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# Virtualization of fluid-dynamics in micro-air assisted extruders for food microfluidic based encapsulation

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## ABSTRACT

Microfluidic technique represents an interesting technological solution for the production of alginate microbeads in pharmaceutical, food and cosmetics industry. Given the characteristic small size of microfluidic devices and the extremely complex dynamics which are at the base of transport phenomena involved in this process, a purely experimental approach is not able to provide all significant data to design cheap but trustable devices. In this work, a virtual approach – based on computational fluid dynamics analysis – has been proposed to analyze the behavior of a micro air-assisted extruder and to provide mechanistic insight into the particle formation dynamics. The particle formation was characterized by a complex periodic dynamics, during which elongations and instabilities brought to merging of liquid droplets, of different main size, into bigger drops.

Jet instabilities dominating the drops break-up dynamics were greatly influenced by geometric parameters: the fluid-dynamic analysis showed that small misalignment can dramatically change the jet breakup dynamics, introducing a completely different framework.

The analysis proposed in this work represents a powerful tool for the design of simple and cheap microfluidic devices, capable of mass production of small (around 300  $\mu\text{m}$  of diameter) and regular alginate microbeads, at least for matrix encapsulation.

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## 1. Introduction

The application of hydrocolloid gel particles is gaining momentum in several fields, including food, chemical, and pharmaceutical industries. Among several possibilities, alginate gel particles are widely used, being non-toxic, biocompatible, biodegradable, cheap, and relatively simple to produce, particularly appreciated as carrier for microencapsulated compounds. Usually, particle size and their shape are crucial for specific applications.

Alginate is often used in both matrix and drop-in-drop encapsulation, and industrial scale production of possibly monodispersed alginate beads with desirable characteristics such as controlled size and shape is a desirable target in food industry.

In order to design systems and develop methods able to produce microdroplets and microbeads holding the desired characteristics, both in terms of product and process, a suitable microfluidic system must be adopted. Its main feature, beside scalability, ease of

operation, ability to keep the production rate while ensuring sterility and avoiding contamination by the solvent, is the rapid, reproducible and controlled formation of uniform droplets. Microfluidics plays an interesting role in micro-beads production: different approaches have been tried to work quite well on a laboratory scale with fluid mixtures of relatively simple rheology. Nonetheless the transfer of such technology to the industrial scale is often challenging, as several additional characteristics are required: devices should be easy to build, possibly arranged in parallel arrays for mass production with minimal interference length between the single devices, the separation between extrusion fluid and micro-beads should be simple, and possibly cheap (Sarghini, 2015).

Several mechanical and chemical methods had been introduced to produce particles with narrow size distribution in a single step approach, (e.g. milling, oil-water and water-oil emulsions, coacervation, spinning disks, atomization, vibrating nozzles) (Schneider et al., 2011; Prüsse et al., 2008).

Among the proposed methods, extrusion-dripping has been well recognized as relatively simple, size-controlled, low-cost, and

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**Nomenclature**

$c_p$	Specific heat, J/kg-K
$C$	Alginate volume fraction
$D_i$	Internal diameter of the microfluidic device, mm
$D_e$	External diameter of the microfluidic device, mm
$D_{4/3}$	Volume mean diameter, mm
$\mathbf{I}$	Unit tensor
$k$	Ostwald–de Waele flow consistency index, Pa s <sup>n</sup>
$\kappa$	Thermal conductivity, W/(m K)
$n$	Ostwald–de Waele flow behavior index
$p$	Static pressure, Pa
$t$	Time, s
$T$	Temperature, K
$\mathbf{u}$	Velocity vector, m/s
$Re_l$	Reynolds number of liquid phase (DP)
$Re_g$	Reynolds number of continuous gas phase (CP)
$We$	Weber number of liquid phase
$\bar{(\ )}$	Alginate volume fraction averaged properties
<i>Greek letters</i>	
$\sigma$	Cauchy stress tensor, Pa/m <sup>2</sup>
$\mu_{eff}$	Effective dynamic viscosity, Pa s
$\rho$	Density, kg/m <sup>3</sup>

scalable formation method for microgels (Burey et al., 2009). In this work the concept of extrusion has been used in a wider sense, referred to a pressure driven drop shaping at the end of a capillary. In the extrusion-dripping, a biopolymer solution is forced through a nozzle (or a needle) in the form of droplets into hardening solution, where droplets gelation is induced in the hardening solution as a result of crosslinking agents (e.g., mineral ions, glutar-aldehyde, or enzymes, Chan et al., 2009). Gelation can be alternatively induced by applying a temperature change (heating or cooling) in case of thermally setting biopolymers, such as gelatin and whey proteins, or complexation with another polymer (e.g., polysaccharides) may occur (Murakami and Takashima, 2003).

Extrusion-dripping method has been used as well to produce microgel particles of biopolymers such as k-carrageenan injected into a potassium chloride solution (ionic gelation) (Perrechil et al., 2011), pectin injected into a calcium rich solution (ionic gelation), chitosan injected into a tripolyphosphate solution (ionic gelation), whey protein injected into a hot liquid (heat-set gelation), and gelatin injected into a cold liquid (cold-set gelation).

If production of large microgels, characterized by diameters greater than 1 mm, can be performed by simple gravity extrusion dripping method (in which a pendant droplet is allowed to grow until it reaches the maximum size that can be supported by surface tension forces at the dripping tip, whereupon the droplet detaches and turns into a microgel particle when it falls into the gelation solution), in order to decrease the microgel size, the droplet break-up must be assisted by external forces, such as the ones developed by pressurized air, electrostatic charges, vibration, rotation, or shear flow of two fluids.

Pressurized air have been widely investigated in alginate microgels, with the setup being less complicated, handling higher viscosity solutions. A typical air extrusion system for alginate microgel formation consists of a pressurized air or gas supply source as a continuous phase CP, an extrusion nozzle, and a gelation bath. In these systems, needle diameter, feed solution viscosity, and the flow rate and properties of the gelling fluid, as the dispersed

phase (DP), determine the microgel particles size (Joye and McClements, 2014).

Microfluidic devices play an interesting role in micro-beads production due to their relatively cheap technology: several of them based on different approaches had been proposed and they work quite well on a laboratory scale with fluid mixtures of relatively simple rheology and well known properties. Nonetheless the transfer of such technologies to the industrial scale often represents a powerful challenge, as several additional characteristics are required: devices should be easy to build, possibly arranged in parallel arrays for mass production with minimal interference length between the single devices, the separation between extrusion fluid and micro-beads should be simple, and possibly cheap (Sarghini, 2015).

Flow focusing is a microfluidic technique where two or more phases of liquid or gases are co-axially focused and then forced through a small orifice. The flow rate of the CP, usually exceeds that of the inner DP, typically by ten to thousand times depending on the fluid characteristics. The DP is thus forced into a narrow jet and obliged by CP confinement to flow at the orifice. Due to the rapid change in pressure chamber to the outlet and the prevailing effects of shear stress, fluid dynamics instabilities in several forms appear, and the jet breaks up into droplets after passing the orifice (Schneider et al., 2011).

Among the available microfluidic device geometries, the flow-focusing type has been commonly used to make monodisperse polymer particles, both spherical and non-spherical. One of the most important factors influencing droplet formation in such microfluidic devices is the confinement as the propagating thread is restricted within the microchannels and is affected by both the geometry and size of the channel (van Steijn et al., 2009). The various confinements let microfluidic device, the cross junction geometry and the flow-focusing junction.

A possible flow focusing configuration, requiring oil as CP, allows a very good control of geometrical properties of beads in the extruding phase, although it involves complicate production processes, as it introduces a barrier to the interaction of alginate solution with the calcium ions solution during external gelation phase and requiring a-posteriori filtering and washing procedure to recover the beads. Moreover, during gelation phase, chain agglomerations of micro-beads can appear (Haeberle et al., 2008).

The possibility of using a gas as CP introduces several advantages if compared with liquid-liquid focusing configuration: easier separations of the different phases, cheaper production and the possibility of using three dimensional arrays for mass production with minimal interference distance. On the other hand, the reduced shear stress at the phase interface and different inertial properties of the gas-liquid mixture requires relative high gas velocities involving gas flow instabilities (Si et al., 2009) and possibly turbulence, with a more difficult control of drop properties.

In such approach, the pressure gradient induced by an outer gas stream is used to confine and ‘focus’ a steady liquid jet of the dispersed phase. When the liquid microjet and the co-flowing gas stream cross a discharge outlet whose diameter is much larger than that of the microjet, a jet instability is generated, inducing DP flow break-up and allowing mass production of ultrafine and almost monodispersed spray, microcapsule or microspheres (beads) (Acero et al., 2012).

This technique was used for active compounds microencapsulation (Gundabala et al., 2013) using a configuration in co-focusing flow (gas-liquid) in which viscous (Cohen et al., 2001; Blanchette and Zhang, 2009) and/or pressure (Gañán-Calvo, 1998) forces stretch an interface until small DP jet is emitted.

One of the first application of air-liquid extrusion for alginate micro-beads production was introduced by Hardikar et al. (1999),

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