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Understanding and predicting the performances of concentration processes in the dairy industry

Research Activities in Process Engineering

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Valparaiso, 6th June 2012
INRA, The French Institute for Agricultural Research (www.inra.fr)

- 1946 → National public scientific and technological establishment under the joint authority of ministries of agriculture and research

- Three main fields: agriculture / food / sustainable development

- Ressources
  - **Scientific resources**
    - 3,800 researchers and scientists
    - 4,800 technicians and research support personal
  - **Links to University and Higher Education**
    - 780 teacher-researchers associated with INRA in its joint research units
  - **Ample training potential**
    - 1000 young trainee-researchers
    - Over 1000 student trainees, grant holders and foreign researchers / each year

- Nation – wide presence (22 centres, each of them divided into several sites (200 hundreds) and experimental units (70))

- Second largest French public research organisation

- Largest European organisation for agricultural research
Research Unit : Science and Technology of Milk and Egg (STLO)

Director : J. Léonil
75 permanents
35 scientists
≈ 22 PhD
+ 7 research teams from the industry
≈ 135 people
- **Milk production**
  - France (2nd range in Europe after Germany)
    - 24 millions of tons (Mt) of milk / year
    - 160 000 employees
  - « Grand Ouest »: 47 % French milk collected (11.3 Mt)
    = Netherlands or Italy
    = half the volume produced in Oceany
  - Brittany; 20 000 producers
    21% French milk collected (5 Mt)
    = production of Denmark or Ireland
Our objectives

Produce knowledge about

Milk components

Food / Ingredients properties

Operation units / Processes
(membrane separation; spray drying; cheese processing; heat treatment)

Functionalities
(foaming, emulsifying, gelling properties)

Digestibility
(nutrition)

Food industry

Consumer, Human being

To control the final quality of products (safety, functional, sensorial and nutritional)
To contribute to create new products / concepts (competitiveness of the industry)
Science and Technology of Milk and Egg

~ 10 permanent people
Dairy Processing team: Background

Fractionation and concentration processes ➔ Preparation and diversification of dairy functional ingredients

Membrane Separation

Spray Drying

- ≈ 40% of the membrane area in food industry

- ≈ 50% of Milk DM ➔ Powder
- ≈ ¼ energy consumption of the dairy industry

Low control of the process ➔ Difficulties
in prediction of the processing parameters
in reduction of environmental impacts
and in control of the quality / characteristics of the products!
Dairy Processing team: Background

**Membrane separation**
- Tangential flow
- Solvent transfer
- Pressure
- Retentate
- Permeate

**Spray Drying**
- Concentrate
- Inlet Air
- Heat transfer
- Water transfer
- Powder

**Properties of the concentrates**

**Performances of the process**
[Productivity, Properties (functional, physical, biochemical, nutritional, …), Environment]
Composition of bovine milk

- Water (870-875)
- Lactose (48-50 g/L)
- Fat (34-44 g/L)
- Minerals (8-9 g/L)
- Proteins (32-35 g/L, expressed as N × 6.38)

Milk components include:

- Fat micelles
- Soluble proteins
- Caseins (80%)
- Soluble proteins (20%)

Casein Micelles: Soft and dynamic colloid

SEM Image (Dalgleish, 2004)

Size distribution (Regnault, 2004)

Structure (McMahon, 2008)
MF 0.1 µm of milk: separation casein micelles / serum proteins

Whole or skimmed milk + Microfiltration (1.4 µm)

**MF 0.1 µm**
Fractionation casein micelles / soluble proteins

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**Retentate**
Native casein micelles

According to VRR:
- casein
- DM, TNM, Ashes, Ca
- Lactose, soluble proteins (%DM)

**Permeate**
Native whey proteins

- pH 6.6
- Lactose
- Ashes
- Soluble proteins

"ideal whey"
Free of residual fat, micro-organisms & CMP
Natural pH of milk
The skimmed milk microfiltration (0.1 µm)

\[ J = 76 \text{ L h}^{-1} \text{ m}^{-2} \]
\[ \tau_w = 100 \text{ Pa} \]

Permeation flux, \( J \)

\[
\begin{array}{c|c|c|c|c|c|c|c|c}
\hline
\text{Wall shear stress, } \tau_w (\text{Pa}) & 0 & 20 & 40 & 60 & 80 & 100 \\
\hline
\text{Filtration time (s)} & 0 & 5000 & 10000 \\
\hline
\end{array}
\]

Fouling resistance \( R_f/R_m \) - \( \beta \)-LG transmission

\[
\begin{array}{c|c}
\hline
\text{Permeation flux, } J \\
\hline
0 & 20 & 40 & 60 & 80 & 100 \\
\hline
\end{array}
\]

\[
\begin{array}{c|c}
\hline
\text{Wall shear stress, } \tau_w (\text{Pa}) & 0 & 20 & 40 & 60 & 80 & 100 \\
\hline
\text{Filtration time (s)} & 0 & 5000 & 10000 \\
\hline
\end{array}
\]

Divergent runs

Le Berre et Daufin, 1996; Gésan-Guiziou et al., 1999; 2000

Ceramic membrane 0.1µm, UTP system, VRR=2, T= 50°C
Objectives

1. To characterize the concentrates and the colloid interaction involved in concentrated regime (independly of the process / isotrope conditions)

2. To characterize, in a non invasive manner, the concentrated structure during the process (*in-situ* characterization/ anisotrope conditions)

3. To adapt / define predictive models relating properties of the concentrated zones and process performances

4. To validate these models at the local and global scales

Strategy

To Control, Improve and Predict the process performances and the product properties

**Properties of the concentrates (proteins)**

- Microscopic (local) scale
- Macroscopic (global) scale

**Process Performances**

(+ Impact of the processes on structural and functional properties of the products)
Objectives

Properties of the concentrates (proteins)

Process Performances

Original Approach

Multi-disciplinary & Multi-scale Approach

Process Engineering

+ Physico-chemistry

+ Soft Matter Physics

+ Impact of the processes on structural and functional properties of the products
The concentration of casein micelles

Why?
- Major protein of bovine milk (80% of milk protein)
- Little information on the behaviour of native casein micelle in concentrated regime
- Major contributor of deposit in skimmed milk filtration

1- To characterize the concentrates and the colloid interaction involved in dense regime

Performance of 0.1 μm microfiltration of skim milk

Characteristics of accumulated layers of micelles

J. Membrane Sci., 2011
The concentration of casein micelles

Why?
- Major protein of bovine milk (80% of milk protein)
- Little information on the behaviour of native casein micelle in concentrated regime
- Major contributor of deposit in skimmed milk filtration

Questions
- What are the interactions involved?
- What are the phenomena that rule the phase transition?

Originality
- Use of tools and concepts of « soft matter physics » to study a complex biological supramolecular structure

1- To characterize the concentrates and the colloid interaction involved in dense regime

Osmotic stress

=> Quantification of interactions
Preparation of highly concentrated dispersions (C>500 g/L)
Methodology – Casein micelles characterization

. Osmotic stress technique


Solvent
(from skimmed milk ultrafiltrate "UF" pH 6.7)

Stressing polymer
(PEG35000)

Dialysis bag
Spectra-Por 12-14 kD

π = π_{PEG}

Equilibrium → π_{bag} = π_{PEG}
(7-30 days)

= casein micelle from "native phosphocaseinate powder", NPC

=> Quantification of interactions

Preparation of highly concentrated dispersions (C>500 g/L)

π reflects the balance of all interactions ("equation of state" of a colloidal system)
The concentration of casein micelles

Equation of state and rheological behavior

Σ interactions

Rheology

3 regimes of compression

1. Liquid -> polydisperse hard-sphere fluid
2. Transition -> ~ close packing (f = 0.7 - 0.9)
3. Gel -> deformation, deswelling of micelle
   -> “soft” and compressible colloid

1- To characterize the concentrates and the colloid interaction involved in dense regime

- Determination of phase transition
- Access to \( \pi(C) \) => Modeling

**In-situ characterization of a deposit in filtration**

**Why?**
- Data needed for the validation of model
- Little work in that domain

**Questions**
- What are the kinetics of accumulation at the membrane surface?
- What are the structure and organisation of the matter in these concentrated layers?

**Originality**
- Coupling of micro-system of filtration with observation and characterization method (small-angle X-ray scattering), SAXS
- Spatio-temporal evolution

To characterize, in a non-invasive manner, the concentrated structure during the process.
In situ characterization of a deposit in filtration

2- To characterize, in a non invasive manner, the concentrated structure during the process

Concentration profile at the membrane surface

- Access to spatio-temporal evolutions of the concentration and structure of micelles in the accumulated layers at the membrane surface
- Promising methodology => to better understand and model filtration

Langmuir, 2008
Prediction of spray drying parameters

Why?

- No methods to determine \textit{a priori} & precisely the parameters of spray drying for food products ➞ necessity of expensive and time consuming experiments
- One major difficulty: drying is non isenthalpe (≠ drying of « pure » water in ideal conditions)

**Reasons:**

- **Energetic losses**
- Need to take into account water availability = \( f(\text{product}) \)

The water in the product is « bounded » to constituents ➞ the removal of water requires extra energy ➞ Decrease in drying performance

Questions

- How to evaluate the energy required to compensate the association of water with the product ?
- How can we predict the spray drying parameters ?
Prediction of spray drying parameters

Method of drying by desorption

Desorption behavior

Energy required to compensate the association of water with the product

+ Overall mass and energy balances established over the entire spray dryer ⇒ prediction of parameters ± 1-5%

Drying Technol. 2011
J. Food Eng. 2009

3- To adapt / define predictive models relating properties of the concentrated zones and process performances

Software SD²P: Spray Drying Parameters Simulation & Determination
(20 licences sold (25% in foreign countries); 14th / 360 licences INRA (software, patents, know-how))
Our specificities / strengths

- Development of specific analytical tools / methodologies
  - Osmotic stress technique
  - Micro-system of filtration coupled with SAXS
  - ‘Dryability’ of dairy and food concentrates (drying by desorption)
    ➔ 1 software SD²P: Spray Drying Parameters Simulation & Determination

- Availability of analytical tools for the characterization of milk and dairy products
  - Rheometers, AFM, FRAP, NMR, Mass spectroscopy, membrane osmometer, …
  - + all classical analyses of dairy products: Chromatography, zeta sizer, Kjeldahl…
  - X-ray with Synchroton Soleil (Gif / Yvette), …

- Availability of well equipped and characterised pilots

➔ Dairy Technology Platform
Dairy Technology Platform

Platform: wide and flexible range of processing technologies

- Heat treatment (pasteurization to UHT-sterilization)
- Homogenization, cream separation, melting...
- Membrane separation modules:
  - Microfiltration
  - Ultrafiltration
  - Nanofiltration and Reverse osmosis
- Two cheese production lines (soft- and hard-type cheeses)
- Spray drying facilities: pilot (1-7 kg of water per hour) and semi-industrial (BIONOV) units
Dairy Technology Platform—MF equipments

- **Microfiltration (5 pilots)**
  - Mineral membranes /organic
  - Pore diameter: 0.1 µm - 12 µm
  - Membrane area: 0.24 m² - 4.6 m²
  - Capacity: 50 L - 2000 L et +

MF Pilot equipped with sensors, gauges
Real data monitoring and storage by a computer

Pilot (S 0.24 m², Vm 4.5 L)

Pilot (S 4.6 m², Vm 52 L)
To understand / follow the triple effect (temperature, RTD and concentration) on the physico-chemical qualities of the dairy concentrate

Pilot workshop: Research and development for vacuum evaporation

One stage falling film vacuum evaporator at 30 kg of water h⁻¹

Vacuum evaporator (GEA-PE)
Dairy Technology Platform– spray dryers

Spray Dryer MOBILE MINOR™

- Spray drying tower
- 5-10 kg of water.h⁻¹
- Equipped of a lot of sensors in all the process lines
  - Air: T°C, Hygrometry, HR, HA, Air flow rate, ...
  - Product: Temperature, Masse flow rate, Viscosity (Extraction Pump Intensity)

- To understand / follow the water transfer of the droplet to the air during spray drying in relation to the biochemical composition
Semi-industrial pilot

Pilot workshop: Research and development for evaporation / drying

Two stage falling film vacuum evaporator at 300 kg of water.h⁻¹

"MSD type" drying tower 80 kg of water.h⁻¹
More than

10 millions tons dairy powders per year;

Only 1 International Symposium on Spray Dried Dairy Products!
More than 10 millions tons dairy powders per year;

Only 1 International Symposium on Spray Dried Dairy Products!

IDF / INRA International Symposium on Spray Dried Dairy Products

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http://colloque.inra.fr/sddp2012

St-Malo, FRANCE
19-21 June 2012
Thank you for your attention!

http://www.rennes.inra.fr/stlo