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).

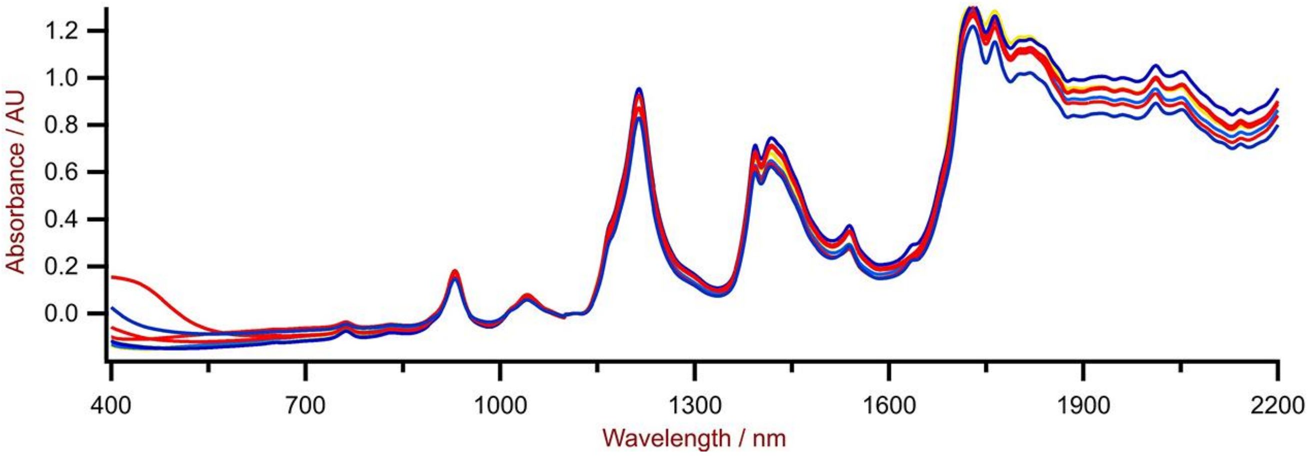

Figure 2.

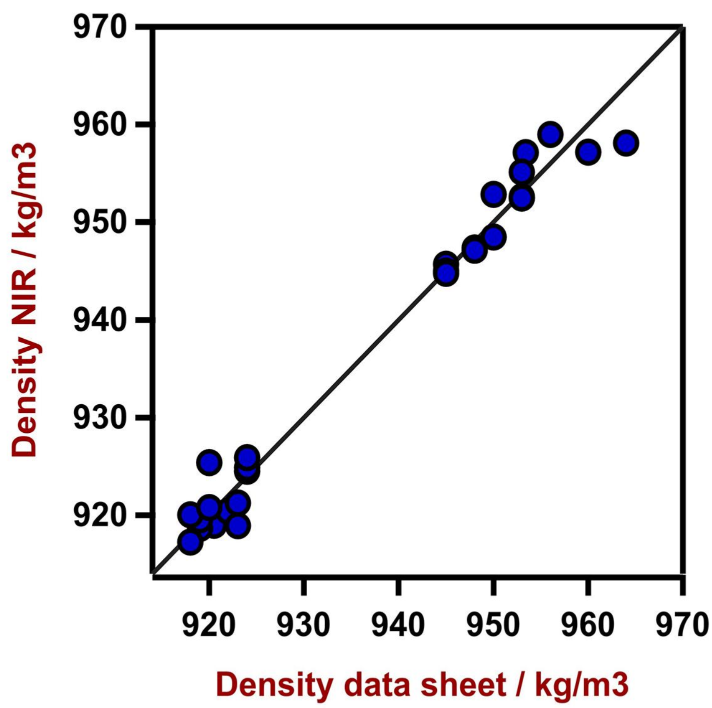

Figure 3.

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

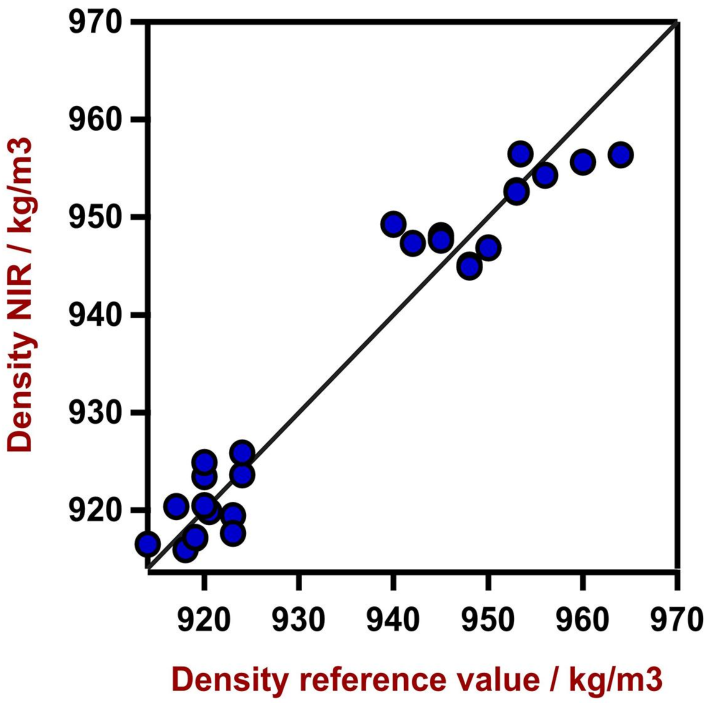

Figure 4.

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**.

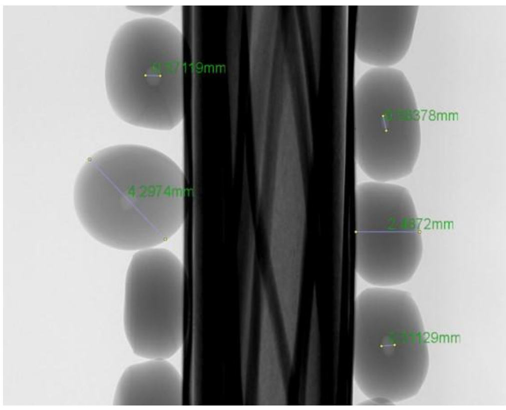

Figure 5. Example of computer tomography (CT) scan of polyethylene pellets showing air bubbles inside the polymer granulate.

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

CONTACT

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CONFIGURATION

DS2500 Solid Analyzer

ラホおよひ生産環境における品質管理用の堅牢な近赤外分光法。

DS2500 Analyzerは、生産チェーン全体に沿った固形物、クリーム、およひオフションとしての液体のルーチン分析に実績のあるフレキシフルなソリューションてす。頑丈な仕様により、DS2500 Analyzerは粉塵、湿気、振動や温度変動に強い為、過酷な生産環境ての使用に理想的てす。

DS2500は400 ~ 2500 nmのスヘクトル範囲全体をカハーし、1分以内に正確て再現性の高い結果を提供します。DS2500 Analyzerは製薬業界の要件を満たしており、簡単な操作により日常的な作業においてユーサーをサホートします。

装置に完全に適応した付属品により、 顆粒のような粒の荒い固形物、またはクリームのような半固形液体サンフルなとのあらゆる困難なタイフのサンフルにおいても、最良の結果を得ることかてきます。固形物の測定においては、9つまてのサンフルのシリースの自動測定を可能にするMultiSample Cupを使用することて、生産性を高めることかてきます。

DS2500

NIRS DS2500 Analyzerを用いた、様々なサンフル位置における反射中の粉末および顆粒のスヘクトル記録のための、大きなサンフル容器てす。