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In-line Monitoring of the Dispersion of Nanofillers in Polymer Composites using Vibration Spectroscopy, Ultrasonic Measurements, In-Line Rheology and Dielectric Spectroscopy

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Nanomaterials often have properties that are superior to conventional microscale composites. Therefore the development of such materials is currently a research area of great interest. In polymer nanocomposites the particles and the macromolecules are mixed on a nanometer-length scale. The nanoscale dispersion in the polymer matrix can improve such properties as optical clarity, strength, stiffness, thermal stability, reduced permeability, or flame retardancy. The possibility of material improvement by adding small amounts of nanofillers has triggered an enormous interest in the commercialization of polymer nanocomposites for a variety of applications, and several of these applications are expected to be successful in the near future.

The key for the success of these applications is to guarantee nanoscale dispersion in the polymer matrix. For effective processing of noanocomposites reliable, rapid and stable process control during extrusion is needed. In order to evaluate the potential of different in-line methods for determination of the degree of dispersion in the extrusion process we tested rheometry, ultrasonics, near infrared (NIR), Raman and dielectric (conductivity) spectroscopy. Therefore recently developed melt-at-dies equipped with different combinations of sensors/probes have been adapted to the end of single and twin-screw extruders. In order to test the sensitivity of the different in-line methods for different types of nanocomposites we have performed experiments on several pretreated montmorillonites with polypropylene and ethylene-vinylacetate and conductive nanocomposites containing carbon black and carbon nanotubes.

The composites based on montmorillonite were extruded through a slit die containing pressure transducers for in-line rheology, ultrasonic transducers, Raman and NIR transmission and/or reflection probes to measure ultrasonic velocity, ultrasonic attenuation, Raman and NIR absorbance/reflectance spectra. A combined NIR/Raman process spectrometer (Sentronic, Germany) and high temperature/high pressure probes where used for the optical measurements. For the ultrasonic measurements we used a setup with PC pulser/receiver plug-in cards. Correlations between the recorded in-line data, especially for the NIR-spectra, and the degree of dispersion of the filler in the polymer melt have been found. To calibrate and validate the in-line methods, off-line transmission electronic microscopy and X-ray scattering was used.

A slit die with dielectric sensors combined with in-line rheometry and ultrasonic transducers was applied to monitor the extrusion of carbon black or carbon nanotube filled polyethylene, polypropylene and/or polycarbonate (carbon black content 0 to 40%, carbon nanotube content 0 to 20%) using an Agilent LCR bridge HP4284A. The sensitivity of the dielectric spectroscopy and conductivity measurements for determination of the carbon content and its distribution was very good, especially in the concentration range of percolation (about 15 vol.-% for carbon black and 1-5 wt-% for carbon nanotubes). The in-line experiments on conductive nanocomposites were compared to off-line dielectric experiments, electronic microscopy and X-ray scattering.