

Washability and Stain Removal: Paints were applied at a wet film thickness of 7 mils to black Leneta Scrub Charts and allowed to dry for a minimum of 3 days. CIELAB color values were measured prior to application of stains. Stains were allowed to set for 2 hours and excess stain was removed by rinsing under cold water. Samples were placed into a Garner Straight Line Washability and Wear Abrasion Machine and scrubbed for 50 cycles with the addition of 10 mL of Formula 409 solution to the premoistened sponges. Samples were rinsed with water and dried. CIELAB color values were measured on areas where the samples were scrubbed and delta E values are reported.

Mudcracking: Paint drawdowns were made using a Sag bar on 1B Leneta charts and allowed to dry for 24 hours. The greatest thickness that did not show cracking is reported.

Dry Adhesion: The test paints were applied to untreated aluminum, cold rolled steel and galvanized steel using a 4 mil Bird applicator. The paints were allowed to dry for 7 days before the lattice pattern described in ASTM D3359B was cut and the pressure sensitive tape applied. The removal of the coating from the substrate was rated using the classification charts described in the ASTM Standard.

Flash Rust: The cold rolled steel panel from the adhesion test was examined after drying overnight. The panels were rated on a scale where 10 = no rust and 0 = heavy rust.

Other standard test methods used: Scrub Resistance ASTM D2486-06, Test Method B; Wet Adhesion to alkyd paints ASTM D6900; Block Resistance ASTM D4946; Chemical Resistance, ASTM D1308, 1 Hour Contact; Impact Resistance, ASTM D2794, 1 Week, R612 CRS Panels; Humidity Resistance, ASTM D2247, R46 Panels; ASTM B117-09 Standard Practice for Operating Salt Spray (Fog) Apparatus - R46 Panels; Prohesion, ASTM G85 Annex, R46 Panels

RESULTS AND DISCUSSION

Architectural coatings

The first half of this study concerns benchmarking the performance of this new coalescent, labeled DP-2200,[8] in representative architectural coatings. Two commercial coalescents were chosen as controls in these experiments: the first is a commonly used petroleum-derived product, and the second is a biobased product.

These coalescents and their characteristics are summarized in *Table 1*. All three materials are considered zero-VOC[9], are insoluble in water at room temperature, and have similar molecular weights ranging between approximately 300 g/mol and 400 g/mol. MFFT depression in representative acrylic and vinyl acrylic latexes are also similar for the three coalescents, making them good benchmarks for each other in this study.

Hansen solubility parameters were used as a tool to predict polymer-solvent compatibility, pigment dispersion and substrate adhesion.[10] One difference between the new epoxy ester technology used in DP-2200 and the two commercial glycol esters in this study is the lower hydrogen bonding ($\delta_{\text{H bonding}}$) and polar ($\delta_{\text{polar}}$) solubility parameters, which indicate a lower level of inter- and intra-molecular permanent dipole and hydrogen bonding interactions. However, it is the dispersion ($\delta_{\text{dispersion}}$) solubility parameter which is most heavily weighted in the calculation of the solubility parameter distance,[10,11] and the closeness of this parameter in all three coalescents suggests they should have similar compatibility within a given resin system.

Three semigloss paints made with ENCOR® 309 vinyl acrylic latex (MFFT 12) were formulated using each of the coalescents as the sole coalescing solvent. These paints had a pigment volume concentration of 23% and volume solids of 36.3%. A coalescent loading of 6 wt % on polymer solids was chosen as this was found to be the lowest coalescent loading needed for DP-2200 to pass a low temperature coalescence (LTC) test at 40 °F. This coalescent loading was also sufficient to pass LTC for the paint formulated with Coalescent 1. However, the paint formulated with Coalescent 2 at 6 wt % did not coalesce at 40 °F, indicating that this coalescent has a lower coalescing efficiency than the other two coalescents in this formulation.

Stability of the paint formulations was assessed with heat aging for 2 weeks at

TABLE 1—Coalescents Used in the Architectural Paint Formulations

COALESCENT	CHEMISTRY	COLOR	BOILING POINT [°C]	HANSEN SOLUBLITY PARMATERS A [MPA1/2] ΔDISPERSION, ΔPOLAR, ΔH BONDING	MFFT IN ALL ACRYLIC RESINB [°C]	MFFT IN VINYL ACRYLIC RESINC [°C]
DP-2200	Proprietary epoxy esters	Colorless	333	16.1, 3.9, 3.7	-1.9	-0.8
Coalescent 1	Proprietary glycol di-ester	Colorless	374-381	16.2, 4.5, 7.1	-1.8	-0.2
Coalescent 2	C-19 Fatty acid mono propylene glycol ester	Yellow	284	16.6, 5.3, 8.7	-0.1	-0.1

[a] Taken from literature[9] values or calculated using the HSPiP software tool.
[b] At 8 wt % coalescent on polymer solids in neat latex. MFFT of binder without coalescent is 14 °C.
[c] At 8 wt % coalescent on polymer solids in neat latex. MFFT of binder without coalescent is 12 °C.

120 °F. All three paints showed only modest changes in Stormer viscosity ($\Delta KU \geq 6$) post aging, indicating good paint stability. Blocking is the term used to describe two freshly painted surfaces sticking together when pressed against each other. It can be a common problem, particularly with binder-rich semigloss paints and can be exacerbated by zero-VOC coalescents that will not evaporate from the film. The film of the semigloss paint formulated with DP-2200 coalescent and Coalescent 2 were found to have block resistance sufficient to resist film rupture (*Figure 1*), whereas the film of the paint formulated with Coalescent 1 had very poor blocking resistance.

Optical properties, including gloss and color of the films, were measured for each semigloss formulation. The graph in *Figure 2* shows that the 60-degree angle and 85-degree angle gloss for the DP-2200 coalescent-containing formulation was comparable to that of the other two formulations. Biobased materials can often add unwanted color to a formulation. Yellowness index measurements (*Figure 2*) indicate that DP-2200 coalescent does not appear to contribute color, as measured by yellowness, to the formulation.

The ability for the paint formulations to accept color and keep color dispersed was determined by measuring tint strength and observing rub-up (*Figure 3*). Tint strength indicates the intensity of color developed when a specific amount of colorant is added to a paint. Rub-up is used to determine whether flocculation is occurring. If the rub-up appears lighter, then TiO_2 flocculation is occurring. If the rub-up area appears darker, then colorant flocculation is occurring. Paint formulated with DP-2200 coalescent maintains tint strength and rub-up properties consistent with those of the other paint formulations.

Stain removal, or washability, is the ease with which a stain, once applied to a substrate, can be removed by using water and a surfactant coupled with a sponge to physically scrub the stain off. The less stain that remains, and the whiter the coating appears, the better the stain removal. Stain removal is measured using CIELAB L*a*b* delta E values. A lower number indicates more complete stain removal. In all three cases, more hydrophobic stains (e.g., ink, crayon, cosmetics) were more easily removed. The removal of both hydrophilic and hydrophobic stains was similar for all of the semigloss paints, regardless of the coalescing agent (*Figure 4*).

FIGURE 1—Block Resistance for Semigloss Architectural Paints (Scored 1–10; 10 = no adhesion, 3 or less = film rupture)

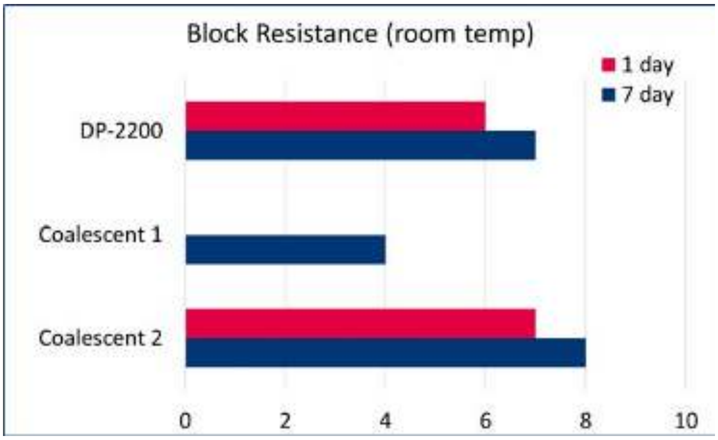


FIGURE 2—Gloss and Yellowness Index Measurements of Semiglоss Architectural Paints

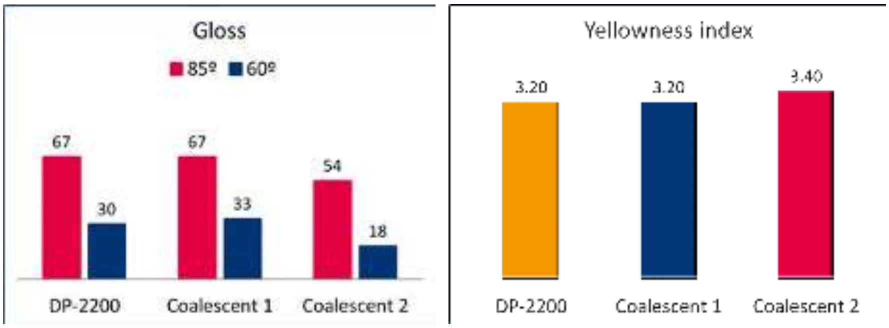


FIGURE 3—Tint Strength and Rub-Up Measurements of Semigloss Architectural Paints

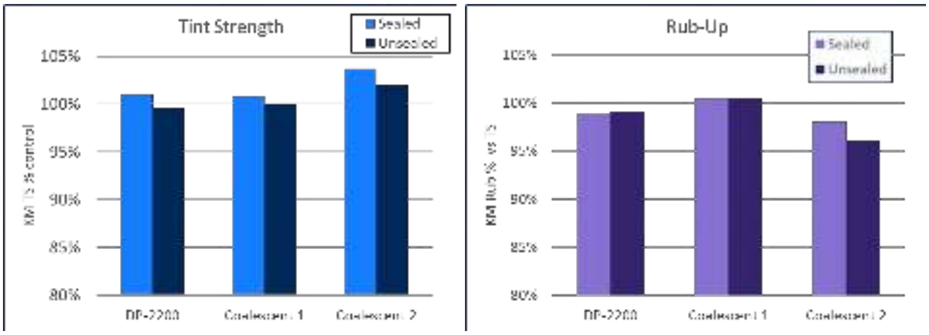
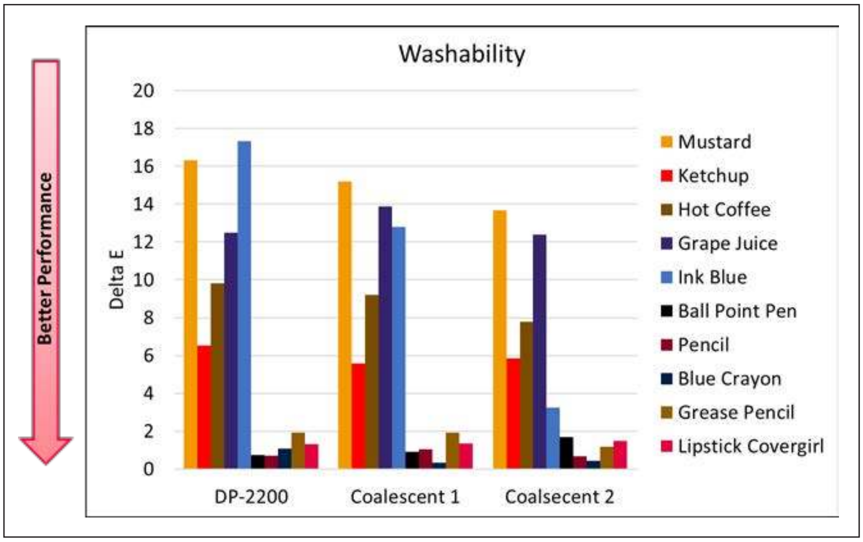


FIGURE 4—Washability Measurements of Semigloss Architectural Paints



The second part of the architectural paint performance evaluation made use of a flat paint made with ENCOR® 626 all-acrylic latex (MFFT 20). A low-VOC paint, at a pigment volume concentration of 58%, was coalesced at a concentration of 9% on latex solids, with volume solids of 33.5%. The same three coalescents were evaluated as before (*Table 1*).

All three paints passed LTC tests at this coalescent loading. Film-formation properties were also evaluated by measuring "mudcracking" of the film. Mudcracking is defined as a film failure that occurs when a thick paint application cracks upon drying the film, similar to a dried riverbed. For this test, a sag bar was used to do a flat drawdown of the paint, and then each film thickness was evaluated for cracking. As evidenced in *Table 2*, the flat paint coalesced with DP-2200 coalescent resisted mudcracking in a thicker film than the other two flat paints.

TABLE 2—Mudcracking Evaluation of Flat Architectural Paints

YOU MUDCRACKING (24 MILS MAX)	DP-2200	COALESCENT 1	COALESCENT 2
mils at failure	>24	16	18

As with the semigloss paint study, color acceptance was assessed for the flat paints. Tint strength comparisons and rub-up comparisons are shown in *Figure 5*. A flat paint formulated with DP-2200 coalescent maintains tint strength and rub-up properties similar to those of the other paint formulations. Dried film appearance was evaluated for color as before. DP-2200 coalescent does not contribute color, as measured by yellowness, to the formulation.

Durability of the paint film, as determined by wet adhesion and scrub resistance, was also assessed for the flat formulations. The paint coalesced with DP-2200 coalescent had good wet adhesion to a gloss alkyd substrate, and scrub resistance comparable to the other flat paints (*Table 3 and Figure 6*).

Finally, stain removal was again measured using CIELAB L*a*b* ΔE values. While stain removal for the flat coatings was not as dramatic as that on the semigloss coatings, the removal of both hydrophilic and hydrophobic stains was similar for all the paints, regardless of coalescing agent.

Industrial Coatings

To understand the performance of DP-2200 coalescent in industrial maintenance coatings, two waterborne metal coatings formulations were chosen. The first formulation was a direct-to-metal coating (DTM) based on a self-crosslinking acrylic emulsion (MFFT 14) designed for very low-VOC coatings.[12]

Two cosolvent blends were evaluated in this formulation: a blend of DP-2200 coalescent and dipropylene glycol butyl ether (DPnB) at a 40/60 ratio and a blend of Coalescent 1 and DPnB at a 40/60 ratio. A total coalescent loading of 3 wt % on polymer solids was used to make paints with a VOC level of less than 25 g/L. Pigment volume concentration was 15.6% and volume solids were 39.3%.

Both low-VOC DTM paints exhibited good stability with little to no changes in viscosity (ΔKU >4) upon heat aging for 2 weeks at 120 °F. Low-temperature film formation was evaluated on drawdowns that were allowed to dry at 40 °F for 24 hours. At the very low coalescent loading used in this formulation, the DP-2200/DPnB coalescent blend was found to be slightly more efficient at low temperature film formation, with a passing film observed on both sealed and unsealed portions of the Leneta charts. The Coalescent 1/DPnB coalescent blend passed on the sealed portion of the chart but displayed severe cracking and wrinkling on the unsealed panels at this temperature. Both paints had similar gloss values, excellent color acceptance and color. Both paints were also found to have good impact resistance, mandrel bend, and blocking resistance.

Adhesion to metal is a critical property for industrial paints, particularly adhesion to a variety of different metals and metal surface treatments. Adhesion was measured by crosshatch testing of films 1 week after application to cold rolled steel (CRS), untreated aluminum and galvanized metal substrates. Both paints had excellent adhesion to CRS and Al panels. The paint formulated with the DP-2200/DPnB coalescent blend had improved adhesion to the galvanized metal substrate (*Table 4*).

Several explanations are possible for this observed improved adhesion. The blend of DP-2200 and DPnB could provide the right combination of plasticization and solvent volatility to enable the film to better wet the rougher galvanized metal surface[13] during coalescence and/or maintain better adhesion upon drying.[14] Reactivity of the epoxy functionality in DP-2200 at the Zn surface is possible,[15] but is not likely considering the data in the final portion of the study described below.

The low-VOC DTM paints were also evaluated across a battery of corrosion testing, as summarized in *Table 5*. Both paints had excellent performance in flash rust and chemical-resistance testing against water, 5% NaOH and 5% HCl. In salt fog and prohesion testing, both cosolvent blends tested also displayed good performance.

The final formulation evaluated in this study was a gloss DTM formulation that was based on a styrene acrylic epoxy modified binder (MFFT 14).[16] This binder was used to evaluate DP-2200 coalescent performance in a different binder chemistry

FIGURE 5—Tint Strength and Rub-Up Measurements of Flat Architectural Paints

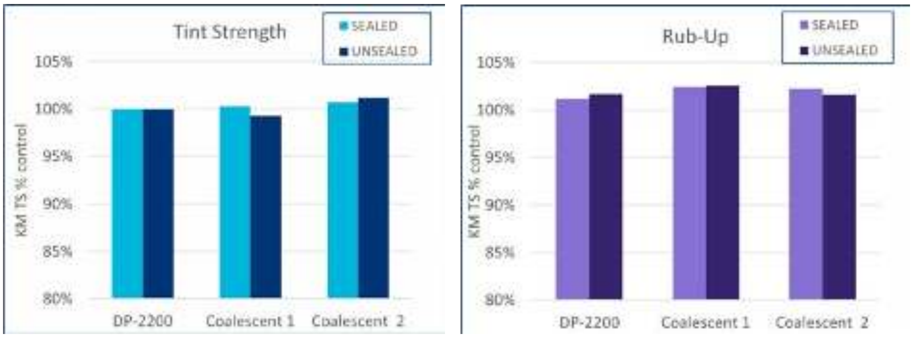


TABLE 3—Wet Adhesion and Scrub Resistance of Flat Architectural Paints

WET ADHESION 4 HR DRY	DP-2200	COALESCENT 1	COALESCENT 2
% Remaining at 500 cycles	100%	97%	30%

FIGURE 6—Wet Adhesion and Scrub Resistance of Flat Architectural Paints

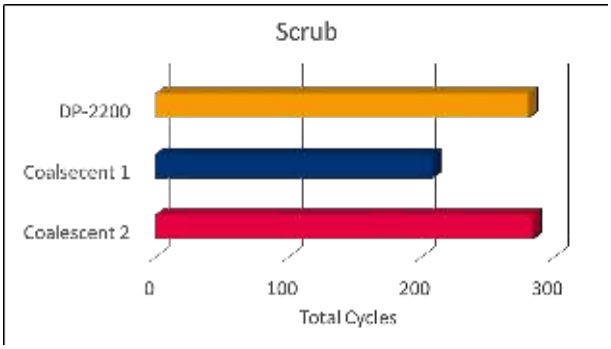


TABLE 4—Adhesion Testing of Low-VOC DTM Industrial Paints

ADHESION, 1 WEEK, (5B = NO FAILURE)	COALESCENT1 / DPNB	DP-2200 / DPNB
CRS	5B	5B
Untreated Aluminum	5B	5B
Galvanized Metal	0B	3B

TABLE 5—Corrosion Testing Results for Low-VOC DTM and Gloss DTM Industrial Paints. (Film thicknesses of 2 mils)

	LOW-VOC DTM GLOSS		GLOSS TOPCOAT		
	Glycol di-ester/DPnB	DP-2200/DPnB	DPnB	DP-2200/DPnB	DP-2200
Humidity Resistance					
ASTM D610 Rust (10 = No Rust)	6	8	10	10	10
ASTM D714 Blisters (10 = No Blisters)	9D	9D	10	10	10
Salt Fog, 500 hours					
ASTM D610 Rust (10 = No Rust)	10	10	6	8	6
ASTM D1654 Scribe Creep (10 = No Scribe Creep)	8	7	7	7	7
ASTM D714 Blisters (10 = No Blisters)	8M	8M	6D	6D	6D
Prohesion, 500 hours					
ASTM D610 Rust (10 = No Rust)	10	10	6	7	7
ASTM D1654 Scribe Creep (10 = No Scribe Creep)	7	7	7	7	7
ASTM D714 Blisters (10 = No Blisters)	8F	8F	6F	10	6F

TABLE 6—Coalescents Used in Gloss DTM Industrial Paint and the Calculated VOC Levels of Each Resulting Paint

COALESCENT	CALCULATED VOC
DP-2200	12 g/L
DP-2200/dipropylene glycol butyl ether (50/50)	53 g/L
Dipropylene glycol butyl ether	94 g/L

as well as to explore the range of possible VOC levels when using DP-2200 coalescent alone, or in a blend with a VOC cosolvent. Total coalescent loading was maintained at 10 wt % on polymer solids with a pigment volume concentration was 15.6% and volume solids of 39.3%.

The three coalescent combinations used in this study, as well as the resulting calculated paint VOC levels, are provided in *Table 6*. The substitution of DP-2200 for DPnB, in whole or in part, resulted in a substantial drop in the calculated VOC level of the paint.

All three paints demonstrated good gloss and adhesion to CRS and Al substrates, and good stability with little change in viscosity (ΔKU $\geq$6) upon heat aging for 2 weeks at 120° F. As before, improved adhesion to galvanized metal was observed in the paint containing the DP-2200/DPnB coalescent blend, as shown in *Table 7*. A similar improvement in adhesion was not realized with the paint containing DP-2200 coalescent alone, suggesting that some synergistic interaction[17] between the levels of DP-2200 and DPnB in these formulations could be present.

The presence of DP-2200 coalescent resulted in a marked increase in impact resistance of the paints, as shown in *Figure 7*. This is consistent with a softer and more flexible film being formed when DP-2200 is present. With the increased film flexibility, a drop in initial block resistance was also observed. However, the block resistance had largely recovered after 7 days and was high enough to prevent film rupture.

As with the low-VOC DTM formulation, all three paints again performed well in flash rust and chemical-resistance testing. The prohesion and salt fog results were slightly improved in the DP-2200/DPnB coalescent blend containing formulation as compared to the other two paints in this series (*Table 5*).

CONCLUSION

DP-2200 coalescent was evaluated in a series of representative architectural and industrial waterborne coatings formulations and benchmarked against several commercial zero-VOC and VOC cosolvents.

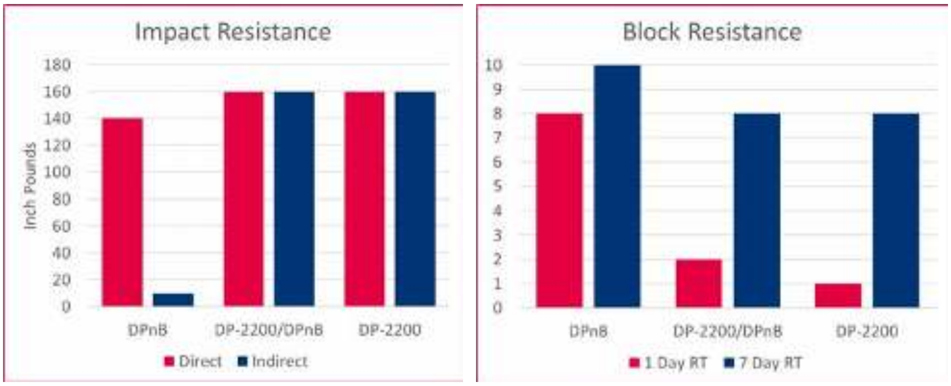
In architectural coatings, this new epoxy esters technology was found to perform well when compared to conventional glycol ester coalescents. Good coalescing efficiency and good paint performance was observed across all parameters evaluated, indicating that DP-2200 is a sustainable alternative to traditional coalescing solvents.

In industrial maintenance coatings, DP-2200 coalescent was found to be an effective coalescing agent, again comparing favorably with conventional glycol esters in different resins. DP-2200 displayed good gloss, adhesion, and corrosion-resistance properties, while providing wide formulation latitude for lowering paint VOC levels as well as increasing biobased content.

TABLE 7—Adhesion Testing of Gloss DTM Industrial Paints

ADHESION, 1 WEEK	DPNB	DP-2200/DPNB	DP-2200
CRS	5B	5B	5B
Untreated Aluminum	5B	5B	5B
Galvanized Metal	0B	3B	0B

FIGURE 7—Impact Resistance and Block Resistance (Scored 1-10; 10 = no adhesion, 3 or less = film rupture) of Gloss DTM Industrial Paints



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