



Application Note AN-NIR-124

Polyethylene content determination in polypropylene pellets by NIRS

Rapid analysis of recycled polyolefins

Next to polyethylene (PE), polypropylene (PP) is the most widely used plastic worldwide. Recycling these polyolefins is problematic, since it is challenging to separate polyethylene from polypropylene using sink float separation. Increasing PE content in PP leads to a poor interfacial bond strength which could negatively impact the mechanical properties of the final molded product.

An efficient method to determine the polyolefin

composition based on near-infrared spectroscopy (NIRS) analysis is presented in this Application Note. The main advantage is the short time to result. Compared to the time-consuming standard method (i.e., Differential Scanning Calorimetry or DSC), the analysis of polyethylene content in polypropylene using near-infrared spectroscopy is performed in just seconds.

EXPERIMENTAL EQUIPMENT

Different polymer blends ($n = 54$) with varying polyethylene content from 0.5–35% were produced in a compounder. The polyethylene content was determined by weighing before the polymer blends were produced. All measurements on the OMNIS NIR Analyzer Solid (Figure 1) were performed in rotation using a large sample cup to average the subsample spectra. This sample setup reduces the influence of the particle size distribution of the polymer particles.



Figure 1. OMNIS NIR Analyzer Solid.

Table 1. Hardware and software equipment overview.

Equipment	Metrohm number
OMNIS NIR Analyzer Solid	2.1071.0010
Large holder OMNIS NIR, 100 mm	6.07402.100
Large cup OMNIS NIR, 100 mm	6.07402.110
OMNIS Stand-Alone license	6.6072.208
Quant Development software license	6.06008.002

RESULT

The obtained NIR spectra (Figure 2) were used to create prediction models for two different polyethylene content ranges. The first correlation diagram (Figure 3) displays the relation between the NIR prediction and the reference values in the range

of 0.5–35.0% PE content. Figure 4 displays the correlation in the polyethylene content range of 0.5–9.0%. The respective figures of merit (FOM) show that by selecting a smaller range, the absolute error can be lower compared to using the full PE range.

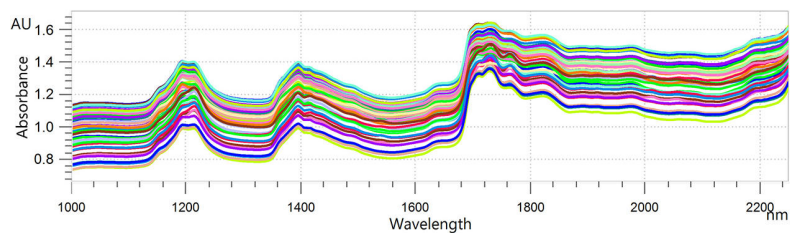


Figure 2. NIR spectra of different polymer blends with varying polyethylene content. Data was obtained with an OMNIS NIR Analyzer Solid.

RESULT PE CONTENT: 0.5–35.0%

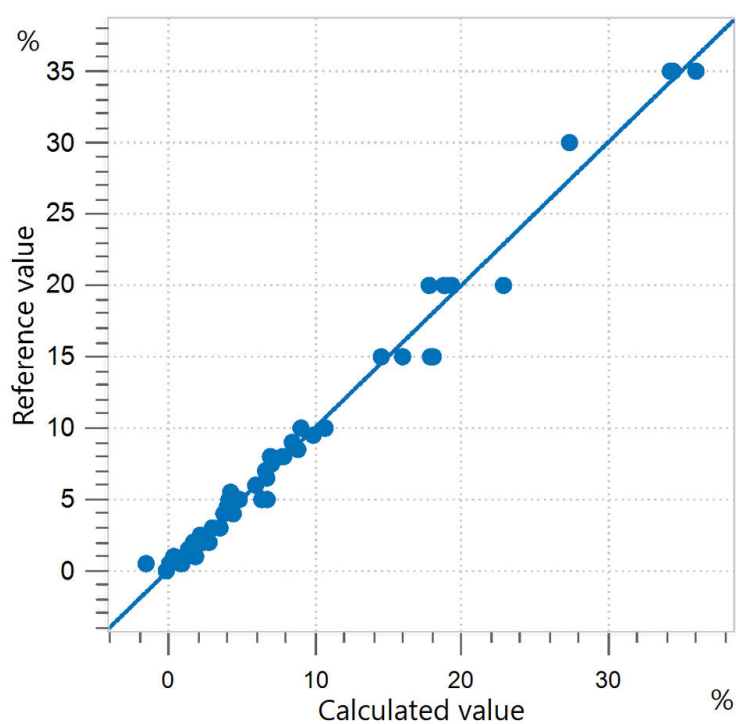


Figure 3. Correlation diagram and the respective FOMs for the prediction of polyethylene content in the range of 0.5–35.0%.

SEC (%)	SECV (%)	R ² CV
0.94	1.10	0.987

RESULT PE CONTENT: 0.5–9.0%

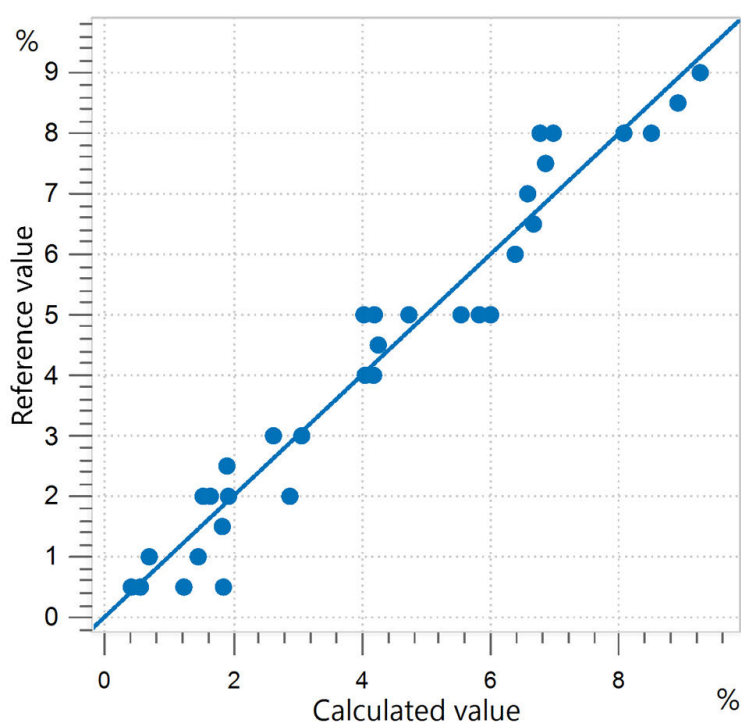


Figure 4. Correlation diagram and the respective FOMs for the prediction of polyethylene content in the range of 0.5–9.0%.

SEC (%)	SECV (%)	R ² CV
0.51	0.60	0.950

CONCLUSION

This Application Note shows the feasibility of NIR spectroscopy for the analysis of polyolefins. Detecting PE concentrations below 5% with the standard Differential Scanning Calorimetry (DSC) method can be challenging and time consuming. Compared to

DSC measurements (**Table 2**), near-infrared analysis saves significant time: one sample measurement is performed in a couple of seconds. Next to PE content, physical parameters like density can also be determined with NIRS.

Table 2. Time to result overview for the parameter of polyethylene content.

Parameter	Method	Time to result
PE content	Differential Scanning Calorimetry	60 min per sample

CONTACT

Metrohm Brasil
Rua Minerva, 161
05007-030 São Paulo

metrohm@metrohm.com.br

CONFIGURATION



OMNIS NIR Analyzer Solid

Near-infrared spectrometer for solid and viscous samples.

Developed and produced in accordance with Swiss quality standards, the OMNIS NIR Analyzer is the near-infrared spectroscopy (NIRS) solution for routine analysis along the entire production chain. Its application of the latest technologies and its integration in the modern OMNIS Software are reflected in its speed, operability and flexible utilization of this NIR spectrometer.

Overview of the advantages of the OMNIS NIR Analyzer Solid:

- Measurements of solids and viscous samples in less than 10 seconds
- Automated multi-position measurements for reproducible results, even with nonhomogeneous samples
- Simple integration in an automation system or link with additional analysis technologies (titration)
- Supports numerous sample vessels



Large holder OMNIS NIR, 100 mm

Large holder for large sample vessel OMNIS NIR, 100 mm (6.07402.110).

Permits unambiguous positioning of the sample vessel and the rotation of the sample vessel.