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THERMOSETTING CHARACTERISTICS OF SOME NEW POLYMETHYLOL EPOXY RESINS

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ABSTRACT

Several new polymethylol epoxy resins have been synthesised in a one pot reaction system and characterised. The new resins are derived from epichlorohydrine, formaldehyde and several aromatic phenols: phenol, biphenylol, bisphenol-A, bisphenol-S, sulphone, and DDT. The rate of crosslinking has been studied at different temperatures (120–180°C) by determining the amount of H_2O evolved from the crosslinking reaction using a DuPont moisture evolution analyser; the activation energy of setting has been determined from Arrhenius plots. The results are 57.1, 50.0, 42.9, 75.2, 11.3 and 74.3 kJ mol^{-1} for resins **I–VI** respectively. The set resins show good thermal stability and are resistant toward chemicals and solvents; these resins also set catalytically at room temperature.

INTRODUCTION

During an earlier research program we synthesised [1,2] and characterised several new polymethylol epoxy resins. These intermediate resins were transferred to several esters and their efficiency as plasticisers was investigated for several polymers (PVC, polystyrene and polymethylmethacrylate) [3–8], promising results being obtained. In this paper the thermosetting characteristics of these resins and other new resins are studied.

EXPERIMENTAL

Materials

Several polymethylol epoxy resins were synthesised in a one pot reaction system and characterised by spectroscopy, elemental analysis, molecular weight determination and colorimetry [9–11]. The resins prepared and

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studied were: **I**, 1,3-bis(2,4,6-trimethylolphenoxy)propan-2-ol; **II**, 1,3-bis(2,6-dimethylol-4-phenylphenoxy)propan-2-ol; **III**, 1,3 bis[4-(3,5-dimethylol-4-hydroxyphenylisopropylidene)2,6-dimethylolphenoxy] propan-2-ol; **IV**, 1,3-bis [4-(3,5-dimethylol-4-hydroxyphenylsulphide)2,6-dimethylolphenoxy]propan-2-ol; **V**, 1,3-bis[4-(3,5-dimethylol-4-hydroxyphenylsulphone)2,6-dimethylolphenoxy]propan-2-ol; **VI**, 1,3-bis[4-(3,5-dimethylol-4-hydroxyphenyltrichloroethylidene)2,6-dimethylolphenoxy]propan-2-ol.

Instruments

A Du Pont moisture evolution analyser, model 903 H, was used in this study after being calibrated with standard materials.

Thermosetting procedure

Several samples from each resin (10–15 mg) were heated at different temperatures (120–180°C). The amount of evolved moisture was determined by the conductivity cell as a function of time. The total amount of H_2O evolved from the crosslinked resin was also determined at the end of each experiment for each resin.

RESULTS AND DISCUSSION

The rate of setting (crosslinking) of the resins was calculated from percentage H_2O loss as a function of time at each temperature; typical results are shown in Figs. 1 and 2 for the resins **II**, **IV**, **V** and **VI**.

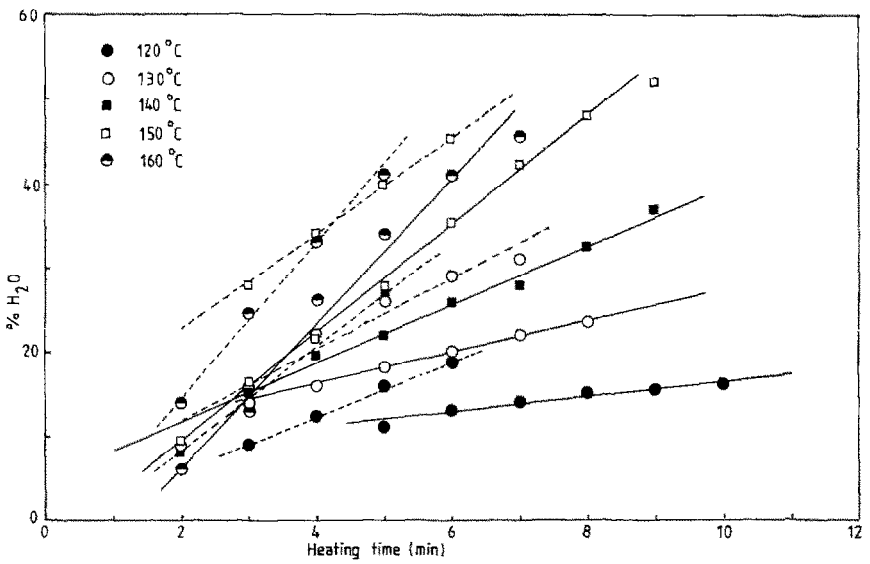


Fig. 1. The effect of temperature on the rate of setting of resin **II** (— — —), **VI** (———).

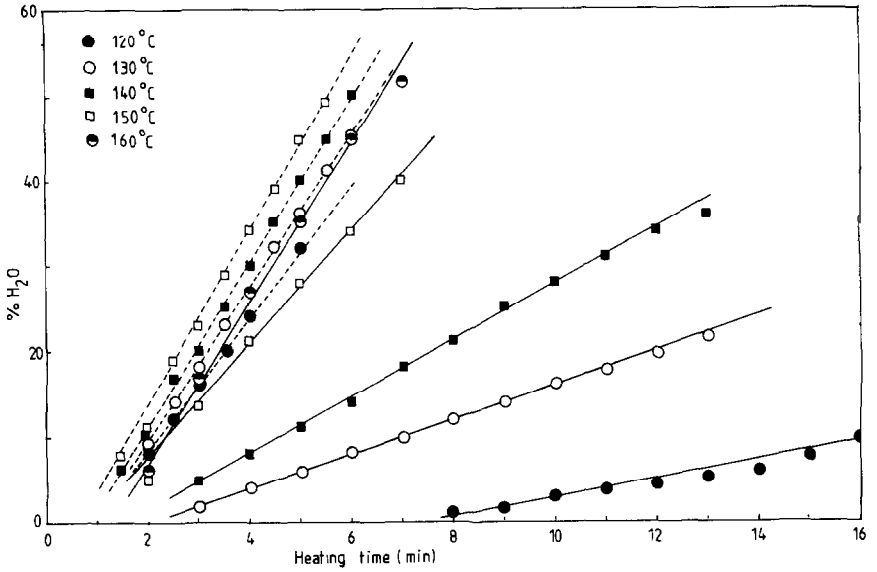


Fig. 2. The effect of temperature on the rate of setting of resin IV (———), V (— — —).

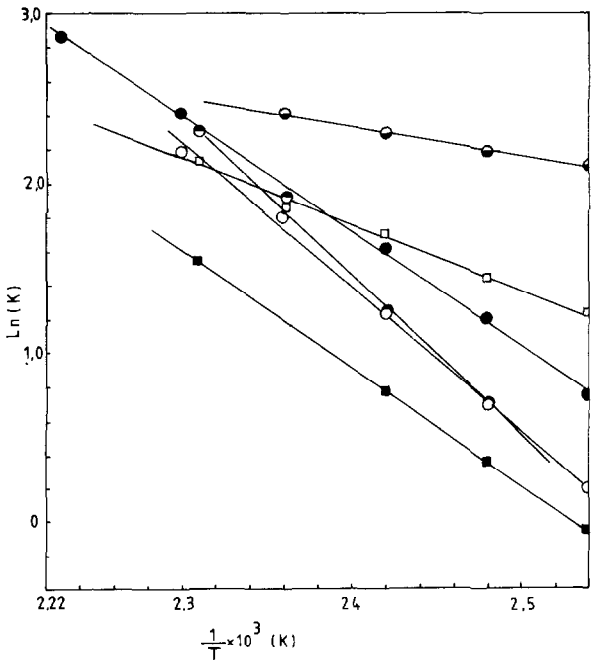
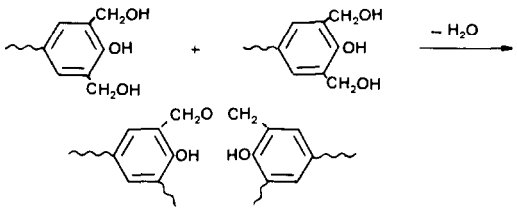


Fig. 3. Arrhenius plot showing the relationship between rate of setting and temperature (K) for resin I (■), II (□), III (●), IV (◓), V (◒), VI (○).



Scheme 1

The rate of crosslinking for each resin was determined from the slopes of these individual curves. The rate of setting was plotted against temperature (K) as shown in Fig. 3. From the slopes of these curves the energy of activation for the setting process was found for each resin and the results obtained were: 57.1, 50.0, 42.9, 75.2, 11.3 and 73.3 kJ mol^{-1} for resins **I–VI** respectively.

The crosslinking reactions take place according to Scheme 1 but as the temperature increases above 180°C another type of crosslinking reaction takes place. This is conversion of the ether interlinkage group to methylene by abstraction of a formaldehyde molecule

$$\sim\!\sim CH_2OCH_2 \sim\!\sim \rightarrow \sim\!\sim CH_2 \sim\!\sim + HCHO$$

The set resins were thermally stable as found from thermogravimetric studies. A typical thermogram is shown in Fig. 4 for resin **III** thermally set at 200°C.

The set resins have attractive golden shiny surfaces, high strength, and are resistant toward solvents and chemicals. The resins also set catalytically at room temperature. These resins might have promising applications as adhesives and laminating materials.

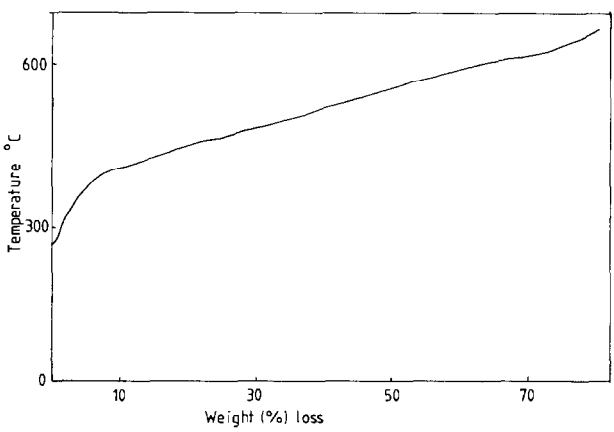


Fig. 4. Typical thermogravimetric curve showing the thermal stability of resin **III** thermally set at 200°C for 1 h.

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