LexMarGlobal

APPLICATION NOTE

POLYOLEFINS

Density of Polyethylene

SUMMARY

The global polyolefin industry produces polymeric materials which have, over the years, replaced many traditional materials such as wood, steel, and ceramics. Polyolefin plants are adapting with more advanced technology to improve efficiency, reduce transition time, and tailor products to meet the ever-increasing demand on mechanical requirements. A fundamental parameter for the production of polyethylene (PE) is the resin's density. Density is directly related to the crystalline packing of the polyethylene molecule chains. Chain length and molecular branching affect this packability. Rapid monitoring of density has been shown to increase on-spec yield and reduce reactor transition times.

Analysis has traditionally been performed via displacement or the gradient column technique (ASTM D792 or ASTM D1505, respectively). However, these methods require time-consuming setups and are prone to errors due to operator dependencies.

Magneflow Industrial Magnetic Resonance (IMR) analyzers from LexMar Global Inc. provide faster, more robust, and reliable solutions for the measurement of density in polyethylene.

LexMar Global Inc. is the world's leading supplier for IMR technology to the polyolefin industry. The company provides robust laboratory instrumentation (MagStation™ II) and is the only manufacturer of fully automated online process equipment (MagModule™ II) used by the world's most efficient polyolefin resin producers.

TECHNOLOGY OVERVIEW

The Magneflow product line utilizes Industrial Magnetic Resonance techniques for the analysis of polyethylene materials. In IMR, a sample is positioned in a strong magnetic field. The nuclei within the sample begin to orient into multiple population states due to the presence of this external magnetic field. This orientation yields a small net magnetic moment within the sample. A short radio-frequency pulse is applied to the sample which rotates this magnetic moment. As the magnetic moment returns to equilibrium after the pulse, the sample emits a Free Induction Decay (FID) signal. This FID signal contains information about the morphology of the material. If multiple morphological domains exist within a single sample, the FID can be modeled as the sum of multiple smaller components. For polyethylene, this approach allows for direct measurement of crystalline, amorphous and interfacial domains within a given sample.

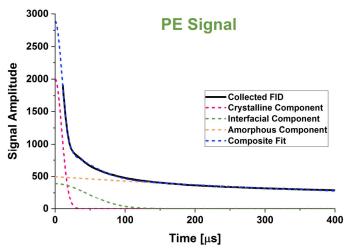


Fig 1. Low density polyethylene FID signal fit to characterize and extract quantitative information from the crystalline, interfacial and amorphous domains.

PROCESS OPTIMIZATION

Polyethylene producers successfully utilize real-time feedback from the MagModule™ II online analyzer to continually monitor density during product transitions. This feedback allows for responsive adjustment of process conditions which minimizes transition times and virtually eliminates target overshoot.

These same producers also continuously monitor density results post transition. This allows plant controllers to utilize closed-loop feedback to optimize reactions and maximize product consistency.

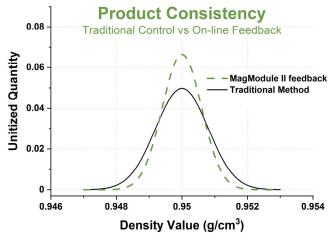


Fig 2. Product consistency variation control utilizing MagModule II online feedback. This real-time feedback enables the production of a higher quantity of on-spec materials and a lower quantity of off-spec materials.

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CALIBRATION AND RESULTS

The laboratory or online Magneflow IMR analyzer is typically calibrated against traditional wet chemistry analysis. It is the customer's choice whether the calibration is performed by LexMar Global Inc. or locally by the polyethylene plant staff. Linear calibrations are easy to generate with a limited number of reference samples as long as the entire product range is represented in the sample set. The calibration models, once established, are stable, typically for years, and only in rare cases require adjustment. The following figures demonstrate typical calibration performances for the Magneflow product line.

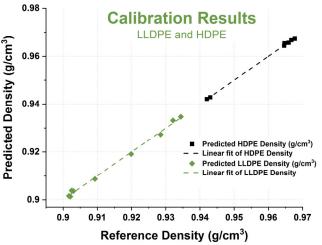


Fig 3. Magneflow technology density predictions for HDPE and LLDPE compared with lab reference data. Independent HDPE and LLDPE calibrations have been plotted together to demonstrate linearity.

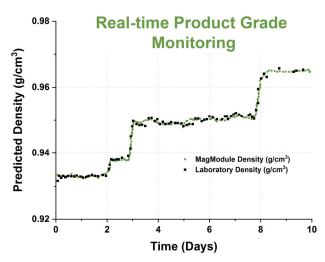


Fig 4. Ten-day trend of online density measurements made by a MagModule II alongside the results reported by the on-site laboratory. The process transitioned through four different product grades. *Note that on day 8 the MagModule II shows the process has lined out before the laboratory results – reducing the potential waste of prime material.*

SAMPLING

Both reactor powders and extruded pellets are sampled from the process for Magneflow IMR analysis. For online analysis, a customized sample extraction panel automatically collects a sample as close to the reactor as possible and conveys it directly to the MagModule II. This happens typically within minutes after polymerization. Conversely, for offline IMR, samples are manually collected and loaded in state-of-the art sample tubes; engineered to expedite sample conditioning and analysis. The samples (25 g) are analyzed using the Magneflow IMR instrument in approximately 10 minutes to provide an accurate density determination. No sample preparation is required and there is no need for gradient column solutions, weighing, or drying. Since the Magneflow IMR technology measures 100% of the sample in the testing probe, the analysis is virtually unaffected by additives, color, product homogeneity, and other matrix effects.

BENEFITS & SERVICES

- Rapid and accurate density analysis of HDPE, LLDPE, and LDPE
- Representative sampling for the most accurate results
- Non-destructive analysis
- No necessity for sample preparation
- Simple, highly repeatable analysis
- Operator independent results
- Solvent-free analysis
- Pre-loaded methods and analysis routines
- Results are fully compatible with DCS and LIMS systems
- Customized solutions for hazardous and non-hazardous installations (MagModule™ II)
- Fully automated 24/7 analysis (MagModule™ II)
- Large sample volumes (25 mL) ensure proper sample

FURTHER INFORMATION

In addition to the analysis of polyethylene density, the MagModule™ II is capable to provide trending information for melt flow rate (MFR.)

Other polyolefin application offerings include:

 Analysis of Polypropylene (PP) homo and copolymers measuring xylene solubles content (XS), ethylene content (C2), flex modulus, and Charpy

In addition to the MagStation™ II and MagModule™ II LexMar Global proudly offers also the following analytical equipment:

- MagStation[™] Lite Benchtop IMR
- Correflow™ 5430, Correstat™ 5410, Correflux™
 5420 Electrostatic Reaction Monitoring



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