Residence Time Distribution Measurement via Video Analysis for Design of Experiment Characterization of Melt Extrusion

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The residence time distribution (RTD) is of importance for extrusion, because it strongly influences the product quality, e.g. degradation of the active pharmaceutical ingredient (API) or content uniformity. The first can be reduced by shorter residence times, while the latter improves with a broadening of the RTD, caused by mixing. Therefore in-line video analysis of a melt extrusion process was developed, which has been mainly used off-line up to now. It represents a robust and inexpensive solution, with equipment costs far below that of Raman or NIR, and without the need of calibration model development.

A video of the extrudate was taken with a camera positioned directly behind the die plate (see Figure 1, left). The analysis of the video consisted of four steps: (1) Segmentation of the image to mask the strands, (2) calculation of average color values for the segmented pixels of each frame, (3) fitting of the obtained color curve with an analytical solution [1] and (4) calculating several RTD measures (e.g. minimal and mean RT) for easier comparison of the results (Figure 1, center).

The calculated RTD measures were used as responses of a DoE, consisting of the process parameters: screw design, screw speed and throughput. As an example, the impact of screw speed and throughput on RT_{FWHM} (full-width at half maximum) is shown in Figure 1, right.

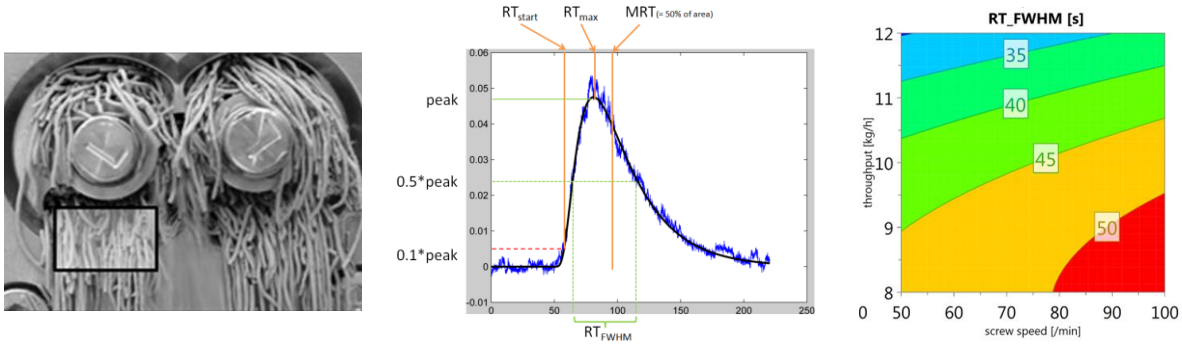


Figure 1: Left: Extrudate and die plate of the extruder. Analysis was performed within the rectangle area. Middle: The measured RTD curve was fit by an analytical solution [1], from which several important RT measures were derived. Right: DoE response for the RT measure full-width at half-maximum (FWHM).

[1] David B. Todd (1975), Residence time distribution in twin-screw extruders, *Polym. Eng. Sci.*, 15:437-443