

Understanding Cellulose Microstructure in Dry Forming Using Synchrotron-Based X-ray Scattering and Tomography

THE INDUSTRIAL CHALLENGE

Dry forming enables resource-efficient fibre packaging, but industrial uptake is limited by poor understanding of how processing conditions affect fibre bonding and performance. Pressure, temperature, and forming time alter cellulose structure across multiple length scales, yet conventional methods cannot resolve these effects, hindering optimisation and driving trial-and-error development.

WHY USING A LARGE-SCALE FACILITY

Structural features controlling fibre bonding in dry-formed materials span nano- to microscale and vary throughout complex 3D structures. Conventional methods such as light microscopy and Scanning Electron Microscopy (SEM) mainly provide surface or averaged information and lack the penetration and sensitivity needed to probe bulk effects. Small-angle X-ray scattering (SAXS) resolves nanoscale features like fibril aggregation, pores, and density fluctuations, while wide-angle X-ray scattering (WAXS) quantifies crystalline cellulose parameters including crystallinity, crystallite size, and microfibril orientation. At synchrotrons, SWAXS enables micrometre-resolved mapping with high statistical robustness. Combined with X-ray tomography, this provides quantitative 3D correlations between nanostructure, fibre arrangement, and local densification, enabling direct structure–process relationships in dry-formed materials.

HOW THE WORK WAS DONE

SAXS and WAXS measurements were carried out at the ForMAX beamline at MAX IV. Dry formed samples produced under varying press conditions were sliced into 1 mm thin pieces with a laser cut and cross sections of 2 by 4 mm were scanned with a 100 µm beam to capture full thickness of the packaging material including the surfaces in direct contact with the heated tool. The in-situ X-ray tomography experiments under three-dimensional compression were conducted at ID19 at European Synchrotron Radiation Facility (ESRF). A miniature 3D compression tool with a diameter of 3 cm was built to fit on the tomography stage. Due to complex compression cell setup

measuring SAXS and WAXS through the cell was not possible. Follow-up scanning X-ray scattering experiments were performed at DESY P.03 at Petra III where it was possible to go down to 30 µm beam size and BL11-NCD-SWEET at ALBA Cells to validate and extend the initial findings.

THE RESULTS AND EXPECTED IMPACT

Clear links were demonstrated between press parameters and cellulose microstructure across multiple length scales, from fibre packing down to the crystalline cellulose unit. SWAXS revealed how processing conditions influence nanoscale organisation and fibre bonding, while in situ X-ray tomography captured the dynamic behaviour of fibres under three-dimensional compression. These results provide quantitative structure–process relationships that enable more precise control of dry forming conditions and improved reproducibility of material performance. The established methodologies form a strong foundation for future studies on additives, thermoforming effects, and product-specific optimisation, while building long-term industrial capability in advanced characterisation—enabling routine, quantitative evaluation of structure–process–performance relationships to support materials development, process tuning, and product design.



Figure. Fiber pad after compression into a 3D shape using the LTH in-situ compression cell.

“Advanced X-ray tools enabled structure-to-process understanding, facilitating optimisation of dry-formed products.” /Polina Naidjonoka, Yangi

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Vinnova’s project No: 2023-02824 **Duration:** Nov 2023 – Dec 2025

Funded by Sweden’s Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.