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Analyzing Recycled Polyethylene Resin for Polypropylene Contamination Using FT-IR

Introduction

The use of post-consumer recycled, high-density polyethylene (HDPE) has been steadily growing over recent years. For example, approximately 974,000,000 pounds of HDPE were recycled in 2011, according to a recent Association of Post-Consumer Plastic Recyclers report (1). Thus, recycled HDPE, a Green, cost-effective alternative to virgin HDPE, has become an important raw material for many HDPE-based products, such as sheet, pipe, decking material, electrical conduit, bottles, etc. Most recycled HDPE is from bottles, many of which have caps and pour spouts made of polypropylene (PP), a material that has similar physical properties to HDPE. Consequently, PP is a common contaminate in post-consumer recycled HDPE resin and can adversely affect end-product performance (e.g., products can be more brittle and susceptible to stress cracking). In order to maintain high quality, it is imperative that processors monitor and control the amount of PP contained in recycled resin. For this purpose, a reliable tool is Fourier Transform Infrared Spectroscopy (FT-IR), and, to that end, the PerkinElmer Spectrum Two™ FT-IR is the easy-to-use, accurate system for measuring the amount of PP in recycled HDPE resin. This Application Report presents the methodology for employing the PerkinElmer Spectrum Two for this analysis.

Experimental

Standards were prepared by Advanced Blending Technologies (Table 1). Samples of virgin HDPE pellets were first dry-blended with virgin homopolymer PP pellets at the percentages shown in Table 1 (covers a range, 3-12%, typically observed for levels of PP in recycled HDPE resin), then hot melt compounded through a ¾-inch single screw laboratory extruder. In order to ensure a homogenous mix, the samples were (i) dry blended and then (ii) re-extruded two additional times for a total of three passes through the extruder.

Table 1. Standards at Four Different Blends of Polyethylene (PE) and Polypropylene

Name	Polypropylene (%)	Polyethylene (%)
12% PP 4	12	88
9% PP 4	9	91
6% PP 4	6	94
3% PP 4	3	97

A PerkinElmer Spectrum Two, fitted with a diamond single-bounce ATR, was used to collect spectra of the standards at 4 cm^{-1} spectral resolution with four co-adds. The PP/HDPE resin pellets were placed/pressed on the diamond crystal and measured directly without any further sample preparation. Each standard was tested 5-6 times using different pellets. PerkinElmer Spectrum Quant software was used to build a calibration method with the standards data. (Spectrum Quant is a powerful software package that includes the linear regression-based Beer's Law, which allows the use of area, peak or maximum peak, or peak ratio with baseline options. The software also has options for quadratic and cubic curve fitting, which were not required for this analysis.)

For this analysis, the PE peak (in black) centered at 719 cm^{-1} (attributed to methylene group rocking) was ratioed against the PP peak (in green) centered at 1,376 cm^{-1} (attributed to methyl group symmetrical bending). Representative spectra (overlaid) are presented in Figure 1. The peak ratios were then used for developing the calibration curve, presented in the next section in Figure 2.
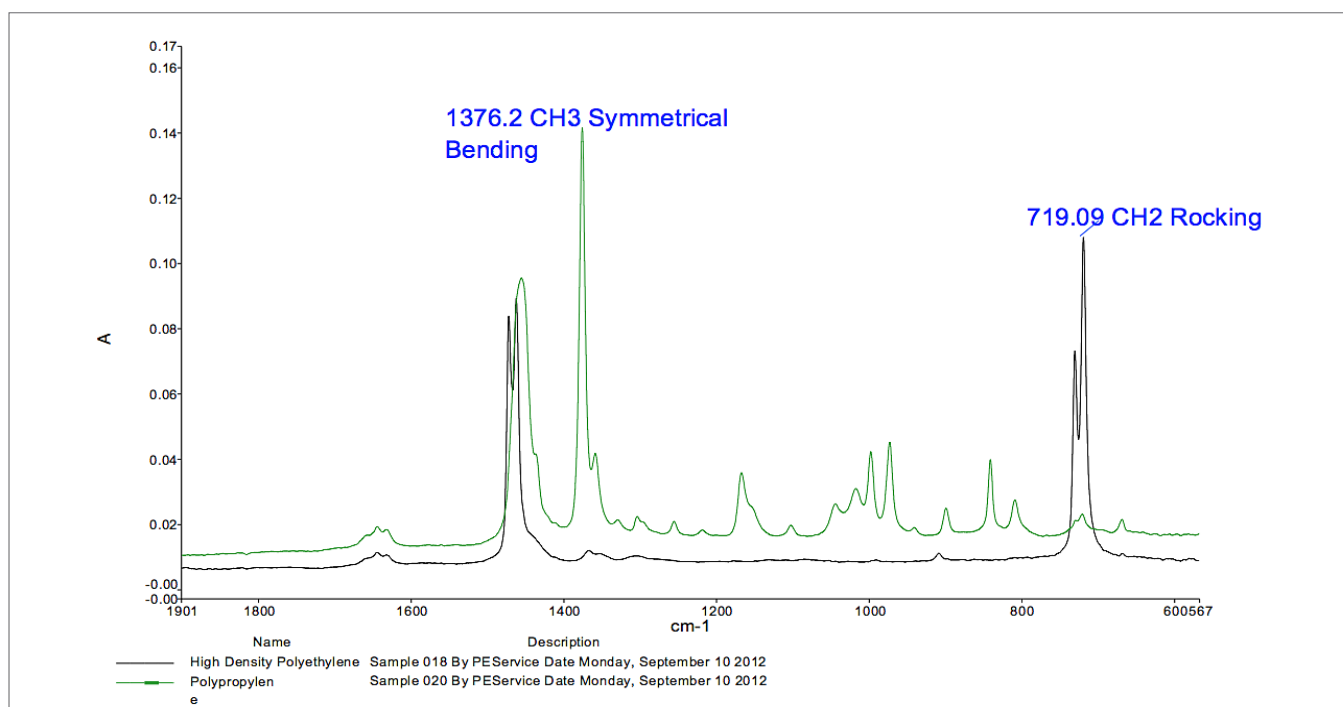

Figure 1. Spectral Comparison of High Density Polyethylene and Polypropylene

Results

Inspection of Figure 2 reveals a good linear fit for calculated PP (based on the peak ratio) and the specified values, yielding a regression correlation of nearly 0.999. The standard error of prediction (SEP) is low, with a value of approximately 0.19%, indicating that the model is robust.

In any quantitative analysis, an important step is validation of the model. Independent validation was done within the Spectrum Quant software. Table 2 presents the validation results for PP. Consistent with the good linear fit in the calibration curve, the residual values are generally low.

Table 2. Validation Results for PP

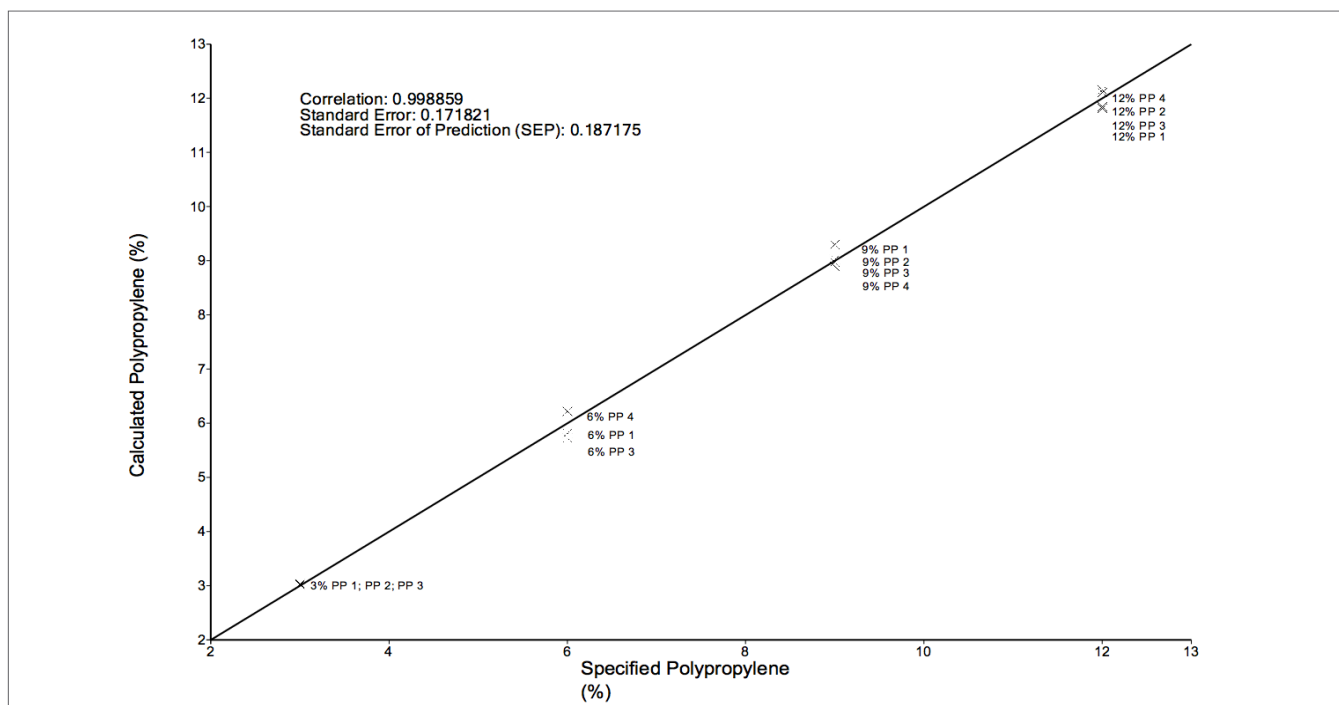| Validation Samples Specified		Calculated	Residual
12% PP validation	12	11.7817	0.218282
9% PP validation	9	9.30215	-0.302153
6% PP validation 1	6	5.60739	0.392614
6% PP validation 2	6	5.70573	0.294273
3% PP validation	3	3.17936	-0.179363

Figure 2. Calibration Model for PP

Conclusion

A feature of the PerkinElmer Spectrum Two with a single-bounce ATR accessory is that high quality spectra can be collected in seconds, without sample preparation, owing to the advanced design and built-in algorithms, which facilitate ease-of-use. These results were quickly generated, in a matter of minutes; the most time-consuming part of the analysis was preparation of the "contaminated" resin standards. In conclusion, the calibration curve showed excellent linearity and robustness, indicating that, for the levels of PP in recycled HDPE measured here, the Spectrum Two is an excellent tool for quick screening analysis. It is noted that, for higher concentrations, a second calibration might be required, bracketing the higher concentrations of PP in recycled HDPE resin.

Reference

1. American Chemistry Council, *"2012 United States National Post-Consumer Plastics Bottle Recycling Report",* 2013, 14 p.

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