

Kevin Biller, President of ChemQuest - Powder Coating Research, discusses the use of hard minerals to enhance powder coating performance

Hard minerals enhance powder coating performance

Powder coatings continue to expand their share of the industrial coatings market mainly because of their high performance combined with excellent economics. The application of powder coatings can be highly efficient as the oversprayed material can be collected and recycled back into the finishing process. This minimises waste streams, which allows powder's environmental profile to align well with both corporate and regulatory sustainability goals.

Experts forecast robust growth of powder coatings. Global sales of powder coatings in 2021 are estimated to be around US\$12.5bn (€10.6bn) with a volume of approximately 5.5bn lbs. (2.5M tonnes). Growth is projected at 3.9 to 5.4% per annum for the next five years.

Abrasion resistance in powder coatings is an important property. Some powders are used to coat items that are subjected to extreme environments such as oil and gas transmission pipelines, whereas other powder coatings are used in decorative applications where appearance is critical. These decorative finishes must maintain their appearance after the handling of parts during assembly and everyday wear and tear.

Improving abrasion resistance is a sought-after goal of powder coating formulators, whether for functional or aesthetic purposes. The use of high hardness mineral fillers is considered one avenue to increase abrasion resistance in powder coatings. This study examines the effect that white fused alumina, nepheline syenite and silicon carbide have on the abrasion resistance of a common powder coating system based on polyester-HAA (hydroxy-alkyl amide) chemistry.

WESTER MINERALIEN

Wester Mineralien is an expert in the refinement of mineral grains. It offers intelligent solutions for abrasion-resistant, scratch-resistant and slip-resistant surface coatings, as well as blasting agents, abrasives, and polishing agents. Wester



has recently ventured into powder coating technology as a supplier of hard minerals to enhance powder coating performance.

Objective:

Evaluate functional fillers in an industry relevant powder coating formulation. Compare performance to a common industry filler and an unmodified control formulation. Evaluate for: processability, application performance, appearance (colour, gloss and surface profile), mechanical properties and abrasion resistance.

Materials:

Carboxyl polyester resin: Crylcoat 2689 – Acid Value: 34, Tg62° – allnex
 Hydroxy alkyl amide: Curing agent – Aalchem
 Polyacrylate flow agent: Estron Chemical
 Benzoin: Degassing agent – Aalchem
 Titanium dioxide pigment: TiPure R-960 – Venator
 Black manganese ferrite pigment: Lansco 8303 – DCL Corp.

Nepheline Syenite: Typical analysis	
	Percent by weight
Silicon Dioxide	60.20
Aluminium Oxide	23.60
Sodium Oxide	10.50
Potassium Oxide	4.80
Calcium Oxide	0.35
Iron Oxide	0.08
Magnesium Oxide	0.02
Loss on ignition	0.42

White Fused Alumina: Typical analysis	
	Percent by weight
Aluminium Oxide	99.78
Silicon Dioxide	10.50
Titanium Dioxide	0.006
Calcium Oxide	0.010
Iron Oxide	0.035
Sodium Oxide	0.200

Silicon Carbide: Typical analysis	
	Percent by weight
Silicon Carbide	98.00
Free Carbon	<0.30
Free Silicon	<0.80
Aluminium Oxide	<0.20
Silicon Dioxide	<1.30
Iron Oxide	<0.20

General Properties: Mineral Fillers			
	Nepheline Syenite	White Fused Alumina	Silicon Carbide
Grade	Minex 4	WFA F 500, WFA F 800	SiC F 500
Colour	White	White	Greyish black
Specific gravity	2.6	3.9	3.2
Mohs hardness	6.0	9.0	9.0-9.5
Particle morphology	Nodular-irregular	Angular-cubic	Angular
Median particle size (µm)	6.8	6.5 (WFA F 800) 12.8 (WFA F 500)	12.5
pH	10	7	7

Formulations									
	1	2	3	4	5	6	7	8	9
COOH Polyester	410.88	366.36	326.50	347.36	295.50	347.36	295.50	357.35	311.48
HAA	21.63	19.28	17.18	18.28	15.55	18.28	15.55	18.81	16.39
Polyacrylate	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Benzoin	2.50	2.50	2.50	2.50	2.50	2.50	2.50	2.50	2.50
TiO ₂	54.00	54.00	54.00	54.00	54.00	54.00	54.00	54.00	54.00
Black Mn-Fe	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00
Nepheline Syenite ¹		46.86	88.82						
WFA-F 500 ²				66.86	121.45				
WFA-F 800 ³						66.86	121.45		
SiC F 500 ⁴								56.34	104.63
	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00
Filler Volume %	0	5	10	5	10	5	10	5	10

1. Nepheline Syenite – 6.8µm, 2. White Fused Alumina – 12.8µm, 3. White Fused Alumina – 6.5µm, 4. Silicon Carbide – 6.8µm.

Nepheline Syenite: Minex 4 – D50: 6.8µm – Sibelco
 White Fused Alumina – F 500 – D50: 12.8µm – Wester Mineralien
 White Fused Alumina: F 800 – D50: 6.5µm – Wester Mineralien
 Silicon Carbide: F 500 – D50: 12.8µm – Wester Mineralien

Formulation platform:

Materials were evaluated in a grey standard durable Polyester-HAA powder coating. Fillers were incorporated at 5 and 10% by volume. Industry standard additives were incorporated at conventional concentrations. Pigmentation consisted of titanium dioxide tinted with an inorganic black pigment.

Processing conditions:

Premix: 8 seconds Vitamix @ low setting
 Extrusion: APV 19mm twin screw extruder
 Zone 1: 100°C
 Zone 2: 100°C
 Screw RPM: 500
 Torque: 45-55%
 Chill Rolls RPM: 18
 Grind: Strand Mill

Sieve: 140 mesh (106µm)
 Application: Nordson Encore LT manual spray gun
 Substrate: 75mm x 150mm Cold rolled steel test panels
 Film Thickness: 50 to 60µm
 Cure: 10min at 200°C

RESULTS:

Processability

All samples processed well using the conventional conditions outlined above. No issues were experienced with extrusion, pulverisation or electrostatic application.

Aesthetics

Gloss was measured at a 60° angle using a TQC Polygloss GL0030. Multiple measurements were taken in various locations across the surface of the finished coatings and then averaged. Gloss was reduced with the addition of all fillers evaluated. Gloss reduction was very similar with the incorporation of Nepheline Syenite, White Fused Alumina F 500 and the Silicon Carbide F 500. The White Fused Alumina F 800 material reduced gloss slightly less.

Surface profile was graded using PCI Smoothness Standards, a subjective visual scale which ranges from 1 (the most texture) to 10 (the smoothest). The addition of 5% of all fillers had essentially no effect on smoothness and the 10% addition of either White Fused Alumina F 500 or Silicon Carbide F 500 caused a slight increase in texture.

Rheology

Melt viscosity was ascertained using the pellet flow technique per ASTM D-4242. 1.0g pellets of each formulation were pressed, adhered to a test panel and baked at the standard curing condition (10min at 200°C metal temperature) at an incline of 65° from horizontal. Longer pellet flow correlates to a lower melt viscosity. Pellet flow was reduced incrementally with the addition of all fillers and correlated well with the surface profile observations.

Colour

Colour was affected versus the control with the addition of all fillers. Nepheline Syenite caused the least shift in colour whereas White Fused Alumina F 500 and White Fused Alumina F 800 caused a moderate shift. The Silicon Carbide F 500 material caused a significant shift in colour. The colour shift in all cases was mainly darker (lower “L” value) and was incrementally greater with higher concentrations of filler.

Mechanical properties

Impact resistance is a rapid deformation test which was performed per ASTM D-2794 using a Gardner Impact Tester. Panels were struck with the tester both on the film surface (direct impact) and from the uncoated side of the panel behind the film (reverse impact). Impact resistance was measured in inch-pounds of force. The largest amount of force applied which did not result in the coating becoming cracked was recorded in the tables below. Impact resistance was generally unaffected by the

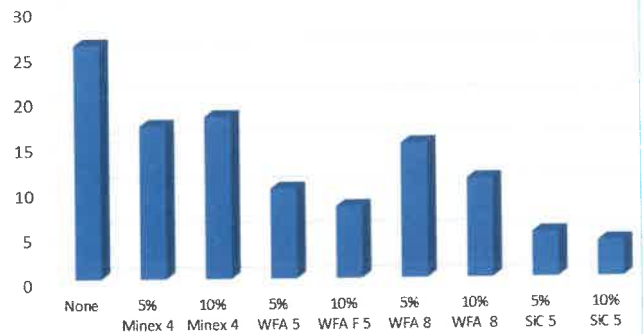
Gloss, Smoothness, Pellet Flow			
Modification vol %	Gloss (60°)	PCI Smoothness	Pellet flow (mm)
None	95.1	4	75
5% Nepheline Syenite	69.5	4	70
10% Nepheline Syenite	55.3	4	62
5% WFA F 500	69.6	4	70
10% WFA F 500	48.5	3	65
5% WFA F 800	72.4	4	70
10% WFA F 800	57.8	4	67
5% SiC F 500	67.1	4	70
10% SiC F 500	47.3	3	63

Colour				
Modification vol %	L*	a*	b*	ΔE (from control)
None	61.75	-0.30	-1.12	N/A
5% Nepheline Syenite	61.08	-0.19	-1.03	0.69
10% Nepheline Syenite	60.42	-0.07	-0.97	1.41
5% WFA F 500	59.78	-0.01	-1.14	2.05
10% WFA F 500	58.81	-0.01	-1.43	3.12
5% WFA F 800	59.32	0.06	-1.07	2.56
10% WFA F 800	58.62	0.05	-1.38	3.32
5% SiC F 500	58.29	-0.20	-1.08	3.47
10% SiC F 500	56.81	-0.36	-1.57	5.13

Abrasion Resistance – ASTM D4060

Modification Vol%	Initial Weight (g)	500 Cycles (g)	Coating Loss (g)	1000 Cycles (g)	Coating Loss (g)
None	44.540	44.529	0.011	44.514	0.026
5% Nepheline Syenite	44.995	44.988	0.007	44.978	0.017
10% Nepheline Syenite	44.897	44.889	0.008	44.879	0.018
5% WFA 5	44.746	44.741	0.005	44.736	0.010
10% WFA 5	44.877	44.874	0.003	44.869	0.008
5% WFA 8	44.738	44.731	0.007	44.723	0.015
10% WFA 8	44.599	44.594	0.005	44.588	0.011
5% SiC 5	44.393	44.391	0.002	44.388	0.005
10% SiC 5	44.309	44.307	0.002	44.305	0.004

Coating Loss (mg.) - 1000 Cycles ASTM D4060



WFA = White Fused Alumina, SiC = Silicon Carbide, 5= F 500, 8= F 800.

Impact Resistance

Modification vol %	Direct	Reverse
None	20	20
5% Nepheline Syenite	20	20
10% Nepheline Syenite	20	20
5% WFA F 500	20	20
10% WFA F 500	20	20
5% WFA F 800	20	20
10% WFA F 800	20	20
5% SiC F 500	20	20
10% SiC F 500	40	40

addition of all of the fillers. A slight increase in impact resistance was observed with a 10% addition of Silicon Carbide F 500. This may fall within the precision of the test method.

Abrasion resistance

Abrasion resistance was evaluated using the Taber Abrasion test method per ASTM D-4060 using CS-17 abrasive wheels. Coating loss was measured after 500 and 1000 cycles. The addition of all fillers provided a significant improvement in abrasion resistance versus the unmodified control. Best performance was observed with the addition of Silicon Carbide F 500 followed closely by White Fused Alumina F 500. Both White Fused Alumina F 800 and Nepheline Syenite provided improvements in abrasion resistance to a lesser degree.

DISCUSSION AND CONCLUSIONS

The primary goal of this study was to determine if the addition of "hard" mineral fillers such as white fused alumina, nepheline syenite or silicon carbide would increase the abrasion resistance of a powder coating. The addition of all fillers evaluated provided a significant improvement in abrasion resistance versus

the unmodified control. Best performance was observed with the addition of silicon carbide followed closely by the large particle size White Fused Alumina (WFA F 500). After 1000 cycles of Taber Abrasion per the ASTM D4060 Test Method, the control lost 26mg, whereas 10% Silicon Carbide (SiC 5) lost 4mg and White Fused Alumina (WFA F 500) lost 8mg.

Adding 10% of the smaller particle size fillers, White Fused Alumina (WFA F 800) and Nepheline Syenite (Minex 4) provided improvements in abrasion resistance to a lesser degree. The WFA F 500 lost 11mg and the Nepheline Syenite provided the least improvement at 18mg lost.

The addition of mineral fillers was expected to influence colour, gloss and smoothness of powder coating samples.

Colour was affected by the incorporation of all fillers. The 10% addition of Nepheline Syenite exhibited a modest effect on colour of around 1.50 ΔE versus the unmodified control. The White Fused Alumina materials added somewhat more colour with a ΔE of around 3.10 to 3.30. The Silicon Carbide material, black in colour, introduced the most colour versus the control with a 5.13 ΔE.

In general, the incorporation of larger particle size mineral fillers into powder coating formulations produces lower gloss. This trend was observed in this study as samples containing the larger particle size fillers exhibited measurably lower specular gloss. Specifically, the 10% by volume additions of both 12.8µm WFA-5 and 12-13µm SiC materials produced 60° gloss of around 48 gloss units, whereas the unmodified control exhibited a 95GU and the smaller particle size fillers, Nepheline Syenite and WFA-8 produced gloss around 55 to 60GU.

Surface profile was graded using PCI Smoothness Standards developed by the Powder Coating Institute. The addition of 5% of all fillers had essentially no effect on

smoothness and the 10% addition of either of the smaller particle size fillers, White Fused Alumina F 500 or Silicon Carbide F 500 caused a slight increase in texture.

Melt viscosity was ascertained using the pellet flow technique per ASTM D-4242. Longer pellet flow correlates to a lower melt viscosity. Pellet flow was reduced incrementally with the addition of all fillers indicating an increase in melt viscosity. These results correlated well with the surface profile observations.

Only minimal effect on impact resistance was observed with the addition of the mineral fillers evaluated. A slight increase in impact resistance was observed with a 10% addition of SiC F 500. This may fall within the precision of the test method.

In conclusion, the incorporation of hard mineral fillers, specifically silicon carbide and White Fused Alumina, provides a significant enhancement of the abrasion resistance of a powder coating. Type and concentration of mineral filler should be carefully evaluated in formulations to determine optimal performance.

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Information: For further information and samples, please contact Wester Mineralien GmbH through their website: www.wester-mineralien.de or by contacting their sales department at: +49 (228) 987 200. Wester Mineralien GmbH, Heerstraße 41, 53347 Alfter-Witterschlick, Germany.