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Evaluation of cellulosic fiber pore structure with thermoporosimetry

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Cellulose, which is the most prevalent polymer on the planet, usually organizes into hierarchical fiber form. Cellulose chains crystallize into 3-5 nm elementary fibrils, which cluster into macrofibrils that are wound and stacked into the layers of the cell wall of plant-based fibers. Cellulosic fibers, often derived from wood (pulp fibers), are a primary building block of many renewable products, including packaging, textiles, composites, and various functional applications. Cellulosic fibers interact strongly with water and form a porous structure under saturated conditions. The pore volume (fiber swelling) is one of the most important material properties pulp fibers. Fiber pore structure relates to several processing and end-use behaviors, including dewatering, rheology, dissolution, transport, dimensional stability.

The pulp fiber pore size distribution (PSD) is very difficult to measure because it is 1. very wide, extending from the nano to the macroscale, 2. transient with respect to moisture content and drying history and 3. Consists of soft, flexible pores which are subject to osmotic effects. Fiber pore structure can be evaluated in the dry state using critical point drying and classical pore evaluation methods, or in the wet state, using more specialized methods. Thermoporosimetry is one of the more attractive methods for evaluating the pulp fiber pore structure in the mesoporous range since samples can be evaluated in the wet state and at any moisture content.

For this application, the author prefers to carry out thermoporosimetry measurements with a semi-continuous evaluation of the freezing exotherm under conditions that avoid super-cooling. The water inside pores less than about 200 nm consists of water which freezes at a depressed temperature and water that does not freeze at all. Evidence suggests the nonfreezing water (NFW) in mesopores is a monolayer at the cellulose interface, while in micropores the NFW is clusters of water too small (less than 500-1000 molecules) to form an ice crystal. Thus, splitting the NFW into these two fractions allows a more nuanced interpretation of the thermoporosimetry data, including estimation of fiber hydrated surface area.

In this presentation, the interpretation of thermoporosimetry data in relation to the fiber hierarchy is discussed. Results are compared to other analysis methods. The control of the fiber hierarchical structure, its relevance and relation to thermoporosimetry measurements are discussed. It is demonstrated that selective addition of charges groups inside the cell wall can be used to control the swelling of the fibril aggregates independently from the bulk cell wall. This effect can be evaluated with thermoporosimetry.

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