Abstract
Simultaneous measurement of coating thickness and conversion of UV-cured acrylate coatings by in-line near infrared spectroscopy

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Introduction

UV curing of acrylate formulations is an efficient and versatile technology to produce polymer coatings with a wide range of potential properties. During technical curing processes, the conversion needs to be controlled continuously, since it may be influenced by a lot of parameters. NIR reflection spectroscopy was used for in-line monitoring of the conversion. However, unintended changes of the thickness of the coating, e.g. caused by variation of the web speed, were found to preclude the correct analysis of the conversion by chemometric methods. In order to correct the conversion data for such changes, two approaches were developed. The first one includes monitoring the variation of the thickness of the layer into the chemometrics calibration model by use of the PLS2 algorithm. This method requires enormous efforts for calibration, because the full range of both conversion and thickness, which can occur during analysis, has to be covered. An alternative method is based on separate spectra, which were recorded before, as well as after UV irradiation by use of two probe heads. The corrected conversion can be directly obtained from the ratio of the integrals of the band of the acrylic double bond at 1620 nm, according to the Beer-Lambert law.

Materials and methods

NIR spectra were recorded with two diode array spectrometers, which can be used in the laboratory (for calibration) as well as for process control. Spectra were taken in reflectance mode against a ceramic retroreflector, in the range from 1405 to 1950 nm. For in-line monitoring, the probe heads were mounted on a pilot-scale roll coating machine directly before and after the UV lamp. Clear and pigmented acrylate formulations were applied to 20 µm OPP foil and cured by UV

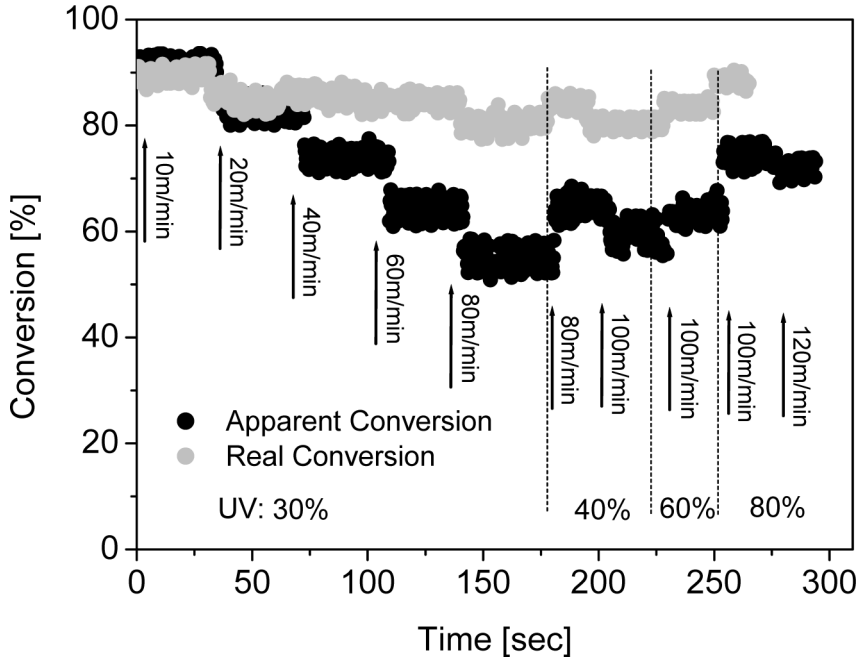

Figure 1. In-line monitoring of the conversion in clear acrylate coatings with one (●) or two (●) NIR probe heads, respectively (data analysis by band integration).

irradiation. Conversion and coating thickness of the calibration samples were determined by FT-IR transmission spectroscopy and a thickness gauge, respectively.

Results and discussion

Figure 1 shows the in-line monitoring of the conversion in an acrylic clear coat after UV irradiation.

Data were analysed with the band integration method. In order to simulate changes of the irradiation dose, both the power of the UV lamp and the line speed were varied repeatedly. The trace with the black symbols (●) was recorded with one probe head only, i.e. without correction of thickness changes (the reference value of the unconverted coating was recorded before the trial). In contrast, the other curve (●) was based on measurements with two probe heads, which shows much less variation. It is obvious that the conversion changes in the former case are mainly due to changes of the coating thickness, resulting from the increase of the web speed. Consequently, the pronounced decay of the conversion is largely an apparent effect only. This effect can be corrected for by use of two probe heads. Results of the chemometrics approach using the PLS2 algorithm are plotted in Figure 2.

In this case, coating thickness and conversion were obtained simultaneously. Reference values, which were determined off-line after the coating trial, show excellent correlation with the recorded NIR data, and verify their high precision.

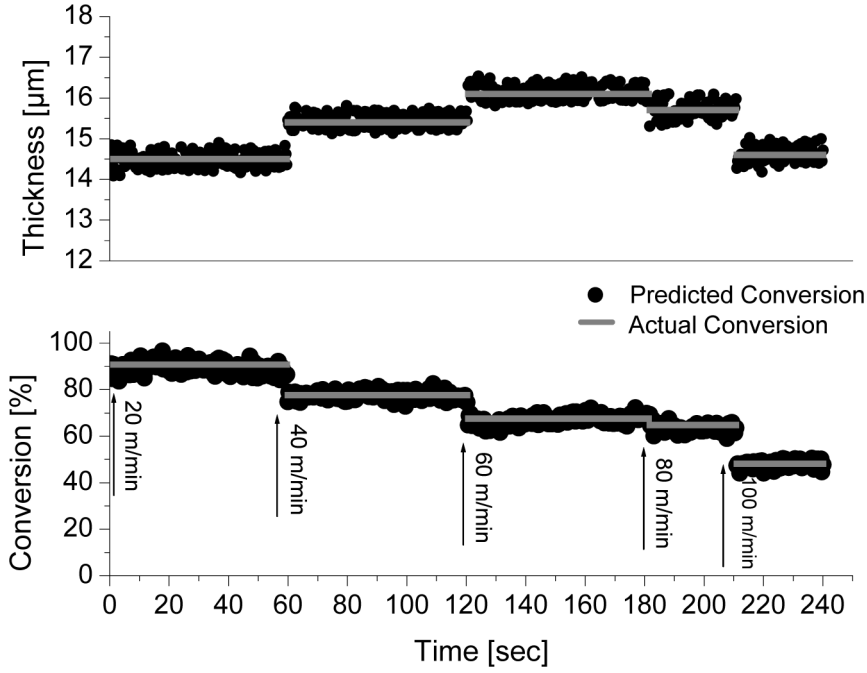

Figure 2. Simultaneous in-line monitoring of the conversion and the coating thickness in a clear acrylate coating by use of the PLS2 algorithm.