IFPRI Research project - ExPowSE

Exploring food powder surface under controlled environment

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I. Proposal context, positioning and objective(s)

I.A. Objectives and research hypothesis

The current inability of most consumers to achieve the recommended daily intake of fruits and vegetables triggers food research toward the generation of new and improved fruit- and vegetable-based products and ingredients. The World Health Organization (WHO) recommends consuming at least 400 g each day to reap their health and nutrition benefits. In 2017, some 3.9 million deaths worldwide were attributable to the lack of fruit and vegetables in the diet (**WHO, 2019**). Actions are needed to **increase the production and consumption of fruit and vegetables and make them more economically accessible to consumers, while generating economic, social and environmental benefits in line with the Sustainable Development Goals**. In declaring 2021 as the international year of fruits and vegetables, the United Nations (UN) general assembly aims to raise awareness of the nutritional and health benefits of fruit and vegetables and their contribution to a balanced and healthy diet and lifestyle (**FAO, 2020**). The diverse range and characteristics of fresh fruit and vegetables and their inherently perishable nature warrants the deployment of conservation techniques in order to: (1) **guarantee their accessibility** throughout the year and in particular outside production periods, (2) **deliver stable products** in non-producing regions by exportation, (3) **reduce food waste** due to seasonality and report the consumption by using drying and powdering methods. However, these operation units govern powder structure and functional properties and it is of paramount importance to master them.

The overall scientific objectives of ExPowSE are:

- Preserving product stability and quality.
- Bringing knowledge in the technofunctionality of food powders using a multiscale approach with a focus on the particle surface. Even if the powder surface is one of the main players during the reconstitution or transport (powder flowability), it has been poorly studied in the literature for fruit and vegetables powders.
- Elucidating physical mechanisms occurring when materials reach the glass transition by increasing temperature and/or relative humidity (RH). Until now, only hypotheses were proposed as the techniques were not able to probe the progressive evolution. Atomic Force Microscopy (AFM) is the technique of choice to gather physicochemical and nanomechanical data during the glass transition phase.

A profound understanding of the **process–structure–function relations** to tailor the **functional properties** of fruits and vegetables powders is then required. A schematic representation of the key concept of the ExPowSE project is displayed in **Figure 1**.

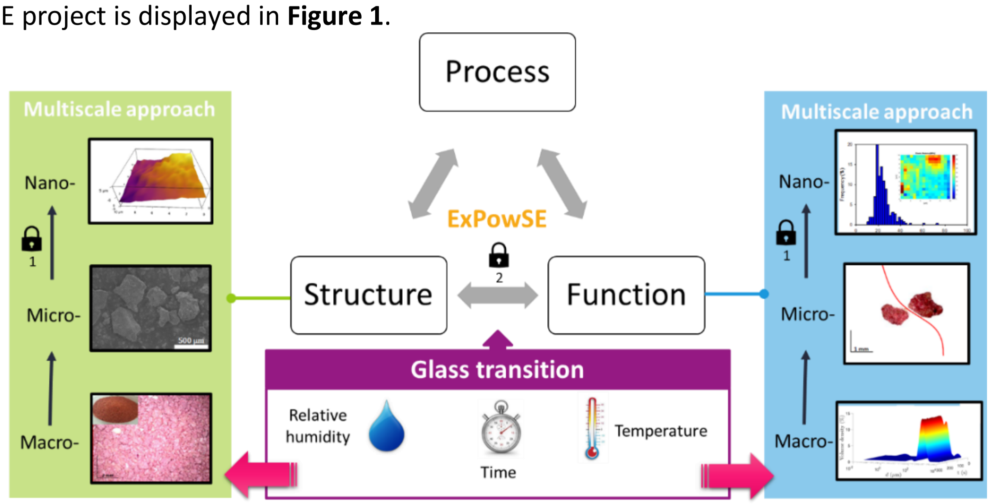

Figure 1: Schematic overview of the ExPowSE project: process–structure–function relations with a multiscale approach.

Research on this project will be performed at a nanoscopic, microscopic, and macroscopic level entailing particle surface investigations with the aim to answer to the following questions:

- How surface modification (topography, chemistry, nanomechanics) can impact the macroscopic behaviour (reconstituability and flowability) of the powder?
- What are the phenomena occurring at particle surface during the glass transition phase?
- How to improve powder stability and quality during shelf-life?

I.B. State of the art

Many types of fruit and vegetables are processed to increase their shelf-life, year-round availability, or to increase their value, which integrates structure-enabling and preservation techniques. Minimal processing includes drying and/or grinding (powdering process) of fruits and vegetables and guarantee that such foods are as nutritious as the food in its unprocessed form (**Fitzpatrick and Ahrné, 2005**). However, fruits and vegetables powders contain a high quantity of low molar mass sugars with low glass transition temperature (T_g) (**Fang and Bhandari, 2011**). The direct consequence is that fruit powders are highly hygroscopic and sticky at high temperatures but also at ambient temperature if the water content is not well mastered. This feature causes the powder adhesion to surfaces and powder caking during the storage, which affects the quality of the final product. **The T_g is one of the most important parameters to consider during powder storage.** Indeed, the phenomenon of glass transition is the gradual and reversible transition of amorphous materials from a hard and "glassy" state into a viscous and rubbery state as the temperature and/or the moisture content is increased. The glass transition is also linked to the water activity and water as a strong plasticizer decreases the T_g. Also, the glass transition temperature depends on the molecular mass as the T_g of monosaccharides, disaccharides, oligosaccharides and polysaccharides increases with increasing their molecular mass (**Roos, 2002**). If the glass transition is reached during powder storage, unexpected phenomena can happen that affect powder functionality such as reconstitution ability.

An important structure-determining component in this context is the particle surface. It should be noted that particle surfaces, in the case of fruits and vegetables powders, are essentially constituted by broken structures. Because of various origins, their particle size and shape distributions, chemical composition, surface composition, and physical properties are highly variable. Therefore, more than one analytical technique is often required to obtain a full set of information about a given scientific question (**Burgain et al., 2017**). Among these questions, the powders flowability and reconstitution are of upmost importance for the industry considering that most powdered ingredients are transported and dissolved or infused before use. For the past few years, numerous powder surface analysis techniques were used to further understand the role of powder surface on functionalities impairments. For example, microscopy techniques such as Scanning Electron Microscopy (SEM), Confocal Laser Scanning Microscopy (CLSM) or even chemical composition techniques such as X-ray Photoelectron Spectroscopy (XPS) are already widely used (**Burgain et al., 2017**). However, **AFM is currently a rising star in the food powder surface analysis field**, mainly for its resolutive capacity. AFM is a versatile tool compared to other surface analysis techniques. For example, AFM allows to study particle surface topography and roughness, surface chemistry and nanomechanics.

In a previous project, we were able to have a better understanding of surface modification after high temperature storage of whey protein isolate and micellar casein powders (**Burgain et al., 2016a, 2016b**). Surface hardening with the development of a poorly dispersible skin layer composed of aggregated micelles was evidenced to be the phenomenon responsible for the reconstitution impairment. However only punctual analyses were possible and the development of an environmental chamber around the AFM will allow for continuous measurements at nanometer scale. **By controlling the temperature and RH, their variation during time in the neighbouring of the sample will provide new insight in the elucidation of mechanisms occurring during powders storage or transport under unfavourable conditions, in particular when they reach the glass transition.** Even if AFM was already applied to dairy powder, application to fruits and vegetables powders is still missing while there is an important industrial stake with the growth of the plant products market and the development of vegetable formulations.

First experiments on fruits and vegetables powders provided hopeful results showing that when approaching glass transition, patches at the surface were crystallising by nucleation and the rest of the matrix presented a decreased elasticity (**Figure 2**). The glass transition event is accompanied by a physical change at the powder surface (modified topography) with a change in the Young modulus (modified nanomechanical properties) (**Palzer, 2007).** Moreover, powder caking related to glass transition is promoted by moisture adsorption which create liquid bridges between hydrophilic groups at particle surface (modified physicochemical properties). **The strength of the technique is that it is now possible to follow the evolution of the same area during dynamic variation of temperature and RH**. These observations confirm the fact that a focus at particle surface is undeniably required to better understand macroscopic phenomenon such as powder flowability, caking or reconstitution. This is particularly true for fruit powders that are highly hygroscopic materials with low glass transition temperature and as a consequence easily affected by ambient temperature and RH. In a recent work, we evidenced that above T_g, a viscous layer around the particle limits water entrance and is the limiting step in the global reconstitution process (**Gaudel et al., 2022**). With AFM, surface structure and chemical properties will be correlated thanks to the combination of surface topography analysis and force measurements.

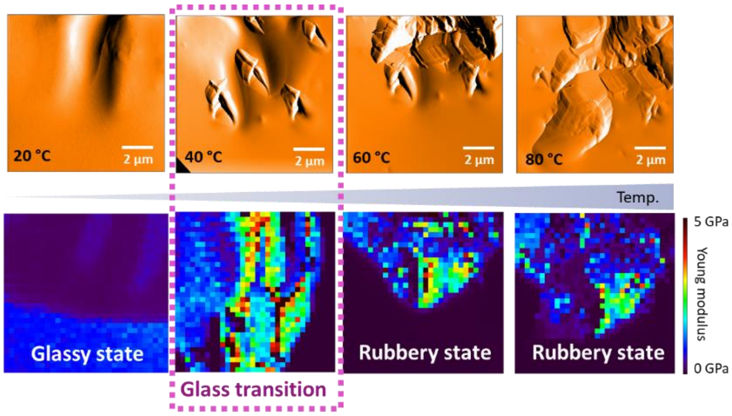

Figure 2: Use of AFM on maltodextrin powders with progressive temperature increase. The same area was analysed in situ during temperature increased (unpublished results from the team).

I.C. Project methodology

The project will be divided into three Work Packages (WP).

WP1: Fruits and vegetables powders screening (months: 0 – 12). The powder functional properties will be investigated at the macroscale by the evaluation of hygroscopicity, flowability and reconstituability. Powder thermal properties will also be determined and linked to powder proximate composition. In this WP, powders will be grouped as a function of their origin, T_g, solubility index, flowability index and only few powders of each group will be investigated in the WP2.

WP2: Probing surface physicochemical and nanomechanical properties by AFM-CE (months: 6 – 30). In this WP, a newly designed Controlled Environmental chamber around the AFM (AFM-CE where temperature and RH can be managed) will be employed. AFM-CE will be employed for the determination of particle surface topography and roughness and for the estimation of nanomechanics and adhesion forces. Even if process parameters define powder surface structure and functional properties, they can be impacted by environmental conditions. The technical and scientific challenge in the ExPowSE project will be to observe and quantify surface evolution during temperature and RH modulation (**Figure 1 - Locker 1**). In fact, the design of the environmental chamber was done in a way to recreate thermal and hygroscopic conditions that can be encountered by powders during transport and storage, but it is also possible to reach higher conditions in order to exceed the glass transition region. However, mechanical features of the AFM tip can evolve with increasing temperature or water droplet can deposit on the tip. All of these technical issues must be mastered to produce reliable data.

WP3: Modelling the process–structure–function relations (months: 24 – 36). Using mechanistic and statistical models, data will be processed in order to establish the process–structure–function relations and describe mechanisms at the basis of surface evolution (as a function of temperature and RH) and impacting powder reconstitution and/ or flowability. The second scientific challenge here will be to link powder surface structure with powder function and to get enough data to model the phenomena occurring and elucidate the underlying mechanisms (**Figure 1 - Locker 2**).

II. Team and link's with the actual IFPRI project

The work packages outlined above will be carried out by a dedicated full time PhD student 100 % funded by the IFPRI Consortium. Materials and consumables will be covered by the LIBio. This project can be leveraged to obtain local funds from the Grand Est region and the University of Lorraine Impact "Biomolecules" program leaded by a LIBio professor. These organizations provide 2-to-1 support for industrial cash grant. The PhD student will be embedded in the LIBio laboratory under the direct supervision of Prof. Claire GAIANI and Jennifer BURGAIN. They received more than of 2 000 k€ funding for projects on which they were principal investigators. Among other important activities, they successfully managed fundamental competitive programs including the French ANR and European projects. They also participated in international collaborations. Finally, they managed many industrial partnerships with leading international food companies (including Nestlé, Lactalis, Bel, Arla Food, and CNIEL).

Nowadays, their research thematic deals with the establishment of links between surface and functional properties of food powders. The objective will be to further develop, the work that was recently initiated into the IFPRI project managed by Claire GAIANI (more particularly in the WP2). The idea is to loop the approach process-structure-function and the development of AFM in controlled environmental.

The ExPowSE team will be managed by Claire GAIANI and Jennifer BURGAIN and composed of one PhD student, one technician (working mainly on the AFM technique) and a specialist of food glass transition (Pr Stephane DESOBRY). Therefore, four permanent staffs will be involved in the team plus one PhD student (IFPRI funds). The complementarity of the team members is clearly an asset for this project.

Finally, this project will be in close collaboration with the IFPRI members with regular meetings and reports. We will test powders of interest for the industry and if possible directly from the industrial partners.

III. References related to the project

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