In-line color measurement of molten thermoplastics during extrusion via a fiber-optics spectrophotometer

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Abstract

The measurement of color in molten plastics has been investigated recently by few researchers. In 1995, Calidonio(1) conducted several experiments of pigmented polyethylene during extrusion. Refinement of this work was presented at the SPE ANTEC meeting of 1996(2)(3). In 1999, Gilmour(4) expanded the previous work by utilizing a low cost Charge Coupled Device Spectrometer. Most recently, Rom-Roginski(5) reported additional work on in-line color measurement using a CCD on several color changeover directions. Despite the above work, limitations in technology have prevented a comprehensive and accurate study of in-line color measurement. This paper presents a breakthrough in technology that allows in-line color measurement in thermoplastic compounding with the same (or better) degree of accuracy than a top-of-the-line laboratory spectrophotometer.

Introduction

Recent development and refinement of fiber-optics spectroscopy have made possible in-line color measurement in thermoplastics' extrudate without the problems associated in the past with high-frequency random noise, variations in response due to lamp-related problems, and the ability of the instrument to discern color over long periods of time.

Equipment

The Equitech Reflection Polymer Melt Probe (RPMP) (figure 1) consists of seven illuminating fibers evenly spaced around the probe circumference. Illumination fibers are angled at 28 degrees from the surface normal. The reflected light is picked up by a single fiber at 0 degrees from the normal. The RPMP optical window is a sapphire hemisphere sealed into titanium housing. The RPMP is capable of withstanding operating temperatures up to 370° C and pressures up to 15,000 PSI (1035 bar). The RPMP effectively solves the problems of high temperatures, pressures, and high-frequency electrical noise found in extruders. The probe is connected via fiber optics to the Equispec™ OCS (online color spectrophotometer) Analyzer that continuously corrects for variations in lamp response and spectrometer drift allowing the instrument to discern small color shifts over long periods of time.

Figure 1. Equitech's Reflection Polymer Melt Probe

The Equispec™ OCS includes a Xe arc flash lamp, an Equitech M3 multi-channel spectrometer, and a control computer. Light from the Xe lamp is divided into two fiber optic bundles, the first serves as a reference channel and the second illuminates the RPMP. The M3 spectrometer is typically configured with four fiber optic inputs. In these experiments only two inputs were used. Channel-1 is connected via a jumper fiber to the reference channel from the Xe lamp. Channel-3 is connected to the RPMP detection fiber. Channels 2 and 4 were not used. Flash lamp triggering, exposure timing, exposure averaging and multi channel CCD readout are controlled by a DSP built into the M3 spectrometer. The simultaneous intensity spectra from channels 1&3 of the M3 are first corrected for dark current and optical crosstalk in the control computer before they are used to generate a reflectance spectrum. The reflectance spectrum is then processed in the usual way to calculate L*, a* and b*.

Figure 2. Equispec™ OCS (Online Color Spectrophotometer)

Experimental & Results

Color (L*, a*, b*) of several thermoplastic resins (ABS, PE) was measured during color compounding operations and a color changeover utilizing the Equispec™ OCS (online color spectrophotometer) Analyzer and a Reflection Polymer Melt Probe (RPMP) inserted in a Dynisco port located in the extruder tooling just before the die as shown in the picture.

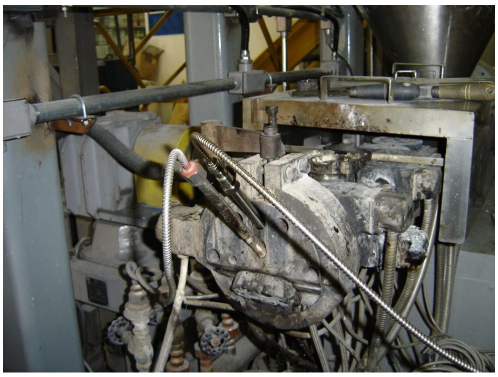

Figure 3. Equitech's Reflection Polymer Melt Probe inside extruder

The first experiment tests the ability of the online system to correlate with laboratory measurements. In-line and real time color measurements of the thermoplastic extrudate were obtained by continuously monitoring L*, a* and b* generated with the in-line color spectrophotometer. Thermoplastic pellets were taken during the extrusion process at determined periods of time. The pellets were injection molded into "color plates" of standard dimensions and measured under controlled conditions using a laboratory spectrophotometer (GretagMacbeth 7000A) with a pulsed Xenon lamp, and a 360nm to 750nm spectral range. Good agreement between the in-line and the laboratory measurements was obtained during each color run as shown in Figure 4.

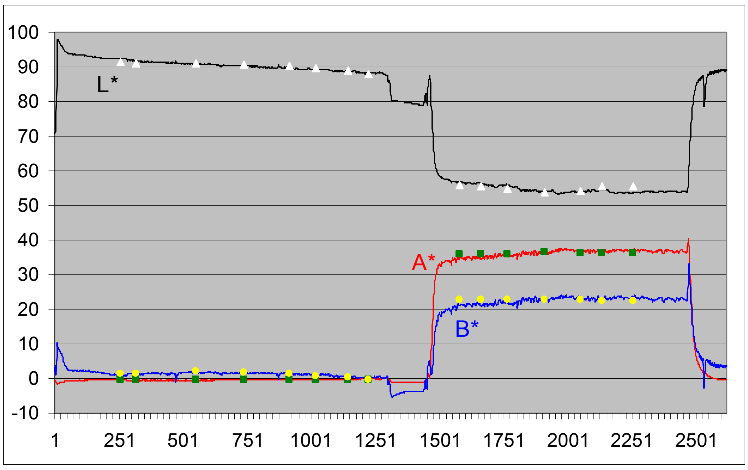

Figure 4. Correlation of Online vs. Laboratory Color Measurements in a thermoplastic compounding operation and spectrophotometer response with a change in color.

The in-line instrument readings were corrected to the laboratory values using linear interpolation. Thus, for each color that was compounded, an in-line governing equation of L*, a* and b* was generated using the laboratory and the measured L*, a* and b* values.

Initially, the correlation was achieved using two color samples that were measured on both the in-line and laboratory color instruments. Sample N°1 was a white or near white sample made of the same base materials as the colored sample of interest. Sample N°2 was a sample of the colored product. The in-line instrument readings are corrected to the laboratory values using linear interpolation. The equations are:

$$L^*_{corrected} = L^*_o \text{ x } (L^*_{wl} - L^*_{cl}) / (L^*_{wi} - L^*_{ci}) + (L^*_{cl} \text{ x } L^*_{wi} - L^*_{ci} \text{ x } L^*_{wl}) / (L^*_{wi} - L^*_{ci})$$
$$a^*_{corrected} = a^*_o \text{ x } (a^*_{wl} - a^*_{cl}) / (a^*_{wi} - a^*_{ci}) + (a^*_{cl} \text{ x } a^*_{wi} - a^*_{ci} \text{ x } a^*_{wl}) / (a^*_{wi} - a^*_{ci})$$
$$b^*_{corrected} = b^*_o \text{ x } (b^*_{wl} - b^*_{cl}) / (b^*_{wi} - b^*_{ci}) + (b^*_{cl} \text{ x } b^*_{wi} - b^*_{ci} \text{ x } b^*_{wl}) / (b^*_{wi} - b^*_{ci})$$

where: $L^*_{wi}, a^*_{wi}, b^*_{wi}$ — Where wi = white in-line
$L^*_{ci}, a^*_{ci}, b^*_{ci}$ — Where ci = colored in-line
$L^*_{wl}, a^*_{wl}, b^*_{wl}$ — Where wl = white laboratory
$L^*_{cl}, a^*_{cl}, b^*_{cl}$. — Where cl = colored laboratory

The same white sample can be used for all colors, so in effect only one sample of each color product need be measured to complete these equations. If the product is white or near white, the equations become unstable. We suggest the following equations in this case.

$$L^*_{corrected} = L^*_o \text{ x } (L^*_{wl} / (L^*_{wi})$$
$$a^*_{corrected} = a^*_o + (a^*_{wl} - a^*_{wi})$$
$$b^*_{corrected} = b^*_o + (b^*_{wl} - b^*_{wi})$$

The equations can be simplified if the white sample is used as the Blank material for the in-line instrument. In this case, $L^*_{wi} = 100$, $a^*_{wi}=b^*_{wi}=0$

The second experiment tests the sensitivity of the online analyzer. The RPMP is placed after the melt pumps from a dual extruder arrangement. Extruder-1 is feeding a 0.4% TiO_2 mixture and extruder-2 is feeding an orange colored mixture. The feed rate of TiO2 to extruder-1 is varied while holding all other conditions constant. The temperature at the RPMP is 270°C and the pressure is 1200 psi. Figure 5 shows the results of the run.

The first adjustment to TiO2 was a small change at point #625 of 0.5% relative to the base feed rate of 0.4%. This translates to a change from 0.4% to 0.402%. This change is easily seen in L* and is approximately 10 time the noise level of the online analyzer. The effect on a* and b* is much less pronounced. The TiO2 feed rate was increased to 0.42% at point #1250, then decreased to 0.412% at point #2500, and finally reset to 0.42% at point # 3850. The effects of these changes is easily seen in all the coordinates L*, a* and b*. The a* and b* plots appear more noisy than the L* plot. This effect is due to the nature of the flow control of the dual extruders. Observe that small fluctuations in a* and b* are highly correlated with each other and inversely correlated with the small fluctuations in L*. But the large changes due to the TiO2 feed rate are all correlated. This indicates that flows of extruder-1 and extruder-2 are inversely oscillating while the control system tries to maintain constant total output. The oscillations are small but easily discernible.

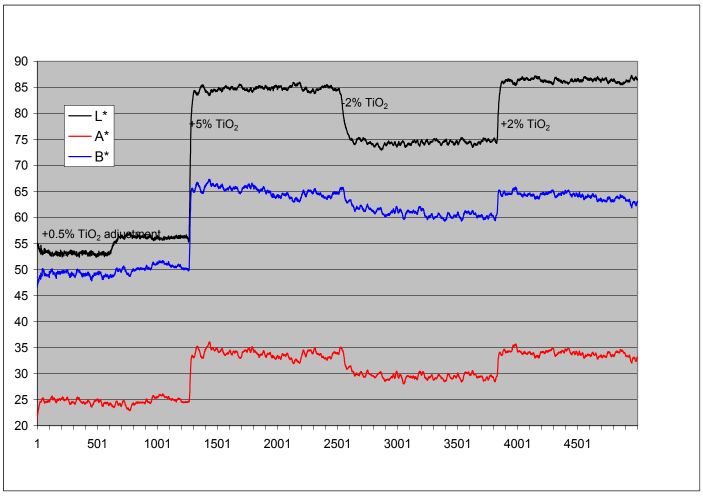

Figure 5. Sensitivity of the Equispec™ OCS to small changes in pigment load in the production of orange nylon with TiO2

Conclusions

The Equispec™ OCS and the RPMP are ideal for measuring color on thermoplastic compounds directly on the extrudate. Both tools are sensitive and accurate for in-line color quality control. The results obtained with the Equispec™ OCS are comparable to those obtained on a top-of-the-line laboratory spectrophotometer. In addition, the Equispec™ OCS and the RPMP are capable of monitoring the performance of thermoplastic extruders as they provide accurate real-time process information such as system time response and system stability.

References

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