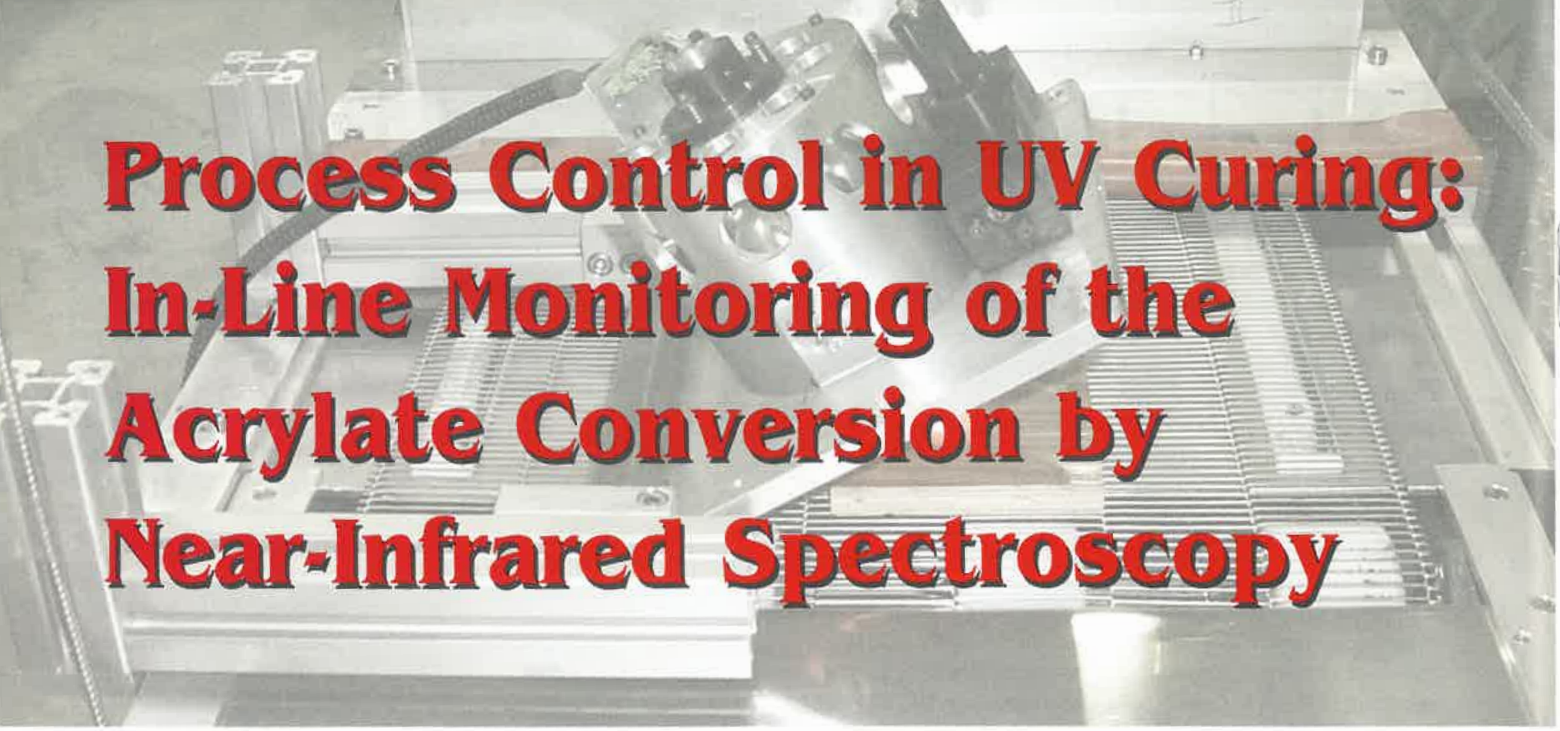

Process Control in UV Curing: In-Line Monitoring of the Acrylate Conversion by Near-Infrared Spectroscopy

by Tom Scherzer,**
Sabine Müller, and
Reiner Mehnert
Leibniz Institute of Surface
Modification (IOM)*

and

Arne Volland and
Hartmut Lucht
LLA Instruments GmbH†

The conversion of double bonds in UV-cured acrylic coatings on various substrates was followed in-line by near-infrared (NIR) reflection spectroscopy. Quantitative data were obtained directly from the intensity of the acrylic overtone band at 1620 nm, which allows very easy calibration of the method. The custom-made probe head of the NIR spectrometer was fitted to several pilot-scale coatings and curing lines, and the conversion was determined in clear and pigmented coatings as well as in scratch resistant nanocomposite layers on the basis of acrylates. It was shown that reasonable conversion data can be obtained with sufficient time resolution at line speeds of at least 120 m/min. Furthermore, the method has been proven to be useful for the monitoring of the conversion in layers from hot-melt adhesives for PSA applications. Thus, for the first time there is an analytical method available which can be reliably applied for process and quality control in technical curing processes.

INTRODUCTION

Process analytical tools have been used in the chemical industry for a long time in order to control the actual state of the process and the quality of the products made.[1,2] In recent years, such systems have also been increasingly employed for the monitoring of various polymerization reactions where they can help to optimize the efficient use of energy and raw materials, and also to ensure that the properties of the produced polymers are within the given specification. Process monitoring and control would also be interesting for radiation-induced polymerization reactions.

The most important parameter for process and quality control of UV and electron beam (EB) curing processes is the conversion of the (meth)acrylic double bonds, since that determines all other functional properties of a coating such as scratch and abrasion resistance, hardness, weathering resistance, chemical stability, the content of extractables, etc. Moreover, for further pro-

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*Permoserstr. 15, D-04318 Leipzig, Germany.

†Schwarzschildstr. 10, D-12489 Berlin, Germany.

**Author to whom correspondence should be addressed. Fax: +49-341-235-2584; email: tom.scherzer@iom-leipzig.de.

cessing of the coating a sufficient conversion is required, and wipe resistance must be attained before stacking or winding.

However, the conversion that is achieved during irradiation depends on a large number of factors such as the applied irradiation dose, which is determined by both line speed and irradiance of the incident UV light (or the electron current in EB curing), and the composition of the reactive formulation, temperature, inertization, and other factors. Only some of the possible influences can be easily controlled. Hence, the compliance of the actual level of the conversion of a coating with the requirements determined by the specific application has to be controlled continuously in order to be able to respond to process variations. For an efficient process and quality control, the immediate and continual availability of current conversion data is an indispensable precondition.

Various analytical methods have been tested with respect of their applicability for in-line measurements of the degree of cure of a coating in a running coating line.[3-5] However, none of them was found to be sufficiently suited for this task. Near-infrared (NIR) spectroscopy is widely used for process control in the chemical industry and in several other commercial applications,[6,7] since it possesses sufficient time resolution and sensitivity for in-line measurements as well as comprehensive and versatile analytic potential. Moreover, efficient analysis of the recorded data stream can be supported by powerful chemometric methods. Dedicated NIR process analyzers based on multichannel detectors are compact, rugged, and comparatively cost-effective. Generally, they are equipped with optical fibers, which allows spatial separation of spectrometer and probe head, and, consequently, easy integration of the instrument into an existing production line.

The analysis of acrylates and methacrylates by NIR spectroscopy is based on the band of the first overtone of the C–H stretching vibration of the (meth)acrylic double bonds, which appears at 1620 nm.[8] In the past, the conversion was followed in several batch polymerization processes[9-11] where the thickness of the layer in which the probe light is absorbed lies in the range of millimeters or centimeters. Some authors also report the investigation of photopolymerization reactions of acrylates[12-14] and methacrylates[15,16] by NIR spectroscopy. However, those investigations were performed on thick samples as well (i.e., a thickness of about 1 to 6 mm) since most of the samples were dental composites. The investigation of thin coatings with a thickness of only a few micrometers is difficult because of the relative low extinction coefficients in the near-infrared. Nevertheless, in this study it will be shown that NIR reflection spectroscopy is able to detect even small variations of the conversion in acrylate coatings studied directly in a running coating line, in spite of the weak absorption and the very low thickness of the layers.

EXPERIMENTAL

In-Line NIR Reflection Spectroscopy

NIR spectra were recorded with a process analyzer system which was developed according to the specific requirements of in-line measurements on thin UV-cured acrylate coatings.[17] It consists of a commercial spectrometer unit (Kusta 4004 P from LLA) and a separate tailor-made probe head which is linked to the spectrometer by a fiber-optic cable.

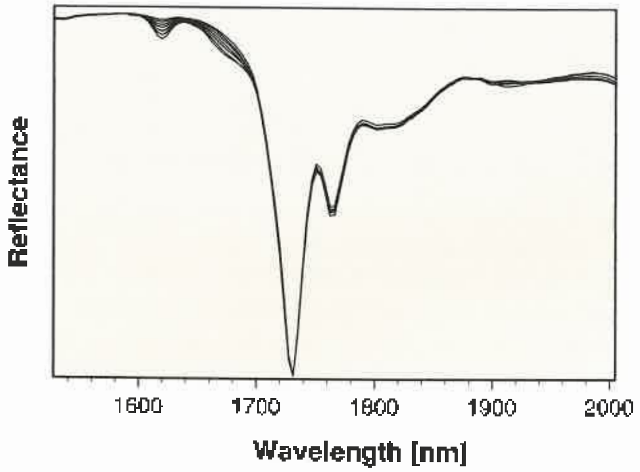
The spectrometer is based on a concave holographic grating and a fast linear photodiode array as detector. The thermoelectrically cooled InGaAs detector consists of 256 elements and covers a spectral region from 1530 to 2000 nm. With its minimum integration time of 56 µsec, the diode array provides the time resolution and the sensitivity which are necessary for in-line monitoring. The tungsten halogen lamp used as the light source for the spectrometer is integrated in the probe head. In order to prevent postcuring of the acrylate coatings by the short-wavelength part of its emission, a UV filter is mounted in front of the probe head. Moreover, it is equipped with a diffuser plate in order to suppress interferences which occur in thin transparent foils of some high-grade polymers. Spectra of transparent film samples are taken against a ceramic retro-reflector behind the foil, which is also attached to the probe head. A more detailed description of the NIR reflection spectrometer system was reported in reference 17.

Application and UV Curing of Coatings

In-line monitoring studies were carried out on various pilot lines at IOM. Acrylate coatings on polymer foils and paper were made on a roll coating machine provided with a Fusion F-600 mercury arc lamp and an LEA electron beam accelerator operated at 150 kV (IOM).[18] Irradiation of coatings on panels and plates was performed on a conveyor, which can be equipped with various UV lamp systems such as an IST mercury arc lamp or an array of fluorescent lamps. The degree of cure was followed in coatings from proprietary clear and pigmented acrylate formulations to be used as functional or protective layers.

Application and UV irradiation of adhesive layers were carried out with a dedicated slot die coating machine. After heating to 90°C in order to get a melt viscosity suitable for processing, the adhesives were applied to a 220 µm thick polypropylene (PP) tape and subsequently irradiated at 308 nm with an XeCl* ex-

Figure 1—NIR reflection spectra of 20 g/m² acrylate coatings on 60 µm LDPE foil after UV irradiation with different doses.



cimer lamp (Heraeus), which was mounted lengthwise in order to increase the applied irradiation dose.

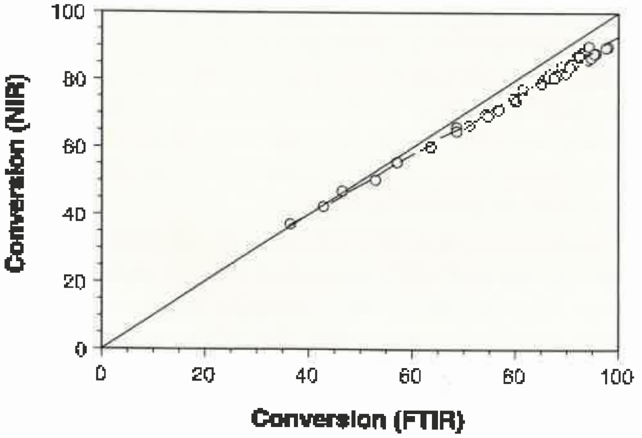
In any case, the NIR probe head was installed above the moving web or conveyor just behind the exit of the UV lamp or the EB accelerator, and spectra were recorded continuously at a rate of 100 spectra/min.

RESULTS AND DISCUSSION

Quantitative Analysis

Figure 1 shows spectra of an acrylic clearcoat on polyethylene foil (LDPE) after UV irradiation at various doses. The acrylate band at 1620 nm and its decay on irradiation can be clearly seen in the spectra. Since the spectra are recorded in reflectance, the method is not limited to transparent materials, but is also suited for opaque substrates as well as for pigmented coatings.

Figure 2—Calibration curve for the conversion in 10 µm acrylate coatings on 30 µm LDPE foil: comparison of results from NIR reflection spectroscopy with reference data from FTIR transmission spectroscopy.



Before NIR spectroscopy can be used for process control, it has to be calibrated to the specific system under investigation. In general, this is done by powerful chemometric approaches such as PLS. However, these methods require large sets of well-defined samples for calibration, which are difficult to provide in UV curing. As a simple alternative for the quantitative analysis of the spectra, the conversion in the acrylate coatings can also be determined directly from the ratio of the integrals of the band at 1620 nm before and after irradiation of the sample. In this case, calibration is done just by recording some spectra of the non-irradiated coating. However, the thickness of the coating has to be constant throughout calibration and in-line monitoring since there is no suitable band in the NIR spectrum which could be used as an internal standard. Therefore, for the correction of the conversion data for potential thickness variations, the coating thickness would have to be measured independently. In principle, this could even be done with NIR spectroscopy. However, this would require a second NIR setup and a specific PLS-based calibration to the thickness for each coatings/substrate combination.

The precision of the integration method was tested by comparing conversion data obtained in this way from the NIR spectra with results from FTIR transmission spectroscopy. Investigations were carried out with 10 µm-thick acrylate coatings on a 30 µm LDPE foil. Samples with a wide range of conversions were prepared. In order to avoid significant postcuring, NIR reflection and FTIR transmission spectra were recorded immediately after UV irradiation. With both methods, spectra were taken at several points of each sample, and the resulting spectra were averaged before further processing. Results are summarized in *Figure* 2. In the complete range of conversions studied, a close linear correlation between the data from the two spectroscopic methods was found. NIR spectroscopy slightly underestimates the conversion in comparison to FTIR spectroscopy, which might possibly be due to baseline effects in either one or both methods. This offset could be easily corrected for, if this is required.

Process Control of the UV and EB Curing of Acrylate Coatings

For in-line monitoring of the degree of cure in thin acrylate coatings on polymer foils and paper, the spectrometer was fitted to a pilot-scale roll coating machine. It was slightly tilted against the web, which, in addition to the diffuser plate, leads to an efficient suppression of interferences in thin transparent polymer foils.

Figure 3—Process monitoring of the UV curing of a 20 g/m² acrylate coating on 20 µm OPP foil with variable irradiance and at various line speeds.

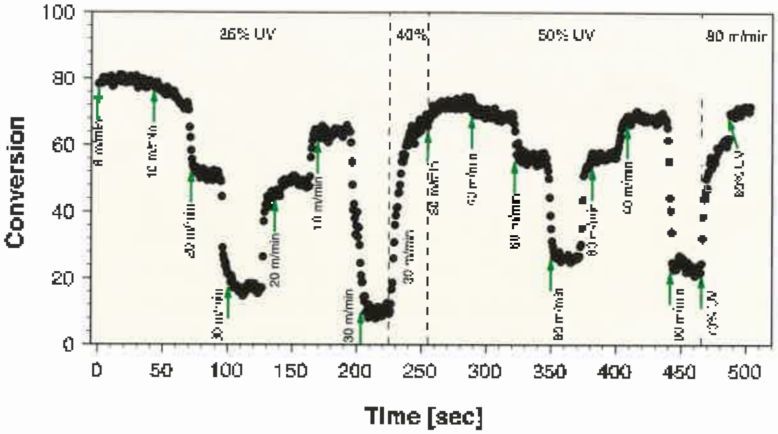


Figure 4—Process monitoring of the UV curing of a 10 g/m² acrylate coating on an 80 g/m² decor paper with variable irradiance and at various line speeds.

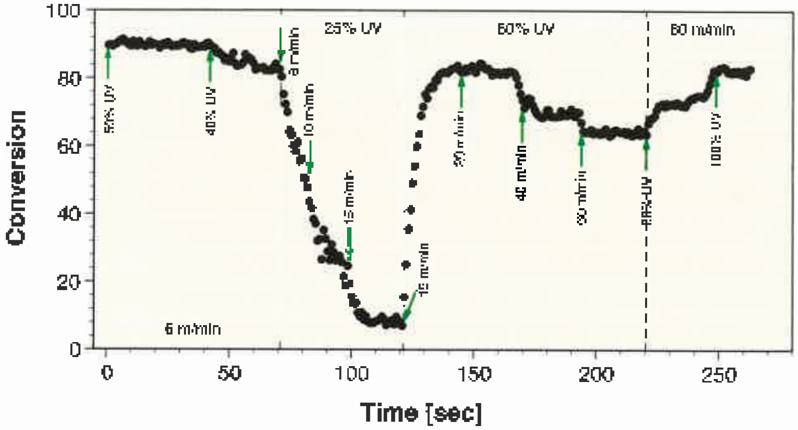
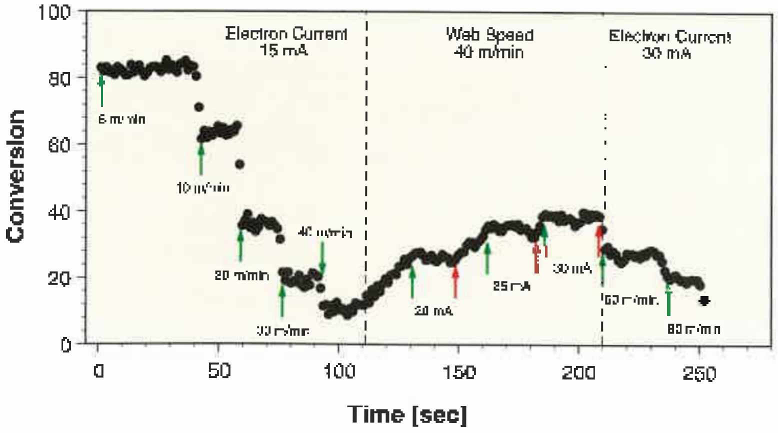


Figure 5—Process monitoring of the EB curing of a 25 g/m² acrylate coating on 60 µm LDPE foil with variable electron current and at various line speeds



Clear and pigmented acrylate coatings were applied to various substrates and cured with UV light or EB radiation. Simultaneously, NIR spectra were recorded. Calibration to the specific sample system was carried out just before the monitoring trial and directly in the coating machine by taking spectra of the non-irradiated coating.

Figure 3 shows the in-line monitoring of the conversion in a clear acrylate coating on OPP foil after UV irradiation. In order to simulate changes of the irradiation dose, both the power of the UV lamp and the line speed were varied repeatedly. The power of the Fusion lamp is given in percent of its maximum output. It can be clearly seen that the conversion increases or decreases according to the resulting irradiation dose. If the line speed is first increased stepwise and then decreased, equal conversion is observed at equal doses. Any change of the line speed leads to an immediate change of the conversion. In contrast, changes of the power of the lamp appear after a delay only (e.g., the period between 225 and 255 sec), which is due to the fact that the response of the UV lamp itself is much slower than that of its electronic control when the power is switched to a higher level.

Analogous monitoring trials were also carried out with acrylate coatings on paper. As an example, *Figure* 4 shows an investigation of the degree of cure in a clearcoat on decor paper. In this case, the paper itself serves as the reflector for the near-infrared radiation. Nevertheless, the results are quite similar to those on transparent polymer foils.

In this study, the monitoring of the conversion was performed at line speeds up to 120 m/min. Even at these high speeds, the scatter of the conversion data is low, i.e., it is in the order of about ± 2 to 4%. The results clearly show that it spite of the low extinction coefficients in the near-infrared and the low thickness of the layers in the range of a few micrometers, NIR reflection spectroscopy is able to detect small variations of the conversion in such acrylate coatings even when they have to be analyzed in a running coating line. The

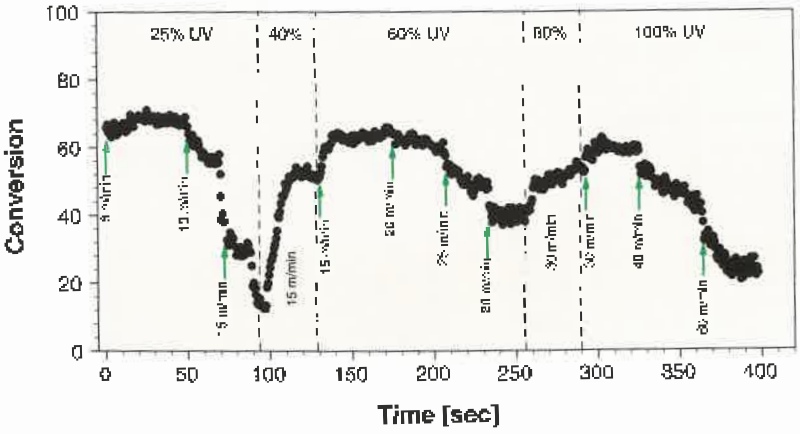


Figure 6—Process monitoring of the UV curing of a 25 g/m² white-pigmented acrylate coating on 60 µm LDPE foil with variable irradiance and at various line speeds.

time-resolution of the data (0.6 sec/spectrum) is sufficient to be used for process control. However, if the coating line has to be operated at much higher speeds, the recording rate of the spectrometer can be further increased without a significant loss of the quality of the spectra.

Results similar to those found after UV irradiation were also obtained after EB curing. *Figure* 5 shows the monitoring of the conversion in an acrylic clearcoat on LDPE foil after EB irradiation. The electron current and the web speed were varied alternately. Either the current was kept constant and the web speed was increased stepwise, or the current was varied at constant speed of the line. These variations of the irradiation dose can be directly observed as changes of the acrylate conversion. Changes of the web speed again lead to an immediate response of the conversion, whereas, after changes of the electron current, its takes some time to set the EB accelerator to the new irradiation conditions. This is reflected in a gradual increase of the conversion as can be clearly seen after each increase of the current in the period between 110 and 185 sec.

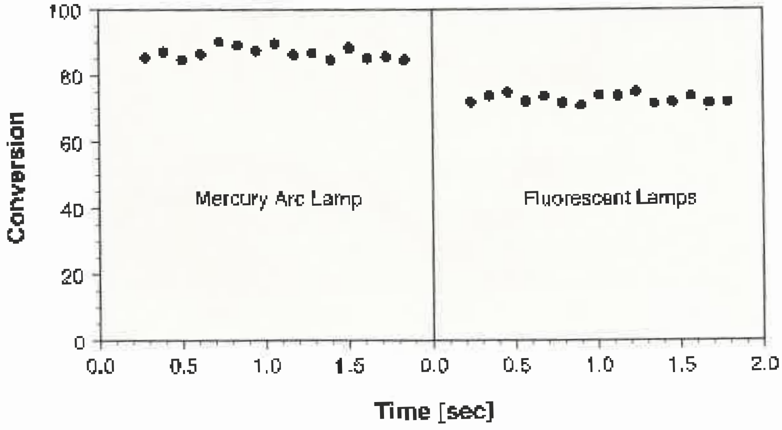


Figure 7—In-line monitoring of the degree of cure in UV-stabilized nanocomposite coatings on MDF panels after UV irradiation with a mercury arc lamp or a set of fluorescent lamps at a line speed of 10 m/min.

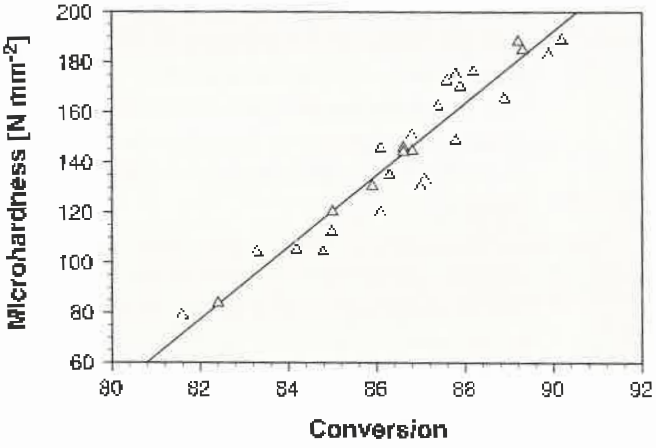
Pigmented systems make up a significant percentage of technical applications of UV-cured coatings, and among them white-pigmented coatings play the most important role. However, the curing of white-pigmented coatings is one of the most difficult problems in UV curing. On the one hand, sufficient amounts of pigment have to be added to the binder formulation in order to achieve sufficient hiding power of the coating. On the other hand, the pigment particles strongly impede the penetration of UV light into the deeper layers of the coating. Moreover, they compete with the photoinitiator for the incident UV light. The resulting conversion gradient may cause serious curing problems such as a lack of adhesion to the substrate. In order to avoid such problems, the curing process has to be carefully optimized. In-line monitoring may help to detect any deviation from these optimum conditions.

Titanium dioxide, the most widely used white pigment, does not significantly absorb in the near-infrared, which allows for monitoring of the acrylate conversion in such coatings in the same way as for clearcoats. A typical result is shown in *Figure* 6. In this trial, the degree of cure in a white coating containing 15 wt% titanium dioxide on polyethylene foil was followed by NIR spectroscopy. As expected, the record is basically not different from those of clear coatings. However, despite roughly similar irradiation conditions, the conversion is, on average, lower than in the clear coatings in *Figures* 3 and 4. This is due to the effect of the stronger conversion gradient within the pigmented coating. The lower conversion in its deeper layers is reflected in a somewhat lower average conversion in the in-line plot.

Process Control of the UV Curing of Nanocomposite Coatings

Nanoparticles of silica or alumina can be used as appropriate fillers in UV-curable acrylate formulations to provide transparent nanocomposite materials. Particles that were previously modified by grafting monomers onto their surface are covalently bound into the acrylate network during UV curing,[19,20] which leads to a marked reinforcement of the organic matrix. One of the outstanding properties of such coatings

Figure 8—Microhardness of 30 µm nanocomposite coatings on glass in dependence on the conversion.



is their excellent scratch and abrasion resistance, used to protect wood, medium-density fiberboard (MDF), and polymers. The mechanical properties strongly depend on the network density, which is mainly determined by the conversion of the acrylic double bonds. For this reason, a continuous control of the degree of cure in the coating is indispensable in order to ensure a consistently high quality coating. Hence, a pilot line for the coating and UV irradiation of panels at IOM was fitted with the NIR process monitoring equipment, and the conversion in acrylic nanocomposite coatings on MDF panels was studied.

The acrylate formulation used here contained modified nanoparticles with an average particle diameter of 30 to 40 nm. Moreover, it was protected against UV aging and degradation by a standard UV stabilizer package, and Lucirin TPO-L was used as the photoinitiator. After application to MDF panels, UV irradiation was carried out either with a conventional mercury arc lamp (IST) or with an array of fluorescent lamps. The emission spectrum of the latter is shifted to somewhat longer wavelengths in comparison to that of the mercury lamp, which is advantageous for the curing of UV stabilized coatings. NIR spectra were taken in-line just after UV irradiation. The rate of spectra recording was increased to nine spectra per second in order to provide enough spectra per panel. *Figure* 7 compares the efficiencies of the two different UV sources to cure the applied acrylate formulation. The results clearly demonstrate that the cost-effective fluorescent lamps (which are normally used for skin tanning) could be effectively used to crosslink such coatings despite their much lower irradiance.

The spectral variation in the NIR spectra does not necessarily need to be related to the acrylate conversion. Alternatively, calibration can be performed directly to the properties of the UV-cured coatings, which are relevant for their application performance provided that they scale linearly with the changes in the spectra. In this respect, the strong effect of the crosslink density and consequently, the conversion on the mechanical properties of the nanocomposite coatings, was studied in more detail. Coatings with a thickness of 30 µm were applied to glass plates and irradiated with various UV doses. Subsequently, the conversion in the layers was determined by NIR spectroscopy, and their Martens hardness was measured using a microhardness tester (Fischerscope H100C with Vickers indentor).

Figure 8 shows the correlation between the hardness of the coating and the acrylate conversion. It is obvious that the microhardness responds extremely sensitively, even to small changes of the conversion. An increase of the conversion by only 8% leads to an increase of the microhardness by about 15%. This strong dependence is due to the specific characteristics of the crosslinking process in highly crosslinked polymer networks where a few more crosslinks lead to a marked effect on the network density and, thus, on the mechanical properties. Moreover, in the conversion range above ~80%, the microhardness was found to be strictly linear to the conversion. Therefore, it could be used as an independent reference method for the calibration of the NIR measurements, which allows the in-line prediction of the hardness of a coating directly from the NIR spectrum.

Process Control of the UV Curing of Pressure Sensitive Adhesives

UV-curable acrylic hot-melts to be used as pressure sensitive adhesives (PSA) are still a rather new development in UV curing technology.[21] The ratio of cohesion and adhesion in these systems can be varied widely not

Figure 9—In-line monitoring of the conversion in a 300 g/m² coating of an acrylic hot-melt adhesive on PP tape after UV irradiation at 308 nm at various line speeds.

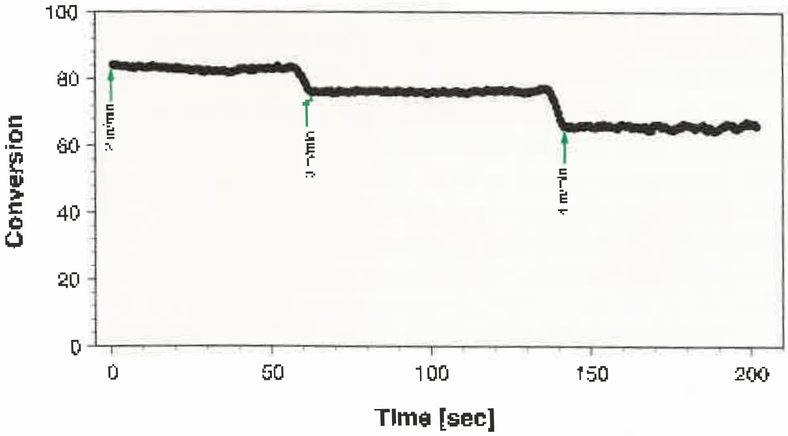
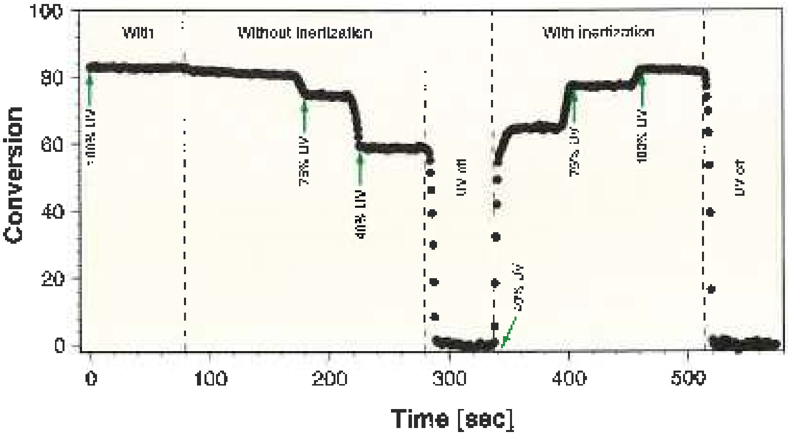


Figure 10—In-line monitoring of the conversion in a 400 g/m^2 coating of an acrylic hot-melt adhesive on PP tape after UV irradiation at 308 nm with variable irradiance at a constant line speed of 3 m/min.



only by the composition of the hot-melt formulation but also by conversion which is achieved during UV irradiation. Accordingly, the adhesive properties of hot-melt layers, i.e., their peel strength and shear strength, were found to respond extremely sensitively even to minor changes of the conversion, which, in turn, strongly depends on the applied UV dose.[22] In particular, this applies to the peel strength. In order to avoid too high or too low conversion, which affects the adhesive properties adversely, an exact and continuous control of the UV curing process is required. Similar to conventional coatings, such as those described previously, NIR reflection spectroscopy also offers a unique possibility to monitor the actual conversion in acrylate-based adhesives. The overtone of the acrylic double bonds likewise appears at 1620 nm as in liquid acrylate monomers and oligomers.

Figures 9 and 10 show two examples of the in-line monitoring of the conversion in acrylic adhesives after UV curing at various web speeds and with various UV intensities, respectively. Due to the very high thickness of the layers, spectra with excellent reproducibility were recorded which results in minor scattering of the conversion data. It can be clearly seen that changes of the irradiation dose due to a variation of the line speed or the irradiance lead to changes of the conversion.

Moreover, the influence of the inertization of the irradiation zone with nitrogen was studied (see *Figure* 10). Purging was found to have a minor but nevertheless detectable effect on the conversion of the acrylic double bonds, which clearly demonstrates the sensitivity of the NIR method. After turning off the purge gas after about 90 sec, a slight decrease of the degree of cure is observed. Furthermore, if the conversion levels at similar irradiance before and after switch-off of the UV lamp are compared, a somewhat higher conversion is found when the irradiation is carried out under nitrogen. The unusually low influence of inertization might be due to the extremely high thickness of the adhesive layers in comparison with conventional acrylate coatings. This limits the penetration of atmospheric oxygen into the deeper lying layers and impedes the replenishment of the exhausted oxygen.

The response of the conversion to any changes of the reaction conditions, in particular after changes of the power of the UV lamp, seems to be quite slow. However, in contrast to the behavior of the mercury arc lamp mentioned above, the excimer lamp responds much faster to control commands, i.e., the delay of the conversion is not due to the inherent properties of the lamp. In fact, it is a consequence of the low line speeds and the lengthwise installation of the excimer lamp being about 40 cm in length that together lead to rather long passage time through the lamp. This long irradiation time is necessary for achieving sufficient crosslinking of the adhesive layers.

CONCLUSIONS

In this study, it was demonstrated that NIR reflection spectroscopy can be effectively used for in-line monitoring of the conversion of double bonds in thin acrylate coatings after UV and EB irradiation. In a number of pilot-scale investigations, the degree of cure was determined in clear and pigmented coatings as well as in nanocomposites on the basis of (meth)acrylates. It was shown that reasonable conversion data can be recorded even at line speeds of at least 120 m/min. Furthermore, NIR reflection spectroscopy has been proven to be useful for monitoring of the conversion in layers from hot-melt adhesives, which is crucial for an efficient control of their adhesive properties. Thus, for the first time, there is an analytical method available which can be reliably applied for process and quality control in technical curing processes.

Some preliminary investigations were also carried out with cationic systems. The overtone band in vinyl ethers is shifted to about 1612 nm, whereas cycloaliphatic epoxies (in contrast to glycidyl ethers) unfortunately do not show a specific overtone or combination band in the near-infrared spectrum which could be used for the monitoring of their conversion. Consequently, only the conversion of the vinyl ether component in cationic formulations can be determined by NIR spectroscopy. Results of these further investigations will be reported in a future publication.

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