PRELIMINARY EVALUATION OF VAPOUR ADSORPTION ON CELLULOSE ETHERS

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The determination of the adsorption of organic vapours by inverse gas chromatography (IGC) has permitted the derivation of polymer surface energies (Anhang & Gray, 1982). To date the interfacial behaviour of cellulose ethers commonly employed in the film coating of tablets has been examined by contact angle goniometry rather than vapour adsorption techniques (Davies, 1985). As part of an IGC investigation into the effect of chemical substitution on the surface energies of cellulose ethers, this study reports on the preliminary evaluation of the sensitivity of IGC to changes in surface adsorption phenomena in the region of the glass transition temperature, Tg for this range of polymers.

The IGC experiments were undertaken with a Hewlett Packard 5890 gas chromatograph equipped with a flame ionisation detector. The carrier gas, nitrogen, was passed through conventional gas chromatography columns (length - 6' : internal diameter - 2mm) individually packed with the commercial grade of polymer, at a flow rate of approximately 20 ml min^{-1}. 0.1 µl samples of the probe (hexane, octane or dodecane) were injected onto the column and the retention times recorded by a Hewlett Packard 3392A integrator. Adsorption readings were taken in one degree increments, 15 degrees either side of the detected Tg value.

Table 1 illustrates the excellent correlation between the Tg values derived by IGC and differential scanning calorimetry (DSC) for four cellulose ethers commonly employed in pharmaceutical formulations. The value of Tg for HPMC and EC also show good agreement with previous DSC studies (Entwistle and Rowe, 1979). Transitions were also observed for HPC at temperatures below the final Tg value in agreement with a previous study by Aspler and Gray (1982). It is of particular interest to this study, however, that IGC displayed a higher sensitivity to the onset of the transition from the glassy to the amorphous state than DSC particularly where the polymer samples were outgased prior to analysis, to remove surface moisture and appreciable amounts of residual volatile impurities from the polymer bulk.

Table 1 : Glass Transitions of Cellulose Ethers

POLYMER	Tg by IGC (±0.5° C)	Tg by DSC (±1.0° C)
Hydroxypropylmethylcellulose (HPMC)	177	177
Hydroxyethylcellulose (HEC)	132	133
Ethylcellulose (EC)	129	129
Hydroxypropylcellulose (HPC)	123	124

This initial investigation has confirmed that IGC is a highly sensitive technique for the study of the nature of the interfacial adsorption of organic vapours for this range of cellulose ethers, and has potential for the elucidation of relationships between chemical substitution and surface free energies for this group of polymers.

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