Xylene Soluble Content in Polypropylene

Process Monitoring and Quality Control | Application #17

The term 'Xylene Solubles' refers to the percentage of soluble species in homo- and copolymers of polypropylene. Its value correlates to the R21 value and to the amorphous content of the polymer. For polypropylene homopolymers, R21 is a measure of tactility parameters like stiffness and hardness. Investigating copolymers, R21 additionally corresponds to comonomer related parameters such as transparency and gloss. Its measurement is widely used to control the polymerization process and to determine the physical properties of final products for Quality Control.

Historically the xylene soluble content (ISO 16152, ASTM 5492) has been measured by extraction.

With this time consuming method, a direct controlling of the polymerization process is not feasible. In contrast, the TD-NMR application is fast, operator independent and requires no harmful solvents or dedicated lab-space.

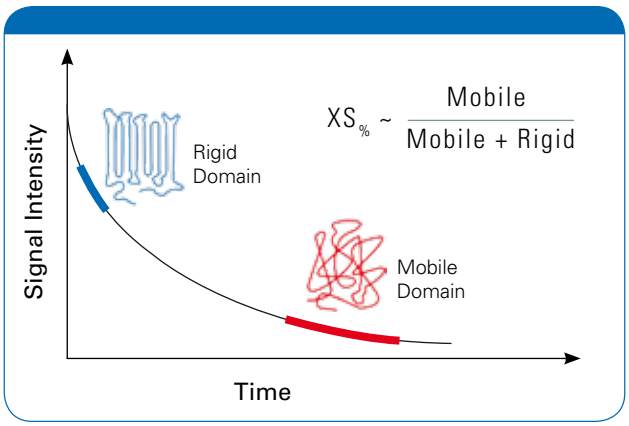
Features and Benefits

- Polymerization process control *at line*
- Minimal sample preparation (pellets and powders are measured *as is*)
- Only 25 minutes from sample taking to validated result
- Daily check instrument validation

Application Method

The Xylene Solubles (XS) application is based on different relaxation times in the rigid and mobile domains of the polymer. Protons in rigid domains return significantly faster to the equilibrium state than protons in mobile domains. Measuring the decay signal at two characteristic times provides a ratio that corresponds with the xylene soluble content.

Measurement



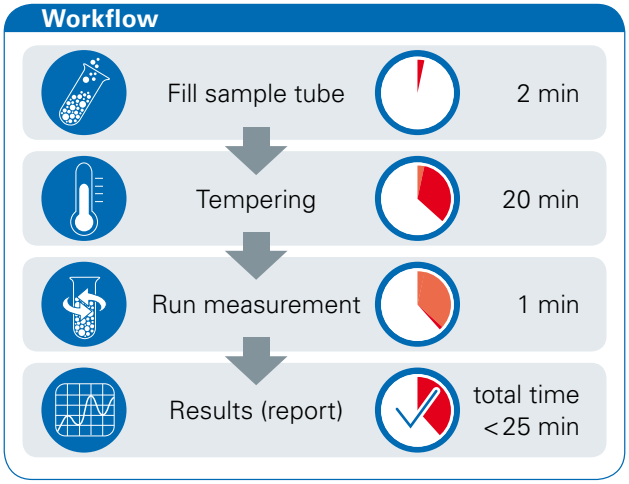
Calibration

The quality of the calibration is generally limited by the accuracy of the reference data and sample homogeneity.

- Calibration range from 0.9 – 30 % xylene soluble content
- Typical correlation factors between 0.94 and 0.9987
- Typical mean standard deviation of repeated measurements below 0.05 wt %

The method is calibrated with 3 to 5 samples of known xylene soluble content. NMR values (ratios) are related to xylene solubles by linear regression. Bruker-certified standard samples are provided for back-up calibration.

Workflow



Recommended Equipment

- minispec mq-one Polymer Analyzer for routine measurements
- minispec mq20 series for R&D and routine measurements
- Tempering bath and/or tempering block
- Automation (optional)

Beyond XS Content in Polypropylene

- The XS method is easily adaptable to other extractables such as decaline or heptane solubles
- Other polyolefins such as polyethylene and polystyrene can be analyzed accordingly
- For samples containing small amounts of xylene solubles a weight normalized measurement (balance required) is available

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