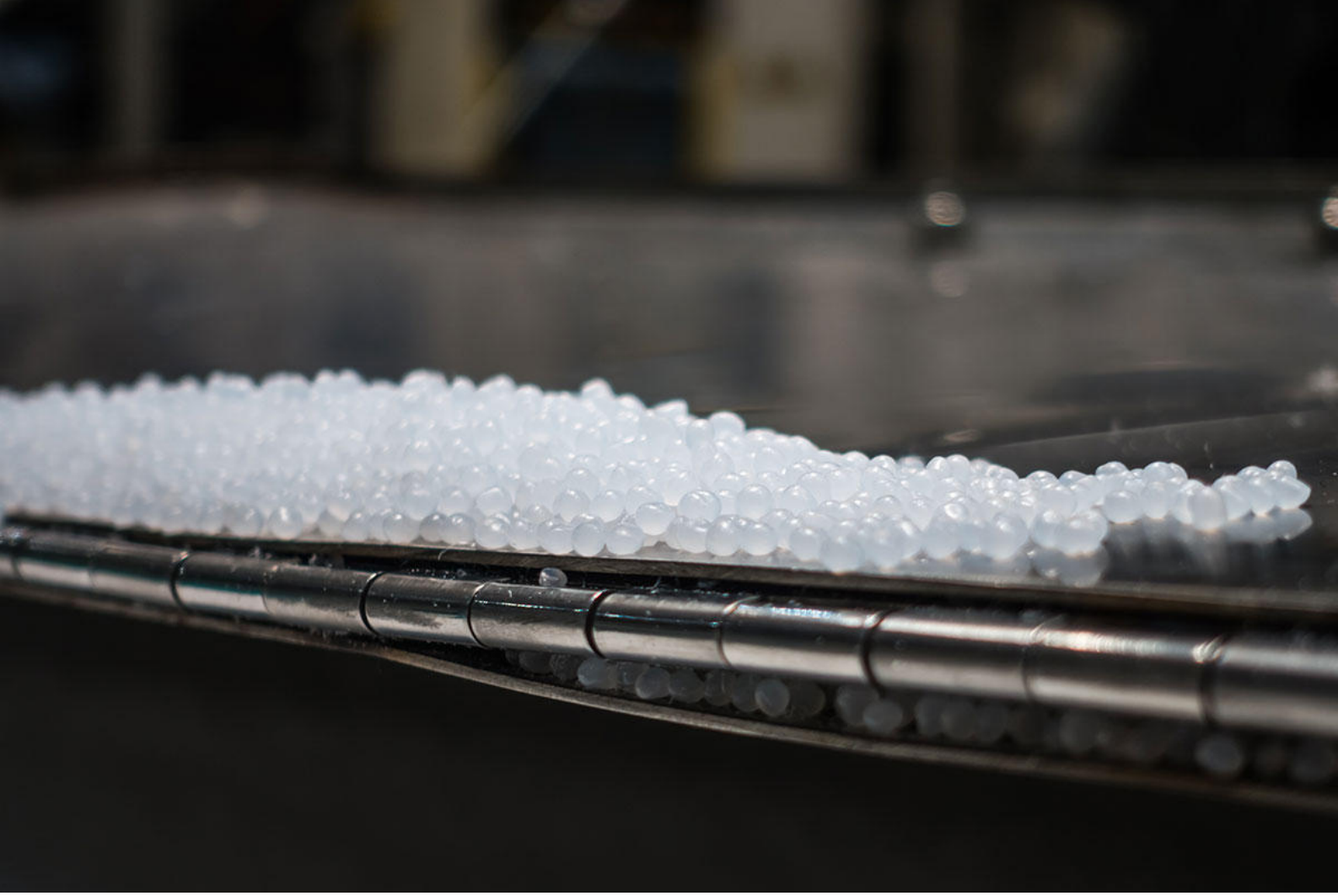


Application Note AN-NIR-119

Moisture content and rheology determination of fluorinated polyolefins using NIRS

Moisture and melt flow rate analysis results within seconds

Ethylene tetrafluoroethylene (ETFE) is a partially fluorinated copolymer of ethylene and tetrafluoroethylene. ETFE is a melt processable polymer, designed to have high corrosion resistance and strength over a wide temperature range. Some key quality parameters that should be monitored during the production of fluorinated polyolefins are melt flow rate (MFR) and moisture content. The measurement of these parameters usually involves using chemicals and can be time-consuming. Near-infrared spectroscopy (NIRS) offers users **rapid and reliable prediction** of key quality parameters in ETFE **without any sample preparation or cleaning procedures**.

EXPERIMENTAL EQUIPMENT

273 ETFE pellet samples with varying MFR and moisture content were measured on a Metrohm near-infrared spectroscopic analyzer in reflection mode. All measurements were performed in rotation using a large sample cup to reduce the stray light effect caused by varying particle sizes. An overlay of the sample spectra is displayed in Figure 1.

RESULT

The obtained NIR spectra (Figure 1) were used to create a prediction model for quantification of MFR and moisture content in ETFE. The quality of the prediction model was evaluated using correlation diagrams (Figures 2 and 3), which display a high correlation between the NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

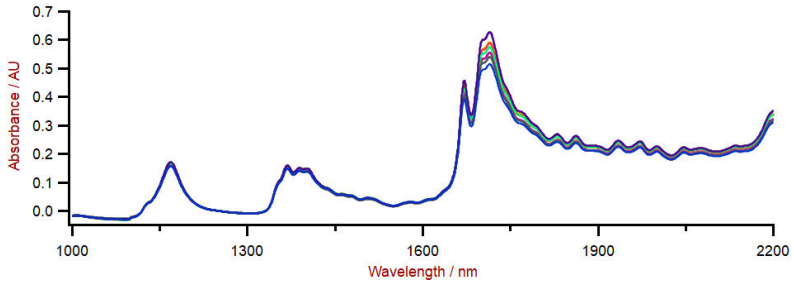


Figure 1. Overlaid NIR spectra of ETFE samples analyzed on a Metrohm near-infrared spectroscopic analyzer with a large sample cup.

RESULT MELT FLOW RATE

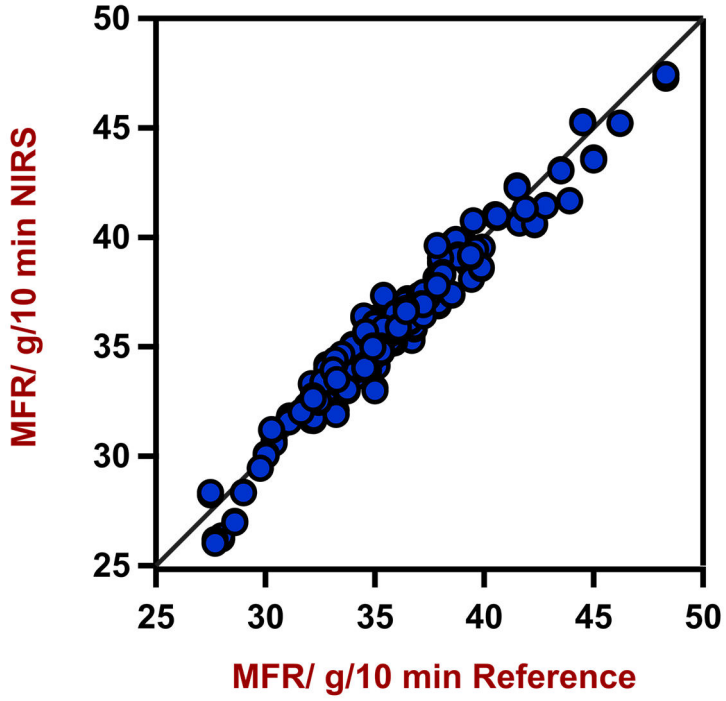


Figure 2. Correlation diagram and the respective figures of merit for the prediction of melt flow rate in ETFE. The MFR lab value was evaluated using a melt flow indexer.

Figures of Merit	Value
R^2	0.9413
Standard Error of Calibration	0.96 g/10 min
Standard Error of Cross-Validation	0.98 g/10 min

RESULT MOISTURE CONTENT

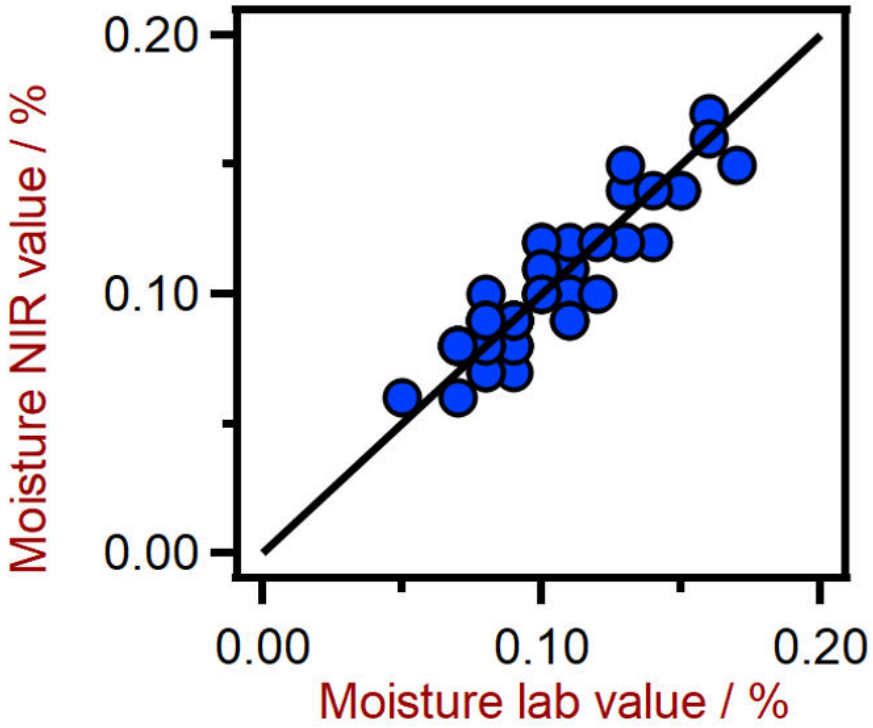


Figure 3. Correlation diagram and the respective figures of merit for the prediction of moisture content in ETFE. The lab value was evaluated by Karl Fischer titration.

Figures of Merit	Value
R^2	0.840
Standard Error of Calibration	0.011%
Standard Error of Cross-Validation	0.012%

CONCLUSION

This Application Note demonstrates the feasibility to determine mechanical properties and perform moisture analysis in fluorinated polyolefins like ETFE using NIRS. In comparison to the standard analytical methods (Table 1), quality control with NIR spectroscopy shows obvious advantages regarding the analysis time. In addition, the sample handling with near-infrared spectroscopy is easier to perform and safer since no chemicals are needed.

Table 1. Time to result overview for the different key quality parameters.

Parameter	Method	Time to result
Melt flow rate	Melt flow indexer	20 min (measurement and cleaning)
Moisture	Karl Fischer titration	3–5 min

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