Application Note AN-NIR-102

Densité des polyoléfines mesurée par spectroscopie dans le proche infrarouge

Analyse de routine simple des granulés de polymère

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).

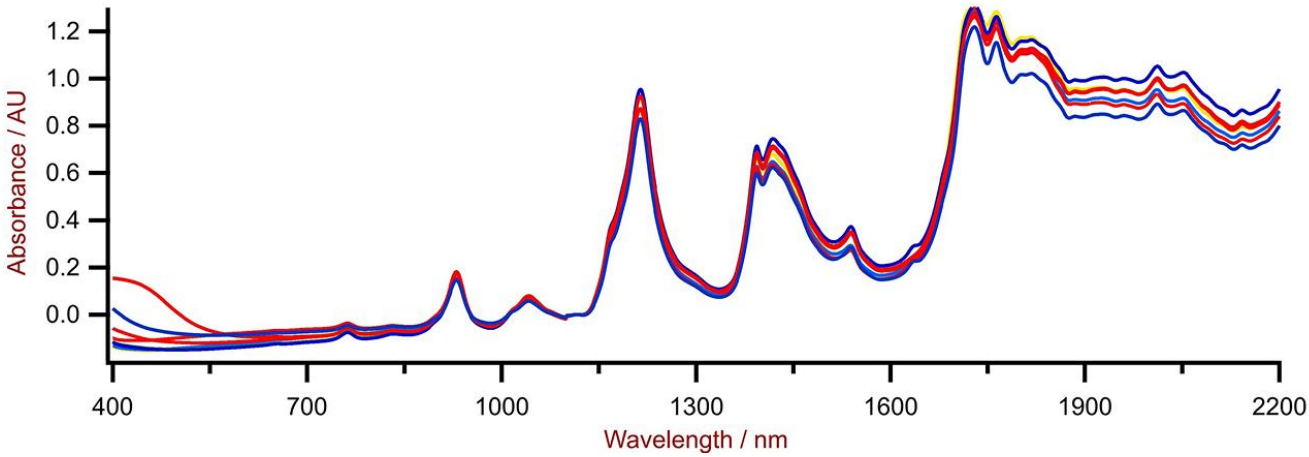

Figure 2.

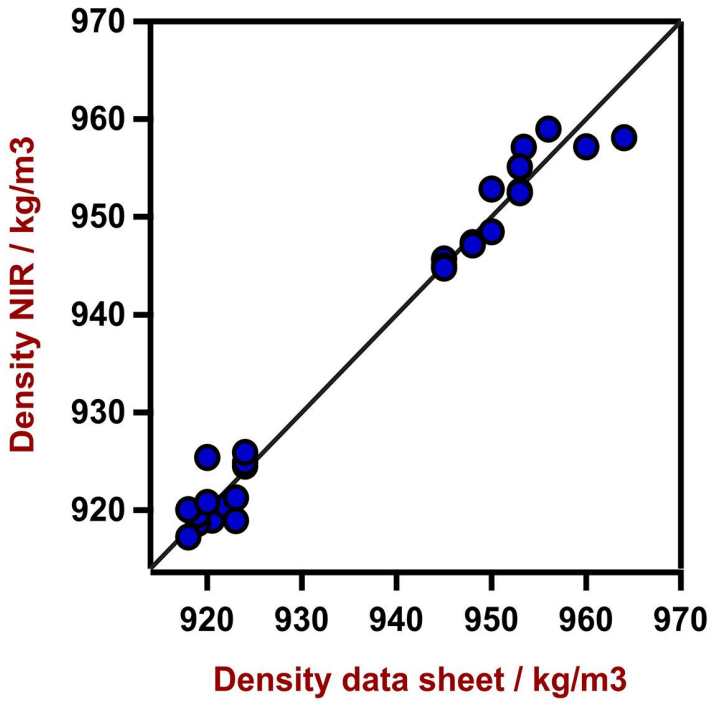

Figure 3.

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

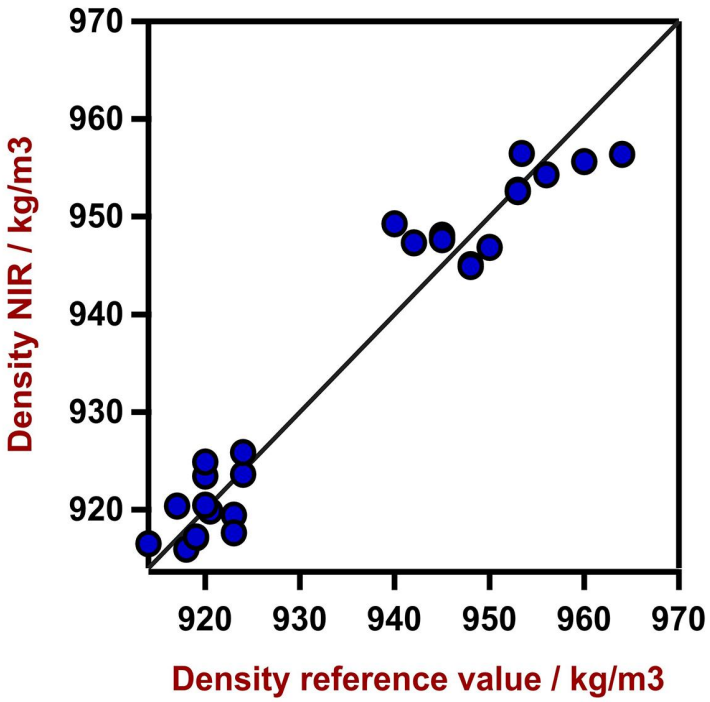

Figure 4.

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

RESULT DENSITY IN PE

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.

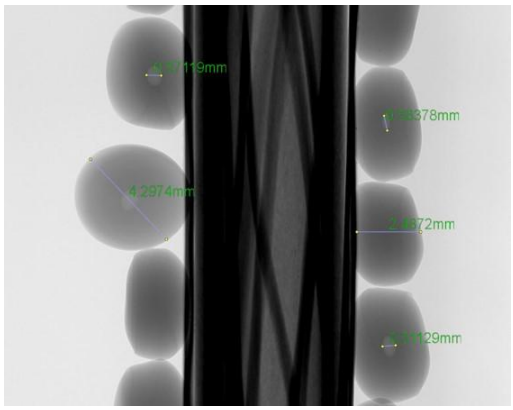

Figure 5. Example of computer tomography (CT) scan of polyethylene pellets showing air bubbles inside the polymer granulate.

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

Spectroscopie proche infrarouge robuste pour le controle de la qualité en laboratoire et en environnement de production.

L'analyseur DS2500 Analyzer est la solution éprouvée et souple destinée aux analyses de routine de matières solides, de crèmes et, en option, de liquides, tout au long de la chaine de fabrication. Sa conception robuste fait du DS2500 Analyzer un appareil insensible à la poussière, à l'humidité, aux vibrations ainsi qu'aux variations de température, et donc particulièrement adapté aux rudes conditions d'un environnement de production.

Le DS2500 couvre l'ensemble de la gamme spectrale de 400 à 2 500 nm et fournit des résultats exacts et reproductibles en moins d'une minute. Le DS2500 Analyzer répond aux exigences de l'industrie pharmaceutique et représente une aide précieuse pour les opérations de routine quotidiennes grace à sa simplicité d'utilisation.

Grace à des accessoires parfaitement adaptés à l'appareil, il atteint des performances sans précédent avec tous les types d'échantillons, quel que soit le défi qu'ils opposent (matières solides à gros grains comme les granulats ou échantillons semi-solides ou liquides telles les crèmes). La productivité lors de mesures de matières solides peut encore etre augmentée par l'utilisation du MultiSample Cup, lequel permet des mesures automatisées en série jusqu'à un maximum de 9 échantillons.

DS2500 Récipient d'échantillon, grand

Grand récipient d'échantillon pour l'enregistrement de spectre par réflexion de poudres et de granulés en différents points de l'échantillon avec le NIRS DS2500 Analyzer.