Thermal Analysis Option

DSC-Photocalorimetry System

Study of Photoinitiated Reactions

Manufacturing organizations are constantly faced with the need to be more competitive and to look for new markets. The challenges are to increase output and reduce costs, and so become more efficient and effective. Part of this involves exploring new ways to improve existing processes. For example, processes involving light curing offer optimization possibilities in the following areas:

- **Time** – light-curing systems shorten processing times and reduce costs
- **Temperature** – temperature-sensitive substrate materials can be coated because light curing is performed at lower temperatures compared to thermal curing
- **Environment** – only negligible amounts of volatile organic compounds (VOCs) are released into the environment

The DSC-Photocalorimetry System enables you to study such process improvements in your laboratory.

Features and benefits

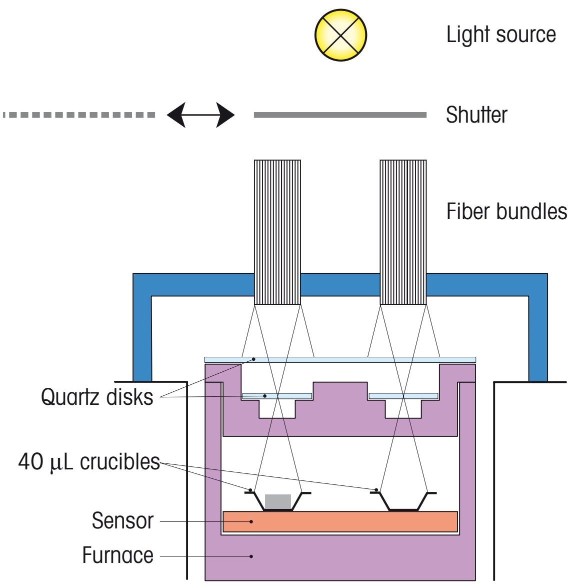
- **Versatile experimental parameters** – the effect of light intensity, wavelength range, temperature, and time on photoinitiated reactions can easily be investigated
- **Simulation of production conditions** – curing times can be studied in the laboratory, which greatly reduces costs
- **Product development optimization** – the stability behavior of materials and aging effects can be quickly determined
- **Modularity** – a DSC X or a DSC82x[e] can be easily upgraded to a DSC-Photocalorimetry System

METTLER TOLEDO

Optical Design
of the DSC-Photocalorimetry System

The photocalorimeter accessory is used in combination with the METTLER TOLEDO DSC X or DSC82x^{e} and extends the application possibilities of DSC into the field of light curing and, in general, to the study of photoinitiated reactions.

DSC-photocalorimetry allows enthalpy changes in a material to be measured during and after exposure to light of certain wavelengths for different periods of time at different temperatures. This means that the effects of light on the behavior of light-sensitive materials used in the plastics, electronics, healthcare, chemical, food and pharmaceutical industries can be investigated.



Cross-section of the optical system and the DSC furnace

Light-activated curing processes and photoinitiators, as well as the influence of UV stabilizers and the effect of light intensity on polymer stability in accelerated testing or aging studies are some of the topics frequently studied.

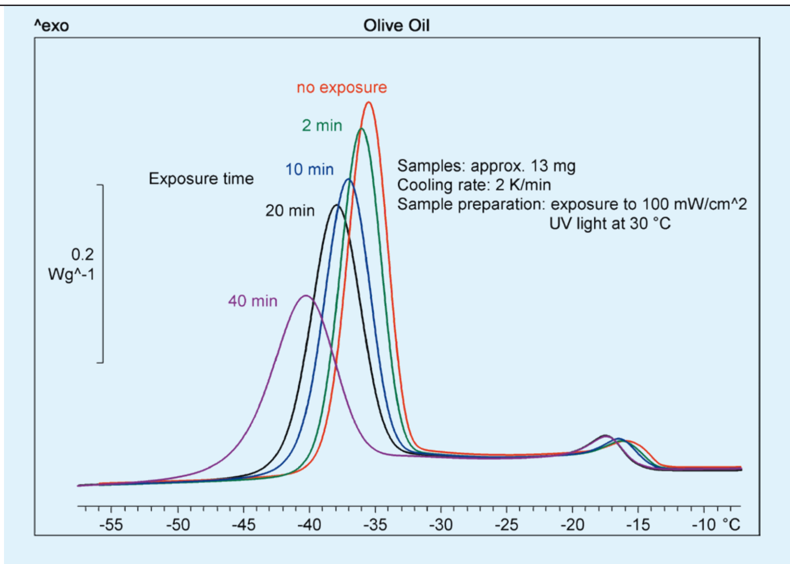
The optical arrangement with the interchangeable light source is designed so that the sample can be exposed

- to a defined light intensity
- of a particular wavelength range
- for different periods of time
- at different temperatures.

The DSC sensor measures the sample temperature and enthalpy changes that occur in the sample during the course of the reaction.

Industry	**Applications**
Plastics (elastomers, thermosets, thermoplastics)	• Influence of UV stabilizers on the stability of the materials • Chemical reactions – polymerization
Electronics	• Curing of resins and adhesives, e.g. in optoelectronic devices
Paint, lacquers, adhesives, coatings	• Curing, establishing optimum process conditions (temperature, light intensity and exposure time)
Healthcare	• Light curing of dental composites
Chemicals (organic and inorganic materials, pharmaceutical products) and food	• Influence of light on stability • Aging effects • Study of fats and oils

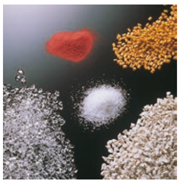
Application Examples

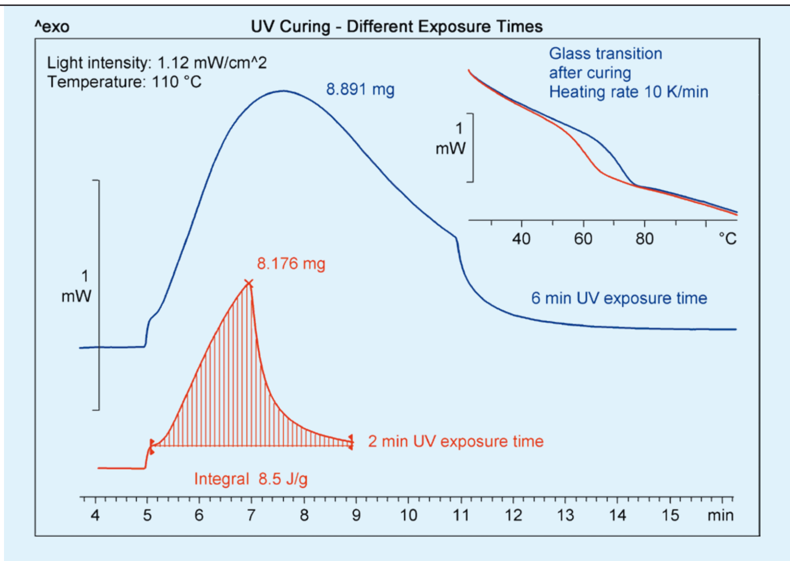


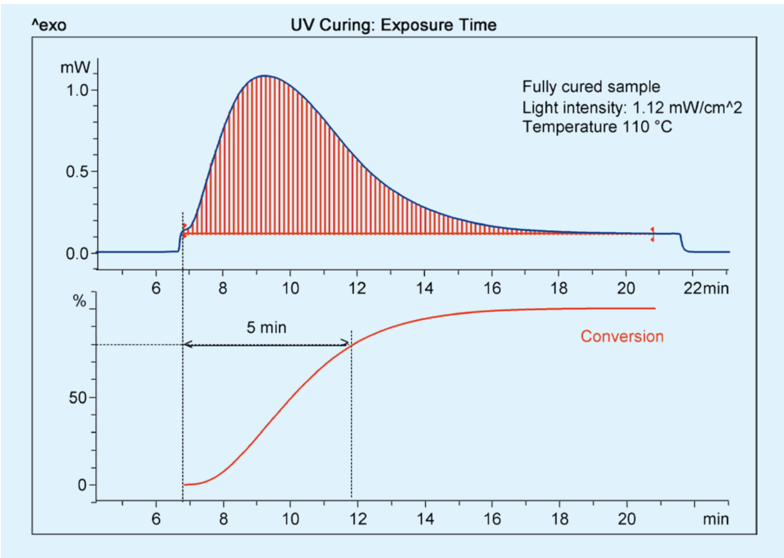
Influence of UV exposure time on olive oil

The action of UV light on foodstuffs can lead to degradation and depolymerization with an accompanying negative effect on aroma and taste.

In the example shown, olive oil was exposed to UV light (100 mW/cm^2) for different periods of time. After exposure, the crystallization behavior of the samples was investigated. The results show that UV light exposure clearly has a significant effect. With increasing exposure time, crystallization occurs at lower temperatures. In addition, the enthalpy of crystallization decreases.





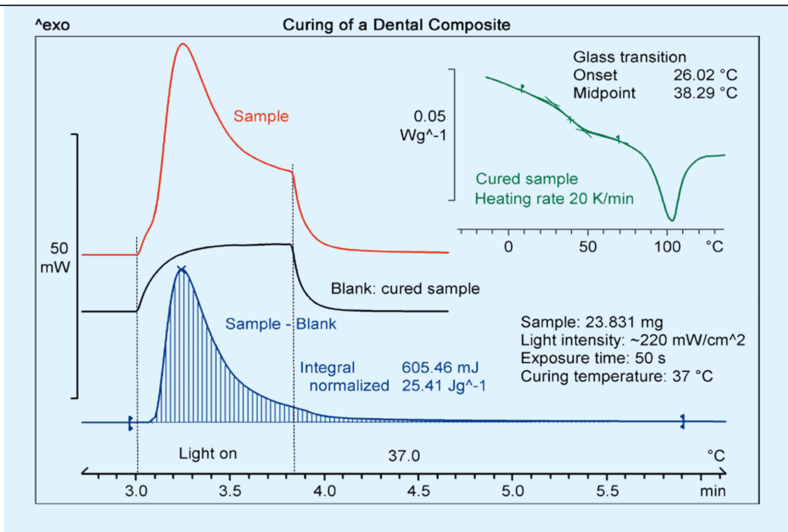


Powder coatings

Powder coating technology is nowadays applied to a wide range of different materials (wood, plastics, metals). Besides the excellent mechanical and chemical properties of powder coatings, their use also offers important ecological advantages. For example, unlike solvent-based paints, only negligible amounts of volatile organic compounds (VOCs) are released into the environment.

The powder coating is usually sprayed onto the substrate and then cured either thermally (typically at about 180 °C) or by means of UV light at lower temperatures. Curing with UV light has the great advantage that temperature-sensitive materials can be coated.

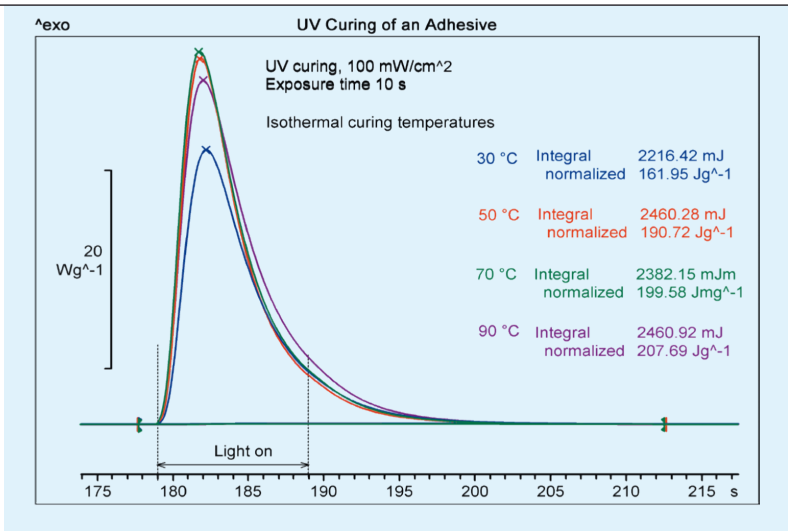
In practice, the main question is how long does the material have to be exposed to UV light in order to achieve an adequate degree of cure or crosslinking. This is illustrated in the example for a powder coating system. Several experiments were first performed to measure the degree of cure for different exposure times. The necessary exposure time can then be simply determined from the requirements set for the degree of cure. In this example, an exposure time of 5 min is necessary for a desired degree of cure of 80%.

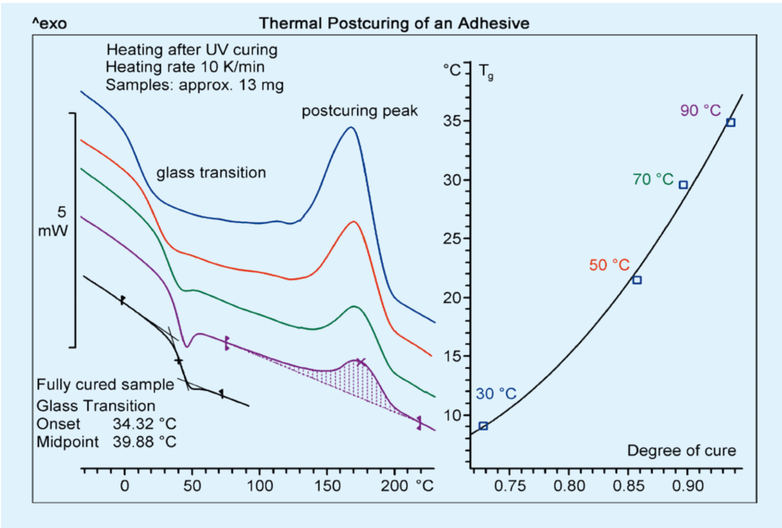


Curing of a dental composite

Light-curing composites are nowadays widely used in dentistry to fill cavities in teeth. DSC-photocalorimetric measurements enable the curing process of these dental fillings to be followed.

This is demonstrated in the example showing the curing of a composite filling material under the action of light. In this application, a white light source with a high percentage of light between 400 and 500 nm was used. The glass transition of the cured composite filling is at about 38 °C and is relatively broad. The endothermic peak following the glass transition is due to the melting of a constituent of the composite.





UV curing of an adhesive

UV-curing adhesives are widely used in the electronics industry. Electronic components can be fixed permanently in place within a few seconds.

The example shows the curing behavior of a technical product at different temperatures. The exposure time was 10 s. The crosslinking process is apparent as a strongly exothermic peak. With increasing temperature, the peak area becomes larger, indicating that the degree of cure increases with increasing temperature.

This was confirmed when the thermal postcuring of the UV-cured sample was investigated. The postcuring is observed as an exothermic peak after the glass transition. The degree of cure of the UV-cured sample can be calculated from its curing and postcuring enthalpies. The inserted diagram shows the relationship between the glass transition temperature and the degree of cure. To achieve a degree of cure of 90%, the curing process must be performed at about 70 °C. This is in fact the temperature recommended by the supplier.

Literature: M. Schubnell, "Curing of powder coatings using UV light", UserCom 19, 13

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