

Application Note AN-NIR-102

用近红外光谱法测量聚烯烃的密度

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).

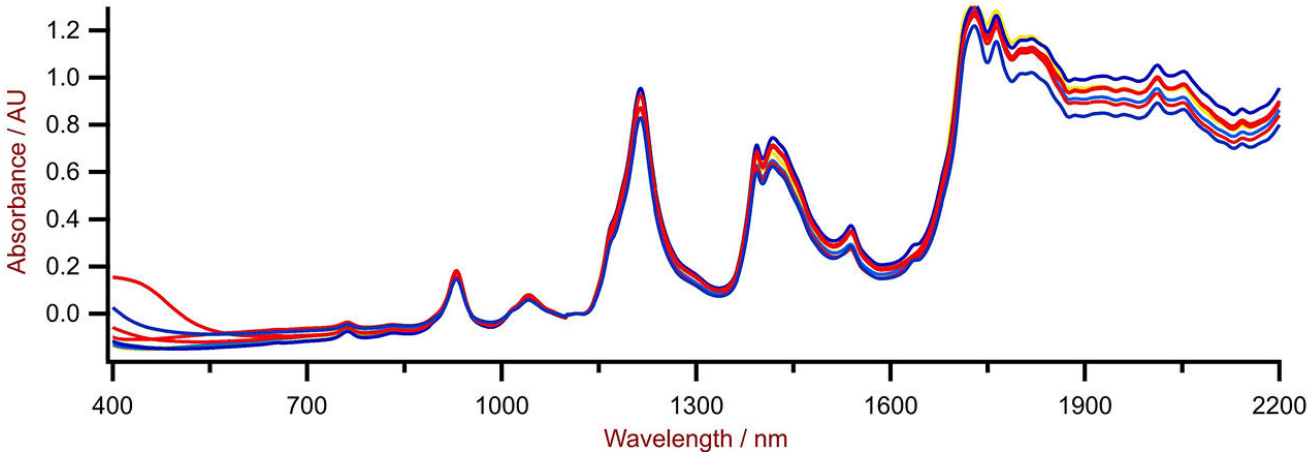


Figure 2.

RESULT DENSITY IN PE

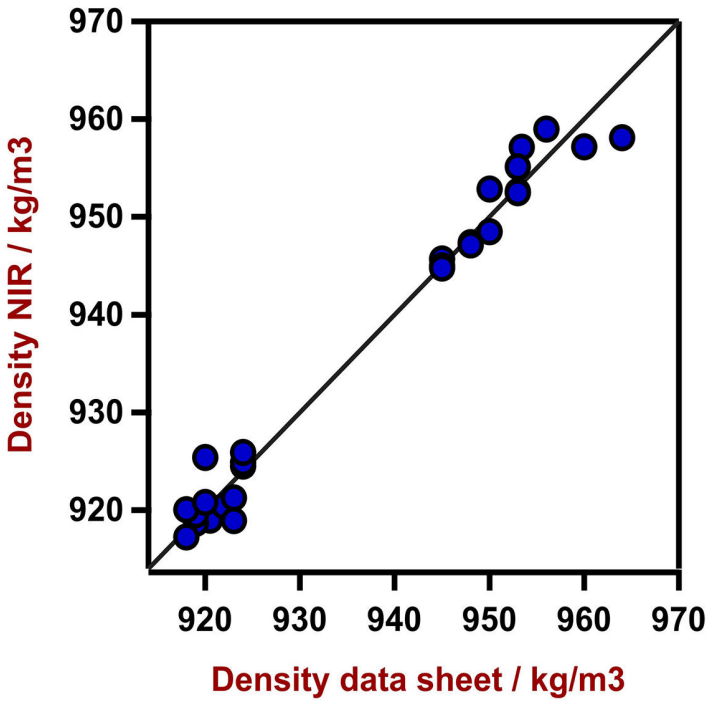


Figure 3.

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

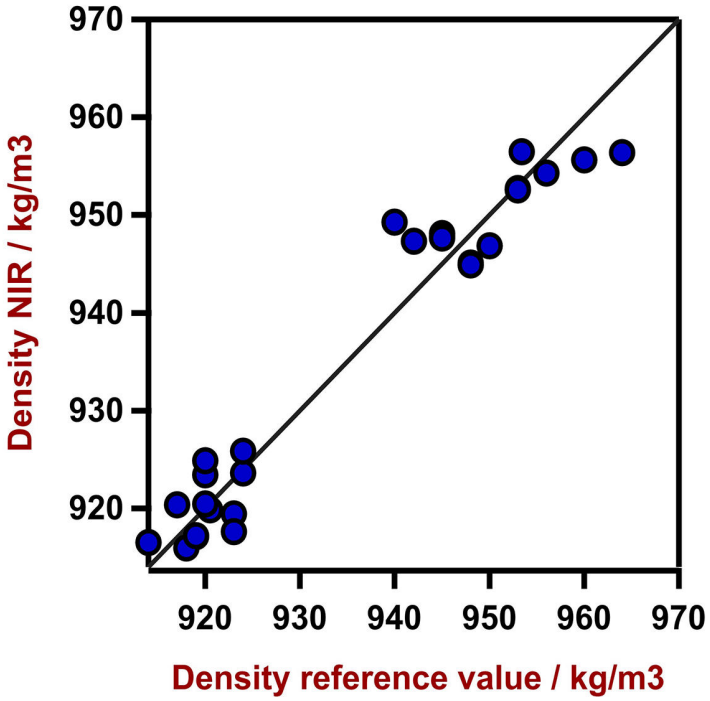


Figure 4.

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.

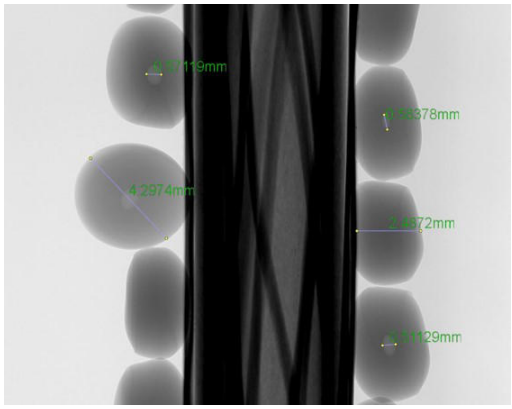


Figure 5. Example of computer tomography (CT) scan of polyethylene pellets showing air bubbles inside the polymer granulate.

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

CONTACT

Metrohm Taiwan Ltd.
開封街二段 46 號 5 樓
108 台北市
info@metrohm.com.tw

CONFIGURATION



DS2500 Solid Analyzer

坚固结实的近红外光谱仪,用于生产环境和试验室中的质量检查。

DS2500 分析仪是经过验证的灵活解决方案,用于对整个生产链过程中的固体、乳膏和液体进行常规分析。其坚固结实的设计使 DS2500 Analyzer 分析仪不受灰尘、湿度、振动和温度波动的影响,因此非常适用于在恶劣的生产环境中使用。

DS2500 涵盖了从 400 到 2500 nm 的整个光谱范围,并能在不到一分钟时间内提供准确和可再现的结果。DS2500 Analyzer 满足制药行业的要求,并由于操作简便而能帮助用户完成其日常工作任务。

由于与设备匹配,附件可以承受任何具有挑战性的样品类型,例如:粒料之类的粗颗粒固体或乳膏之类的半固体样品,可获得结果。测量固体的时候,使用 MultiSample Cup 可以提高生产率,可以自动批量测量多 9 个样品。



DS2500

用于在不同样品位置处使用 NIRS DS2500 Analyzer 采集粉末和颗粒反射光谱的大号样品容器。