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Neutron imaging for the investigation of the lyophilisation of amorphous bulk solids

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Lyophilisation refers to the sublimation of ice below the triple point of water. It is employed for dehydrating biopharmaceuticals and high-value foods in frozen state as the structural and nutritional attributes are not affected. The sublimation front divides the dried area from the frozen area. The knowledge about the sublimation front is important to understand process characteristics and to ensure the product quality. However, the development of the sublimation front, especially in particulate matter, is not yet fully understood and the existing models are contradictory and based on different assumptions. No experimental validation of the existing models exists so far. Therefore, it is the aim to study the sublimation front by in-situ neutron imaging. The experiments were carried out in the Antares beamline at FRMII using maltodextrin particles of two different particle sizes and concentrations. Sublimation for finer particles (x= 70 µm) was investigated by radiography. For the larger particles (x = 3550 µm) continuous tomographic measurements were carried out.
With the reconstructed 3D volumes, we could demonstrate the structure of the drying fronts, whereas with the radiographic images we could estimate the dynamic ingress of the sublimation front. It was shown that for small particles the sublimation front first occurred at the bottom of the particle bed and moved to the top. For large particles multiple sublimation fronts were found.

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