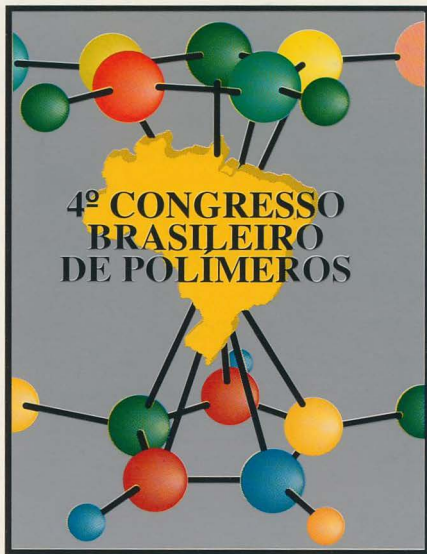# 4º CONGRESSO BRASILEIRO DE POLÍMEROS

Salvador, 28 de setembro a 2 de outubro de 1997

Promoção:

Associação Brasileira de Polímeros

CHARACTERIZATION OF EPOXIDE RESIN/HYDROXYPROPYL CELLULOSE NETWORKS BY DSC

Maria Inez G. de Miranda, Clara I. D. Bica and Dimitrios Samios

Instituto de Química/UFRGS- Cx. Postal 15003
91501-970 Porto Alegre/RS –Brasil   e-mail: IGRAF@if.ufrgs.br

Introduction

With the purpose to obtain epoxide systems with a liquid crystalline component, we have studied semi-interpenetrating networks which result from the curing of the epoxide resin in the presence of hydroxypropyl cellulose[1] which is known to displays liquid crystal behaviour[2]. The techniques employed were differential scanning calorimetry (d.s.c.) and dynamic mechanical thermal analysis (d.m.t.a). In the present study we investigated the effect of different heating rates in thermal transitions of epoxy systems with varius contents of hpc by applying d.s.c. The properties of thermoset networks depend upon the conditions under which the cure reactions occur and also to relaxation processes thermally activated inside the polymeric matrix[3-7]. The differences can be related to the method of the solvent removal employed[7] and when another component is present (polymer, inorganic filler[4]) as hydroxypropyl cellulose[2].

Experimental

The samples studied were prepared from solvent casting of diglycidyl ether of bisphenol A (DGEBA)/4,4'-diaminediphenylmethane(DDM) and hydroxypropyl cellulose (hpc). The epoxy resin DGEBA (Ciba-Geigy) had an epoxy equivalent of 190 g.eq^{-1} with 82-92% of monomeric material (data furnished by the supplier). DDM (Ciba-Geigy) had a amine equivalent of 90 g.eq^{-1}. The hydroxypropyl cellulose used was manufactured by Aldrich with nominal molecular weight of 100,000 gmol^{-1}. Stoichiometric mixtures of DGEBA/DDM (epoxy system) and hpc were prepared separately until complete dissolution in chloroform. The hpc solution (25 g.l^{-1}) was added in epoxy system solution (250 g.l^{-1}) according to the required compositions. The final solution of DGEBA/DDM and hpc was prepared through mixing of the original solutions. All solutions were carried out under constant stirring and at room temperature during four hours. The films were left for 7 days at ambient conditions to allow for the evaporation of excess solvent before vacuum was applied (10 hours) and afterwards were cured for 60 minutes at 120°C. The glass transition temperature (Tg) and the cholesteric-isotropic transition of the samples were monitored using a Perkin-Elmer DSC-4. The samples (10 mg) were preheated from -40 to 230°C at a heating rate of 10, 20 and 30 °C/min and quenched to -40 °C. The second scan was performed at the same conditions and registered. The Tg data were determined as the midpoint of the transition range. T_{LC} values are the peak temperatures of the cholesteric-isotropic transition.

Results and Discussion

In Figure 1 the Tg^{epoxy}, the hpc glass transition (Tg^{hpc}) and the cholesteric-isotropic transition (T_{LC}) of the epoxy/hpc blends, at different heating rates, are showed. The epoxy/hpc blends show two transitions from 10 to 50 %wt.hpc. At concentrations lower

than 50 %wt.hpc until 10 %wt.hpc the Tg^{epoxy} and the cholesteric-isotropic transition (T_{LC}) are assigned. At concentrations higher than 50 %wt.hpc the T_{LC} and Tg^{hpc} transitions can be detected. Concerning the peak of the cholesteric-isotropic transition of hpc no significant changes can be observed but one can notice an increase of the epoxy glass transition temperature (T_g^{epoxy}) from 0 to 10 %wt.hpc with an increasing of the heating rates. The shift of the epoxy-Tg to higher temperatures can be explained as an ordering of the epoxy matrix due to the presence of the cholesteric hydroxypropyl cellulose. On the other hand the interactive forces between the epoxy system and hpc can also be assigned to Hydrogen bonding due to the presence of hydroxyl groups of hpc. It is known hydroxyl groups helps in the opening of the epoxy ring, through hydrogen forces, during the state of transition of the curing reaction[7]. The hpc glass transition (Tg^{hpc}) can be detected at concentrations higher than 50 %wt.hpc for higher heating rates (30 and 40 °C/min) and does not seem to show any composition dependence.
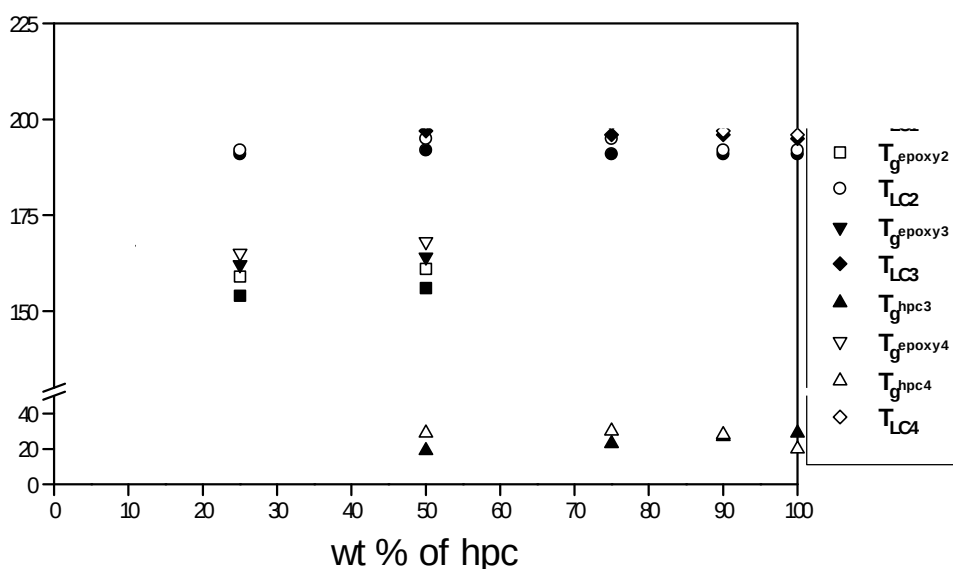

Figure 1. Behaviour of Tg, cholesteric-isotropic transition (T_{LC}) and Tg^{hpc} of epoxy/hpc blends at different concentrations of HPC and heating rates **1**(10 °Cmin^{-1}), **2**(20 °Cmin^{-1}), **3**(30 °Cmin^{-1}) and **4**(40 °Cmin^{-1}).

References

1. M.I.G. Miranda, C.I.D. Bica, l. l. Freitas, D. Samios, 1997, *Polychar-5,*Anais P-16*, Denton-Texas.*
2. T. Rials, W. Glasser, *J. Applied Polym. Sci*., 1988, 36, 749.
3. M. I. G. Miranda and D. Samios, *Eur. Polym*. J., 1997, 33:3, 325.
4. M. I. G. Miranda, C. Tomedi, C. I. D. Bica and D. Samios, *Polymer,* 1997, 38, 1017.
5. M.I.G. Miranda, C.I.D. Bica, D. Samios, *Polymer,* 1997, in press.
6. S. Tarifa, A. Bouazizi, *J. Thermal Anal.,* 1997, 48, 297.
7. Mondragon et all., *J. Applied Polym. Sci*., 1995, 55,75.

Acknowledgements