Performance Study of 3M™ Ceramic Microspheres W-410 in a Water-Based Acrylic Flat Architectural Paint

Lesbia E. Girón, 3M Advanced Materials Division

Abstract

3M™ Ceramic Microspheres are functional additives used to enhance performance in a variety of industrial and architectural paints and coatings. The purpose of this study was twofold: to determine the effect of 3M™ Ceramic Microspheres W-410 when added to a given paint; and to determine the optimal amount of 3M ceramic microspheres to improve performance and quality in a flat paint formulation.

The results of the study demonstrate that the addition of 3M ceramic microspheres W-410 increases dispersibility, scrub resistance, washability, burnish resistance and gloss control in a water-based flat architectural paint.

Introduction

3M™ Ceramic Microspheres are inert, thick-walled white spheres made of alkali alumino-silicate ceramic and featuring a fine particle size distribution. Intrinsically hard, 3M ceramic microspheres offer higher strength than any other spherical filler or extender. Because spheres have the lowest surface area to volume ratio of any shape, 3M ceramic microspheres roll easily over one another – like microscopic ball bearings – allowing high packing density. This contributes to lower viscosity, improved flow and sprayability, higher solids and reduced resin demand. It is therefore possible maintain viscosity when reformulating a paint with 3M ceramic microspheres in place of irregularly shaped fillers, even at higher filler loadings.

3M ceramic microspheres afford paint manufacturers the ability to reduce costs through lower resin demand while enhancing overall paint performance. These engineered additives also contribute customer-pleasing functional properties (i.e., better hiding, scrub resistance and easy washability), adding value and providing a market advantage for paint manufacturers.

This study was conducted in order to better understand the performance of 3M ceramic microspheres in flat, water-based architectural paint systems. Dispersibility, washability, gloss control, burnish and scrub resistance properties were evaluated. The data presented here is designed to aid paint manufacturers in determining the optimal amount of 3M ceramic microspheres for a given paint formulation. The goal was to produce a high-performance, high-quality formulation optimized for customer-pleasing properties.

Table 1 Properties of 3M™ Ceramic Microspheres W-410

True Density	2.4 g/cc (20.0 lbs/gal)
Bulk Density	1.5 g/cc (12.6 lbs/gal)
Particle Size (microns)	Median: 4
	Top: 24
Whiteness (“L” Value) [ASTM D2244]	95+ (Hunter L,a,b scale)
Crush Strength	> 4,200 kg/cm^2 (>60,000 psi)
pH [ASTN E70]	9.0 – 12.0
Hardness [Mohs Scale]	7
Softening Point	1,020°C (1,870°F)
Refractive Index [Becke Line]	1.53
Dielectric Constant	3.19
UV Light Transmission	UV Transparent to 250 nm
Thermal Conductivity	2.3 W/mK

Summary

A flat paint formulation was selected both for its value to paint manufacturers, as mentioned previously, and because of the complexity of its high pigmentation level in comparison with other paint types. 3M ceramic microspheres W-410 were added in varying volume percentages (0 – 20 vol%) to the starting formulation (PVC 40 – 60%) in order to determine the optimal addition for obtaining the highest paint performance.

The results of this study show that, for a water-based flat paint formulation, optimal performance was achieved by adding 5 vol% of 3M ceramic microspheres W-410.

Experimental

Materials

The paint used as the starting formulation was a medium-performance water-based acrylic flat paint with a Pigment Volume Concentration (PVC) of 40%, identified here as DG-1. This paint was made with an acrylic latex binder and pigments/extenders as listed in Table 2; note that the starting formulation as described in Table 2 is the control, and does not incorporate 3M ceramic microspheres.

Table 2 Starting Point Formulation DG-1

Product	Commercial Name	Weight (lbs)	Volume (gal)
Grind			
Water	–	95.61	11.45
Dispersant	Tamol™ 850	7.06	0.70
Potassium trypoliphosphate	–	1.06	0.05
Surfactant	Triton™ CF-10	1.54	0.18
Defoamer	Rhodoline® 643	0.70	0.10
Titanium dioxide	Ti-Pure® R-706	123.35	3.69
Zinc oxide	Eagle Zinc 417W (Alfa Aesar 99.99%)	17.62	0.37
Nepheline syenite	Minex® 4	65.29	3.00
Calcined aluminum silicate	Burgess Iceberg®	43.53	1.98
Calcium carbonate	Duramite®	65.29	3.01
Ceramic microspheres	3M™ Ceramic Microspheres W-410	0.00	0.00
Thickener	Attagel® 50	3.52	0.18
Biocide	Skane™ M-8 Mildewcide	1.52	0.18
Hydroxyethylcellulose (2.5% in water)	Natrosol™ 250 MHR	71.01	8.46
Letdown			
Ester alcohol	Eastman Texanol™	6.64	0.84
Propylene glycol	–	4.57	0.53
Binder	Rhoplex™ VSR-50	276.90	31.29
Defoamer	Rhodoline® 643	2.50	0.35
Thickener	Acrysol™ RM-2020 NPR	12.27	1.41
Final Paint	**Density 11.80 lbs/gal**		
	PVC 40%		

Procedure

The starting paint formula was prepared in two stages: 1) grind; and 2) letdown. The dispersion of the grind stage was performed for 20 minutes at 1600 rpm using a high dispersion mixer equipped with a Cowles blade. Once the grinding stage was completed, the fineness of dispersion was measured by Hegman-type gauge according to ASTM D1210, using an ADM-2 Automatic Drawdown machine.

The resin, coalescent, defoamer, biocide and thickeners were added during the letdown stage. The viscosity of the paint was subsequently measured with a Digital KU-2 T-Stormer Type Viscometer.

Table 3 lists the control formulation and five variants incorporating different volume percentages of 3M™ Ceramic Microspheres W-410. All formulations were prepared using the same procedure as DG-1, as described previously. The PVC values for the five variant formulas ranged from 40 – 56%. All prepared samples had initial viscosities in the range of 95 – 100 Krebs.

Table 3 Addition level of 3M™ Ceramic Microspheres W-410

Paint Formulation	Vol% Ceramic Microspheres W-410	PVC
DG-1*	0	42
DG-2	2.5	40
DG-3	5.0	40
DG-4	10	40
DG-5	15	49
DG-6	20	56

*Starting point formulation

Panel preparation for washability, scrub and burnish resistance tests

Drawdowns were performed on black plastic panels from Leneta Company using a 0.18 mm (7.0 mil) clearance film applicator. Panels were then air dried in a horizontal position for 7 days prior to testing.

Panel preparation for opacity

Paint drawdowns were made on 3B Opacity Charts from Leneta Company using a 3 mil bird gauge. After the samples were completely dry, a Color Flex EZ spectrophotometer was used to determine the opacity of the samples by contrast ratio measurement, as shown by the following equation:

$$\mathbf{y} = \frac{\mathbf{y}_{\text{black backing}}}{\mathbf{y}_{\text{white backing}}} \times 100$$

Table 4 Additional Test Methods

True Density	Method	2.4 g/cc (20.0 lbs/gal)
Scrub Resistance	ASTM D2486 Test Method A	A 0.5 mm shim was used instead of 0.25 mm. A non-scratch Scotch-Brite™ pad was attached to the bottom of the brush. Standard abrasive scrubbing media was used as indicated in the ASTM test method.
Burnish Resistance	ASTM D6736	None
Specular Gloss	ASTM D523	None
Washability	ASTM D4828	A blue Paper-Mate® Pen was used (see test method in Appendix). Lines were cleaned until they were no longer visible; the number of passes was recorded at that point.

Results and Discussion

Dispersibility

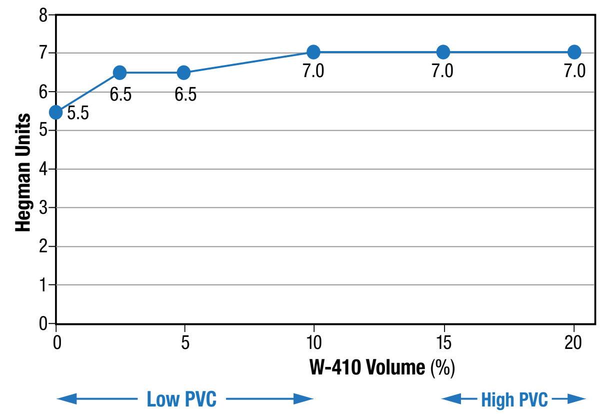
The Hegman Grind test measures the occurrence of large, coarse particles that can result in blotchy color. By helping to evenly space pigments, 3M ceramic microspheres can improve color acceptance; tight packing allows a smooth, even finish.

As shown in Figure 1, the dispersibility of a paint formulation increases with higher loadings of 3M ceramic microspheres W-410. The control formula DG-1, which does not contain ceramic microspheres, has the lowest Hegmen grind at 5.5 HU. Adding 2.5 and 5 vol% 3M ceramic microspheres W-410 yields a higher value (6.5); and at 10 vol% filler loading, formula DG-4 reaches a Hegman grind value of 7. While formulas DG-5 and DG-6 incorporate higher loadings of 3M ceramic microspheres and have higher PVCs, they maintain the same high Hegman value as DG-4. The Hegman grind does not increase above 7 with loadings >10 vol% of 3M ceramic microspheres W-410.

The increase in fineness of grind shown in Figure 1 can likely be attributed to the replacement of high volume percentages of calcium carbonate – which has a large particle size – with 3M ceramic microspheres, which offer fine particle size distribution.

These results are consistent with the theory that 3M ceramic microspheres provide good particle spacing and are easily dispersed. Together, these two qualities improve the stability of the dispersion, thereby limiting the potential for re-agglomeration and flocculation.

Figure 1 Fineness of Grind with 3M™ Ceramic Microspheres W-410



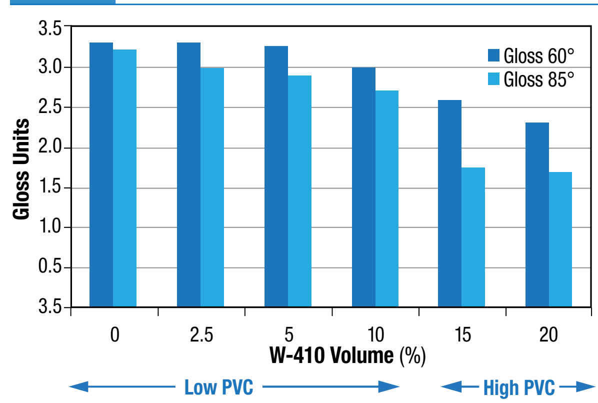
Gloss

3M™ Ceramic Microspheres control glossiness by providing surface texture. The degree at which ceramic microspheres W-410 reduce gloss for a water-based flat architectural paint is shown in Figure 2. For this particular type of paint, glossiness (also known as sheen) at an incidence angle of 85° is the most relevant value. For reference, measurements were also taken at 60°.

The volume percentage of ceramic microspheres W-410 is inversely proportional to sheen in the paint film. For lower loadings, ceramic microspheres W-410 gradually reduce gloss while maintaining PVC in the same range as the starter formulation. With 10 vol% ceramic microspheres, DG-4 delivers the most gloss reduction at 40% PVC.

Higher PVC formulations (DG-5 and DG-6, at 49% and 56%, respectively) demonstrate a dramatic reduction in gloss with higher loadings of ceramic microspheres W-410.

Figure 2 Effects of 3M™ Ceramic Microspheres W-410 on Paint Gloss



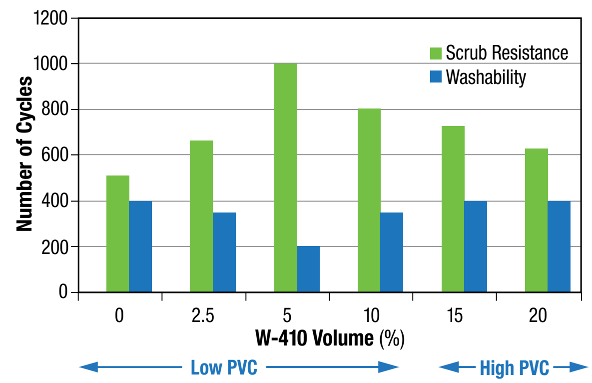
An important advantage of 3M ceramic microspheres is that these additives can control gloss without significantly increasing viscosity. All formulations evaluated in this study maintained an initial viscosity in the range of 94 – 98 Krebs (KU).

Scrub resistance and washability

While soft, irregularly-shaped particles break or wear away, 3M ceramic microspheres are hard and durable; their spherical shape may help prevent the particles at the paint surface from being abraded when scrubbed. This helps keep painted surfaces smooth. The formulations incorporating ceramic microspheres demonstrated improved scrub resistance compared to the starting formulation DG-1, as shown in Figure 3. The green bars denoting scrub resistance show the number of passes until failure; the higher the value, the more resistant the paint is to abrasion.

The best scrub resistance properties were obtained with an addition level of 5 vol% ceramic microspheres W-410 (formula DG-3), which withstood 1000 scrub cycles before the paint failed – doubling the resistance of the starting point formulation. Formulas DG-5 and DG-6, with 15 and 20 vol% ceramic microspheres W-410 and PVCs of 49 and 56, respectively, also exhibited higher scrub resistance than the control.

Figure 3 Effects of 3M™ Ceramic Microspheres W-410 on Washability and Scrub Resistance



The washability test evaluates how easily soil can be removed from a coated surface, and whether washing leaves behind any change in appearance. The spherical shape of 3M ceramic microspheres may help prevent dirt from becoming trapped beneath the particles, as is more common with irregularly shaped fillers. The blue bars in Figure 3 show the number of cycles until a soiled drawdown panel is cleaned; the lower the value, the easier it is to wash the painted surface.

The best washability performance was also obtained at an addition level of 5 vol% ceramic microspheres W-410 (formula DG-3). Whereas the control formula needed 400 wash cycles to remove the soilant from the painted surface, formula DG-3 was clean after only 200 cycles. Formulas DG-5 and DG-6, on the other hand, exhibited the same washability as the starting point formula, which has a lower PVC and higher resin content. As film porosity increases, the staining media (ink) penetrates deeper. Less porous paints are not only easier to clean, but also better retain uniformity of sheen when washed.

With a 5 vol% loading of 3M ceramic microspheres W-410, formulation DG-3 clearly exhibits the optimal balance of scrubbability and washability properties.

Burnish Resistance

When a painted surface is repeatedly brushed against or rubbed, it can become glossy or burnished. This is especially noticeable – and undesirable – in flat architectural paints containing a high amount of fillers. Burnishing is a function of both film cohesiveness and material hardness. 3M ceramic microspheres impart coating hardness to better protect against burnishing. In addition, the tight packing density of 3M ceramic microspheres can help provide a more uniform surface compared to paints with irregularly shaped fillers, which can protrude and reflect light.

In this test, a painted surface is repeatedly rubbed and subsequently measured for gloss, as shown in Figure 4; the higher the percentage of gloss increase, the more burnished the surface. Once again, formula DG-3 yields optimal results: a 5% volume loading of 3M ceramic microspheres W-410 exhibited the least amount of burnishing at both 60° and 85° angles of incidence. All formulations containing 3M ceramic microspheres exhibited improved performance compared to the control formula DG-1, which was least resistant to burnishing.

Figure 4 Effect of 3M™ Ceramic Microspheres W-410 on Burnish Resistance

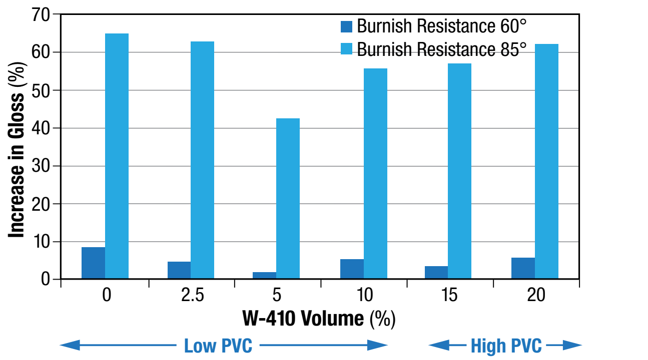
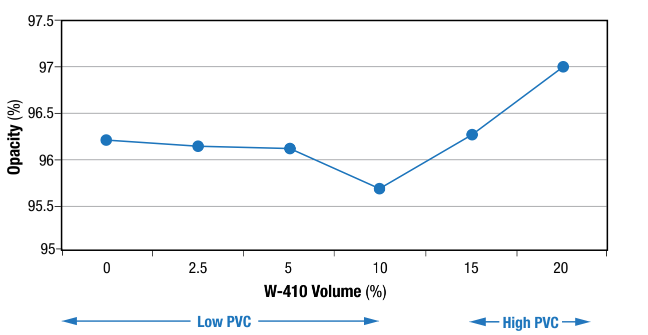


Figure 5 Effect of 3M™ Ceramic Microspheres W-410 on Opacity



Opacity

Opacity affects a paint's ability to conceal the previous coating, and is an indicator of how much paint will be needed to cover a specific area. Formulations containing 3M™ Ceramic Microspheres can be optimized to increase opacity or hiding power.

Figure 5 shows that opacity decreases slightly with the addition of ceramic microspheres W-410 at either 2.5 vol% or 5 vol% (formulas DG-2 and DG-3) in comparison with the control formulation. When a 10 vol% loading of ceramic microspheres W-410 reduced the amounts of other fillers (calcium carbonate, calcined kaolin, Minex®), opacity was negatively affected. As expected, opacity increased at high loadings of ceramic microspheres (15 and 20 vol%).

Conclusion

As shown by the tests performed for this study, the addition of 3M ceramic microspheres increases dispersibility, gloss control, washability, scrub and burnish resistance in a water-based flat architectural paint. These enhancements are made possible by the unique spherical shape and intrinsic hardness of 3M ceramic microspheres, which offer fine particle size, high packing density and a smooth, uniform paint surface finish. In this study, the best overall performance was obtained at 5 vol% 3M ceramic microspheres W-410 in an acrylic, water-based formulation with 40% PVC.

Addition levels of 10 – 20 vol% are possible if the major filler component is substituted with 3M ceramic microspheres, as is the case of formulas DG-5 and DG-6 in this study. In terms of opacity, these higher loadings (10 – 20 vol%) are favorable, even if other fillers are present in very low amounts. However, low to medium loadings of 3M ceramic microspheres W-410 slightly decrease opacity in comparison with the control formulation with a similar PVC.

By replacing some or the majority of other fillers with high loadings (15 – 20 vol%) of 3M ceramic microspheres W-410, it is possible to increase the PVC of an acrylic water-based formulation (i.e., from 40% to 49 – 59%) while reducing the amount of resin and still producing a higher-performance paint compared to the original formula. In this way, 3M ceramic microspheres can help paint manufacturers reduce costs with lower resin demand, enhance overall paint performance and deliver customer-pleasing functional properties.

Videos of these and other tests performed in the 3M Paints & Coatings Laboratory are available online at **www.3M.com/paintsandcoatings**. For specific recommendations on optimizing your formulations with 3M™ Ceramic Microspheres, contact your 3M technical service representative.

3M Center
St. Paul, MN 55144-1000
1-800-367-8905
www.3M.com/engineeredadditives

Please recycle. Printed in USA.

Issued: 9/13 9328HB
98-0212-4234-6