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Evaluating pigment dispersion for better color in plastics

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Scanning electron micrography and image analysis assess coloration mixing quality during polymer resin extrusion.

Color is a key appearance characteristic of plastics. To produce plastics with the desired output colors, compounders blend polymer resins with color pigments in parts per hundred parts of resins during extrusion. A minute deviation in the output color could cause an entire production lot to be rejected, with high wastage and delays.[1,2] During compounding, both dispersive and distributive mixing processes generally take place simultaneously. The former breaks agglomerates up into the primary particle size, whereas the latter process increases the randomness of the spatial distribution of pigments within the polymer matrix without any further change in their size.[3] Inadequate mixing of color pigments in a polymer matrix, whether dispersive or distributive, raises color mismatch issues. Possible causes of inadequate pigment mixing and color deviations include variations in color formulation,[4] processing parameters,[1] degradation behavior, primary particle size and spread, variable refractive index regions,[2] and the effect of the processing aides on rheological properties.[5]

Various techniques have been used to investigate factors that affect color in plastics.[4,6,7] We have successfully employed environmental scanning electron microscopy (ESEM) and image analysis to analyze pigment dispersion in a high-chroma polycarbonate grade. These techniques have not previously been reported as used for this purpose. (Gunde and coworkers used a similar technique but for powder-coated, plasma-etched samples.[8])

The polycarbonate was compounded under variable processing conditions on a technology line of SABIC IP at Cobourg Plant, Ontario, Canada. We took thin sections from injection-molded plaques, imaged them with ESEM (FEI Quanta FEG 250), and analyzed the images obtained. Knowing the optimal settings for processing variables such as temperature, feed rate, and screw speed is extremely important for obtaining the desired output color. However, transparent polymer resins do not permit scattering of incident light, and achieving the desired opacity, brightness, and whiteness requires blending with white pigments (mostly titanium dioxide).[2] Optical theory suggests the most efficient scattering occurs when the pigment particle size is slightly less than half the wavelength of incident light. The visible spectrum wavelength range (0.4–0.7μm) suggests a particle size of 0.2–0.35μm will optimize scattering.[9–13]

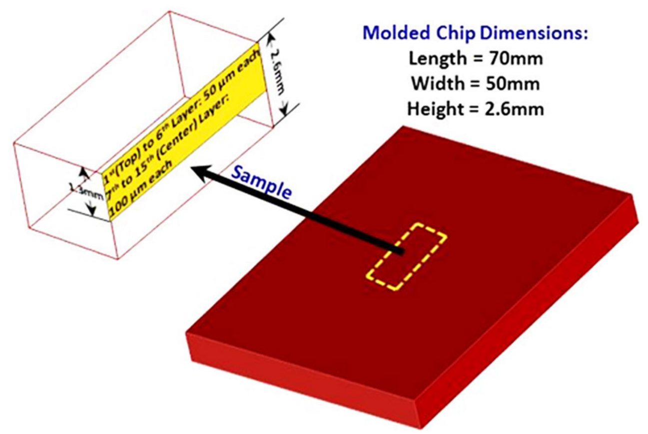

Figure 1. Schematic of molded plaque and location of thin sections.

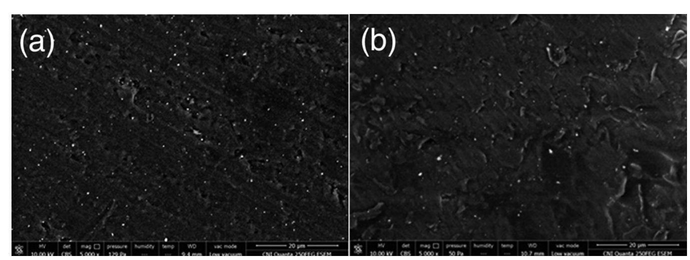

Figure 2. *(a) Top-layer (that is, of the first thin section cut from the sample chips) environmental scanning electron microscopy (ESEM) image from a run 7 sample chip at 5000× magnification. (b) Top-layer ESEM image from a run 17 sample chip at 5000× magnification.*

To evaluate pigment dispersion, we chose molded plaques from two experimental runs (specifically, runs 7 and 17), executed at 240°C and 300°C, respectively, fixing the screw speed at 900rpm and feed

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rate at 23kg/h. Polycarbonate resins are more sensitive to temperature than to the shear rate and, although their rheology is non-Newtonian, they display very low shear-thinning behavior (that is, their viscosity decreases only a little as the shear rate increases) compared with other thermoplastics.[14] We measured the color of the molded plaques on a spectrophotometer (X-Rite CE7000) and used a rotary microtome (SLEE CUT 6062) to cut each plaque into 15 thin sections (see Figure 1). We processed the ESEM images with ImageJ®software to evaluate pigment dispersion (see Figure 2).[15]

The particle size distribution graphs shown in Figure 3 and the interparticle distance distributions shown in Figure 4 clearly indicate that, compared with run 7, run 17 contains more agglomerates exceeding 0.5μm in diameter, fewer particles of diameter 0.2–0.35μm, and a lower spatial randomness of particles. (Randomness is measured in terms of weighted average of nearest neighbour distance among particles as reflected in Figure 4.) These characteristics reduced the scattering power of the pigments and eventually negatively affected L*,[16] the brightness axis in the International Commission on Illumination (CIE) L*a*b* color space, where a* and b* are the green/red axis and blue/yellow axis, respectively. Figure 5 shows the color data in CIE L*a*b* color space, which clearly indicates run 7 stands closer to the reference (the target color) than run 17. We attribute this to better dispersion of pigments in the polymer matrix.

In summary, we have shown that our technique is useful for detecting small differences in pigment particle size and spatial distributions in a polycarbonate resin grade. The technique correlates these with process variables regarding color deviation and can further be employed as a tool to optimize compounding process for plastics coloration. We also

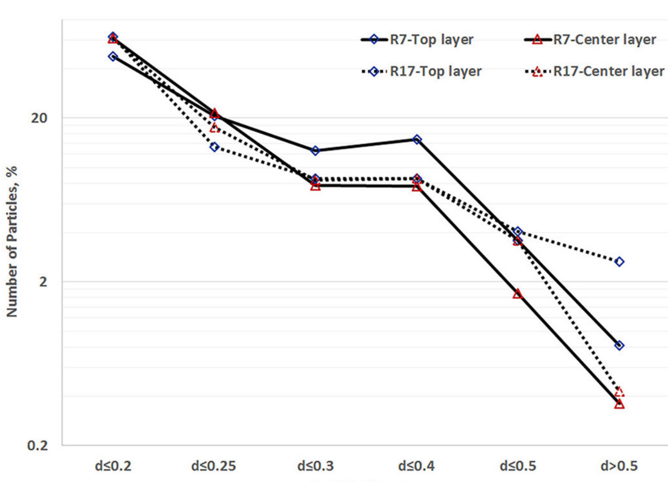

Figure 3. *Plots of particle size (diameter, d) distribution for different layers and runs. R7, R17: Data from run 7 and run 17, respectively.*

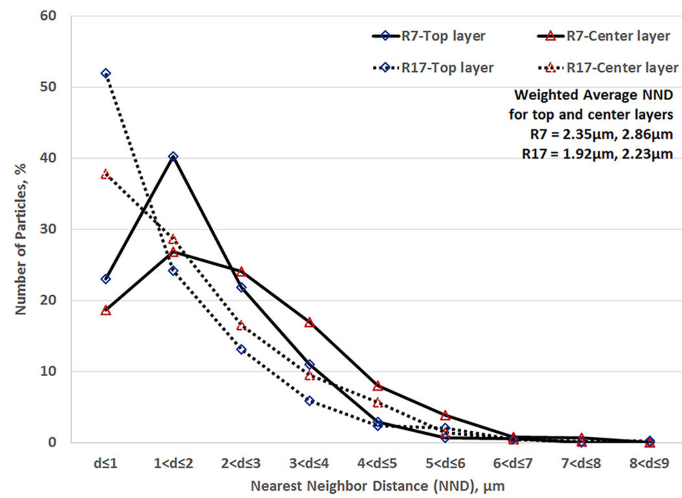

Figure 4. *Plots of nearest neighbour distance for different layers and runs.*

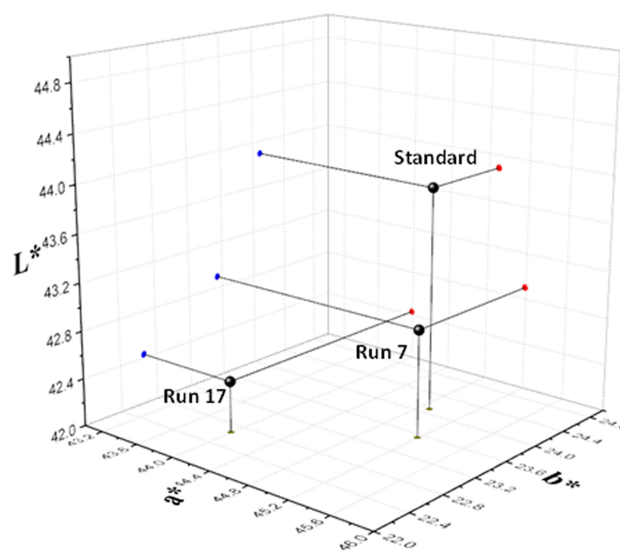

Figure 5. *Sample color data and the standard reference in International Commission Illumination (CIE) L*a*b* color space, where L* is the illumination axis, and a* and b* the green/red and blue/yellow axes, respectively.*

plan to examine the effect on pigment dispersion of other processing conditions, such as the screw speed and feed rate, so that we can determine an optimal set of process variables for consistent plastics coloration.

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References

1. D. Ross, M. Rabbani, S. Ahmed, J. Alsadi, and G. Rizvi, *A study on effects of various parameters on colours during compounding of plastics*, **Proc. ANTEC**, pp. 427–433, 2011.
2. B. Mulholland, *Effect of additives on the color & appearance of plastics*, **Proc. ANTEC**, pp. 1334–1340, 2007.
3. J. W. Ess and P.R. Hornsby, *Characterisation of distributive mixing in thermoplastics compositions*, **Polym. Test. 6**, pp. 205–218, 1986.
4. L. Klebanov and M. Kosarzycki, *A technique comparison for the quantification of color concentrate in a polyacetal component* 2008.
5. E. C. Achilleos *et al.*, *Role of processing aids in the extrusion of molten polymers*, **J. Vinyl Add. Technol. 8** (1), pp. 7–24, 2002.
6. I. Groves, T. Lever, and N. Hawkins, *Determination of carbon black pigment in nylon 66 by TGA*. Thermal Analysis Application Brief Number TA-122, TA Instruments Ltd UK. http://www.tainstruments.co.jp/application/pdf/Thermal_Library/Applications_Briefs/TA122.PDF
7. D. Grewell, G. Srinivasan, M.R. Kessler, and N. Kieffer, *Case study of color variation of thermal formed automotive body components*, **Proc. ANTEC**, pp. 1763–1767, 2008.
8. M. K. Gunde, M. Kunaver, M. Mozetic, and A. Hrovat, *Method for the evaluation of the degree of pigment dispersion in powder coatings*, **Powder Technol. 148**, pp. 64–66, 2004.
9. R. M. Christie, *Pigments, dyes and fluorescent brightening agents for plastics: an overview*, **Polym. Int'l 34**, pp. 351–361, 1994.
10. Technical information on DuPont™ Ti-Pure® titanium dioxide http://www2.dupont.com/Titanium_Technologies/en_US/index.html Accessed 1 April, 2015.
11. R. Levinson, P. Berdahl, and H. Akbari, *Solar spectral optical properties of pigments—Part II: survey of common colorants*, **Solar Energy Mater. Solar Cells 89** (4), pp. 351–389, 2005.
12. B. R. Palmer, P. Stamatakis, C. G. Bohren, and G. C. Salzman, **J. Coat. Technol. 61**, pp. 41–47, 1989.
13. E. S. Thiele and R. H. French, *Computation of light scattering by anisotropic spheres of rutile titania*, **Adv. Mater. 10**, pp. 1271–1276, 1998. doi:10.1002/(SICI)1521-4095(199810)10:15¡1271::AID-ADMA1271¿3.0.CO;2-
14. J. M. Margolis, **Engineering Plastics Handbook**, McGraw-Hill, 2006.
15. ImageJ, *Image Processing and Analysis in Java*, Research Services Branch, NIH 2014, http://rsb.info.nih.gov/ij/
16. A. R. Robertson. *Colorimetry – Part 4: CIE 1976 L* a* b* Colour Space*. CIE DS 014-4.3/E. Vienna, Austria: CIE Central Bureau, 2007