

REEXAMINING STRUCTURE AND CRYSTALLINITY IN CELLULOSE

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In recent years, several new analysis tools have become available that enable researchers to study the boundaries of structure, polymorphism and crystallinity in materials. Many of these tools have been discussed in prior PPXRD symposium and include quantum chemical calculations (1), studies on amorphous materials (2), full pattern matching techniques (3) and applications of cluster analyses (4,5).

The study of cellulose by diffraction methods offers many challenges and opportunities. The challenges arise from the characteristics of the micro and macro molecular structure. Cellulose is typically fibrous, where the fibers consist of microfibrils with extreme aspect ratio's. The microfibrillar structure can be highly complex consisting of regions of variable crystallinity and polymorphic forms (6). Small levels of natural impurities can further complicate the analyses since many are known to interact with the structure and morphology. The cellulose molecule itself has three hydroxyl units per glucan monomer, that can give rise to a myriad of hydrogen bonding possibilities and many reaction products when cellulose is processed and chemically treated. Overall, while cellulose is one of the most common materials it can be very complex in chemical diversity, structure and reaction chemistry resulting in a wide range of physical properties and performance attributes.

In this presentation the authors have applied the new tools (1-5) to this old problem. This includes the determination of the crystalline polymorphs and the amorphous structure via methods described in references 1 and 2. Subsequently cluster analysis and pattern matching was applied to study pulp and paper as well as pharmaceutical tablets.

(1) J. A. Kaduk, P. Langan, "Crystal structures and powder patterns of celluloses I α , I β , and II", private communication. To be published. Earlier results presented at PPXRD2, **2002**, abstract available at www.icdd.com/ppxrd/02

(2) S. Bates, "Analysis of Short Range Order in X-ray Amorphous Indomethacin Using X-ray Powder Diffraction and Pair-Wise Distribution Function" PPXRD3, February **2004**. Abstract available at www.icdd.com/ppxrd/03 and a related presentation at www.icdd.com/ppxrd/05

(3) T. Degan, "XRPD Pattern Matching: Probability Based Versus Image Comparison" PPXRD5, **2006**. Abstract available at www.icdd.com/ppxrd/05.

(4) C. Gilmore, "The Search for Polymorphs and Salts Using High Throughput Powder Diffraction, Spectroscopy and Other Techniques, PPXRD4, **2005**. Abstract available at www.icdd.com/ppxrd/02

(5) T. G. Fawcett, S. N. Kabekkodu, "Mapping Polymorphs by Combining Cluster Analysis with Diffraction Databases", PPXRD05, **2006**, Abstract available at www.icdd.com/ppxrd/05

(6) R. H. Atalla, B. L. Vander Hart, **1984**, Science, 223, 283

