ESRF

ESRF	**Experiment title:** Time-resolved SAXS/WAXS studies of the influence of Temperature and plasticizers on the crystallisation kinetics of sucrose	**Experiment number**: SC-1390
Beamline: ID02	**Date of experiment**: From: 12/6/2004 to: 15/6/2004	**Date of report**: 26/2/2005
Shifts: 9	**Local contact(s)**: Dr. T Narayanan	*Received at ESRF:*

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Report:

This report describes the application of time-resolved x-ray diffraction techniques to investigate the influence of an anti-plasticizer (polyvinylpyrrolidone (PVP)) on the crystallization kinetics of sucrose at several temperatures at beamline ID02. The sucrose samples were loaded in an aluminium differential scanning calorimeter (DSC) pan which was injected into a pre-heated x-ray diffraction sample stage using a stepper-motor controlled arm. During data collection, the sample was oscillated in the beam to minimize the effect of the preferred orientation of the crystallites formed during the crystallization process. The time-resolved SAXS/WAXS data

were recorded during the in-situ crystallisation of sucrose at several temperatures above glass transition temperature (T_g). ESRF CCD detectors, with a typical exposure time of 0.01 seconds per frame and a variable gap between the frames, were used (depending on the rate of crystallisation). WAXS data were analysed to determine the crystallisation kinetics by using an algorithm developed to extract the crystallinity data from the diffraction pattern. Figure 1 shows the crystallisation kinetics of pure sucrose at temperatures ranging from 130°C to 160°C. It can be seen from Figure 1 that the induction time for crystallisation is not very sensitive at temperatures less than 150°C. Also it is important to note that the degree of sigmoidality of crystallisation kinetics increases with decreasing temperature. Figure 2 shows the crystallisation kinetics of sucrose colyophilized (freeze-dried) with 1% PVP. It clearly shows that the addition of 1% PVP increases the induction time and the degree of sigmoidality. PVP reduces temperature sensitivity of the induction time at the higher temperatures. Figure 3 shows that the addition of 1% PVP is equivalent to an increase of ~20°C in the crystallisation temperature of sucrose.

Figure 1

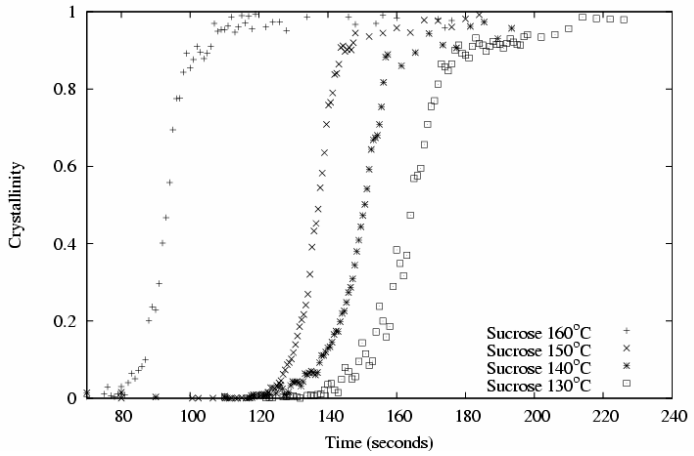

Figure 2

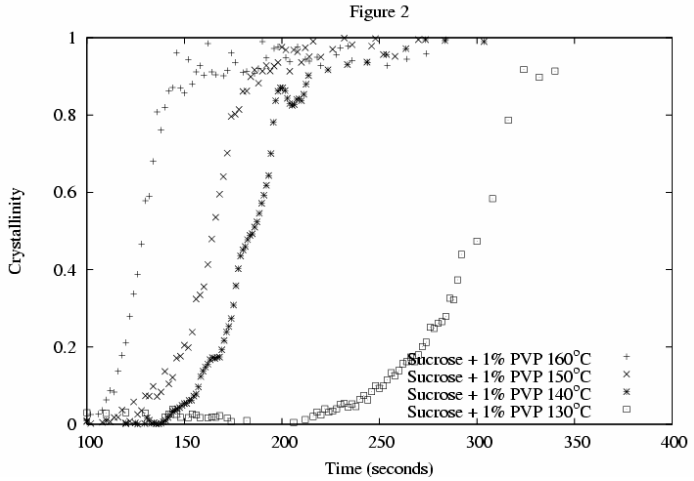

Figure 3