Thermokinetic investigations on two-step curing reactions in melt

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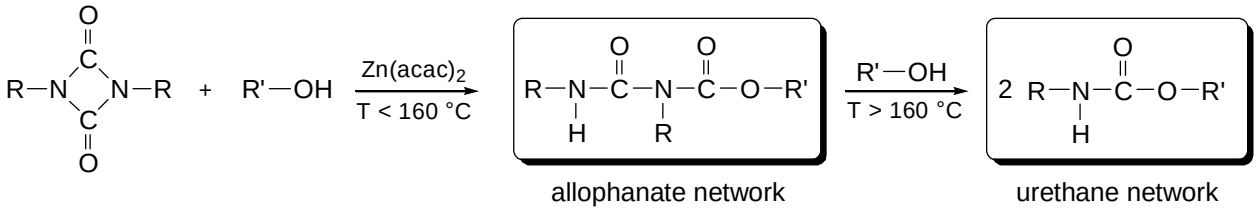
The curing process of low temperature thermoset powder coatings was found to proceed via a two step mechanism [1]. In presence of a suitable catalyst (e. g. zinc(II)-acetylacetonate) the reaction between uretdione cross-linkers and OH-functionalized polyesters to PUR powder coatings occurs via an allophanate structure which was build at temperatures below 150 °C by ring opening of the uretdione ring. This allophanate network exhibit already excellent mechanical and optical properties of the laquer layer. At higher temperatures the allophanate was converted into the resulting urethane network. Applying adequate temperature programs enables the selective separation of the two reaction steps.



Herein we report about the formal kinetic investigation of this two-step curing reaction in melt. The kinetic parameters were determined by non-isothermal DSC measurements. The aim of our work was to find formal kinetic reaction models for two step reaction between uretdiones and OH-groups to make some predictions about the reaction behaviour.

We started our investigations with a low molecular model system in which the OH-functionalized component was replaced by 1,8-octanediol. The determination of the kinetic parameters (activation energy, pre-exponential factor) was carried out using the "NETZSCH-Thermokinetics" software. Afterwards some predictions for the formation of the allophanate network were made and verified using isothermal DSC measurements and FT-IR-spectroscopy.

As a further step the investigations were expanded to polymeric systems, where an OH-functionalized was used. However the used polyester contained some synthetically caused COOH terminal groups which inhibited the activity of the catalyst. These COOH groups were esterfied by addition of an epoxide in a previous reaction. Again suitable formal kinetic reaction models were searched to describe as well as possible the two step curing process. By use of the kinetic parameters some predictions of the curing process were done. The results of these predictions were verified using isothermal DSC measurements and FI-IR spectroscopy.

In addition the influence of different preparative methods of the polymeric model systems were examined. The polymeric model systems were prepared by the use of a lab kneeder and by the use of a lab extruder, which was of more practical importance in view of the preparation of powder coatings.

References

[1] Lehmann, F.; Gedan-Smolka, M.; Lehmann, D., Farbe und Lack 106, p. 62 (2000)