Extract content of polyamides

1 Question

The question is frequently raised regarding the residual monomer or extract content in a PA 6.

In fact, residual monomer content and extract content are two different issues. In the case of PA 6, the question of residual monomer content refers only to the free caprolactam, while the extract content can include all extractable components in the plastic. Therefore, in addition to caprolactam, it also encompasses its oligomers, i.e. dimers, trimers, tetramers, etc., as well as any possible additives.

The numerical values depend greatly on the selected measuring method. The most critical aspect in this context is the nature of sample preparation.

2 Measuring methods

All measuring methods used in practice separate the polymer from the low-molar fractions, either by dissolving them from the plastic with a solvent or by dissolving the entire sample and precipitating the high-molar fractions. Finally, the quantity of extract is determined and then compared to the original sample quantity.

The differences lie in the details of procedure and in the substances detected as extract. For example, differences exist in the:

- Samples (whole or ground granules, the size and shape of the particles also plays a role)
- Solvent (methanol, water, ...)
- Temperature and duration of extraction
- Temperature, pressure and duration of rotary evaporator
- Temperature, pressure and duration of vacuum drying in the drying cabinet
- Analysis of extract quantity (e.g. concentration by evaporation of the solvent or spectroscopic methods)

to name but a few.

All of these, and possibly other parameters must be specified in order to effectively interpret the numerical values. Different solvents or particle sizes, for instance, result in significantly different values.

Although standards exist to govern these preparation and measurement procedures, they often leave a certain degree of latitude, or are carried out in different ways in practice. Deviations of this kind are not specified at all in some cases, or at most by the reference "...based on..."

Extraction often is conducted on whole granule particles (pellets). This results in the soluble components near the surface being detected, but not those in the interior of the granules. They remain in the granules and are not included in the analysis.

To avoid this situation, the granules can be ground into a defined type of powder, making it possible to much more effectively capture these interior regions of the particles as well. The quantity of substance extracted is significantly larger as a result. This preparation method is prescribed by EN ISO 6427.

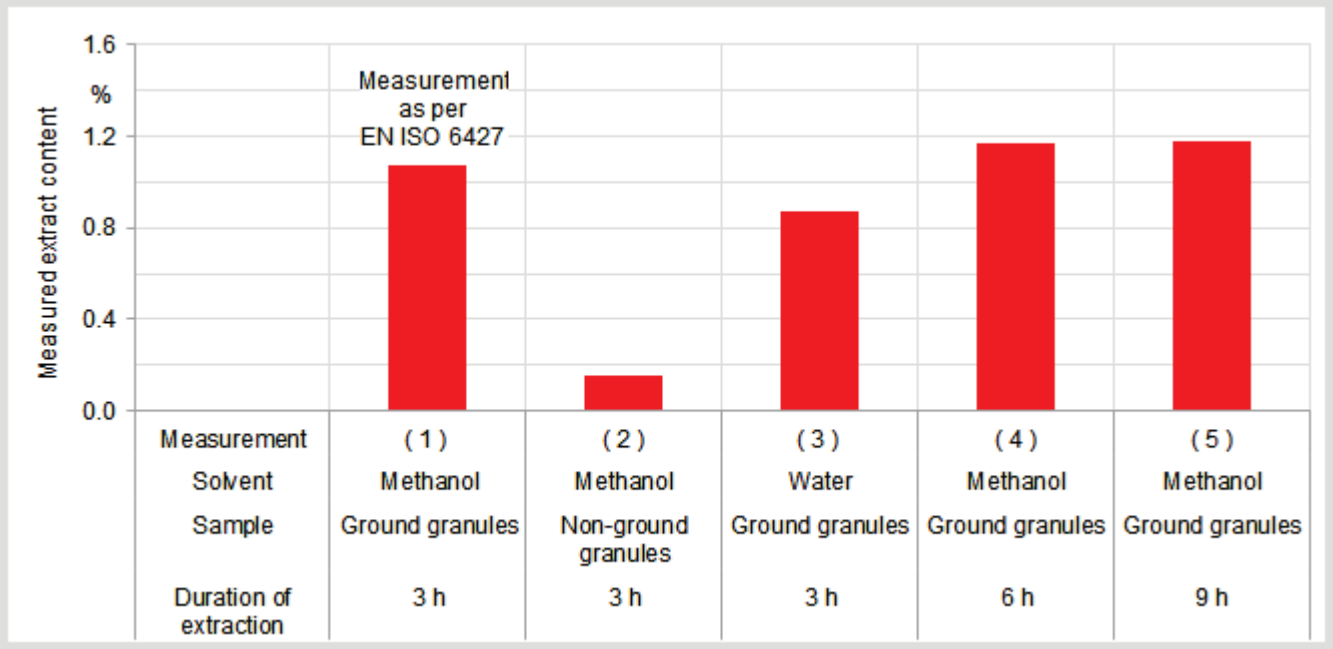

Fig. 1 Extract content, measurement results using different methods

Fig. 1 compares the results of measurements on a uniform granule sample, but using different methods.

Most noticeable here is the difference between the measurement results for the ground (1) and non-ground (2) granules. The ratio of measurement values of 1 : 7, as determined here, can deviate significantly in some cases. It depends on numerous parameters not discussed in detail here.

The selected solvent also influences the measurement results. In this case, water is a poorer solvent for the extracted substances, so the measurement results obtained with water (3) are somewhat lower than those with methanol (1).

The measurement results obtained under EN ISO 6427 must not be viewed as the absolutely correct answer to the question of “extract content,” because this method most likely does not capture the entire content of extractable substances. Extending the extraction duration – e.g. from 3 h as specified in the standard to 6 h (4) – slightly increases the extract content. However, extending it again to 9 h no longer improves extraction (5). On the other hand, this confirms that no artifact – such as a breakdown of the polymers into low-molecular substances – is falsifying the measurement result.

3 Comparison of various products

The next figure, Fig. 2, shows which extract contents are actually found in the materials based on measurements to EN ISO 6427 of our Durethan® and products from other manufacturers.

It must be emphasized in this case that the measured values shown are not typical values, but rather the result of an individual measurement.

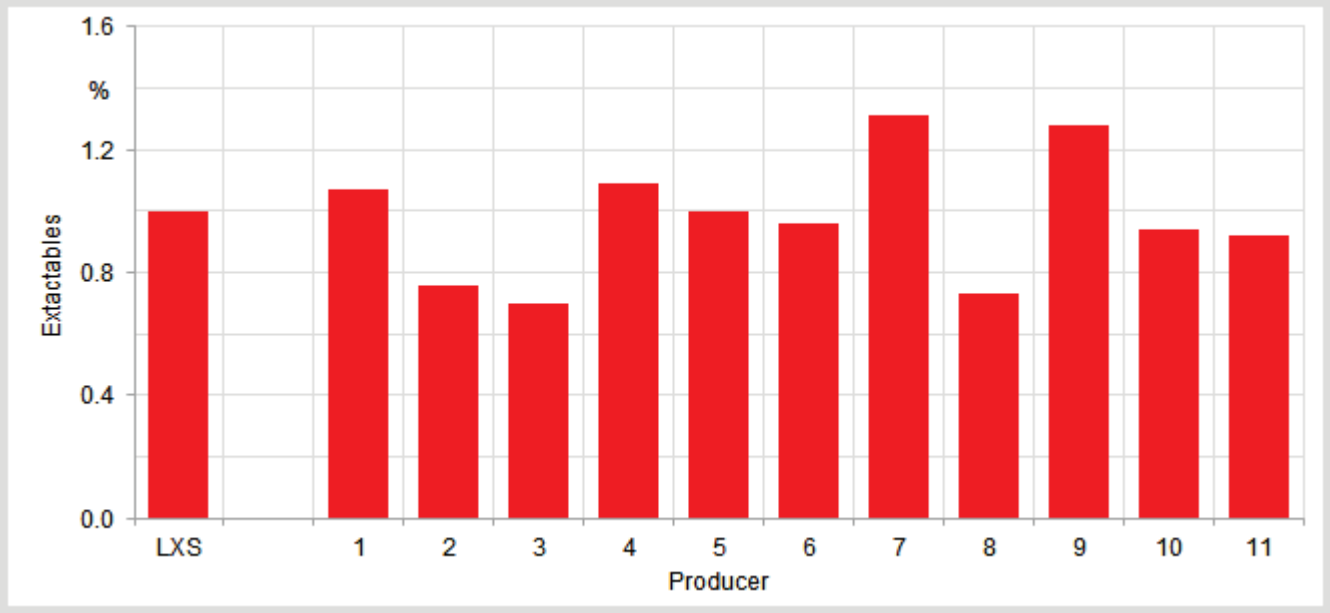

Fig. 2 Extract content, measurement results using different methods

The figure shows that the extract content of LANXESS products is comparable to that of products from other manufacturers. Many of these manufacturers indicate a value of 0.6% without specifying their measurement and preparation methods. Some expressly specify that the test sample involved non-ground granules. It is notable that, in all samples an extract content of more than 0.6% was found using EN ISO 6427.

These results again underline the fact that precise knowledge of the measurement method is important for interpreting the results. Similarly, a comparison of measurement values cannot provide useful information without detailed knowledge of the method.

For PA 6 materials produced according to state-of-the-art processes, we expect extract contents of over 0.6% when tested in accordance with EN ISO 6427. In contrast, lower values indicate the use of other parameters or methods, and should therefore not be used for a direct quantitative comparison with values measured by this standard.

LANXESS regularly measures the extract contents of its PA 6 resin as part of monitoring and controlling its production process. The extract contents typically are between approx. 0.8 to 1%, measured in accordance with EN ISO 6427, i.e. ground granules (0.5 – 0.7 mm) and with extraction for 3 hours with methanol.

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Medical
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Note:
The information contained in this publication is current as of November, 2016. Please contact LANXESS Corporation to determine if this publication has been revised.