APPLICATION NOTE

Thermal Analysis

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Analysis of Recycled Polyethylene Using the PerkinElmer Pyris™ DSC 9

Introduction

Differential scanning calorimetry (DSC) provides information about key thermal characteristics of polymers including insights into glass transitions, melting point, recrystallization and curing among others. When characterizing polymers, the PerkinElmer Pyris™ DSC 9 provides a simple yet robust solution to differentiate between different grades of the same polymer based on known thermal characteristics. This work will demonstrate how DSC can be used to easily determine the grade of a sample of recycled polyethylene, as well as detect the present of a low concentration of polypropylene in a recycled polyethylene sample.

Experimental

5 mg (+/- 0.5 mg) samples of polyethylene were cut to give a flat shape, providing ideal thermal contact for DSC analysis. Samples were crimped into standard aluminum pans (02190041) prior to analysis using the DSC 9 differential scanning calorimeter (Figure 1). All samples were measured using the following temperature program:

- Hold for 1 minute at 50 °C
- Heat from 50 to 180 °C at 20 °C/min
- Hold for 1 minute at 180 °C
- Cool from 180 to 50 °C at 20 °C/min
- Hold for 1 minute at 50 °C
- Heat from 50 to 180 °C at 20 °C/min

This method involves first heating all samples past their melting temperature, then cooling them down at a controlled, consistent cooling rate. This accomplishes two key tasks. The first is ensuring optimum thermal contact between the sample and pan. The second is essentially 'erasing' any thermal history the sample might have due to previous processing. This provides far more comparable results than would otherwise be available.

Figure 1.
PerkinElmer Pyris™
DSC 9 Differential Scanning Calorimeter.and
Established PDEs for Elemental Impurities.

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Results and Discussion

Results from DSC analysis of virgin low-density polyethylene (LDPE) and high-density polyethylene (HDPE) are shown in Figure 2. For both samples, Pyris™ software was used to calculate the melting onset temperature as well as the peak melting temperature.

This data may be used as a reference in order to determine the grade of an unknown polyethylene sample. Figure 3 shows the data obtained by a sample of polyethylene with an unknown grade.

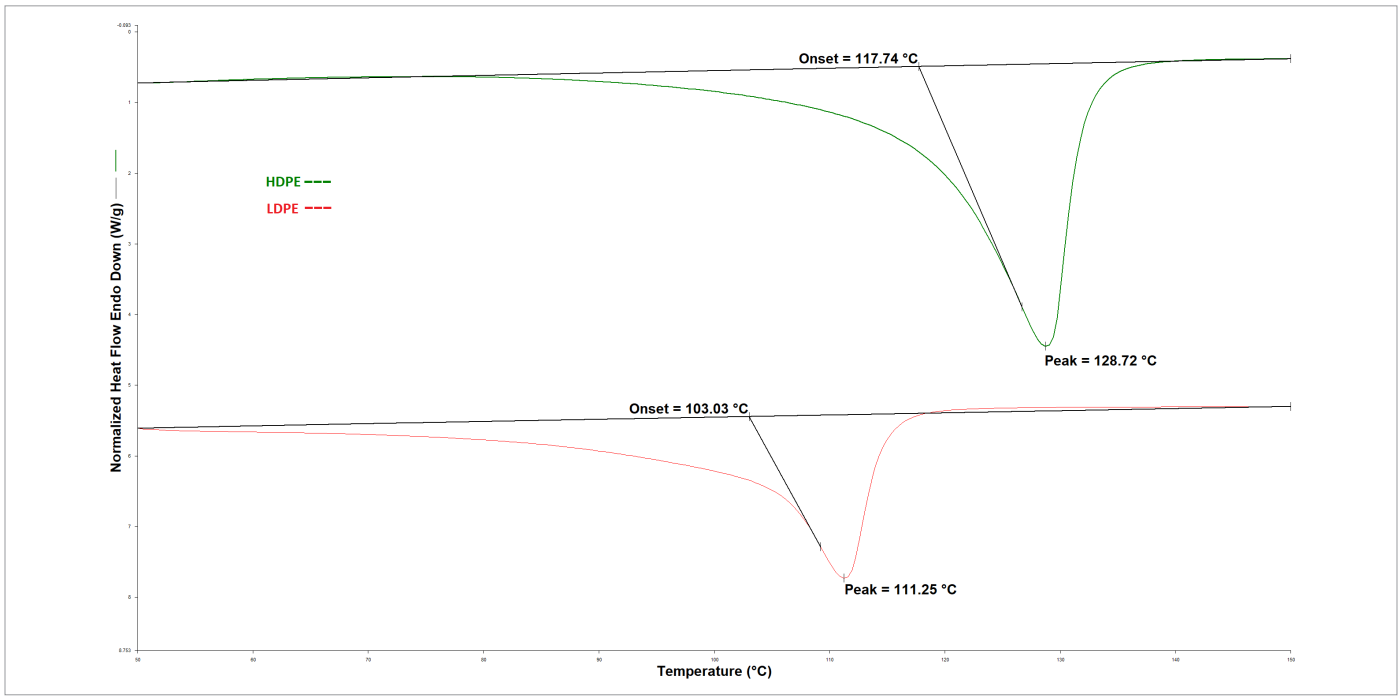

Figure 2. DSC results from virgin polyethylene samples.

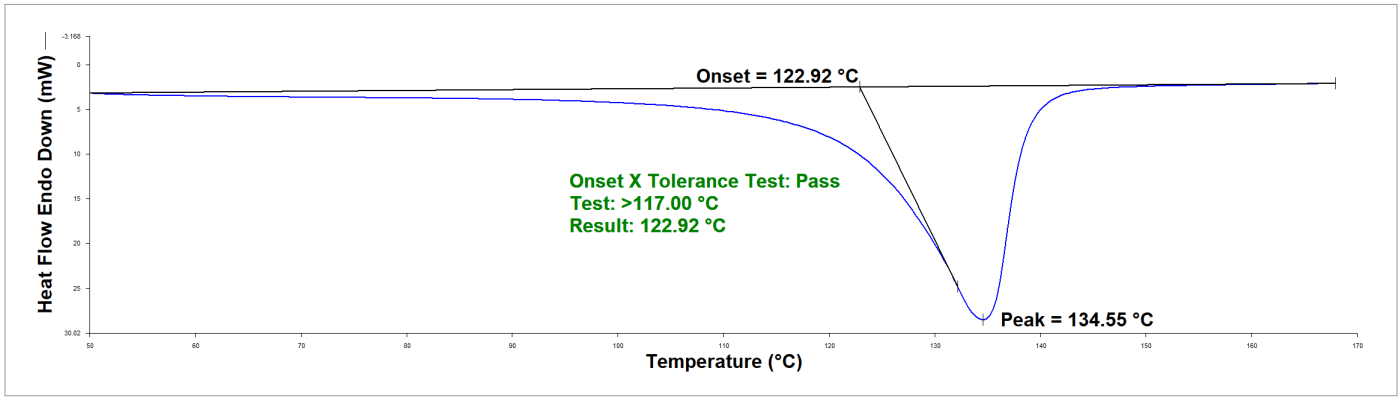

Figure 3. DSC data obtained from a recycled polyethylene sample.

Pyris software allows users to set tolerance test limits. In this case, the limit can be set such that anything with a melt onset temperature above 117 °C (as found from analysis of the virgin polymer) may be deemed to be HDPE. This provides a simple and streamlined method for quickly determining the grade of polyethylene.

Another test that may be employed with recycled polyethylene is the detection of polypropylene. Figure 4 shows the DSC collected from a sample that is suspected to contain a small quantity (approximately 2%) of polypropylene.

From this data we can determine the grade of polyethylene by looking at the melting onset temperature (LDPE due to an onset of 101 °C) as well as clearly identifying the peak with a melting point of 162 °C.

Summary

The PerkinElmer Pyris DSC 9 provides a robust yet high-performance solution for polymer analysis. Pyris software may be used to automate data analysis to give a simple yes or no answer regarding the identity of a sample. Furthermore, the high sensitivity of DSC allows for the detection of contaminant polymers even at low concentrations.

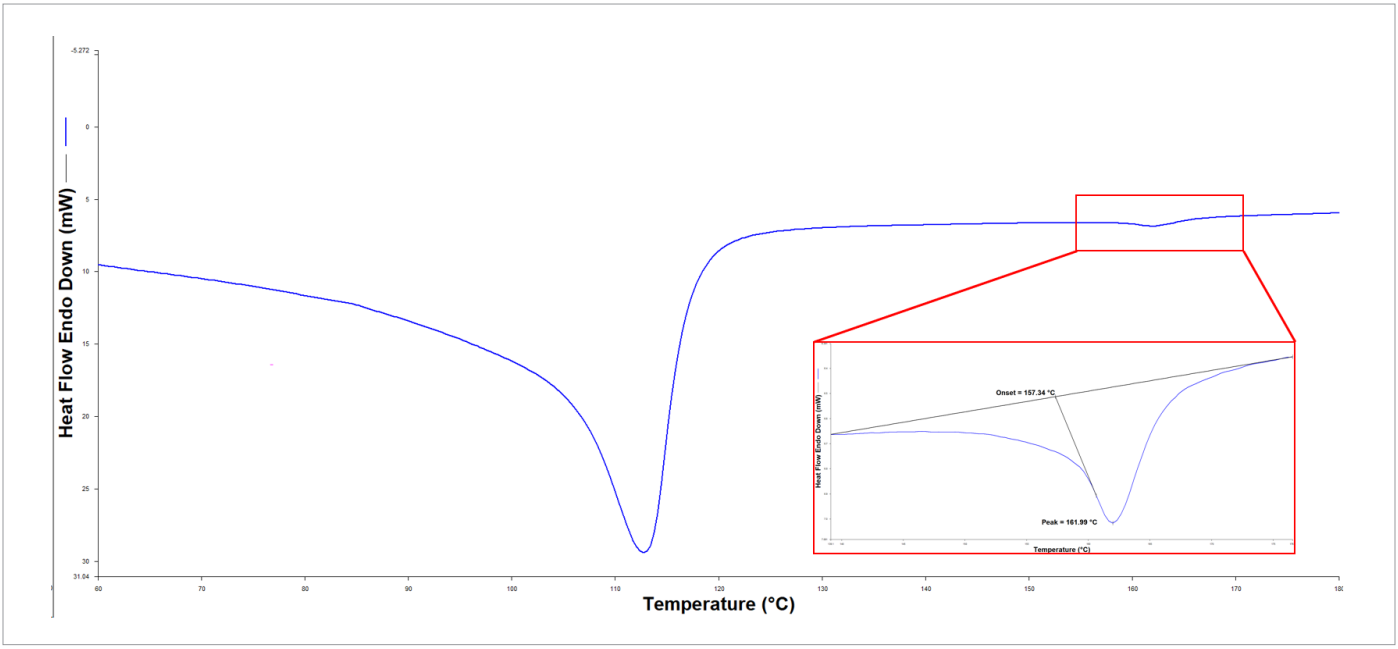

Figure 4. DSC data from a sample of recycled polyethylene suspected to contain a small quantity of polypropylene.

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