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## 3D observation of (nano)-cellulose fibers network by multiscale imaging techniques: from nanometer to millimeter scale.

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The end of single plastic packaging is scheduled in Europe around 2040.

Cellulosic materials such as paper and cardboard are today the only viable bio-sourced alternative, biodegradable and already recycled, which can reach a mass market. Paper is a multiporous material with a large surface area accessible to contaminants. The transport of contaminants via the gas phase or cycles of (ad)sorption/desorption between the different components (shipping boxes, paper, and board), between the fibers and finally between the food particles (powder, grains, flake) is supposed to be the critical factor in food packaging. The roles of the connectivity of voids, micro-, and macro-pores, and the effects of the entanglement of fibers, specific surface area, surface composition, and relative humidity are poorly understood.

The aim of the present work is to characterize the 3D microstructure of a new type of paper for packaging. To reach the desired barrier properties, this new material combines a 200 micron thick paper with a 20 micron thick MFC film: (i) the paper is made of cellulose fibers with a diameter size range of a few micrometers and millimeters, (ii) the MFC film is made of a network of microfibrillated cellulosic fiber with sizes of about 20 to 50 nm in diameter and 500 to 1500 nm in length and ensures the barrier properties. The MFC film is stucked on the paper or cardboard following a wet lamination technique [1].

In the past, several imaging acquisition techniques have been used to characterize the cellulose material such as paper and MFC film [2,3]. These techniques reveal relevant information about the microstructure but depend on the object desired to observe and the pixel size achieved by the source. Therefore, in our case, since the fiber size range is wide and depends on each layer, a single imaging technique will not reveal the microstructural information of the whole bilayer network. Thus, several techniques must be combined to accomplish the full 3D characterization of the bilayer network.

In the present work, the bilayer material has been imaged by either micro or nano synchrotron tomography, and using a FIB-SEM. The 3D X-ray images were performed in ID16B and ID19 beamlines at European Synchrotron Radiation Facility (ESRF). ID16B beamline was used to scan the MFC film at a pixel size of 25 nm/pixel and ID19 beamline was used to characterize the paper and interphase between paper and MFC layer at a pixel size of 0.36  $\mu\text{m}$ /pixel. In parallel, the MFC layer was also scanned using a FIB-SEM technique to reach smaller pixel size (5 nm/pixel) and to highlight details of the MFC film. Despite some artifacts captured by the imaging techniques which hindered the segmentation task, we present for the first-time full 3D reconstruction of this new material. The microstructural properties (porosity profile, specific surface area...) have been then computed. The obtained results suggest that there is no visible pore connectivity in the MFC film which is consistent with the results of nitrogen adsorption/desorption.

### Participation

In-Person

### References

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