Advantages of Siloxane in Low-VOC Architectural

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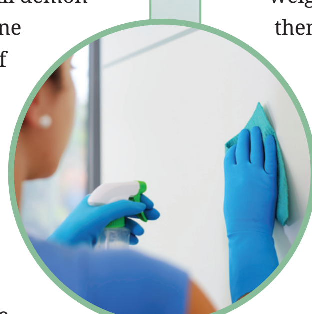
Siloxane-based additives are broadly used in the coatings industry to provide a variety of performance benefits. As the industry continues to move toward lower volatile organic content (VOC) or near-zero VOC, there is renewed and continued interest in siloxane-based additives due to the efficiency of its many chemistries.

Because siloxane-based additives exhibit diverse end properties in coatings, a range of siloxane-based surface control additives generated through structure modifications can be used as highly effective additives in coatings. Utilization of these siloxane surface control agents in coatings significantly lowers surface tension, improving wetting of substrates, while avoiding the environmental implications of other chemistries that are traditionally used in this space.

In this article, we will demonstrate the use of siloxane additives as a means of improving early block resistance. In the higher pigment volume concentration (PVC) systems, the use of siloxane additives will be shown to improve stain-resistance and scuff-resistance properties, as well as positively impact other difficult to achieve end-coating properties.



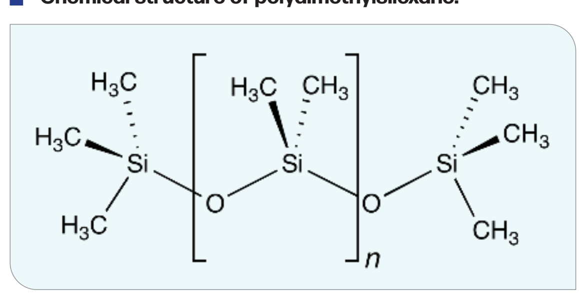
Introduction

Siloxane-based chemistry presents a route to surface control that can enable a broad range of coatings with varied properties. This range of coating attributes can be attained through chemical modification of siloxane-based molecules that can differ in general structure, molecular weight, size of the siloxane domains, and the relative amount of siloxane in the molecule; additionally, the nature of the organo-modification plays a dramatic role in the behavior of the resulting siloxane polymer.[1]

In **Figure 1**, the structure of an unmodified polydimethylsiloxane (PDMS) molecule is shown. To understand how a range of end attributes can be generated based upon a range of structural modifications to this "parent" polydimethylsiloxane molecule, it is important to recognize that the basic siloxane molecular structure, with silicon-oxygen backbone linkages and repeating units, can occur as fully methylated as shown in this figure. In this unmodified state, the siloxane molecule is quite flexible as well as both hydrophobic and oleophobic in nature, and its surface energy measures approximately 20 mN/m.[2-4]

Because polysiloxanes can be synthesized to have varied molecular weights with shorter or longer siloxane blocks, it is possible to tune them for a particular end use. Siloxane-based wetting agents typically have short siloxane backbones that are modified with hydrophilic pendant groups to increase their water solubility, while longer chained silicone polyether structures with pendant groups or modified end groups can be designed for applications such as defoamers or deaerators. In each scenario, the organic

FIGURE 1
Chemical structure of polydimethylsiloxane.



Surface Additives

Coatings

In the higher pigment volume concentration (PVC) systems, the use of siloxane additives will be shown to improve stain-resistance and scuff-resistance properties, as well as positively impact other difficult to achieve end-coating properties.

modifications can impact the hydrophilic versus hydrophobic nature of the molecule. For instance, the presence of ethoxylated pendant or end groups will increase the molecule's hydrophilicity while a high degree of propoxylation typically results in a more hydrophobic siloxane.[1]

Polyether-modified siloxanes with larger siloxane blocks tend to be more hydrophobic and oleophobic, and this increases their surface activity in waterborne coatings; therefore, both 100% active liquid polyether-modified siloxanes and emulsions of higher molecular weight and crosslinked polyether-modified siloxanes have been used to modify the surface activity of waterborne coatings. Historically, these types of molecules have been used to improve surface slip, flow, and leveling,[5] but, more recently, siloxane surface control agents have also been shown to significantly improve scratch and block resistance of wood coatings.[1]

These findings prompted us to further explore the use of polyether-modified siloxanes in architectural coatings where we have now discovered that early block resistance, as well as stain and scuff resistance, can be improved by using emulsions of polyether-modified siloxanes and 100% active polyether-modified siloxanes. In some cases, use of polyether-modified siloxanes intended for extreme hydrophobing[6] effect have been found to provide particular benefit.

Results and Discussion

Experimental

The siloxane-based surface control additives (SCAs) used in this study were commercial products sold by Evonik Corporation under the TEGO® Glide and TEGO® Phobe brand names, and they were used as received. General characteristics of the SCAs are shown in **Table 1**.

Commercial architectural paints were purchased from local retail stores and were

TABLE 1
Characteristics of Siloxane-Based Surface Control Additives

	Activity	Siloxane Structure	Relative Molecular Weight of Siloxane	Relative Size of PDMS Segments	Relative Degree of Organo-Modification
SCA#1	65% (emulsion)	Smaller Particle Size Crosslinked PDMS-1	Very High	Very High	Low
SCA#2	65% (emulsion)	Larger Particle Size Crosslinked PDMS-1	Very High	Very High	Low
SCA#3	65% (emulsion)	Smaller Particle Size Crosslinked PDMS-2	Very High	Very High	Low
SCA#4	100%	Comb	High	High	High
SCA#5	100%	Linear	Medium	High	High
SCA#6	100%	Linear	Low	Medium	Medium
SCA#7	100%	Linear	Low	Medium	Medium
SCA#8	55% (emulsion)	Very Hydrophobic Amino-Functional Siloxane	High	Very High	Low
SCA#9	55% (emulsion)	Very Hydrophobic Modified Siloxane Resin	High	Very High	Very Low

TABLE 2
Waterborne Vinyl-Acrylic High PVC (62%), Low-VOC Interior Architectural Paint

Raw Material	Supplier	Function	Mass (g)
Water		Solvent	27.94
Ammonia		pH Modifier	0.16
Natrosol™ Plus 330	Ashland	Thickener	0.40
TEGO® Dispers 715 W	Evonik	Dispersant	0.44
CARBOWET® 109	Evonik	Surfactant	0.18
AIRASE® 4500	Evonik	Defoamer	0.66
Ti-PureTM R-706	Chemours	Pigment	16.42
DrikaliteTM $CaCO_3$	Imerys	Pigment	14.36
MINEX® 4	Covia	Pigment	14.36
Mix for 20 minutes. Grind to Hegman 4.			
ENCOR® 309	Arkema	Vinyl-Acrylic Latex Emulsion	18.07
Water		Solvent	2.85
TEGO® ViscoPlus 3000	Evonik	Thickener	2.17
Optifilm™ Enhancer 400	Eastman	Coalescent	0.80
Propylene Glycol		Coalescent	0.82
Rhodoline® FT 100	Solvay	Freeze/Thaw	0.37
TOTAL			**100.00**

TABLE 3
Waterborne Vinyl Acetate-Ethylene (VAE) 52% PVC, Low-VOC Interior Paint

Raw Material	Supplier	Function	Mass (g)
Water		Solvent	17.57
AMP-95™	Angus	pH modifier	0.13
TEGO® Dispers 715 W	Evonik	Dispersant	0.58
CARBOWET® 109	Evonik	Surfactant	0.18
TEGO® Foamex 9	Evonik	Defoamer	0.18
Ti-Pure™ R-706	Chemours	Pigment	17.51
OPTIWHITE™ $CaCO_3$	Burgess	Pigment	8.75
MINEX® 4	Covia	Pigment	11.38
ATTAGEL® 50	BASF	Clay thickener	0.44
Mix for 20 minutes. Grind to Hegman 4.			
VINNAPAS® EF 8001	Wacker	Vinyl Acetate-Ethylene (VAE) Copolymer Dispersion	24.51
TEGO® ViscoPlus 3010	Evonik	Thickener	0.29
TEGO® ViscoPlus 3030	Evonik	Thickener	0.93
TEGO® Foamex 9	Evonik	Defoamer	0.26
Water		Solvent	17.29
TOTAL			**100.00**

The cleaned scuffed areas were assessed comparatively versus uncleaned scuff marks using visual inspection as well as colorimeter measurements.

thoroughly mixed on a Red Devil Paint Shaker and allowed to rest overnight before use. Two different high-PVC waterborne interior architectural coating formulations were prepared using the formulations presented in **Tables 2** and **3**. In all cases, the surface control additives were post-added to the paint formulation at the stated use level, and the resulting paint was mixed well at 800 rpm for 15 minutes using an IKA Eurostar 60 overhead mixer. All paints were allowed to stand overnight before application and testing.

For block-resistance testing, all coatings were applied to sealed black and white Leneta 5C opacity charts in a controlled temperature and humidity (CTH) room held at 25 °C and 50% relative humidity using a 6-mil applicator bar. Block resistance was tested according to ASTM Method D4946-89 (Reapproved 2017) with varied durations of cure: 4 hours, 24 hours (1 day), and 7 days under both oven block testing (at 50 °C) and room temperature (RT) block testing conditions. Ratings were assigned according to the ASTM Methods D4946-89 with the highest rating of 10 assigned to coatings with no tack and perfect performance; lower ratings were given depending upon the difficulty of separation of the charts, reduction in tack and seal, as well as performance. The lowest rating of 0 reflects 75-100% loss of seal and very poor performance.

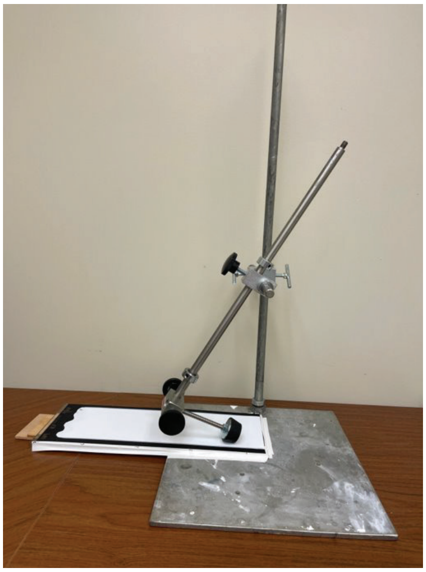
Appearance of the initial coating was rated visually on a scale from 0 to 10, with 10 being a perfect, defect-free film. Initially, a single coating layer was applied to a sealed black/white Leneta chart drawn down using a 7/10 Dow bar in a CTH room. Charts were cured in the CTH room for 7 days and then a second coating layer was applied. The dried films were then assessed for appearance attributes.

Stain-resistance testing was performed by applying each coating to a black plastic scrub test panel (Leneta Form P121-10N) in a CTH room and the coatings were cured for 7 days. After 7 days, a range of household stains were applied in a horizontal line across the scrub panel with approximately 1-inch distance between each stain application. Stains included the following hydrophobic and hydrophilic types of household stains: washable marker, crayon, ballpoint pen, red lipstick, ketchup, mustard, red wine vinegar, and brewed coffee.

Stains were applied and allowed to sit on the paint surface for 5 minutes, after which a paper towel was used to absorb the wet stains. A damp sponge saturated with 5 mL of Fantastik® surface cleaner was then rubbed with consistent pressure back and forth 30 times down the center of the panel, and the panel was finally rinsed with tap water and gently patted dry. Each stain was then assessed for stain appearance where 0 indicates complete stain removal; 1 signifies partial stain removal, and 2 represents zero stain removal. An overall score (in total) was then calculated, with a lower value reflecting better stain resistance of the coating.

Scuff-resistance testing was performed by applying each paint to a black plastic scrub test panel (Leneta Form P121-10N) in a CTH room and coatings were cured for 7 days. After 7 days, scuff marks were applied via use of the apparatus shown in **Figure 2**. The scuff marks were applied by impinging a black chemical stopper on the surface. The pendulum was dropped at a height of four inches from the panel surface.

FIGURE 2
Scuff-resistance testing apparatus.



Typically, four to eight marks were applied across the panel. The scuff mark from the black chemical stopper was intended to mimic a scuff mark from the sole of a shoe on a wall.

After application of the scuff marks, any surface residue was gently brushed from the surface using a Kimwipe™, and then each panel was cleaned by hand rubbing over the scuff mark in a circular fashion for 20 seconds using a sponge soaked with Fantastik® household cleaner. The cleaned scuffed areas were assessed comparatively versus uncleaned scuff marks using visual inspection as well as colorimeter measurements. Visual assessment ratings were as follows: a score of 0 for full removal of the mark, indicating it visually not present; scores of 1 to 2 for partial removal and a score of 3 for no change with cleaning. Colorimeter measurements were performed using a BYK Gardner Spectro-Guide measuring L*a*b* color space and ΔE (Delta E).

FIGURE 3
Block-resistance test results for commercial 100% acrylic latex exterior semigloss paint (Comm A) containing siloxane-based surface control additives.

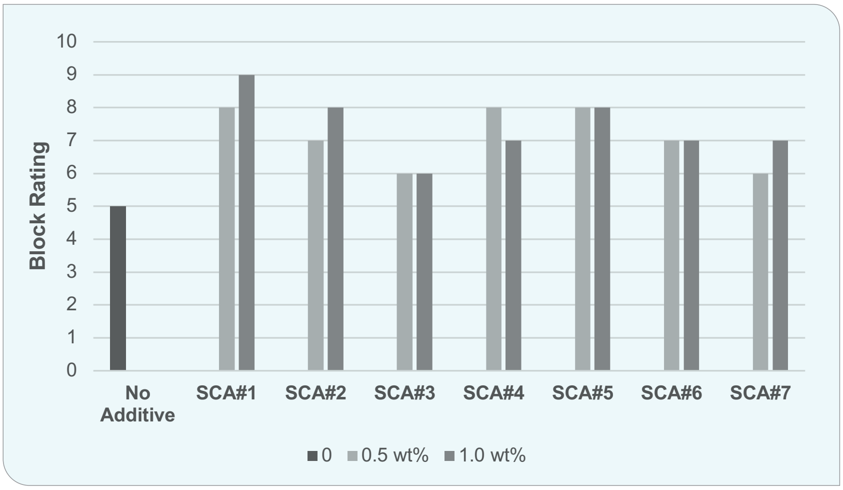
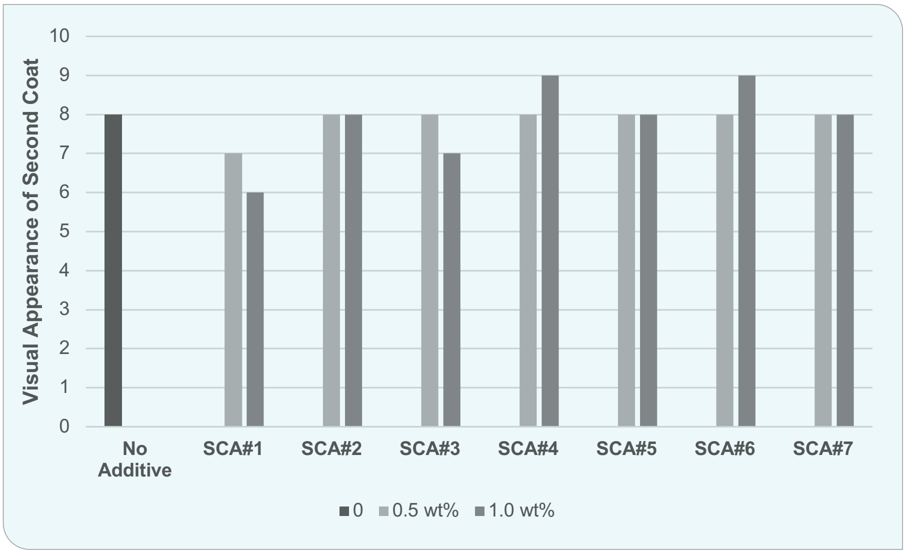


FIGURE 4
Visual appearance of second coats of commercial 100% acrylic latex exterior semigloss paint (Comm A) containing siloxane-based surface control additives.



Improvement of Early Block Resistance Using Siloxane-Based Surface Control Additives

In an initial screening study, a commercial 100% acrylic latex exterior semigloss ultra-pure white base paint (Comm A) was evaluated using post-addition of several of the siloxane-based SCAs listed in **Table 1** at 0.5 and 1.0 wt %. Each coating was assessed for appearance, block resistance under standard block conditions (ASTM D4946-89; 7 days) and recoatability. As can be seen in **Figure 3,** the block resistance of the paint improves with addition of 0.5 wt % of any of these SCAs, and the higher 1.0 wt % use level of SCA#1, SCA#2, and SCA#7 further improves block resistance.

However, when using siloxane-based additives, one also needs to consider the potential of these additives to cause craters and recoatability issues due to incompatibility of the additive in the coating throughout the drying process. Therefore, each of the paints evaluated in **Figure 3** were also evaluated for recoatability. As shown in **Figure 4**, while SCA#1 did the best at improving block resistance, it had a deleterious effect on the coating's recoatability; similarly, at the higher use

FIGURE 5
Results of ambient block testing of tinted commercial interior semigloss (Comm B).

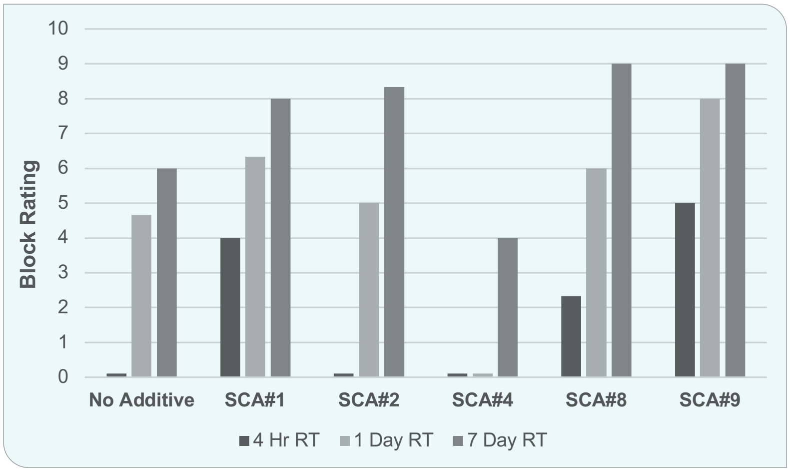


FIGURE 6
Results of oven block testing of tinted commercial interior semigloss (Comm B).

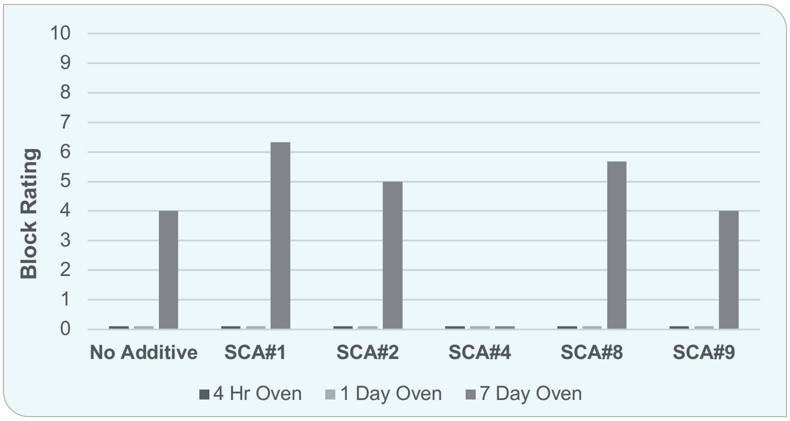


FIGURE 7
Results of ambient block testing of tinted commercial interior semigloss (Comm C).

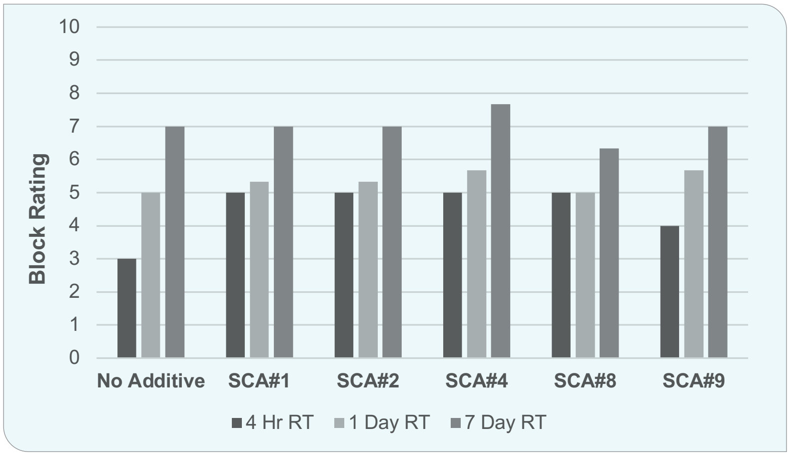
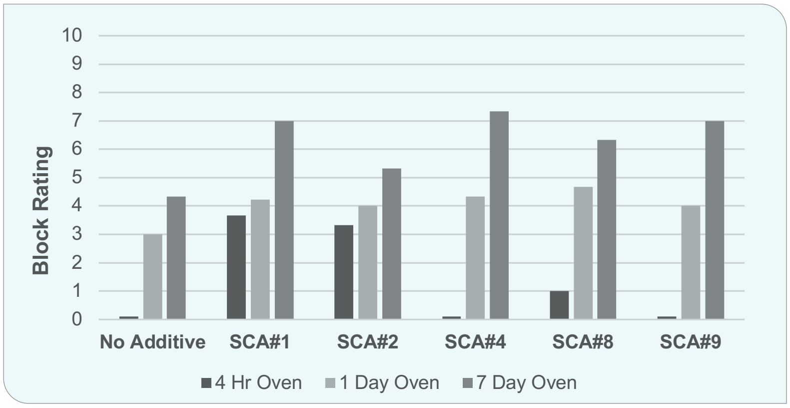


FIGURE 8
Results of oven block testing of tinted commercial interior semigloss (Comm C).



level, SCA#3 also began to cause craters and negatively impacted recoatability. For the 100% acrylic latex exterior semigloss Comm A, a 0.5 wt% use level of either of the two 100% active organo-modified siloxanes SCA#4 or SCA#5 provided the most efficient improvement in block resistance with the least impact on the paint's ability to be recoated.

Based on these first promising results, additional studies of the effects of siloxane-based SCAs on improving block resistance were undertaken, and two additional very hydrophobic siloxane emulsion SCAs were included in the evaluations. For these studies, four commercial low-VOC acrylic interior semigloss paints were evaluated both as tinted paints and as the untinted white base paints. Because the block testing resistance of the white base paints exhibited comparable to very slightly better block resistance to that of the corresponding tinted paints, only the results for the tinted paints are discussed here. Siloxane-based SCA use levels of 1.0 wt % were employed in these initial studies. Results for the block-resistance testing of the four tinted paints (Comm B, Comm C, Comm D, and Comm E) are shown in **Figures 5** through **12**.

Commercial semigloss paint Comm B displays poor ambient-temperature-cure block resistance, particularly within the first 24 hours **(Figure 5)**, and its hot block resistance is extremely poor **(Figure 6)**. However, the four siloxane-emulsion SCAs (#1, #2, #8, and #9) can improve the paint's ambient-temperature-cure block resistance to some extent, and SCA#9–a silicone-resin emulsion originally developed as a hydrophobizing agent–does particularly well in this regard. Interestingly, the 100% active organo-modified siloxane SCA#4 worsens the block resistance of this paint under 1-day and 7-day ambient block conditions, and early hot block resistance is not able to be positively impacted by any of these SCAs.

Commercial semigloss paint Comm C displays somewhat better block resistance than Comm B, particularly within the first 4 hours at room temperature **(Figure 7),** and its hot block resistance within the first 24 hours is fair **(Figure 8)**. All five SCAs studied can improve the paint's 4-hour ambient-cure block resistance to some extent, and the crosslinked PDMS-1 emulsions, SCA#1 and SCA#2, significantly improve the challenging 4-hour hot block resistance. Interestingly, the 100% active organo-modified siloxane SCA#4 does an excellent job of improving the ambient-cure block resistance of this paint, but neither SCA#4 nor SCA#9 can improve the 4-hour hot-block resistance of this paint.

Commercial semigloss paint Comm D has far better overall block resistance than Comm B or Comm C, particularly within the first 4 hours at room temperature (RT), as shown in **Figure 9,** and its hot-block resistance within the first 4 hours is good **(Figure 10)**. However, the crosslinked PDMS-1 emulsion SCA#1 and the extremely hydrophobic SCA#9 can significantly improve the 4-hour hot-block resistance of this paint.

Commercial semigloss paint Comm E has the best block-resistance behavior of the four commercial paints tested, particularly within the first 4 hours **(Figure 11).** Its hot-block resistance within the first 4 hours is amongst the best available on the market **(Figure 12)**. However, all five of the siloxane-based SCAs tested boost the room-temperature and hot-block resistance of this paint, with SCA#1 and SCA#9 providing the best overall results at the 1.0 wt % use level in this paint.

A summary of the results of ambient and 50 °C block testing for the four tinted commercial interior semigloss paints (Comm B, Comm C, Comm D, and Comm E) is shown in **Table 4.** Blocking ratings between 0 and 2 were assigned as “poor” (red), 3 to 5 as “fair” (yellow), 6 to 7 as “better” (light green), and 8 through 10 as “best” (dark green).

Improvement of Stain and Scuff Resistance Using Siloxane-Based Surface Control Additives

Low-VOC architectural coatings also tend to exhibit poorer stain-resistance and scuff-resistance properties due to the lower glass transition temperature (T_g) binders based on softer polymers that are often required in these systems. In order to understand the impact of siloxane-based SCAs on flat architectural paints, a series of siloxane-based SCAs were evaluated as post-additions at 0.5, 1.0, and 2.0 wt % use levels in the two architectural paint formulations shown in **Table 2** (62% PVC vinyl-acrylic) and **Table 3** (52% PVC VAE).

FIGURE 9
Results of ambient block testing of tinted commercial interior semigloss (Comm D).

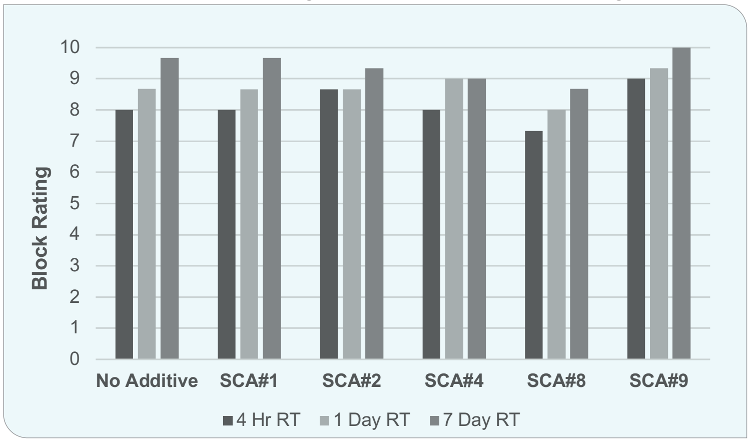


FIGURE 10
Results of oven-block testing of tinted commercial interior semigloss (Comm D).

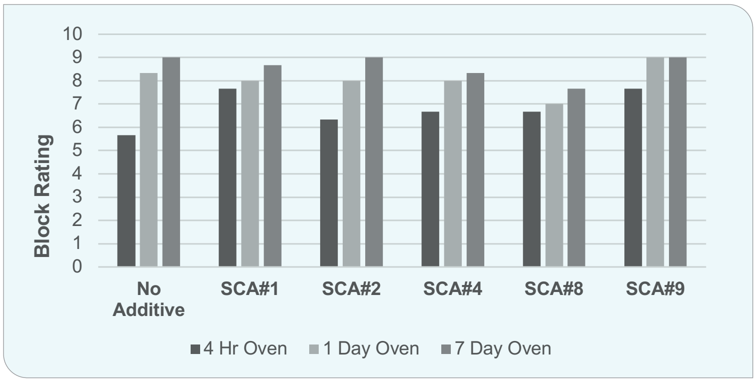


FIGURE 11
Results of ambient block testing of tinted commercial interior semigloss (Comm E).

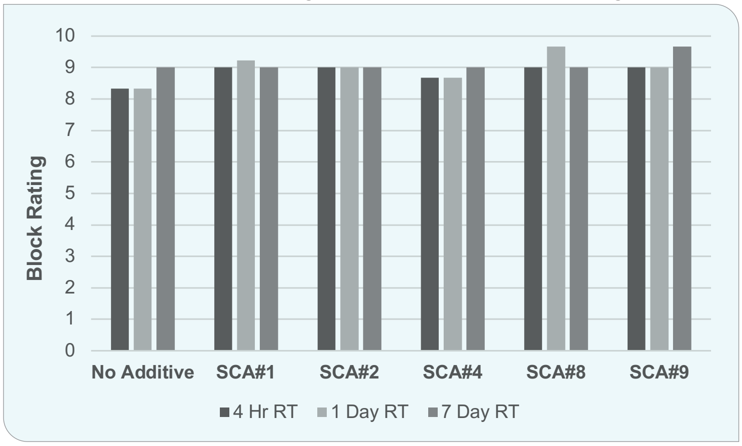
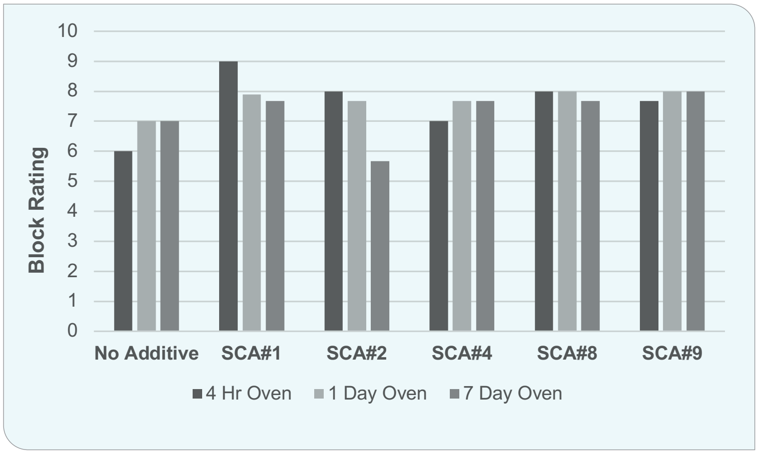


FIGURE 12
Results of oven-block testing of tinted commercial interior semigloss (Comm E).



Figures 13 and **14** show the stain-resistance performance for the interior architectural paint formulations shown in **Tables 2** and **3** containing SCAs at 0.5 wt % use level. In all cases for the vinyl-acrylic paint in **Table 2,** an improvement—indicated by a lower number—was observed for stain-resistance performance due to significant improvements in washable marker and crayon removal. Ladder studies were also performed with this formulation using 0.5, 1.0, and 1.5 wt% of the siloxane-based SCAs. Further improvements in stain resistance were seen at the higher use levels; however, compatibility in the system should be considered at higher levels.

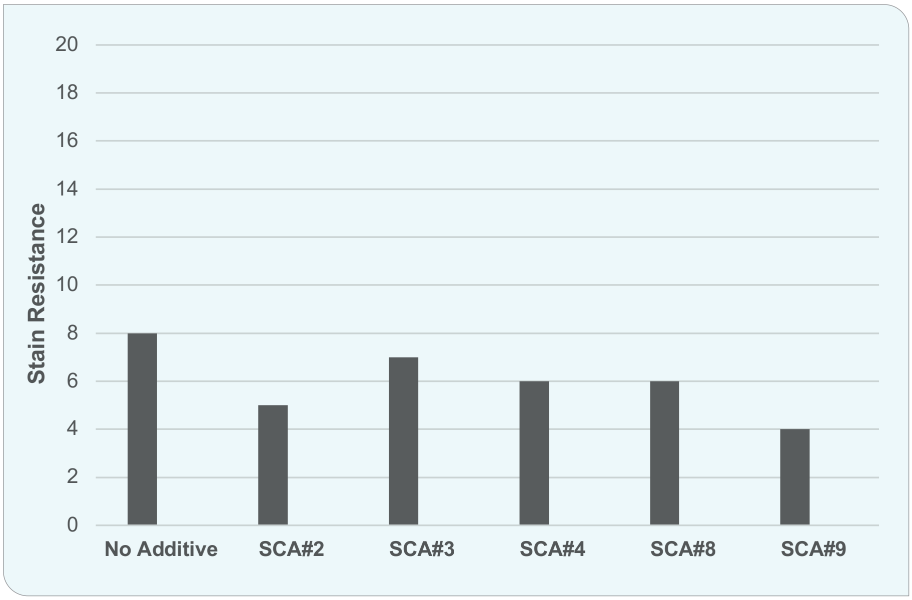
In the case of the VAE flat paint in **Table 3,** which is based upon a lower T_g VAE resin system, there were stain-resistance improvements seen with some, but not all, of the SCA additives **(Figure 14)**. This is likely because the softness of the resin dominates the behavior of this low T_g paint.

TABLE 4
Summary of Ambient and 50 °C Block Testing Results for Four Tinted Commercial Interior Semigloss Paints

Commercial Semigloss Paint	Block Testing Conditions	No Additive	+ 1.0 wt% SCA#1	+ 1.0 wt% SCA#2	+ 1.0 wt% SCA#4	+ 1.0 wt% SCA#8	+ 1.0 wt% SCA#9
Comm B	4 Hr RT	poor	fair	poor	poor	poor	fair
	1 Day RT	fair	better	fair	poor	better	best
	7 Day RT	better	best	best	fair	best	best
	4 Hr 50 °C	poor	poor	poor	poor	poor	poor
	1 Day 50 °C	poor	poor	poor	poor	poor	poor
	7 Day 50 °C	fair	better	fair	poor	better	fair
Comm C	4 Hr RT	fair	fair	fair	fair	fair	fair
	1 Day RT	fair	fair	fair	better	fair	better
	7 Day RT	better	better	better	best	better	better
	4 Hr 50 °C	poor	fair	fair	poor	fair	poor
	1 Day 50 °C	fair	fair	fair	fair	fair	fair
	7 Day 50 °C	fair	better	fair	better	better	better
Comm D	4 Hr RT	best	best	best	best	better	best
	1 Day RT	best	best	best	best	best	best
	7 Day RT	best	best	best	best	best	best
	4 Hr 50 °C	fair	best	better	better	better	best
	1 Day 50 °C	best	best	best	best	better	best
	7 Day 50 °C	best	best	best	best	best	best
Comm E	4 Hr RT	best	best	best	best	best	best
	1 Day RT	best	best	best	best	best	best
	7 Day RT	best	best	best	best	best	best
	4 Hr 50 °C	better	best	best	better	best	best
	1 Day 50 °C	better	best	best	best	best	best
	7 Day 50 °C	better	best	better	best	best	best

(RT is room temperature.)

FIGURE 13
Stain resistance of 62% PVC vinyl-acrylic flat paints containing 0.5 wt % SCA.



In the case of the VAE flat paint in Table 3, which is based upon a lower T_g VAE resin system, there were stain-resistance improvements seen with some, but not all, of the SCA additives (Figure 14).

FIGURE 14
Stain resistance for 52% PVC VAE flat paints containing 0.5 wt % SCA.

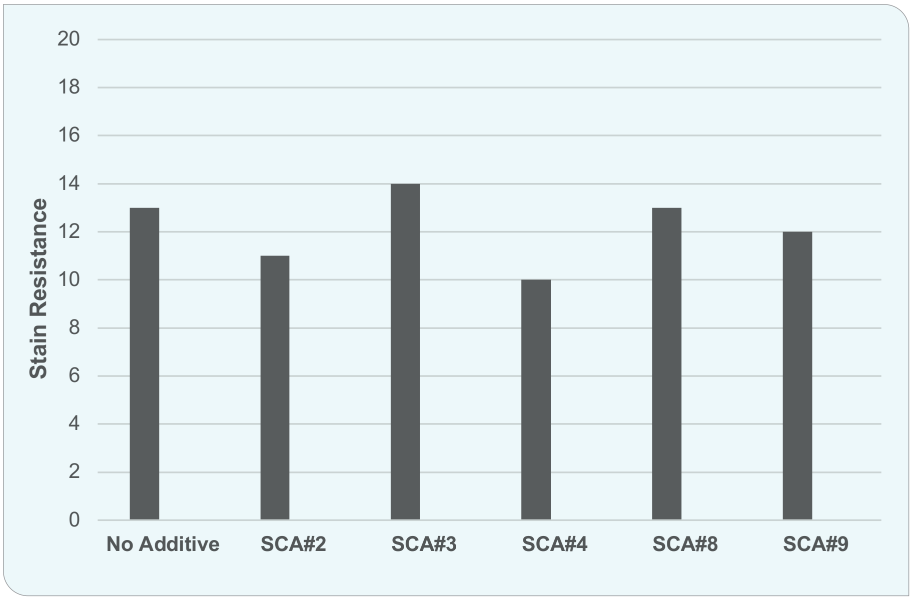


FIGURE 15
Visual assessment of scuff resistance for 62% PVC vinyl-acrylic flat paint (0 = best).

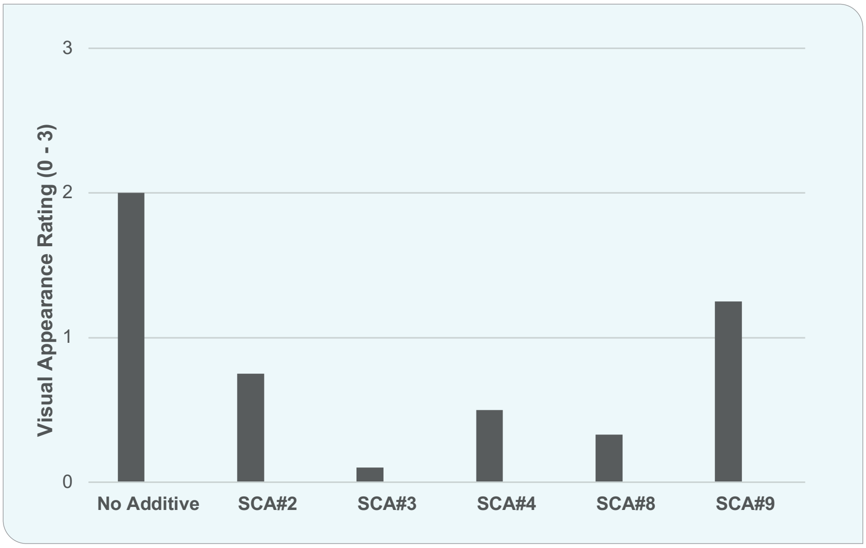


FIGURE 16
Scuff resistance by colorimeter (Delta E) for 62% PVC vinyl-acrylic flat paint.

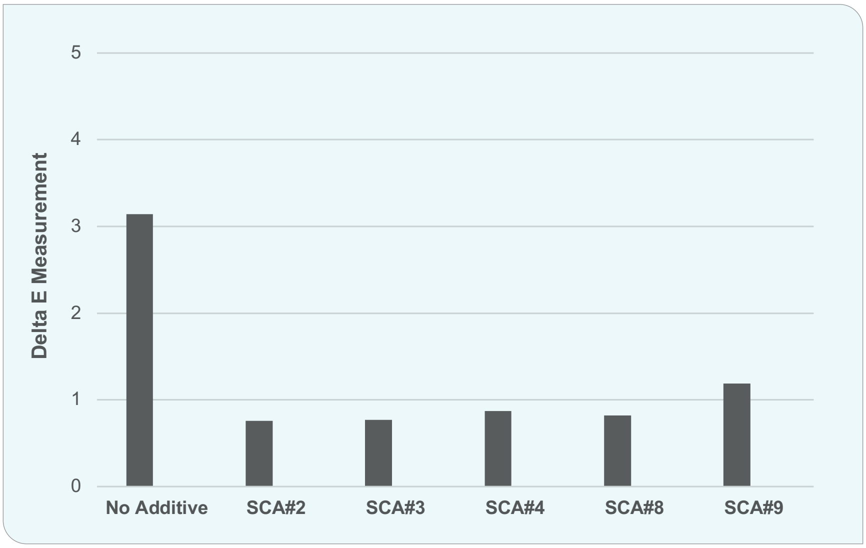
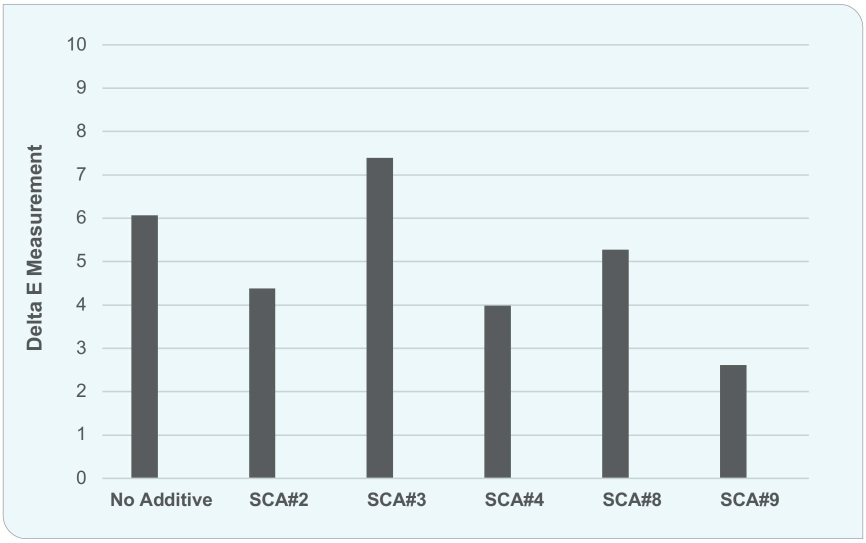


FIGURE 17
Scuff resistance by colorimeter (Delta E) for 52% PVC VAE flat paint.



Scuff-resistance testing of the same two flat paint formulations was also performed. **Figures 15** and **16** show the scuff-resistance performance for the 62% PVC vinyl-acrylic flat paint **(Table 2)**, and **Figure 17** shows the results for the 52% PVC VAE-based flat paint **(Table 3)**; the same SCAs utilized in the stain-resistance studies above were evaluated for their impact on scuff resistance. In the case of the vinyl-acrylic flat paint, the coatings containing SCAs showed improvements in scuff-resistance properties both by visual inspection **(Figure 15)** and by colorimeter measurements **(Figure 16)**. All the emulsion-based SCAs performed quite well in this system with very low ratings, indicating good scuff-resistance performance. **Figure 17** shows the performance of the SCAs in the 52% PVC VAE-flat paint. In this case, improvements in scuff resistance were observed with all SCAs investigated except for SCA#3 (the crosslinked PDMS-2 emulsion).

A summary of the results of stain- and scuff-resistance testing in the two low-VOC interior flat paints is shown in **Table 5**. Stain-resistance ratings of 16 to 20 were assigned as "poor" (red), 11 to 15 as "fair" (yellow), 6 to 10 as "better" (light green), and 0 to 5 as "best" (dark green). Scuff resistance ΔE values greater than 5 were assigned as "poor" (red), 3 to 5 as "fair" (yellow), 1 to 3 as "better" (light green), and less than 1 as "best" (dark green).

Conclusions

Siloxane-based SCAs were evaluated in several semigloss and flat architectural coatings to better understand the SCAs' impact on low-VOC paints, specifically with regards to block, stain- and scuff-resistance performance. These types of properties can be severely impacted in low-VOC paints due to the tendency of these types of formulations to use lower T_g binders to circumvent the need for VOC-contributing coalescing agents and solvents. While fluorosurfactants have been used in the past to address these challenges, these additives are facing increasing environmental pressure. Therefore, additional formulating tools like siloxane-based SCAs would be advantageous if they could help achieve the needed properties.

This work has shown that block resistance in low VOC semigloss interior and exterior architectural paints can be significantly and positively impacted by adding siloxane-based SCAs at relatively low levels

TABLE 5
Summary of Stain- and Scuff-Resistance Testing in Two Low-VOC Interior Flat Paints

Interior Flat Paint	Block Testing Conditions	No Additive	+ 0.5 wt% SCA#2	+ 0.5 wt% SCA#3	+ 0.5 wt% SCA#4	+ 0.5 wt% SCA#8	+ 0.5 wt% SCA#9
62% PVC Vinyl-Acrylic	Stain Resistance	better	best	better	better	better	best
	Scuff Resistance	fair	best	best	best	best	better
52% PVC VAE	Stain Resistance	fair	fair	fair	better	fair	fair
	Scuff Resistance	poor	fair	poor	fair	poor	better

(0.5-1.0 wt %). Difficult-to-achieve early (4-hr) block resistance can be improved by up to 4 units when adding an effective siloxane SCA, and improvements in both hot and ambient block resistance at longer intervals of testing are also observed. In addition to improved block resistance, addition of siloxane-based SCAs to higher PVC flat architectural paints has been shown to improve stain- and scuff-resistance properties of the paints.

Siloxane-based surface control additives can provide a route to difficult-to-achieve final architectural paint properties such as early block resistance, as well as stain and scuff resistance. This preliminary work has demonstrated that several different siloxane chemistries, particularly siloxane emulsions, show promise in this regard, and additional evaluations and optimization studies are planned. This research has also highlighted two new hydrophobic siloxane-emulsion surface control additives, SCA#8 and SCA#9, which have been shown to be useful tools for formulating architectural paints. Further exploration to better understand exactly how these siloxane SCAs cause these improved properties are certainly warranted.

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