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Combination of NIR, Raman, Ultrasonic and Dielectric Spectroscopy for In-line Monitoring of the Extrusion Process

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The results of our recent activities in the field of extrusion process control^{1,2,3} by optical, ultrasonic and dielectric spectroscopy will be presented.

A slit die (Fig. 1) containing pressure transducers for inline rheology, ultrasonic transducers, Raman and NIR transmission and/or reflection probes was used to measure ultrasonic velocity, ultrasonic attenuation, Raman and NIR absorbance/reflectance spectra during the extrusion process. A combined NIR/Raman process spectrometer (Sentronic) and high temperature/high pressure probes were used for the optical measurements. For the ultrasonic measurements we used a setup with PC pulser/receiver plug-in cards.

To test the system, PE/PS blends ranging from 0% to 100% PS content were measured. The resulting spectra for each method were used to build up a calibration set using partial least squares (PLS) analysis. The calibration set can then be used to predict the concentration of an unknown composition. The resulting prediction errors are in the order of 1% (PS content) for NIR, 1% for Raman, and 2% for ultrasonic spectroscopy. Furthermore the system was applied to other blends (e.g. ABS/SAN), nano composites, and composites of PP/chalk.

Furthermore, a slit die with dielectric sensors was applied to monitor the extrusion of carbon-black filled PE (carbon black content 0 to 40%), using an Agilent LCR bridge HP4284A.

The sensitivity of this method for determination of the carbon black content and its distribution was very good, especially in the concentration range of percolation (about 20 wt %).

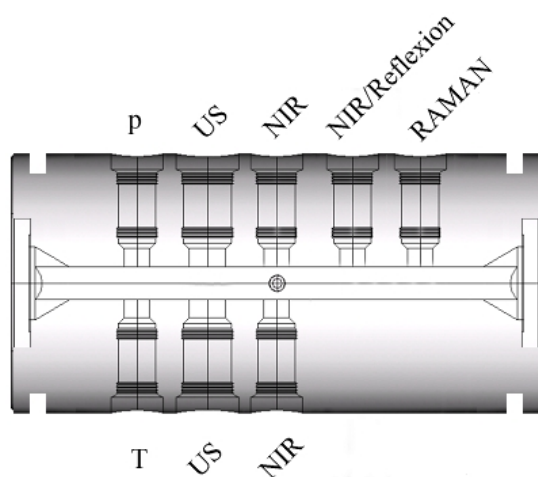


Fig. 1: Combined cell for inline monitoring containing Raman and NIR transmission and/or reflection probes, pressure (p), temperature (T) and ultrasonic transducers (US).

¹ I. Alig, D. Lellinger, R. Lamour, J. Ramthun, *Kunststoffe* **5** 2000, 96 (*plast europe* **5** 2000, 31)

² I. Alig, D. Lellinger, K. Wassum, *Kunststoffe* **6** 2003, 53 (*plast europe* **6** 2003, 24)

³ I. Alig, D. Lellinger, K. Wassum, *Kunststoffe* **12** 2003, 62 (*plast europe* **12** 2003, 33)