In situ X-ray study of the dissolution of cellulose with ionic liquid under mild conditions.

Dr Florian Meneau
http://lattes.cnpq.br/5349860579246534

CNPEM,
LNLS, Laboratório Nacional de Luz Sincrotron

Out of the most important challenges to the chemical industry nowadays is the effective use of cellulose for the production of fibers, paper, cosmetic, innovative polymeric materials, chemicals and biofuels. Indeed, cellulose, the most abundant bio-renewable material, consists of polydisperse linear glucose polymer chains which form hydrogen-bonded supramolecular structures (Figure 1). Due to its structure, a number of mechanisms protects the cellulose against chemical and biological transformation, cellulose is insoluble in water and in most common organic liquids. In order to be transformed, it requires to be first fractionated/dissolved, using highly toxic solvents. However, it has been demonstrated that imidazolium-based ionic liquids (ILs) can be used as green solvents for cellulose[1]. Ionic liquids are a class of environmentally friendly compounds, composed of ions only and with melting point below 100°C.
Moreover, we know that not only the crystallinity of cellulose but also its size and surface texture are influencing the final cellulose conversion and then the products selectivity.

In this context, the project will consist in a systematic study of the influence of the dissolution conditions (ionic liquid, temperature, reaction times) on the crystalline structure of cellulose.
The influence of the treatment conditions on the cellulose crystalline structure will be studied by *in situ* Small and Wide Angle X-ray Scattering (SAXS/WAXS) and Scanning Electron Microscopy (SEM).
We will use ionic liquids of the imidazolium family as solvent to treat the microcrystalline cellulose at temperatures below 200°C and for different reaction times. The effects of the ionic liquid chain length, the nature of its anion and the reaction temperature and time will be studied.
Combined *in situ* SAXS/WAXS experiments, which will be carried out at the LNLS facility, will enable to determine the degree of crystallinity and the eventual coexistence of the two cellulose crystal structures, and on a larger length scale, to gain information about the size, surface roughness of the microcrystals. *Ex situ* SEM experiments, which will be carried out at the LN-NANO, will complement the X-ray studies (Figure 2).
Regenerated cellulose, precipitated from the ionic liquid with an adequate anti-solvent, will be studied by *ex situ* SAXS/WAXS and SEM techniques.

The results obtained on the model cellulose will then be compared to biomass samples. The correlation between the degree and type of crystallinity, shape and morphology of the cellulose and their influence on the final cellulose conversion will be studied in a second step.

[1] RP Swatloski et al, Journal of the American Chemical Society, 2002, 124, 4974.

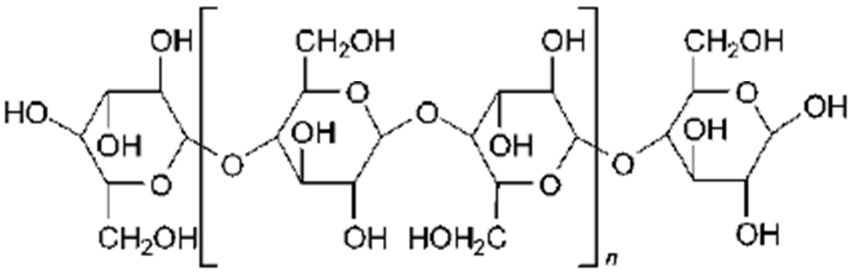

Figure 1: Cellulose polymer chain. (from ref 1)

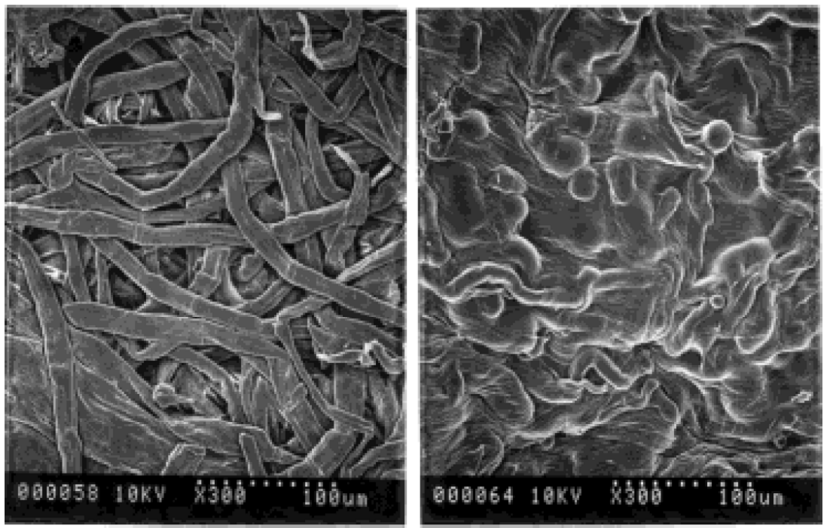

Figure 2: Scanning electron micrographs of initial cellulose (left) and regenerated one after dissolution.in ionic liquid (right), the morphology and texture of the material has evolved into a more compact (from ref 1)