



Near-infrared Technology for Analysis of Multilayer Plastics

Analytical Spectral Devices, Inc.

Multilayer plastic films used for packaging of food and pharmaceuticals is gaining in popularity.

The properties of the multilayer plastic is tailored to the application by adjusting the composition and thickness of the individual layers. Typically, the core layer provides strength, while the outside layer(s) acts as water vapor and/or oxygen barriers. While these barrier layers are essential for maintaining the quality of the packaged product, they are also quite costly. Thus, the ability to monitor and control the thickness of these layers impacts both product quality and overall cost.

ASD's QualitySpec Pro near-infrared analyzer is an excellent tool to not only verify the identity of each layer and determine its thickness, but also to verify the identity and quality of incoming raw materials prior to their introduction into the coextrusion process. Due to its dependability and ruggedness, QualitySpec Pro is ideally suited to perform these measurements either at-line or near-line, with rapid and precise results so product specifications are more tightly controlled. QualitySpec Pro is a cost-effective quality control tool for minimizing both the use of costly barrier materials and the production of out-of-spec product.

Experimental

All data were collected with an ASD QualitySpec Pro NIR analyzer using a fiber optic reflectance probe. Spectral averaging was set to 20 and with an acquired scan every 0.1 seconds, each measurement took two seconds to collect. The first sample set consisted of three layer coextruded plastic films, placed between a Spectralon reference panel and the tip of the bifurcated reflectance probe for spectrum collection. The second sample set consisted of several different plastics in pellet form. Spectra were collected by placing the tip of the fiber optic reflectance probe in contact with the sample. In practice, either of these measurements can be made at-line and in-place, in the receiving area or on the production floor.

Results and Discussion

Coextruded Multilayer Films. Twenty different multilayer plastic samples with measured layer thickness values were obtained and absorbance spectra were measured using an ASD QualitySpec Pro NIR analyzer. Each of the three layers analyzed (skin, core, and seal) had a unique composition. In general, NIR spectra of multilayer plastics have broad and overlapping peaks

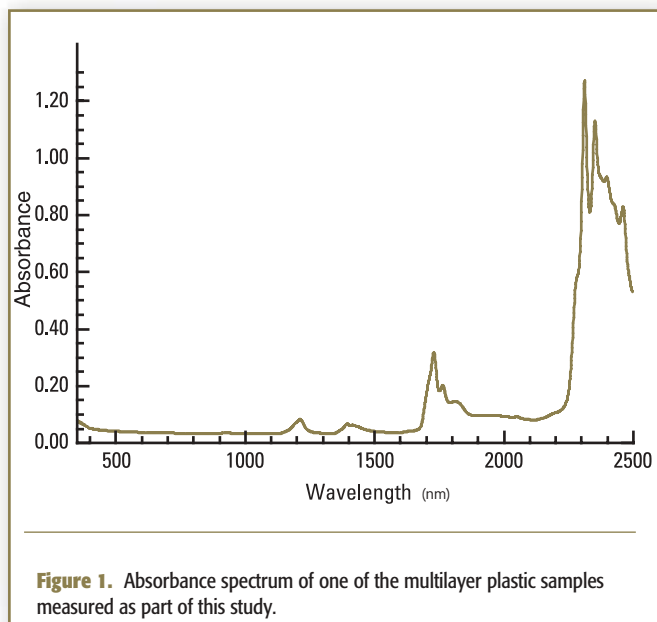


Figure 1. Absorbance spectrum of one of the multilayer plastic samples measured as part of this study.

associated with combinations and overtones of molecular vibrations of the many C-H as well as the N-H and O-H bonds found in these large molecules (see Figure 1).

The ASD QualitySpec Pro NIR analyzer was calibrated using Grams 32/AI chemometrics software which interfaces in real-time with the ASD QualitySpec Pro. The best calibration was obtained using the partial least squares regression approach applied to first derivative absorbance spectra. A full cross-validation was performed to insure the integrity of the calibrations. The calibration for each of the three layers was evaluated by comparing the actual and predicted layer thickness values (Figure 2).

These results attest to the utility of ASD's QualitySpec Pro NIR analyzer for the analysis of multilayer plastics. From Figure 2 it can be seen that QualitySpec Pro is capable of producing results similar in accuracy to the time-consuming and costly laboratory methods used to make the reference measurements.

Plastics Raw Material ID. Thirty samples each of three different types of plastic in pellet form were obtained. The types of plastics analyzed are as follows: Polyethylene Terephthalate (PETE), High Density Polyethylene (HDPE), and Polypropylene (PP). Even though the spectral differences were, in some cases, subtle (see Figure 3), all samples were correctly identified using the Grams 32/AI chemometrics software in discriminate mode (see Figure 3).

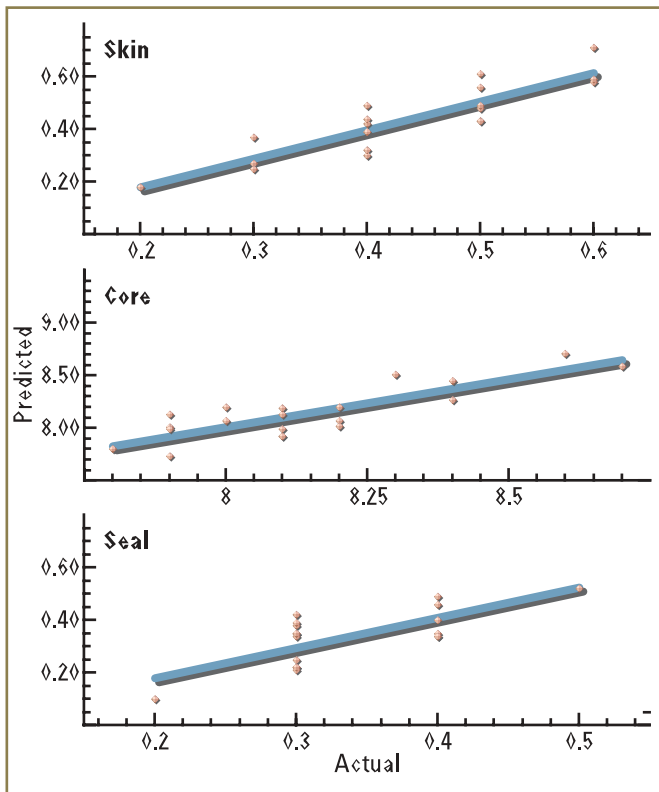


Figure 2. Predicted values of the skin, core and seal layer thickness derived from the NIR calibration are plotted against actual measured values.

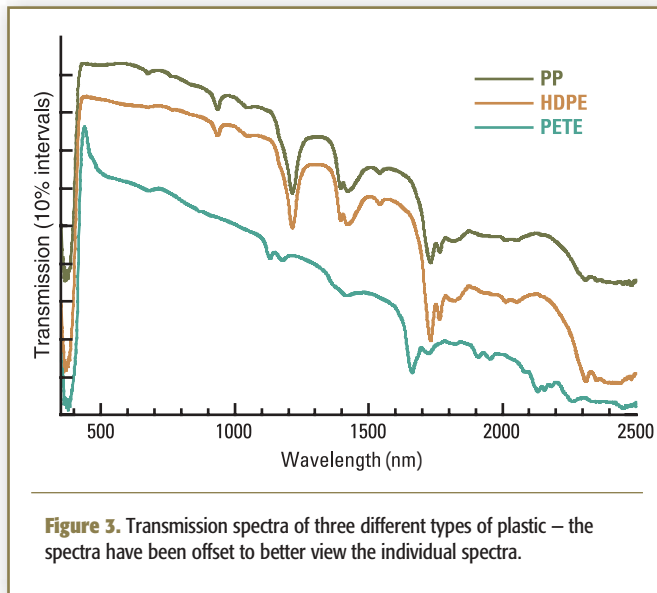


Figure 3. Transmission spectra of three different types of plastic – the spectra have been offset to better view the individual spectra.

Conclusion

The results of this experiment demonstrate that the ASD QualitySpec Pro NIR analyzer can measure the thickness of individual layers of multilayer plastic film and can discriminate chemically different, but visually similar, plastics. Since this analysis method is non-invasive, non-destructive and very fast, the ASD QualitySpec Pro NIR analyzer is an ideal QA/QC tool for the qualitative and quantitative analysis of industrial plastics. QualitySpec Pro’s ruggedness allows the user to make measurements at any convenient location in the production environment. For more information about the QualitySpec Pro NIR analyzer please contact Analytical Spectral Devices.

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