

MICROCALORIMETRIC STUDIES OF WATER VAPOUR SORPTION IN MICROCRYSTALLINE AND MICROFINE CELLULOSE POWDERS

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Whilst water in its various forms plays critical roles in pharmaceutical formulation, processing and product performance, the association, structure and form of water in polymeric materials lacks fundamental definition. Recent work (Zograffi et al 1984) using microcrystalline cellulose (MCC) and water sorption isotherms has highlighted this feature and suggested that water vapour might be taken up by a mechanism similar to that proposed for starch (Van den Berg, 1981). This study examines the effects of powder pretreatment on the mechanism of water vapour sorption for two high grade cellulose powders using a microcalorimetric technique.

Enthalpy of water vapour sorption (ΔH_a) was obtained (LKB Sorption Microcalorimeter 2107) at 20°C using a flow cell using a grade of spray dried MCC (Avicel pH 101) and a microfine cellulose (MFC) (Elcema P050) which is milled to achieve a size specification. Humid air (R.H. range 10-70%) was passed at a controlled rate over previously dried powder samples. Fig. 1. shows graphs of differential ΔH_a versus powder moisture content (derived from previously determined water vapour sorption isotherms at 20°C).

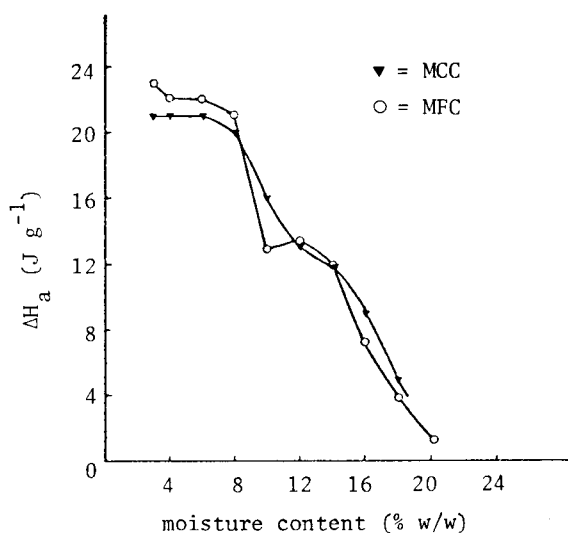


Fig. 1. Differential ΔH_a versus moisture content for MCC and MFC.

Three sections can be distinguished in both graphs. For MCC an initial level ΔH_a value occurs from 3-8% w/w moisture content indicating a homogeneously sorbed water. A second plateau less well defined at lower ΔH_a is observed between 10-14% w/w and above this moisture level, the continuous decrease in ΔH_a suggests the sorbed water gradually resembles bulk water. The breaks in the curve correspond to calculations of stoichiometric ratios of 1:1 and approximately 2:1 of water to repeating anhydroglucose units, assuming a crystallinity figure of 63% (Nakai et al, 1977), the fact that water is taken up by the amorphous regions of cellulose (Stamm, 1964) and a calculated water vapour monolayer capacity of 0.0346 gH₂O/g MCC (from BET analysis of water vapour sorption isotherm). The mechanism of sorption appears to initially involve an interaction between a single water vapour molecule with each repeating sugar unit followed by a second, weaker and possibly less homogeneous associated water molecule with each repeating unit, subsequently followed by less well defined multilayers of water molecules. This follows the pattern reported for starch (Van den Berg, 1981). Interestingly, for MFC the value of ΔH_a for the initial plateau is higher, and the second plateau region is sharper than for MCC. These phenomena can be attributed to differences in powder pretreatment, inducing, for example, different degrees of crystallinity.

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