AutoEvaluation of DSC Curves: The New Evaluation Function for UV-Curing

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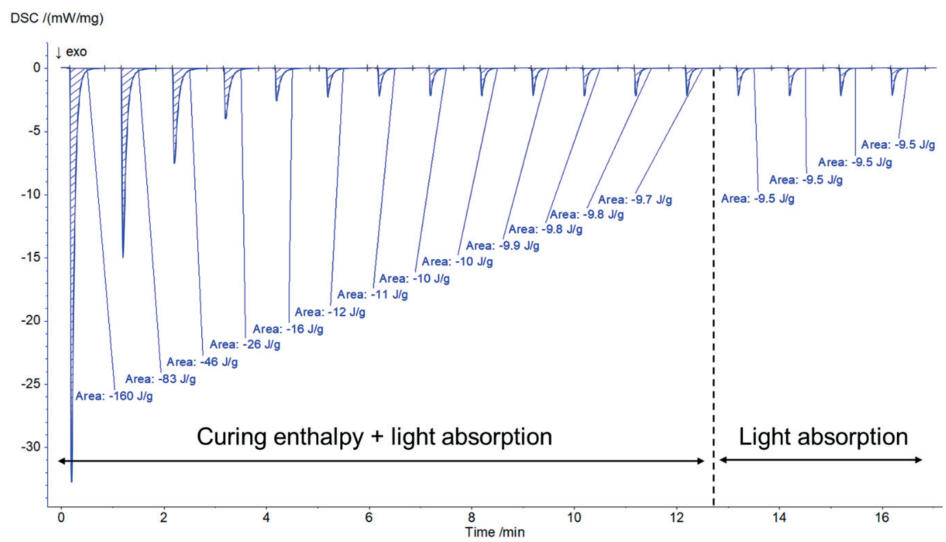
As of *Proteus*® Version 9.5, the new *AutoEvaluation* function, UV-Curing for DSC curves, is available. As usual, it can be selected in the analysis, but it can also be directly integrated into the measurement methods.

Introduction

In light or UV curing of polmyers, a light source initiates a photochemical reaction, leading to changes in the mechanical and chemical properties. Typically, a light-curable polymer system consists of monomers and/or oligomers, as well as a photoinitiator. When exposed to UV light, the photoinitiator decomposes and forms radicals or cations, which initiate polymerization or cross-linking [1].

To make a curing process energy-efficient while ensuring that the system fully reacts, it is necessary to characterize the effect with regard to light intensity, pulse duration, and, if applicable, the number of pulses. The cross-linking effect is an exothermic process that can be measured using differential scanning calorimetry (DSC). This involves coupling of a DSC with a calibrated light source. Typically, multiple pulsed light exposures initiate cross-linking, and the differential scanning calorimeter measures the resulting reaction heat. This method allows for precise measurement of the effect and the relationships mentioned before. However, it requires correction of the heat signal introduced by the light pulse itself. Since the light-induced heat transfer to the sensor differs significantly between an empty metallic crucible and a crucible filled with a sample, it is not practical to perform a typical correction measurement with empty crucibles.

Against this background, it is advisable to expose the sample beyond the end of the cross-linking process and to carry out as many exposure events as necessary until a constant enthalpy value is reached (see figure 1). This value corresponds to the heat introduced by the light pulse (light absorption) and can subsequently be used for correction. Until now, the correction had to be done in a relatively laborious manner, either in *Proteus*® or manually using a separate data processing software tool.



1 DSC curve of UV paint with multiple light exposures

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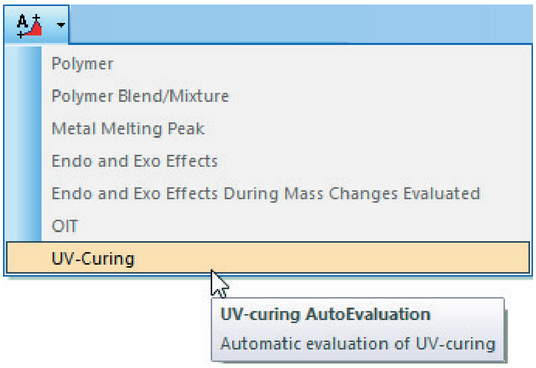
The New *AutoEvaluation* Function: UV-Curing

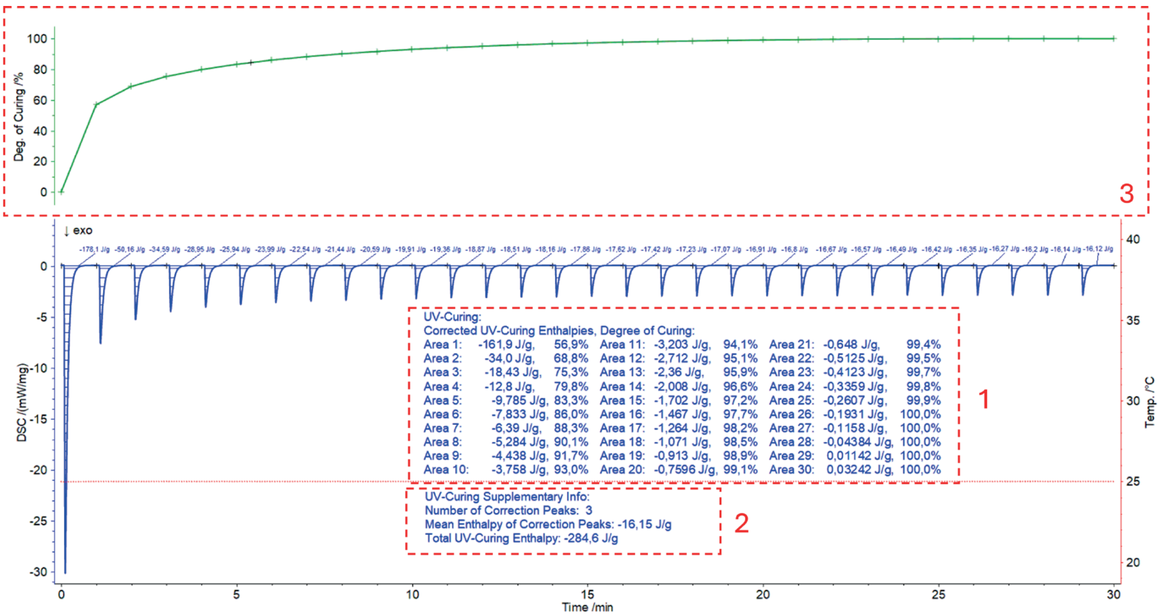
The correction of the heat effect introduced by UV exposure, as well as analysis of the degree of cross-linking, can now be performed fully automatically using the new *AutoEvaluation* function UV-Curing in *Proteus*® as of version 9.5. This option can be found as a new sub-item in the *AutoEvaluation* selection window (figure 2).

By selecting the measurement curve and then clicking on the UV curing function, the fully automatic analysis begins. In this process, the areas of the individual exposure events are evaluated, and an average value is calculated from a user-defined number of the last peak areas, which serves to correct the heat effect introduced by the light pulse.

Figure 3 shows the result for an automatic analysis of a UV paint. It was exposed to 30 light pulses, each for 1 second, at an isothermal temperature of 25°C with defined intensity. As shown, a tabular listing of the corrected reaction enthalpies and the respective percentage cross-linking degree of each exposure is generated (1). Furthermore, the user is provided with important information regarding the total enthalpy of the UV curing process (sum of the corrected reaction enthalpies), as well as the number of correction peaks used and their average value (2). Additionally, the progression of the percentage cross-linking degree can be displayed as a curve (3).



2 *AutoEvaluation* icon in *Proteus*® analysis version 9.5 with UV-Curing function



3 Fully automatic analysis of a UV paint using the new *AutoEvaluation* function UV-Curing

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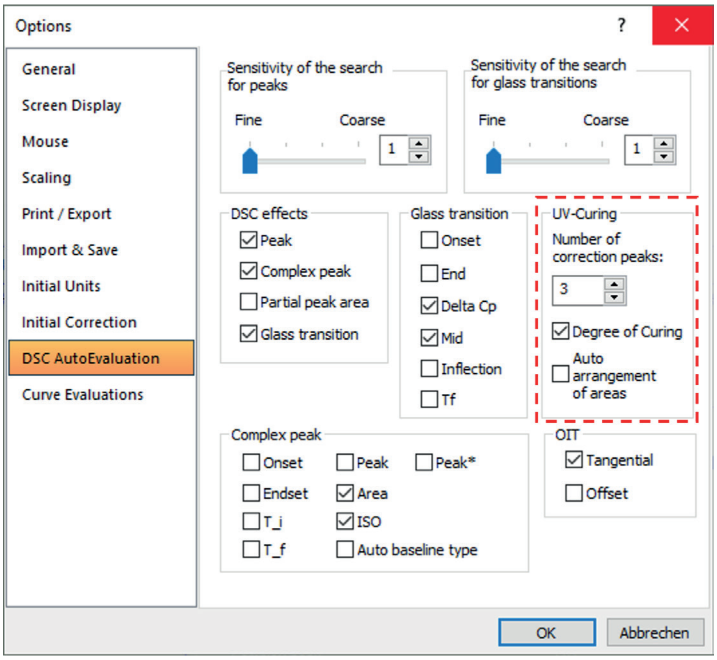
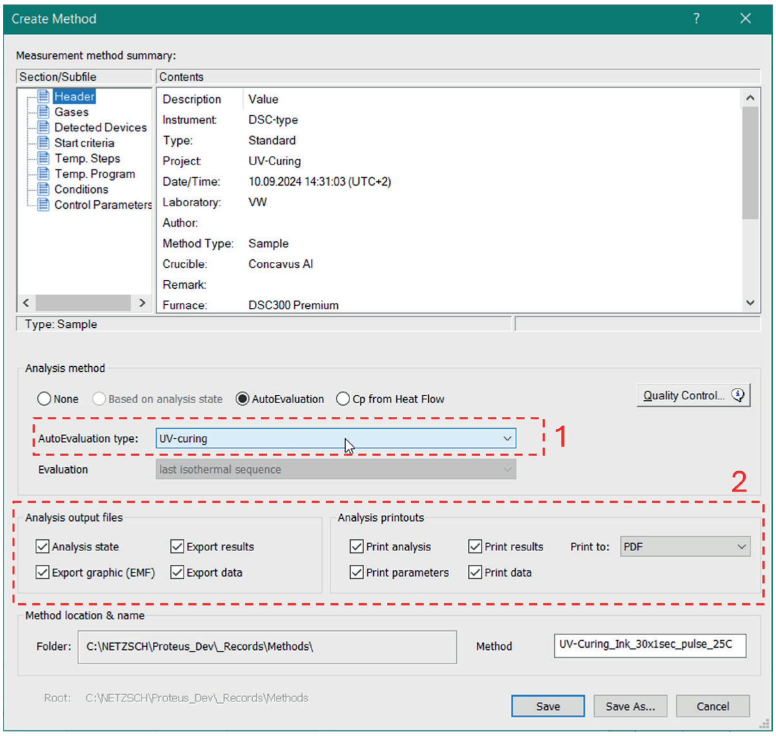
The user can define in the *AutoEvaluation* settings how many areas should be used to calculate the average correction value and whether an analysis of the crosslinking degree should be performed (see figure 4). These settings can be found in the options of the *Proteus*® software.

The UV-Curing function integrates seamlessly into methods via selecting in the final overview window during method generation (see figure 5 (1)). Additionally, the familiar functions for direct result output, such as creating text and image files, are supported (2). This can significantly reduce the evaluation effort, especially for users with a large number of measurement tasks, as the explained evaluation results are generated fully automatically at the end of the measurement and can be exported if necessary.



4 Settings of the *AutoEvaluation* funktion UV-Curing



5 Measurement method summary

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Summary

The expansion of the NETZSCH *Proteus*® software in version 9.5 with the *AutoEvaluation* function UV-Curing significantly simplifies the analysis of measurements for light-curing materials. There is no longer a need to transfer parts of the calculation steps to other programs, nor does the user need to manually add the values of the individual areas.

By starting the UV-Curing function, an automatic correction of the heat introduced by the light pulse is performed, as well as an evaluation of the partial enthalpies, a display of the total reaction enthalpy, and the percentage of cross-linking. The *AutoEvaluation* function UV-Curing can be directly integrated into methods, further reducing the effort required for analysis.

Sources

[1] E. Reichmanis, J. Crivello: Photopolymer Materials and Processes for Advanced Technologies. In: Chem. Mater. Band 26, 2014, S. 533–548.

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