Journal of Thermal Analysis, Vol. 45 (1995) 573-576

A NOVEL DIFFERENTIAL SCANNING CALORIMETRIC METHOD TO STUDY THE COMPLEXATION OF β-CYCLODEXTRIN WITH STEROID IN SOLID STATE

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(Received February 11, 1994; in revised form September 3, 1994)

Abstract

This communication demonstrates that, by simply monitoring the evaporation of water molecules included in β-cyclodextrin cavity, complexation of β-cyclodextrin with steriod in solid state can be studied. The reported method is simple, rapid and novel.

Keywords: β-cyclodextrin, complexes, DSC

Introduction

Cyclodextrins (*CDs*), the oligomers of glucose have been subjected to extensive studies. The reason for this enormous efforts to investigate *CDs* can be traced to their remarkable ability to form inclusion complexes with a variety of molecules ranged from organic or inorganic compounds of neutral, ionic nature to noble gases [1–4].

Normally the characteristic properties of the included components vary as a result of complexation [5, 6]. Complexation is usually monitored by studying the variation in properties of the guest molecules.

It is well known that water molecules from the *CD* cavity get released as a result of complexation of the guest [7]. It is quite reasonable to presume that water molecules inside the cavity evaporate at a higher temperature than the free water molecules. In that sense scanning calorimetry would give a precise insight into the state of water in *CD* and *CD*–guest complex. To the best of our knowledge, this simple approach of monitoring the nature of water in *CD* as a route to understand the complexation has not been attempted. This note addresses our effort in this direction.

0368–4466/95/ $ 4.00

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Experimental

β-Cyclodextrin (β*CD*) and cholesterol obtained from Sigma Chemicals, USA, were used as received.

β*CD* and cholesterol in 1:1 ratio were mixed thoroughly in the form of a paste by adding 2–3 drops of water. This mixture was kept under a dynamic atmosphere of nitrogen for about 30 min to get a dry powder. β*CD* was also kept similarly under nitrogen.

A DuPont 990 thermal analyser system in conjuction with a 910 DSC cell was used for obtaining the curves. About 6–10 mg of the components were non-hermetically sealed in aluminium pans and heated from 30 to 200°C at a heating rate of 10 deg·min^{-1} under a dynamic atmosphere of nitrogen.

Results and discussion

Figure 1A shows the DSC scan of β*CD*. A peak centered around 130°C which dissappears in the second run (Fig. 1B) of the same sample after cooling to the room temperature, can be assigned to the evaporation of water molecules

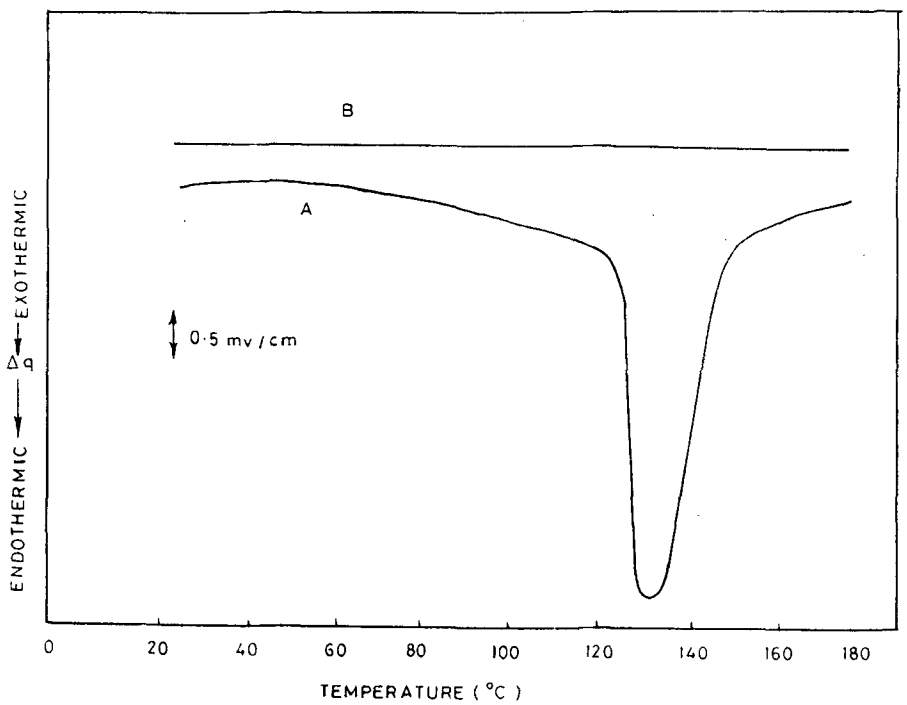

Fig. 1 DSC curve of β*CD* (A), DSC curve of same material after cooling to room temperature (B)

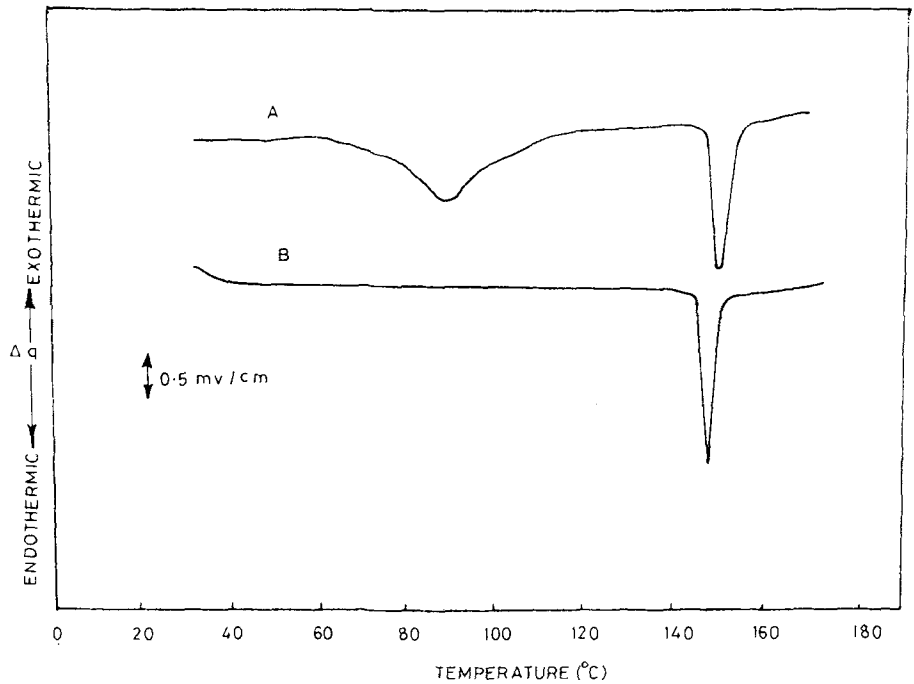

Fig. 2 DSC curve of β*CD*–cholesterol mixture (A), DSC curve of cholesterol (B)

from the cavity of β*CD*. The increased temperature could be due to the shielding of the water molecules by β*CD* cavity.

Figure 2A depicts the curve of β*CD*–cholesterol system. It is interesting to see that water in this case evaporates at about 95°C. The evaporation temperature would have been 130°C if these water molecules were inside the β*CD* cavity. Moreover the intensity of the peak is reduced. This is indeed expected in the sense that, free water molecules evaporate at a faster rate when subjected to drying under nitrogen. The absence of any peak around 130°C shows that the cavity of β*CD* does not contain any water molecules. The peak at 151°C can be assigned to the melting of cholesterol. As per the curve shown in Fig. 2B, the melting point of cholesterol is 149°C. A slight increase (about 2°C) in melting point of cholesterol can be due the action of β*CD* cavity as a protective thermal shield. The thermal behaviour apparently indicates that cholesterol replaces water molecules to form the inclusion complex with β*CD*.

The ability of β*CD* to form inclusion complexes with steroids is well known [8]. The technique discussed here is a novel, simple and rapid method providing direct insight into the complex formation in solid state.

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Zusammenfassung — Vorliegend wird dargelegt, daß mittels eines einfachen Verfolgens der Verdampfung von Wassermolekülen aus ß-Cyclodextrin-Hohlräumen die Komplexbildung von ß-Cyclodextrin mit Steroiden im festen Zustand untersucht werden kann. Die beschriebene Methode zeichnet sich durch Schnelligkeit, Einfachheit und Neuartigkeit aus.