CARBON BLACKS

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High-Color Carbon Blacks for High Performance Coating Applications

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Introduction

High jet masstone is a requirement of high-end performance coating applications such as automotive OEM or automotive refinish. The demand for increased jetness has heightened with the emergence of new resin technology. For this reason, raw material and dispersion equipment suppliers have looked for ways to improve their products. In this respect, Cabot Corporation is no exception and continues to supply the market with innovative ideas for black pigments that meet current market needs.
This technical paper presents a summary of information regarding the effect certain fundamental properties of carbon black may have on coatings performance.

It also outlines information on Cabot's core line of high-color carbon black pigments, including the well-established MONARCH 1300 and MONARCH 1400 carbon blacks. Also covered are two new pigments, MONARCH 1500 carbon black and the revolutionary EMPEROR 2000 carbon black. While MONARCH 1300, MONARCH 1400, and MONARCH 1500 carbon blacks are oxidized pigments, EMPEROR 2000 carbon black is based on Cabot's proprietary surface modification technology.
This brochure demonstrates the ability of EMPEROR 2000 carbon black to impart superior jetness in high performance coating applications. The test criteria used to collect and to compare performance data on these grades are highlighted in Section II of this brochure.

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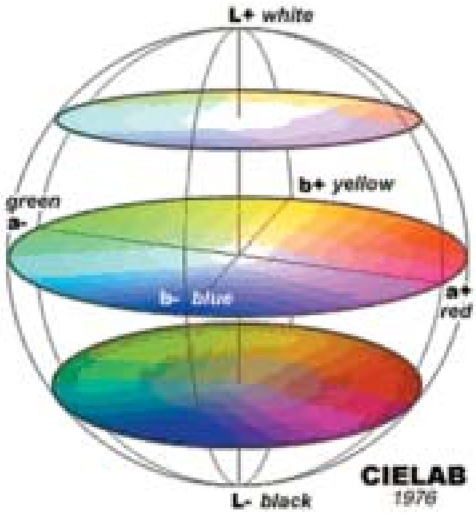
I. Carbon Black Properties and Coating Performance

One of the major reasons carbon black is used in coating applications is for its color properties. Color development can be measured by converting the color spectral reflectance data into three-dimensional color space, as provided by the Hunter Color formula measurements, as shown here.

In automotive topcoat applications, extreme black (low L* values) and deep blue undertones (low b* values) are the desired colors. The key properties of carbon black that can affect this color goal are:

1. Primary particle size/surface area
2. Primary aggregate shape/morphology
3. Surface chemistry

Hunter Color Definition

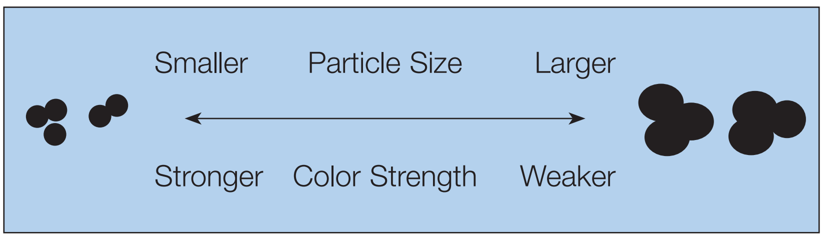


Effect of Primary Particle Size

Most pigments absorb and scatter portions of the visible light spectrum. The color of paint depends on how well the pigment is able to absorb and scatter light. Carbon black can be used to help increase the amount of light absorbed by a coating because it can absorb and scatter light more effectively than many other pigments.

As a carbon black's primary particle size decreases, more surface area becomes available to incident light.

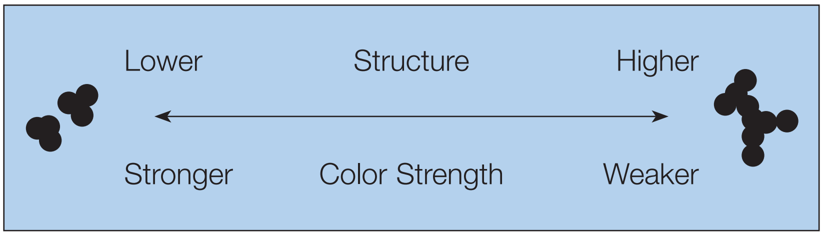
Also, primary aggregates tend to be smaller, assuming a constant structure level, resulting in a finer carbon black. The overall effect of the finer carbon black particle size is increased light absorption and more efficient light scattering, giving a blacker or "jetter" color.



Effect of Structure

A carbon black characterized by aggregates composed of many primary particles with considerable branching or chaining is referred to as a "high structure" black. Conversely, an aggregate having relatively few particles forming a more compact unit is a "low structure" black.

As structure increases, the absorption and scattering efficiencies are decreased. Thus, in primary particles of the same size, a high structure black will exhibit lower jetness than a black having low structure.



Effect of Surface Chemistry

Increasing the volatiles content (that is, chemisorbed oxygen complexes) on the surface of carbon black will generally increase dispersibility and lower viscosity in liquid systems. The dispersibility of carbon black can also be enhanced through the adsorption of a limited amount of moisture on its surface.

To some extent, both the volatiles content and adsorbed moisture can function as surfactants, whereby the surface is more readily "wetted" by the vehicle. However, as the volatiles content of carbon black increases, its surface becomes more acidic. Thus, care should be taken when formulating coatings systems.

Physical form

Most commercial high-color carbon blacks are available in either fluffy or pellet form. The pellet form handles more easily (reduced dusting) and normally costs less than the fluffy form. However, the process of densification, which is used to create the pellet form, tends to pack carbon black agglomerates more closely, making dispersion more difficult than with the fluffy form.

II. High-Color Carbon Blacks: Cabot MONARCH/ BLACK PEARLS 1300 & 1400, MONARCH 1500, and EMPEROR 2000 Carbon Blacks

Properties of an ideal high-color black for coatings

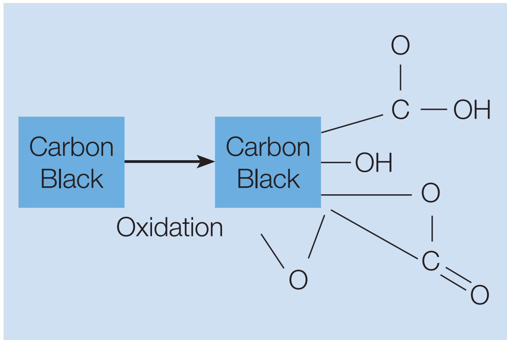
An ideal high-color carbon black should provide superior "jetness" in high-color enamels or lacquer coatings. As explained above, key properties of such a carbon black would include sufficiently fine-sized primary particles for increased light absorption and lower structure for increased light scattering.
The volatiles content and moisture level also can be adjusted to improve dispersibility. When these properties are obtained, superior "jetness" in high performance coating systems can be achieved.

MONARCH/BLACK PEARLS 1300 & 1400, MONARCH 1500 and EMPEROR 2000 Carbon Blacks

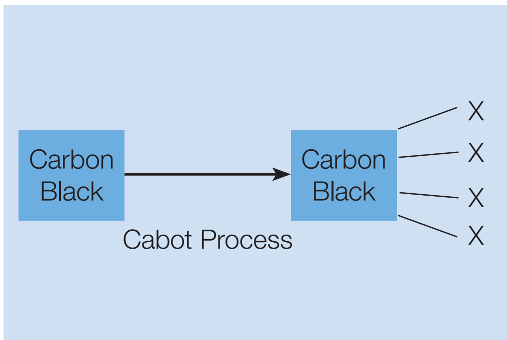
MONARCH/BLACK PEARLS 1300 & 1400, MONARCH 1500 and EMPEROR 2000 are commercially available Cabot highcolor carbon blacks. Because of their properties, Cabot believes that they are able to impart superior "jetness" for high performance coating applications. They exhibit a very fine primary particle size (9-13 nanometers) for increased light absorption combined with a low structure (90-100 DBP) for increased light scattering. Together, these properties help to achieve superior color "jetness" and improved gloss in high performance coating systems. Additionally, the specifications for volatiles content and moisture levels of these Cabot carbon blacks are optimized to help maintain dispersibility.
MONARCH/BLACK PEARLS 1300 & 1400, MONARCH 1500 are Cabot oxidized high-color carbon blacks. These high-color carbon blacks are produced through the process of chemisorption of oxygen complexes onto the carbon black surface.

EMPEROR 2000 carbon black is Cabot's newest pigment black and is produced using an innovative chemical modification technology. This patented technology allows specific functional groups to be grafted onto the surface of the carbon black particle. For EMPEROR 2000, the function of the attached groups is twofold: first, it helps to anchor adsorbed surfactants and dispersants that otherwise can float free of the carbon black surface and affect coating performance, and secondly, it improves the ease of dispersion and dispersion stability of the carbon black yielding the desired coating jetness with a strong blue undertone. The new technology is illustrated here.

Conventional Commercial Carbon Black After-treatments



Cabot's New Surface Treatment (Chemical Modification)



Performance Comparison Study

Cabot's MONARCH 1300, 1400 & 1500, and EMPEROR 2000 carbon blacks were first tested in high-solids automotive topcoat acrylic formulas for this comparison study. An in-house Cabot application development group developed the formulations based on high-solids acrylic polyol resin suitable for one-component thermoset coatings. The formulation also contained a reactive melamine formaldehyde crosslinker. The carbon black dispersing agent used was a solution of high molecular weight blocked copolymer with cationic activity. A mixture of aromatic and ester solvents was used to help control the viscosity of the coating formulation.

The above carbon blacks were evaluated also in water-borne acrylic latex automotive base-coat formulas. This formulation also was developed by our in-house applications development group. In addition to the acrylic resin, the formulation consisted of a melamine formaldehyde resin as a crosslinker, a non-ionic low molecular weight dispersing agent and, for film formation, a mixture of propylene glycol n-butyl ether and dipropylene glycol n-butyl ether solvents.

The formulations, performance properties and test results of both the high-solids acrylic and water-borne acrylic enamels are described in the sections A and B below. Additionally, accelerated weathering results are given for the water-borne acrylic enamel.

High-Solids Acrylic Enamel

Millbase Formulation:

	Amount (Parts)	Raw Material	Supplier
Butyl Acetate	17.5	Solvent	Eastman
DisperBYK 161 (30% N.V.)	32.5	Dispersion Aid	BYK-Chemie
Carbon Black	10.0	Pigment	Cabot Corp.
Setalux 27.1597 (80% N.V.)	40.0	Resin	Akzo Nobel

Butyl acetate, DisperBYK 161 and carbon black were premixed using good agitation to wet-out the pigment. Setalux 27.1597 was then added under good agitation. Once the resin was incorporated, the millbase was premixed at 4,000 RPM for 20 minutes. The millbase was then charged to a horizontal mill along with 0.6-0.8 mm zirconium silicate media. The millbase was ground at 10m/sec. tip-speed to achieve particle sizes of <5 microns on the Hegman Scale.

Letdown Masterbatch Formulation:

The following materials were mixed together under good agitation.

Setalux 27.1597	290.0	Resin	Akzo Nobel
Cymel 325 (80% N.V.)	110.0	Crosslinker	Cytec
Butyl Acetate	100.0	Solvent	Eastman

Finish masstone formulation:

To prepare the finish formulation, 10 parts of the millbase and 50 parts of the letdown masterbatch were added together using good agitation for approximately 20 minutes before application.

Finish Masstone Formulation Constants:

Carbon back loading in millbase (%)	10
Dispersant/Carbon black solids ratio	0.975/1.00
Pigment/Binder solids ratio	0.028/1.00
Crosslinker %, solids	25
Carbon black loading on total formulation (%)	1.67

Performance Properties of High-Solids Acrylic Enamel

Paint viscosities were adjusted with Aromatic 100 to 30 seconds with a No.4 Ford cup. The paints were then sprayed out with a 665SX66SD nozzle using air assist applied at 35 psi. The final coating film thickness and curing schedule were as follows:

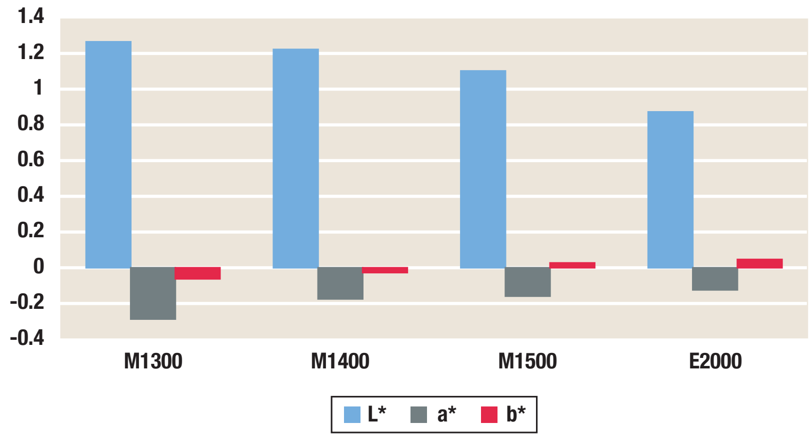
Monocoat Application:
Masstone

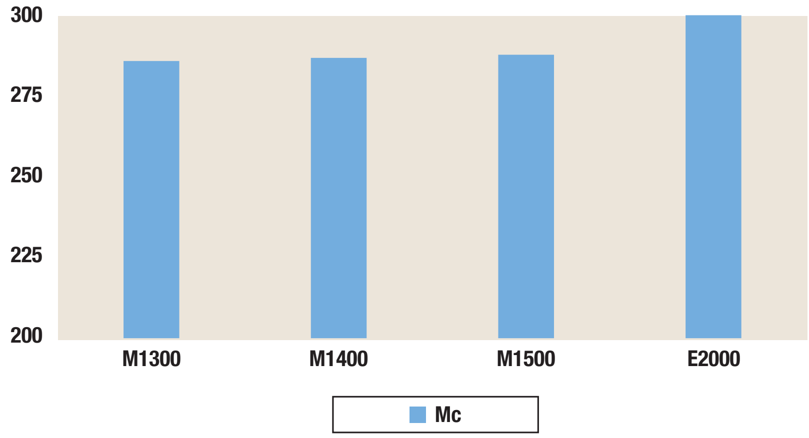
	DFT, mil	Flashoff Time	Bake time
Monocoat	1.0 - 0.02	20 min at 23°C	30 min at 140°C

After curing, the high-color pigment black panels were measured for color development and gloss. Color measurements were determined using a Hunter Labscan colorimeter using (45,0) geometry, CIELab equation, D-65 illuminant, and 10 degree observer. Gloss measurements were determined using BYK Gardner Glossmeter. Similar gloss readings were obtained for each of the carbon black formulations. The color development results are shown in the following table and in the charts below. The results illustrate the increased jetness achieved when using MONARCH 1500 and especially EMPEROR 2000 carbon blacks. Both L* values and Mc values are improved over those of MONARCH 1300 & 1400 carbon blacks.

Masstone

	MONARCH 1300	MONARCH 1400	MONARCH 1500	EMPEROR 2000
DFT†, mil/micron	1.0/25.0	1.0/25.0	1.0/25.0	1.0/25.0
Gloss- 20 Degree	82	82	83	84
Gloss- 60 Degree	91	91	90	90
Color Development †				
L*	1.29	1.22	1.12	0.9
a*	- 0.25	- 0.14	- 0.13	- 0.10
b*	- 0.06	- 0.02	0.03	0.06
Mc	288	289	291	300





Water-borne Acrylic Latex Enamel

Millbase Formulation:

The following materials were mixed together under good agitation.

	Amount (Parts)	Raw Material	Supplier
Water	71.6	Water	-
D-1441	8.9	Dispersion Aid	Baker Petrolite
Triton™ X-100	4.0	Surfactant	Dow Chemical
Nopco NS-1	2.0	Defoamer	Cognis
Carbon black	13.5	Carbon black	Cabot

The above materials were mixed together at 4,000 RPM for 20 minutes. The mixture was then charged to horizontal mill along with 0.6-0.8 mm zirconium silicate media and milled using 10m/sec. tip-speed to achieve particle sizes of <5 micron on the Hegman Scale. The millbase was then adjusted to a pH of 7.0-7.5 with ammonia prior to letdown.

Letdown Masterbatch Formulation:

A masterbatch letdown was prepared using the following procedure. First, materials 1-6 in the table below were mixed together using good agitation until a homogenous mixture was obtained. Next, this mixture was added to 1404 parts of NeoCryl XK-100 acrylic resin. Finally, a solution of 150 parts water and 4 parts ammonia (28 %) was added to the mixture, also using good agitation, to complete the masterbatch.

	Parts		
Cymel 373	116.5	Amino resin	Cytec
Arcosolv DPnB	56.2	Co-solvent	Lyondell
Arcosolv PnB	84.2	Co-solvent	Lyondell
Dehydran 1293	8.0	Defoamer	Cognis
BYK 348	4.0	Wetting agent	BYK-Chemie
BYK 024	2.0	Foam breaker	BYK-Chemie
Neocryl XK-100	1404.0	Acrylic resin	Neoresins

Finish masstone formulation:

To prepare the finish formulation, 20 parts of the millbase and 182.9 parts of the letdown masterbatch were added together using good agitation for approximately 20 minutes before application.

Finish tinting formulation (10/90):

To prepare the finish tinting formulation, 5 parts of the millbase concentrate formulation and 45 parts of the finish white base (TrueValue Weatherall 100% Acrylic Latex GHP-9 White) were add together under good agitation for approximately 20 minutes before application.

Formulation Constants:

Carbon back loading in millbase (%)	13.5
Dispersant/Carbon black solids ratio	0.25/1.00
Total solids by weight in final letdown (%)	35.0
Pigment/Binder solids ratio	0.04/1.00
Crosslinker %, solids	15
Carbon black loading on total formulation (%)	1.33

Performance Properties of Water-borne Acrylic Latex Enamel

Paints were sprayed out using a 665SX66SD nozzle with air assist applied at 35 psi. The final coating film thickness and curing schedule was as follows:

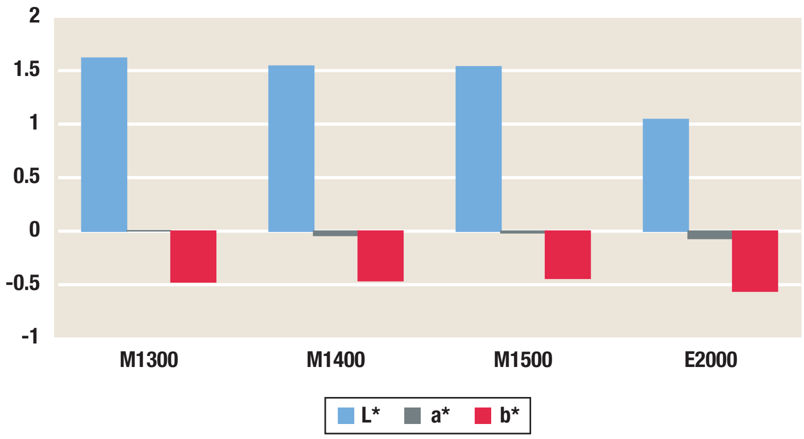
Monocoat Application:

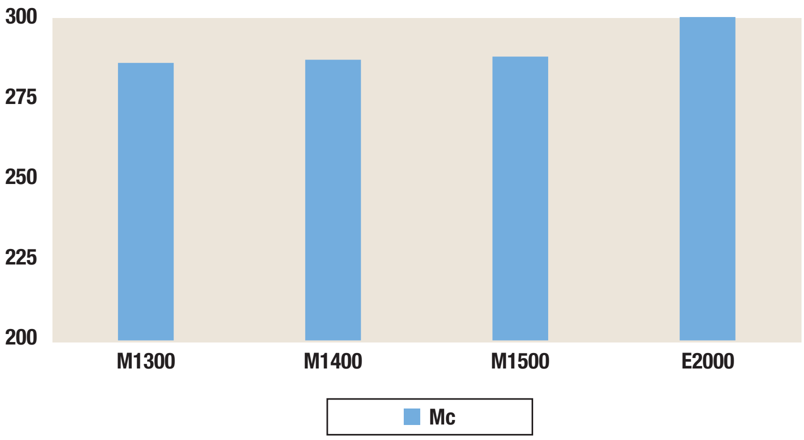
	DFT†, mil	Flashoff Time	Bake time
Monocoat	1.0 ± 0.02	5 min at 23°C	30 min at 140°C

After curing, high-color pigment black panels were measured for color development and gloss. Color measurements were determined using a Hunter Labscan colorimeter using (45,0) geometry, CIELab equation, D-65 illuminant, and 10 degree observer. Gloss measurements were determined using BYK Gardner Glossmeter. Similar gloss readings were obtained for each of the carbon black formulations. The color development results are shown in the following table and in the charts below.
The results illustrate the increased jetness achieved when using MONARCH 1500 and especially EMPEROR 2000 carbon blacks. Both L* values and Mc values are improved over those of MONARCH 1300 & 1400 carbon blacks. Further, EMPEROR 2000 exhibits better blue tone than all other carbon blacks used in this formulation.

Masstone

	MONARCH 1300	MONARCH 1400	MONARCH 1500	EMPEROR 2000
DFT†, mil/micron	1.0/25.0	1.0/25.0	1.0/25.0	1.0/25.0
Gloss- 20 Degree	82	82	83	84
Gloss- 60 Degree	91	91	90	90
Color Development †				
L*	1.51	1.41	1.43	0.76
a*	0.09	0.42	0	0.08
b*	- 0.46	- 0.43	- 0.44	- 0.53
Mc	284	285	287	321

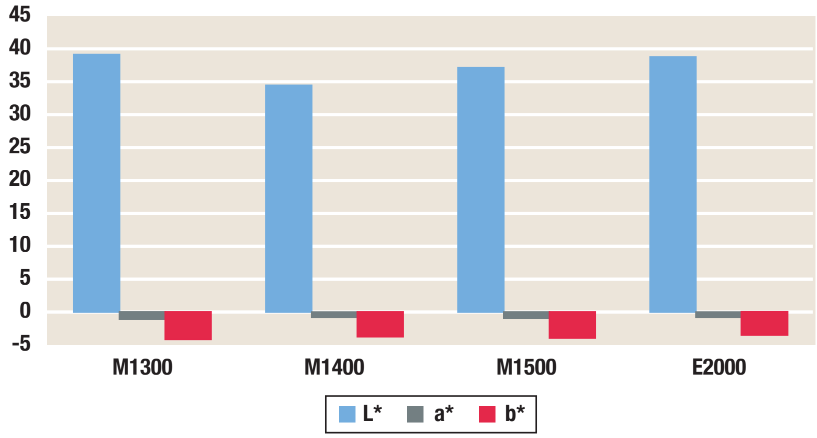




Tinting

	MONARCH 1300	MONARCH 1400	MONARCH 1500	EMPEROR 2000
DFT†, mil/micron	1.0/25.0	1.0/25.0	1.0/25.0	1.0/25.0
Color Development †				
L*	39.37	39.23	40.55	37.49
a*	- 1.4	-1.08	- 1.18	- 1.01
b*	- 3.85	- 3.5	- 3.75	- 3.4

Accelerated weathering was performed with a QUV tester. The test was based on the ASTM 4587-91 method using UV-B fluorescent lamps. The tester was set at 4 light cycles followed by 4 condensation cycles. After 1000 hours of exposure, the panels were removed for evaluation. Weathering results are shown in the table below. Similar results are obtained for MONARCH 1300, 1400 and 1500, while EMPEROR 2000 showed improved resistance to whitening and color retention.



Hours-1000	MONARCH 1300	MONARCH 1400	MONARCH 1500	EMPEROR 2000
Chalking	No effect	No effect	No effect	No effect
Blistering	No effect	No effect	No effect	No effect
Whitening	Moderate	Moderate	Moderate	Slight
Color Development †				
L*, initial/final	1.63/3.19	1.47/2.13	1.42/2.98	1.27/1.38
a*, initial/final	- 0.18/- 0.34	- 0.30/- 0.32	- 0.11/- 0.19	- 0.19/- 0.15
b*, initial/final	- 0.29/- 1.13	- 0.18/- 1.08	- 0.18/- 0.68	- 0.35/- 0.84
Gloss retention, %	100	100	96.8	100

III. Formulation Guide

The following modifiers were also used in our evaluations of carbon black color performance:

Dispersion Agent

Since high-color blacks consist of small particle sizes, high surface areas, and high structures, dispersion and stability are much more difficult to achieve than with other carbon black grades. Dispersion agents are highly recommended for dispersion and stability of high-color blacks in coating systems.
The following dispersion agents and usage levels were used in our evaluation:

For Solvent-Borne Systems:

Dispersion Agent	Amount (parts per 1 part carbon black)
DisperBYK 161	0.75 -1.00
DisperBYK 2000	0.50 - 0.75

Or a combination of Solsperse 32500 (0.50 - 0.60) and Solsperse 5000 (0.10 - 0.20 parts per 1 part carbon black) may be used.

For Water-Borne Systems:

Dispersion Agent	Amount (parts per 1 part carbon black)
TEGO Dispers 760W	0.40 - 0.60
D-1441	0.20 - 0.40

Surfactant

Surfactant addition is also required in order to increase the dispersibility and stability of water-borne formulations. The following surfactants were used in our study of the water-borne acrylic latex system:

Surfactant	Amount (parts per 1 part carbon black)
Triton™ X-100	0.3
AMP-95	0.3

Leveling Agent

Since substrates can be difficult to wet-out, especially with water-borne coatings, wetting agents are required for waterborne formulations so that good, continuous film formation may be achieved. Wetting (or leveling) agents can also eliminate film defects, such as orange peel, cratering or shrinkage. The following leveling agents were used in our comparison study:

For Solvent-Borne Systems:

Leveling Agent	Amount (wt. % of total wt. Finish formulation)
BYK 358	0.3 - 0.6

For Water-Borne Systems:

Leveling Agent	Amount (wt. % of total wt. Finish formulation)
BYK 346	0.3 - 0.6
BYK 348	0.2 - 0.4
Efka 3570	0.2 - 0.4

Defoamer

Foaming in water-borne formulations is extremely difficult to control. Adding defoamer is a must to minimize the foaming effect in these systems. The following defoamers were used in our evaluation of water-borne acrylic latex systems:

Defoamer	Amount (wt. % of total wt. Finish formulation)
Dehydran 1293	0.3 - 0.5
Dehydran 1620	0.3 - 0.5
Nopco NS-1	0.1 - 0.2
BYK 024	0.1 - 0.2

Dispersion Process

Optimum dispersion of carbon black is necessary in order to achieve superior color development in coatings formulations. All agglomerates must be broken down to primary aggregates to realize the full potential of the optically functional units. Any degree of dispersion less than the optimum will result in poorer jetness. The energy required for optimum dispersion must be supplied in the form of some type of media mill.

Definitions:

DFT = Dry Film Thickness

L* is a measure of the lightness/darkness (lower numbers indicate darker color)

b*is a measure of blue/yellow (lower numbers indicate bluer color)

a* is a measure of red/green (lower numbers indicate a greener color) Mc: Mc is the Color Dependent Black Value and was developed by K. Lippok-Lohmer (Farbe + Lack, (1986), vol. 92, p. 1024). It is defined by the equation Mc = 100[log(Xn/X) - log (Zn/Z) + log (Yn/Y)], where X, Y, and Z are measured tristimulus values. The Mc value correlates well with the human perception of increased jetness. As the Mc value increases, the jetness of the masstone increases.

Raw Material Suppliers*

Setalux™ 27.1597	Akzo Nobel 800.292.2308 www.akzonobel.com
Solsperse® 5000 Solsperse® 32500	Avecia 704.672.9920 www.aveciaadditives.com
BYK® 024 BYK® 346 BYK® 348 BYK® 358 DisperBYK® 161 DisperBYK® 180 DisperBYK® 2000	BYK-Chemie 203.265.2086 www.byk-chemie.com
Triton™ X-100 AMP-95™	Dow Chemical 800.447.4369 www.dow.com
D-1441	Baker Petrolite 781.335.6668 www.bakerhughes.com
Dehydran® 1293 Dehydran® 1620 Nopco® NS-1	Cognis 215.628.1000 www.cognis.com
Cymel® 202 Cymel® 373	Cytec Industries, Inc. 847.652.6013 www.cytec.com
EFKA® 3570	EFKA Additives 440.943.4200 www.efka.com
Arcosolve™ PnB Arcosolve™ DPnB	Lyondell 888-777-0232 www.lyondell.com
NeoCryl™ XK-100	NeoResins (Avecia) 978.658.6600 www.neoresins.com
TEGO Dispers™ 760W	TEGO Chemie 800.446.1809 www.additiveweb.com

** All raw materials used were North American versions.*

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