ESRF

ESRF	**Experiment title:** Time-resolved SAXS/WAXS mercerization studies of cellulose using the flow	**Experiment number**: SC-1667
Beamline: ID02	**Date of experiment**: From: 5/3/2005 to: 8/3/2005	**Date of report**: 13/2/2006
Shifts: 9	**Local contact(s)**: Dr. T Narayanan	*Received at ESRF:*

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Report: The purpose of this investigation was to exploit the high-brilliance of the ID02 beam-line and the development in time-resolved techniques to follow the molecular reconfiguration during the mercerization of native cellulose. A longstanding issue of high fundamental and applied importance has been the relationship between cellulose I and cellulose II structures and in particular the transformation of cellulose I into cellulose II during the industrial processing. As can be seen from the data recorded under this proposal reported bellow we have been able follow this structural transformation in real time and demonstrated the existence of an amorphous intermediary. The identification of this pathway is of crucial importance since by further time-resolved experiments it offers the possibility of refining the mercerization conditions to optimise cellulose II production whilst minimising damaging environmental impact.

WAXS data was recorded during in-situ mercerization experiments on beam line ID02 using the Keele CCD detectors with 40 milliseconds exposure time. Native cellulose pulp supplied by Innovia Films PLC and Whatman cellulose I filter paper were used in the in-situ mercerization experiments where the concentration of NaOH was varied from 5% to 30%. A typical plot of the relative content of each crystalline phase within a sample over a 60 second experiment is shown in figure 1. It can be seen from this figure that during the mercerization process, there is in increase in the amorphous (*) component prior to the formation crystalline cellulose II (+). These results clearly show that during the mercerization, initially cellulose I is converted from crystalline to amorphous phase before re-crystallises in to cellulose II. We have also observed that cellulose IV polymorph is formed instead of cellulose II from amorphous cellulose depending on the temperature and concentration of the NaOH used in the mercerization. In addition to polymorphic changes during the mercerization, analysis of SAXS revealed that during the mercerisation the size and orientation of the pores also changes.

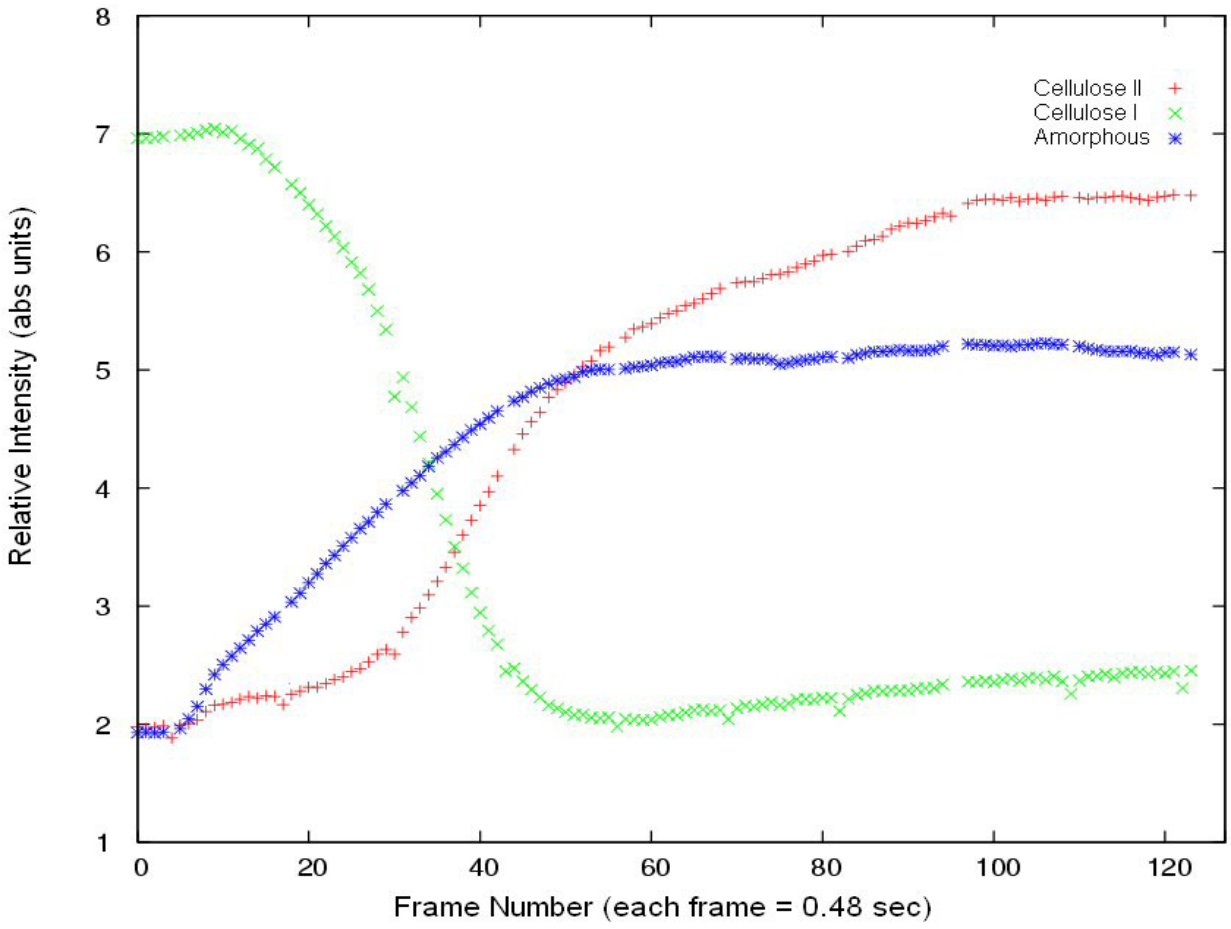

Figure 1: Relative amount of Cellulose I, Cellulose II and Amorphous Cellulose in a sample which was mercerized at room temperature with 20% NaOH.