Application Note AN-NIR-102

Density of polyolefins measured by near-infrared spectroscopy

Simple routine analysis of polymer pellets

Aside from melt flow rate, density is the most important parameter to describe the properties of polyethylene (PE) materials. PE stiffness, rigidity, and heat resistance increase with higher density. Various testing methods exist for density in PE – the most common is by density balance, measuring the buoyancy in a liquid (ASTM D792). This test is easy to perform, but the method contains a variety of measurement errors sources, such as specimen fixation corrections, temperature changes, or air bubbles within the sample pellets.

Trapped air bubbles formed during polymer pellet production result in lower density values when measured with the buoyancy method. In contrast, near-infrared spectroscopy (NIRS) is a fast analytical technique which shows a low influence on density measurement error if any air bubbles are present in the sample material.

EXPERIMENTAL EQUIPMENT

29 different polyethylene samples with varying density were measured on the Metrohm NIRS DS2500 Solid Analyzer (Figure 1) as well as with the buoyancy method described in ASTM D792. All measurements on the DS2500 Solid Analyzer were performed in rotation to average the subsample spectra. This setup with the DS2500 large sample cup reduces influences from the particle size distribution of the polymer pellets. Data acquisition and prediction model development were performed with the software package Vision Air Complete.

Table 1. Hardware and software equipment overview.

Equipment	Metrohm number
DS2500 Solid Analyzer	2.922.0010
DS2500 large sample cup	6.7402.050
Vision Air 2.0 Complete	6.6072.208

Figure 1. Metrohm NIRSDS2500 Solid Analyzer used for determination of density in PE pellets.

RESULT

The obtained Vis-NIR spectra (Figure 2) were used to create a prediction model for the density value determination in PE pellets. To verify the quality of the prediction model, correlation diagrams were created which display the correlation between the Vis-NIR prediction and primary method values received from the supplier (Figures 3–4).

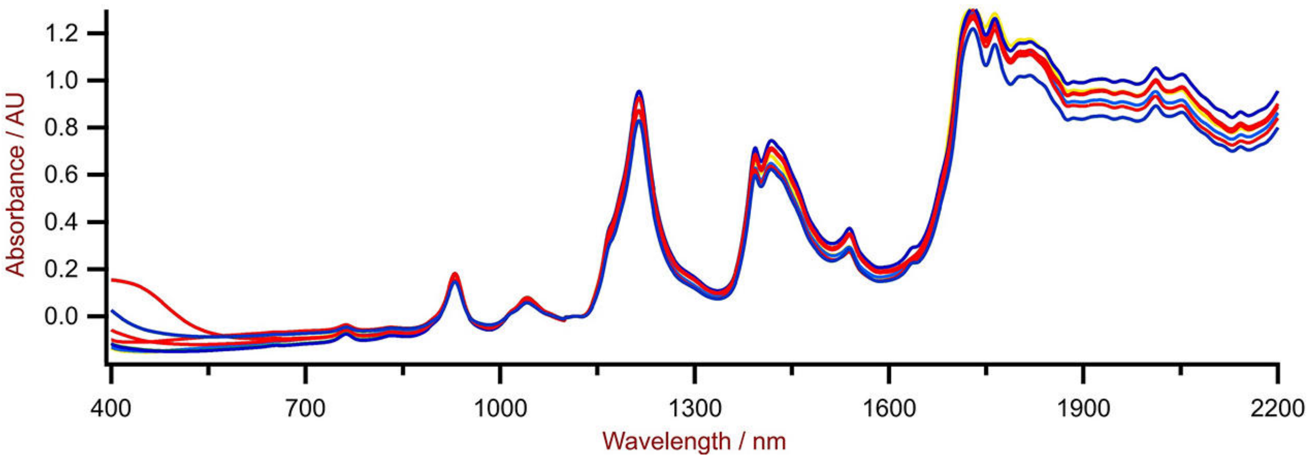

Figure 2.

RESULT DENSITY IN PE

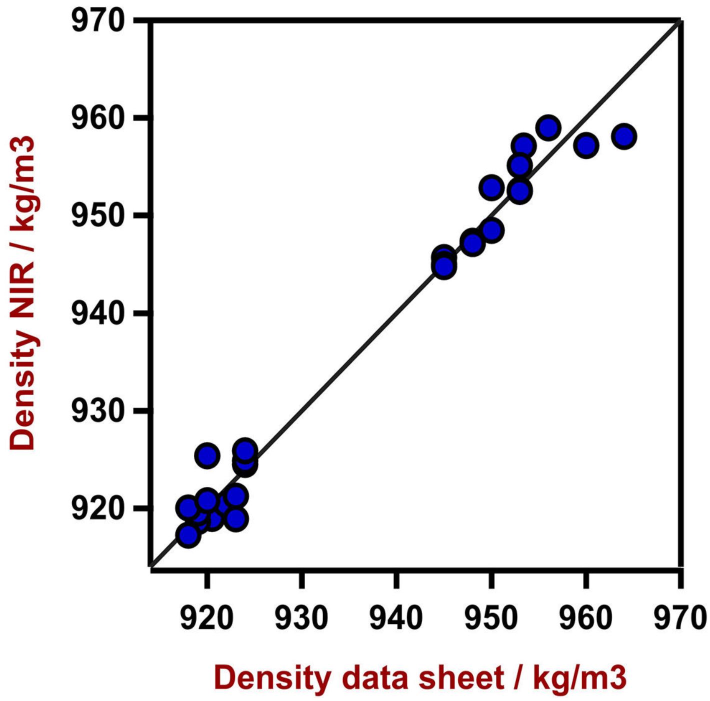

Figure 3.

Figures of Merit	Value
R^2	0.979
Standard Error of Calibration	2.48 kg/m^3
Standard Error of Cross-Validation	3.42 kg/m^3

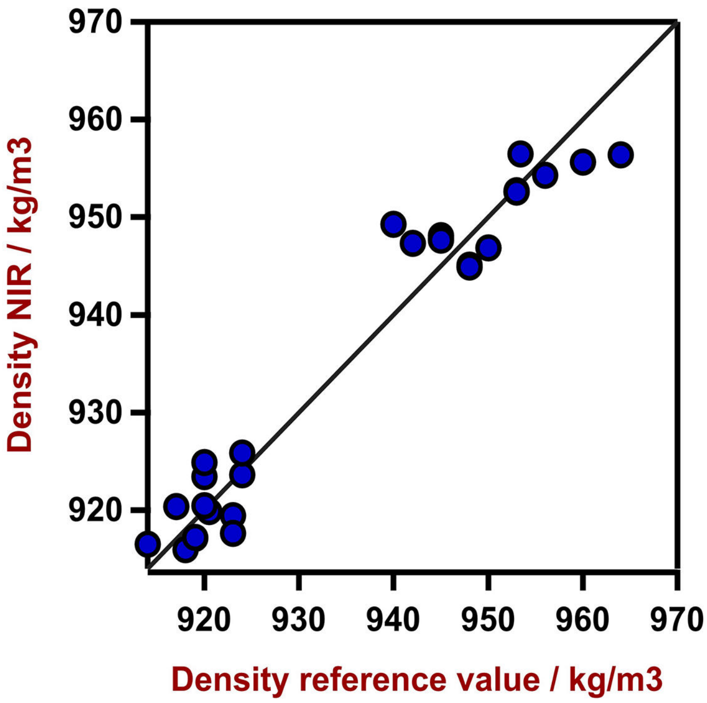

Figure 4.

Figures of Merit	Value
R^2	0.948
Standard Error of Calibration	3.95 kg/m^3
Standard Error of Cross-Validation	6.00 kg/m^3

In addition to the NIRS analysis, the density of the pellets was measured with the density balance in the laboratory. These results deviated even more from the reference values of the supplier, compared to the NIRS results (Table 2). This can be explained due to the appearance of air bubbles in some of the polymer pellets, visible in the CT scan displayed in Figure 5. The respective figures of merit (FOM) of the NIRS analysis related to the reference data from the polymer production facility is displayed in Figure 3. The correlation of the density balance measurements performed in the lab with the predicted NIRS analysis is displayed in Figure 4.

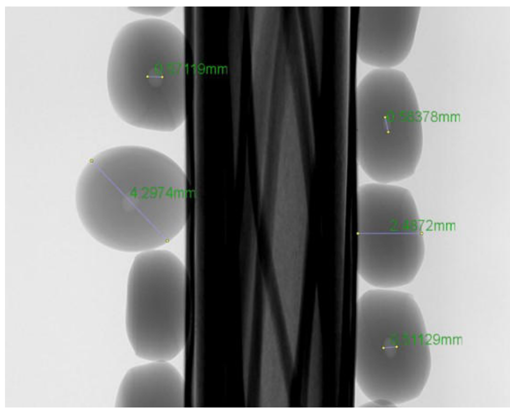

Figure 5. Example of computer tomography (CT) scan of polyethylene pellets showing air bubbles inside the polymer granulate.

CONCLUSION

This Application Note shows the feasibility of NIR spectroscopy for the analysis of density in polyethylene granulates. Compared to the standard method (Table 2), NIRS analysis shows a lower prediction error when air bubbles are present in polymer pellets. In addition, sample handling with near-infrared spectroscopy is easier to perform and therefore less error-prone.

Table 2. Comparison of density prediction with NIRS and density balance according to ASTM D792.

	Density: producer	Density: lab balance	Density: NIRS	Air bubbles present
Sample 1	953 kg/m^3	941 kg/m^3	952 kg/m^3	Yes
Sample 2	950 kg/m^3	935 kg/m^3	953 kg/m^3	Yes
Sample 3	918 kg/m^3	917 kg/m^3	915 kg/m^3	No

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CONFIGURATION

DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.

The DS2500 Analyzer is the tried and tested, flexible solution for routine analysis of solids, creams, and optionally also liquids along the entire production chain. Its robust design makes the DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.

The DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The DS2500 Analyzer meets the demands of the pharmaceutical industry and supports users in their day-to-day routine tasks thanks to its simple operation.

Thanks to accessories tailored perfectly to the instrument, optimum results are achieved with every sample type, no matter how challenging it is, e.g. coarse-grained solids such as granulates or semi-solid samples such as creams. The MultiSample Cup can help improve productivity when measuring solids, as it enables automated measurements of series containing up to 9 samples.

DS2500 large sample cup

Large sample cup for the spectral recording of powders and granulates in reflection at various sample positions using the NIRS DS2500 Analyzer.