

# ENCAPSULATION BY MEMBRANE EMULSIFICATION

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## PRESENTATION LAYOUT

- **How can we produce drops and turn them into particles?**
- **Conventional ways to produce drops**
- **Drop by Drop devices to produce drops**  
**Microchannel & Membrane emulsification**
- **Specific encapsulations – from drops to particles**

**IF WE COULD CREATE A DROP  
THEN WITH THE ADITIONAL  
TREATMENT WE COULD GET THE  
SPHERICAL PARTICLE**

**HOW TO MAKE A DROP?**



## MAKING EMULSIONS - DROP-BY-DROP

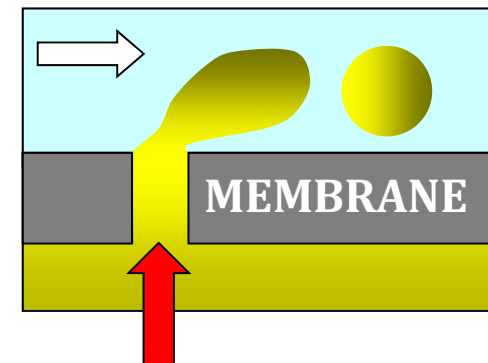
### Microchannel emulsification



Injection of dispersed phase  
through microgrooves.

*Kawakatsu et. al. 1997*

### Membrane emulsification



Injection of dispersed phase through  
membrane.

*Patent - Asher and Tsien 1980*

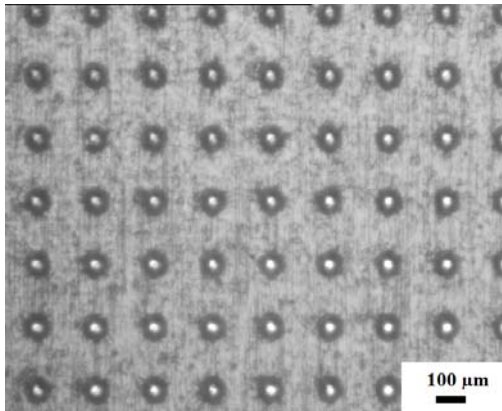
*Nakashima et. al. 1991*

They use **low energy** per unit volume and give  
**monosized** distribution.

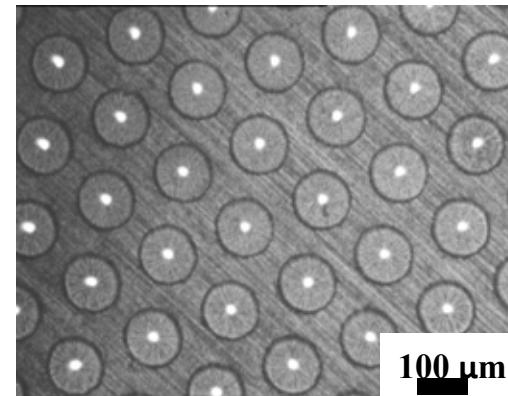
# MEMBRANE EMULSIFICATION

## METAL MEMBRANES

### Stainless steel membranes



### Nickel membranes

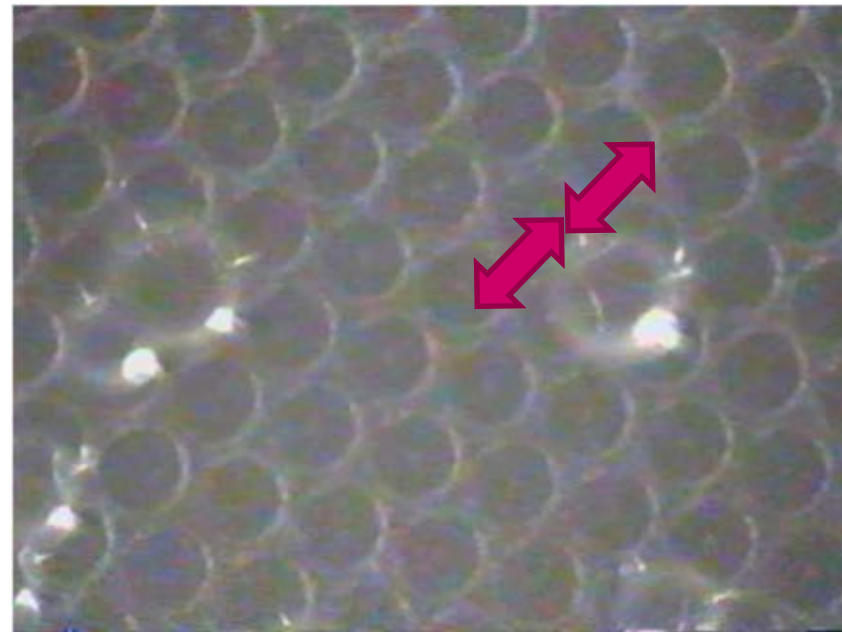
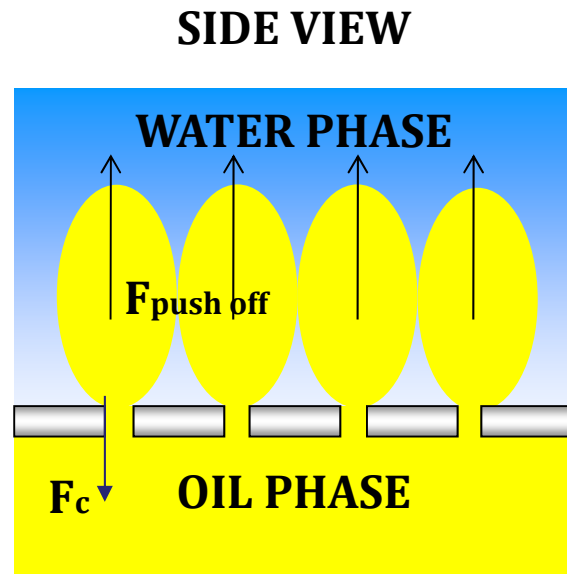


**Both used at Loughborough**

# MEMBRANE EMULSIFICATION

## NO SHEAR STRESS ON THE MEMBRANE SURFACE

TOP VIEW



Hydrophilic membrane

$D_{50}=200 \mu\text{m}$

Scaling up – **possible**  
Productivity – **high**

*Kosvintsev et al. 2008*

## **SHEAR STRESS ON THE MEMBRANE SURFACE**

### **Movements of continuous phase:**

- **STIRRING**
- **CROSS FLOW**
- **PULSATIONS OF THE CONTINUOUS PHASE**

### **Movements of the membrane:**

- **VIBRATIONS**
- **ROTATIONS**
- **TORSIONAL MOVEMENTS**



# EXPERIMENTAL RESULTS

- 1. COMPLEX COACERVATION**
- 2. POLYMER PARTICLES FOR DRUG DELIVERY**
- 3. INORGANIC SILICA PARTICLES**

# 1. COMPLEX COACERVATION

## O/W emulsion

### Motivation for the work:

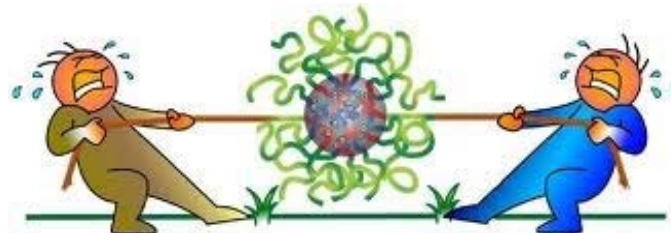
**Currently batch production**

**High polydispersity of the product and usually too big droplet size**

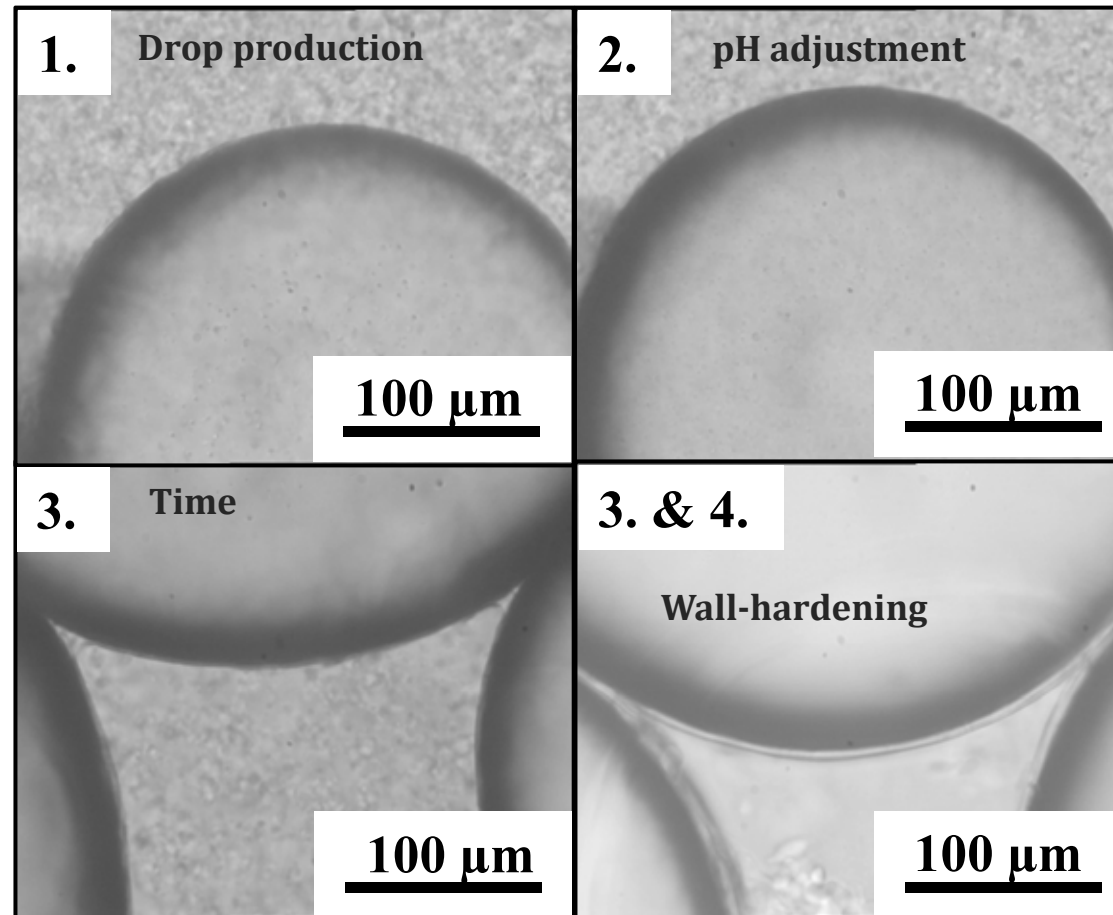
**Need for gelatine alternative**

## 1. COMPEX COACERVATION

1. **Drop production** in hydrocolloids solution
2. Coacervation (phase separation) implying the formation of a coacervate phase – **pH adjustment**
3. Wall formation by aggregation of the hydrocolloid around droplets of the emulsified hydrophobic material – **time, room temperature**
4. **Wall-hardening**, which is generally achieved by cross-linking the hydrocolloid forming the wall



# 1. COMPLEX COACERVATION



# 1. COMPLEX COACERVATION – Oil encapsulation

## FISH GELATINE CAPSULES

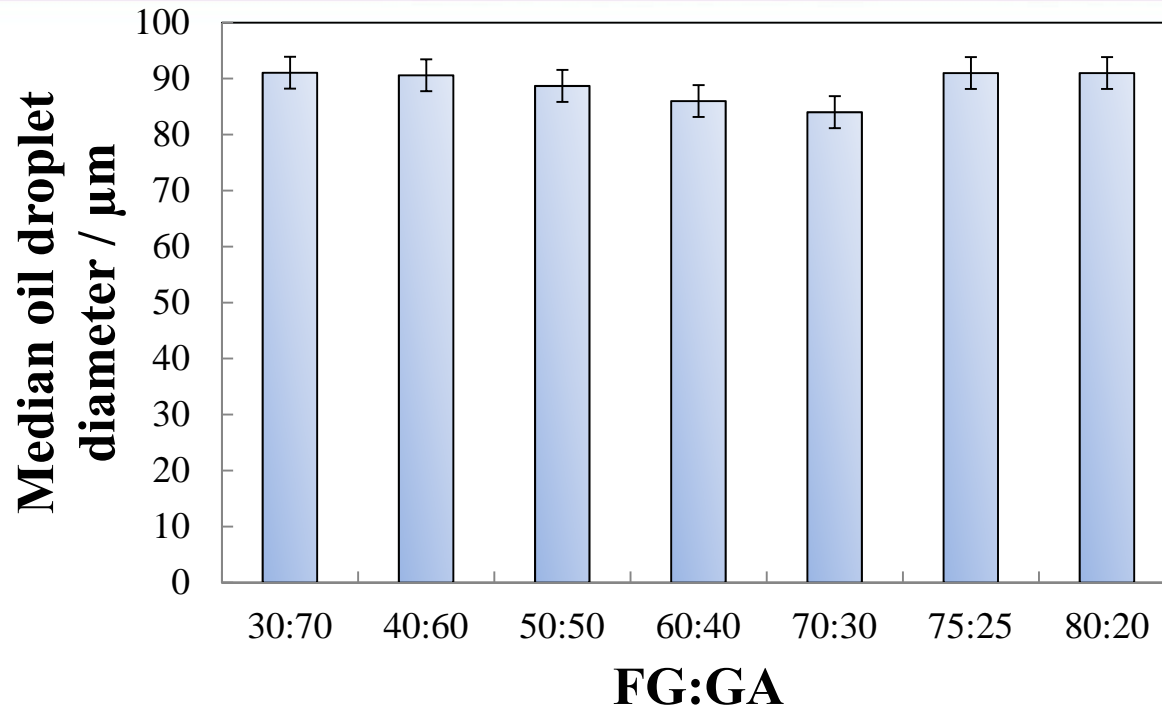
### WHY FISH GELATINE?

- **ROOM TEMPERATURE** - less energy compared to alternative gelatine types and
- New possibilities for encapsulation of **VOLATILE COMPOUNDS**
- Increased **CONSUMER** consent for religious or diet reasons and health safety

*Piaccentini et al., 2013*

*ITM-CNR @ University  
of Calabria, Rende*

## DIFFERENT RATIOS OF FG:GA – PARTICLE SIZE



**Shear = 6 Pa**

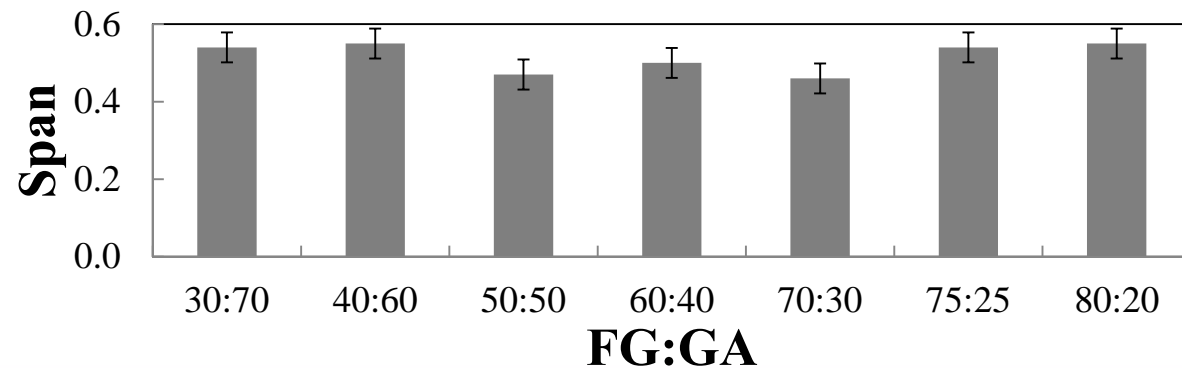
**Injection rate = 1.5 ml/min**

**pH=3.5**

**Room temperature**

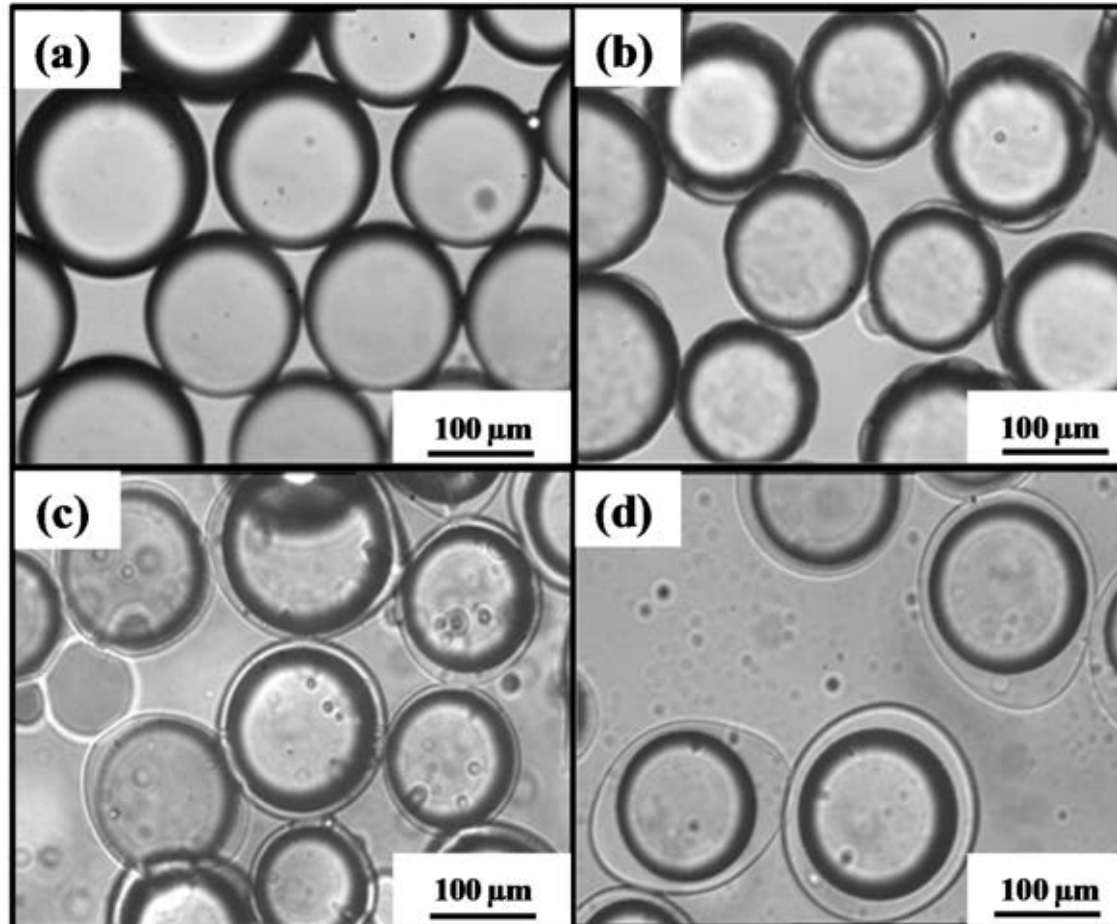
$$\text{Span} = \frac{D_{90} - D_{10}}{D_{50}}$$

*Piaccentini et al., 2013*



# DIFFERENT RATIOS OF FG:GA FOR MICROCAPSULES

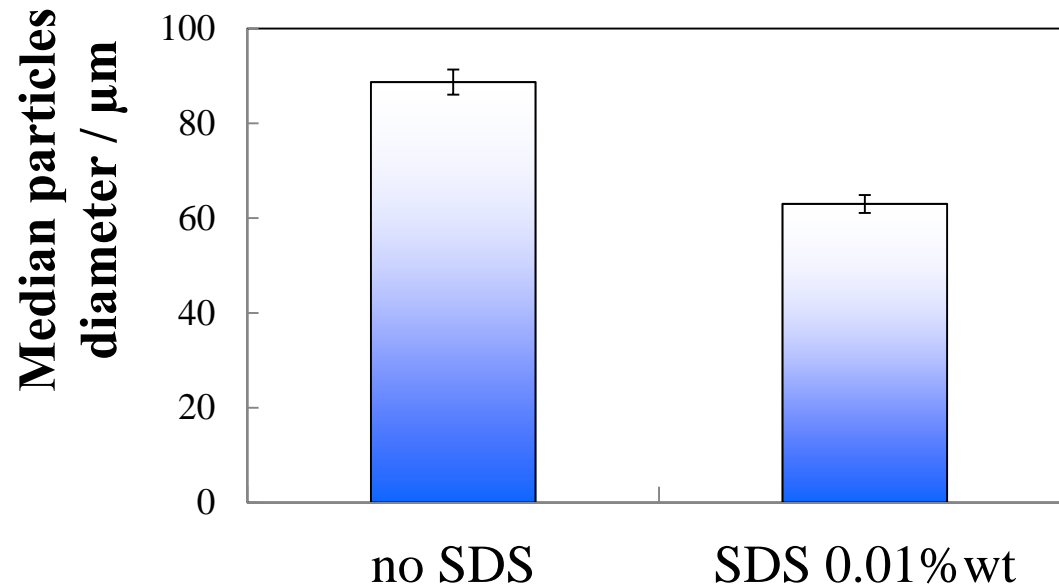
## ROOM TEMPERATURE



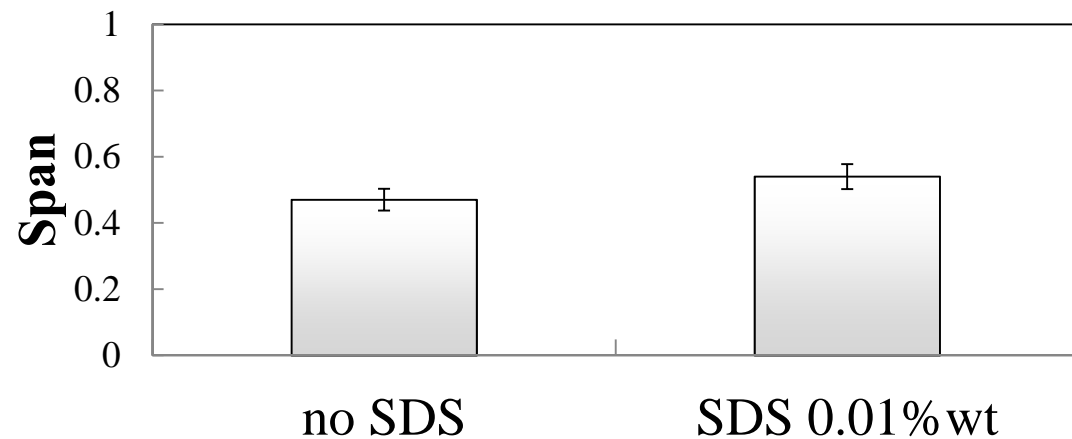
*Piaccentini et al.,  
2013*

**FG:GA** (a) 30:70; (b) 40:60; (c) 80:20; and (d) 50:50.

## PARTICLE SIZE CONTROL WITH SURFACTANT ADDED



Shear = 6 Pa  
Injection rate = 1.5 ml/min



*Piaccentini et al., 2013*

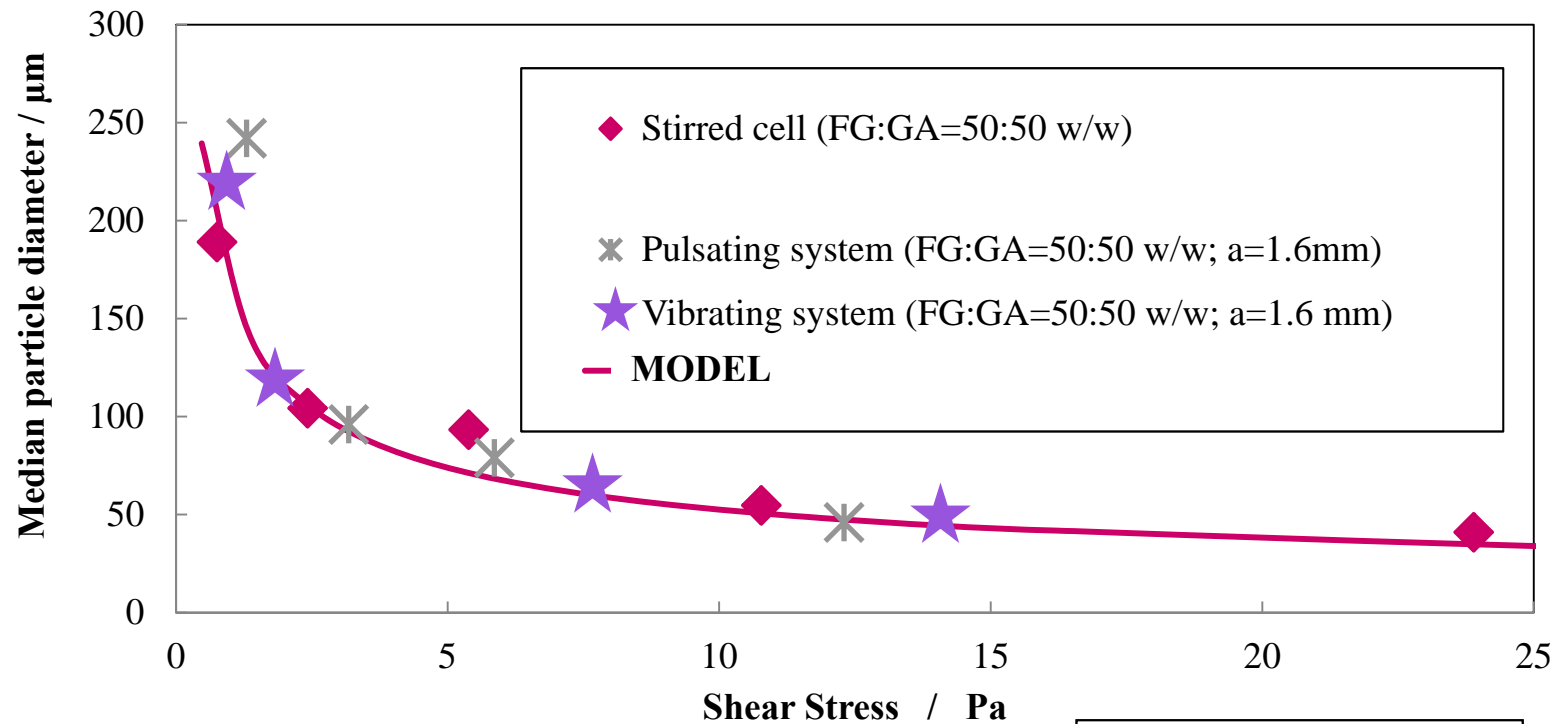


# 1. COMPLEX COACERVATION

## DISPERSION CELL, PULSATING & VIBRATING SYSTEM

Dispersed phase: **Sunflower oil**

Continuous phase: **Fish gelatine (FG) and Gum Arabic (GA)**



Flux through the pulsed membrane =  $3\text{L h}^{-1}$

*Piaccentini et al., 2013*



## **2. ANTYCANCER DRUG ENCAPSULATION**

Aim to encapsulate water soluble peptide

## **2. ANTYCANCER DRUG ENCAPSULATION**

### **O/W & W/O/W**

#### **Motivation for the work:**

**Currently batch production**

**Low uniformity of the produced particles using conventional emulsification methods**

**Need for higher encapsulation efficiency**

**Anticancer drug - extremely expensive & temperature sensitive)**





### **3. SILICA PARTICLES** **W/O emulsion**

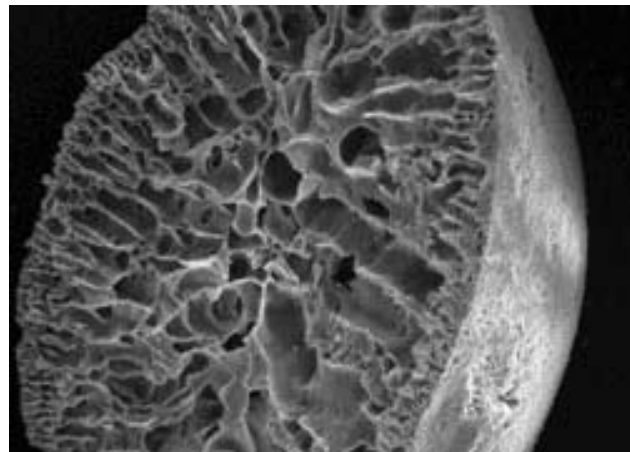
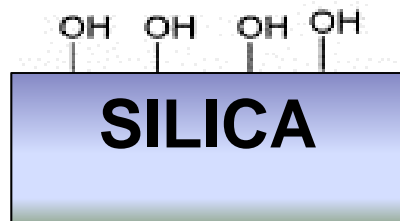
**Aim to produce spherical silica particles  
with high surface area and internal structure**

## 3. SILICA PARTICLES

### W/O emulsion

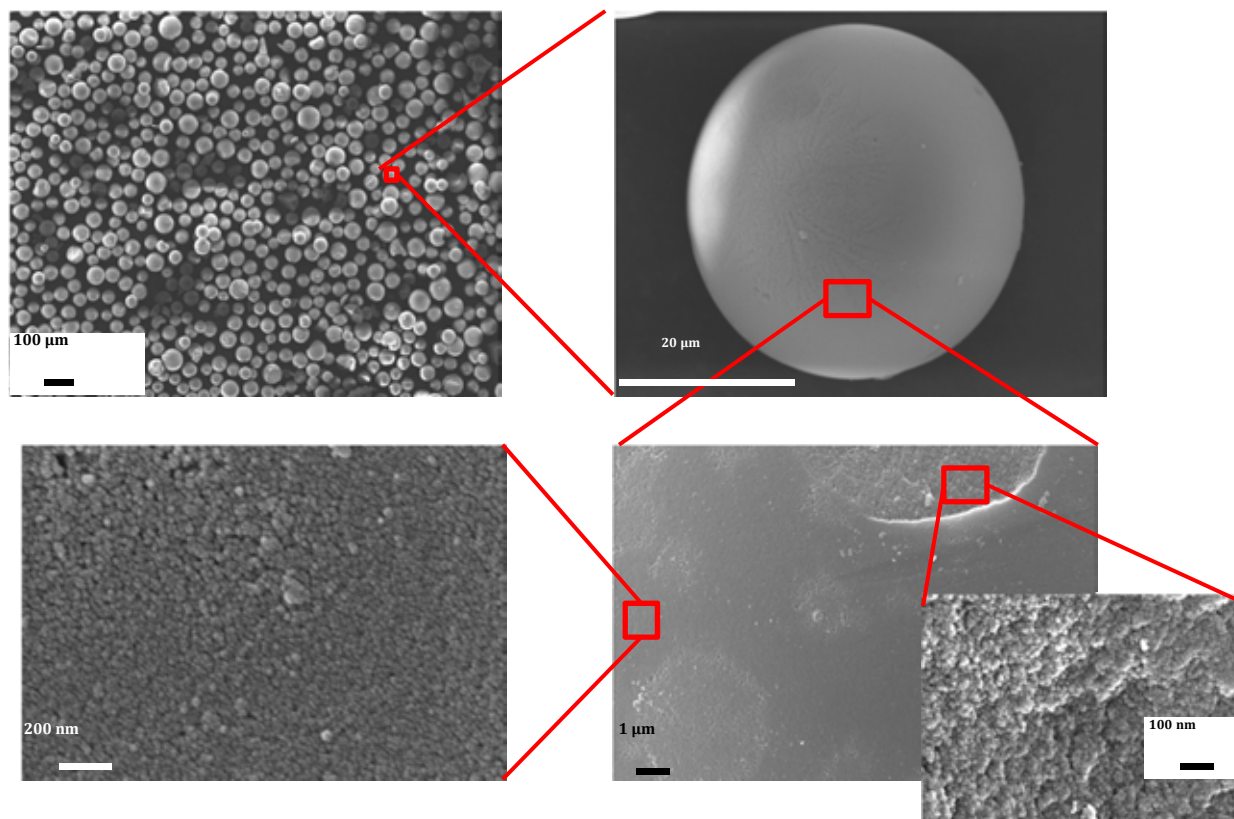
#### Motivation for the work:

- Be able to produce novel ion exchange materials
- Use of porous silica for delivery of oil soluble drugs



## 3. SILICA PARTICLES for drug delivery

SILICA PARTICLES WITH  $D_{50}=40\mu\text{m}$  AFTER DRYING



## CONCLUSIONS

### WHY MEMBRANE EMULSIFICATION?

- **High productivity using microsieve membrane**
- **Uniform particles sizes 10 – 1000  $\mu\text{m}$**
- **Suitable for encapsulation of sheer sensitive compounds**
- **Scaling up possible providing larger membrane area**



## ACKNOWLEDGEMENTS

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<http://www.micropore.co.uk/>

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