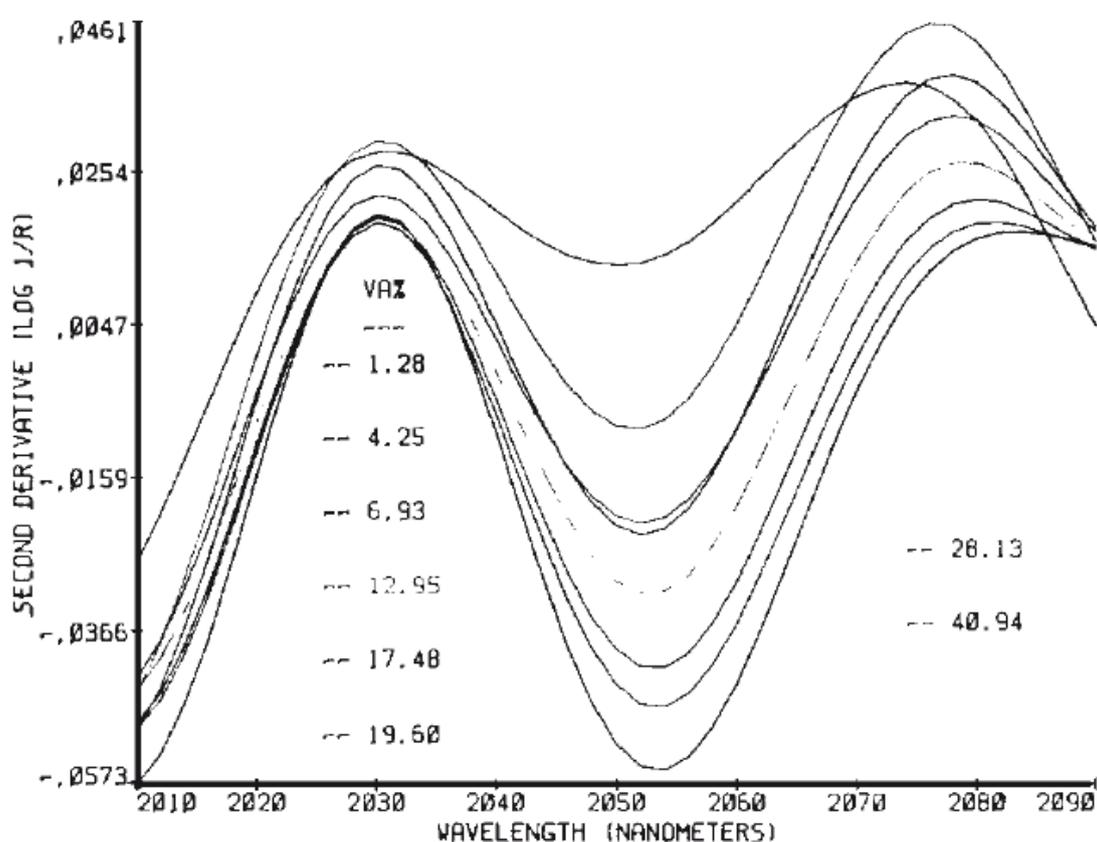


Analysis of copolymer levels in polymer pellets by near-infrared spectroscopy

Ethylene-Vinyl Acetate Copolymer



This Application Note describes the determination of copolymer levels in polyethylene (PE) and polyvinylacetate (PVA) pellets using NIRS. Without any sample preparation and within less than 30 s, NIRS determines the composition of the polymer blends investigated. By using second derivative spectra and linear least-squares regression, straightforward quantitative analyses are possible.

Method description

Introduction

Thermal polymers are typically supplied in the form of chips or pellets. Some of the very properties desired for polymers such as strength, solvent resistance, acids, etc. make analysis of these materials in this form extremely difficult. Typical methods of analysis require dissolution of the pellets, which can take from several hours to over one day depending on the material.

An analysis method which did not require any sample preparation would be advantageous in terms of speed and ease of analysis, and reduction of errors from irreproducible sample preparation.

Near-infrared (NIR) spectroscopy is uniquely suited for quantitative analysis of polymers, since no sample preparation is required. Furthermore, it is nondestructive.

The example shown will demonstrate the use of NIR for determination of copolymer levels in polymer pellets, measured without any sample modification.

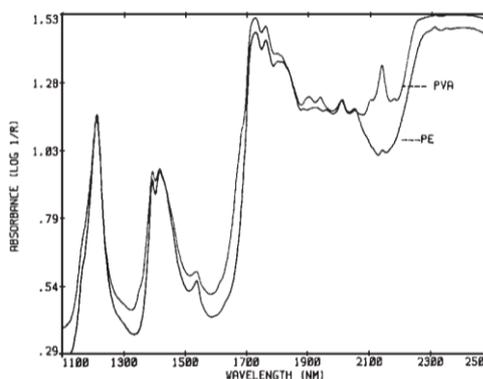
Experimental

The NIR spectra of the samples were measured using a NIRS XDS RapidContent Analyzer in reflectance mode from 1100–2500 nm. The samples were simply poured into a hopper for analysis. Analysis of the entire hopper contents occurs unattended by an operator. The obtained spectrum is the average of all scans. This sampling technique diminishes variations caused by non-homogeneous sample packing, which allows quantitative analysis to be performed without grinding the samples.

Results and discussion

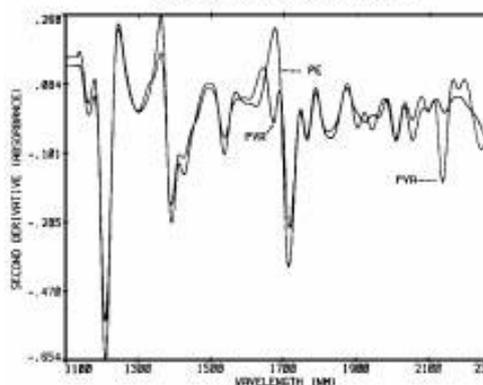
The NIR spectra of polyethylene (PE) and polyvinyl acetate (PVA) were collected to determine the spectroscopic contributions of each of the copolymers, and are shown in Figure 1.

Figure 1
Copolymer Components



Due to the broad, overlapping absorptions typical in this spectral region, the spectra of the two polymers appear very similar. By conversion to the second derivative, shown in Figure 2, the features in each spectrum are enhanced, making spectral comparison easier. (Also note that this math treatment inverts absorption peak maxima to second derivative peak minima.)

Figure 2
Copolymer Components



Figures 3 and 4 isolate unique absorbance features for PVA and PE.

Method description

Figure 3
Copolymer Components

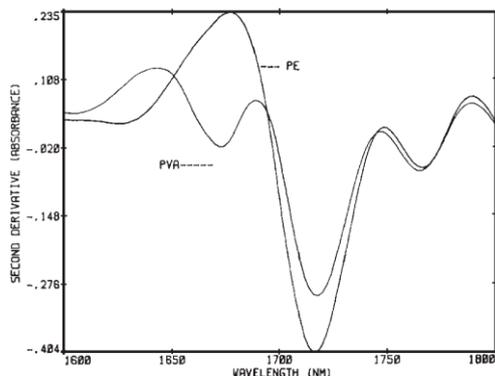


Figure 4
Copolymer Components

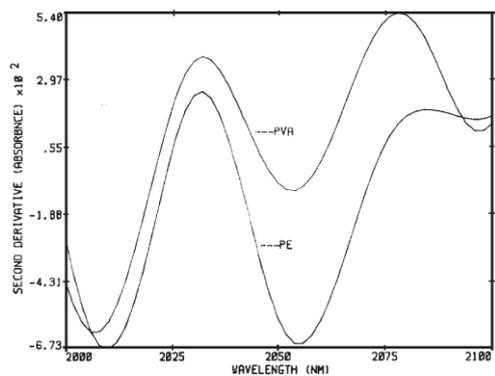
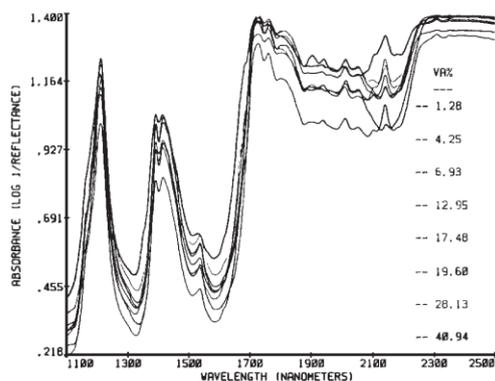


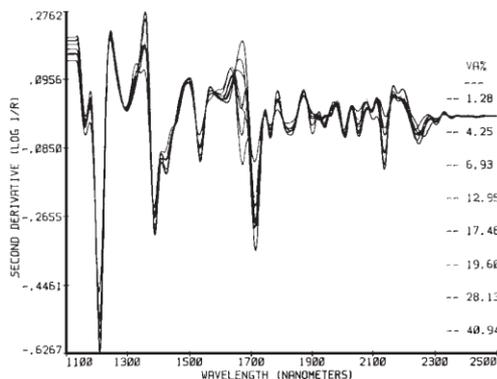
Figure 5 shows the absorbance (log 1/reflectance) spectra for ethylene vinyl acetate (EVA) copolymer pellets.

Figure 5
Ethylene-Vinyl Acetate Copolymer



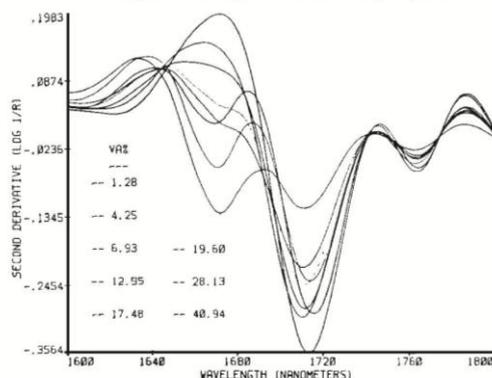
The baseline changes dramatically due to variations in the intensity of radiation scattered from the pelletized samples. The baseline variations can be compensated by conversion of the spectra to the second derivative, shown in Figure 6.

Figure 6
Ethylene-Vinyl Acetate Copolymer

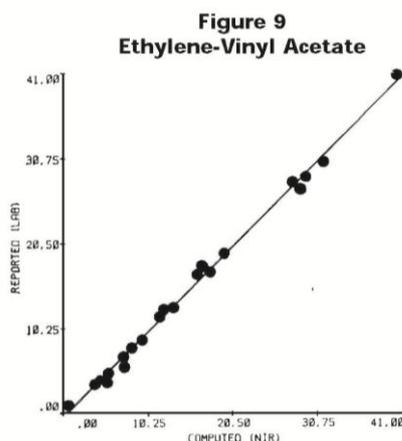
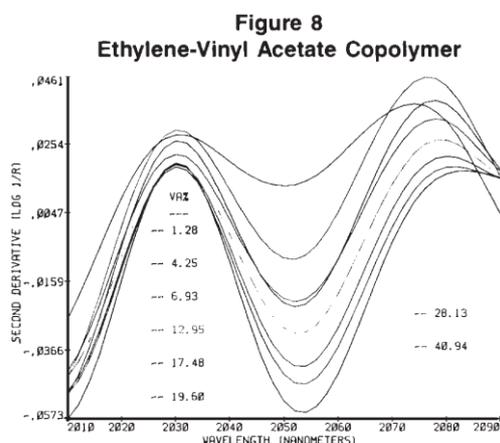


With the baseline variations compensated, the chemical differences due to the amount of copolymer can now be seen. The increase in a vinyl acetate absorption near 1680 nm is isolated in Figure 7, and the corresponding decrease in an ethylene absorption at 2050 nm is shown in Figure 8.

Figure 7
Ethylene-Vinyl Acetate Copolymer



Method description



A linear least-squares regression was performed using the second derivative spectra, and the known vinyl acetate levels; results are shown in Table 1.

Table 1

Wavelength [nm]	Constants	R	SEC
2042	K(0) = 21.153 K(1) = 139.029	0.99	0.7

In this table, K(0) and K(1) are the intercept and slope of the regression line, respectively. R is the correlation coefficient and SEC is the standard error of calibration.

A comparison of the known values and NIR results are shown graphically in Figure 9.

Conclusions

The analysis of polymer pellets by NIRS has the advantage that the sample preparation step is removed, with no degradation in accuracy of the determination. This was demonstrated for samples of a copolymer blend, whose baseline variations appear at first to be too large for quantitative analysis to be performed on the pellets as-is.

Through the averaging of large areas of the samples, and the use of derivative math treatments, quantitative analyses can be performed, as demonstrated in this study.

Other copolymer pellet determinations that have been performed using NIR spectroscopy include ethylene/propylene, styrene/butadiene, and butylene/terephthalate. Polymer additives such as antioxidants, stabilizers and slip agents are also being successfully monitored by NIR in pelletized samples.