



Microfluidics techniques to design encapsulated ingredients

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Naples, October 11, 2012



FOOD MICROSTRUCTURING

- The design of novel food micro-structures aimed at the quality, health and pleasure markets will probably require unit operations where the scale of the forming device is closer to the size of the structural elements (i.e., 1–100 μm).
- One particular technique to provide bioavailability of nutraceutical or controlled release of active principles is encapsulation



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Journal of Food Engineering 67 (2005) 3–11

JOURNAL OF
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Why food microstructure?

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Received 10 October 2003; accepted 1 May 2004



MICROENCAPSULATION

➤ Encapsulation is the packaging of small particles of solid, liquid or gas, also known as the core, within a secondary material, also known as the shell or coating, to form small capsules.

➤ Microcapsules are usually classified in 

- Nanocapsules (less than 100 nm)
- Microcapsules (in the order of microns)

➤ In the past encapsulation was used to mask the unpleasant taste of certain ingredients and also to simply convert liquids to solids.



MICROENCAPSULATION

- All three states of matter (solids, liquids, and gases) may be microencapsulated. This allows liquid and gas phase materials to be handled more easily as solids.
- Microencapsulation may be achieved by a myriad of techniques, with several purposes in mind.
- Substances may be microencapsulated with the intention that the core material be confined within capsule walls for a specific period of time.
- Alternatively, core materials may be encapsulated so that the core material will be released either gradually through the capsule walls, known as controlled release or diffusion, or when external conditions trigger the capsule walls to rupture, melt, or dissolve.



MICROENCAPSULATION

- Ingredients in foods are encapsulated for several reasons.
- Historically, the most important application was flavoring.
- Most flavorings are volatile; therefore encapsulation of these components extends the shelf-life of products by retaining within the food flavors that would otherwise evaporate out and be lost.
- Some ingredients are encapsulated to mask taste, such as nutrients added to fortify a product without compromising the product's intended taste.



MICROENCAPSULATION

- Alternatively, flavors are sometimes encapsulated to last longer, as in chewing gum.
- The amount of encapsulated flavoring required is substantially less than liquid flavoring, as liquid flavoring is lost and not recovered during chewing.
- Flavorings that are comprised of two reactive components that, when encapsulated individually, may be added to the finished product separately so that they do not react and lose flavor potential prematurely.
- Some flavorings must also be protected from oxidation or other reactions caused by exposure to light.

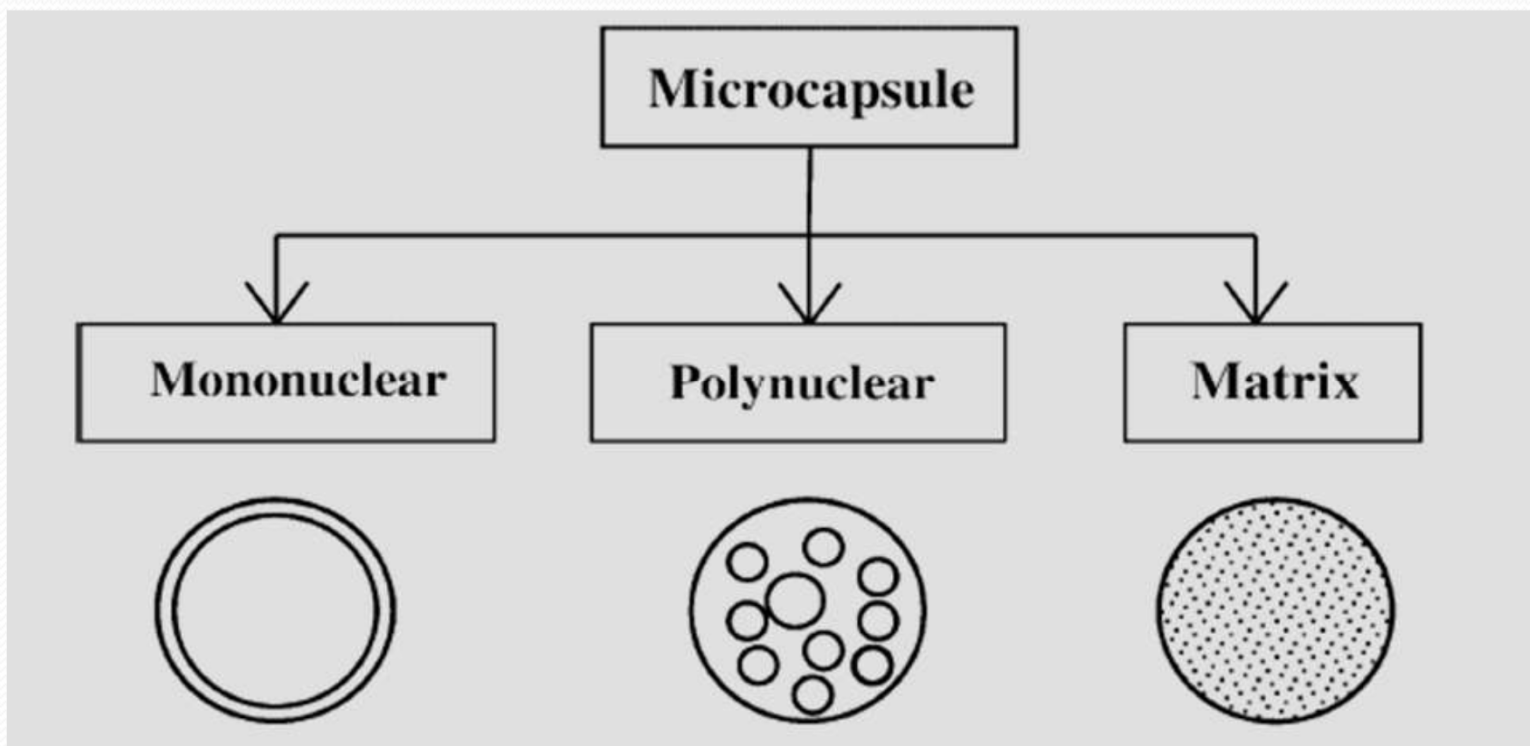


MICROENCAPSULATION

- **Morphology of Microcapsules:** the morphology of microcapsules depends mainly on the core material and the deposition process of the shell.
- 1- **Mononuclear** (core-shell) microcapsules contain the shell around the core.
- 2- **Polynuclear** capsules have many cores enclosed within the shell.
- 3- **Matrix encapsulation** in which the core material is distributed homogeneously into the shell material.
- In addition to these three basic morphologies, microcapsules can also be mononuclear with multiple shells, or they may form clusters of microcapsules.

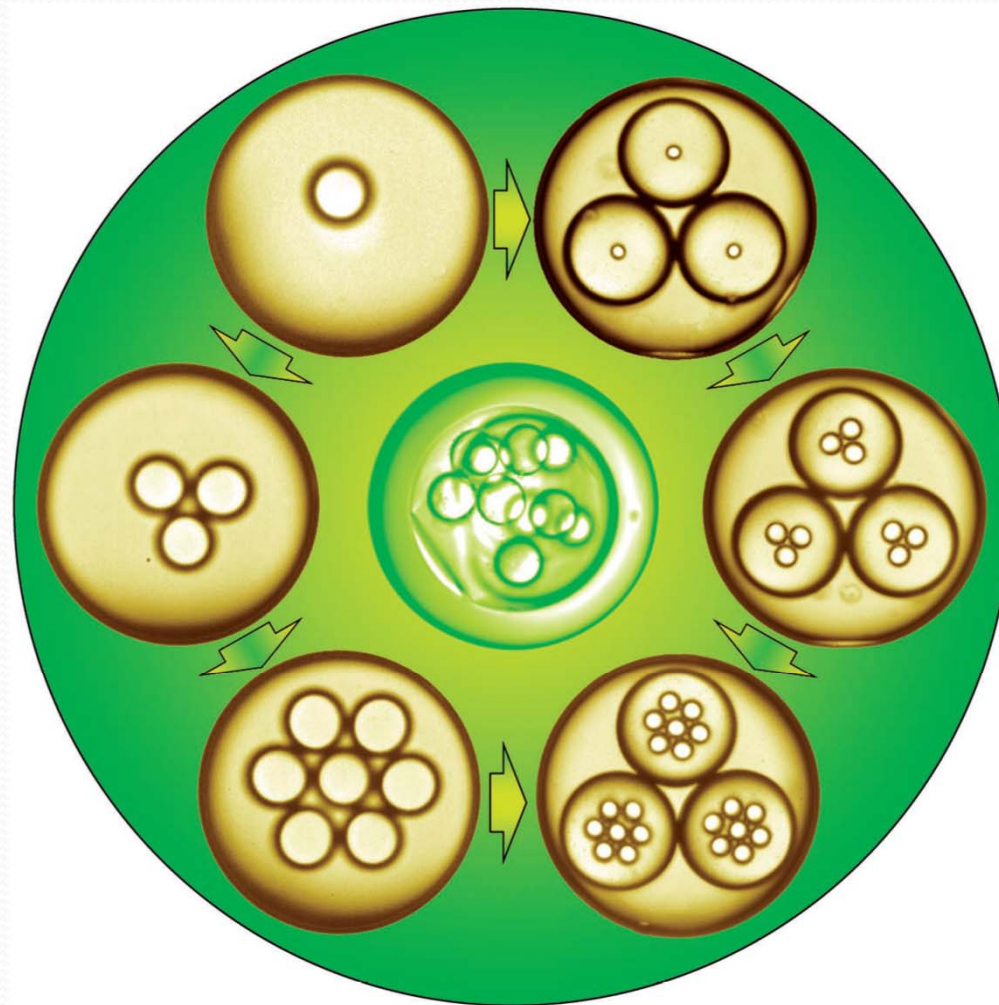


MICROENCAPSULATION





MICROENCAPSULATION





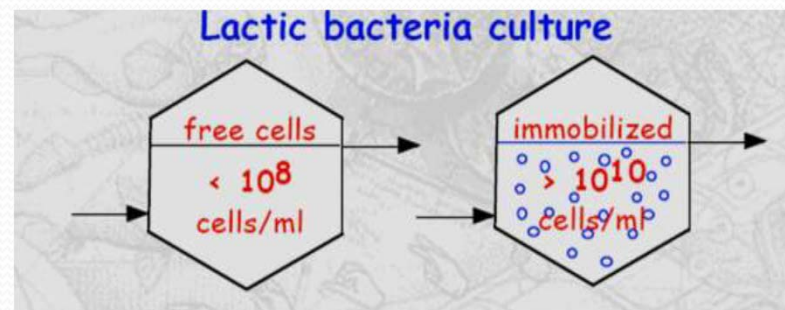
BENEFIT OF MICROINCAPSULATION

1- Microorganism and enzyme immobilization.

Enzymes have been encapsulated in cheeses to accelerate ripening and flavor development.

The encapsulated enzymes are protected from low pH and high ionic strength in the cheese.

The encapsulation of microorganisms has been used to improve stability of starter cultures.





BENEFIT OF MICROINCAPSULATION

2-Protection against UV, heat, oxidation, acids, bases

(e.g. colorants and vitamins). e.g. Vitamin A / monosodium glutamate appearance (white) protection (water, T, light)

3- Improved shelf life due to preventing degradative reactions

(dehydration, oxidation).

4-Masking of taste or odours.

5- Improved processing, texture and less wastage of ingredients.

- Control of hygroscopy
- enhance flowability and dispersibility
- dust free powder
- enhance solubility



BENEFIT OF MICROENCAPSULATION

6 - Handling liquids as solids

7 – Growing demand for nutritious foods for children which provides them with much needed vitamins and minerals during the growing age. Microencapsulation could deliver the much needed ingredients in children friendly and tasty way.

8 - Enhance visual aspect and marketing concept.



BENEFIT OF MICROENCAPSULATION

9 – Farmaceutical controlled and targetted release of active ingredients.

Many varieties of both oral and injected pharmaceutical formulations are microencapsulated to release over longer periods of time or at certain locations in the body.

Aspirin, for example, can cause peptic ulcers and bleeding if doses are introduced all at once. Therefore aspirin tablets are often produced by compressing quantities of microcapsules that will gradually release the aspirin through their shells, decreasing risk of stomach damage.

10- Microencapsulation allows mixing of incompatible compounds.



COATING MATERIAL

Coating material properties:

- ✓•Stabilization of core material.
- ✓•Inert toward active ingredients.
- ✓•Controlled release under specific conditions.
- ✓•Film-forming, pliable, tasteless, stable.
- ✓•Non-hygroscopic, no high viscosity, economical.
- ✓•Soluble in an aqueous media or solvent, or melting.
- ✓•The coating can be flexible, brittle, hard, thin etc.



COATING MATERIAL

Coating materials:

- ✓ **Gums:** Gum arabic, sodium alginate, carageenan.
- ✓ **Carbohydrates:** Starch, dextran, sucrose
- ✓ **Celluloses:** Carboxymethylcellulose, methycellulose.
- ✓ **Lipids:** Bees wax, stearic acid, phospholipids.
- ✓ **Proteins:** Gelatin, albumin.



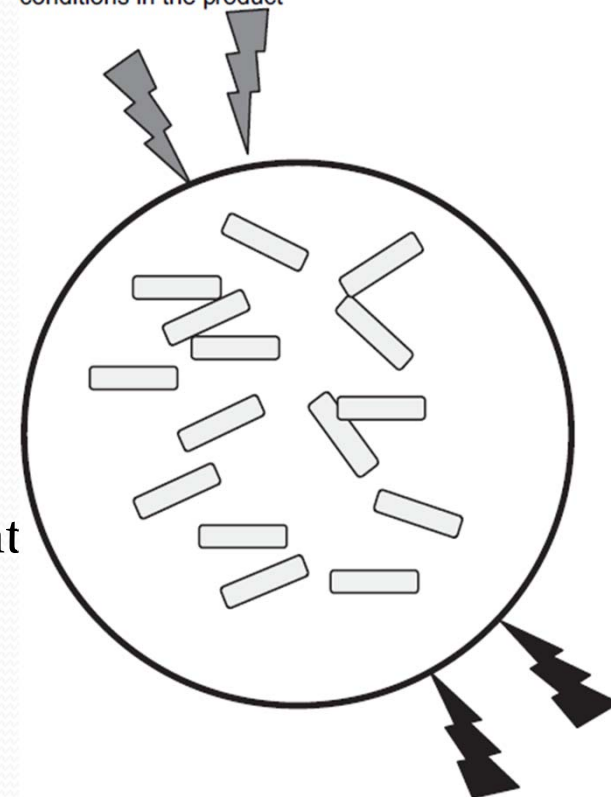
RULE OF THE SHELL

Protection of the active ingredients against:

- ✓ pH
- ✓ Oxygen
- ✓ Osmotic Pressure
- ✓ High temperature
- ✓ Shear stress
- ✓ Enzymatic activity

- ✓ Improved handling of the active ingredients
- ✓ Possibility to introduce hydrophilic ingredient in hydrophobic food matrix and vice versa

Protection against adverse conditions in the product

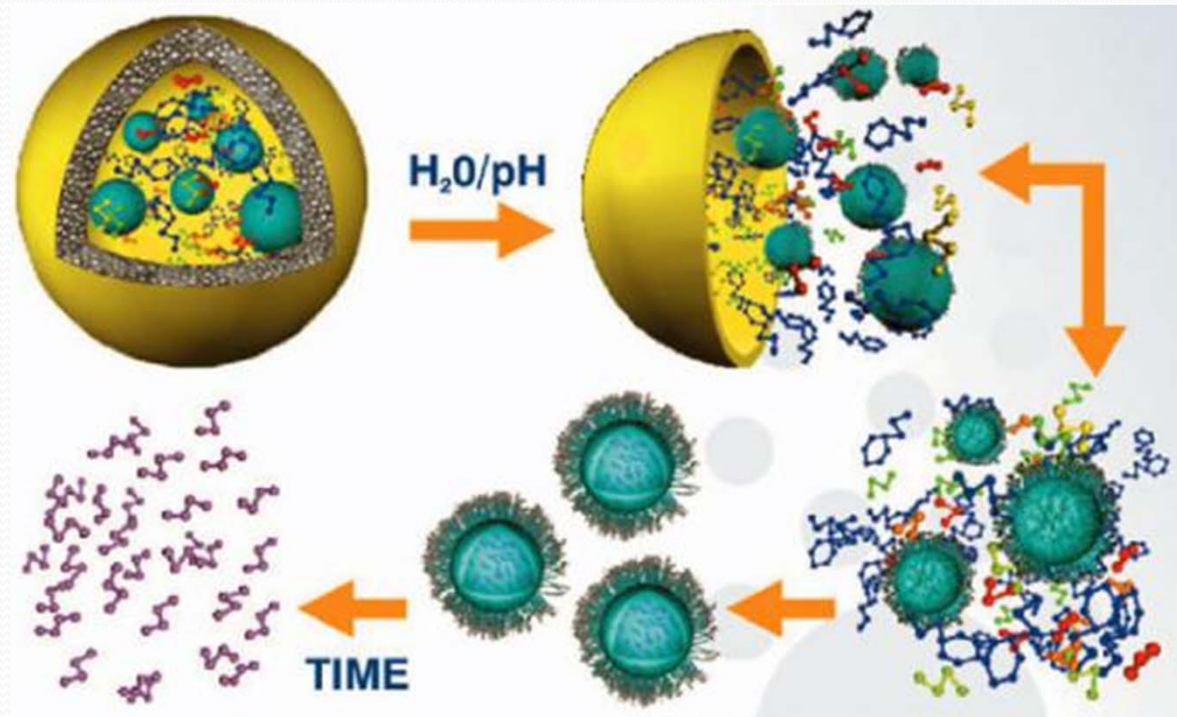


Protection against deleterious conditions in the upper gastrointestinal tract



DIFFERENT SHELL, DIFFERENT BEHAVIOR

➤ Control over the release kinetic.

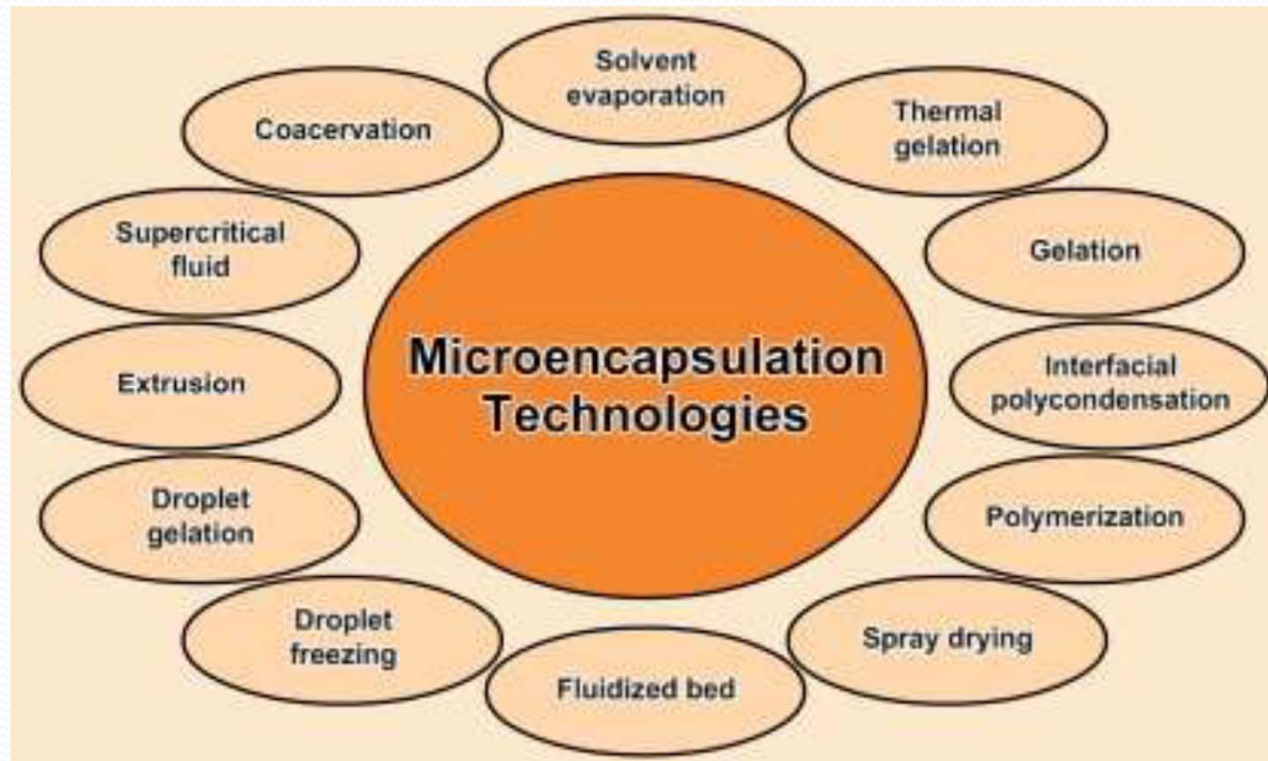


As results:

- ✓ Improved Shelf life of the active ingredient
- ✓ Increased bioavailability



MAIN TECHNOLOGIES



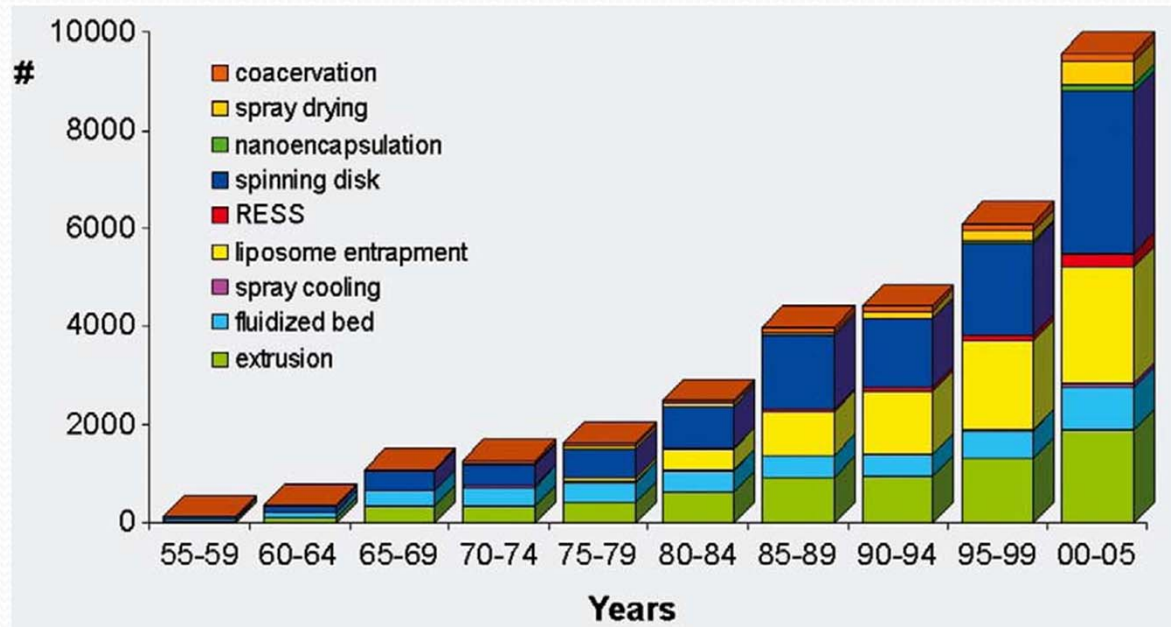


MAIN TECHNOLOGIES

Physico - Chemical Processes	Physico - mechanical Processes
Coacervation (2 – 1200 μm)	Spray-drying (5 – 5000 μm)
Polymer-polymer incompatibility (0.5 – 1000 μm)	Fluidized- bed technology (20 – 1500 μm)
Solvent evaporation (0.5 – 1000 μm)	Pan coating (600 – 5000 μm)
Encapsulation by supercritical fluid	Spinning disc (5 – 1500 μm)
Encapsulation by Polyelectrolyte multilayer (0.02 – 20 μm)	Co-extrusion (250 – 2500 μm)

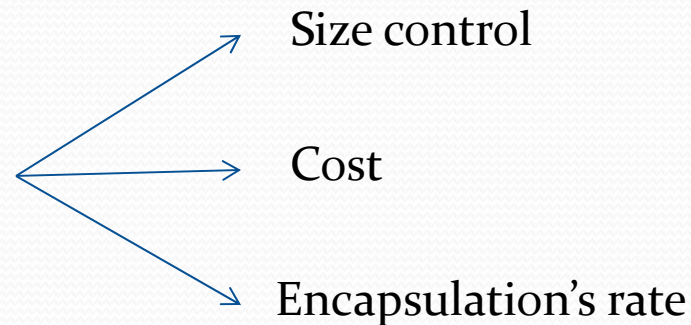


MAIN TECHNOLOGIES



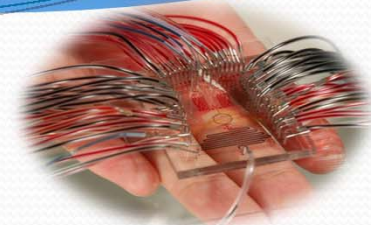
Increased number of encapsulation's research every year in all fields.

Limits of traditional technologies





MICROFLUIDICS



Microfluidics —**the science of designing, manufacturing, and operating devices and processes that deal with small amounts of fluids (10^{-6} to 10^{-9} l)**—has the potential to significantly change the way of processing dispersed food systems.

Microfluidic devices can be identified by the fact that they have channels with at least one dimension smaller than 1 mm. The devices themselves have dimensions ranging from millimeters down to micrometers.

Food Biophysics (2008) 3:1–15
DOI 10.1007/s11483-007-9043-6

REVIEW

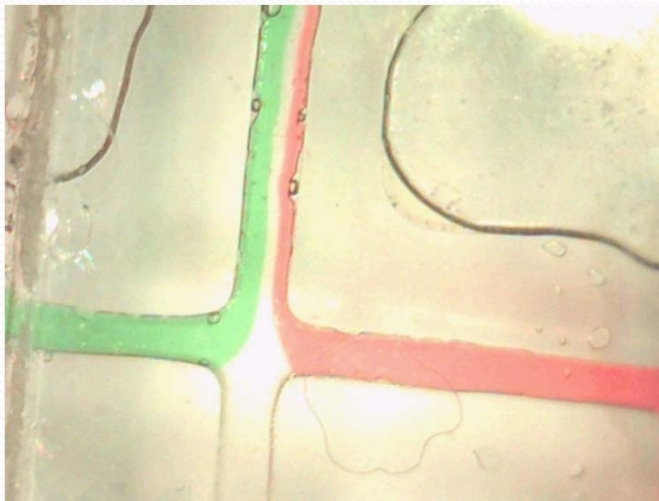
Applications of Microfluidic Devices in Food Engineering

O. Skurtys • J. M. Aguilera

Received: 27 April 2007 / Accepted: 28 August 2007 / Published online: 26 September 2007
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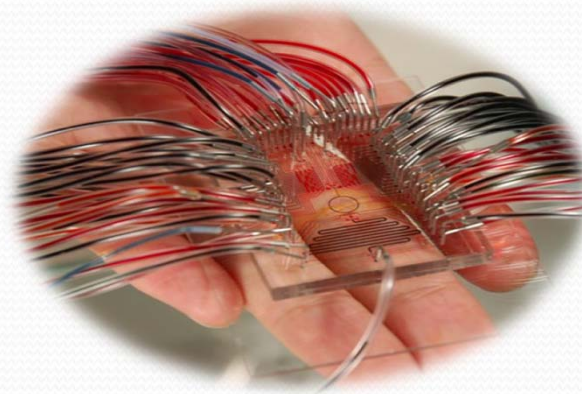
MICROFLUIDICS BASES



- Low Reynold's number \rightarrow laminar flow
- Viscous forces overwhelm inertial forces
- No mixing in microchannel;

Main features:

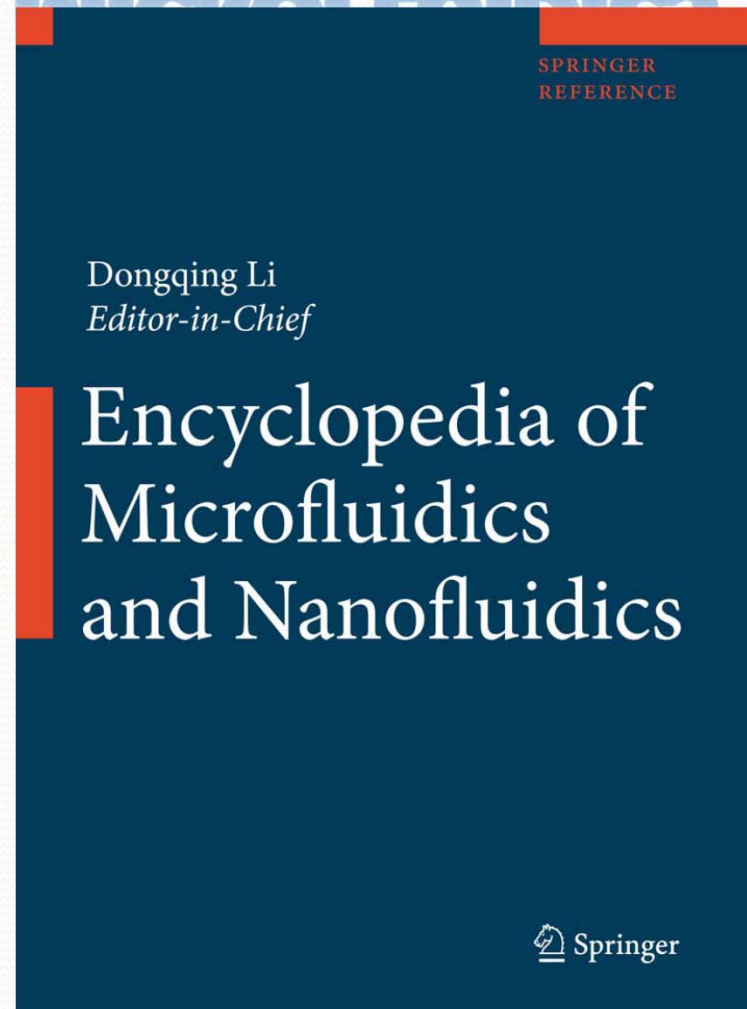
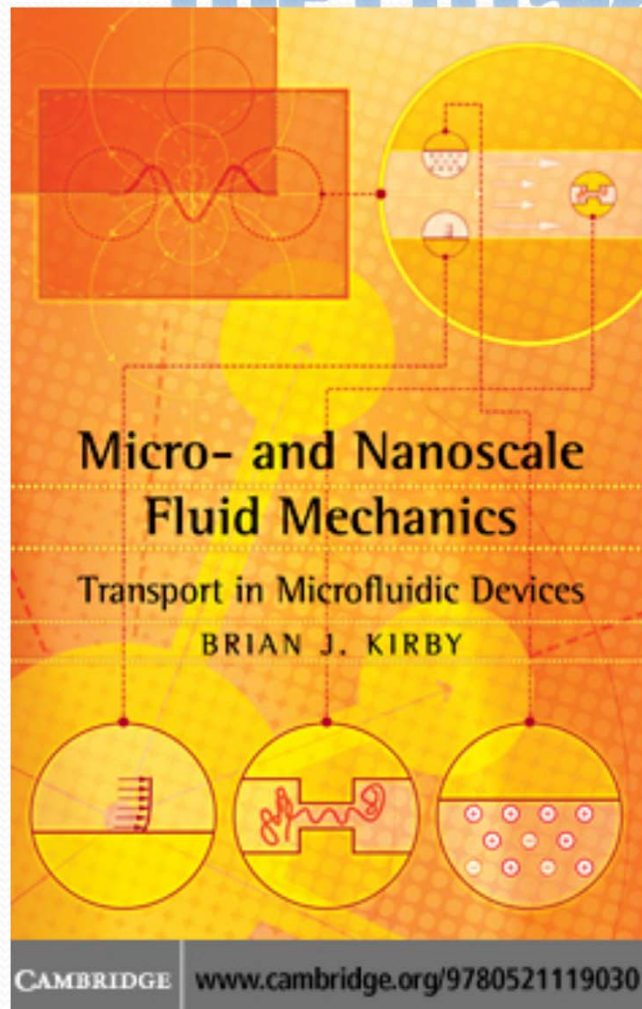
- ✓ Small device
- ✓ Small volume used
- ✓ Cheap device



➤ The scaling-up's question



THE PHYSICS OF MICROFLUIDICS





THE PHYSICS OF MICROFLUIDICS

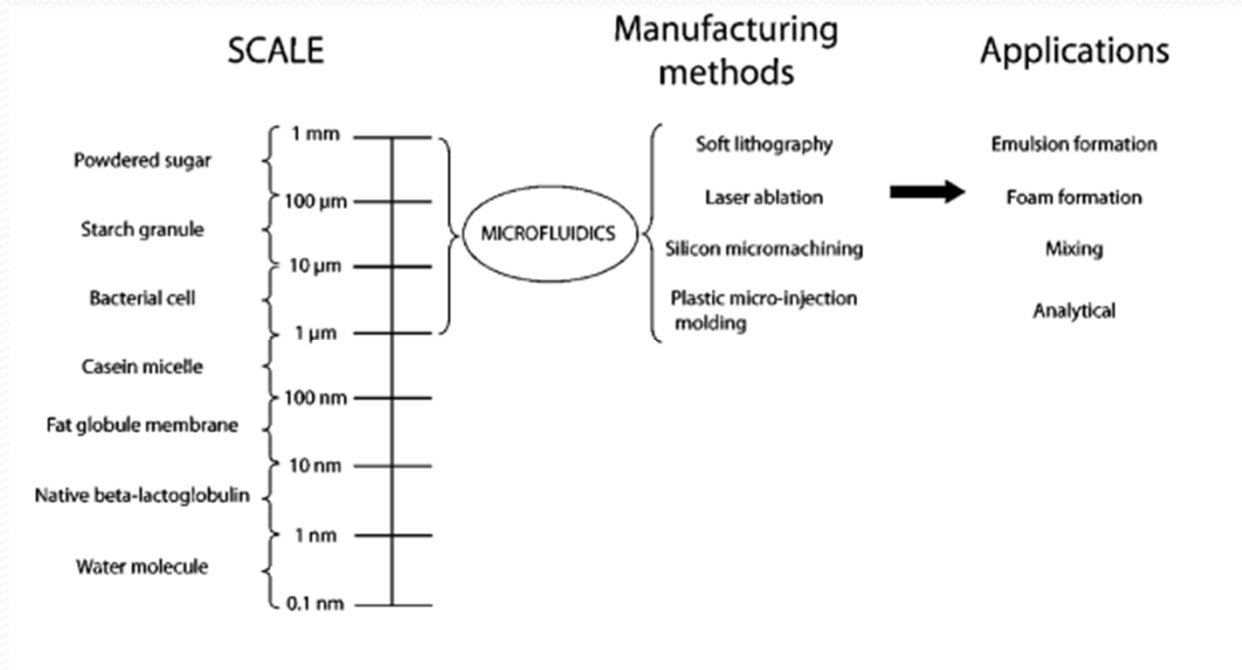
The behaviour of dispersed phases, either gas–liquid (foams) or liquid–liquid (emulsions), common in many macroscopic food systems is relatively well understood.

At levels below the micrometer scale, some effects negligible at the macroscopic level become important; for example, those related to **surface tension, energy dissipation and fluidic resistance**.

Moreover, different from the macro-scale, a special attention must be paid to the wetting phenomena of the fluid on the substrate.

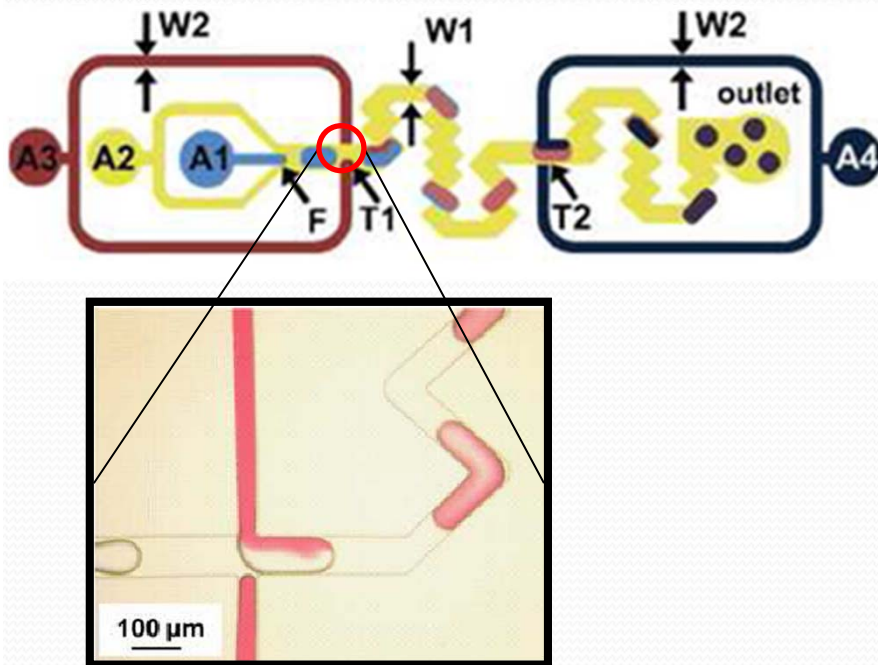


THE PHYSICS OF MICROFLUIDICS



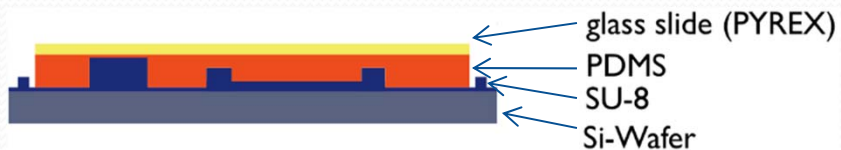


MICROFLUIDICS ENCAPSULATION PROCESS



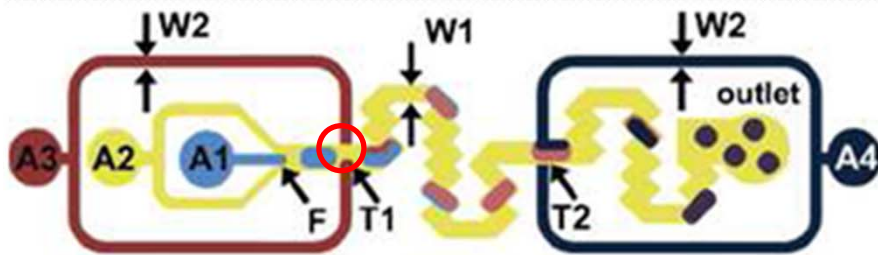
- Alginate
- Droplet formation depends by flow rates.
- Rule of the Ca^{++}

➤ Soft-litography technique

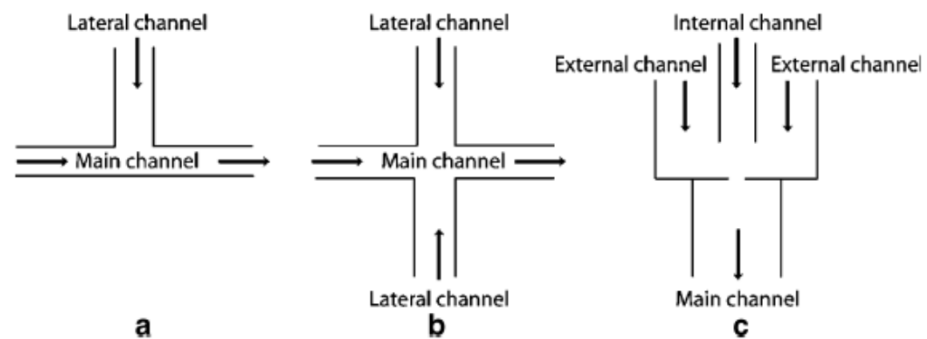




MICROFLUIDICS ENCAPSULATION PROCESS



- T-Junction and Cross Junction





MICROFLUIDICS ENCAPSULATION PROCESS

- T-Junction and Cross Junction movies



MICROFLUIDICS ENCAPSULATION PROCESS

➤ Soft-lithography techniques



The concepts of soft lithography have been developed by Whitesides et al. at Harvard. Soft lithography is so called because it utilizes cast-moulded stamps made from flexible materials.

- 1)The process begins with the creation of a master.
- 2)The master is made by etching a blank—normally a silicon wafer—with a negative photoresist. This gives a raised pattern of nanometer-sized features on the silicon wafer that corresponds with the required channels in the polymer stamp.
- 3)A liquid polymer is then poured on top of the silicon wafer mould. The polymer usually used is the transparent elastomeric PDMS.
- 4)The polymer is heat cured and peeled off the mould.



MICROFLUIDICS ENCAPSULATION PROCESS

➤ Soft-lithography techniques



Annu. Rev. Mater. Sci. 1998. 28:153–84
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SOFT LITHOGRAPHY

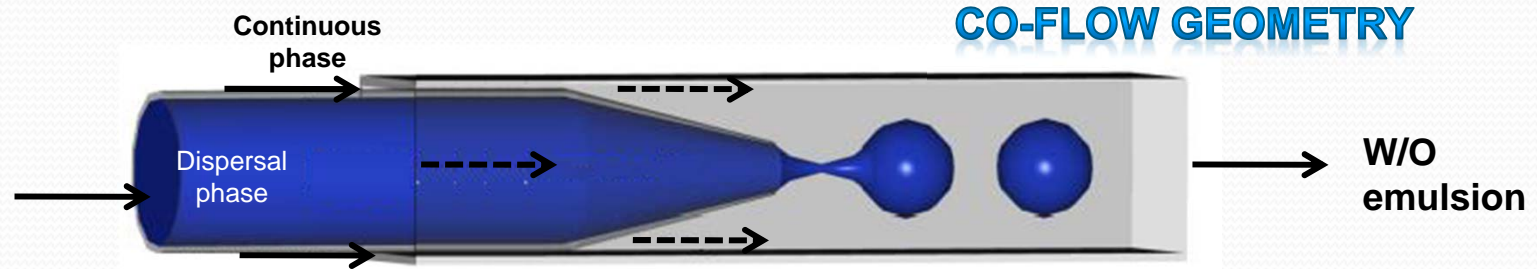
Younan Xia and George M. Whitesides

Department of Chemistry and Chemical Biology, Harvard University, Cambridge,
Massachusetts 02138; e-mail: gwhitesides@gmwgroup.harvard.edu

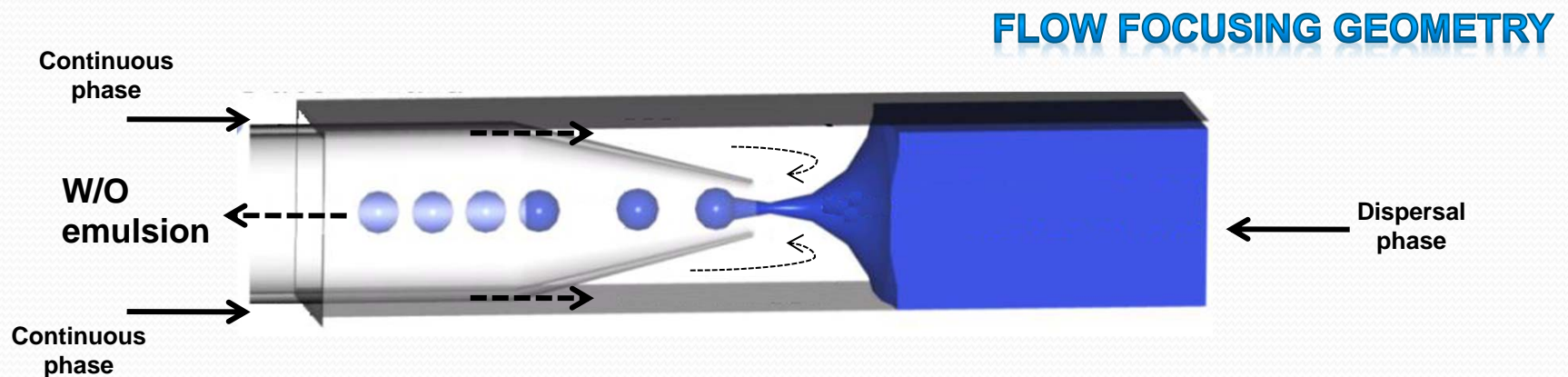
KEY WORDS: patterning, microfabrication, nanofabrication, elastomers, self-assembled monolayers

Microfluidic devices for focusing and cofocusing flows

Capillary microfluidics presents a way to controllably generate drops of one liquid in another immiscible liquid in devices that consist of coaxial assemblies of glass capillaries.



Schematic of co-flow microcapillary devices for making emulsion droplets



Schematic of flow focusing microcapillary devices for making emulsion droplets

Dripping and jetting condition in microfluidic devices

- In the co-flow geometry when the fluids flow at low rates, individual mono-disperse drops are formed periodically at the tip of the capillary orifice, in a process termed dripping.



Image of drop formation at low flow rates (dripping regime)

- If we increase the flow rate of either fluid beyond a certain critical limit, the result is a jet, a long stream of the inner fluid with drops forming downstream.

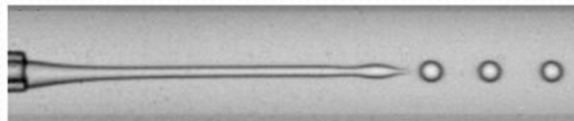


Image of a narrowing jet generated by increasing the flow rate of the continuous fluid above a threshold value, while keeping the flow rate of the dispersed phase constant

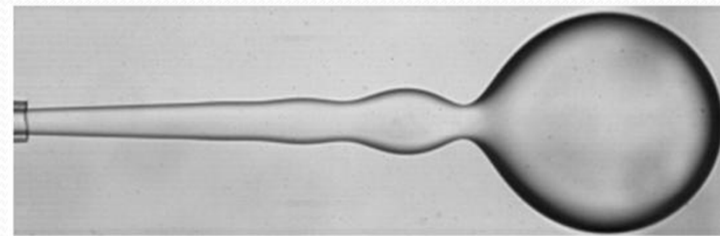
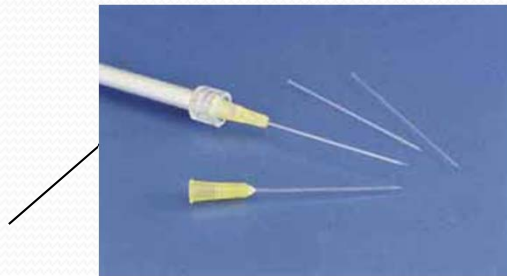


Image of a widening jet generated by increasing the flow rate of the dispersed fluid above a threshold value, while keeping the flow rate of the continuous phase constant.

Siliconizing glass microcapillary

One of the inherent advantages of these devices is that their wettability can be easily and precisely controlled by a surface reaction with an appropriate surface modifier



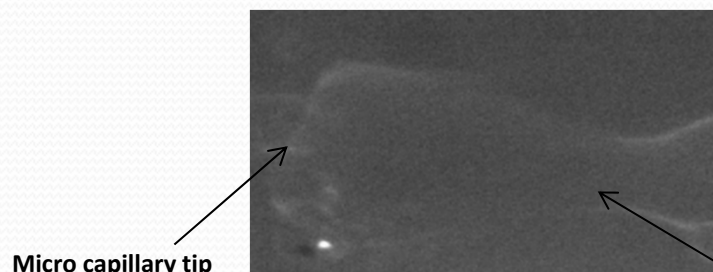
Micro capillary pulled (ID = 80 μm)



microcapillary glass are gently placed in a silicizing solution (dimethyldichlorosilane in chloroform)



Microcapillary are left dry overnight and transferred in oven a 60-80 $^{\circ}\text{C}$

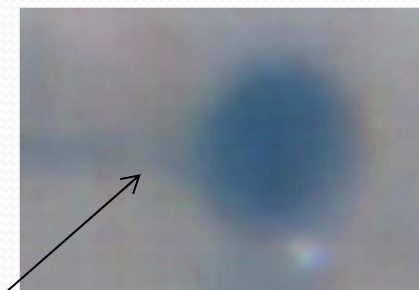


Micro capillary tip (ID 80 μm)

Incorrect drop formation at the tip of the inner capillary glass tube

Incorrect Break up and droplet formation

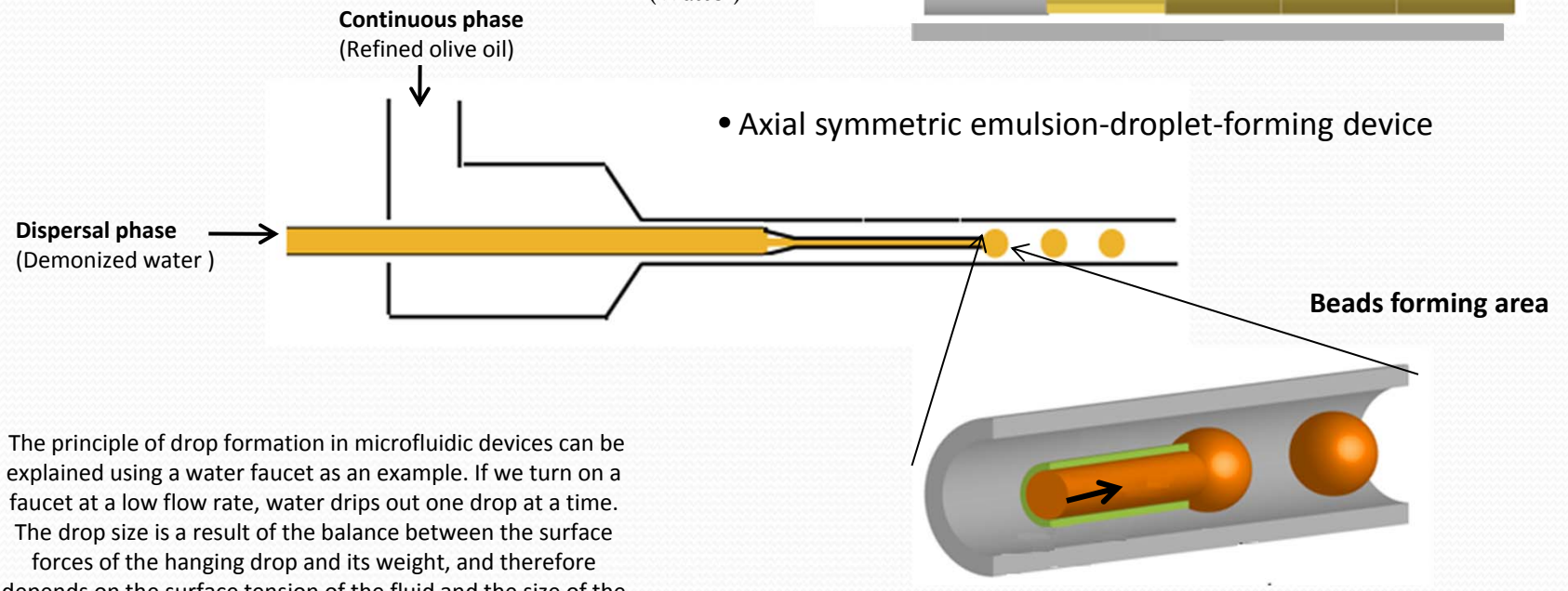
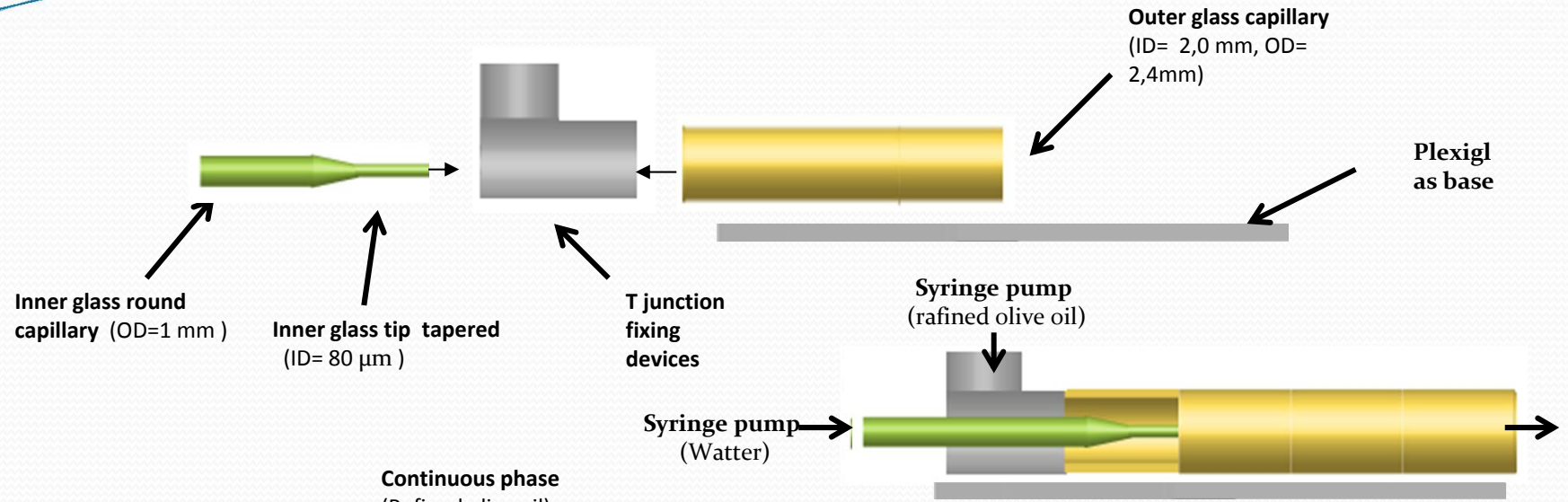
Siliconizing



Micro capillary tip (ID 80 μm)

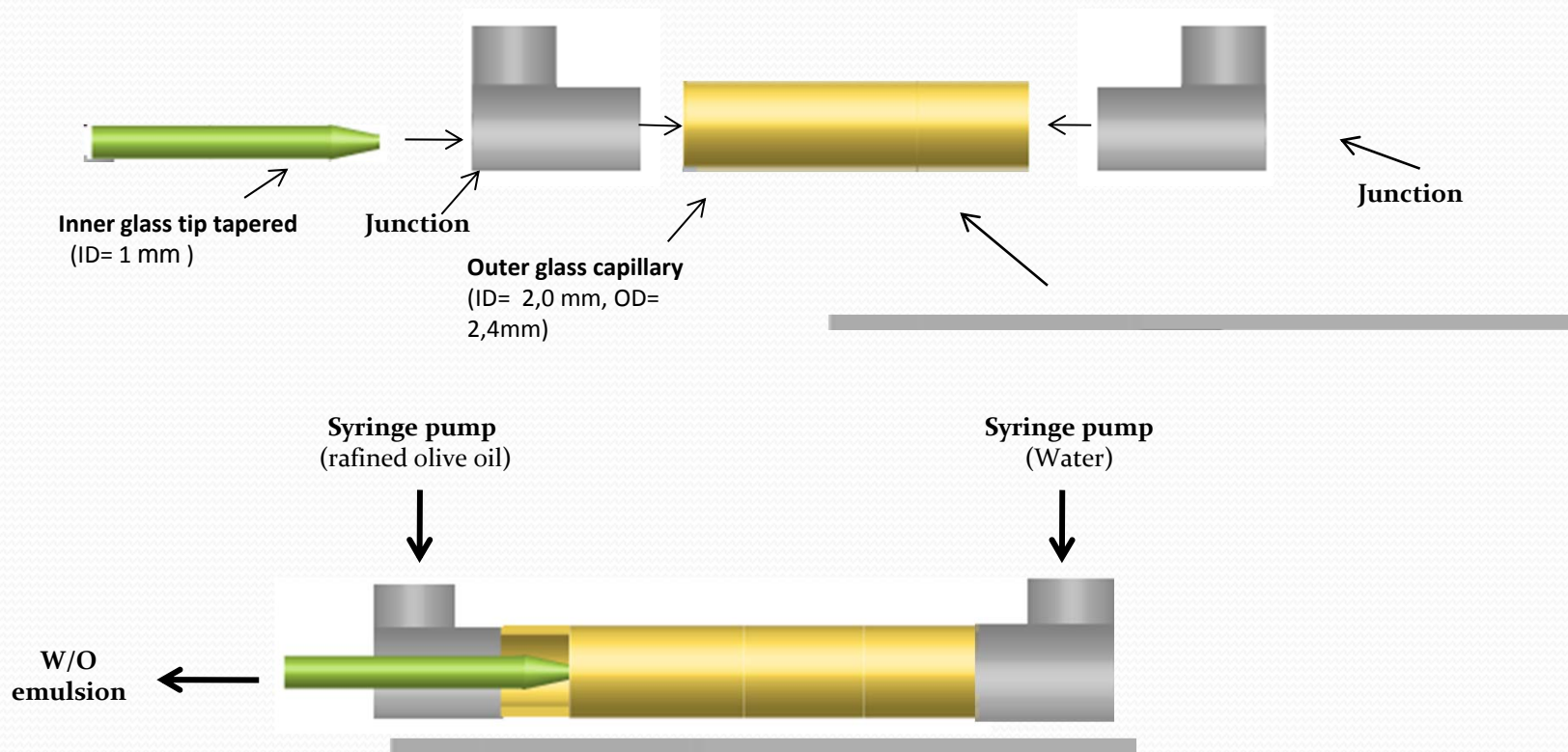
Correct drop formation after silanizing treatment

Design and Fabrication



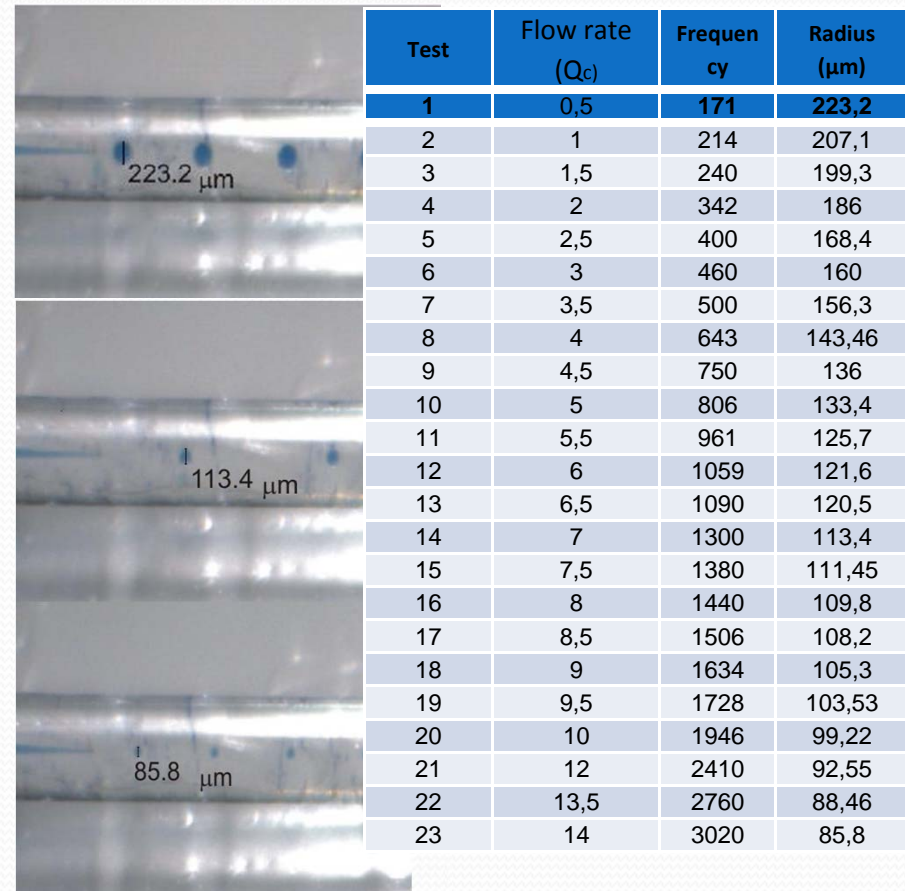
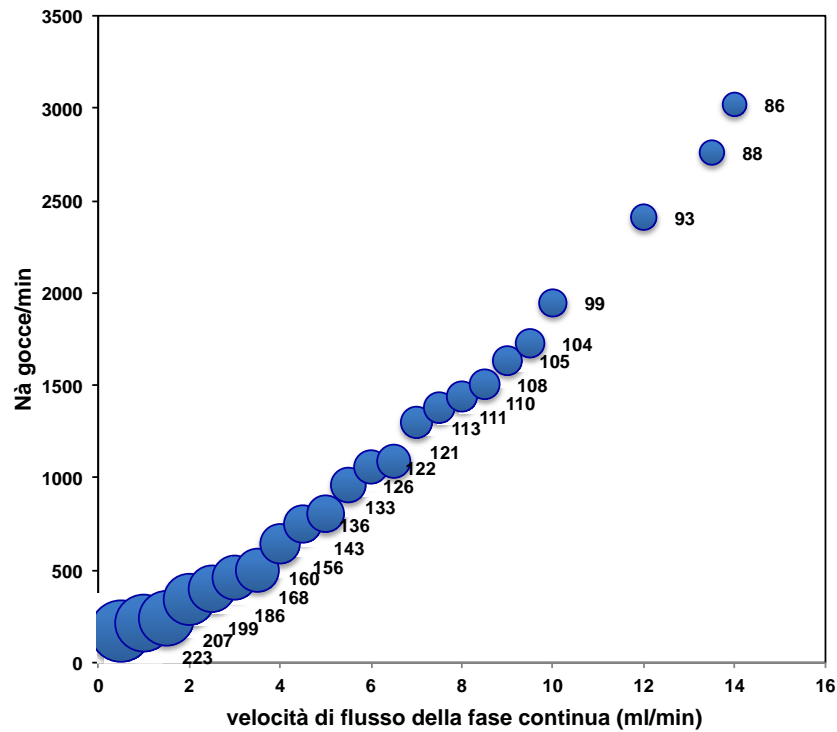
The principle of drop formation in microfluidic devices can be explained using a water faucet as an example. If we turn on a faucet at a low flow rate, water drips out one drop at a time. The drop size is a result of the balance between the surface forces of the hanging drop and its weight, and therefore depends on the surface tension of the fluid and the size of the faucet.

Design and Fabrication

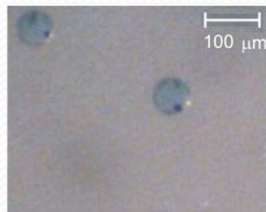


EMULSIONS FORMATION IN A CO-FLOW MICROFLUIDIC DEVICES

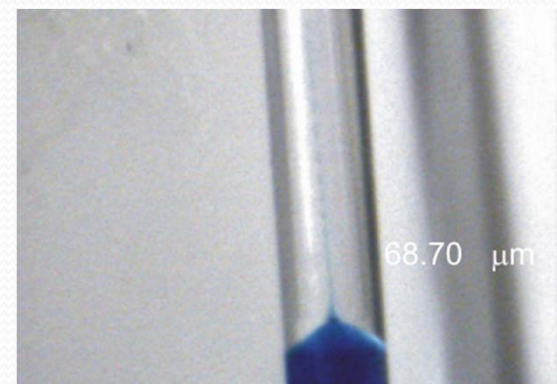
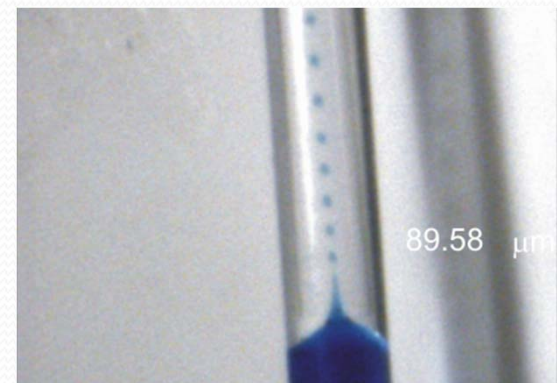
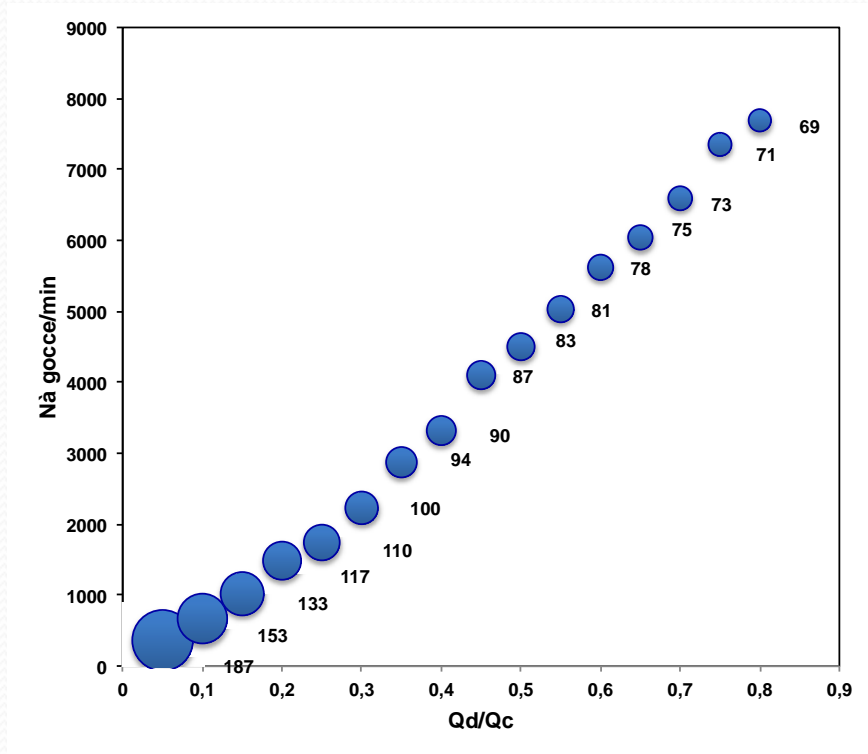
Established the conditions that allow the dripping. It's possible by varying the flow rate of the continuous phase observe drop formation of different size



Mono-disperse droplets formed using a co-flow microcapillary device.

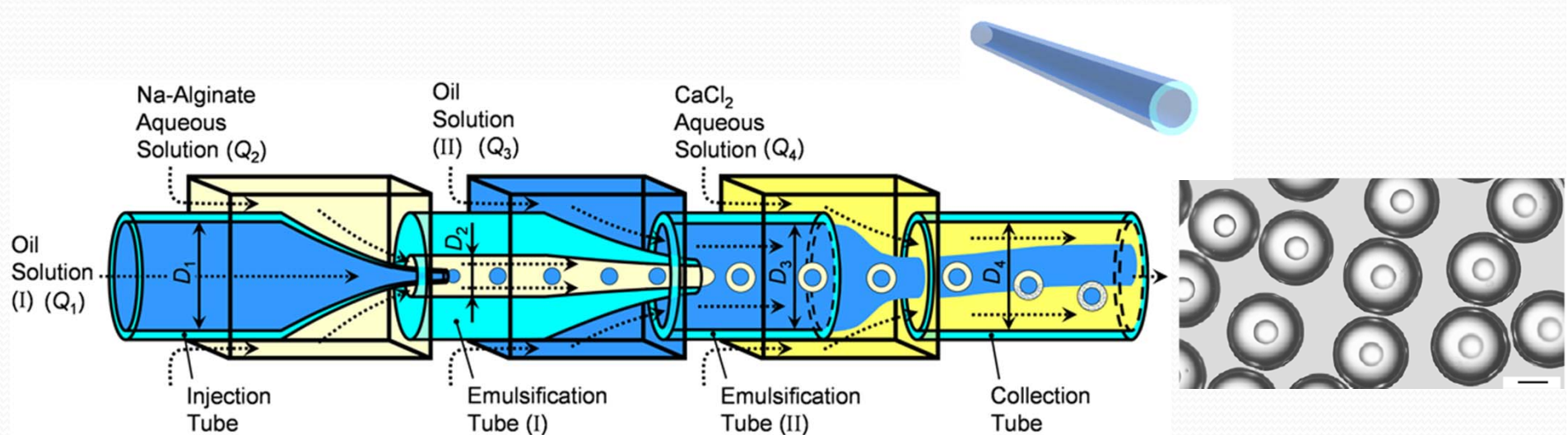


EMULSIONS FORMATION IN A CO-FOCUSING MICROFLUIDIC DEVICES





ENCAPSULATION PROCESS WITH OIL CORE



1. preparation of single emulsions
2. preparation of monodisperse O/W/O double emulsions
3. gelation reaction

Focusing device
creation:

- borosilicate
- micropuller
- microforge



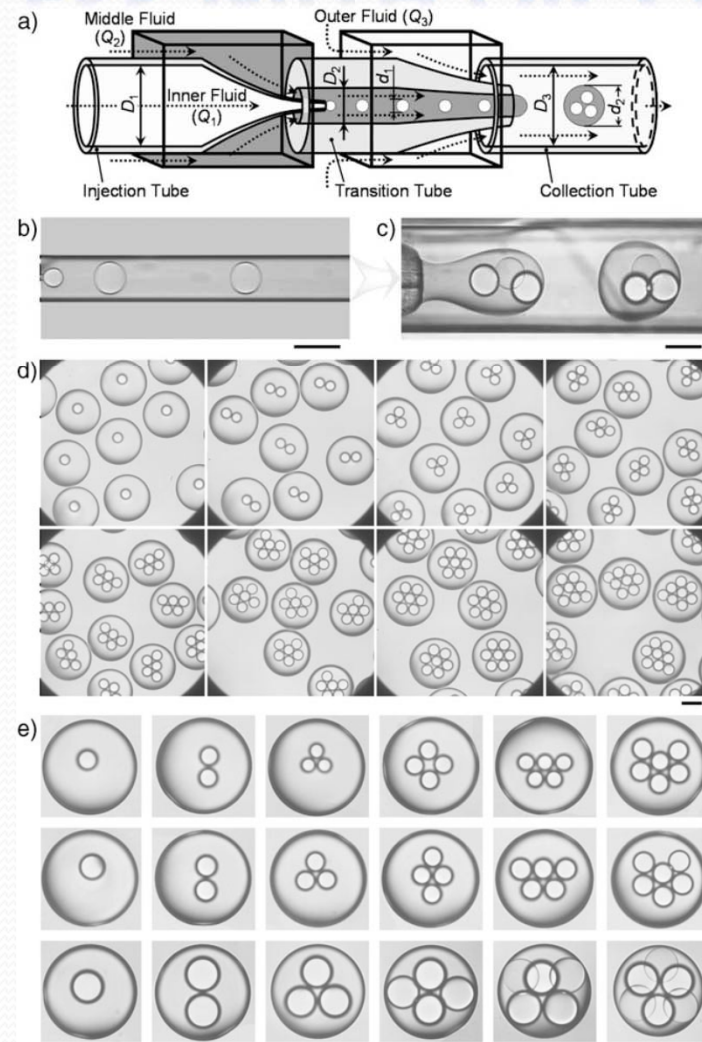
ENCAPSULATION PROCESS WITH OIL CORE

Microfabrication

DOI: 10.1002/anie.200701358

Controllable Monodisperse Multiple Emulsions**

Liang-Yin Chu,* Andrew S. Utada, Rhutesh K. Shah, Jin-Woong Kim, and David A. Weitz*





Encapsulation in food fields is becoming even more important for his many applications. In fact microfluidic's encapsulation allow an encapsulation:

- Effective
- Cheaper
- With capsules's size of the order of tens micron

Nevertheless some issues related to the scaling-up procedure of the tecnology, microfluidic's encapsulation suggest the ability to connect food fields with the health's one, and provide to food technologist an additional important tool for ensure food's quality and safety as well.

CONCLUSION

