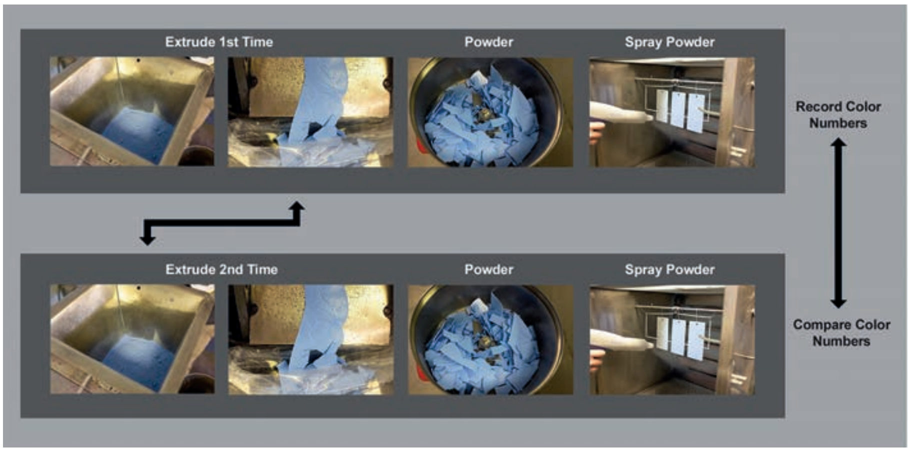
Mark Ryan, The Shepherd Color Co, discusses the dispersion of Complex Inorganic Colour Pigments (CICPs) in powder coatings

Controlling colour with high-performance inorganic pigments

The previous article (*PPCJ*, April 2020, p24) discussed the general properties of the class of high-performance pigments known as Complex Inorganic Colour Pigments (CICPs). These pigments are known for their outstanding inertness and resistance to chemicals, migration and weathering. They also have some inherent colouring properties advantageous for powder coatings. The control of colour in powder coatings depends on the predictability of the dispersion of the coloured pigments. CICPs have dispersion properties that produce colour that is controllable and stable. CICPs give controllable colour because the relatively higher use-rates as compared to organic pigments allow more advantageous mixing ratios. This can be demonstrated in the tinting of near white colours. Stability is beneficial if the powder coating needs to be re-extruded to correct for other colourants performance. The CICPs will not develop as much colour as standard organic pigments. Stability can be demonstrated by comparing a light blue tint colour made with either standard phthalo blue (PBl15.1), or Cobalt Blue (PBl28).

Dispersion of colour pigments in powder coatings is similar to that of dry pigment powders in plastics compounding. Dry pigment is added to resin in powder or pellet form and the mixture (along with other additives) is extruded. Because the powder coating resin systems are usually thermoset and not thermoplastic like common plastic colour compounding (PVC, PE, PP), the temperature setting must be carefully controlled and timed to allow the plastic to liquify and incorporate the pigments but not to exceed the temperatures and times needed to cure (crosslink) the resin systems. This leads to rather short residence times for the pigment incorporation time compared to thermoplastic compounding.

When comparing powder coating pigment dispersion to liquid coatings dispersion, the high-shear and amount of energy that can be put into a liquid coating in a small media mill is sufficient to not only disperse the pigment but to also reduce the pigment particle size. Powder coating dispersion has high enough shear to disperse the pigment but not reduce the primary pigment particle size. CICPs are viewed by some as having the reputation of being 'hard to grind' in liquid coatings. This can stem from the fact that their high specific gravity makes keeping them suspended difficult in low viscosity systems (not an issue in powder coatings) and setting the colour standard to require the grinding step to go past de-agglomeration and de-aggregation to actually reducing the pigment particle size, which is very energy intensive to achieve and stabilise.



Explanation of testing procedure

Since the pigment dispersion step in powder coating production is so short, experiments were run to compare the colour development and controllability of CICPs versus organic pigments. In both experiments that were run, the raw materials were blended together and extruded in a 19mm twin screw extruder. Part of the batch was then completed while a portion was re-extruded and then completed. The panels from the second run were compared with the initial run as standard.

The formula used for the two coatings is shown in **Table 1**. One thing to note is the colouring strength difference between the inorganic CICP and the organic phthalo blue used to make this light blue. **Table 2** shows the absolute colour numbers for the first run through the extruder and the deltas for the panels from the second run through the extruder. The CICP PBl28 (Cobalt Blue), known commercially as Shepherd Blue 385, shows very little colour change from the first to the second run. The organic phthalo blue tint shows some difference in colour (DE=0.26), mainly in the increase in strength (chromaticy and b* value).

BLUE	Polyester Resin	438.03	438.03
	TGIC	32.97	32.97
	Flow additive	6.00	6.00
	Benzoin	3.00	3.00
	Blanc Fixe Micro	60.00	60.00
	Ti02	48.00	59.00
	PBl28 Cobalt Blue	12.00	
	PBl15:1 Phthalo Blue		1.00
		600.00	600.00

Table 1. Light blue tint polyester/TGIC formula

While a DE=0.26 may not at first seem like a large difference, it largely depends on the tolerance that you are working with (**Figure 1**). If your first shot isn't right to match a colour and you need to correct it, that colour shift may be half or a quarter of the colour specification. More importantly,

Table 2. Colour shift from re-extrusions for blue tints

Blue Tint with TiO2	L	a	b	C	h	DL	Da	Db	DC	Dh	DE
PBl28 Cobalt Blue	75.16	-8.48	-25.09	26.48	251.32	-0.04	-0.02	-0.06	0.06	0.00	0.08
PBl15:1 Phthalo Blue	70.49	-16.59	-30.48	34.7	241.44	-0.02	-0.03	-0.26	0.24	0.10	0.26

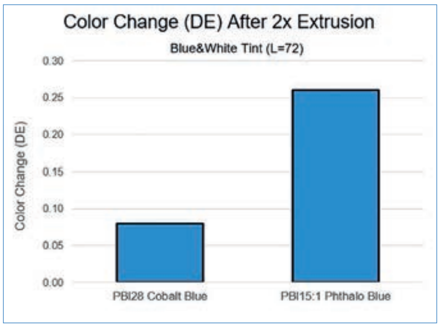


Figure 1.

the colour shift would have to be compensated for in any kind of correction. Also, depending on the colour difference, the addition/correction of colourant to the material could be extremely small and difficult to evenly add.

The colour strength and dosing for correct colour is even more apparent when trying to tint and tone near-white colours. Most powder coatings if made with straight TiO_2, even ones specially engineered for a blue undertone, will have a yellow cast to them due to the yellowness inherent in the resin. Toning the whites to appear 'whiter' is often done with small amounts of violet. The addition of violet doesn't actually make the white brighter but it reduces the positive a* value to a more neutral value. The closer to 0,0 in the a*,b* values, the neutral and 'whiter' the colour.

Two ways were examined to affect this colour change in a TGIC-free polyester coating. One used organic PV23 (Carbazole violet) and the other the inorganic PV16 (Manganese phosphate) known commercially as Shepherd Violet 11. The formulas used are seen in **Table 3**. As seen with the blue experiment, the organic pigment is much stronger than the inorganic pigment. So much so that the pre-blend of the PV23 and TiO_2 had to be made at a ratio of 59:1 (TiO_2 to PV23) and then that was used as a colourant.

The colour results from these formulas can be seen in **Table 4**. As in the blue experiment, the material was run through the extruder and then run again so that three extrusions of the material were made, to see how processing affected colour. Instead of the first extrusion being used as a standard, a formula with no violet and only TiO_2 was used as the standard. The deltas to this straight white are shown in **Table 4**.

The results show that very little violet is needed to improve the appearance of the standard white, which has a notable cast with a b* value of 2.0. Initial violet levels used were too high and were cut in half to get closer to the neutral values of 0,0 (a*,b*). The PV16 needed to be used at a level of 1/200 (0.5%) based on TiO_2, while the stronger organic PV23 needed to be used at a level of 1/12,500 (c. 0.017%). This very low use rate necessitated the use of a pre-blend.

The colour shift from the multiple extrusions is interesting because the colour shift is similar for both violets. This implies that the resin is adding more colour to the system with each extrusion pass and the violets are being 'dragged' more yellow (see **Figure 2**). While the a* and b* values are more neutral, the L* value is dropping. Anytime an absorbing pigment is added to a broadly scattering pigment, the reflectance goes lower, here in the wavelengths associated with yellow because of the absorbing pigments violet tone.

The key is that the inorganic PV16 manganese phosphate doesn't require the additional step of making a pre-blend mix. Dosing such a small amount of organic PV23 (0.03 on a straight pigment basis) into that quantity of white would be problematic (See **Figure 2**).

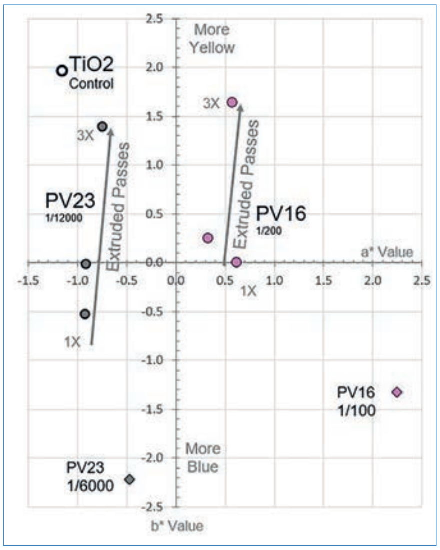


Figure 2. Toning whites with violet

These experiments have shown the benefits to using high-performance CICPs due to their colour dispersion stability and controllability. While CICPs may not be used in every powder coating formulation, they provide a tool box of 'problem-solving' pigments. While this article focused on colour dispersion properties of the CICPs, their largest use is in outdoor coatings that need the ultimate in weathering resistance. Shepherd Color has a long history of CICPs in durable coatings and the next article will cover weathering results from high durability powder coatings.

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	Control White	1/100 PV16	1/6000 PV23	1/200 PV16	1/12000 PV23
Polyester	325.38	325.38	325.38	325.38	325.38
HAA	17.13	17.13	17.13	17.13	17.13
Flow aid	5.00	5.00	5.00	5.00	5.00
Benzoin	2.50	2.50	2.50	2.50	2.50
TiO2	150.00	148.50	148.50	149.25	149.25
PV16 Manganese Violet		1.50		0.75	
PV23 9:1 Wh:VL Pre-mix					
PV23 59:1 Wh:VL Pre-mix			1.50		0.75
	500.0	500.0	500.0	500.0	500.0

Table 3. Above. Toning of TGIC whites with violet

	Times Extruded	L*(DL*)	a*(Da*)	b*(Db*)
Control TiO2		96.7	-1.2	2.0
PV16 1/100	1x	-3.3	3.4	-3.3
PV16 1/200	1x	-2.1	1.8	-2.0
PV16 1/200	2x	-2.5	1.5	-1.7
PV16 1/200	3x	-2.5	1.7	-0.3
PV23 1/6000	1x	-2.9	0.7	-4.2
PV23 1/12000	1x	-1.8	0.2	-2.5
PV23 1/12000	2x	-1.9	0.2	-2.0
PV23 1/12000	3x	-2.2	0.4	-0.6

Table 4. Left. Colour results for toning of whites

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