DEHYDRATION KINETICS OF HYDOUS SUGARS TO ANHYDROUS SUGARS IN ETHANOL BY DSC

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1. Introduction
The formation of the solid crystalline phase of sugars plays an important role in many food products. In order to control the crystal structure of sugar, it is necessary to remove the water of crystallization in the sugar. Recently, a new dehydration method using ethanol as a solvent was investigated by Ohashi et al. (2007), and new porous crystals were obtained. Presently in the literature, little is known about the use of ethanol in dehydration of sugar crystals, particularly the kinetic aspects and the consequences on the crystals size and shape. In the present work, the dehydration of sugar in ethanol was performed by differential scanning calorimetry (DSC) in high pressure crucible pans filled with ethanol. In this study, several sugars, i.e. trehalose, glucose, and lactose were used in their hydrous forms. The dehydration reactions were carried out in the DSC pan in isothermal condition and in the increasing temperature method at constant rate (Kissinger’s method). The influence of water content in ethanol on the reaction kinetics and the physical aspects of the crystals were investigated.

2. Materials and Methods
Dihydrate Trehalose (Moisture content MC=9.50%), α-Monohydrate Lactose (MC=5.00%) and D-(+)-Glucose Monohydrate (MC=9.28%) were purchased. Three ethanol-water mixtures (MC=0.4%, 2.5% and 4%) were prepared by adding distilled water to a high purity ethanol. The dehydration kinetic analysis was carried out with differential scanning calorimetry (DSC) under isothermal condition and the increasing temperature method at constant rate (Kissinger’s method). Sugar crystal of 2.0±0.1 mg in hydrous form was packed in high pressure crucible pans with 10±0.1 µl of an ethanol-water solution. In the isothermal condition, the pan was heated quickly to a prescribed temperature Tc, until the dehydration reaction completes. The endothermic heat flow vs. time was recorded with Muse Measurement software. For Kissinger’s method, the pan was heated from 30 to 140°C at several heating rates. The DSC curve of the endothermic heat flow vs. temperature was measured. For both methods, nitrogen was used as a purge gas at 30 ml/min. Avrami equations were used to determine the kinetic constant “k” in isothermal conditions, and Arrhenius equation was used afterwards to evaluate the activation energies. For non-isothermal conditions, Kissinger’s method was applied (Kissinger, 1957). Scanning electron microscopy was used to investigate the microstructural properties of the sugar crystals.

3. Result and Discussion
The constant temperature method and Kissinger’s method gives an overall evaluation of the apparent activation energy of dehydration and presumably reveal two different dehydration reactions in the glassy and rubbery state. Ethanol with 2.5%MC shows the fastest reaction speed and the lowest activation energy for the three studied sugars. Ethanol with 4%MC shows the biggest crystal after reaction for trehalose (Figure 1) and lactose. This dehydration method with ethanol by DSC could be applied to evaluate the kinetic parameters of dehydration of other crystalline drug compounds.

Fig. 1 Effect of water content of ethanol on the morphology of anhydrous sugar crystals. (a) trehalose dihydrate, (b) anhydrous trehalose with ethanol containing 0.4% MC, (c) 2.5% MC and (d) 4% MC. (e) anhydrous glucose with ethanol containing 2.5% MC. (f)anhydrous lactose with ethanol containing 2.5% MC.

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