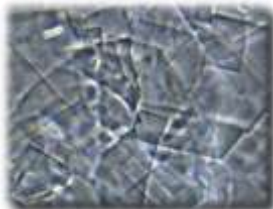


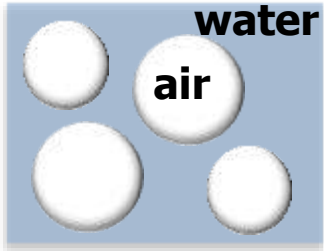
# Edible oil foams stabilized by crystalline particles

**Dr. Anne-Laure Fameau**

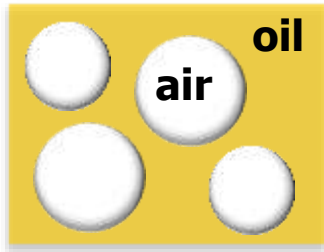


# Outline

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## **1. Aqueous foams**



## **2. Differences between aqueous & non-aqueous foams**



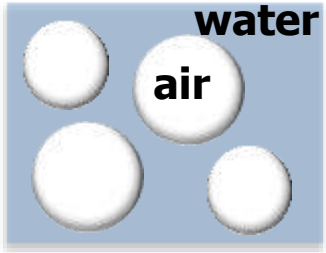
## **3. Formulation rules for edible oil foams**



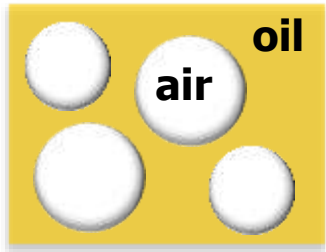
## **4. Effect of co-crystallization on oil foam properties**

# Outline

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## 1. Aqueous foams



## 2. Differences between aqueous & non-aqueous foams



## 3. Formulation rules for edible oil foams



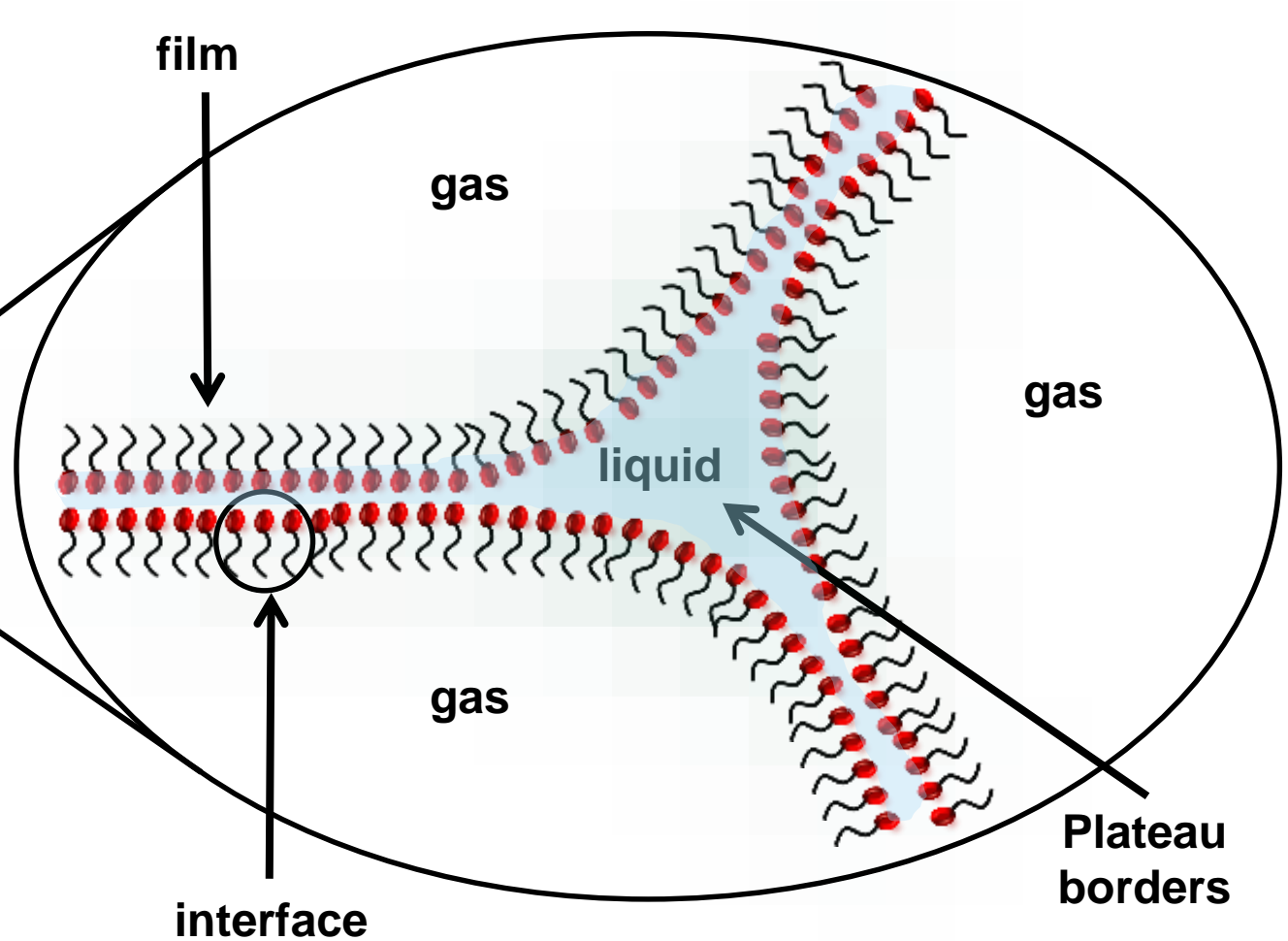
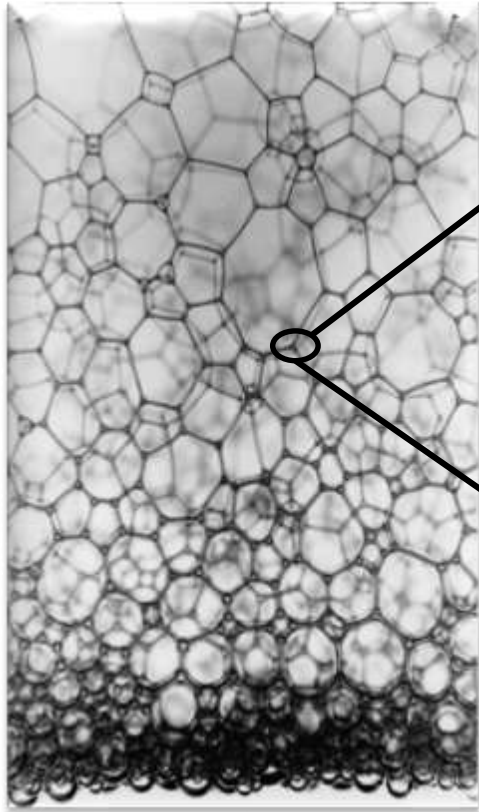
## 4. Effect of co-crystallization on oil foam properties

# Aqueous Foams for what?



# What is an Aqueous Foam?

**FOAM**

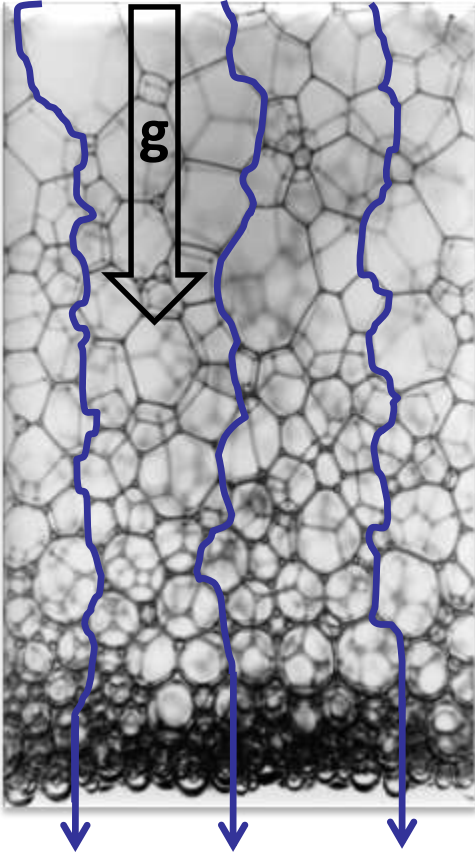




# Mechanisms of foam destabilization

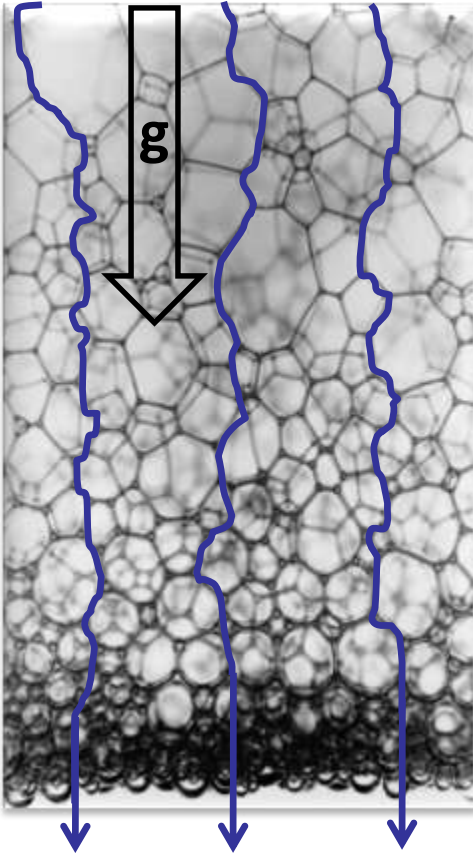
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Drainage

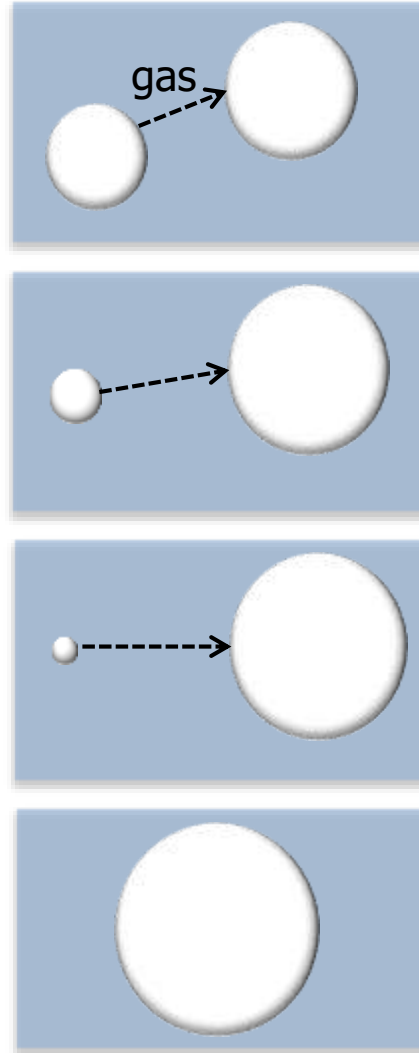


# Mechanisms of foam destabilization

Drainage

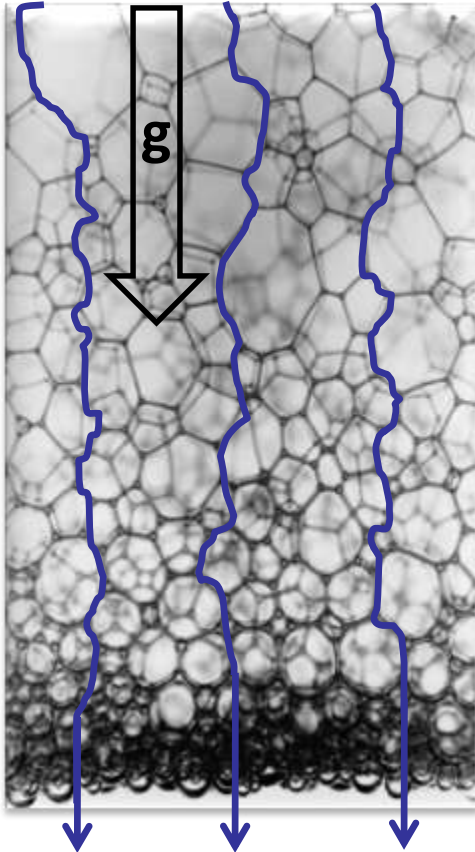


Coarsening

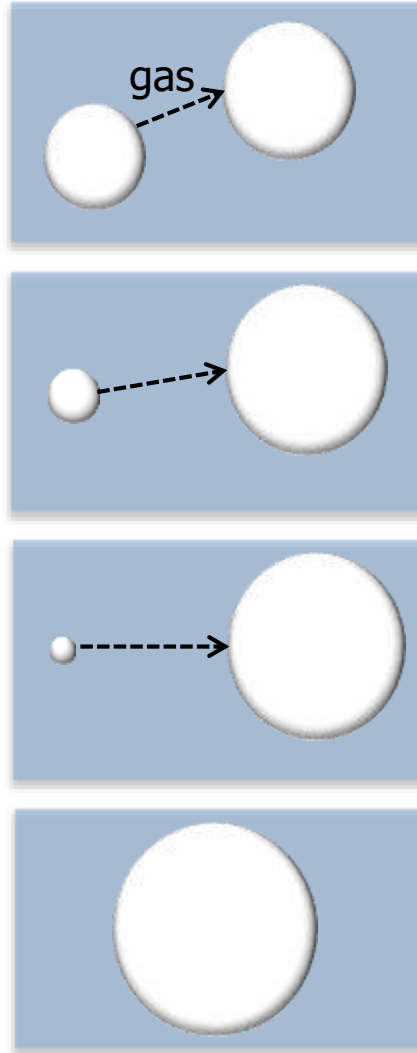


# Mechanisms of foam destabilization

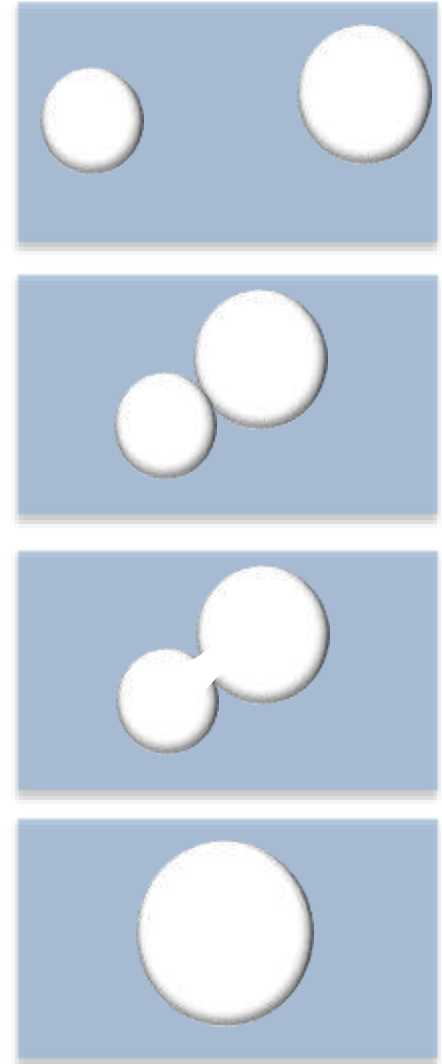
Drainage



Coarsening



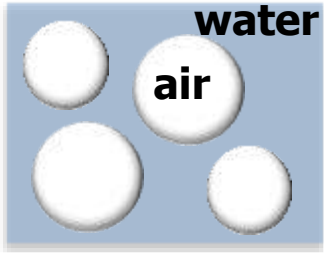
Coalescence



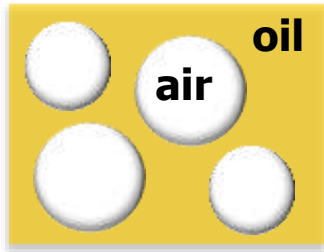


# Outline

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## 1. Aqueous foams



## 2. Differences between aqueous & non-aqueous foams



## 3. Formulation rules for edible oil foams

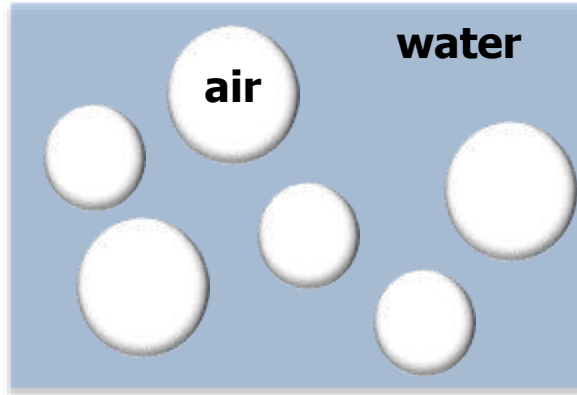


## 4. Effect of co-crystallization on oil foam properties

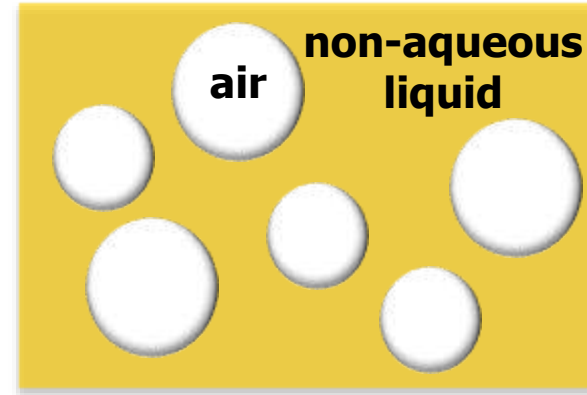
# What is a non-Aqueous Foam?

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## Aqueous Foams

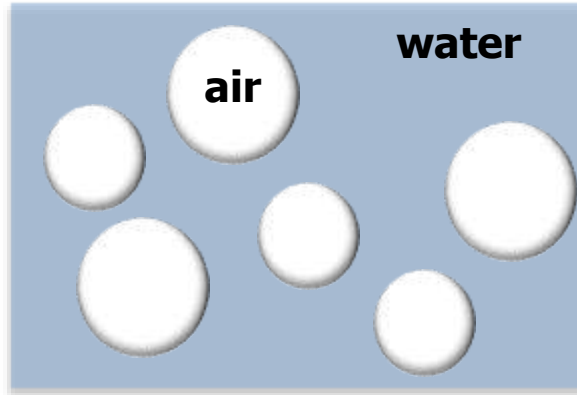


## Non-Aqueous Foams

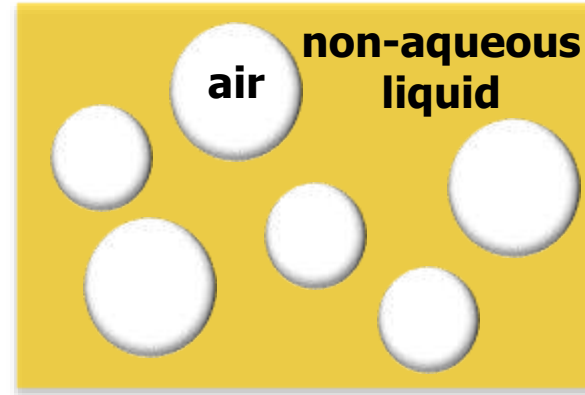


# What is a non-Aqueous Foam?

## Aqueous Foams



## Non-Aqueous Foams



## Where can we find Non-Aqueous Foams?



**Cosmetic industry**



**Petroleum industry**

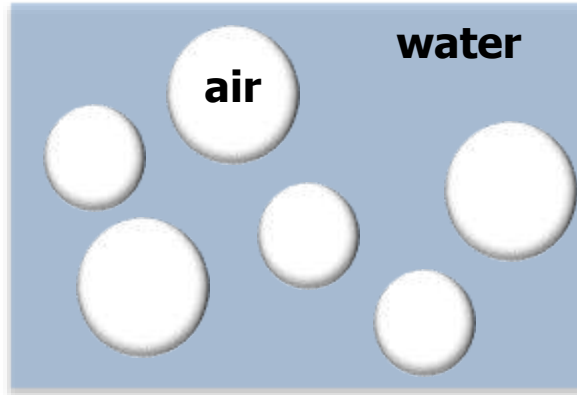


**Food industry**

**Few data available on the formulation, production and characterization of Non-Aqueous Foams**

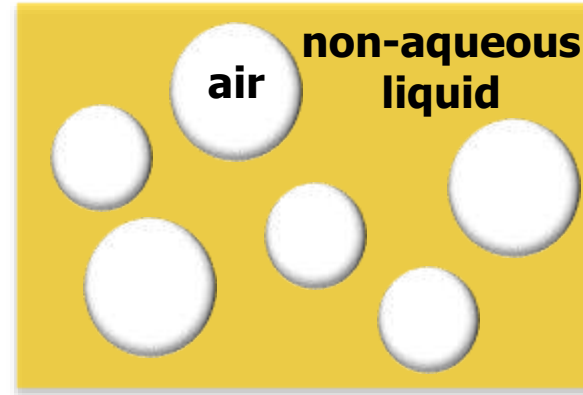
# Aqueous/Non-Aqueous Foams: Differences

## Aqueous Foams



**Maximum Liquid fraction  
< 1 %**

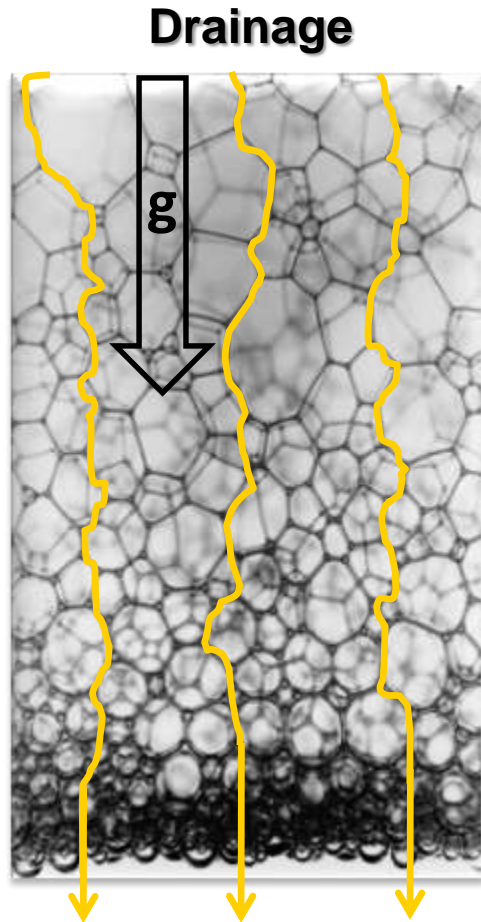
## Non-Aqueous Foams



**Maximum Liquid fraction  
around < 10-20%**

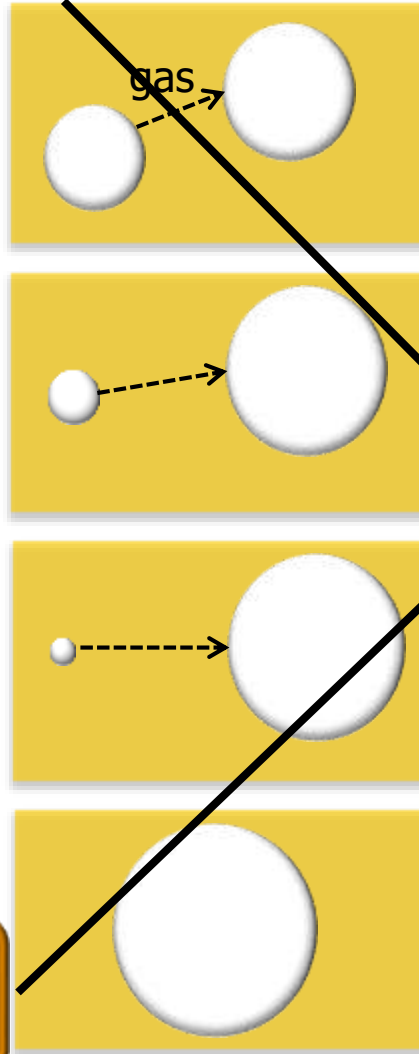
**Large difference in the liquid fraction inside the foam**

# Aqueous/Non-Aqueous Foams: Differences

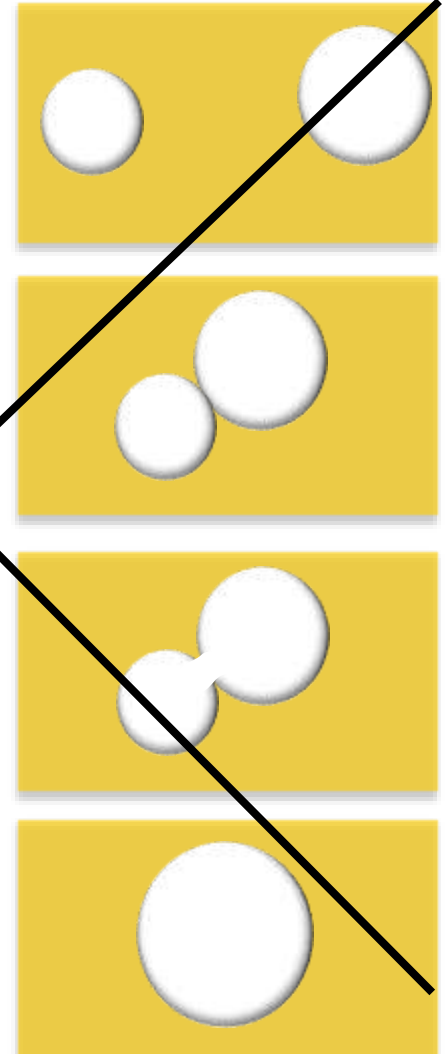


**Drainage most important destabilization mechanism**

**Coarsening**

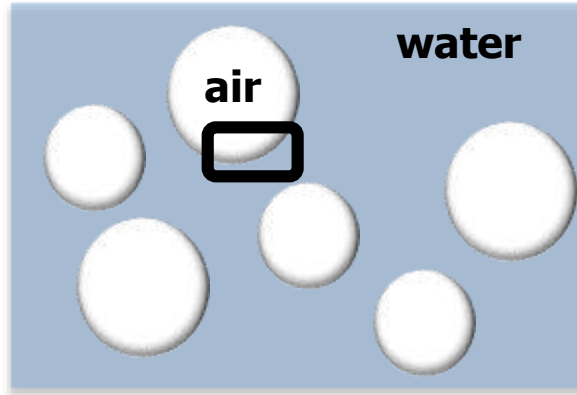


**Coalescence**



# Aqueous/Non-Aqueous Foams: Differences

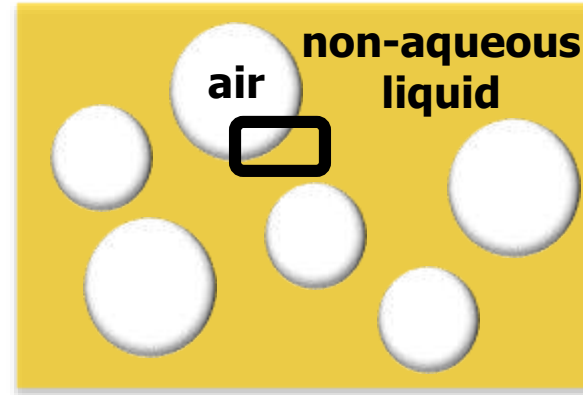
## Aqueous Foams



**72 mN.m<sup>-1</sup> at 25°C**



## Non-Aqueous Foams



**5-35 mN.m<sup>-1</sup> at 25°C**

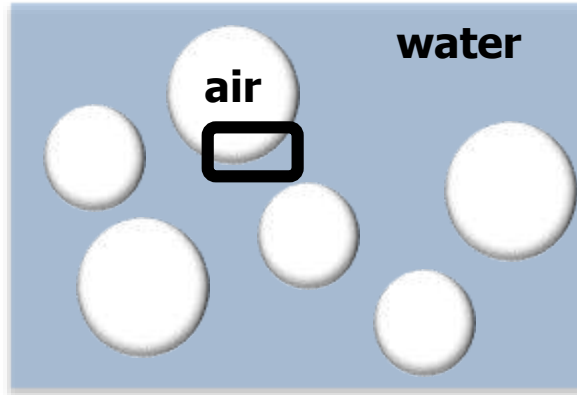


**Large difference in the surface tension of the solvents**



# Aqueous/Non-Aqueous Foams: Differences

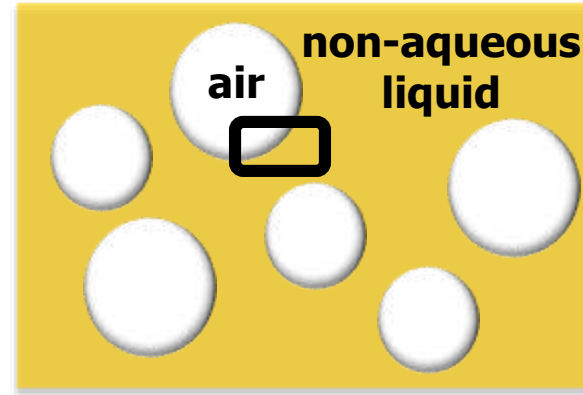
## Aqueous Foams



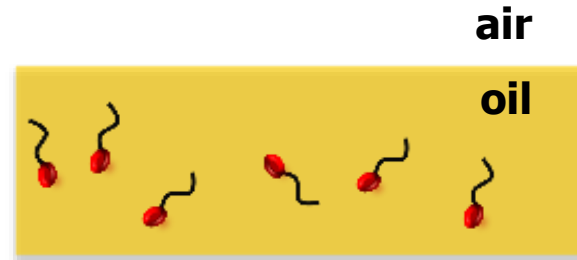
30-40 mN.m<sup>-1</sup> at 25°C



## Non-Aqueous Foams



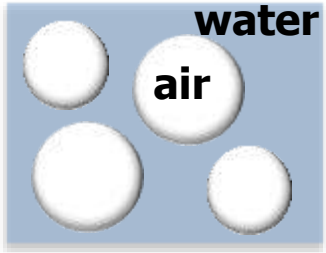
No adsorption



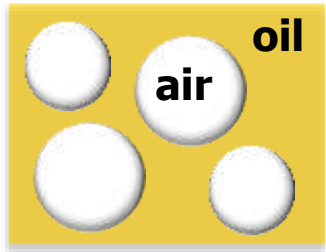
**For oil systems, the low surface tension makes the adsorption of hydrocarbon-based surfactants energetically unfavourable.**

# Outline

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## 1. Aqueous foams



## 2. Differences between aqueous & non-aqueous foams



## 3. Formulation rules for edible oil foams



## 4. Effect of co-crystallization on oil foam properties

# How to obtain Oil Foams?

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## Fluorocarbon-based surfactants



**Low Foam  
Stability**

B. Binks, et al., Coll Surf A, 2010.

# How to obtain Oil Foams?

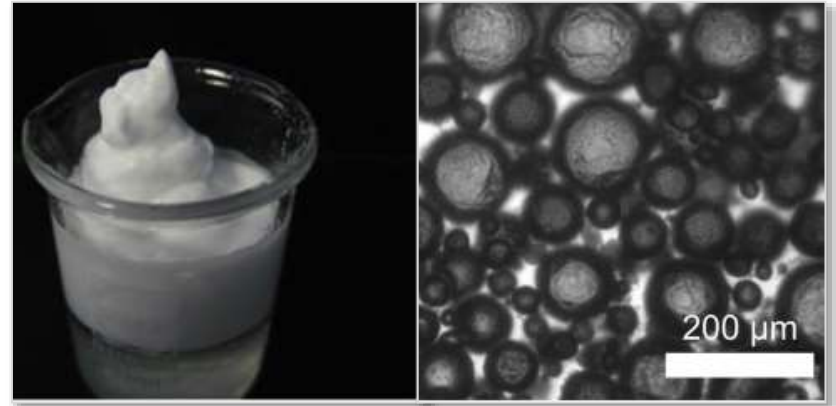
## Fluorocarbon-based surfactants



**Low Foam  
Stability**

B. Binks, et al., Coll Surf A, 2010.

## Partially Oleophobic Particles (Surfaces coated with Fluoro groups)



**Ultrastable Foam**

R. Murakami, et al. Advanced Functional Materials, 2010.

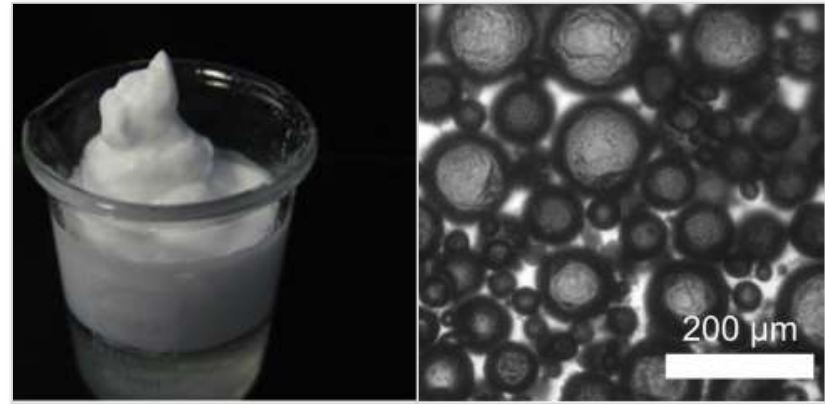
# How to obtain Oil Foams?

## Fluorocarbon-based surfactants



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Stability**

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**Ultrastable Foam**

B. Binks, et al., Coll Surf A, 2010.

R. Murakami, et al. Advanced Functional Materials, 2010.

## Crystalline particles of fatty components (Mono-, Di-, Triglycerides, fatty acids, fatty alcohol & sucrose esters)

**Ultrastable & based on Food grade components**

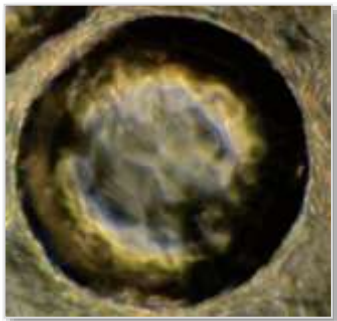
L. K. Shrestha, et al., Langmuir, 2006.

A-L. Fameau et al., Langmuir, 2015.

Y. Lui, et al., JCIS, 2021.

M. Callau et al., Food Chemistry, 2020.

**A-L. Fameau, Book chapter. Handbook of Molecular Gastronomy, 2021**



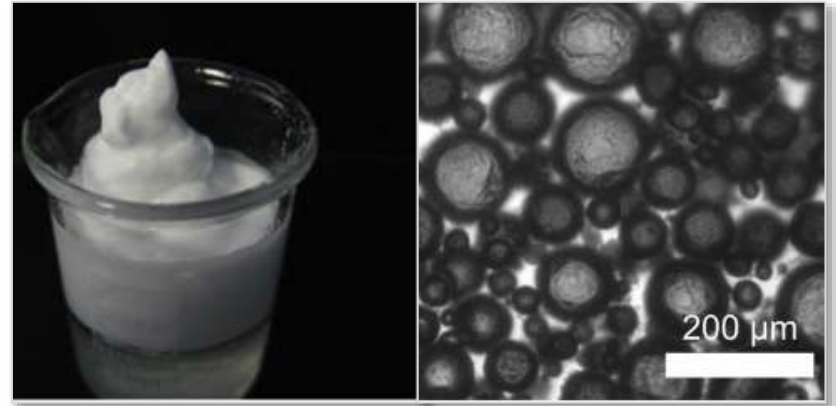
# How to obtain Oil Foams?

## Fluorocarbon-based surfactants



**Low Foam Stability**

## Partially Oleophobic Particles (Surfaces coated with Fluoro groups)

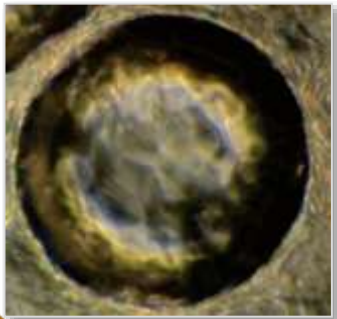


**Ultrastable Foam**

B. Binks, et al., Coll Surf A, 2010.

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## Crystalline particles of fatty components (Mono-, Di-, Triglycerides, fatty acids, fatty alcohol & sucrose esters)



**Ultrastable & based on Food grade components**

L. K. Shrestha, et al., Langmuir, 2006.

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Y. Lui, et al., JCIS, 2021.

M. Callau et al., Food Chemistry, 2020.

**A-L. Fameau, Book chapter. Handbook of Molecular Gastronomy, 2021**



# How to obtain crystalline particles?

## Vegetable oils



+

## Fatty components



- **Mono-, Di-, Triglycerides**
- **Fatty acids**
- **Fatty alcohol**
- **Sucrose esters**

# How to obtain crystalline particles?

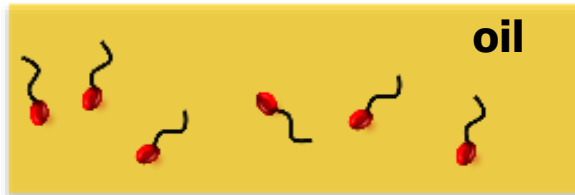
## Vegetable oils



## Fatty components



- Mono-, Di-, Triglycerides
- Fatty acids
- Fatty alcohol
- Sucrose esters



**Fatty component  
soluble at high  
temperatures**



# How to obtain crystalline particles?

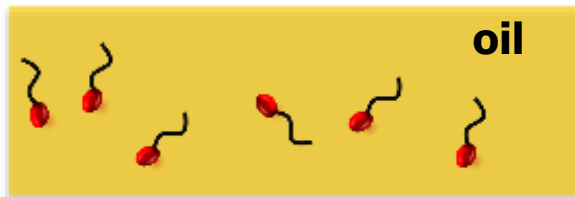
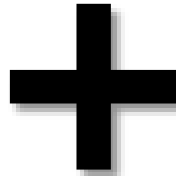
## Vegetable oils



## Fatty components

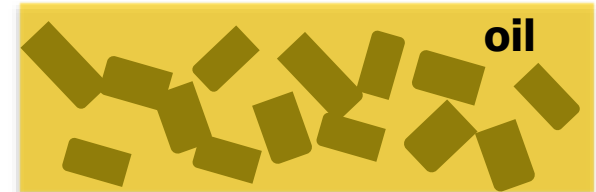


- Mono-, Di-, Triglycerides
- Fatty acids
- Fatty alcohol
- Sucrose esters



Fatty component  
soluble at high  
temperatures

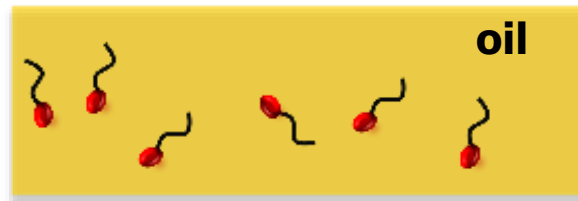
Temperature



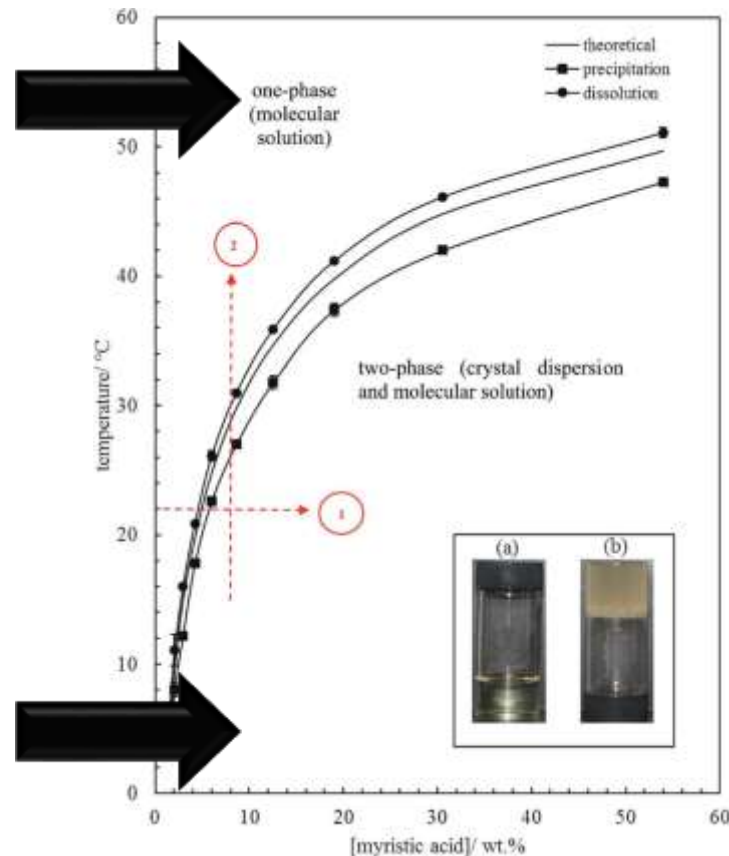
Crystalline particles

**Oleogel formation due to the formation of crystalline particles**

# How to obtain crystalline particles?

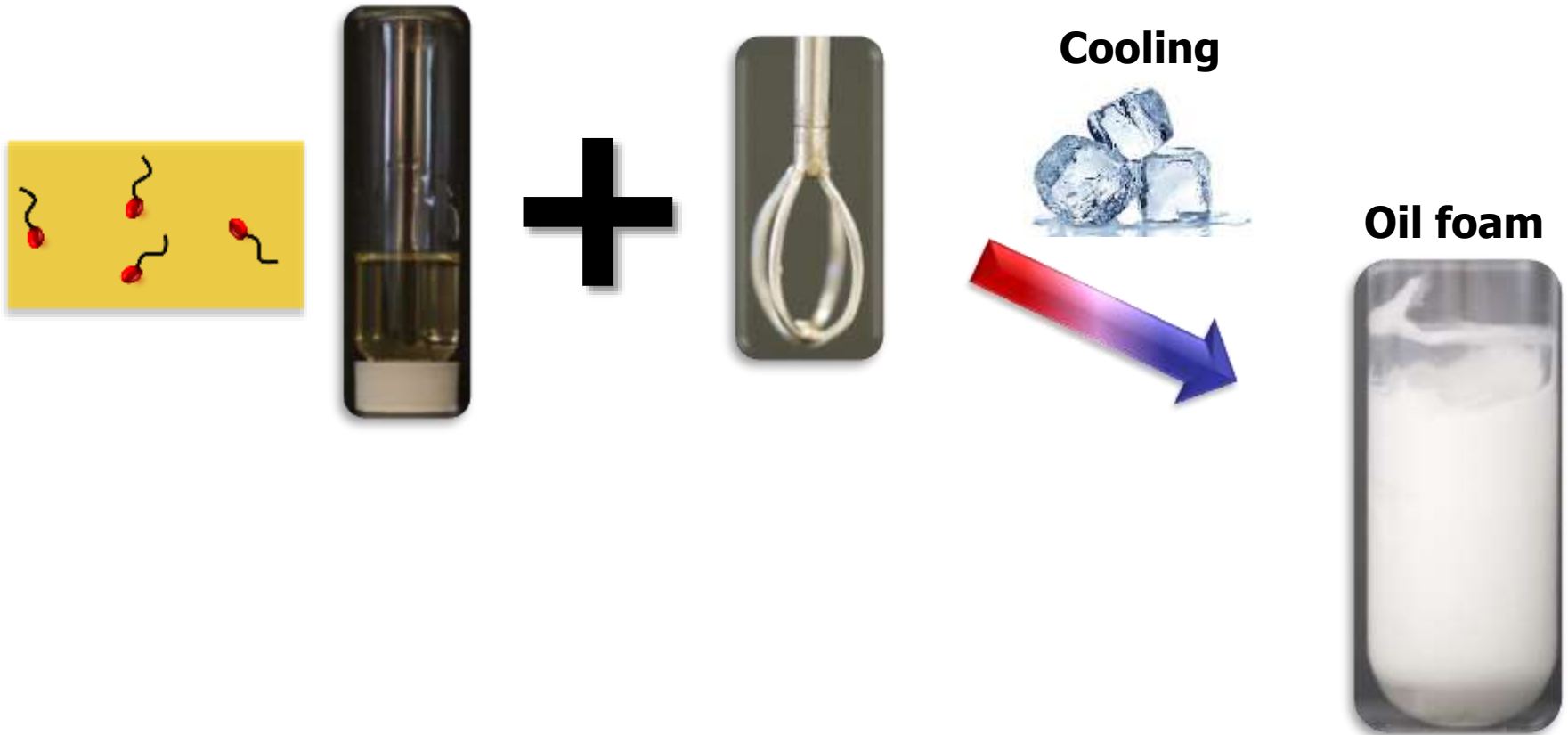


**Crystalline particles**

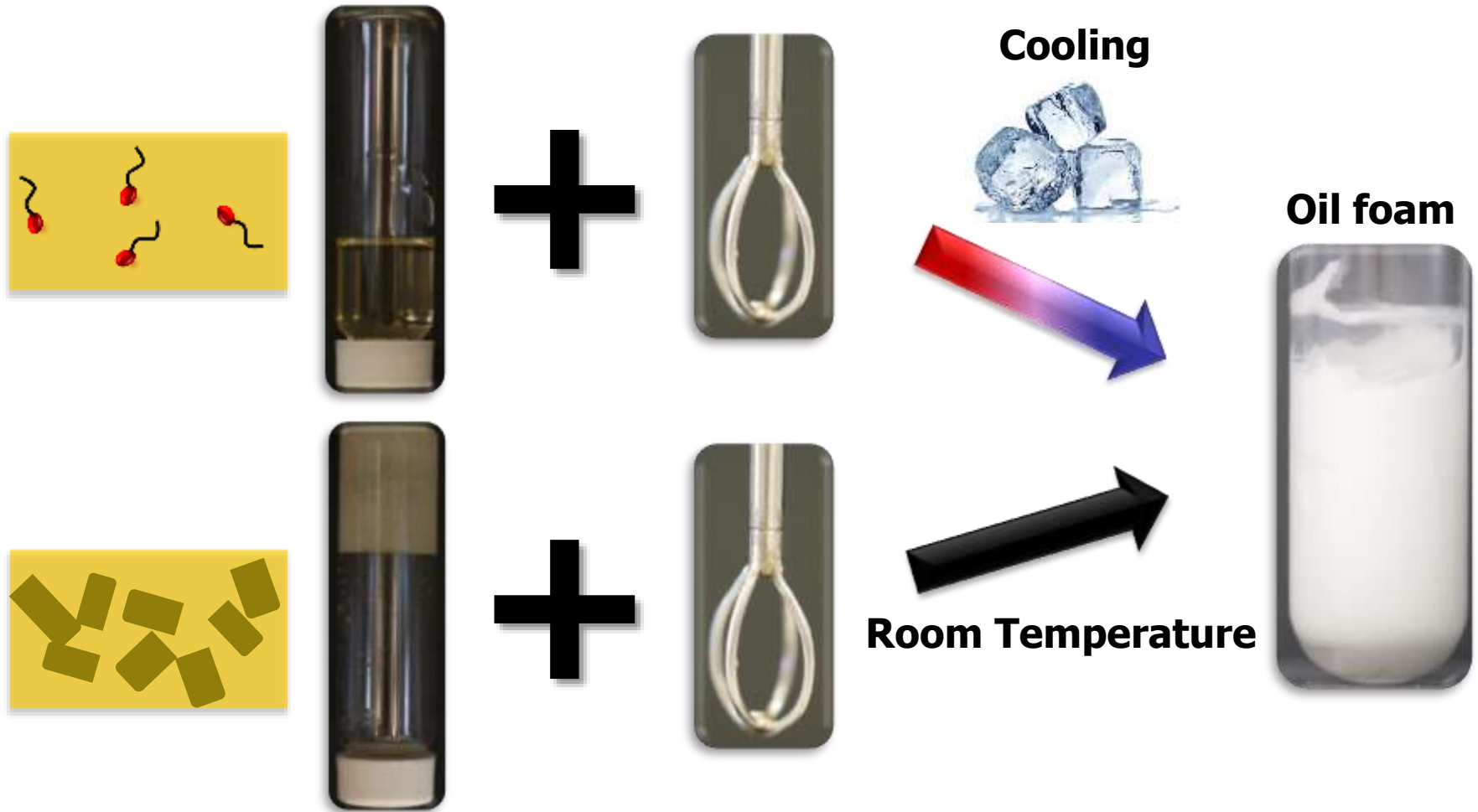


**To produce crystalline particles: determination of the solubility limit for each fatty component in a given oil.**

# How to produce Oil foam?

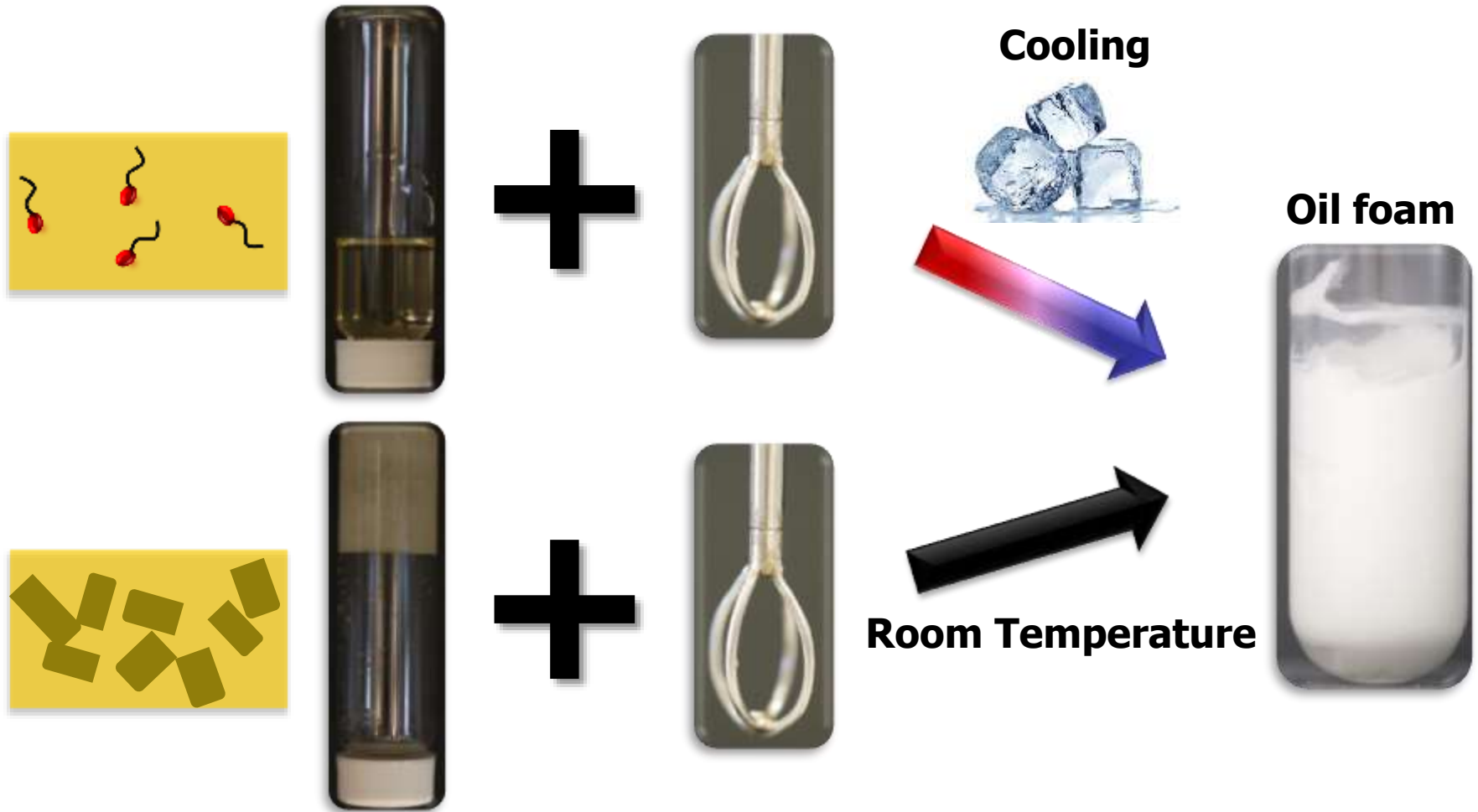


# How to produce Oil foam?





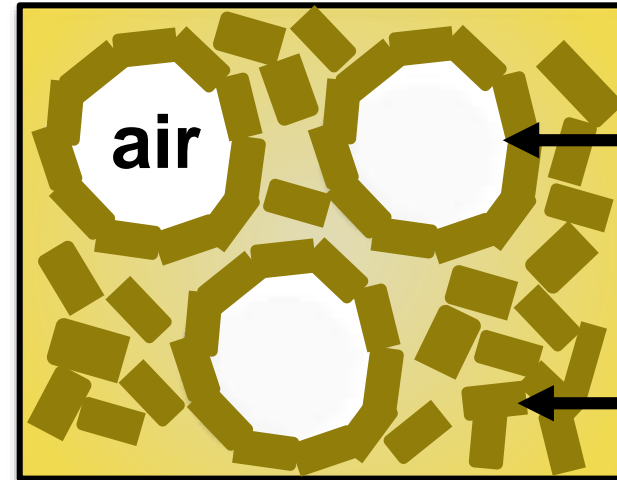
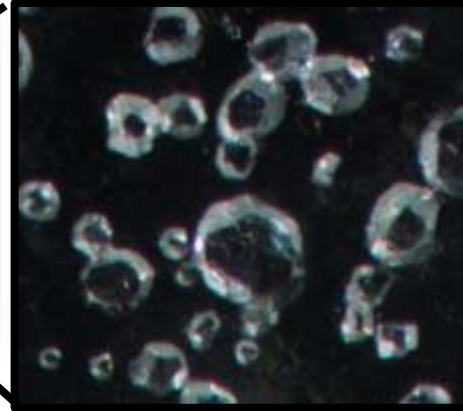
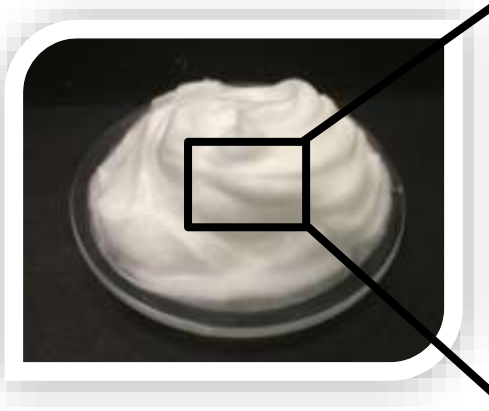
# How to produce Oil foam?



**Two ways to produce oil foams by whipping: (1) by cooling during the oleogel formation or (2) directly from the oleogel**

# How to stabilize Oil foam?

**OIL FOAM**

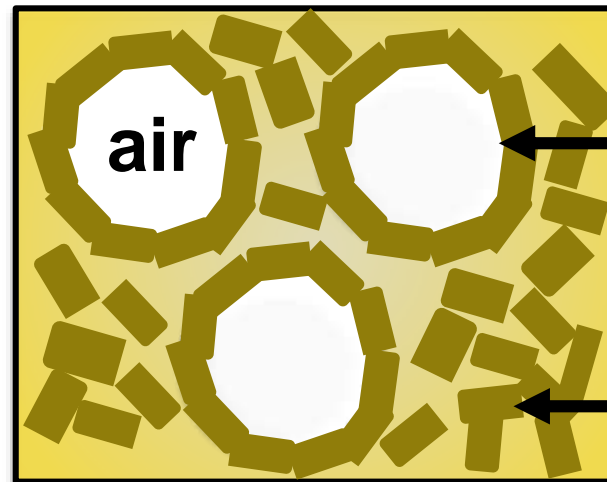
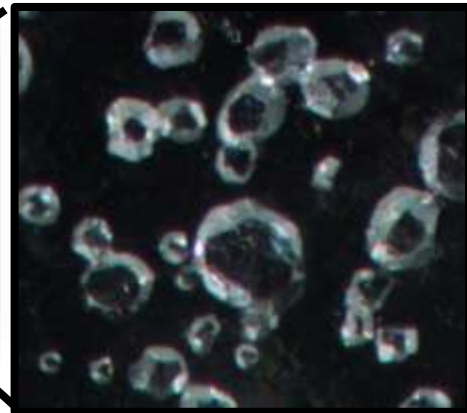
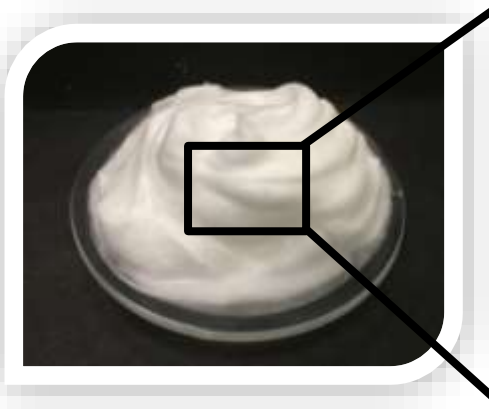


bubble

crystalline  
particle

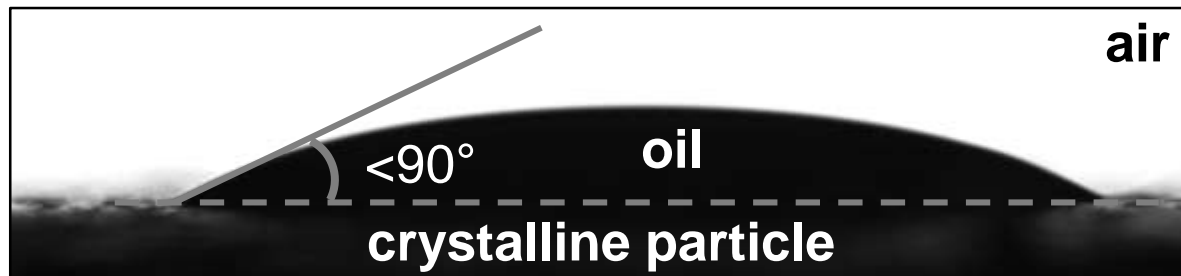
# How to stabilize Oil foam?

OIL FOAM



bubble

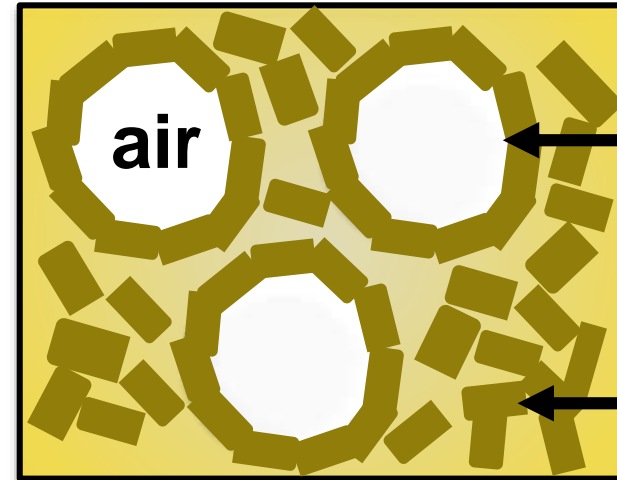
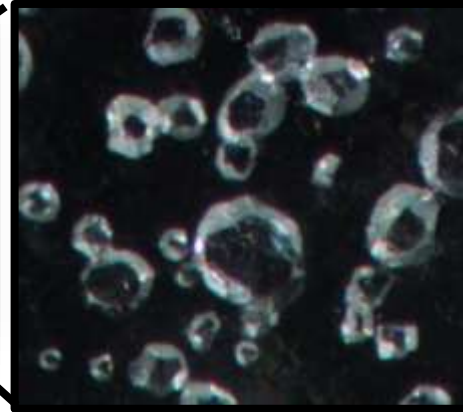
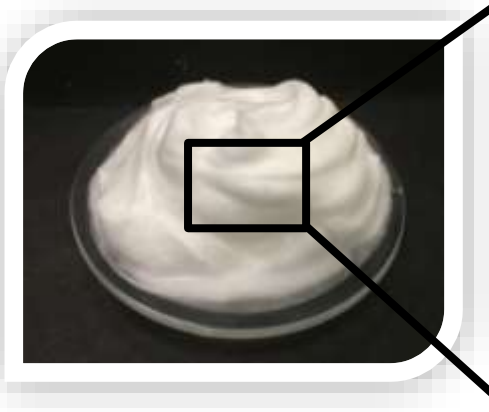
crystalline  
particle



**Key parameter for crystals to adsorb at the air-oil surface is to exhibit a suitable contact angle  $< 90^\circ$ .**

# How to stabilize Oil foam?

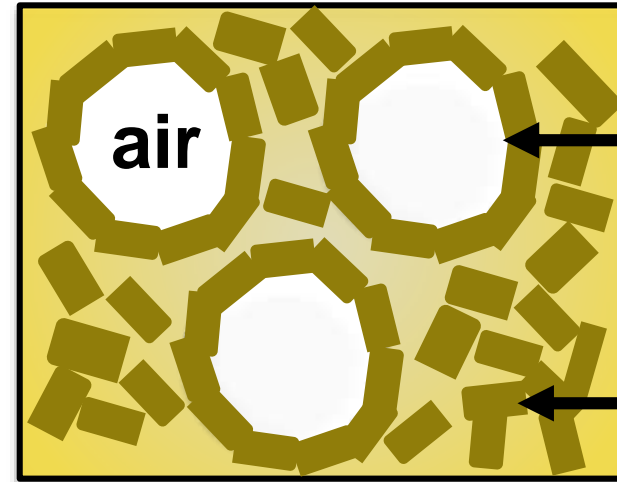
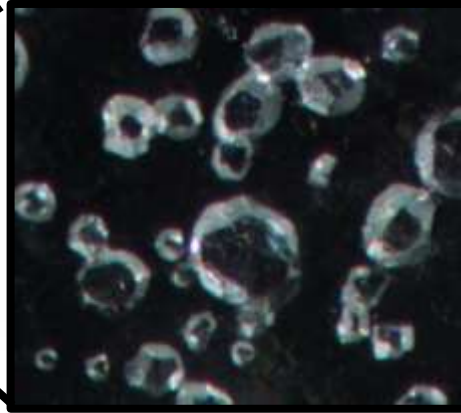
**OIL FOAM**



**Size & shape of crystalline particles depend on: cooling rate, shearing rate & nature of fatty component.**

# Effect of the particles shape?

## OIL FOAM



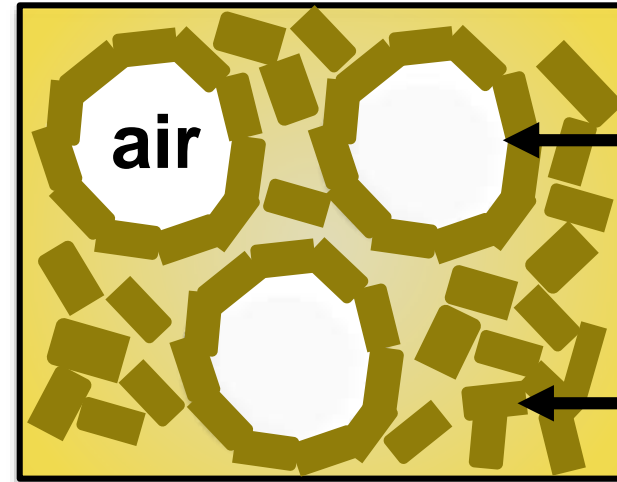
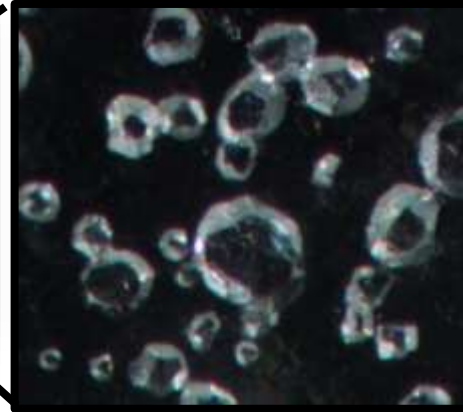
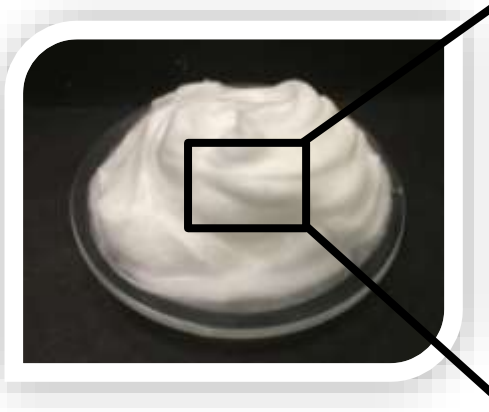
  
fiber

  
platelet

  
spherulite

# Effect of the particles shape?

## OIL FOAM



bubble

crystalline  
particle



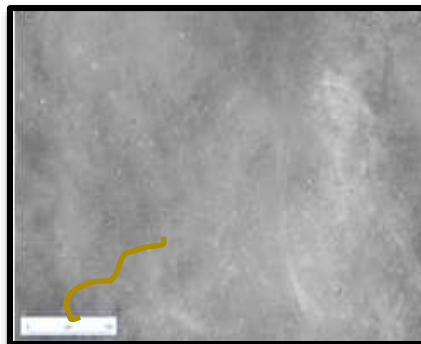
fiber



platelet



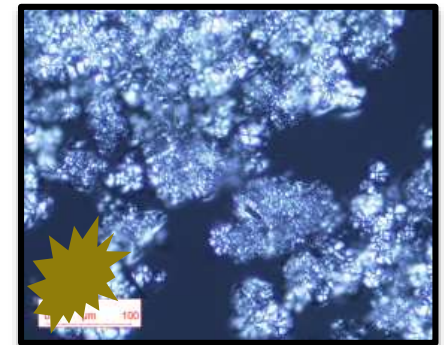
spherulite



12-hydroxystearic acid



stearic acid

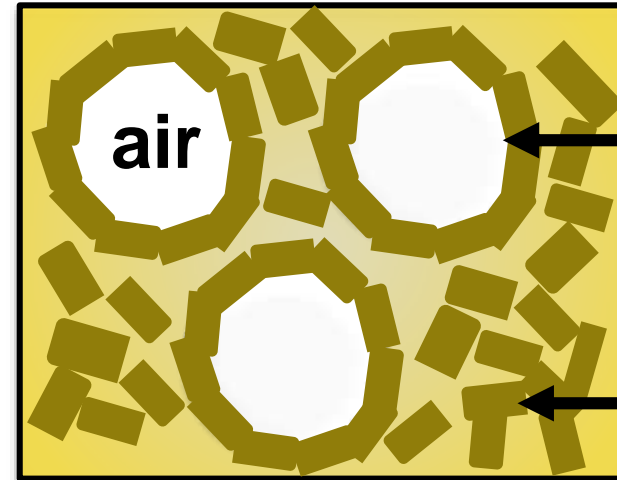
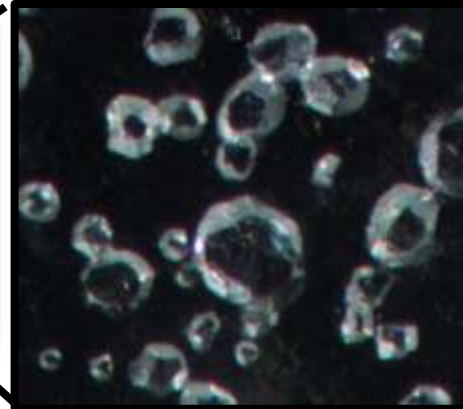
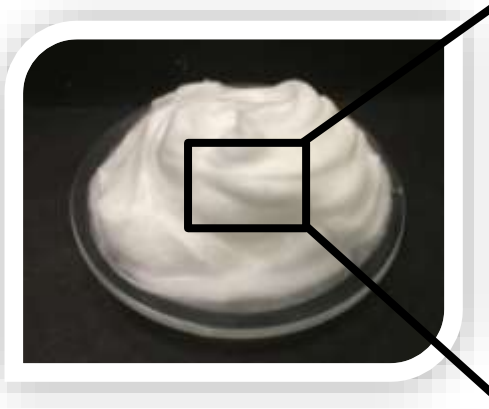


trihydroxystearine



# Effect of the particles shape?

## OIL FOAM



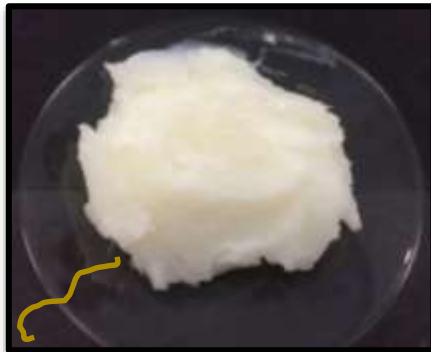
bubble

crystalline  
particle

  
fiber

  
platelet

  
spherulite



12-hydroxystearic acid



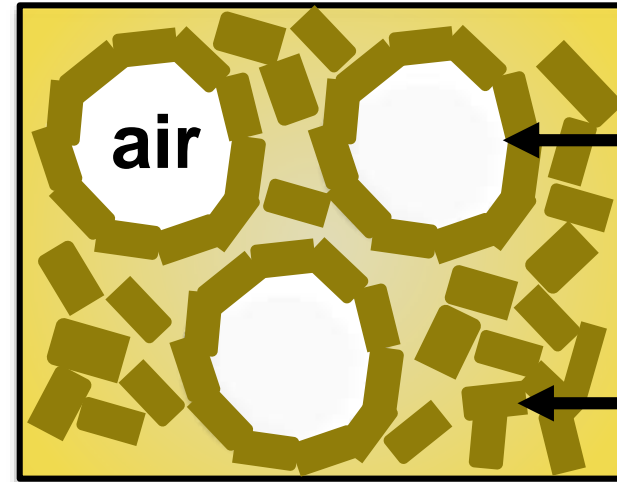
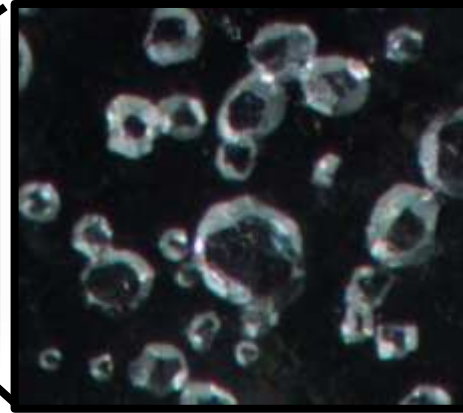
stearic acid



trihydroxystearine

# Effect of the particles shape?

## OIL FOAM



bubble

crystalline  
particle

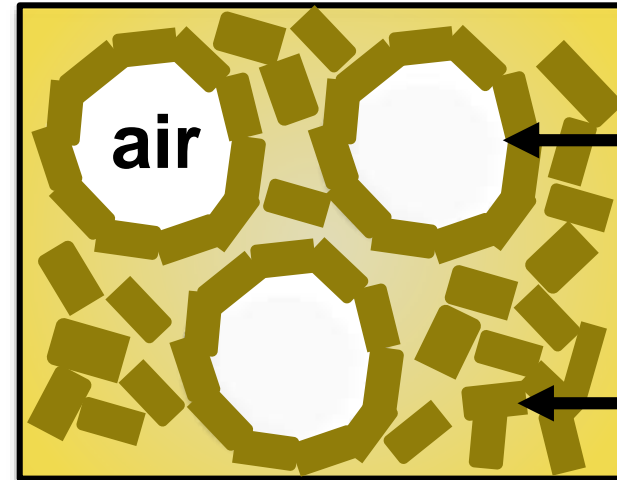
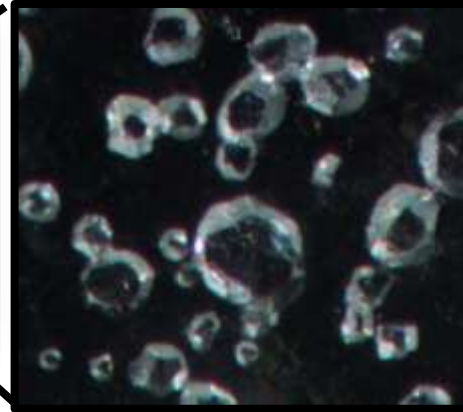
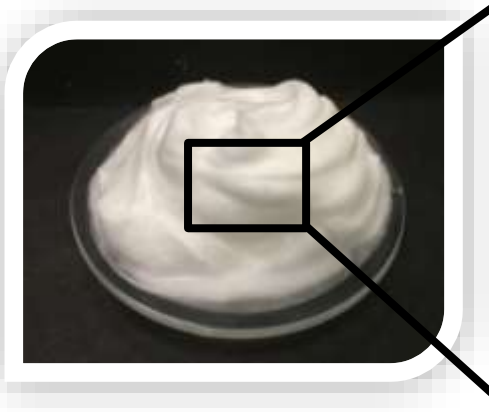
- Shape of the crystalline particles:



platelet

# Effect of the particles size?

**OIL FOAM**



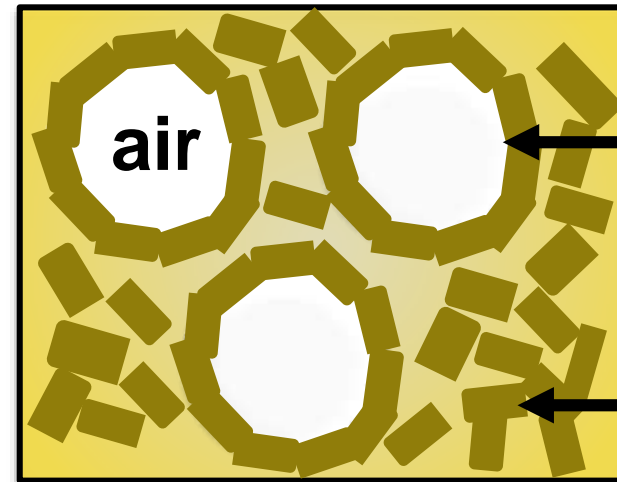
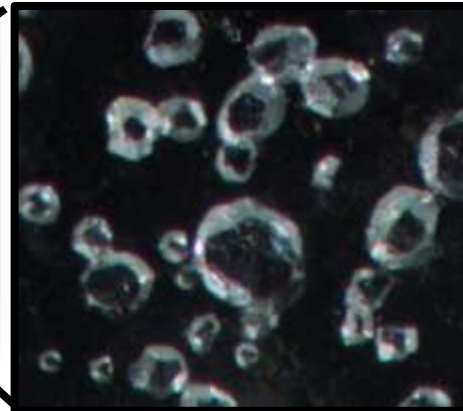
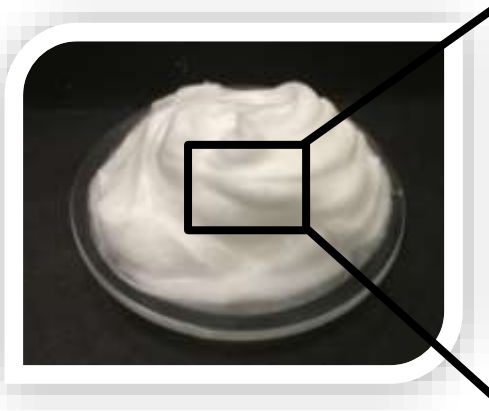
bubble

crystalline  
particle

**Size & shape of crystalline particles depend on: cooling rate, shearing rate & nature of fatty component.**

# Effect of the particles size?

OIL FOAM



bubble

crystalline  
particle

**Size & shape of crystalline particles depend on: cooling rate, shearing rate & nature of fatty component.**

**Fatty alcohol & Fatty acid**  
(stearic acid/stearyl alcohol)



platelet



Fast cooling



platelet



# Effect of the particles size?

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**Fatty alcohol** & **Fatty acid**  
(stearic acid/stearyl alcohol)



**Slow cooling**



**Fast cooling**

# Effect of the particles size?

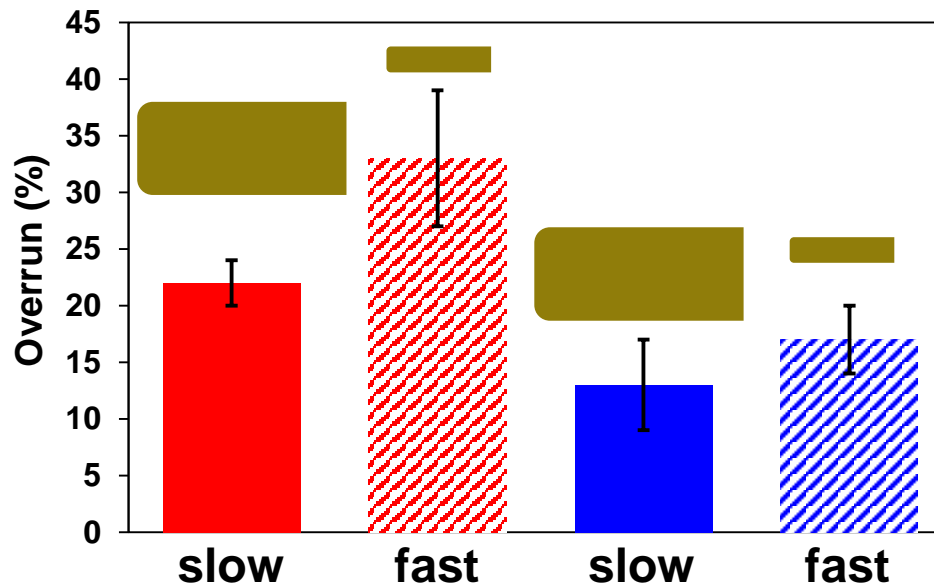
**Fatty alcohol & Fatty acid**  
(stearic acid/stearyl alcohol)



**Slow cooling**

**Fast cooling**

$$\text{Overrun (\%)} = \frac{V_{\text{foam}} - V_{\text{oil}}}{V_{\text{foam}}} \times 100$$



# Effect of the particles size?

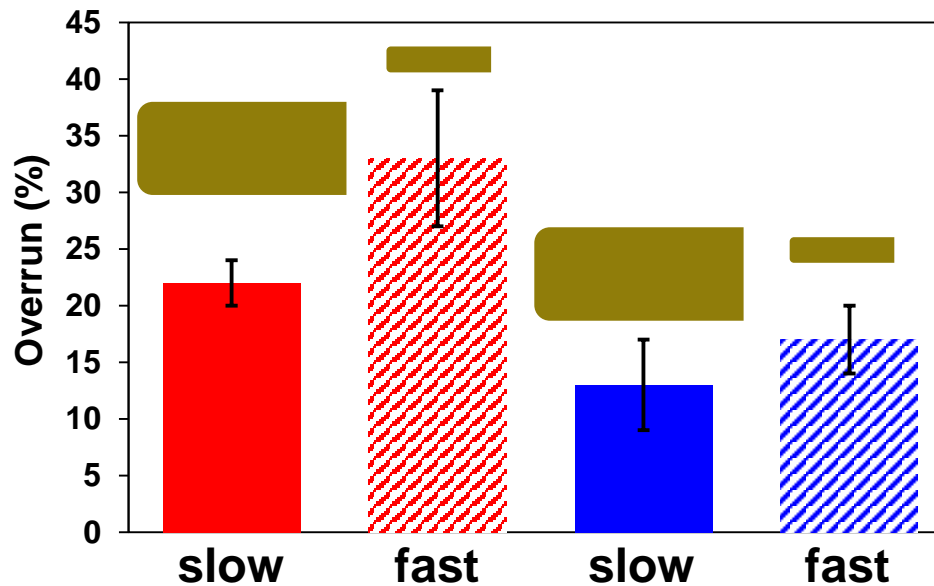
**Fatty alcohol & Fatty acid**  
(stearic acid/stearyl alcohol)



Slow cooling

Fast cooling

$$\text{Overrun (\%)} = \frac{V_{\text{foam}} - V_{\text{oil}}}{V_{\text{foam}}} \times 100$$

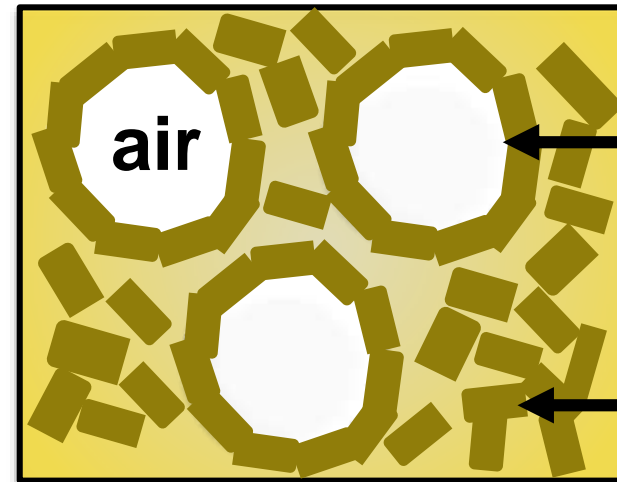
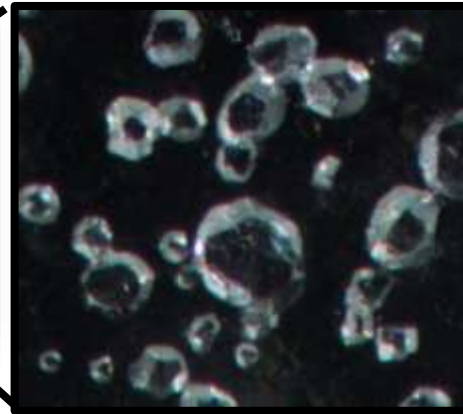
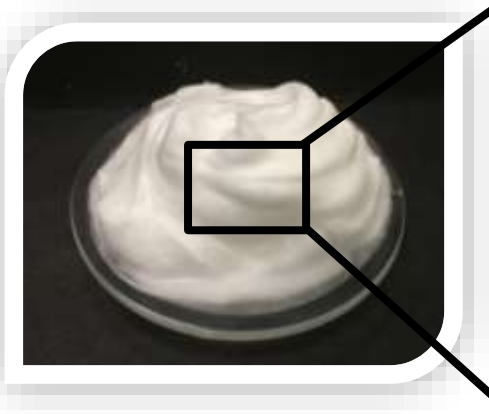


**Size, important parameter: small crystals higher overrun & foam stability**



# Formulation rules

## OIL FOAM



bubble

crystalline  
particle

- Crystalline particles with suitable **contact angle  $< 90^\circ$**

- Shape of the crystalline particles:



platelet



spherulite



fiber

- Size of the crystalline particles:



big

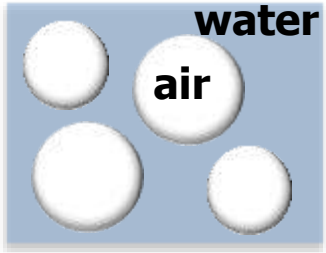


small

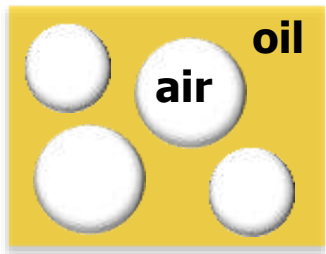
**Key parameters for crystalline particles depend on: cooling rate, shearing rate & nature of fatty component.**

# Outline

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## 1. Aqueous foams



## 2. Differences between aqueous & non-aqueous foams



## 3. Formulation rules for edible oil foams



## 4. Effect of co-crystallization on oil foam properties

# Effect of co-crystallization?

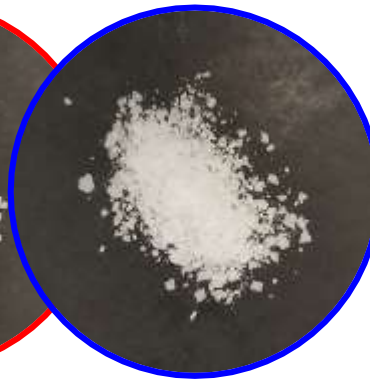
90 wt.%  
Sunflower oil



Stearyl  
alcohol



Stearic  
acid



10 wt. %

R = SO:SA



Oleogel

Oil foam



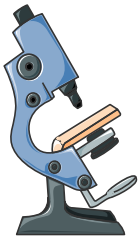
# Effect of co-crystallization?

---

**R= 10:0**

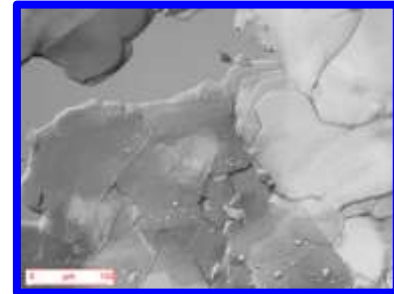


**Fatty Alcohol**



DIC  
x20

**R = 0:10**



**Fatty Acid**

# Effect of co-crystallization?

**R = 10:0**

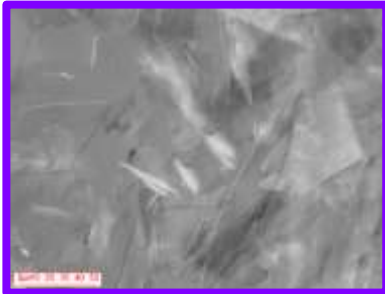


**Fatty Alcohol**

**R = 5:5**



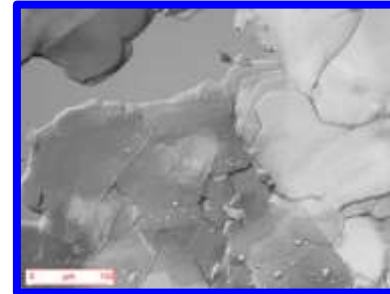
**R = 3:7**



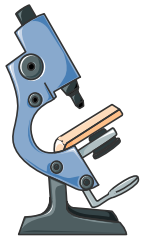
**R = 2:8**



**R = 0:10**



**Fatty Acid**



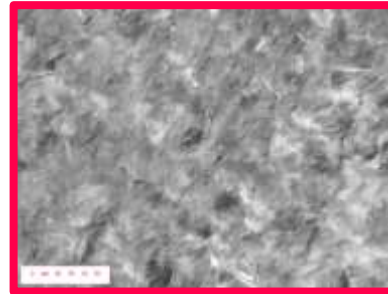
DIC  
x20

# Effect of co-crystallization?

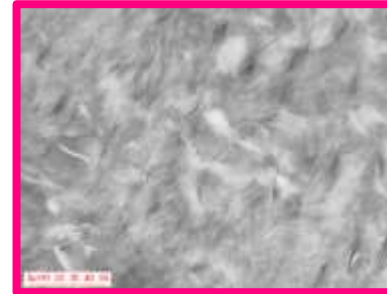
**R = 10:0**



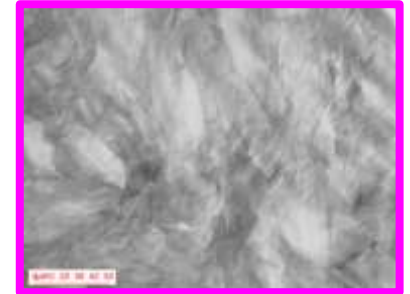
**R = 8:2**



**R = 7:3**

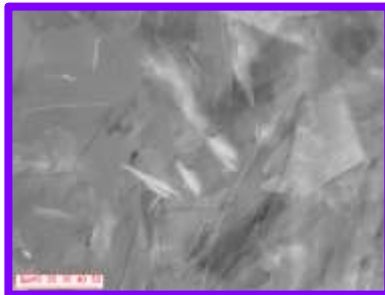


**R = 5:5**



**Fatty Alcohol**

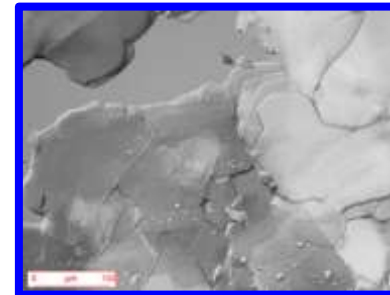
**R = 3:7**



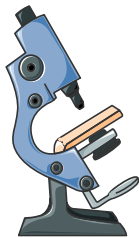
**R = 2:8**



**R = 0:10**



**Fatty Acid**



DIC  
x20

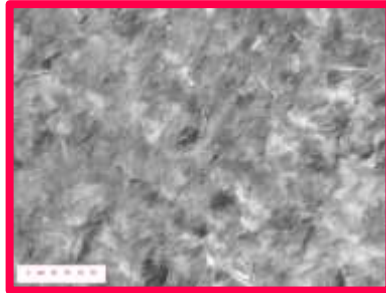
# Effect of co-crystallization?

**R = 10:0**

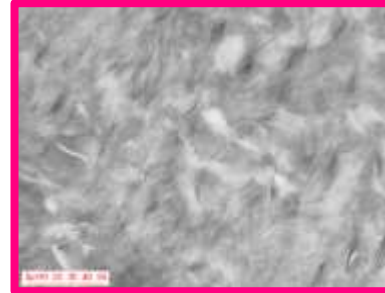


**Fatty Alcohol**

**R = 8:2**



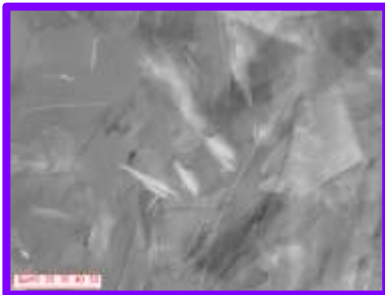
**R = 7:3**



**R = 5:5**



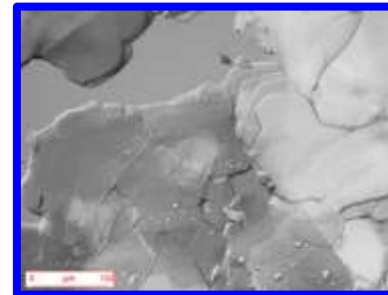
**R = 3:7**



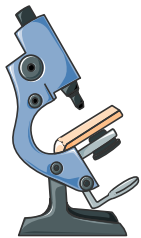
**R = 2:8**



**R = 0:10**



**Fatty Acid**

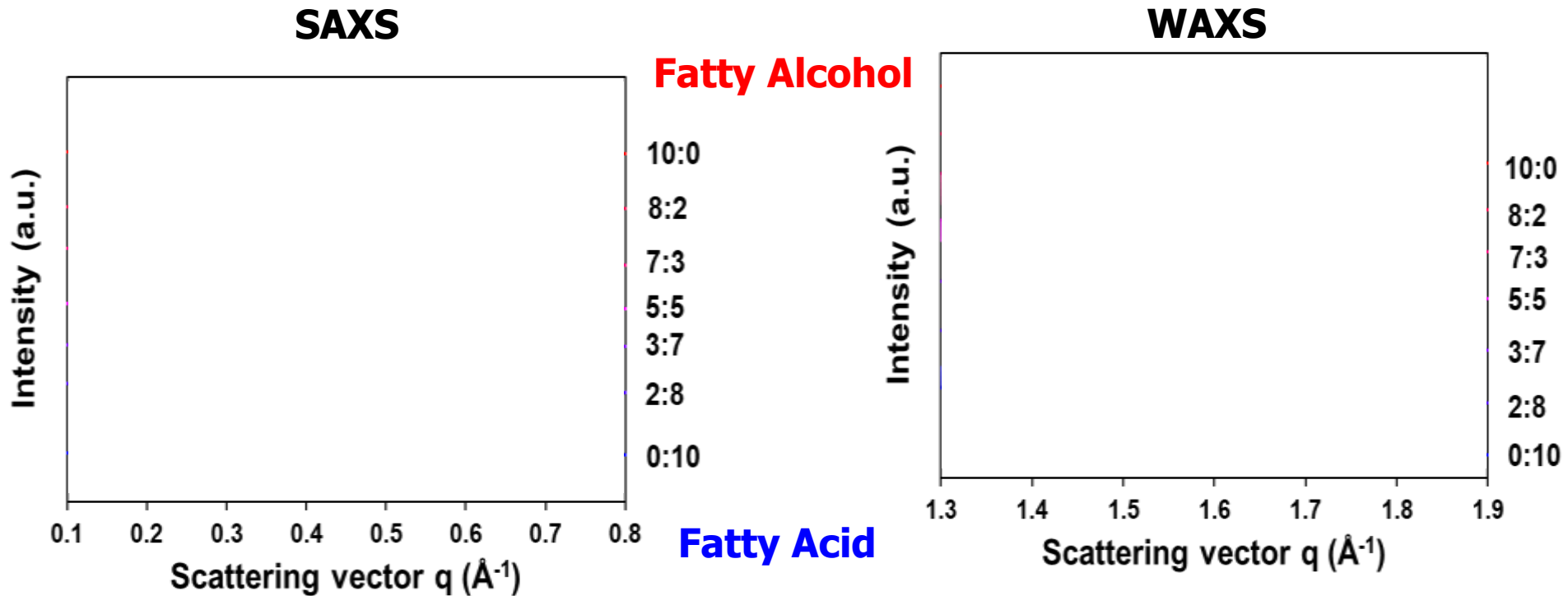


DIC  
x20

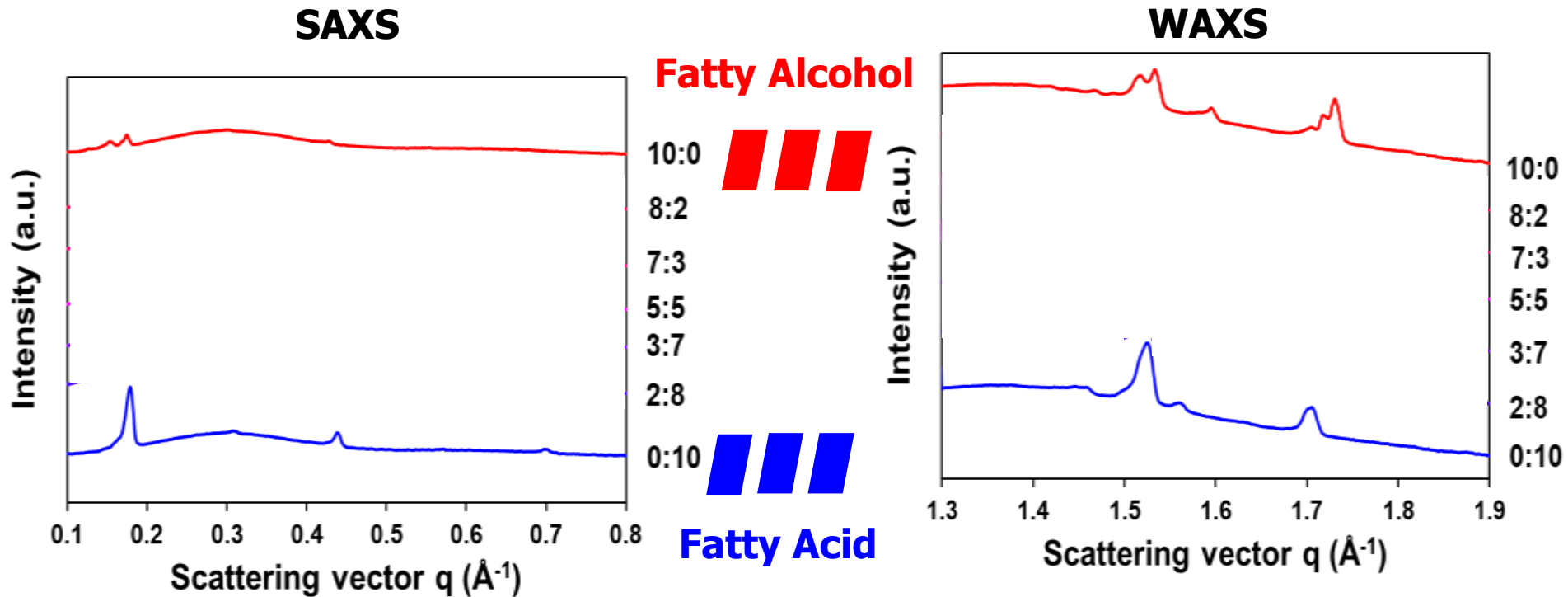
**Platelet-shape for all R, but the size of crystals varies with R:  
two R with very small crystals**



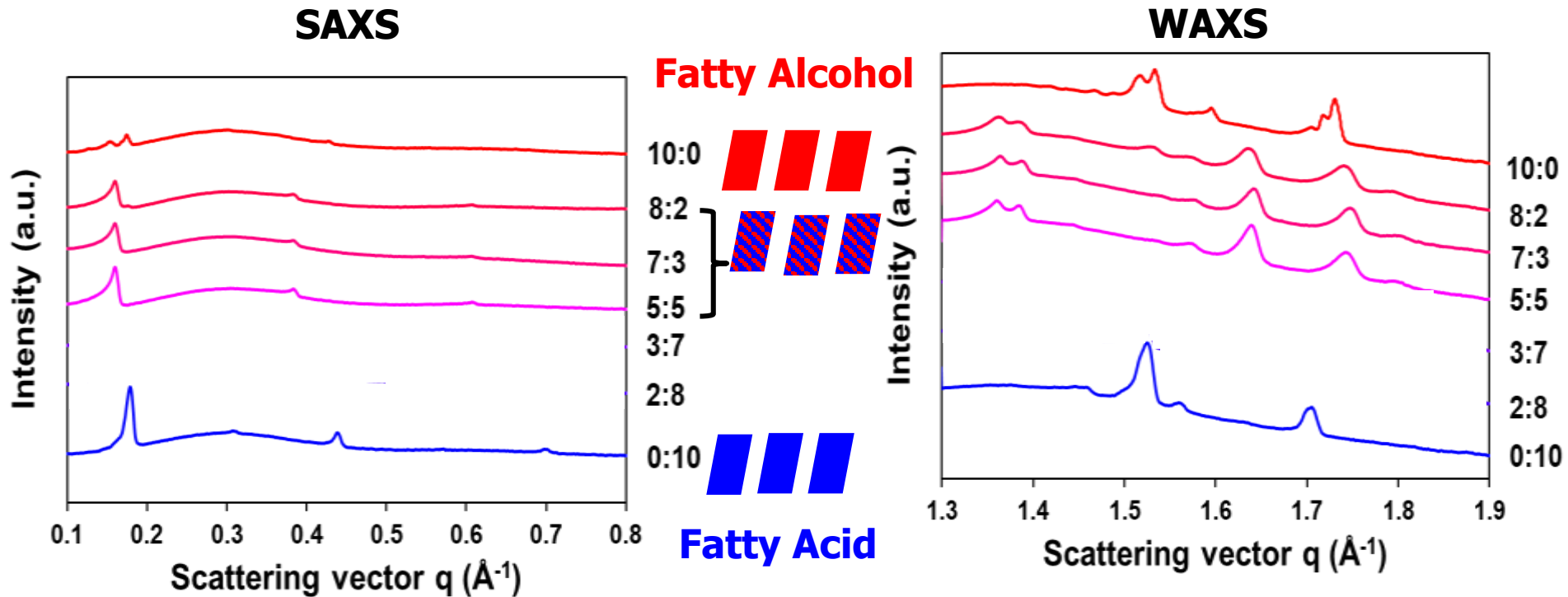
# Effect of co-crystallization?



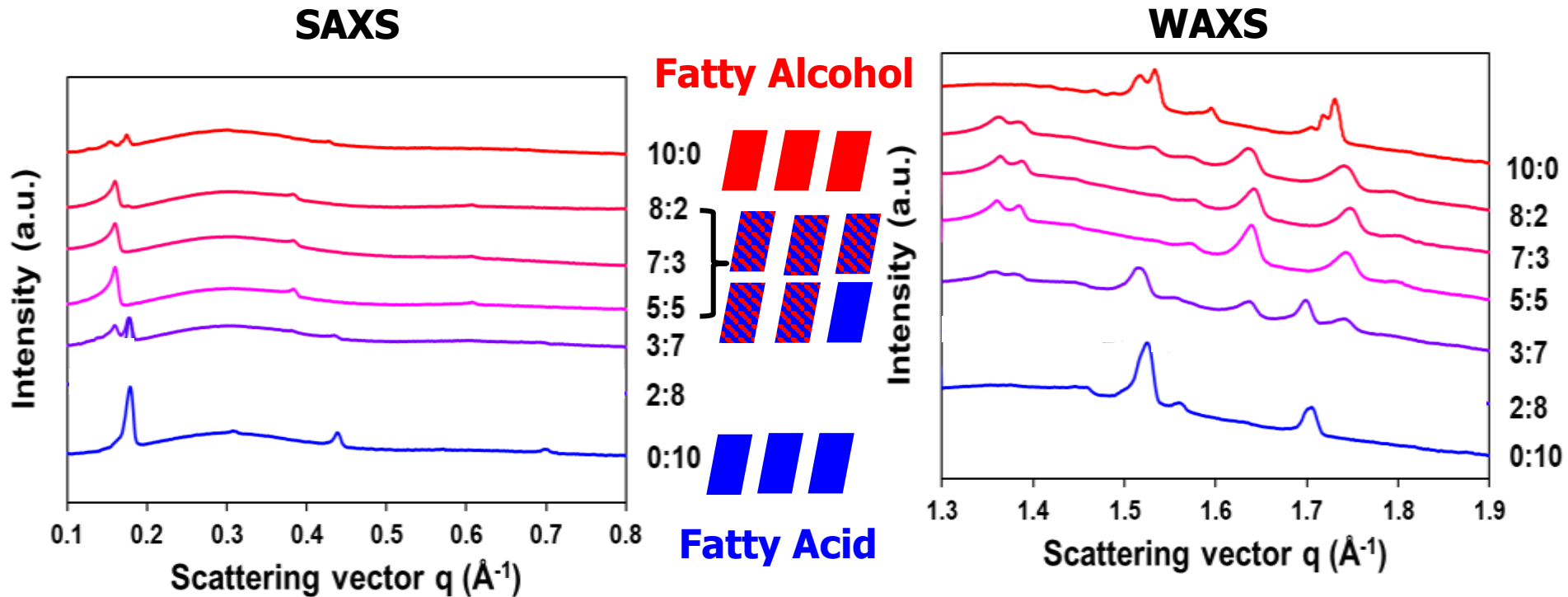
# Effect of co-crystallization?



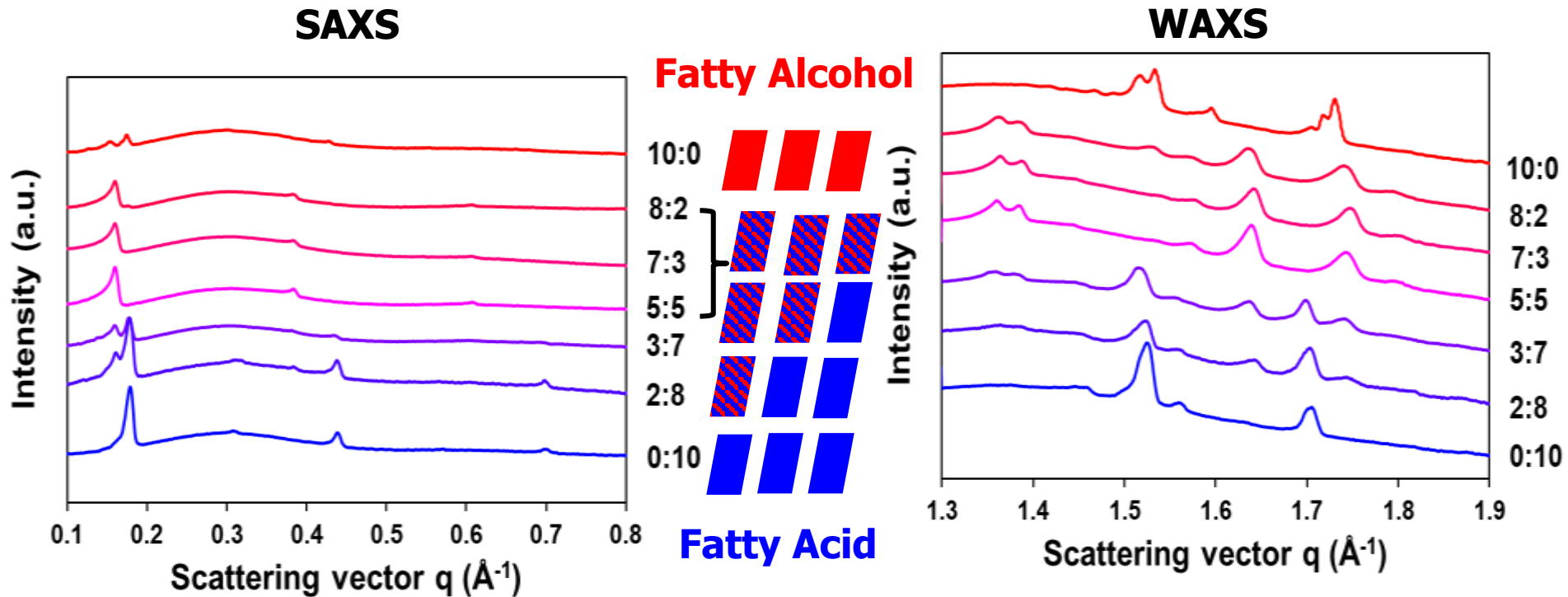
# Effect of co-crystallization?



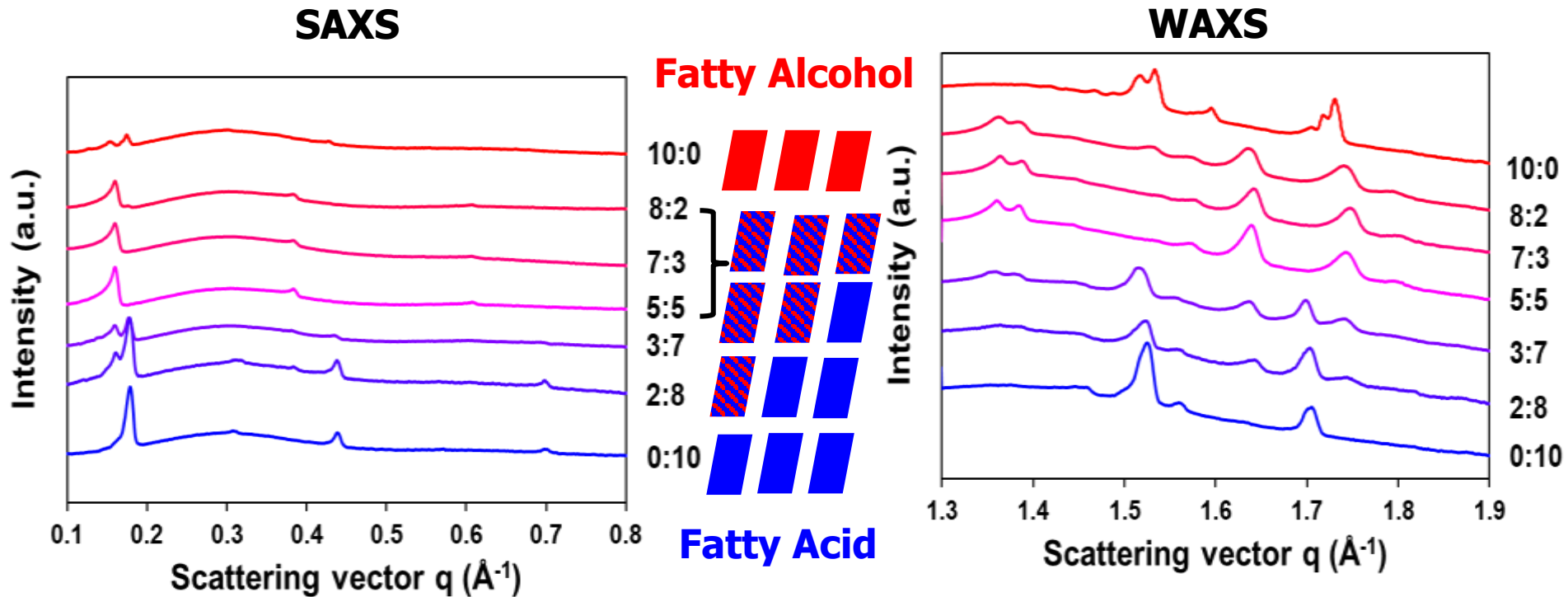
# Effect of co-crystallization?



# Effect of co-crystallization?



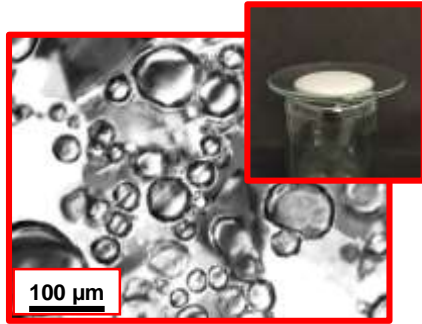
# Effect of co-crystallization?



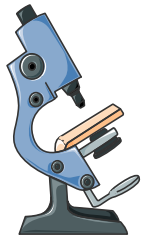
**Mixed crystals for R= 8:2, R = 7:3 with the smallest crystals**

# Effect of co-crystallization?

**R= 10:0**

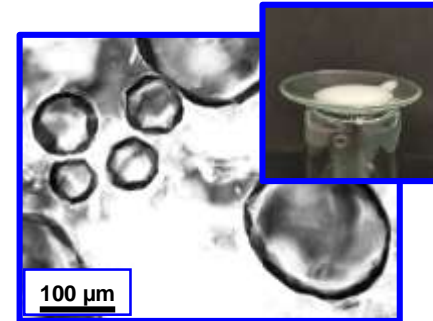


**Fatty Alcohol**



DIC  
x20

**R = 0:10**

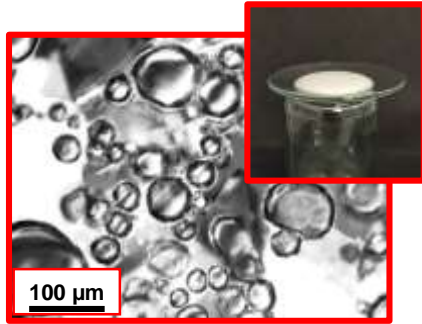


**Fatty Acid**



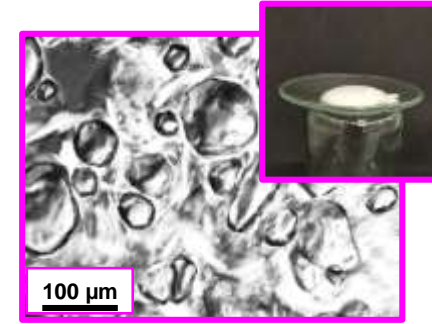
# Effect of co-crystallization?

**R = 10:0**

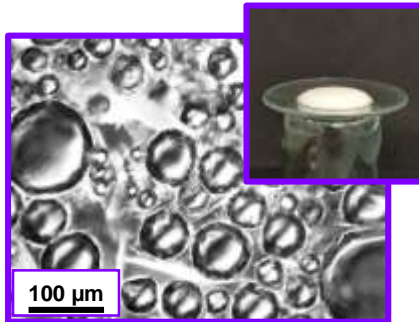


**Fatty Alcohol**

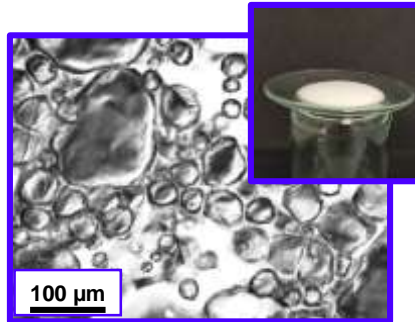
**R = 5:5**



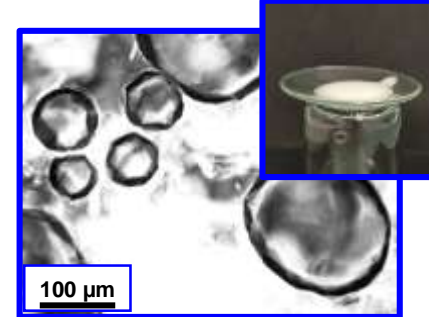
**R = 3:7**



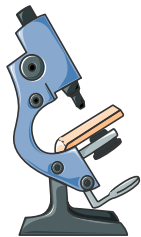
**R = 2:8**



**R = 0:10**



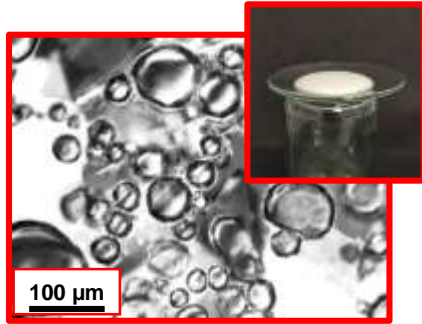
**Fatty Acid**



DIC  
x20

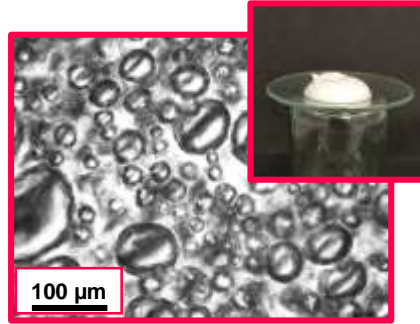
# Effect of co-crystallization?

**R = 10:0**

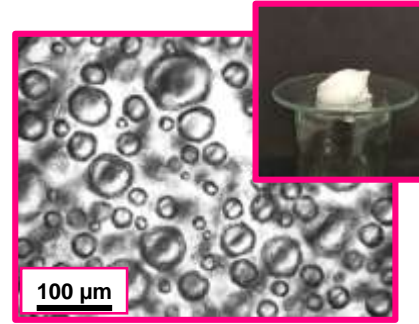


**Fatty Alcohol**

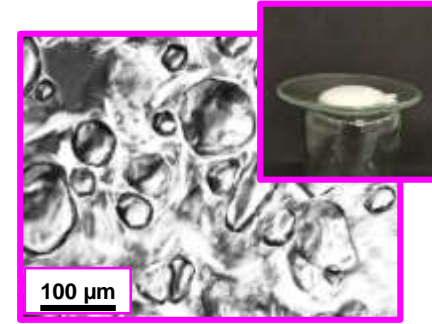
**R = 8:2**



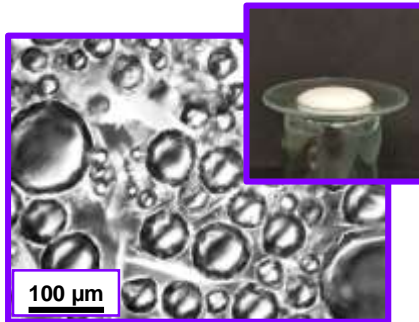
**R = 7:3**



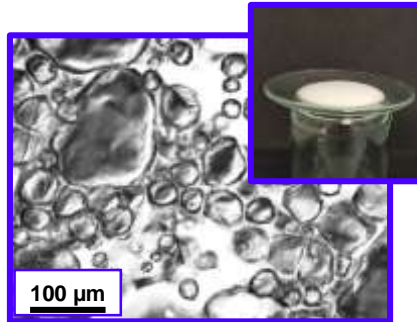
**R = 5:5**



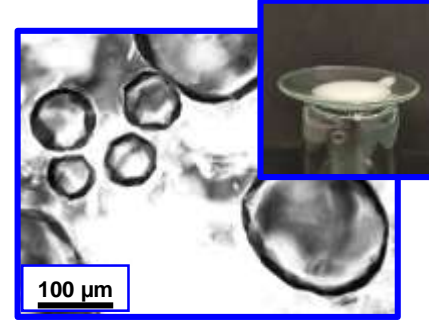
**R = 3:7**



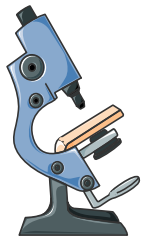
**R = 2:8**



**R = 0:10**



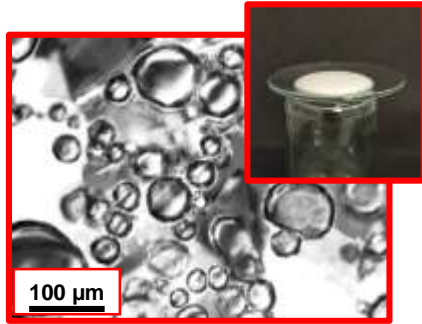
**Fatty Acid**



DIC  
x20

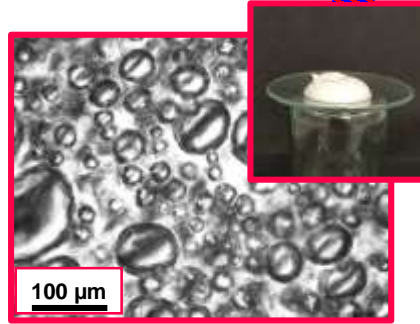
# Effect of co-crystallization?

**R = 10:0**

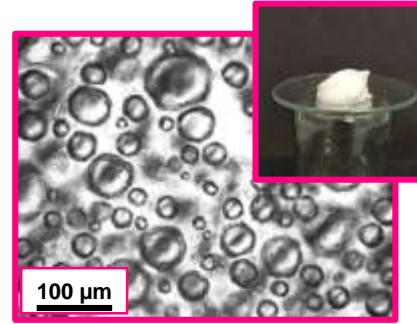


**Fatty Alcohol**

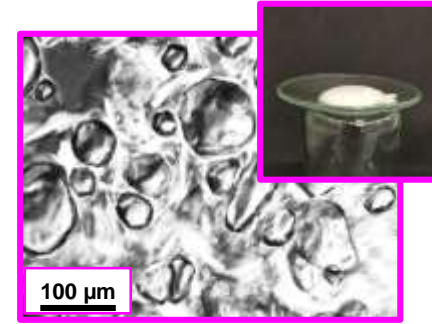
**R = 8:2**



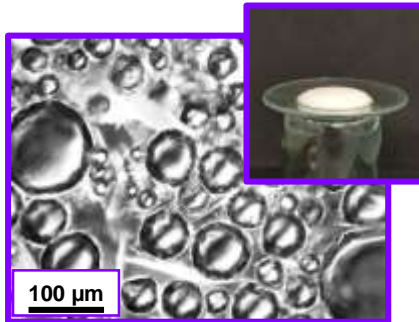
**R = 7:3**



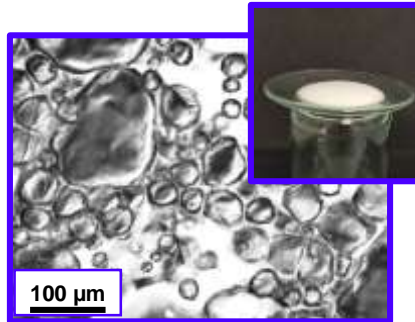
**R = 5:5**



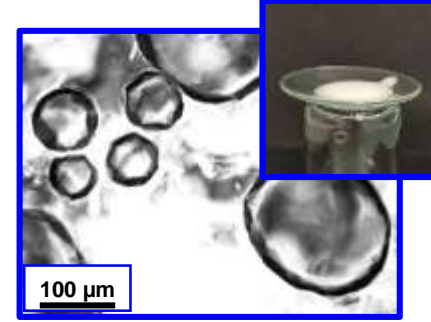
**R = 3:7**



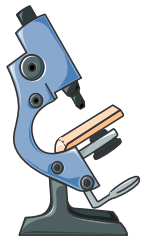
**R = 2:8**



**R = 0:10**



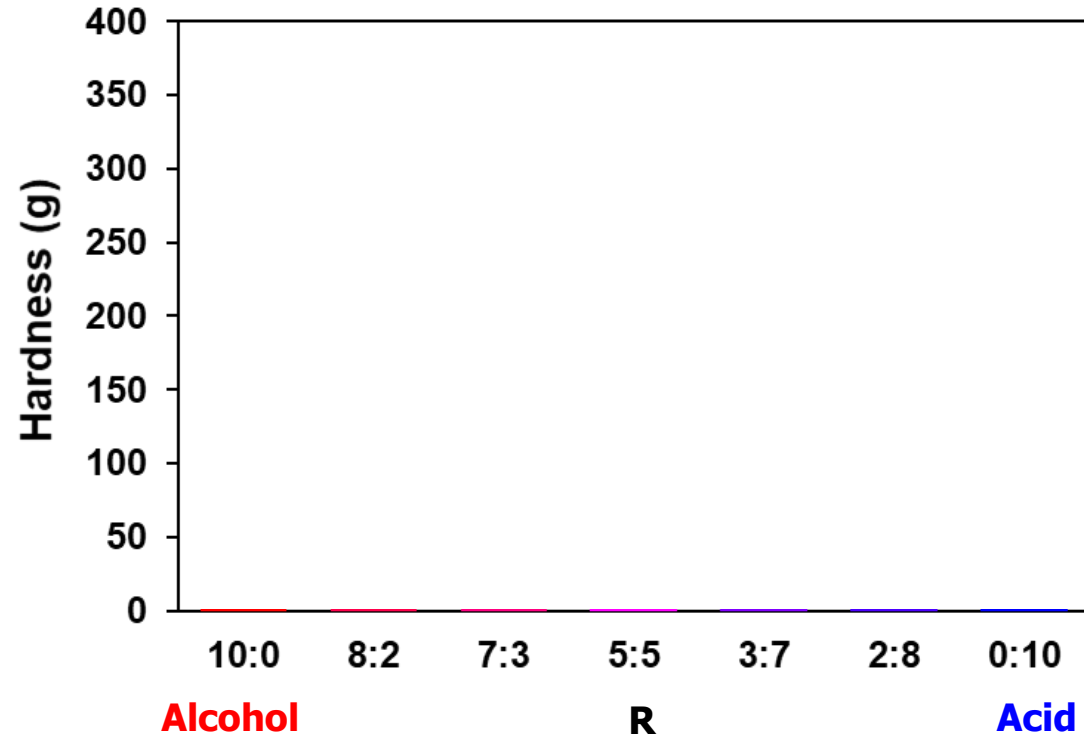
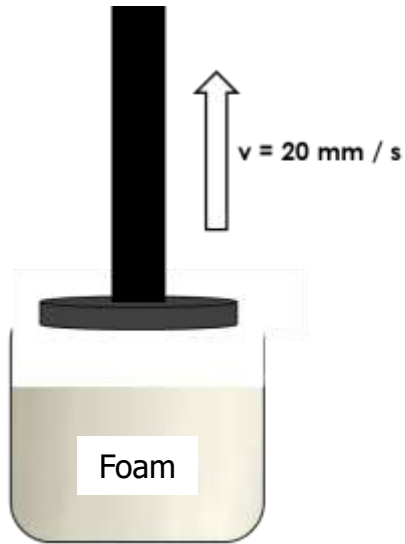
**Fatty Acid**



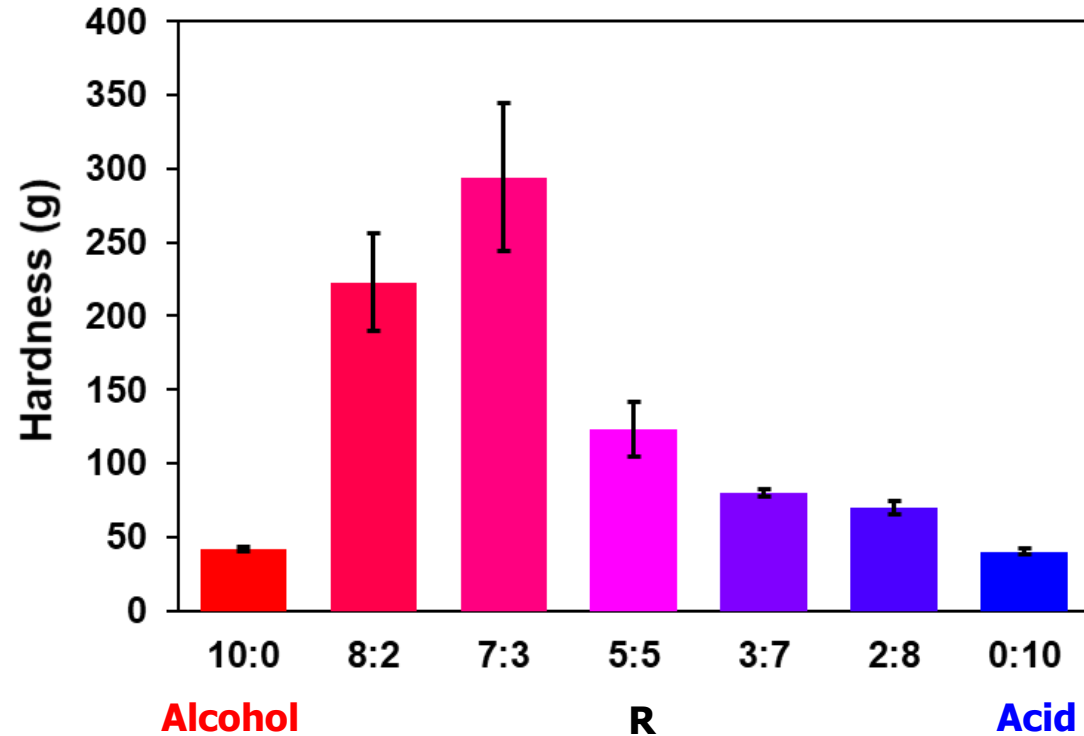
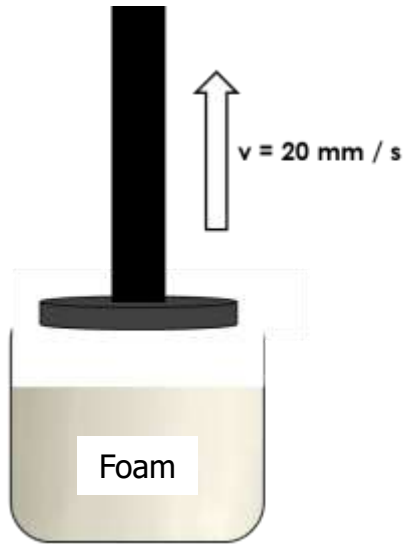
DIC  
x20

**High Overrun and small bubbles for mixed crystals  
(R = 8:2 & R = 7:3)**

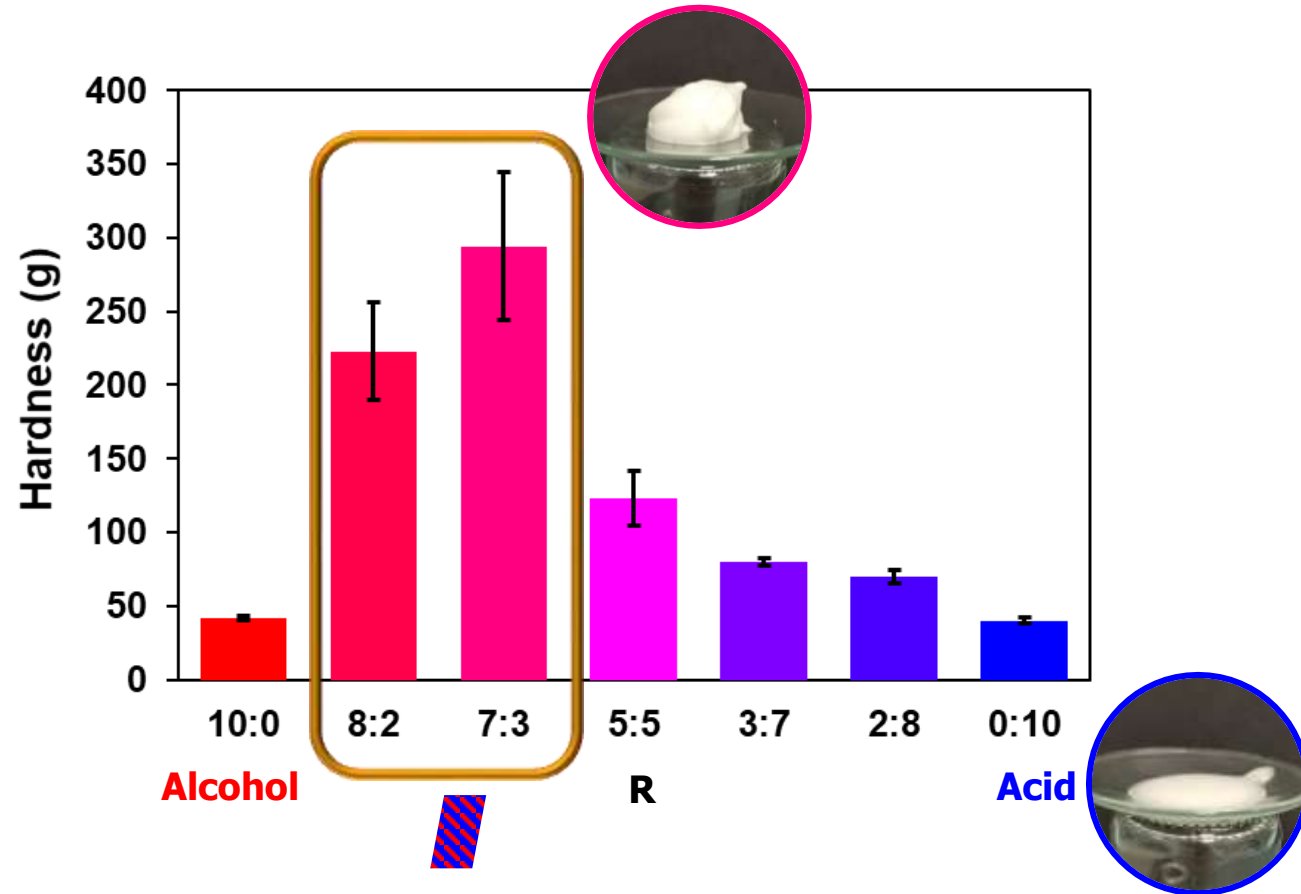
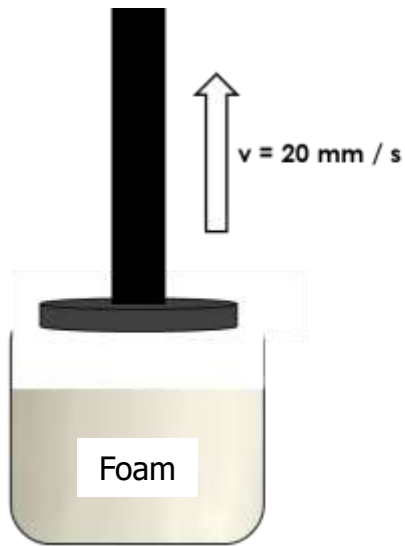
# Effect of co-crystallization?



# Effect of co-crystallization?



# Effect of co-crystallization?

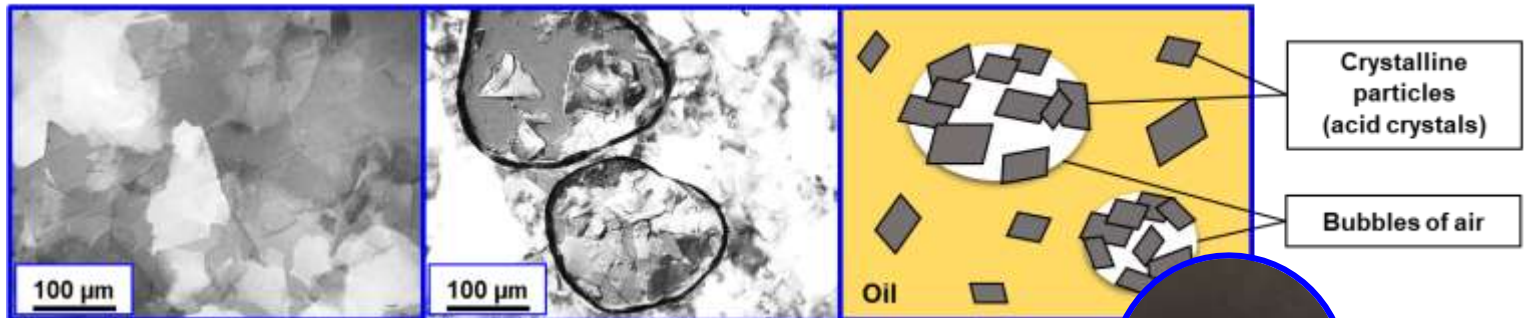
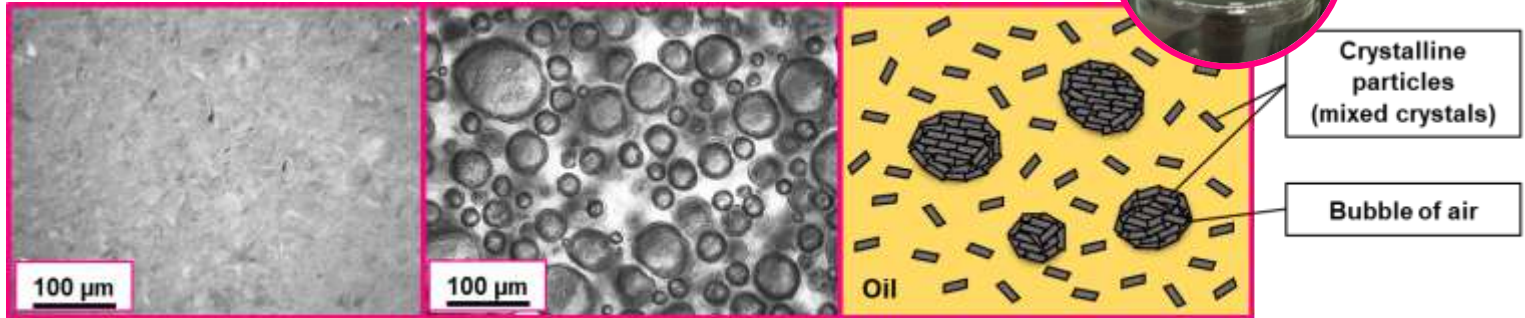


**High hardness of foams for mixed crystals  
(R= 8:2 & R = 7:3)**



# Effect of co-crystallization?

**R = 7:3**



**R = 0:10**

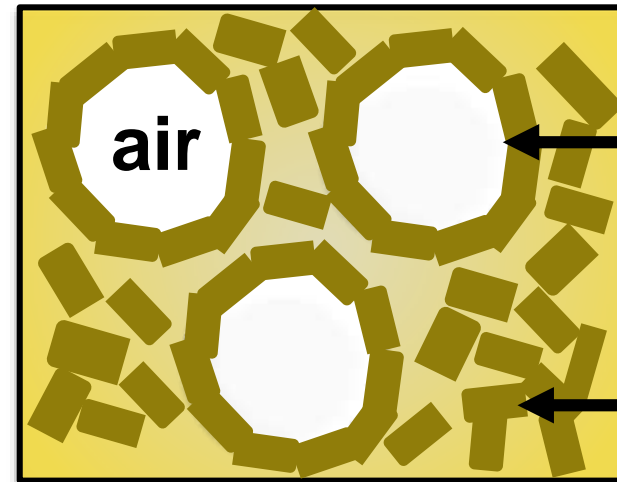
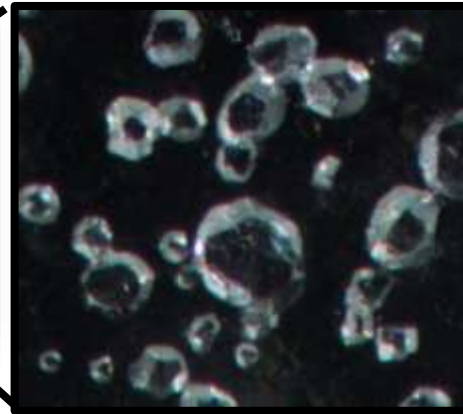
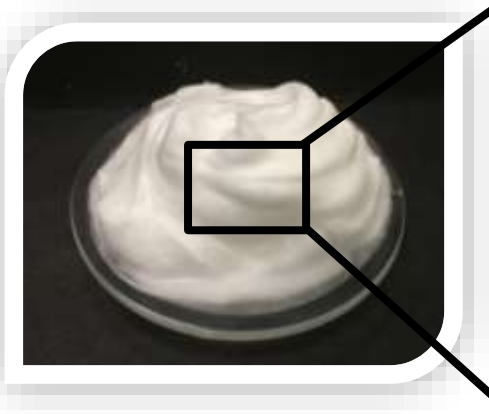


**Co-crystallization of fatty acids/fatty alcohols improves foam properties by the presence of small mixed crystals in high quantity**








# Conclusion

## OIL FOAM



bubble

crystalline  
particle

- Crystalline particles with suitable **contact angle  $< 90^\circ$**
- Shape of the crystalline particles:
  -   
platelet
  -   
spherulite
  -   
fiber
- Size of the crystalline particles:
  -   
big
  -   
small
- Co-crystallization: **increase the quantity & decrease crystals size**

# Acknowledgments

---

**Orlin Velev (NCSU, USA)**  
**Stephanie Lam (NCSU, USA)**

**NC STATE UNIVERSITY**

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**Cédric Gaillard (INRAE, France)**



**Arnaud Saint-Jalmes (IPR, France)**



**Marion Callau (L'Oréal, R&I)**

