Characterization of high protein anisotropic structures using Rheological fingerprint by large-deformation Lissajous curves

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ABSTRACT

Our food system needs to change to provide more sustainable diets in the future. In this transition, new formulations are being developed with limited knowledge of their impact on structure and texture development during processing and storage. It is imperative not only to learn the details of the structure but being able to follow the change dynamics to ultimately control processing. In many process operations, where structures are being developed, the deformation can be large, resulting in a non-linear response from the material. Large amplitude oscillatory shear (LAOS) allows measurement of this non-linear viscoelastic behavior. In this work, LAOS rheology was evaluated as a means to characterize the non-linear mechanical response of anisotropic soft food materials during processing. Pizza cheese was used as a model system, due to its well known macroscopic anisotropy of the protein fibers, as a result of the cheese stretching process. A newly developed software was created based on data visualization theory to better highlight non-linear dynamics between samples. This presentation will demonstrate how novel features of the visualization software can enhance the understanding of non-linear rheological data, providing better tools to compare sample responses and how to link these responses to structural differences.

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Fig. 1. Schematic illustration of the strain sweep test at a fixed frequency. This sweep test can be used for determining the linear and nonlinear viscoelastic region. In the linear region, the oscillatory shear test is called SAOS (small amplitude oscillatory shear), and the application of LAOS (large amplitude oscillatory shear) results in a nonlinear material response. Illustration and text borrowed from [1].
Fig. 2. The three-dimensional space of Lissajous curves of a viscoelastic material, and the two-dimensional projections from either the elastic (strain vs. stress) or viscous (strain rate vs. stress) perspective.

Fig. 3. Lissajous curves from the elastic perspective at different strain amplitudes ranging from 0.1 to 100%.
Fig. 4. Pipkin diagram of normalized Lissajous curves of pizza cheese samples during the processing and storage at 0.1–100% strain amplitude. Sample taken in longitudinal direction to protein fibers. L1 = curd, L2 = during processing, L3 = final cheese, L4 = final cheese stored 1 week and L5 = final cheese stored 2 weeks. Values for maximum stress, stiffening ratio and thickening ratio indicated above individual graphs.

Fig. 5. 2D Layover plot of Lissajous curves for pizza cheese during processing and storage at 1% (a) and 100% (b) strain amplitude. Cheese samples taken in the longitudinal direction to protein stretches. Sample key: L1 = (dark blue), L2 = during processing (purple), L3 = Final cheese (green), L4 = final cheese stored 1 week (brown) and L5 = final cheese stored 2 weeks (light blue). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
Fig. 6. Stiffening index of the five cheese samples: Sample key: L1 = (dark blue), L2 = during processing (purple), L3 = Final cheese (green), L4 = final cheese stored 1 week (brown) and L5 = final cheese stored 2 weeks (light blue). Corresponding confocal images of sample L2 and L3 where protein and fat regions are colored in green and red, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 7. Pipkin diagram of normalized fingerprint Lissajous curves of pizza cheese sample stored one week, strain amplitude raining 1–1000% and temperature 20–40 °C.
Fig. 8. Similarity Network map showing mechanical responses of a final cheese sample analyzed at the day of production at 5–80 °C (symbols) and 100–1000% (colors) strain amplitude. Red dots correspond to a reference point of a purely elastic material. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 9. Peak force derived from hardness experiment in double compression test performed by Texture Profile Analysis (TPA). The large deformation is performed in both longitudinal (blue) and transverse (purple) to protein fibers in the cheese. Responses of the five samples taken during production and storage of the pizza cheese. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
Fig. 10. Area layover plots of Lissajous curve responses for the large deformation performed at 100% strain amplitude by LAOS rheology in both longitudinal (blue) and transverse (purple) to protein fibers in the cheese. Responses of the four samples taken during production, from where anisotropy is introduced, and through storage of the pizza cheese.

Fig. 11. Illustration of cheese sampling and testing directions for texture profile analysis (TPA) and oscillatory rheology.
CRediT authorship contribution statement

**Julie Frost Dahl:** Conceptualization, Methodology, Investigation, Writing – original draft. **Sandra Beyer Gregersen:** Writing – review & editing, Project administration. **Ulf Andersen:** Writing – review & editing. **Hans-Jörn Schulz:** Software, Visualization, Data curation. **Lasse Sode:** Software, Visualization, Data curation. **Jonas Madsen:** Software, Visualization, Data curation. **Milena Corredig:** Conceptualization, Writing – review & editing, Supervision.

Data availability

Data will be made available on request.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References


Julie Frost Dahl received her engineering degree in Dairy Science and Technology from the University of Copenhagen, Denmark in 2021. She is now a PhD Fellow at Department of Food Science at Aarhus University, Denmark. Her research activities are focused on processing protein rich ingredients in multiphase systems, with an initial focus on methodological developments.

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Milena Corredig has been a Full Professor in Food design and technology since 2006, at University of Guelph and since 2019 at Aarhus University, in the FOOD department. She is currently also GIFood Center Leader. Prof. Corredig completed her Bachelor studies at the University of Milano, Italy, and her Master and PhD in Food Science from the University of Guelph, Canada. From 1998 to 2003 she was a researcher, and lecturer at the University of Georgia, in Athens, Georgia, USA. In 2003 she was offered a position as Research Chair in Dairy Technology at the University of Guelph. From 2013 to 2018 she was head of R&D for Gay Lee Foods, a dairy Company owned by 1400 Farmers, in Toronto, Canada, responding to the CEO.

To date, Prof. Corredig has published more than 250 peer reviewed publications, and gave as many public and conference presentations. She also held many professional positions. Within the University domain, she has been Department director of graduate studies, and acting Department Chair. She is also member of the editorial board for Food Hydrocolloids, the Journal of Dairy Research and Journal of Food Texture.

Her research priorities are food design and processing, connecting the domains of food biochemistry, food processing and material science. When designing foods, it is paramount to understand the molecular and supramolecular properties of the ingredients, and how they interact with the other components of the matrix during processing, supply chain and consumption. By knowing such details it is possible to design more functional foods, healthier foods and high quality products, both fresh and preserved products. This will also be vital to improve or create new functional ingredients from raw materials, taking into consideration also the use of byproducts in a new, circular food economy. A better understanding of the interactions between raw materials, ingredients, alternative processing approaches, storage and distribution, will provide new tools to create more sustainable food products in the future.