REAL TIME DETERMINATION OF MORPHOLOGIC AND MECHANICAL PROPERTIES OF POLYMER NANOCOMPOSITES IN THE MELT BY SPECTROSCOPIC AND ULTRASONIC METHODS

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ABSTRACT

Extrusion is one of the most applied technologies for polymer processing to create polymer nanocomposites (NC) for application in automotive, electrical and packaging industrial sectors. These nanostructured materials have great advantages in comparison to traditional polymer materials, so that properties like tensile strength and modulus, barrier and surface properties, electrical properties and flame retardency will be improved.

Principal points of this paper are the real time determination of particle size, dispersion and mechanical properties of polymer NC in the melt by Ultrasonic measurements and NIR spectroscopy during extrusion¹⁻⁴. For the correlation of these in-line data to morphologic and mechanical properties we used different off-line techniques like transmission electron microscopy (TEM), rheology, RAMAN-Imaging and mechanical test procedures. The polymers used are polypropylene (PP) and polyamide 6 (PA6) with different modified layered silicates as nanofillers and different nanoparticles. All experiments were done at twin screw extruders with measuring adapters at the die and side outlet adapters at different positions along the extruder. All adapters can be assembled with sensors for ultrasonic measurements, for NIR, UV/VIS and RAMAN spectroscopy.

A crucial point for NC is the determination of the degree of exfoliation as an indicator for the dispersion of the nanofiller in the polymer matrix. Our concept was to measure the shear thinning exponent (STE) from shear viscosity measurements as a quantitative degree for the state exfoliation. The STE was than correlated with in-line NIR and Ultrasonic measurements using multivariate data analysis to predict the shear thinning exponent. After this calibration, we monitored with both methods the NC and we could determine the degree of exfoliation for different modified layered silicates in PP and PA6. The investigation of the NC at different process conditions like screw speed, temperature and output could show the strong influence of these changes on the morphology of the NC.

Furthermore we investigated the dispersion process in real time along the extruder at different measurement points. The NIR and the Ultrasonic data at these different positions illustrate the improvement of the exfoliation of the layered silicates in the PP matrix along the extruder.

Based on experiments on labscale extruders, we could establish a successful scale-up of both methods to an industrial extruder to control concentration and the degree of exfoliation of layered silicates in PP during the production process of polymer nanocomposites.

References:

- (3) J. Müller, S. Kummer, D. Fischer Meas. Sci. Technol. 2009, 20, 097002.
- (4) D. Fischer, S. Kummer, J. Müller, B. Kretzschmar Macromol Symp 2011, in press.

⁽¹⁾ I. Alig, D. Fischer, D. Lellinger, B. Steinhoff *Macromol Symp* 2005, 230, 51-58.

⁽²⁾ D. Fischer, K. Sahre, M. Abdelrhim, B. Voit, V.B. Sadhu, J. Pionteck, H. Komber, J. Hutschenreuter, *Compt Rendus Chem* **2006**, *9*, 1419-1424.