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Deniz Z. Gunes

Nestlé Research Center, Institute of Material Science, Vers-chez-les-Blanc, CH-1000, Lausanne 26, Switzerland

Abstract

The advent of Microfluidics as a research field radiating toward all other material science disciplines triggered a number of paradigm shifts, which changed visibly the research horizon in Food science. Food engineering as a discipline, which was sometimes criticized for the conservatism of its approaches and aspirations, started finding some important benefits from using Microfluidics as a research accelerating, idea testing, and micro-scale prototyping provider of tools, allowing to probe the relationship between microstructure and end material properties, and building adequate design rules. This short review will attempt to demonstrate this by taking examples in Food structure design with: emulsion and foam stability studies; structures with complex interfaces; controlled delivery systems; analytical approaches and tools based on microfluidics.

I. Introduction

Food science long suffered from the fact that, unlike pharmaceutical products, most food products are commodities, raising the question how far scientific research on food's most common properties, could further impact the industry and consumer's lives. Those properties, i.e. for the most obvious ones, texture, appearance, nutritional quality, stability, aroma profile, result from the multiscale interactions between its components, which is subject to the same laws as any soft material [18]. With the recent popularization of Microfluidics, specific and multiple answers to the above question were given within only a few years in articles published in international science journals, while Food science has been part of the journey of fast development of microfluidic tools. The aim of this review is to show that Microfluidic approaches actually offer unique insight capabilities into food engineering from a soft matter perspective [18,19], while providing tools and a new field for innovative design of processes and structures. They help widening both the applied industrial immediate potential and the research horizons, in particular in the direction of improved nutrition. This paper is organized as follows. I. Introduction. II. Emulsions and foams: structure design by flow control, and study of stability. III. Colloidal structures from control on complex dynamic interfaces: for encapsulation and stabilization. IV. Microfluidics based analytical tools. IV.1. Structure and property probing in food. IV.2. Molecular and biological sensing. V. Conclusion: what's next for Food and Microfluidics ?

II. Emulsion and foams: structure design by flow control, and study of stability

The use of microfluidic techniques opened a large domain of food structure design potential, within which the access to monodisperse food emulsions and foams has been one well-known entry door. Previously available techniques for obtaining monodisperse emulsions relied on either fractionation between different size range populations by use of depletion interactions [1], or centrifugation in a density gradient fluid [2]. The advent of Microfluidics made accessible to every scientist simple approaches to produce μm - to mm -sized emulsions and foams, whose behavior could then be studied in greater details

than without proper control on size distributions. Simple geometries were demonstrated to be functional, e.g. needle-based - with cross-flow [11] or co-flow geometry [10], on-chip with a T-junction [3,5], flow-focusing geometry [7], or flow through a nozzle [4].

Special designs for achieving particular local flows on-chip were used for coalescence studies e.g. while inducing controlled drop collisions with adjustable contact time [20,23,24] (fig.1). An important subcase is the possibility to control a local flow in a close-packed chain of drops, thereby simulating local capillary relaxation induced flows, which can cause avalanches of coalescence propagation in concentrated emulsions [21,22] (fig.1b). Coalescence studies at droplet scale would otherwise require the use of a computer-controlled four-roll mill [31] or an Atomic Force Microscope (AFM) [32]. Similar approaches are valid to study foam stability against coalescence, whereby it is critical to control the initial size distribution of bubbles, as well as gravity effects, because of the coupling between destabilization phenomena (drainage, coalescence, Ostwald ripening) in a foam. Proper design of the sample environment allows studying path-dependent phenomena involving surface-active entities, whose adsorption energy is very large compared to Brownian energy, as in the case of Pickering stabilization [34,36]. The use of 2-dimensional (2D) cells with a control on the size distribution allows quantification of the efficiency of various stabilizers against foam coarsening, and against each of the destabilizing phenomena, e.g. Ostwald ripening, while coalescence events can be counted using image analysis [33,38]. The link between single bubble dissolution behavior in a microfluidic chamber, which could be studied at high resolution in chemical potential gradient, and foam coarsening, was demonstrated [35,37]. It is worth noting the possibility to take into account specific 2D effects on drainage [39].

Figure 1. Illustration of the use of Microfluidics for emulsion coalescence studies. (a) Schematics and images showing the principle of on-chip oil-in-water drops coalescence study (from [23]). (b) Image showing part of a microfluidic geometry with on-going propagation of coalescence events of water-in-oil drops, induced by a local extensional flow – arbitrary example showing the probability of coalescence and its propagation within a chain of drops, as a function of the separation drop-drop capillary number, Ca_{sep} (from [22]).

The monodisperse character permitted by Microfluidics was proven to be necessary to better exploit the potential of many techniques for emulsion studies, e.g. in the case of diffusion-NMR for size and polydispersity characterization, as well as for NMR imaging [41,42,43]. Other studies became possible thanks to the fine size control permitted, e.g. the influence of bubble packing symmetry on monodisperse foam (or emulsion) rheology [44].

Geometries were engineered to induce passive drop detachment by capillary pinch-off, e.g. from high aspect ratio pores [29] or by step-emulsification [30,79]. These geometries possess the inherent property that they can be parallelized by numbering-up only the pore design, while using a cross-flow for conveying the drops produced towards the exit of the chip [80,30,79,88], since the size produced would not depend strongly on the cross-flow stress. One drawback however originates from the low operating transmembrane pressure, which introduces possible pressure coupling between adjacent pores potentially making the drop production irregular. Proper choice of design symmetry is necessary to avoid inactive pores [58]. However, throughput levels reached were satisfactory for high-added value applications, e.g. encapsulation of expensive actives in monodisperse capsules [81]. Larger throughput monodisperse emulsification was achieved by removing the microchannel structure while keeping the step drop formation principle [50].

By introducing a large stress cross-flow up to higher than the critical capillary number for drop/bubble detachment at wall, and using a numbered-up design, industrially realistic throughput levels became possible [45]. Continuous tuning of size by adjusting the capillary number for proper drop/bubble formation at wall was then possible [46]. This approach proved to be most powerful when considering the efficiency of bringing surface-active species on bubbles (drops) for stabilizing foams (emulsions) in a complex environment, which was applied in practice e.g. for ice-cream stabilization [47,33]. This question is known to be critical in food structure design, e.g. in ice-cream, for two reasons: the kinetic (based on diffusion coefficient) competition and the thermodynamic (based on adsorption and desorption energies) competition for structuring at interfaces [48]. By controlling the wettability of pores, membrane emulsification allowed the production of double-emulsions, which were then considered as the basis for low-fat food emulsions such as mayonnaise or spreads [46,51], as well as for controlled delivery [83].

Researchers using microfluidics rapidly realized that the high degree of control offered by laminar flows, came at a cost e.g. a low throughput or parameter constraints, which may not be the best compromise depending on the specific application considered. A typical example is the case of emulsion property design though drop size control, when not targeting strict monodispersity. Extensions of those principles initially developed for slow flows were realized. One example is membrane emulsification in the jet regime, using a strong cross flow detaching drops at high capillary number from capillary threads: good, yet not perfect, control of the production quality was maintained, whilst achieving higher throughputs [49].

Properly addressing the question of how the different techniques cited above compare in delivering controlled and narrow size emulsions, would deserve a dedicated paper. We can however briefly underline some differences here. Single microfluidic channels allow to achieve low standard deviations in size, typically 1-3%, even for multiple emulsion formation [82]. Parallelized microfluidics in practice allows to reach less than 5% standard deviation [90], but introduces a difficulty regarding the fouling control of each channel [124]. For productions, which do not need complex geometries, numbered-up but interdependent microfluidic pores allow to achieve low polydispersity, in particular when using capillary relaxation for drop detachment [30,79]. A fraction of the pores may however be inactive due to low operating transmembrane pressures [58]. Another technique using step emulsification, but suppressing the single pore geometry, may overcome the latter issue – and its scale up may be easier [50]. The above-cited approaches contrast with membrane emulsification techniques making use of more complex and less controlled pore structures, whereby we direct readers to dedicated reviews e.g. [125]. The emulsion size distribution then depends on the pore size distribution, on top of

other factors e.g. the role of vortices at higher Reynolds number [126], or the probability of coalescence [127]. A more simple industrial set-up to achieve moderate size distribution of emulsions is *repeated membrane emulsification*, which involves several passes through the same (or type of) membrane, generally starting from a coarse emulsion [128].

III. Colloidal structures from control on complex dynamic interfaces: for encapsulation and stabilization

By providing unprecedented level of control on most system's parameters i.e. flow, composition and thermodynamics, microfluidic techniques made possible the discovery and study of new functionalities from already existing materials. More than a decade ago, micro-engineered channels allowed the formation of particles with controlled size, shape and composition through the use of tuned and uniform conditions in microflows, with controlled gradients [12,100]. The basic principles were further developed in the direction of interfacial structure formation of microcapsules for e.g. controlled delivery in food using biopolymer capsule shells, based e.g. on electrostatic complex formation [53,54]. Such structures are thought to also have promising nutritional [95,97,98], agricultural [96], and biomedical applications [94]. One example is how Microfluidics allowed to determine the parameters for controlling the size and shell thickness of food-grade biopolymer capsules with tunable properties [52]. The achieved degree of control allowed to hypothesize a diffusion-controlled mechanism of capsule membrane thickness (δ) growth with time (t), since a $\delta \sim t^{1/2}$ dependence was observed. The approach could be extended to capsule fabrication using minimally processed materials, e.g. with pure coffee materials. The capsules were then more rigid and their content's release could be temperature-triggered [86,87]. For both types, the capsules were shown to be able to consolidate, at the condition that the shell could grow rapidly enough to compensate for the very low interfacial tension. The most important general learnings from the above studies have some general character. Uniform sizes and close control of capsule formation conditions, leaving enough time for capsule formation on chip under well-determined hydrodynamic flows, permitted better comparisons, in contrast to batch stirring conditions, for which size and elasticity vary on several orders of magnitude [16]. Highly controlled monodisperse conditions were preferred to form the more rigid and temperature sensitive capsules, while there were not found to be necessary to form the more deformable ones, if highly uniform shell properties were not required. In the latter case, Microfluidics helped to find the conditions under which they can form, which could then be translated into macroscopic processing conditions.

The possibility to operate Microfluidics at low flow stresses was emphasized for encapsulation, e.g. of cells, whereby uniform size and properties of gel microbeads should give a higher control on cell stress [8]. More recent studies described the use of interfacial flow tuned to form liposomes, using microstructured channels and membranes [92,93]. Electrospraying technology, which can apply to liposome and other capsules manufacture, also benefited from Microfluidics insights to engineer hybrid liposome/protein microcapsules [61]. In general, the multilayering type of approach is now widely explored to achieve complex or multiple functions in controlled delivery, in particular using the layer-by-layer adsorption approach [94]. A microfluidic solution was recently demonstrated to solve the major issue of the latter approach, which is the multiple adsorption/washing steps at the forming capsule's surface, whereby a continuous solution was proven [99].

Another promising approach in Food science to achieve quite versatile solutions for controlled delivery applications, as well as for calorie reduction without too strong compromise on taste, is the

water-in-oil-in-water double-emulsion. Microfluidics enabled scientists to finely control the conditions of double-emulsion drop formation either in one or two steps, depending on the technique used [6,82,85,51], whilst capillary co-flows were proven to have their chip-based equivalent [84]. Thanks to monodispersity, fine quantification of osmotic swelling kinetics provided evidence of a two-stage double-emulsion swelling process, allowing to improve the process control [51]. Membrane emulsification techniques were proven to be capable of reaching a high degree of encapsulation, in particular in the case of vibrated membrane [91].

The use of concentric multiple flow-focusing does not only apply to generate multiple emulsions [85,89], but also multiphase structures for a variety of purposes e.g. encapsulation and controlled delivery [89,90]. They were also used for controlling foam stabilizing structures: in one example, food coacervates of acacia gum and a milk protein, β -lactoglobulin, are brought to interact near an interface with air, under physico-chemical conditions favoring their electrostatic complexation. The result is a foam having a structure stabilized by coacervates forming thick lamellae separating bubbles, which can also be generated at rates relevant for industrial processing [55].

IV. Microfluidics based analytical tools

IV.1. Structure and property probing in food

In this paper, we stress that a major benefit from microfluidic techniques is certainly their use for analytical purposes, i.e. for quantifying well-defined material properties, and further for studying complex phenomena [58,101,102]. We recall the possibility of comparing the role and efficiency of various components, amongst which are stabilizers of emulsions and foams against coalescence [33,38], and Ostwald ripening [37]. Recently, the coverage mechanism of a single bubble by interfacially stabilizing particles under confined flow was investigated, revealing multiple regimes depending on particle size, flow rate, microchannel shape, as well as aggregation degree of surface-active particles [56]. A pressure-controlled chamber included in the device could be used to probe the rheological stabilization imparted by the particles collected at the bubble surface [35,37]. In its principle, the approach is similar to osmotic stress studies, which are important for biopolymer shell capsule dispersion [62], or double-emulsion stability [51]. Another device allowing application of a DC current at a microfluidic junction, was proven to be a method to characterize thin film stability at high capillary pressure [40]. Furthermore, we stress here the relevance of combining a microfluidic approach with AFM characterization of hydrodynamic and colloidal properties of drops and bubbles [78].

On-chip mechanical characterization also developed in several directions. Drop deformability and shape relaxation measurements allowed the development of on-chip interfacial tensiometry [63] using simple geometries, making it possible to access fast surfactant adsorption kinetics under flow [105] (fig.2a). Further developed geometries should be useful for even more controlled characterization, at a local stagnation point [106], or by varying the flow type [107] (fig.2b). In this spirit, proper on-chip quantification of single microcapsule interfacial elastic modulus evidenced the difficulty of producing biopolymer shells with uniform thickness and properties [57] (fig.2c). The possibility to characterize a large number of objects individually gave access to quantification of properties, which possess a high level of data scattering, e.g. the modulus of gel particles under formation [103], or the adhesive properties of hydrogel microcapsules [64]. On the other side, the study of single particle micromechanics also presents benefits, e.g. for revealing the restructuring of particle aggregates under controlled stress [65].

Figure 2. Diverse geometries for drop, particle and capsule mechanical studies. (a) Microfluidic geometry section used for interfacial tension characterization from drop deformability (from [63]). (b) Sketch of geometry of a microfluidic equivalent of a 4-roll mill (from [107]). (c) Image showing a T-junction geometry (left) and corresponding flow velocity field (in the absence of particle) used for capsule mechanics studies (from [16]).

Microfluidic tools permitted important insight into powder dissolution properties. Complex capillary flows develop during capillary suction of water into powders occurring simultaneously with powder dissolution [122]. Real understanding of the phenomena and building of models is possible only by the use of model capillary geometries, where the contributions to the pressure build-up as well as transient effects, in particular the important question of bubble trapping, can be studied at the local scale [66] as well as for collective effects [67,68]. This could be combined with further capabilities e.g. the miniaturization of calorimeters [69], to allow measurement of dissolution enthalpies on-chip, which are known to be important parameters for particle dissolution, in particular in link with the ratio of amorphous to crystalline mass [123]. On the other side, microfluidic tools can allow faster and less expensive experimental determination of spray-drying conditions before process upscaling, which are most suitable for optimizing particle properties [70]. Furthermore, extreme conditions of drying can be more easily controlled and explored, possibly leading to novel structures [59,60].

Perhaps a more complex part of on-chip studies address the interface with classical soft matter structure characterization tools. Advanced techniques were developed for the characterization of on-chip static or flowing materials, e.g. wide-angle [76] and small angle X-ray scattering [75,76,77], Raman imaging [75], or dynamic light scattering [74]. They allow studying transitions and building phase diagrams on-chip [75], which yet needs to be applied to food. Crystallization under finely controlled conditions within drops of controlled size, allows quantification of the nucleation probability and the growth kinetics [71,73], and measurement of the mutual diffusion coefficients of particle components during drying [72]. The use of the ensemble of techniques described in this section represents considerable acceleration and insight potential for engineering food structure for functionality. As for examples, such approaches were used to demonstrate the functionality of antifreeze protein to block ice crystal growth [129], or to evidence the potential of using controlled conditions of lipid emulsion crystallization for generating non-spherical fat particles [130].

IV.2. Molecular and biological sensing

Microfluidic methods were developed for molecular and biological sensing of a large variety of molecules and structures. Benefits targeted are wide, including primarily improved food safety and allergenicity, food nutrition, agricultural efficiency, and extending to biomedical diagnosis, generally allied with improved cost-efficiency by reducing the use of expensive reagents. Some well-known device examples for the rapid detection of unsafe elements in food include: food-borne pathogens e.g. the well-known bacteria *Escherichia coli* or *Salmonella* [108,109,110], antibiotics potentially present in particular in dairy food [58,111], toxic ions and in particular metal ions (Hg^{2+} , As^{3+} , and more), including nitrates [112]. Most used base techniques for the rapid on-chip detection of bacteria are combined dielectrophoresis and impedance analysis [110], and fluorescence based optical detection [109]. Chemiluminescence and absorbance also present benefits [114], as well for ion detection [112]. There are many reviews dedicated to the topic on-chip bacterial detection and immunoassays [110,113,118], even regarding the detection of specific pathogen types e.g. mycotoxins (e.g. from fungal source) [115], and we direct the interested readers to those. Note that mass spectrometry (MS) can be highly efficient for e.g. antibody or more generally protein detection, with the challenge of integration of the detection unit on chip [114] – an earlier step being the interfacing of MS with on-chip microfluidics [120]. Very high sensitivity was also achieved using on-chip magnetic bead-based assays [121]. Platforms were developed, which permit the anchoring of cells at well-defined locations on-chip, for subsequently stressing them with controlled chemical potential gradients [13].

Simplicity of use, accuracy and portability are important strengths of these on-chip analysis devices [110,113]. Consequently, the development of point-of-care devices for food quality analysis [116] slowly progresses toward a reality. Significant progress was made as well in the development of paper-based microfluidic testing [117], whereby current challenges include integration of detection units while achieving quantitative output analysis [119].

V. Conclusion: what's next for Food and Microfluidics ?

We have so far reviewed literature, which shows how microfluidic techniques have enriched the horizons of food science and engineering within only approximately a decade of use in the community. They allow accelerated research for finding design rules for food structure generation in wet conditions or from powders, whilst permitting to describe the phenomena complexity thanks to a controlled environment. This final section is an attempt to identify some bolder lines targeting specific scientific horizons, where opportunities may be richer for the next decade of research. This attempt is based on a simple analysis of overlap between research needs and multiple evolving technical capabilities.

A first, realistic and high potential use of Microfluidics, is expected to further develop from the improved characterization of local structure and complex phenomena during phase transitions. This is not limited to phase diagram studies, but promises in particular for investigating out-of-equilibrium phenomena and metastable structures, e.g. morphology development during drying [28] or re-wetting [27], even under reactive conditions. This should impact the design of spray-drying towers and conditions used therein, as well as powder dissolution conditions in machines.

A second direction of strong potential development is expected from the convergence between on-chip control of complex, reactive biological environments on one side, and on-chip capabilities for structural and rheological studies on another side. Certain types of studies, e.g. the digestion of food,

need a high degree of control of many parameter types, including those already cited above (flow and thermodynamic ones), but also further parameters e.g. the contact time between components [26,25]. Lipid digestion studies recently revealed most interesting features from dynamic X-ray analysis e.g. for the case of milk [104]. The access to a high degree of environment control using Microfluidics, both chemically and physically [14,15], should permit to generate higher quality data, but also open the way towards high throughput screening of digestion efficiency as a function of composition. A further, promising but challenging step, would be in the direction of linking digestion efficiency to bioavailability [17].

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