



Contribution ID: 59

Type: Oral Presentation

Use of advanced imaging techniques as a valuable tool to analyze the freeze-drying process in more detail in situ

Tuesday, 31 May 2022 14:35 (15 minutes)

Lyophilization or freeze-drying is commonly applied to stabilize (bio-)pharmaceutical substances and high value foods for long-term storage. The freeze-drying cycle typically consists of three stages: i: freezing ii: primary drying and iii: secondary drying [1]. The freezing step is the most crucial one because the performance of the overall process vastly depends on the freezing step. Since the freezing parameters fixes the morphological structure of the dried material, they directly influence the pore size distribution and the connectivity of the porous matrix. Hence, it affects the heat and mass transfer through the dried cake, which effects the drying rates of both, the primary and secondary drying stages [1,2].

While the influence of pore size on the freeze-drying process is already known, surprisingly other structural parameters like pore orientation or shape is still not sufficiently investigated. Next to the microstructure, process design as well as critical properties of the formulation are important. Drying above critical parameters can lead to changes in microstructure and thus, directly influence the overall drying process and product quality [3].

In this study we will present the influence of the pore structure on the overall drying kinetics and how the pore structure will change during drying. This investigation is carried out in a lyomicroscope. Furthermore, for the first time our self-developed freeze-drying stage [3,4] inside a 4D μ -CT (DynaTOM, TESCAN) to observe structural changes in 3D with a high temporal resolution in-situ is used. Here, maltodextrin and sucrose solutions with different solid concentrations ($c = 0.05$ w/w, $c = 0.2$ w/w and $c = 0.3$ w/w) were used as model substances. In order to estimate the structural changes during the drying process, an in-house image processing code is applied to analyze microstructural parameters like pore size, orientation and shape. The change in the morphological structure at the different drying stages is discussed. The experimental data will further be transformed into irregular pore networks and used for further modelling of drying. The results will lead to guidelines for a faster freeze-drying process with high product quality.

Acceptance of the Terms & Conditions

[Click here to agree](#)

MDPI Energies Student Poster Award

Yes, I would like to submit this presentation into the student poster award.

Country

Germany

References

- [1] Aurélie Hottot, Séverine Vessot, and Julien Andrieu. 2004. A Direct Characterization Method of the Ice Morphology. Relationship Between Mean Crystals Size and Primary Drying Times of Freeze-Drying Processes. *Drying Technology* 22, 8, 2009–2021. DOI: <https://doi.org/10.1081/DRT-200032717>.
- [2] J. A. Searles, J. F. Carpenter, and T. W. Randolph. 2001. The ice nucleation temperature determines the primary drying rate of lyophilization for samples frozen on a temperature-controlled shelf. *Journal of pharmaceutical sciences* 90, 7, 860–871. DOI: <https://doi.org/10.1002/jps.1039>.
- [3] Sebastian Gruber, Nicole Vorhauer-Huget and Petra Foerst. 2021. In situ micro-computed tomography to study microstructure and sublimation front during freeze-drying. *Food Structure* 29, DOI: <https://doi.org/10.1016/j.foostr.2021.100213>
- [4] Mathias Hilmer, Sebastian Gruber, and Petra Foerst. 2020. Development of a Freeze-Drying Stage for In-Situ μ -CT Measurements. *Processes* 8, 7, 869. DOI: <https://doi.org/10.3390/pr8070869>.

Time Block Preference

Time Block B (14:00-17:00 CET)

Participation

In person

Primary author: GRUBER, Sebastian (Technical University of Munich, Chair of Process Systems Engineering)

Co-authors: THOMIK, Maximilian (Otto-von-Guericke University, Magdeburg); Dr VORHAUER-HUGET, Nicole (Otto-von-Guericke University, Magdeburg); Mr COPPENS, Frederik (TESCAN XRE, Belgium); Prof. TSOT-SAS, Evangelos (Otto-von-Guericke University, Magdeburg); Prof. FÖRST, Petra (Technical University of Munich, Chair of Process Systems Engineering)

Presenter: GRUBER, Sebastian (Technical University of Munich, Chair of Process Systems Engineering)

Session Classification: MS10

Track Classification: (MS10) Advances in imaging porous media: techniques, software and case studies