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Data Article

Visualization data on the freezing process of micrometer-scaled aqueous citric acid drops

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A R T I C L E   I N F O

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Keywords:
Emulsi\textipa{\textregistered}fi\textipa{\textregistered}ed aqueous citric acid
Freezing
Freeze-induce phase separation (FIPS)
Freeze-concentrated solution (FCS)
Lyophilization/freeze-drying

A B S T R A C T

The visualization data (8 movies) presented in this article are related to the research article entitled "Freezing and glass transitions upon cooling and warming and ice/freeze-concentration-solution morphology of emulsi\textipa{\textregistered}fi\textipa{\textregistered}ed aqueous citric acid" (A. Bogdan, M.J. Molina, H. Tenhu, 2016)\textsuperscript{[1]}. The movies recorded in-situ with optical cryo-microscopy (OC-M) demonstrate for the first time freezing processes that occur during the cooling and subsequent warming of emulsi\textipa{\textregistered}fi\textipa{\textregistered}ed micrometer-scaled aqueous citric acid (CA) drops. The movies are made publicly available to enable critical or extended analyzes.

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Specifications Table

Subject area
Pharmaceutics, Biotechnology, Tissue Engineering

More specific subject area
Freezing step in lyophilization

Type of data
Movies

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How data was acquired: Movies were recorded using an Olympus BX51 optical cryo-microscope equipped with a Linkam cold stage, Linksys32 temperature control and video capture software.

Data format: Raw, processed

Experimental factors:
- Emulsified micrometre-scaled CA/H2O drops were prepared by magnetic stirring of CA/H2O solutions with a Halocarbon-0.8-oil/lanolin matrix.

Experimental features:
- Emulsion samples were placed in between a standard 75 x 25 mm microscope slide and a cover glass. OC-M measurements were performed at the cooling and warming rate of 3 and 5 K/min between 193 and 300 K.

Data source location:
- University of Helsinki, Finland

Data accessibility: Movies are presented in this article.

### Value of the data

- The movies provide visual insights into the physical chemistry of freezing dispersed aqueous solutions and can be used by other researchers who work with freezing phenomenon in fields ranging from life sciences and biotechnology to geophysics and high-altitude ice clouds.
- The movies demonstrating the freezing process were recorded *in-situ* using OC-M and can be compared to freezing results obtained with other techniques, for example, confocal fluorescence microscopy as well as to results obtained by computer simulations.
- Since the movies are first of their kind, they can be used/give an impetus in/for the development of further experiments in different fields of science and technology where freezing phenomena play important role.

### 1. Data

The movies presented in this data article provide the visualization evidence of a freeze-induced phase separation (FIPS) into pure ice and a freeze-concentrated solution (FCS) which occurs during the freezing of micrometer-scaled CA/H2O drops. These movies also demonstrate how the ice/FCS morphology of frozen drops changes with decreasing drop size.

### 2. Experimental design, materials and methods

We prepared 10–60 wt % CA solutions by mixing > 99% anhydrous citric acid (Merck) with the corresponding amount of ultrapure water. For emulsion preparation we used an oil-surfactant matrix consisted of 80 wt % halocarbon 0.8 oil (Halocarbon Products Corp.) and 20 wt % lanolin (Sigma Aldrich). CA/H2O/oil-surfactant-matrix of 1/10 by volume were subjected to magnetic stirring at different speeds in order to obtain CA/H2O drops of different size distributions [1]. In our measurements, we used methodology based on a ‘2-dimensionál’ solution strategy designed for the *in-situ* observation of FIPS and ice/FCS morphology by applying OC-M [2,3].

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A.B. thanks University of Innsbruck for using OC-M techniques. Starting from February 2014, A.B. obtained a small financial support from Arbeitsmarktservice (AMS, Innsbruck, Austria) during 6 months. After that time, this research did not receive any financial support or specific grant from funding agencies in the public, commercial, or not-for-profit sectors.
Transparency document. Supporting information

Transparency data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2016.11.037.

Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2016.11.037.

References