A STUDY OF THE EFFECTS OF DIRT PICK-UP RESISTANCE IN LOW-VOC

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As the coatings industry moves towards lower volatile organic content (VOC) or near-zero VOC, achieving good film formation in waterborne systems without sacrificing other coating properties has become challenging. Low-VOC coatings are softer and tackier due to the use of low T_g resin, which makes them more susceptible to capturing dirt and dust, especially in warmer and more humid climates. Of all the tradeoffs in low-VOC formulations, dirt pick-up resistance is one of the most noticeable changes, particularly in traditional exterior house paints. Although dirt pick-up is a complex process, the interaction of the resulting paint film with environmental particles, weather, and other variables is ultimately defined by the surface properties, which affect the accumulation of dirt. This study will explore the effects of siloxane and silica additive technologies on dirt pick-up resistance using an accelerated test method, and the mechanism of action of these additives will be discussed.

Introduction

Dirt pick-up resistance (DPUR) is a topic of high interest in exterior architectural coatings. Consumers want paint that can resist staining while also being easy to clean. In developing dirt-resistant paints, outdoor exposure is necessary for providing real-world data; however, these tests can take many months, or years, before a coating's performance can truly be understood. Hence, accelerated DPUR tests are critical to enabling formulation development and providing insights into how various components may contribute to the DPUR of a coating. Unfortunately, there is currently no standard accelerated DPUR method in the coatings industry; several methods focus on a dry deposition of a dirt source, meanwhile methods developed in wetter climates feature dirt application using a dirt/water slurry.[1] In general, existing accelerated DPUR methods follow a procedure consisting of 1) applying the paints to panels, 2) curing the coated panels under conditions of controlled temperature and humidity, 3) exposing the cured panels to ultraviolet (UV) irradiation and higher temperature, 4) applying and removing the dirt from the conditioned panels, and 5) analyzing

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the extent of dirt pick-up and removal from each panel. However, the dirt application and removal step has been acknowledged to be the greatest source of low reproducibility for these methods.[2] In this work, a new accelerated DPUR test method was developed to facilitate studying both initial pickup of dry dirt and how well that dirt could be rinsed off when subjected to a rinsing process.

While siloxanes have traditionally been utilized to enhance surface slip, flow, and leveling,[3] recent research has demonstrated that siloxane surface control agents can also significantly boost block resistance of coatings.[4,5] In a similar vein, spherical silica particles have been found to increase burnish and wet scrub resistance of architectural coatings due to their effect on the dry coating film.[6]

These results motivated us to investigate the impact of various polyether-modified siloxanes, emulsions of higher molecular weight and crosslinked siloxanes, aqueous dispersions of fumed silicas, and spherical precipitated silicas on dirt adhesion and its release.

Experimental

A commercial low-VOC waterborne, self-crosslinking acrylic exterior satin paint was chosen to evaluate various siloxane and dispersed silica additives. Siloxane-based surface control additives (SCAs) were post-added at 0.50 wt % to the commercial paint, mixed well, and the paints were allowed to stand overnight before use. Similarly, dispersed silica additives (DSAs) were post-added at 1.0 wt % to the commercial paint, mixed well, and the paints were

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allowed to stand overnight before use. The characteristics of the additives tested are summarized in **Tables 1** and **2**.

A low-VOC waterborne, self-crosslinking acrylic exterior flat paint with a 76% PVC was formulated (**Table 3**) and it was used to evaluate the effects of full volume-to-volume replacement of calcium carbonate filler with spherical silica fillers (SPHs) of varying particle size (**Table 4**). Similarly, once mixed, the formulations in **Table 3** were allowed to rest overnight to release residual foam from mixing.

The commercial paint samples to which had been post-added various siloxane and dispersed silica additives and the coating formulations in **Table 3** were applied to scrub charts using a 150-mm wide bird-type film applicator for a 4-mil wet film thickness. After two days of drying in a standard conditioning atmosphere of 21–25 °C and 45–55% relative humidity (ASTM D4332-22), the scrub charts were trimmed to the test panel size (12.7 cm x 10.8 cm; center of coating) and cured in a Q-SUN Xe-3 Xenon Test Chamber (Ser. No. 20-34603-79-X3HBSE; from Q-Lab) at 40 °C and 45% relative humidity using a full-spectrum sunlight filter with an irradiance of 0.89 W/m^2 at 340 nm for three days. The test panels were removed and allowed to rest for two hours before dirt deposition. A number of studies have revealed that typical outdoor dirt particles that soil exterior coatings have a number median diameter of approximately 100 nm.[7-9] Hence, for this study, Lamp Black 101 (carbon black from Orion Engineered Carbons), and BAYFERROX® 509 (iron oxide from Lanxess) were chosen. These dirts were applied via fine-mesh sifters to achieve a complete and uniform coverage of the test panels. After one day, the dirt was removed by lifting the panels upright and tapping 15 times against the benchtop. The panels were then rinsed at an upright position with 15 misting sprays of DI water from a spray bottle at a distance of 20 cm. The residual water was removed by tapping the panels 15 times, after which they were returned to a horizontal position and dried overnight.

To evaluate DPUR, the coatings industry typically uses colorimeters to measure the color change of the test panels between pre- and post-dirt application.[1] However, these instruments have small measuring spots, which would not be representative of the DPUR of the entire test panel. Therefore, the dirt pick-up of the entire coated panel surface (excluding 1 cm on each edge for sample handling) both before and after the rinsing process was captured using a controlled camera setup, as seen in **Figure 1**. To minimize sample-to-sample deviation, a mounted camera and lab jack ensured consistent focal length, a lightbox provided consistent lighting, and a secured corner frame assisted in sample placement. Pictures of each test panel were taken following dirt removal (“Before Rinse”) and after drying from the rinsing process (“After Rinse”). The percentage of the test sample with dirt remaining on its surface was quantified by using an “Above/Below” image threshold adjustment feature in the public domain scientific image analysis software, ImageJ from the National Institutes of Health, Bethesda, MD. Examples of original pictures of test panels and their corresponding conversion to black-and-white images by ImageJ are shown in **Figures 2** and **3**.

TABLE 1
Characteristics of Siloxane Additives

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

TABLE 2
Characteristics of Fumed Silica Dispersions

	Activity	Surface Treatment of Active Silica	Surface Area of Active Silica (m^2/g)	pH (5% in Water)	Stabilizing Agent
DSA#1	20%	None	200	10.0	Ammonia
DSA#2	20%	Proprietary	Proprietary	10.5	DMEA
DSA#3	20%	DDS	260	10.5	DMEA

TABLE 3
76% PVC Waterborne Low-VOC Acrylic Exterior Flat Paint Formulations

Raw Material	100% $CaCO_3$ / 0% SPH (v/v)	0% $CaCO_3$ / 100% SPH (v/v)
	Mass (g)	
Grind		
DI Water	124.28	133.17
Cellulose Thickener	1.88	2.02
Polyacrylate Dispersing Agent	0.92	0.98
Biocide	1.49	1.60
pH Modifier	0.48	0.51
Siloxane Defoamer	0.49	0.53
Wetting Agent /Grind Aid	1.00	1.00
Titanium Dioxide	53.32	57.14
Wollastonite	23.08	24.74
Calcined Aluminum Silicate	92.44	99.05
DI Water	11.81	12.65
Calcium Carbonate	124.86	0.00
Spherical Silica	0.00	99.10
Coalescent	2.98	3.19
Let-Down		
100% Acrylic Latex Binder	65.23	69.89
Siloxane Defoamer	0.50	0.53
Total	**504.76**	**506.1**

TABLE 4
Particle Sizes of Calcium Carbonate and Spherical Precipitated Silica Particles

Particle	d_{50} (μm)	d_{95} (μm)
$CaCO_3$	7	–
SPH#1	10	18
SPH#2	14	24
SPH#3	17	32

FIGURE 1
Inside view of controlled camera setup.

FIGURE 2
Panels coated with commercial paint without additives: (A) the original photograph after carbon black was tapped off but before rinse and (B) the corresponding black/white conversion image (threshold of 135) showing 54.8% dirt remaining on the coating; (C) the original photograph after carbon black panel was rinsed and (D) the corresponding black/white conversion image (threshold of 135) showing 63.5% dirt remaining on the coating.

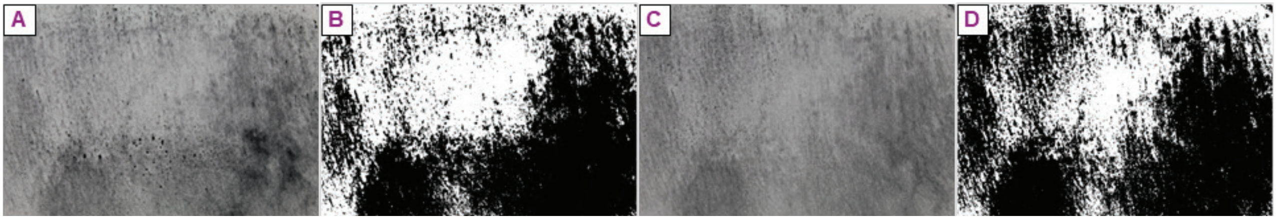
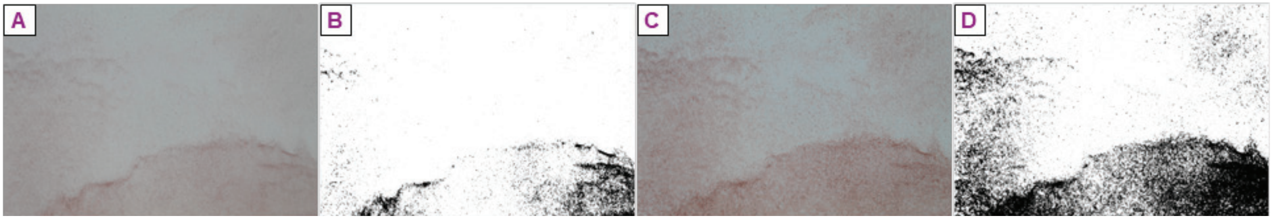


FIGURE 3
Panels coated with commercial paint without additives: (A) the original photograph after iron oxide was tapped off but before rinse and (B) the corresponding black/white conversion image (threshold of 140) showing 5.1% dirt remaining on coating; (C) the original photograph after iron oxide panel was rinsed and (D) the corresponding black/white conversion image (threshold of 140) showing 22.0% dirt remaining on the coating.



Gloss measurements at 60° and 85° were taken on the coated scrub charts using a Micro-TRI-gloss (Cat. No. 4446, Ser. No. 1083656, from BYK Additives & Instruments).

A Drop Shape Analyzer (DSA100 model FM3200; Ser. No. 20055608; from KRÜSS) with ADVANCE software was used for the analysis of contact angle of the coating surfaces. HPLC grade water (15 µL) onto the surface of the clean, cured coating to observe the contact angle of the liquid over time (60 s). The DSA100 high-speed camera captured data every second for the full minute of monitoring. By observing each droplet over time, a pattern of "wetting-out" could be seen as each droplet's contact angle decreased over the time period. Six droplets were measured for each sample.

The porosities of the coatings were determined by using a mineral oil absorption test. Coatings were prepared on pre-weighed half-length scrub charts using a 150-mm-wide bird-type film applicator for a 3-mil wet film thickness. The test panels were cured like those made to study DPUR (two days in a standard conditioning atmosphere of 21–25 °C and 45–55% relative humidity [ASTM D4332-22] and three days in the Q-Sun). The panels were weighed again to obtain the mass of the coating film on the charts. Low viscosity mineral oil (viscosity = 11.84 cSt at 40 °C; specific gravity = 0.8451; from Spectrum Chemical) was applied to the coating panel using a 150-mm wider bird-type film applicator for a 2-mil wet film thickness. The oil sat on the surface for three minutes, and then all residual oil and sheen were wiped off using non-abrasive, absorbent towels. The panel was weighed again. The nonvolatile content (by weight) of the paint formulations were determined according to ASTM D2369-20. Finally, the porosity of the paint coatings was calculated according to ASTM D6583-13.

The roughness of the coating formulations was measured using a HOMMEL ETAMIC Waveline 5 handheld profilometer (Ser. No. 151947; from Jenoptik). Parameters Ra, Rz, and Rz_{max} were taken in accordance with ISO 21920 (formerly ISO 4287). Seven measurements were recorded for each sample.

Scanning electron microscopy (SEM) was used to evaluate topographical and elemental analysis of the paint formulations with (and without) spherical silica particles. Test panels of each formulation were applied to Leneta 5C charts and cured as before. A portion of each test panel was cut out from the chart for surface analysis. All specimens were coated with a layer of gold/palladium alloy via sputter deposition prior to their analysis. Topographical SEM analysis was conducted using a Hitachi S-4800 field-emission SEM operated at 15 kV accelerating voltage to obtain secondary electron (SE) images at 100x magnification of the top surfaces. Meanwhile, a JEOL JSM-5910LV SEM operated at 15 kV accelerating voltage coupled with a Thermo Noran System Six Si-drifted EDS detector was used to collect X-ray maps at 500x magnification of the coating surfaces.

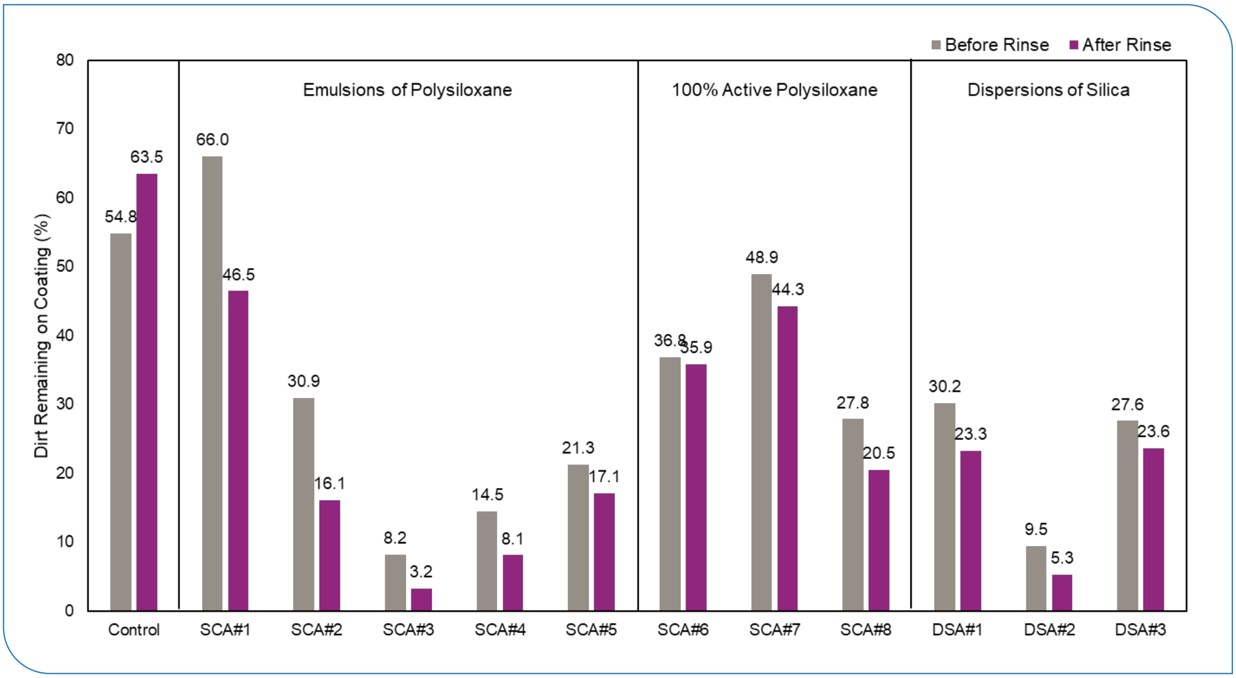
Results and Discussion

Evaluation of DPUR of Exterior Coatings Featuring Siloxane and Silica Technologies

The carbon black dirt pick-up results of the commercial satin paint with various SCAs and DSAs are shown in **Figure 4**. Nearly all additives improved the initial DPUR (before the rinsing process) compared

FIGURE 4

Dirt pick-up of carbon black on commercial exterior satin paints containing various siloxane and dispersed silica additives.



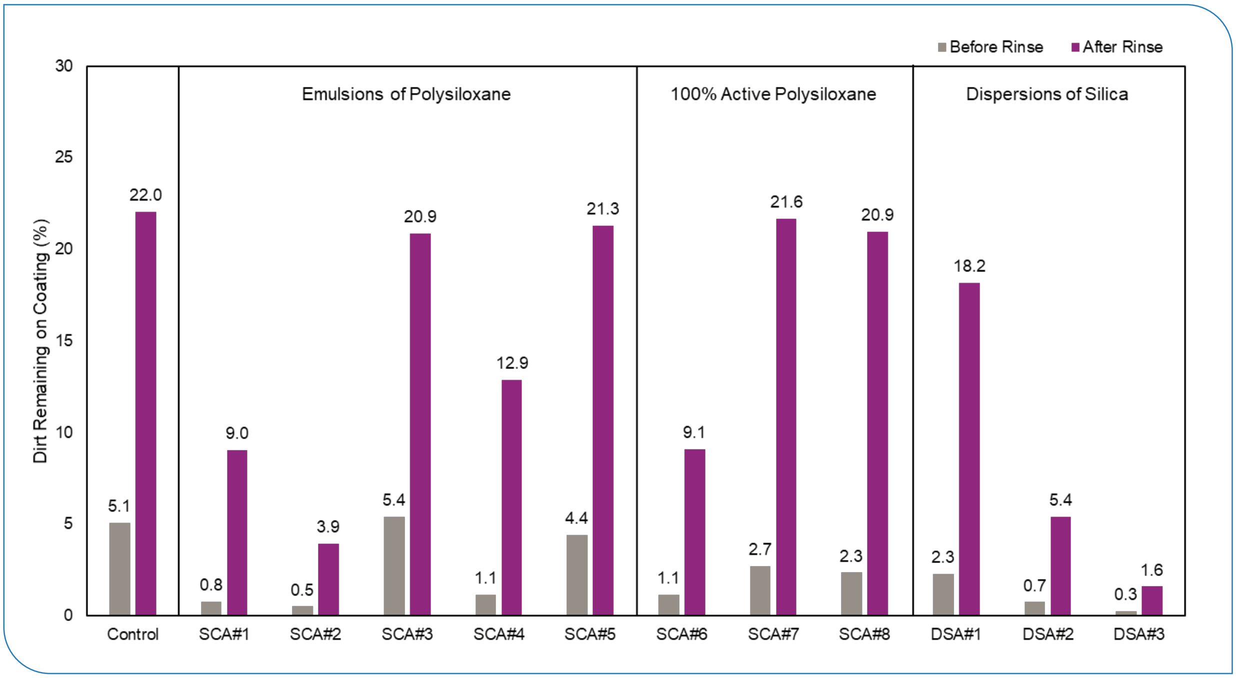
to the control paint with no additional additives, and the rinsing process demonstrated at least some dirt removal from the test panels. Among the five siloxane emulsions tested, SCA#3, SCA#4, and SCA#5 performed the best, exhibiting significantly lower initial dirt pick-up with carbon black. These are high molecular weight, crosslinked siloxanes that have been stabilized in a waterborne emulsion form; it is believed that they function as silicone particles on the coating surface, with particle size and physical characteristics playing a crucial role in their performance. SCA#1, which is an emulsion of a very hydrophobic amino-functionalized siloxane had the worst DPUR against carbon black; while SCA#2, which is a hydrophobic siloxane resin, demonstrated moderate improvement. Interestingly, the best rinsability was observed with SCA#1 and SCA#2, both of which are based on very hydrophobic siloxanes, showing the greatest differences between the pre- and post-rinse dirt pick-up. In comparison to the five emulsion polysiloxanes, SCA#6, SCA#7, and SCA#8 were less effective than most of the emulsion polysiloxanes. SCA#6, SCA#7, and SCA#8 are 100% active polysiloxanes and typically exhibit a film effect, with the length of the siloxane chain and organic modification controlling system compatibility and interfacial behaviors. The dispersions of silica DSA#1, DSA#2, and DSA#3 also showed improved DPUR with carbon black, with DSA#2, based on a proprietary functionalized silica demonstrating the most significant improvement.

The initial iron oxide dirt pick-up was low for each commercial test paint sample, with a maximum of 5.4% dirt remaining on the coating surface before the rinsing cycle (**Figure 5**). Unlike carbon black which showed some dirt removal upon rinsing, the iron oxide dirt dispersed across the coating surface upon water misting, resulting in greater dirt coverage on the test panels after the rinsing process, as seen in **Figure 3**. Among the five siloxane emulsions tested, SCA#1 and SCA#2, both very hydrophobic with a low degree of organic modifications, as well as SCA#4, which has a larger particle size than SCA#3 and SCA#5, exhibited great DPUR improvement against iron oxide. SCA#6 performs best among the three 100% active polysiloxanes. The dispersions of silica DSA#2 again demonstrated great initial iron oxide resistance and showed only a small increase in color after rinsing. DSA#3 outperformed all additives tested in this series, showing only 0.3% iron oxide initially on the coating surface and 1.6% after rinsing.

Tests in this section led us to believe that the particle effect plays a crucial role in DPUR in this system, as the most significant improvement was seen with emulsified polysiloxanes and dispersed silicas.

The effects of volume-to-volume replacement of calcium carbonate filler with SPHs of varying particle size were studied. These spherical silica particles were produced through a novel precipitation process and possess unique properties such as very low surface area, low oil absorption, and an extremely narrow particle size distribution. Previous studies found that these spherical precipitated silica particles are particularly efficient in increasing the burnish resistance and wet scrub resistance of interior architectural coatings, as well as in providing matting and physical property improvements in powder coating formulations.[10,11]

FIGURE 5
Dirt pick-up of iron oxide on commercial exterior satin paints containing various siloxane and dispersed silica additives.



In this 76% PVC exterior flat paint formulation, DPUR against iron oxide was significantly improved by using these spherical silica particles. It can be observed that as the silica particle size increases, less dirt remains on the coating surface (**Figure 6**). Notably, the largest spherical silica particle, SPH#3, significantly improved DPUR against iron oxide with only 0.5% iron oxide remaining on the surface before rinse and 1.0% after rinse. However, these spherical silica particles did not improve the DPUR of the coating to carbon black (**Figure 7**).

Surface Characterization: Analysis of Gloss, Contact Angle, Porosity, and Roughness

Gloss measurements were conducted on panels coated with both the commercial exterior satin paint and the 76% PVC exterior flat paint containing some of the additives. In the commercial exterior satin paint, there were negligible differences (≤ 2 units) in both the 60° and 85° gloss measurements between the blank test panel and the test panels of the paints to which siloxane and dispersed silica additives had been post-added (**Table 5**). Meanwhile, full volume-to-volume replacement of the calcium carbonate filler with larger particle size spherical silica (SPH#2 and SPH#3) in the 76% PVC exterior flat paint resulted in a slight reduction of gloss.

While surface hydrophobicity is believed to play a large role in reducing dirt pick-up,[12] contact angle analysis of the commercial paint samples to which various siloxanes had been post-added did not reveal any correlation between the contact angle of water on the coating and the dirt pick-up of carbon black or iron oxide (**Table 5**). In the 76% PVC exterior flat paint formulation, the use of spherical silicas caused a significant increase in the contact angle of water on the coating. Given the

FIGURE 6
Dirt pick-up of iron oxide on 76% PVC exterior flat paint formulations with spherical silica fillers.

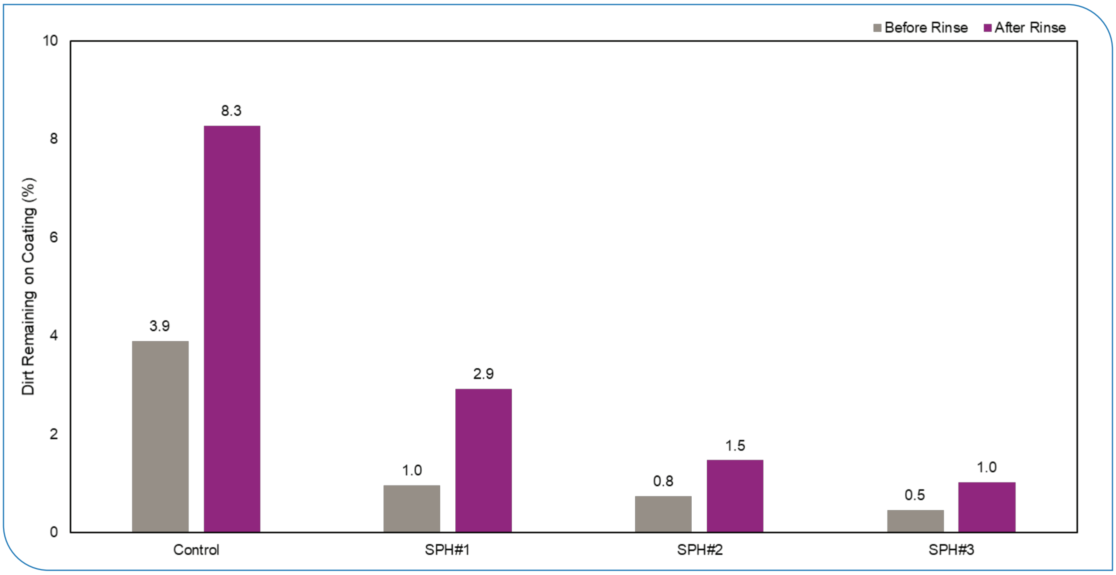


FIGURE 7
Dirt pick-up of carbon black on 76% PVC exterior flat paint formulations with spherical silica fillers.

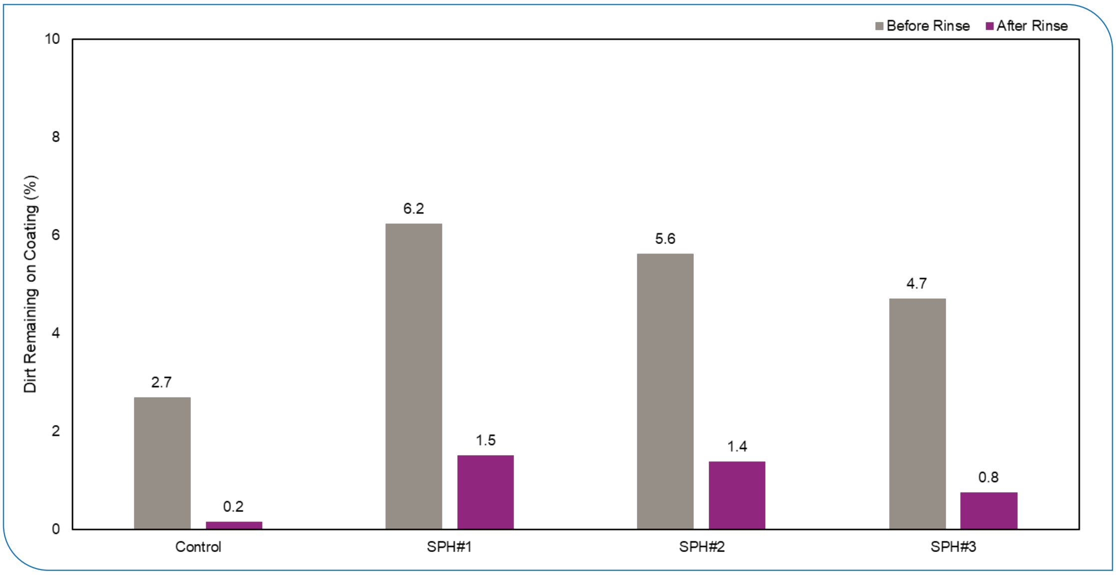


TABLE 5
Gloss and Contact Angle Measurements and Dirt Pick-up of Coating Surfaces

Sample	60° Gloss	85° Gloss	Water Contact Angle (°)	Carbon Black Before Rinse (%)	Carbon Black After Rinse (%)	Iron Oxide Before Rinse (%)	Iron Oxide After Rinse (%)
Commercial Exterior Satin Paint with SCAs and DSAs							
Blank	13.7	33.4	85	54.8	63.5	5.1	22.0
SCA#1	13.2	32.4	93	66.0	46.5	0.8	9.0
SCA#2	13.2	32.5	88	30.9	16.1	0.5	3.9
SCA#3	13.3	32.8	93	8.2	3.2	5.4	20.9
SCA#4	12.9	32.9	88	14.5	8.1	1.1	12.9
SCA#5	14.3	35.4	94	21.3	17.1	4.4	21.3
DSA#2	12.3	31.8	84	9.5	5.3	0.7	5.4
76% PVC Exterior Flat Paint Formulations with Spherical Silica							
Control	2.4	5.8	83	2.7	0.2	3.9	8.3
SPH#1	2.4	4.7	100	6.2	1.5	1.0	2.9
SPH#2	2.3	3.1	104	5.6	1.4	0.8	1.5
SPH#3	2.3	2.8	101	4.7	0.8	0.5	1.0

hydrophilic nature of the spherical silica particles, this increase in coating hydrophobicity prompted further investigation.

The surface character of the 76% PVC exterior flat paint formulations with SPH was then further probed by measuring porosity and surface roughness (**Table 6**). Full volume-to-volume replacement of calcium carbonate with the spherical silica particles resulted in a minor increase in the porosity of the coatings. As the particle size of silica increases, the surface roughness also increases. Previous studies have noted that differences in surface roughness did not seem to influence the dirt pick-up of the paint coatings.[1,12] However, the surface roughness differences observed in those reports were found to be in the sub-micron range. The difference in the surface roughness in **Table 6** range from 1–5 μm in Rz value; therefore, we hypothesize that the roughness differences stemming from increased particle sizes of the spherical silica—which leads to an increase in surface area of the resin binder which lies on top of the particles of the coating—is the reason for increased carbon black dirt pick-up compared to that of the control formulation.

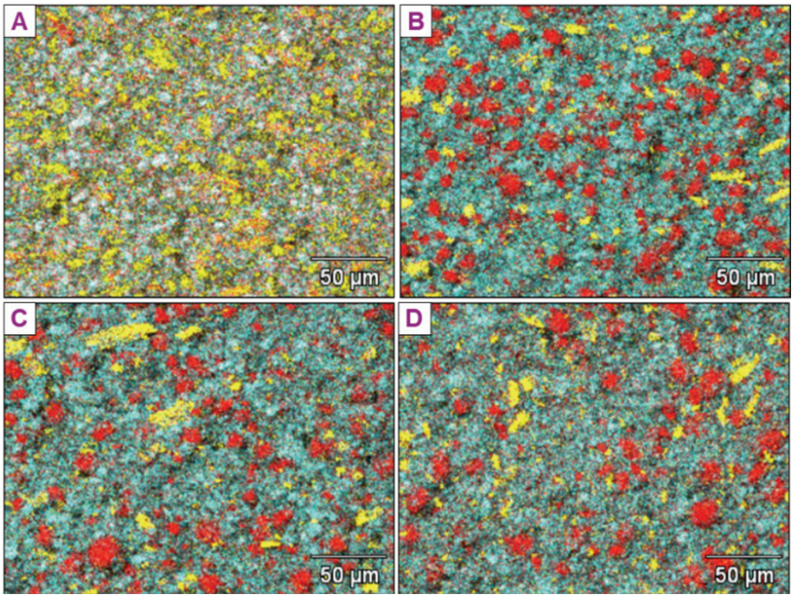
SEM of 76% PVC Exterior Flat Paint with Spherical Silica Particles

Figure 8 displays topographical SEM images with element mapping. In the

TABLE 6
Porosity and Roughness Measurements for 76% PVC Exterior Flat Paint Formulations with Spherical Silica

Silica	Porosity (%)	Ra (μm)	Rz (μm)	Rz_{max} (μm)
None (Control)	19	1.4	10.9	12.9
SPH#1	22	1.6	12.1	14.8
SPH#2	22	1.9	14.3	16.4
SPH#3	21	2.0	15.8	20.5

FIGURE 8
Surface topographical SEM EDS images (500x magnification) of 76% exterior flat paint formulations containing calcium carbonate (A), SPH#1 (B), SPH#2 (C), SPH#3 (D). [Key: red = Si; yellow = Ca; green = Ti; blue = Al]



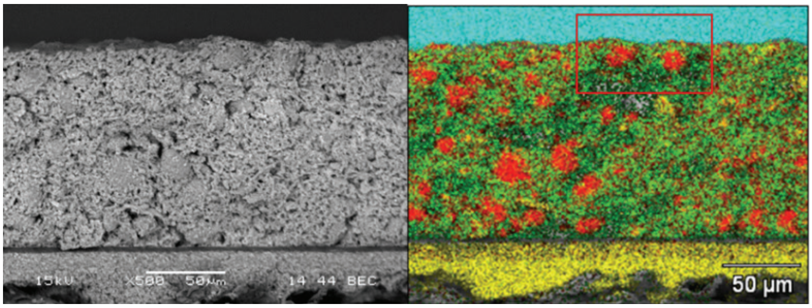
original flat formulation with calcium carbonate (A), a strong signal for calcium was observed as expected. In the images for the samples containing the spherical silicas (B, C, and D), the calcium signals were significantly reduced because calcium carbonate has been replaced with the silicas. The remaining calcium signals were most likely due to wollastonite, which contains calcium and is usually a bladed, columnar, or fibrous morphology, consistent with the images B, C, and D. The round silicon areas in B, C, and D confirmed the existence of spherical silica near the surface. Because these spherical silicas are known to have a very narrow particle size distribution, the size difference between the silicon areas was most likely due to the extent that the silica particles were protruding from the surface relative to the penetration depth of the electron beam (1 to 2 µm).

In addition, the spherical silica was seen to be evenly dispersed throughout the film as confirmed by cross-section SEM (**Figure 9**). Small amounts of Ti were observed on the silicas, likely due to the binder wetting the surface and forming a film carrying TiO_2. Some spherical silicas were not fully exposed at the surface, but instead covered by a thin layer of binder, which was further confirmed in the cross-section SEM. With the two highlighted silica particles, it is clear that there was a layer of Ti on top of the spherical silica. By combining the contact angle and roughness data, we can develop a hypothesis to explain the observed DPUR phenomenon. The use of spherical silica increased the surface roughness and surface area, requiring more binder to cover a rougher surface. This results in an increase in hydrophobicity and more affinity to carbon black but leading to less deposition of iron oxide. Although the three spherical silicas are untreated and hydrophilic in nature, the surface chemistry of the spherical particles could be irrelevant since they are covered by the binder/TiO_2.

Conclusions

A new accelerated DPUR test method was developed to evaluate both the dirt pick-up and removal of two industry-relevant dirts: carbon black and iron oxide. Using this new method, the effects of both siloxane-based additives and silicas on the DPUR of a commercial exterior satin paint as well as a 76% PVC exterior flat paint were studied. Results have shown that DPUR can be significantly and positively impacted by both siloxanes and silicas. The effect of particles on the coating's surface is believed to be a contributor to improved DPUR, as was seen with the commercial paints containing aqueous emulsions of hydrophobic and crosslinked siloxanes as well as silica dispersions and with the internally produced paint formulations containing the spherical silica fillers. This preliminary work has demonstrated that several different siloxane and silica chemistries show promise in improving DPUR and could be useful tools for formulating exterior architectural paints. Investigation into the reproducibility of this new method is underway in our labs. Furthermore, these additives will be evaluated in an extended outdoor exposure study to understand whether these accelerated dirt pick-up resistance results translate to performance in the field.

FIGURE 9
Cross-sectional SEM image of 76% exterior flat paint formulations with SPH#3. [Key: red = Si; yellow = Ca; green = Ti; blue = Al]



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