*Journal of Thermal Analysis and Calorimetry, Vol. 83 (2006) 1, 199–206*

# *TTT* ANALYSIS OF A POWDER COATING SYSTEM BASED ON POLYESTER RESIN AND TRIGLYCIDYL-ISOCYANURATE
## Part I. Gelation of matrix and powder

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The gelation of a powder coating and its matrix based on a saturated carboxyl functional polyester resin and triglycidyl isocyanurate of different diastereomer racemate composition was investigated by rotational viscometry. The iso-$T_{\mathrm{cure}}$ diagrams have been determined directly by isothermal viscometric measurements and the gelation curves for the *TTT*-diagram have been constructed. To the measured section of the gelation curves power functions could be fitted and with their help the reactivity of the investigated systems were compared. The ratio of the two diastereomer racemates of TGIC has a significant effect on the reactivity of the coating, as it was also supported by DSC measurements. $\beta$-TGIC is of highest reactivity, and by increasing its ratio in TGIC, reactivity increases, and adversely effects the performance of the powder coating. Commercial TGIC-s have similar reactivity, comparable to that of high $\alpha$-TGIC. The reactivity of the matrix is higher than that of the powder.

***Keywords:*** *gelation, powder coating, TGIC isomers, time–temperature–transformation analysis*

## Introduction

During the setting of reactive systems many events take place simultaneously such as chemical reactions, elimination of low molecular materials, such as side products of the curing or simply evaporation of solvents, and thermal or thermooxidative degradation. Time–temperature–transformation (*TTT*) analysis is a useful tool to investigate the different processes during the cure supplying information to the processor or user of thermosetting systems, and giving a deeper understanding of material behaviour during the cure. The *TTT* diagram shows the relation between the time, temperature, and the conversion of curing. One of the most important iso-conversion line is the gelation curve.

The formation of network polymers is preceeded by gelation. The theory of gel formation was studied by Flory [1–3], Stockmayer [4], Silberberg [5], Winter *et al.* [6]. Tajima and Crozier [7] studied the chemorheology of an amine-cured epoxy resin, and used the Williams–Landel–Ferry equation [8] to describe the temperature-dependence of the viscosity. For tetrafunctional branching units Flory's classic analysis resulted a conversion of 58% at the gel point [3]. Mijović *et al.* [9] applied a mechanistic kinetic model to calculate the time needed to reach 58% conversion of an epoxy-amine thermosetting system, which was in perfect accordance with the gel time from Flory's model. Van der Linde *et al.* [10–12] used thermoanalytic (DSC) and thermoviscoelastic (rheometric) measurements in order to describe the kinetics of the setting of polyester-based powder coating systems. Vinnik and Roznyatovsky used calorimetry for their kinetic method in order to investigate the mechanism of epoxy-amine curing reaction [13, 14]. Neag and Prime applied time-temperature superposition technique for the investigation of powder coating cure [15]. Nuñez *et al*. determined the gel times of an epoxy-amine curing system as a function of temperature by using solubility test [16]. Halász and Vorster [17] used dynamic stress rheometry for the evaluation of the curing kinetics of carboxyl functional polyester–TGIC powder coating and distinguished three stages of curing by determining the activation energy and rate constants from the rheological parameters.

Rotational viscometry is a practical tool to follow the curing behaviour of a thermosetting system before and up to gelation. Isothermal curing can be carried out in the rotational viscometer at a given temperature, and the change in apparent viscosity can be measured during the curing process. The isothermal viscosity curves directly supply the iso-curing temperature (iso-$T_{\mathrm{cure}}$) diagrams determined by viscometry. These curves also enable to determine the points of the gelation curve, which is part of the *TTT* diagram. Further on the activation energy of the curing reaction can also be determined [8, 18].

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*1388–6150/$20.00*

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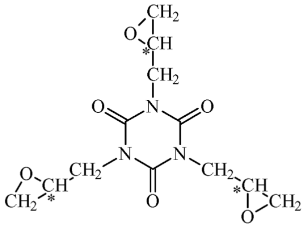
Triglycidylisocyanurate (TGIC) is a trifunctional epoxy compound of high reactivity with the following structure (Scheme 1):



**Scheme 1**

TGIC has three chiral carbon atoms, the number of optical isomers should be eight ($2^3=8$). Due to its symmetrical structure, four antipodes may be distinguished, in two the configuration of the chiral carbon atoms is the same, i.e. RRR or SSS, and in the other two the configuration of one chiral carbon atom is opposite to that of the other two chiral carbon atoms, i.e. RRS and SSR. Consequently two diastereomer racemates of TGIC exist, namely α-TGIC consisting of the RRS/SSR and β-TGIC consisting of the RRR/SSS antipodes. The physical and chemical properties of the two diastereomer racemates are different [19, 20]. The binary phase diagram enables the determination of the content of the two diastereomer racemates in TGIC [21].

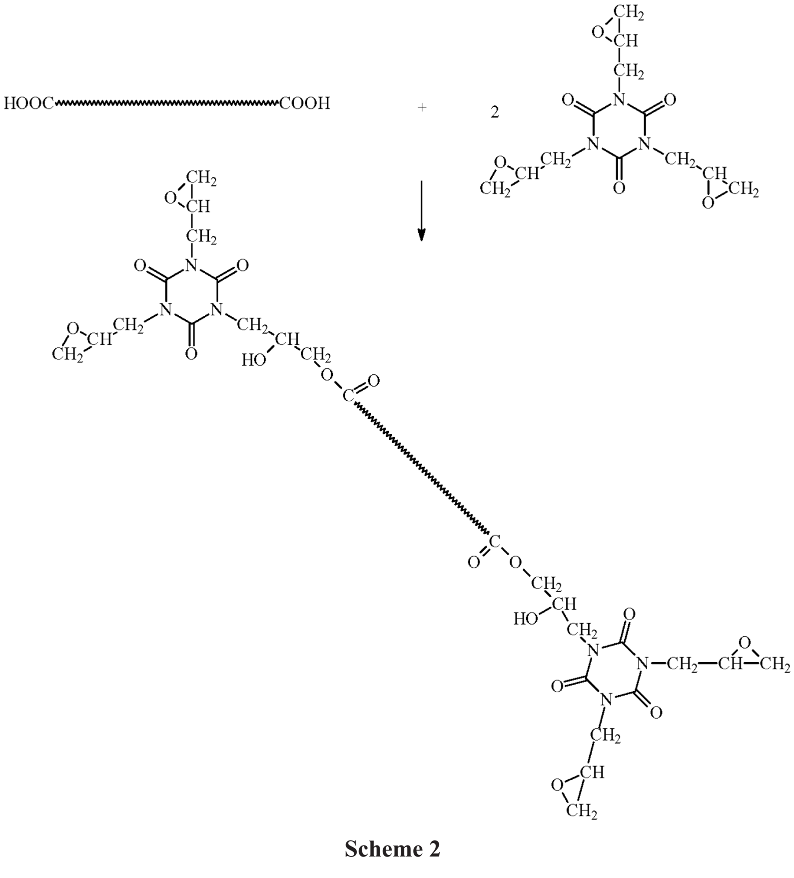
The ultimate goal of our experiments is the determination of the time–temperature–transformation diagram of the powder coating system based on a saturated, carboxyl functional polyester resin and triglycidyl-isocyanurate. The aim of this work is the analysis of the rheological behaviour of the thermosetting system, and the determination of the gelation curve both for matrix and for powder. Further on our goal is the elucidation of the effect of the diastereomer racemate composition on the curing behaviour of matrix and powder.

## Chemical reactions during the curing process

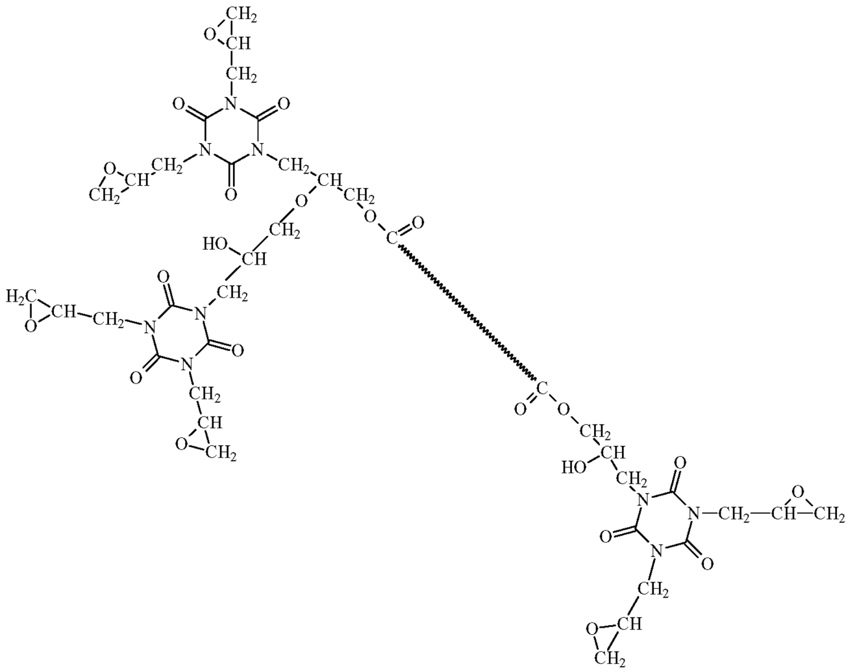
The main cross-linking reaction is the addition of the carboxyl groups of the polyester resin to the epoxy groups of TGIC (Scheme 2).

Since the main cross-linking reaction results the formation of secondary OH groups, these latter may also add to the epoxy groups of TGIC (Scheme 3).

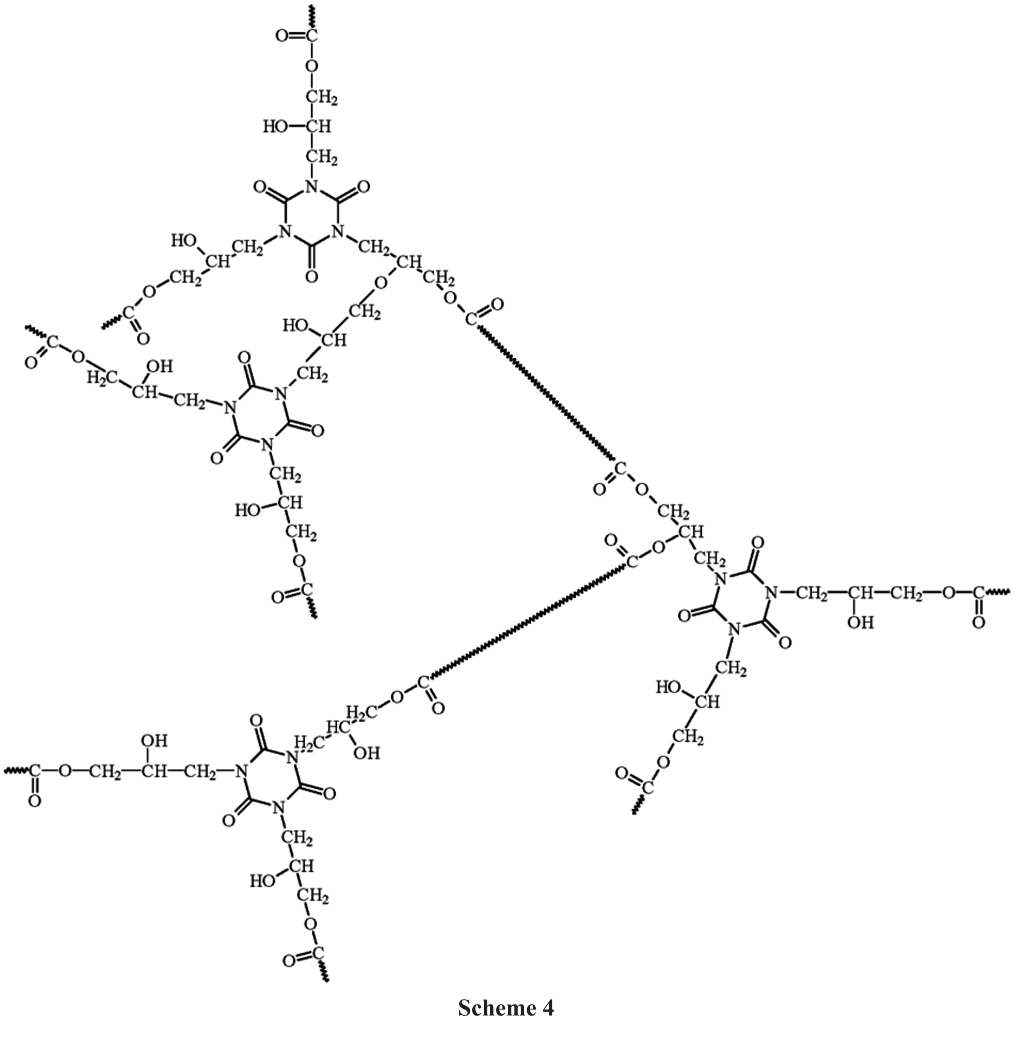
In later stage of cross-linking the reaction of secondary OH groups and of the carboxyl groups of the polyester resin may also occur with the elimination of water. All the above reactions result the following structure (Scheme 4).



**Scheme 2**



**Scheme 3**



**Scheme 4**

## Experimental

### *Materials*

Uralac P2400

Saturated, carboxyl functional polyester resin; Supplier: DSM Resins.

Properties given by the supplier: Acid number (ISO 3682): 30–37 mg KOH/g; $T_g$ (Mettler TA 2000): 63°C.

Measured parameters: Melt viscosity: at 170°C 27.39 mPa s.

Araldit PT 810

Triglycidyl-isocyanurate; Supplier: Ciba Geigy

Properties given by the supplier; Epoxy group content ≥9.3 mmol $g^{-1}$; Epoxy equivalent mass ≤107 g $ekv^{-1}$; Melting point: 94±3°C; Powder density: 0.72 g $cm^{-3}$; Melt viscosity: at 150°C, 20 mPa s; at 120°C, 80 mPa s; at 100°C, 320 mPa s; Gelation time: cca. 90 s (180°C, Uralac P2400/Araldit PT810 93/7 by mass).

Measured parameters: Melting point above the eutectic one: 112°C; β-TGIC content: 22.5% [21]; Melt viscosity: at 165.9°C 16.3 mPa s; at 140.6°C, 28.8 mPa s; at 129°C, 52.4 mPa s.

Tepic G

Triglycidyl-isocyanurate; Supplier: Nichimen Co.

Properties given by the supplier; Epoxy group content ≥9.09 mmol $g^{-1}$; Epoxy equivalent mass ≤110 g $ekv^{-1}$; Melting range: (Auto Mettler): 90–125°C; Density: 1.42 g $cm^{-3}$ (at room temperature); 1.33 g $cm^{-3}$ (at 120°C); Colour (Gardner): <1; Viscosity (JIS K 6833): <100 mPa s; Hydrolisable chlorine: <2 %.

Measured parameters: Melting point above the eutectic one: 122°C; β-TGIC content: 36.5% [21]; Melt viscosity: at 170°C, 10.77 mPa s; at 167°C, 11.8 mPa s; at 157°C 12.4 mPa s; at 137.5°C, 31.34 mPa s; at 131.7°C, 47.2 mPa s.

β-TGIC (RRR/SSS)

Prepared at the laboratory of the Department of Plastics and Rubber Technology of the Budapest University of Technology and Economics [22–24]. Melting point: 153°C (determined by DSC); Melt viscosity: at 167°C, 6.45 mPa s.

α-TGIC (RRS/SSR)

Prepared at the laboratory of the Department of Plastics and Rubber Technology of the Budapest University of Technology and Economics [22–24]. Melting point: 99.8°C, α-TGIC content >99% [21]. Melt viscosity: at 167°C, 4.7 mPa s; at 158°C, 15.7 mPa s; at 140°C, 31.3 mPa s; at 130°C, 44.3 mPa s.

TGIC of high β-TGIC content

Prepared at the laboratory of the Department of Plastics and Rubber Technology, Budapest University of Technology and Economics [22–24]. Melting point above the eutectic one: 136°C, β-TGIC content: 61% [21].

### *Preparation of the materials*

Matrix materials were prepared by homogenizing 60 g of the components in an internal mixer (Brabender plastograf Walzenkneter Typ PL 38) at 100°C for 30 min at a rate of 30 $min^{-1}$. The mass ratio of polyester resin to TGIC was 93:7, which equals to the ratio of 1.2 mole epoxy to 1 mole carboxyl groups.

For powder coatings the following components were used: Uralac P2400 (polyester resin); TGIC; Titanium dioxide; Bariumsulphate; Benzoin; Optical whitener; Modaflow III (flowing agent).

The components after weighing them in, were extruded at 100°C (residence time 15–20 min), ground and sieved. Two formulations have been made, in which the TGIC was different. One was made with TGIC of 61% β-TGIC content and the other formulation contained a commercial TGIC, Tepic G.

### *Methods*

Functional group analysis

The epoxy group content of the pretreated formulations was measured by the pyridinhydrochlorid method according to MSZ 10195/7-78.

Differential scanning calorimetry

The enthalpy of curing was measured by DSC analysis. A Perkin Elmer DSC7 was used. The measurements were carried out with 5 mg sample in purging nitrogen (40 mL $min^{-1}$) at a rate of 5°C $min^{-1}$.

Rheology

Rheological measurements took place with a Rheotest RV 2 rotational cone- and plate viscometer. Cone diameter was 36 mm, range of shear rate $10^{-2}$–$10^{2}$ $s^{-1}$. Temperature of the material was measured directly with a thermoelement inserted into it.

## Results and discussion

### *Functional group analysis and DSC*

The reaction of epoxy groups in matrix during homogenization in Brabender plastograf at 100°C for 30 min has been tested by functional group analysis. The samples after homogenisation were cured in the differential scanning calorimeter and the heat of reaction measured. Aim of these experiments was the evaluation of the reactivity of TGIC with different ratio of diastereomer racemates. The results are shown in Table 1.

From the results of Table 1 follows that β-TGIC has the highest reactivity, which means that the conversion of the reaction of epoxy groups is nearly 50% during homogenization, and the heat of curing is the least compared with the other TGIC's. This is disadvantageous due to the possibility of side reactions during the successive curing process, and the preliminary increase in viscosity, as supported by our rheological measurements. To side reactions belongs the esterification of carboxyl- and secondary hydroxyl groups, with elimination of water, which consumes heat. This is the other reason of the lowest heat of reaction with β-TGIC measured for the curing after homogenisation. The reactivity of the commercial TGIC, Tepic G is higher than that of Araldit PT810 due to its different diastereomer racemate composition.

### *Rheology*

Isothermal curing of the homogenised mixtures have been carried out in the rotational viscometer at different temperatures, and the change of apparent viscosity with time of curing was detected. Aim of these measurements was the determination of the gelation curve for each system and the elucidation of the difference in reactivity of TGIC's with different diastereomer racemate composition. The isothermal curing of the matrix containing the polyester resin and β-TGIC at different temperatures is shown by Fig. 1.

Figure 1 shows the effect of temperature on the course of curing time-apparent viscosity diagrams, when β-TGIC is the curing agent. At 130°C the diagram can be fitted with a polynomial of second order (Table 2). The point of gelation at this temperature could not be determined, since during the time of measurement no gelation occurred. The viscosity at $t$=0 min can be determined as 72 Pa s, which is close to the measured value of 64 Pa s. At 138°C two reaction stages can be observed. During the beginning stage up to 5 min the viscosity increase is slow, and the curve can be fitted with a polynomial of second order. The viscosity at $t$=0 at 138°C is calculated as 5 Pa s, while the measured value is 4.8 Pa s. Above 5 min the viscosity increase with the time of reaction is steep, and follows an exponential curve (Table 2). The time of gelation is determined as the abscissa of the intercept of the straight lines fitted to the beginning and final section of the diagram. At 157°C the first reaction stage is shorter, and lasts up to 2 min. The polynomial fitted to the curve is of second order. The apparent viscosity at $t$=0 was calculated as 17.9 Pa s, while the measured viscosity is 17.2 Pa s. The second section of the reaction starts at 2 min, and follows an exponential curve (Table 2). At 168°C the first reaction stage lasts only up to 1 min reaction time. The curve fitted to this section is also a polynomial of second order. The viscosity at $t$=0 min was found to be 6.5 Pa s, the measured one 6.3 Pa s. The reaction after 1 min follows an exponential curve (Table 2). At 170°C the first reaction stage lasts only up to 0.5 min, the polynomial is of second order. The calculated and measured viscosity at $t$=0 min is 2.8 Pa s. The second section follows an exponential curve (Table 2).

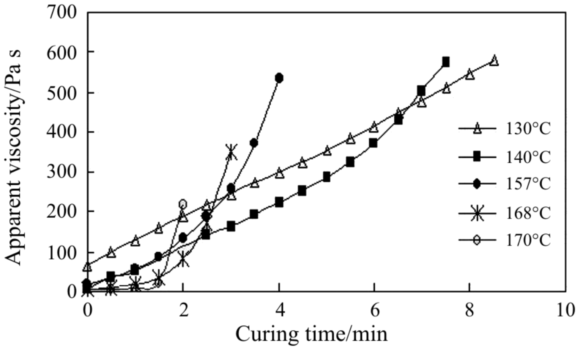


**Fig. 1** Iso-$T_{\mathrm{cure}}$ diagrams of Uralac P2400–β-TGIC matrix determined by rotational viscometry

Figure 2 represents the iso-$T_{\mathrm{cure}}$ diagrams of the matrix containing Uralac P2400 and α-TGIC.

**Table 1** Conversion during homogenization of Uralac P2400 and TGIC (mass ratio 93 to 7) in Brabender Plastograf at 100°C for 30 min and enthalpy of successive curing

| Curing agent | Epoxy group content/mmol $g^{-1}$ | | Conversion of epoxy groups during homogenization/% | Enthalpy of successive curing/J $g^{-1}$ |
|---|---|---|---|---|
| | before homogenization | after homogenization | | |
| β-TGIC | 0.707 | 0.372 | 47.4 | –22.85 |
| α-TGIC | 0.686 | 0.467 | 31.9 | –29.90 |
| Araldit PT810 | 0.642 | 0.464 | 27.7 | –31.62 |
| Tepic G | 0.642 | 0.369 | 42.5 | –26.52 |

**Table 2** Equations of the curves fitted to the beginning and final section of the curing time – apparent viscosity diagrams of Uralac P2400-TGIC matrix

| $T$/°C | Curing agent | Equation of the curve fitted to the beginning section | | $R^2$ | Equation of the curve fitted to the final section | | $R^2$ |
|---|---|---|---|---|---|---|---|
| 130 | β-TGIC | $\eta=0.5425t^2+54.579t+71.914$ | | 0.999 | no final section | | – |
| 130 | α-TGIC | $\eta=2.6659t^2+27.211t+125.03$ | | 0.999 | no final section | | – |
| 138 | β-TGIC | $\eta=0.8155t^2+50.619t+4.9983$ | $t<5$ min | 0.999 | $\eta=68.699e^{0.2833t}$ | $t\geq5$ min | 1 |
| 140 | α-TGIC | $\eta=4.75t^2+10.463t+75.024$ | $t<5$ min | 0.998 | $\eta=53.572e^{0.3109t}$ | $t\geq5$ min | 0.997 |
| 157 | β-TGIC | $\eta=14.745t^2+23.211t+17.924$ | $t<2$ min | 0.997 | $\eta=32.085e^{0.7002t}$ | $t\geq2$ min | 0.999 |
| 157 | α-TGIC | $\eta=15.149t^2-18.719t+28.982$ | $t<3$ min | 0.979 | $\eta=11.886e^{0.7761t}$ | $t\geq3$ min | 0.999 |
| 168 | β-TGIC | $\eta=16.398t^2-7.2454t+6.5143$ | $t<1$ min | 0.998 | $\eta=3.13e^{1.5884t}$ | $t\geq1$ min | 0.998 |
| 168 | α-TGIC | $\eta=8.45t^2+4.399t+3.262$ | $t<2$ min | 1.00 | $\eta=5.7825e^{1.5121t}$ | $t\geq2$ min | 0.997 |
| 170 | β-TGIC | $\eta=9.324t^2-1.562t+2.76$ | $t<0.5$ min | 1.00 | $\eta=1.9565e^{1.631t}$ | $t\geq0.5$ min | 0.997 |
| 170 | α-TGIC | $\eta=7.619t^2-7.4629t+8.6467$ | $t<1$ min | 0.999 | $\eta=3.3086e^{0.9919t}$ | $t\geq1$ min | 0.999 |

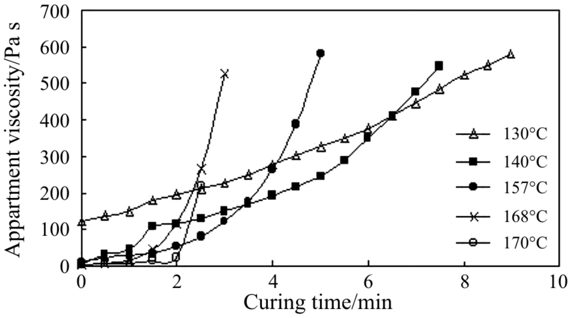


**Fig. 2** Iso-$T_{cure}$ diagrams of Uralac P2400 and α-TGIC matrix determined by rotational viscometry

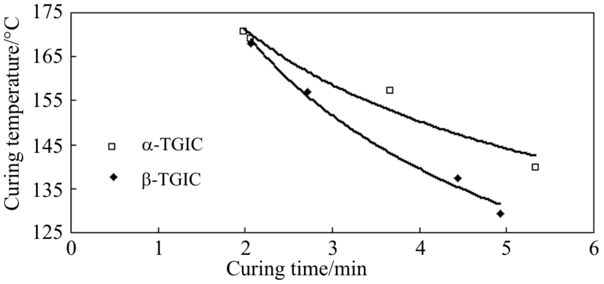


**Fig. 3** Gelation curve of the system containing the matrix polyester resin Uralac P 2400 prepared with β-TGIC and α-TGIC

The coarse of curing time-apparent viscosity diagrams is similar to that of the matrix prepared with β-TGIC, although the reactivity of β-TGIC is higher than that of α-TGIC. This is proven by representing the equations of the curves fitted to the beginning and final section of curing time-apparent viscosity diagrams of matrices prepared with β-TGIC and α-TGIC, respectively (Table 2). If we compare the equations fitted to the final reaction stage at a given temperature, the values of the exponents are comparable for both TGIC's, although the coefficient of the equation is higher for β-TGIC, meaning that the curve is steeper due to higher reactivity. From Figs 1, 2 and Table 2 it is also clear that with the increase of temperature the time of the first reaction stage is decreasing, and the exponential of the second reaction stage is steeper, i.e. the exponent is increasing. The assumption may be made that during the first stage of curing the main cross-linking reaction, i.e. the addition of carboxyl groups to the epoxy groups predominates, while during the transient and final reaction stage side reactions will be advanced.

The difference in reactivity of the two diastereomer racemates of TGIC are also represented by the gelation curve of the two matrix prepared with β-TGIC and α-TGIC (Fig. 3) and by the equation of the curves fitted to the measured section of the gelation curves (Table 3).

The difference in reactivity highly effects the performance of the powder coating as will be shown later.

The iso-$T_{cure}$ diagrams of the matrices prepared with the two commercial TGIC-s, i.e. Araldit PT 810 and Tepic G are shown in Figs 4 and 5.

**Table 3** Equations of the curves fitted to the gelation diagram of coating systems determined by isothermal curing by viscometry

| Material | Equation of the curves fitted to the gelation diagrams | $R^2$ |
|---|---|---|
| Uralac P 2400–β-TGIC matrix | $T_{gel}=196.20\ t_{gel}^{-0.2085}$ | 0.996 |
| Uralac P 2400–α-TGIC matrix | $T_{gel}=186.34\ t_{gel}^{-0.1322}$ | 0.997 |
| Uralac P 2400–Araldit PT 810 matrix | $T_{gel}=189.95\ t_{gel}^{-0.1666}$ | 0.998 |
| Uralac P 2400–Tepic G matrix | $T_{gel}=195.78\ t_{gel}^{-0.1970}$ | 0.998 |
| Uralac P 2400–Tepic G powder | $T_{gel}=228.55\ t_{gel}^{-0.1793}$ | 0.999 |
| Uralac P 2400-high (61%) β-TGIC powder | $T_{gel}=223.77\ t_{gel}^{-0.2869}$ | 0.999 |

The reactivity of the two commercial TGIC-s is much lower than that of β-TGIC, as represented by the gelation curve of the matrix resins on Fig. 6 and by the equations of the fitted curves in Table 3. Tepic G is somewhat more reactive in the curing with the polyester, than Araldit Pt 810, which may be due to the higher β-TGIC content of Tepic G.

Figures 7 and 8 represent the iso-$T_{\text{cure}}$ diagrams of the powder coatings prepared with Tepic G and a mixture of TGIC diastereomer racemates with high (61%) β-TGIC content.

The gelation curves of the powder coating prepared with Uralac P2400 and Tepic G and of the matrix of same composition are represented by Fig. 9. The equations fitted to the measured section of the gelation curves are given in Table 3. It is evident, that the powder has less reactivity compared to that of the matrix.

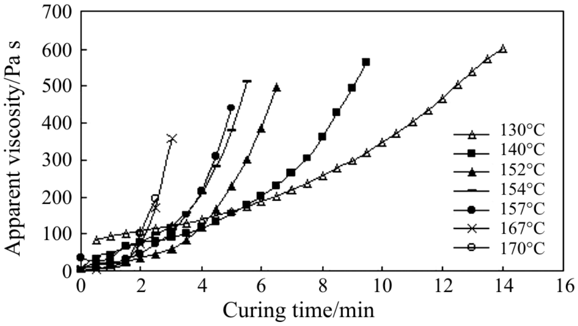


**Fig. 4** Iso-$T_{\text{cure}}$ diagrams of Uralac P2400-Araldit PT 810 matrix determined by rotational viscometry

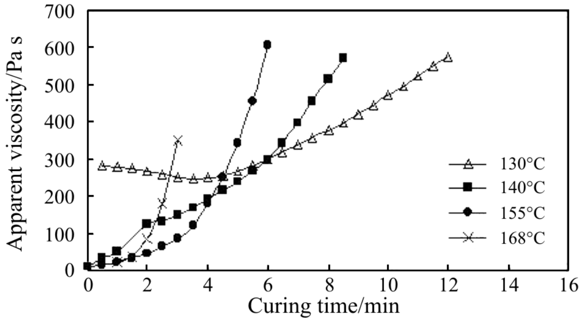


**Fig. 5** Iso-$T_{\text{cure}}$ diagrams of Uralac P2400-Tepic G matrix determined by rotational viscometry

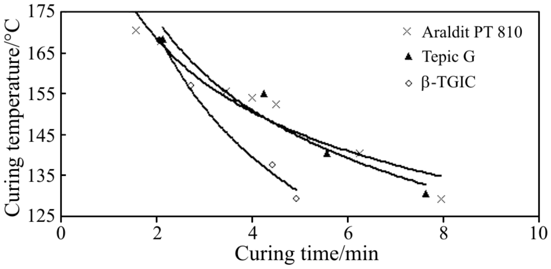


**Fig. 6** Gelation curve of the matrices prepared of Uralac P2400 with Araldit PT 810, Tepic G and β-TGIC

The gelation curves of the powder coatings prepared with Uralac P2400-Tepic G and Uralac P2400-high (61%) β-TGIC are compared in Fig. 10.

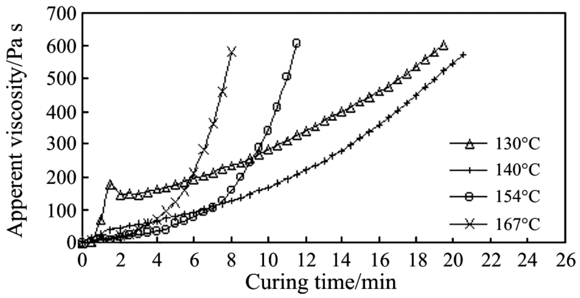


**Fig. 7** Iso-$T_{\text{cure}}$ diagrams of powder coating prepared with Uralac P2400-Tepic G matrix determined by rotational viscometry

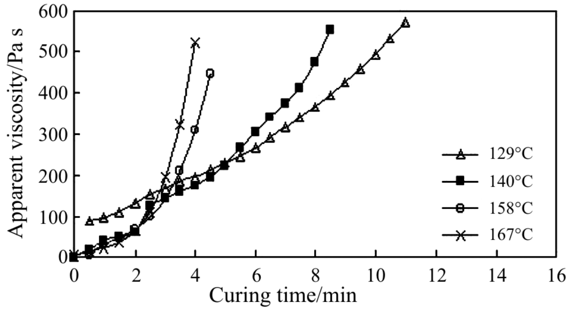


**Fig. 8** Iso-$T_{\text{cure}}$ diagrams of powder coating prepared with Uralac P2400-high (61%) β-TGIC matrix determined

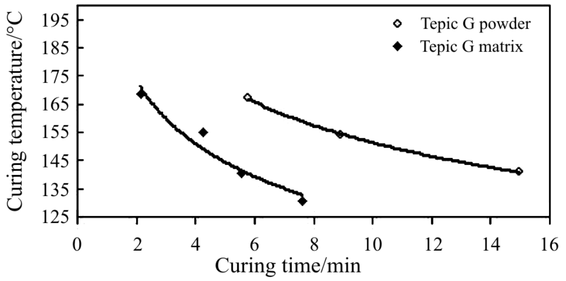


**Fig. 9** Gelation curve of the matrix resin Uralac P2400-Tepic G, and of the powder coating prepared of the same matrix

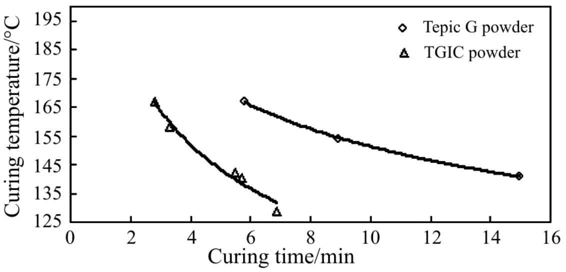


**Fig. 10** Gelation curve of the powder coating prepared of Uralac P2400-Tepic G, and of the powder coating prepared with Uralac P2400-high (61%) β-TGIC

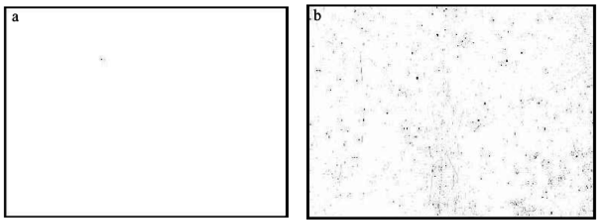


**Fig. 11** Surface of powder coating prepared with a – the matrix Uralac P2400 and Tepic G, b – the matrix Uralac P2400 and high (61%) β-TGIC

The powder containing the mixture of the two diastrereomer racemates with high (61%) β-TGIC content has the reactivity, too high to avoid blistering formation during the cure. This is due to the side reaction of direct esterification via the carboxyl and secondary OH groups accompanied with the elimination of water during the curing process. The high viscosity of the material does not allow the escape of the bubbles from the system. This is represented by the scanned picture of the two coatings, i.e. the one prepared with Tepic G and the other prepared with the isomer mixture with high (61%) β-TGIC content (Figs 11a and b).

## Conclusions

The isothermal curing of a saturated carboxyl functional polyester resin and triglycidyl-isocyanurate of different sources has been monitored in a cone and plate rotational viscometer and the iso-$T_{\text{cure}}$ diagrams have been determined directly from the measurements. The iso-$T_{\text{cure}}$ diagrams served for the determination of the gelation curve of each system by reading the abscissa of the intercept of the straight lines fitted to the beginning and to the final section of each isothermal viscosity curve. The temperature of curing and the time of gelation supply a pair of points of the gelation curve. The measured section of the gelation curves could be fitted by power equations. The conclusion may be drawn that the ratio of the two diastereomer racemates of TGIC has a significant effect on the reactivity of the coating system. β-TGIC is of highest reactivity, and the increase of its ratio in TGIC increases the reactivity and adversely effects the appearance and performance of the powder coating. The reactivity of the matrix is higher than that of the powder. Commercial TGIC-s have similar reactivity, which is comparable to that of high α-TGIC. The reactivity of Tepic G is somewhat higher than that of Araldit PT810 due to its higher β-TGIC content.

## Acknowledgements

Authors thank the Hungarian Scientific Research Fund OTKA T025589, T037988 and DAK3/2000 for financial support, Mária Mara and István Hatházi for their help.

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Received: January 20, 2005
In revised form: March 16, 2005

DOI: 10.1007/s10973-005-6421-5