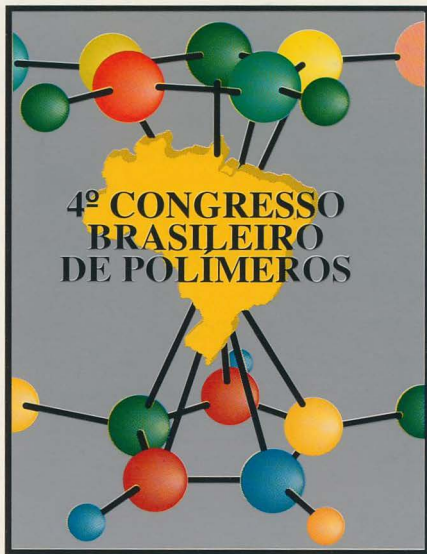


# 4º CONGRESSO BRASILEIRO DE POLÍMEROS

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Promoção:



Associação Brasileira de Polímeros

# NETWORK FORMATION PROCESS OF 1,4-BUTANEDIOL DIGLYCIDYL ETHER WITH *CIS*-1,2-CYCLOHEXANEDICARBOXYLIC ANHYDRIDE BY TEMPERATURE SCANNING BRILLOUIN SCATTERING

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## Introduction

The dynamics of curing process of 1,4-butanediol diglycidyl ether (EP) with *cis*-1,2-cyclohexanedicarboxylic anhydride (CH) and the initiator triethylamine (TEA) has been studied by Temperature Scanning Brillouin Spectroscopy technique (TSBS).<sup>1</sup> In this experiment the frequency shift ( $\omega_B$ ) and width ( $\Gamma_B$ ) of the Brillouin lines are related to the hypersound velocity ( $U$ ), which is a time- and temperature-dependent property. The drastic phase change observed during the network formation enables the choice of the hypersound velocity as an observable for the reaction kinetics. A series of samples with ratios of molar fraction ( $X = X_{CH}/X_{EP}$ ) ranging from 0.50 to 10.08 and constant TEA concentration was investigated. The obtained  $U$  was used as an indicator of the extent of the reaction through a conversion factor ( $\alpha$ ), which was determined by relating the initial and the final hypersound velocities of the cure process.

## Experimental

The EP, CH and TEA were provided by Aldrich Chemical with p.a. purity. The samples were prepared with a ratio  $X = X_{CH}/X_{EP}$  ranging from 0.50 to 10.08. The liquid mixtures of CH e EP were filtered through a 0.22- $\mu\text{m}$  Millipore filter into the light scattering cuvettes in order to remove dust. During the TSBS measurements every sample was submitted to a temperature scanning with a heating rate of 1 °C/min<sup>1</sup>. The temperature ranged from 30 °C to 190 °C. A series of spectra were registered. The backscattered light (approx.  $\theta = 180^\circ$ ) was analyzed in the polarized scattering geometry with a piezoelectrically scanned triple-pass Tandem Fabry-Perot Interferometer<sup>2</sup>. The parameters  $\omega_B$  and  $\Gamma_B$  were computed by a fit of the spectra with a Lorentzian function.

## Results and Discussion

The  $\omega_B$  and  $\Gamma_B$  of the Brillouin lines can be related to the hypersound velocity  $U$  and to the absorption coefficient  $\varepsilon$  as follow:

$$U = \omega_B/K \qquad \varepsilon = 2\pi\Gamma_B/U,$$

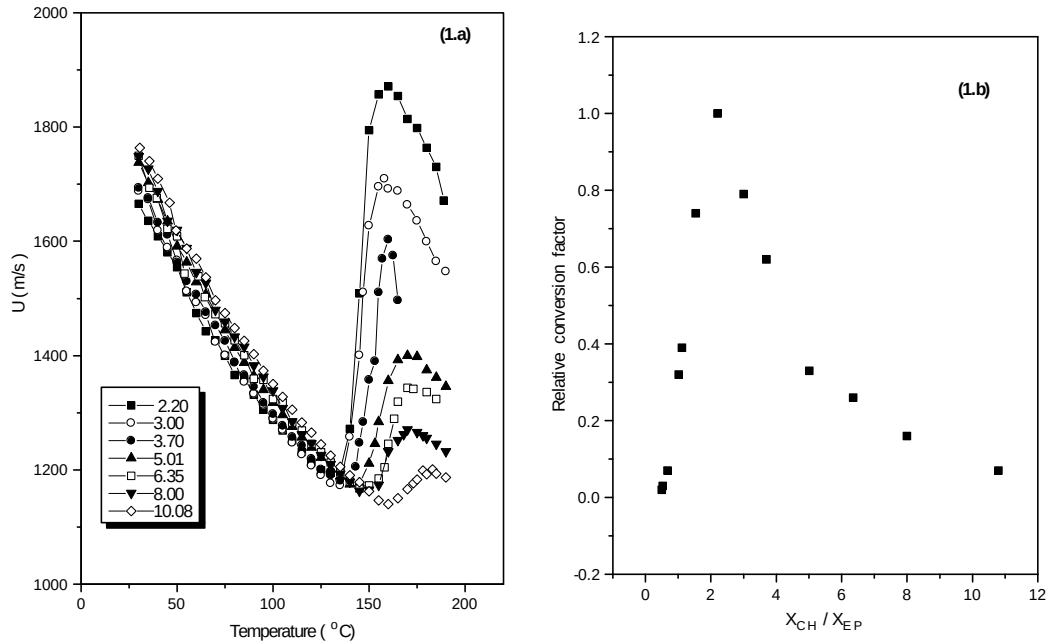
were  $K$  is defined as the scattering wave vector<sup>1</sup>. The determined  $U$  values for the samples with  $X$  between 2.20 and 10.08 are shown in Figure 1.a. A drastic decrease of the slope  $dU/dT$  can be observed with increasing  $X$ . The temperature dependence of the initial and final  $U$  in this series is affected by the composition of the initial and final system. The extent of the cure process was obtained in an active temperature range between the minimum and the maximum experimental values of  $U$  ( $U_{min}$  and  $U_{max}$ ).

The relative conversion factor  $\alpha_{rel}$  for all the samples compositions ( $X$ ) could be determined taking in consideration the  $U_{min}$  and  $U_{max}$  for the hypersonic velocity of the measured system and  $U_{min}$  and  $U_{max}$  of the standard sample ( $X = 2.20$ ):

$$\alpha = \frac{U_{max(X)} - U_{min(X)}}{\dots}$$

$$U_{max(2.20)} - U_{min(2.20)}$$

The values of  $\alpha_{rel}$  were plotted vs. the molar fraction ratio  $X$  in Figure 1.b.



**Figure 1. a)** Hypersonic velocity curves vs. temperature for different molar fraction ratio ( $X = X_{CH}/X_{EP}$ )  
**b)** Final relative conversion factors  $\alpha_{rel}$  vs. molar fraction ratio ( $X = X_{CH}/X_{EP}$ ).

The data and the behavior obtained by the recently introduced TSBS technique<sup>1</sup> suggest that this new experimental technique demonstrates a high sensitivity and capability for the study of curing and generally phase change including gelification and vitrification processes.

## References

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2. D. Fioretto, L. Palmieri, G. Socino, L. Verdini, *Phys. Rev. B*, 1994, 50 (1), 605.

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