

CELLULOSE FIBRIL AGGREGATION STUDIES OF *Eucalyptus* DISSOLVING PULPS USING ATOMIC FORCE MICROSCOPY

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Dissolving pulp is the end product of acid-bisulphite pulping and bleaching processes, and constitutes the raw material for the production of cellulose derivatives such as viscose and cellulose acetate. Only limited information (and based almost entirely based on softwood species) is available on the desirable characteristics of dissolving pulp.

At a cell wall level, cellulose chains form fibrils about 4 nm in diameter and these, in turn, form fibril aggregates of varying diameters cemented together by a matrix of hemicelluloses and lignin. While the exterior of these aggregates is immediately available to react with chemicals during the production of cellulose derivatives, crystalline and para-crystalline domains within the centre are not¹. During pulping and bleaching, lignin and hemicelluloses bound to cellulose fibrils are removed, allowing closer association between fibrils. Recent studies on Norway spruce pulp using atomic force microscopy (AFM) have reported increased cellulose fibril aggregation during processing, and a concomitant decrease in surface area available for chemical reaction^{1,2}. These findings were subsequently confirmed by studies on *Eucalyptus* using solid-state nuclear magnetic resonance (NMR). Therefore it seems critical to monitor ultrastructural changes in the arrangement of cellulose fibril aggregates (CFA) during pulping and bleaching in order to determine the impact of processing conditions upon the aggregation observed, and how these changes affect the reactivity of pulp. Similarly, it is also important to determine the extent to which wood quality further influences CFA diameters.

The work presented will report on the use of (AFM) to study the CFA characteristics of dissolving pulp of a clone of *Eucalyptus* grown in compartments with contrasting growth rates (low and high), at different stages of the pulping process (solid wood, unbleached [raw] and fully bleached) to different end-product content of alpha cellulose (92 and 96%).

Pulp fibres were rapidly frozen in nitrogen slush and freeze-dried in order to prevent distortion of cell wall structure caused by air-drying. The fibres were then embedded in epoxy resin and microtomed transversely into 1 µm-thick sections. These sections were imaged in tapping mode using a MDT Solver P47H AFM. Image analysis using watershed segmentation was used to measure CFA diameters on the AFM images.

AFM images of the wood, raw and fully bleached 96 alpha grade pulp from the low quality site are shown in figures 1, 2 and 3, respectively. Following pulping, CFA of material from the low site quality increased significantly ($p < 0.05$) from 17 nm in solid wood to 24.5 nm in raw pulp, and again to 27.3 nm in fully bleached

pulp. Similar trends were observed for material from the higher site quality. Marginal differences were noticed between the CFA diameters in fibres fully bleached to 96% alpha cellulose purity pulp from low and high site qualities. These findings are supported by solid-state NMR data from the same samples. However, additional replication and image analysis are required to draw unequivocal conclusions about increases in CFAs due to processing to 92 or 96% α cellulose content. At present, the reactivity of the pulp is being assessed and will be discussed.

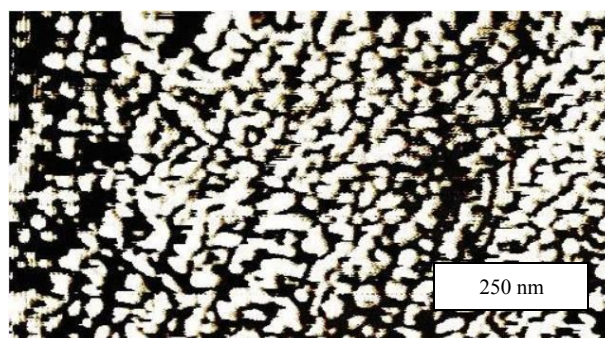


Figure 1. Wood prior to pulping

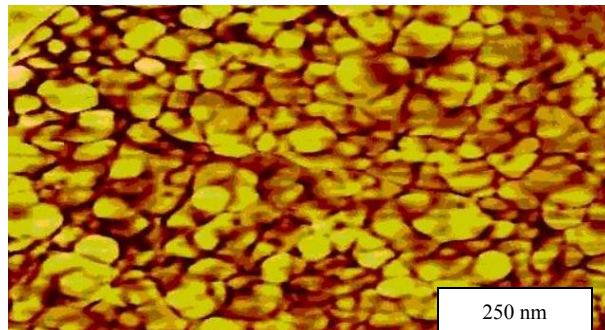


Figure 2. Unbleached (raw) pulp

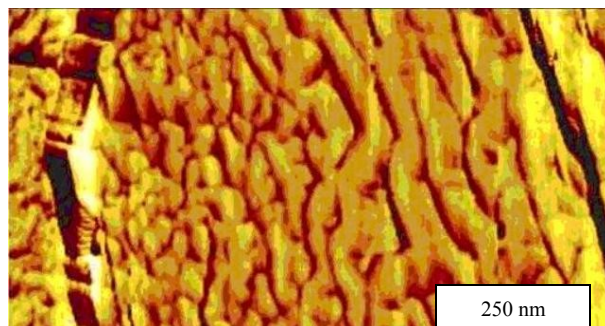


Figure 3. Fully bleached 96 alpha grade

References

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2. Fahlen, J. and Salmen, L. (2003) *J. Mat. Sci.*, **38**, 119-126.

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