

# Continuous Monitoring of Process Parameters in UV Curing Processes

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Dr. Tom Scherzer (IOM Leibniz-Institut für Oberflächenmodifizierung) received the Paul Dufour Award at the RadTech Europe Conference in Nice, France (held Oct. 14-15, 2009), for this paper on continuous monitoring of process parameters in UV curing processes.



RadTech Europe Conference Programme Committee Chairman Dr. David Helsby (right), RadLab, hands the prestigious Paul Dufour Award to Dr. Scherzer.

Most of the functional properties of UV-cured coatings strongly depend on the conversion. However, the degree of the conversion, which is achieved by UV irradiation, is influenced by numerous variables. The most important one is the applied irradiation dose, which is determined by irradiance and line speed. Other parameters which may have an effect on the conversion are the composition of the lacquer formulation, as well as the ambient conditions during irradiation such as temperature, inert atmosphere, humidity and others. Only some of these influences can be easily controlled. Therefore, continuous monitoring of the conversion during the coating process would be of great importance.

However, the properties of the coatings do not depend only on the conversion, but also on the thickness of the applied layer. The thickness, in turn, is affected by several parameters such as viscosity, temperature and web speed. This way, unintended fluctuations of the thickness can occur in technical coating processes. For this reason, the determination of the coating thickness is an important

issue in coating technology as well. Moreover, if the coating thickness is monitored in the process, it can be made sure that the thickness does not exceed a given maximum value, which leads to a more economic use of the raw materials. On the other hand, the thickness must not fall below a specific minimum, because the properties of the coating and, consequently, the quality of the product depend on the layer thickness. Currently, the control of the applied thickness is mostly carried out offline by gravimetric determination of the coating weight. However, it is apparent that this approach is not able to respond to sudden changes of the thickness in the process.

Process control has been used in the chemical industry for a long time,<sup>1-3</sup> which allows for a better control of reaction processes as well as of the physical and chemical properties of the products made. In particular, process control systems have been applied for the monitoring of various polymerization reactions.<sup>4-6</sup> There, they can help to optimize the consumption of energy, raw materials and reaction time. Moreover, they ensure higher quality products.

Near-infrared (NIR) spectroscopy is a powerful analytical method, which is widely used for process control.<sup>7-8</sup> This spectroscopic technique is characterized by high sensitivity, comprehensive analytical potential, and excellent accuracy

of the results. The analysis of the recorded data may be supported by chemometric methods.<sup>9</sup> Today, the equipment employed for in-line monitoring is rugged, compact, solid and cost-effective. Most NIR process spectrometers are equipped with an optical fiber, which allows for a spatial separation of probe head and spectrometer. Thus, integration of such instruments into a production line is easily possible.

In the past, only few authors reported on investigations of the monitoring of UV-induced polymerization reactions by NIR spectroscopy.<sup>10-13</sup> Probably, this is related to the inherent problems of such studies. The typical thickness of UV-cured coatings is in the range of some micrometers only and, therefore, much lower than in most other applications of NIR spectroscopy. In combination with the quite low extinction coefficients in the near-infrared, the detection of NIR spectra of such thin layers and the continuous in-line monitoring of various process parameters is a challenging task. Previously, we reported our first results on the monitoring of the conversion of acrylate coatings under process conditions.<sup>13-14</sup> In that study, accidental changes of the thickness, which may occur in technical coating processes, were not considered. However, such changes may not only result from variations of the ambient conditions, but they are also inevitably induced by changes of the coating machine's line speed. These fluctuations of the thickness may have a detrimental effect on the correct determination of the conversion.

In the current study, we will demonstrate how NIR spectroscopy can be used for the continuous determination of the thickness of thin UV-cured coatings. Moreover, the influence of thickness changes on the

precision of in-line measurements of the conversion will be discussed. Finally, it will be shown how this effect can be corrected by specific measuring methods.

## Experimental

Samples were made from a clear acrylate formulation, which was based on an aliphatic urethane di-acrylate (EB 270, 60 wt-%), an amine-modified polyether acrylate (EB 81, 30 wt-%) and TPGDA (10 wt-%). Lucirin TPO-L was used as photoinitiator. The formulation was applied to 20  $\mu\text{m}$  thick polypropylene foil (OPP) by using an automatic film application machine and a set of Baker applicators, which allowed for a variation of the thickness from 5  $\mu\text{m}$  to 50  $\mu\text{m}$ . If samples with different conversions had to be prepared, the photoinitiator content was varied from 0.25 wt-% to 1 wt-%, and the coatings were cured at various irradiation doses (i.e., by variation of UV intensity and line speed). Irradiation was carried out under nitrogen in a UV-curing unit equipped with a 120  $\text{W cm}^{-2}$  mercury lamp (IST Metz). The irradiation dose per pass was 700  $\text{mJ cm}^{-2}$  at a conveyor speed of 10  $\text{m min}^{-1}$  and an irradiance of 620  $\text{mW cm}^{-2}$ .

In-line monitoring studies were carried out on a pilot-scale, roll-coating machine. It was equipped with a 160  $\text{W cm}^{-1}$  mercury lamp (PrintConcept UV Systeme, maximum power 8 kW, adjustable between 30 and 100 %). At full power, the irradiation dose at 10  $\text{m/min}$  was determined to be 850  $\text{mJ cm}^{-2}$ . In order to achieve coatings with different conversions and thicknesses, the roll gap, UV intensity and web speed were varied.

Spectra were recorded with a Kusta 4004 P process spectrometer (LLA, Berlin, Germany). This instrument is based on an InGaAs photodiode array detector with 256 elements. It was set

to cover the spectral region from 1,470 nm to 1,950 nm. The reflection probe head is linked to the spectrometer unit by a fiber-optic cable. Spectra were taken in transflection mode against a ceramic retroreflector behind the sample. A more detailed description of the spectrometer system is given elsewhere.<sup>12</sup> For in-line monitoring, the NIR probe head was installed above the moving web behind the UV lamp and very close to a guide roll in order to minimize the influence of vibrations of the polymer foil. NIR spectra were recorded continuously at a rate of 140 spectra  $\text{min}^{-1}$ . Some of the trials were carried out with two process spectrometers, which allowed for the correction of the conversion for the influence of the variation of the thickness of the coating. The second probe head was mounted directly after the roller application unit in order to record the spectra of the uncured layer.

The conversion of the double bonds after UV irradiation was determined with FTIR transmission spectroscopy. Spectra were recorded at several positions of the sample and averaged. The acrylate conversion was determined from the band of the  $\text{CH}_2$  scissor deformation at 1405  $\text{cm}^{-1}$ . The actual values of the coating thickness after irradiation were measured with a digital thickness gauge with a resolution of 0.2  $\mu\text{m}$ .

## Monitoring of the Thickness of UV-Cured Coatings

Quantitative analysis of NIR spectra is mostly carried out with multivariate calibration methods such as partial least square regression (PLS),<sup>9</sup> which are able to detect even minor variations in the spectra. The use of such chemometric techniques first requires the setup of a specific calibration model. This model has to relate the variation in the spectral data to the parameter of interest (e.g., the thickness of the applied coating).

FIGURE 1

### PLS calibration curve for the thickness of UV-cured acrylic clear coatings on OPP foil

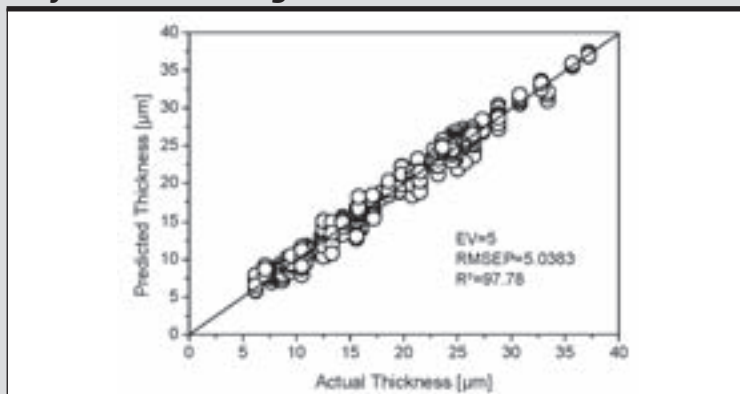
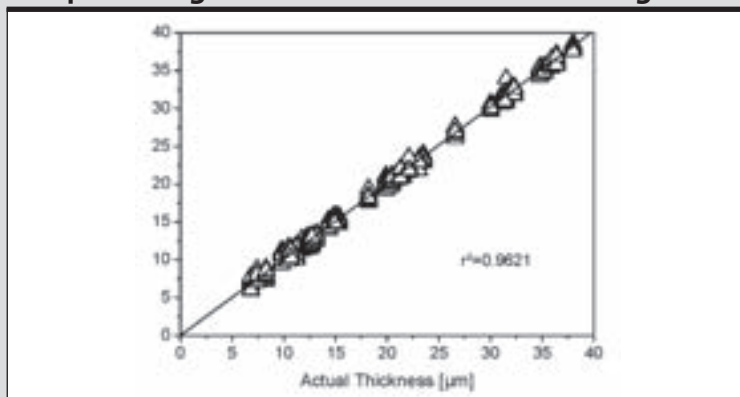


FIGURE 2

### Prediction of the layer thickness of independent test samples using the PLS calibration shown in Figure 1



Therefore, samples with coating thicknesses in the range from 5 to 50  $\mu\text{m}$ , in steps of 5  $\mu\text{m}$ , were prepared for calibration. For each specific thickness, 10 samples were prepared in order to get sufficient samples for creating a stable and powerful calibration model. After UV curing, NIR spectra were recorded. Typically, about 1,000 NIR spectra were accumulated during one scan, while moving the sample slowly through the focus of the probe beam. These single spectra were averaged

before further processing. In this way, each sample was measured 10 times. Consequently, 100 mean value spectra per thickness were recorded. Moreover, the actual thickness of the coatings was determined with a digital thickness gauge.

In the next step, the NIR spectra were pre-treated by normalization and application of the first derivative. Then, the PLS algorithm was applied to the data set. After regression, the root mean square error of prediction

(RMSEP) and the coefficient of determination  $R^2$  were calculated. The resulting calibration curve is shown in Figure 1. Details of the chemometric calculations are described in Reference 15.

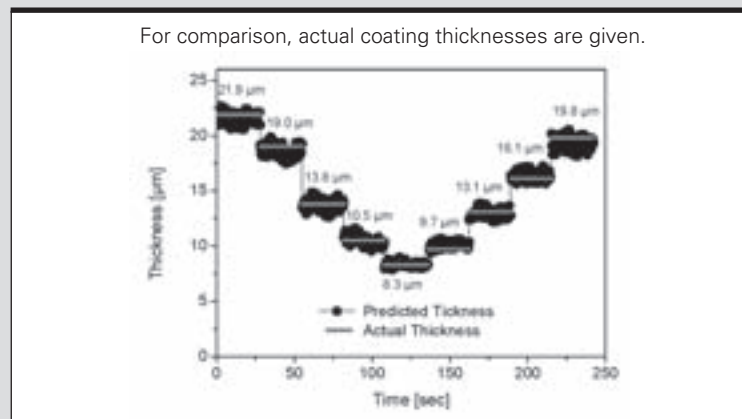
The performance of the developed model was tested with an additional set of independent test samples. Results are given in Figure 2. The coating thickness predicted with NIR spectroscopy is plotted against the thickness determined with the digital thickness gauge. The results clearly prove that the prediction can be carried out with high precision.

For in-line monitoring of the thickness of acrylic clear coats on OPP foil, the probe head was mounted on a pilot-scale, roll-coating machine. Particular attention was paid to the geometric arrangement of probe head and foil in order to make sure that it was identical to the arrangement in the lab. This is of utmost importance, since even minor differences between both arrangements would substantially affect the precision of the prediction. Important parameters which influence the spectral response and, consequently, the predictive power, are the distances between light source and foil or reflector, respectively, as well as the tilt angle of the probe head against the web.

First, the predicting power of the calibration model was tested at a constant line speed of 40 m/min. The coating was applied to OPP foil, and NIR spectra were recorded continuously right after UV curing. In order to simulate changes of the thickness during a real coating process, the nip between the applicator rolls was stepwise reduced and increased. Results are shown in Figure 3. In addition to the results obtained by NIR spectroscopy, the actual coating thicknesses are given, which were determined off-line after the end of

FIGURE 3

### In-line monitoring of the thickness upon stepwise decrease and increase of the nip at a line speed of 40 m/min



immediately after each change of the web speed. It can clearly be seen that the coating thickness significantly increases with increasing web speed, (i.e., from about 4 µm at 6 m/min up to 11 µm at 50 m/min). Again, excellent correlation of the NIR spectroscopic data with the thickness values determined offline was observed.

The results demonstrate that NIR spectroscopy, in spite of the low absorption coefficients in the near-infrared, is able to detect even small changes of the thickness of acrylic coatings under in-line conditions. The time resolution of the data recording (2.5 spectra/s) is sufficient for in-line monitoring. If measurements at higher speeds are necessary, the recording rate of the NIR spectrometer can be further increased without a significant loss of quality of the spectra. Thus, process control at line speeds well above 100 m/min can be realized.

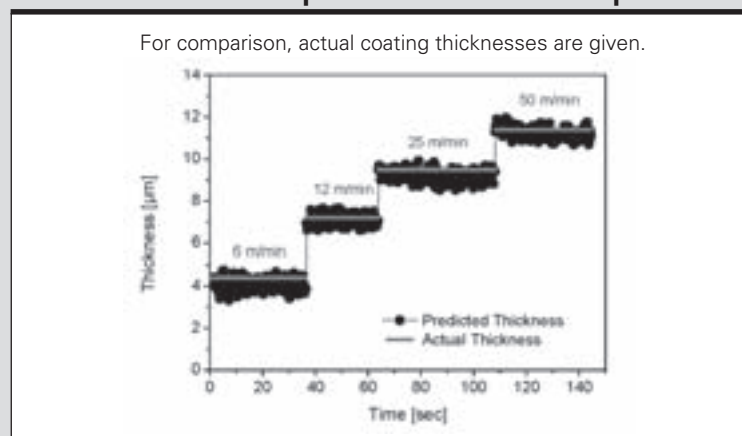
### Effect of Changes of the Coating Thickness on the Monitoring of the Conversion

For the calibration to the conversion in acrylic coatings, 160 samples with different conversions were prepared and characterized by both NIR and FTIR spectroscopy. Various PLS models were built up from the spectral data using the conversion values from FTIR spectroscopy as reference data. In order to optimize the calibration model, different kinds of preprocessing of the spectra were tested. The model with the lowest RMSEP and the highest  $R^2$  was selected for in-line analysis. The resulting calibration curve is shown in Figure 5. The chemometric procedures are discussed in more detail in Reference 16.

The performance of the developed model was tested with an additional set of independent test samples, (i.e., these samples had not been included in the calibration process).

FIGURE 4

### In-line monitoring of the thickness upon stepwise increase of the line speed with constant nip



the roller application trial. It can be seen that both the predicted and the actual thicknesses decrease or increase according to the changes of the nip. Thickness changes as low as 1.4 µm can be clearly detected by NIR spectroscopy. Thus, the close correlation between actual and predicted thickness as well as the immediate spectral response after any change in thickness suggest that the

predicting power of the chemometric model is sufficient for in-line monitoring of the thickness of acrylic clear coatings.

It is well known that the thickness of the coating increases with increasing web speed in case of application by roll coating. Therefore, the calibration model was also tested at different web speeds from 6 to 50 m/min (Figure 4). Similar to the results shown in Figure 3, the spectral response can be observed

FIGURE 5

### PLS calibration of the NIR spectra to the conversion of 10 $\mu\text{m}$ thick acrylic clear coatings

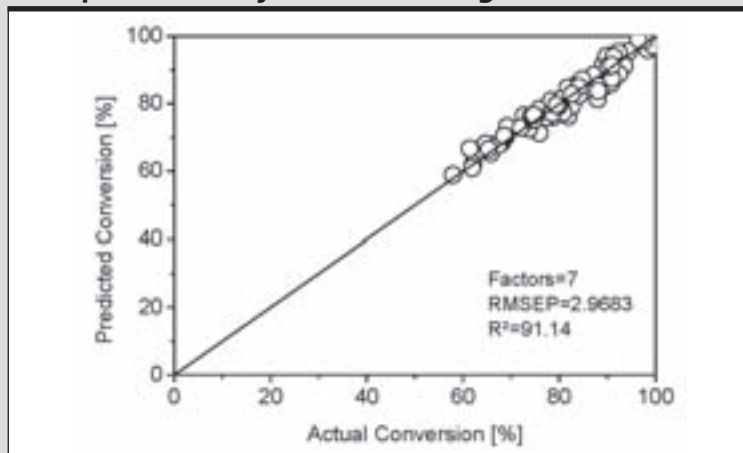
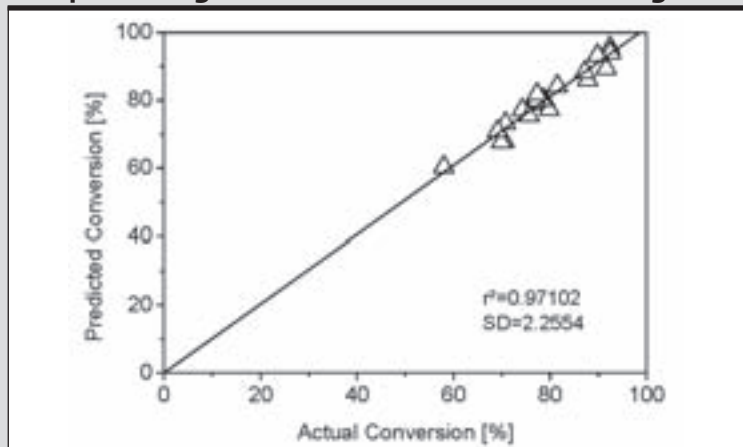


FIGURE 6

### Prediction of the conversion of independent test samples using the PLS calibration shown in Figure 5



Their conversion was predicted with the created chemometric model and compared with FTIR reference data. Results are given in Figure 6. The close correlation of the predicted conversion (NIR) with the actual conversion (FTIR) clearly demonstrates the high predicting power of the created calibration model.

The chemometric calibration model was used to follow the conversion in acrylic clear coats during the coating

process on a roll-coating machine. In order to vary the irradiation dose, the line speed was stepwise increased, whereas the power of the UV lamp remained constant. The resulting changes in conversion were followed by NIR spectroscopy. At each line speed, spectra were recorded for one minute. From each spectrum, the conversion was predicted by use of the PLS calibration, which is shown

in Figure 5. The mean values for each line speed are plotted in Figure 7. For comparison, reference values (FTIR spectroscopy) are given. Moreover, the thickness of the layers is shown, which was measured off-line as well.

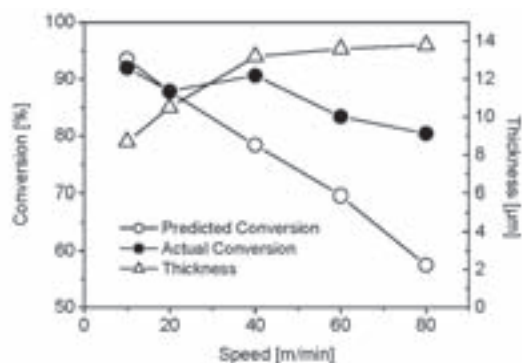
At low web speeds ( $\leq 20$  m/min), a close correlation between the predicted and the actual conversion was observed. With increasing line speed, however, both curves increasingly diverge. Simultaneously, the thickness of the layer applied to the foil was found to increase from about 9 to 14  $\mu\text{m}$ , which is only due to the increase of the line speed (constant nip). However, the increase of the thickness has a destructive effect on the prediction performance of the PLS calibration. This is due to the fact that the calibration to the conversion was carried out with samples with a constant nominal thickness of 10  $\mu\text{m}$  (the resulting thickness of the layers after UV curing was even lower due to the inherent shrinkage during polymerization). Consequently, correct predictions of the conversion during in-line monitoring were only obtained at low line speeds in which the applied thickness corresponds to the thickness during the calibration procedure. With increasing thickness, the predicted conversion more and more deviates from the real conversion.

In order to get accurate results for the conversion in thin acrylic coatings, a correction of the influence of changes of the coating thickness was necessary. Basically, there are two possibilities to consider such changes in the quantitative evaluation. On the one hand, two probe heads may be used, which record NIR spectra of the coating before and after UV curing, respectively. Quantitative analysis of the spectra is based on the integration of the band of the first overtone of the C-H stretching vibration of the vinyl bond, which appears at 1,620 nm in the NIR spectrum of acrylates.

## FIGURE 7

### Effect of the line speed on the prediction of the conversion of acrylate coatings applied to OPP foil

For comparison, the actual conversion (from FTIR spectroscopy) and the coating thickness are shown.



The conversion after UV curing can be directly estimated according to the Beer-Lambert law from the ratio of the band integrals before and after irradiation. In this way, the effect of unintended thickness changes is averaged out.

On the other hand, variation of the thickness of the layer can be directly included in the chemometric calibration model, which is certainly the more sophisticated solution. This can be done by use of the PLS2 algorithm, which is able to predict two or more parameters from the same input data.<sup>9</sup> However, this implies that the enormous efforts for calibration even more increase since the calibration samples have to cover the full range of both conversion and thickness, which can occur during analysis.

In the present study, both methods were tested. The performance of both approaches will be discussed in the next paragraph.

### Compensation of the Effect of Thickness Changes by Use of Two Probe Heads

Since the setup with two measuring points (i.e., probe heads) cannot be

used in combination with chemometric evaluation algorithms, quantitative analysis has to be carried out on the basis of the band integration method, which had already been used previously.<sup>12-14</sup> Figure 8 demonstrates the precision of this method by comparing the conversion data obtained in this way from the NIR spectra of acrylate coatings, which were irradiated with various UV doses, with conversion

data of the same samples, which were determined with FTIR spectroscopy.

A linear correlation between the conversion data from NIR and FTIR spectroscopy was found. Moreover, no offset was observed, which implies that the two data sets are completely equivalent. Therefore, band integration of NIR spectra may be regarded as an easy, rapid and, in most cases, practicable method to get quantitative conversion data, which is an alternative to chemometric approaches. Accordingly, the influence of unintended changes of the thickness of the layer, (e.g., caused by changes of the web speed), can be easily compensated for by recording NIR spectra of the coating before and after UV curing and determination of the conversion from the ratio of the band integrals. During merging of the two data streams coming from the two probe heads, a time offset had to be considered in order to include the delay time between the measuring points.

By use of this technique, the conversion in clear acrylic coatings on OPP foil was followed. In order to test the capability of the method to compensate for changes of the

## FIGURE 8

### Conversion in 10 µm acrylate coatings

Comparison of values obtained from NIR reflection spectra by band integration with reference data from FTIR transmission spectroscopy.

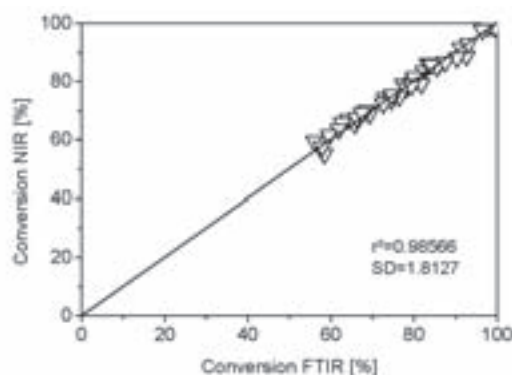
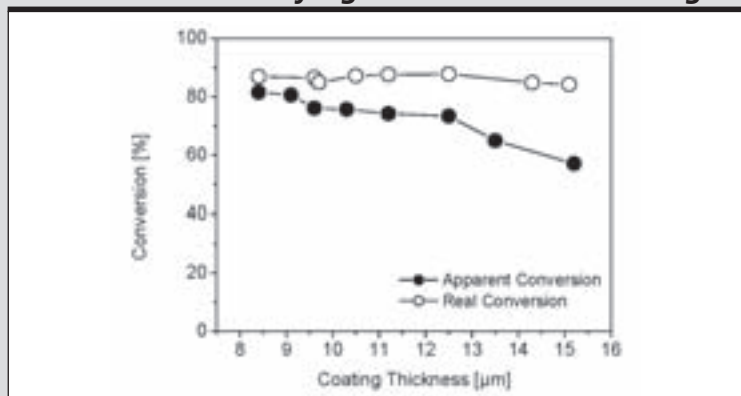


FIGURE 9

### Conversion in clear acrylate coatings with (○, 2 probe heads) and without (●, 1 probe head) correction of the varying thickness of the coating



thickness, the thicknesses of the layer were purposely varied by variation of the nip between the applicator rolls. In contrast, the web speed and the irradiation dose were not changed during this trial. For comparison, an additional test was carried out. In that case, spectra were recorded with only one probe head, (i.e., with that behind the UV lamp). Spectra of the uncured layer were taken just before switching on the UV lamp by collecting data at constant nip (i.e., the narrowest nip) for at least one minute. The band integrals determined from these spectra were averaged and the mean value was used as reference value for band integration during in-line monitoring. The results of both tests are compared in Figure 9.

It can be clearly seen that the conversion, which was determined with the setup consisting of two probe heads, does not depend on the thickness of the applied coating. This is due to the low thickness of the coatings studied here and the high reactivity of the phosphine oxide photoinitiator. This observation was also found to be in accordance with

random off-line measurements of the conversion by FTIR spectroscopy. This result clearly proves the power of the method based on two probe heads for measuring the acrylate conversion accurately in spite of any variations of the thickness of the layer, (i.e., the effect of changes of the thickness is completely compensated).

If only one probe head is used, a decrease of the conversion with

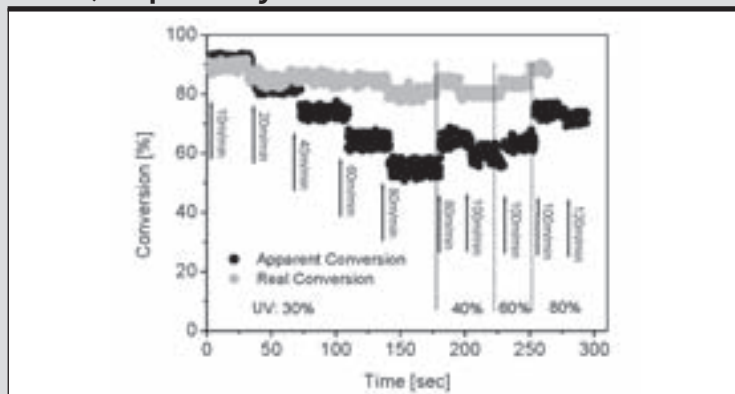
increasing thickness of the coating is observed. Consequently, the difference between this conversion and the conversion resulting from the trial monitored with two probe heads increases with increasing coating thickness. However, the decrease of the conversion is only due to the change of the thickness and does not represent a real decay of the conversion. This result shows that the conversion can only be monitored with one probe head if it can be guaranteed that the thickness of the layer does not change during the coating process. In most technical coating processes, it is, however, almost impossible to eliminate fluctuations of the thickness of the layer, which will lead to apparent changes of the conversion.

The two setups with one or two probe heads, respectively, were then used for in-line monitoring of the acrylate conversion in a coating process, which was carried out at various line speeds and UV intensities. Quantitative evaluation of the data was performed by band integration. A record of a typical test trial is shown in Figure 10.

At first, the speed of the web was stepwise increased at constant

FIGURE 10

### In-line monitoring of the conversion in clear acrylate coatings with one (●) or two (○) NIR probe heads, respectively



irradiance. During the second stage, both the power of the UV lamp and the line speed were alternately varied up to 120 m min<sup>-1</sup> at the end of the trial. After each change of dose, an almost immediate response of the conversion was observed, which shows that the NIR method is sufficiently fast for in-line monitoring.

In case only one probe head was used for monitoring, the changes of the coating thickness (which were induced by the increasing web speed) lead to an apparent decrease of the conversion. This is due to the fact that the reference data of the uncured layer (which were taken in advance) are only correct for the thickness at the beginning of the experiment. Consequently, the pronounced decay of the conversion is largely an artifact.

However, if the conversion is determined from a pair of spectra, which were recorded immediately before and after UV irradiation, the influence of thickness changes is completely eliminated. Accordingly, the trace, which was recorded with

two probe heads, shows much less variation of the conversion, particularly during the first stage of the trial made up of speed changes only. In contrast, changes of the irradiance are clearly reflected in the conversion data. These results correlate quite well with the findings shown in Figure 9.

### Simultaneous Monitoring of Thickness and Conversion

Instead of the use of two probe heads, the effect of thickness changes can be compensated for by use of specific chemometric methods. The PLS2 algorithm<sup>9</sup> is able to predict two parameters from one data set, if the expected variation ranges of both parameters are covered by the calibration samples. For the development of a PLS2 model for the calibration of the NIR spectra to both the conversion and the coating thickness, 160 samples with conversions from 50 to almost 100% and with nominal thicknesses between 5 and 20 μm were prepared. Again, reference data were obtained by FTIR spectroscopy

and the thickness gauge, respectively. After recording of the NIR spectra, the PLS2 model was built up, optimized and tested. Details of the calibration procedure as well as calibration curves are given in Reference 17.

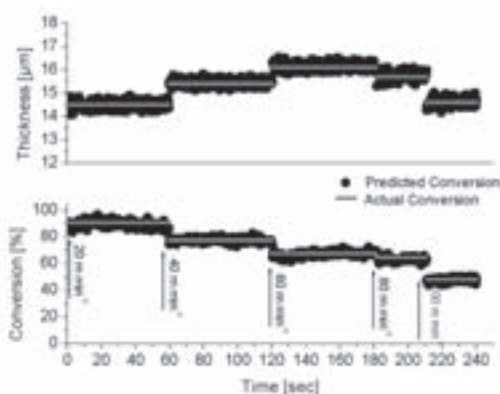
The created PLS2 calibration model was used to follow both the conversion and the coating thickness of acrylic clear coats during a coating trial on a pilot-scale, roll-coating machine. In order to vary the conversion, the web speed was stepwise increased from 20 to 100 m min<sup>-1</sup>, while the power of the UV lamp was kept constant. However, the increase of the web speed automatically leads to an increase of the thickness of the acrylate layer, which is followed by a decrease at higher line speeds. The resulting changes of both parameters were monitored by NIR spectroscopy. Spectra were recorded continuously at a sampling rate of 140 spectra min<sup>-1</sup>. At web speeds up to 60 m min<sup>-1</sup>, spectra were recorded for one minute, at higher speeds for 30 seconds only. Both the conversion and the thickness were predicted from each spectrum by use of the PLS2 calibration. Results are shown in Figure 11. For comparison, reference values, which were determined off-line after the machine trial, are provided. They clearly prove the precision of the predictions from the NIR spectra. So, this powerful chemometric approach is not only able to correct the influence of thickness changes on the conversion, but both parameters in fact can be really monitored simultaneously.

### Conclusions

It was demonstrated that NIR reflection spectroscopy can be effectively used for in-line monitoring of important process parameters in UV-curing technology such as the conversion and the thickness of the coatings. Moreover, in combination with sophisticated chemometric approaches, it is a powerful tool for the

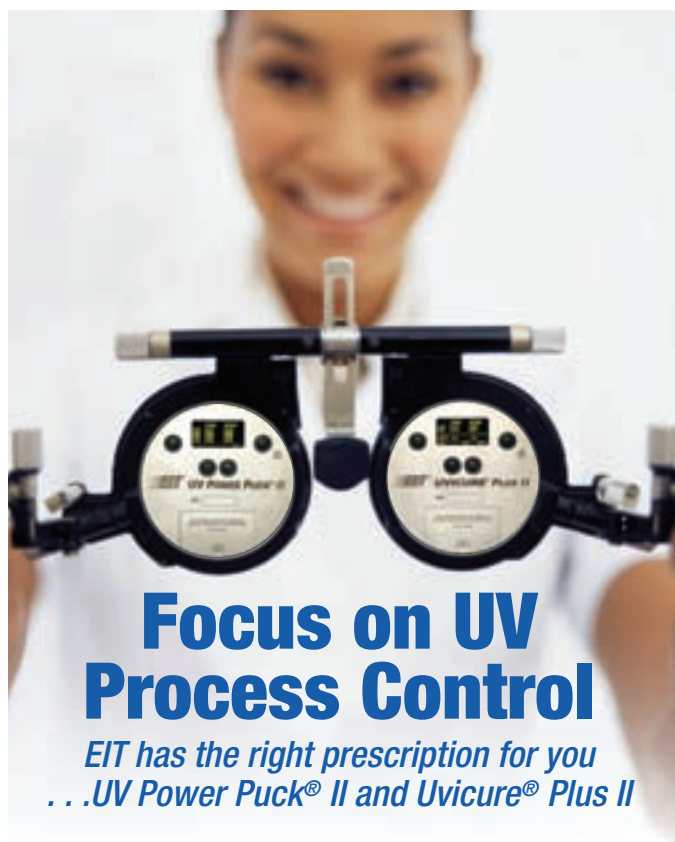
## FIGURE 11

### Simultaneous in-line monitoring of the coating thickness (top) and the conversion (bottom) of a clear acrylate coating on a roll coating machine



For comparison, actual values of both parameters, which were determined off-line, are shown.



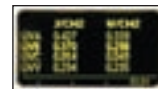


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monitoring of problems with complex interrelationships between several parameters. In the present study, it was used to compensate for the effect of changes of the thickness of the coating on the determination of its conversion, which may occur inevitably after changes of the line speed. Hence, in the future NIR spectroscopy could be used as a versatile and powerful method for process and quality control in technical curing processes.

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