A MULTISCALE STUDY OF POWDER RECONSTITUTION

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Tristan FOURNAISE

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Planning of PhD work and achieved deliverables

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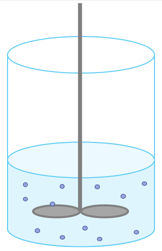
1. Screening of a large variety of powders

RECONSTITUTION PROCESS

Wetting → Swelling → Dispersion → Dissolution



→ RECONSTITUTION KINETICS BY GRANULOMORPHOMETRY

→ POWDER RECONSTITUTION STEPS BY CONDUCTIMETRY & MACRO-IMAGING

→ POWDER CLASSIFICATION

- Easy
- Intermediate
- Poor

Year 1 (January 2019 – January 2020)

(1) Powder classification according to their reconstitution behavior

(2) For selected powders: reconstitution kinetics in different conditions of temperature, stirring…

(3) Statistical correlations between the numerous powder characteristics and their reconstitutability

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1. Screening of a large variety of powders

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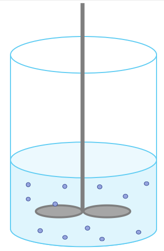

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→ POWDER RECONSTITUTION STEPS BY CONDUCTIMETRY & MACRO-IMAGING

→ POWDER CLASSIFICATION

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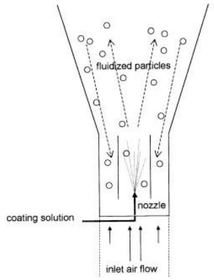
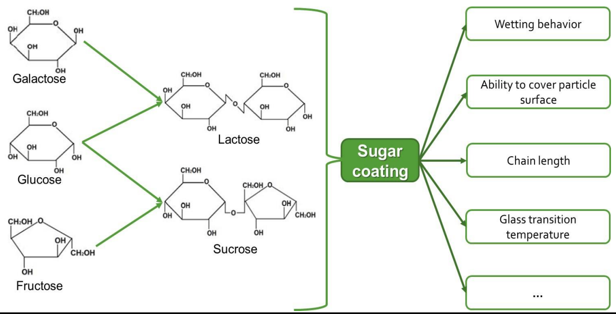
2. Surface composition (micro & molecular scale)

Selection of representative powders from each class of reconstituability

✖ ✖ ✖

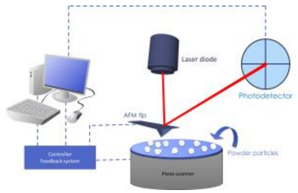
SUGAR COATING





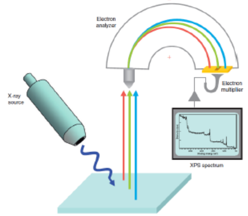
SURFACE MAPPING

Atomic force microscopy (AFM) : topography, chemical forces, nanoindentation



SURFACE COMPOSITION

X-ray photoelectron spectroscopy (XPS) : surface elemental composition



Year 2 (January 2020 – January 2021) ongoing

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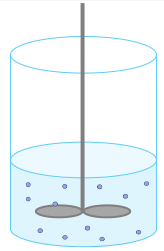
HD WORK AND
ERABLES

Year 3 (January 2021 – January 2022)

1. Screening of a large variety of powders

Reconstitution process

Wetting → Swelling → Dispersion → Dissolution



→ Reconstitution kinetics by granulomorphometry

→ Powder reconstitution steps by conductimetry & macro-imaging

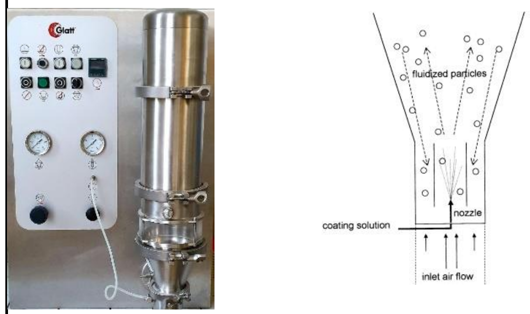
→ Powder classification

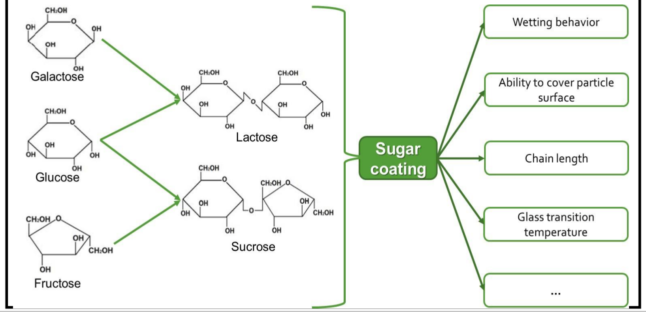
- Easy
- Intermediate
- Poor

2. Surface composition (micro- & molecular scale)

Selection of representative powders from each class of reconstituability

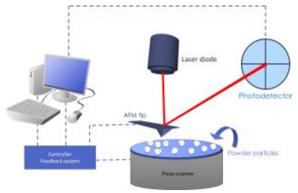
Sugar coating





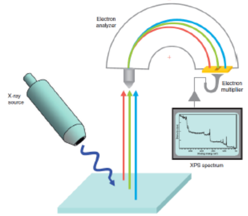
Surface mapping

Atomic force microscopy (AFM) : topography, chemical forces, nanoindentation



Surface composition

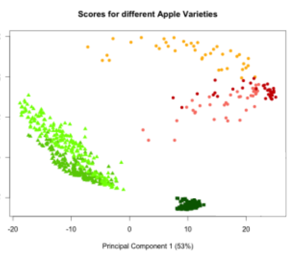
X-ray photoelectron spectroscopy (XPS) : surface elemental composition



MOLECULES Nano- | PARTICLES Micro- | POWDE Macro-

MULTISCALE STUDY

Experimental data from 1. & 2.



Semi-empirical models

3. Development of a reconstituability index, in order to predict reconstitution times

2

POWDER CLASSIFICATION ACCORDING TO THEIR RECONSTITUTABILITY (YEAR #1: 2019)

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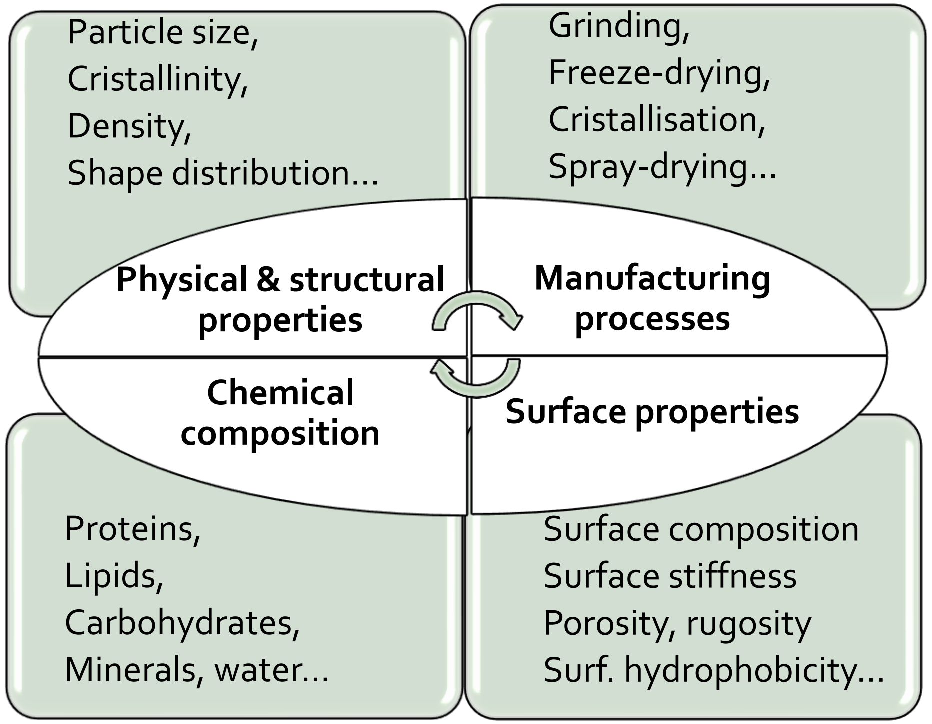
SCREENING OF A LARGE VARIETY OF POWDERS

About **50 food powders** were selected :
- Sugar: icing, fine, granular,
- Pea proteins: normal and micronized,
- Polenta: fine and large,
- Instant coffee: fine and large,
- Carboxymethylcellulose, flour,
- Fumed silica,
- Starch,
- Cocoa powders: medium and high fat content, alkalized or not

…



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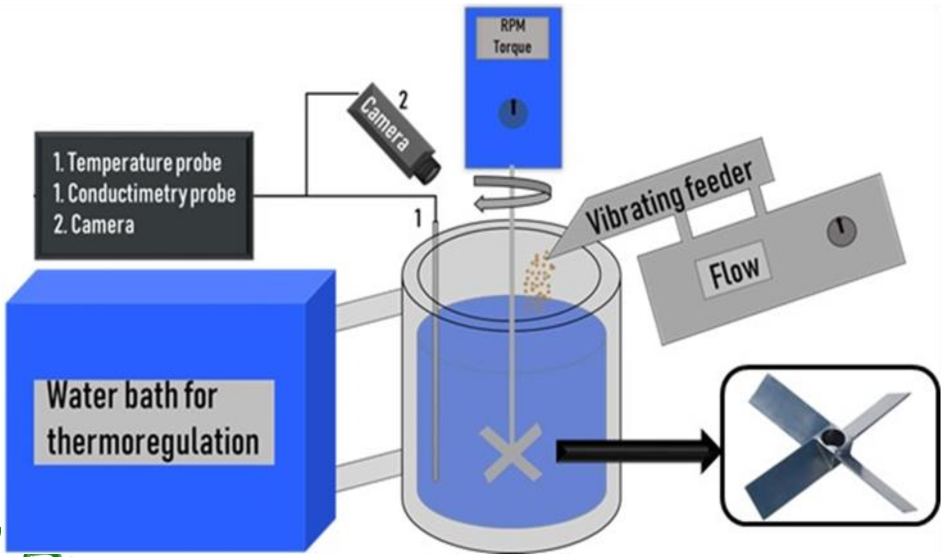
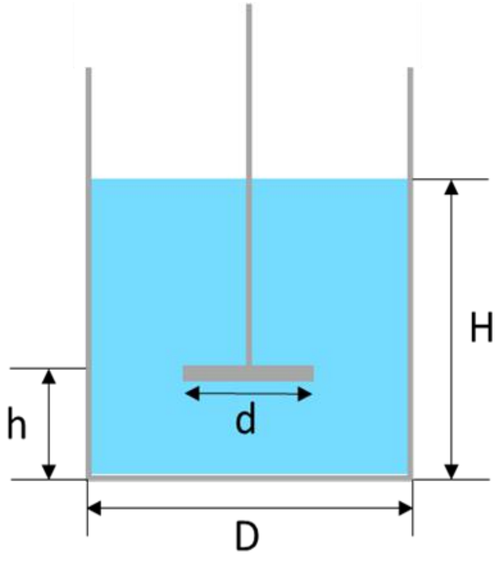
RECONSTITUTABILITY FOLLOWED BY CONDUCTIMETRY

1 - Powder reconstitution kinetics followed by conductimetry

- thermostated jacketed glass reactor
- mechanical stirring with a 5.08 cm diameter impeller
- powder added by a vibratory feeder

Powder reconstitution experiments were performed with distilled water at 25.0 ± 0.2 °C in the following conditions: 10.0 % (w/w) powder/water mass ratio with a total mass (powder + water) fixed at 2.650 kg, 800 rpm stirring rate and 3.6 $g.s^{-1}$ feeding rate.





Reactor design and reconstitution conditions based on the literature

(Galet et al., 2004; Goalard et al., 2006; Mitchell et al., 2015, 2019; Schober & Fitzpatrick, 2005)

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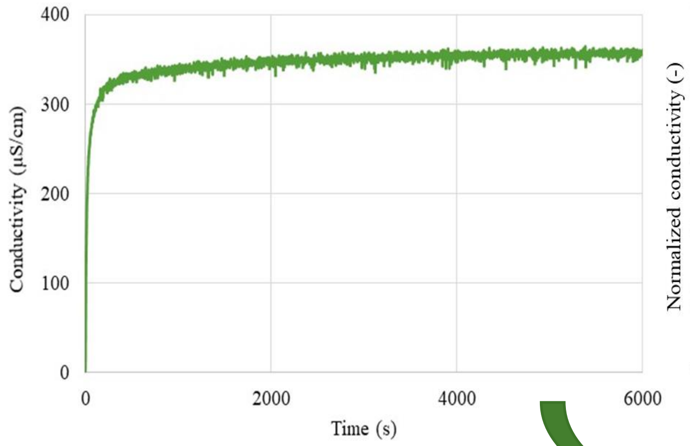
Reconstitutability followed by conductimetry

2 - Conductivity normalization

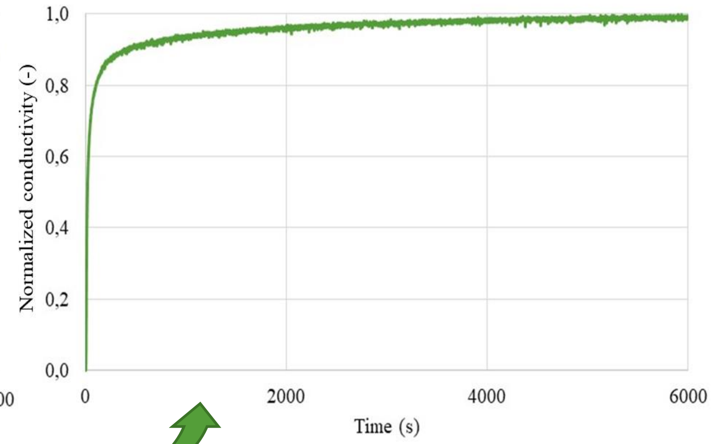
- Conductivity evolved from an almost null initial value (real value inferior to the conductimeter sensitivity) to a final value that depended from powder nature.
- Averaged conductivity values were normalized according to:

$$c(t) = \frac{\kappa(t) - \kappa_{ini}}{\kappa_{fin} - \kappa_{ini}}$$

With: c(t) designates normalized conductivity (-);
κ(t) stands for conductivity at time t ($\mu S.cm^{-1}$);
κ_{ini} represents the initial conductivity, i.e. distilled water conductivity, almost null ($\mu S.cm^{-1}$);
κ_{fin} corresponds to the final conductivity ($\mu S.cm^{-1}$).



Before normalization
(mean of three repetitions)



After normalization

RECONSTITUTABILITY FOLLOWED BY CONDUCTIMETRY

3 - Curve fitting

The normalized conductivity curves were fitted using the least squares method solved by the Levenberg Marquardt iteration algorithm with the Hill model :

$$c(t) = c_0 + (c_\infty - c_o)\frac{t^n}{k^n + t^n}$$

With: c_0, initial normalized conductivity (%);
c_∞, final normalized conductivity (%);
t, time (s);
k (s) and n (-), Hill model parameters.

Initial and final normalized conductivities being equal to 0 and 1, respectively, the Hill model could simply be rewritten as :

$$c(t) = \frac{t^n}{k^n + t^n}$$

k corresponds to the time needed to reach 50 % normalized conductivity. The slope of normalized conductivity curve at *t* = *k* was found to be equal to $\frac{n}{4k}$, then it can be considered that the model parameter *n* gives an indication of the powder reconstitution rate once wetted/sinked, i.e. in the dispersion/solubilization steps of powder reconstitution. Last, powder reconstitution time was deduced from obtained Hill model by taking the time needed to reach 95 % normalized conductivity, it was then noted $t_{95\%}$.

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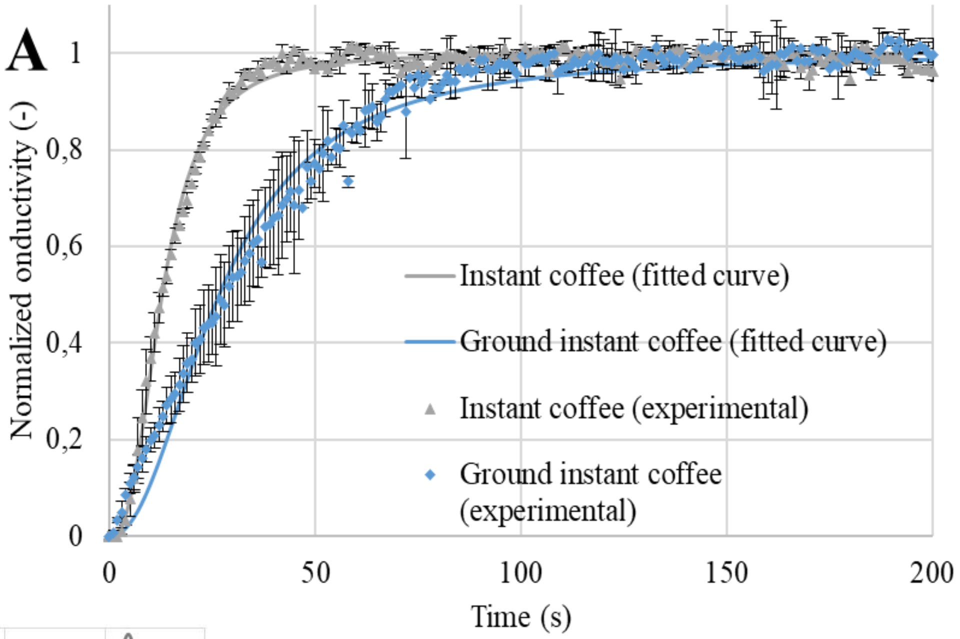
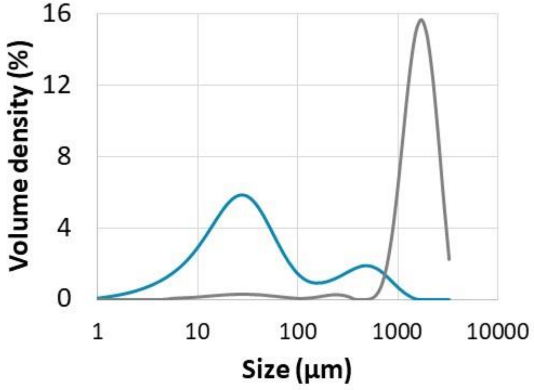
Reconstitutability followed by conductimetry

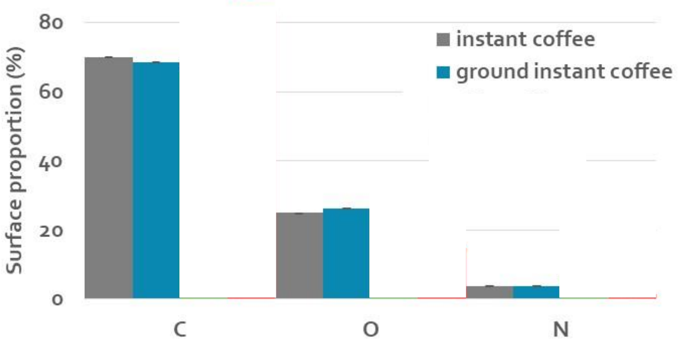
Impact of particle size on reconstitutability

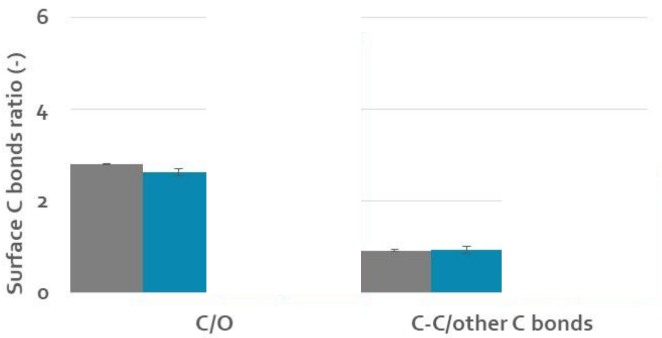
Instant coffee powder

Ground instant coffee powder









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RECONSTITUTABILITY FOLLOWED BY CONDUCTIMETRY

Instant coffee powder

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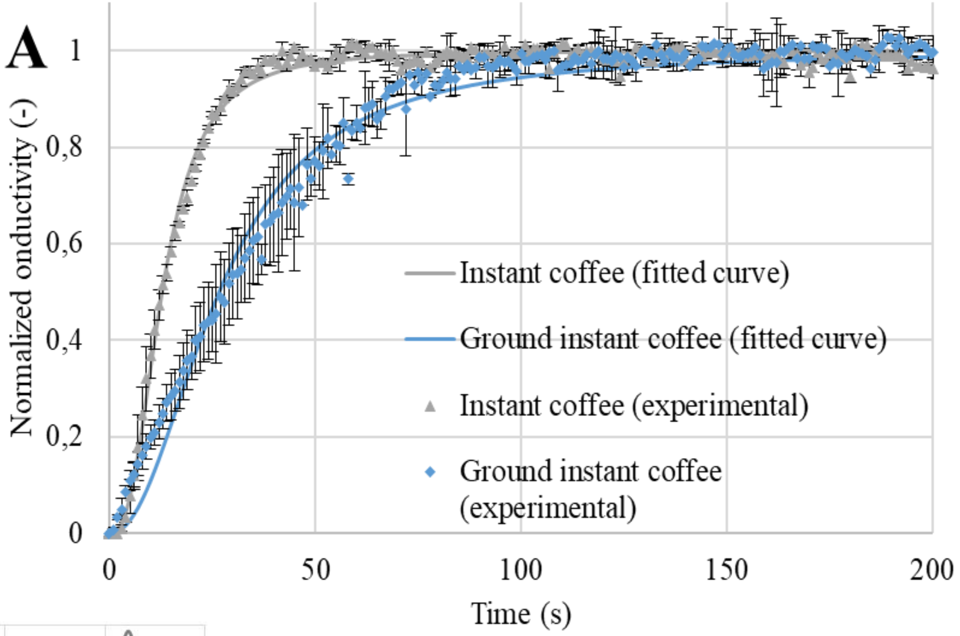
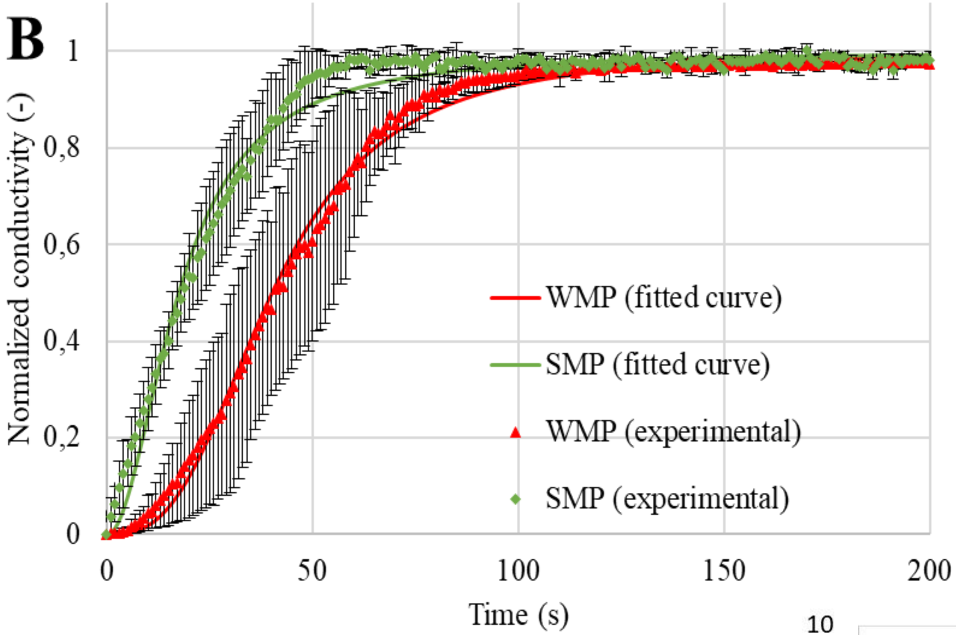
Impact of particle size on reconstitutability

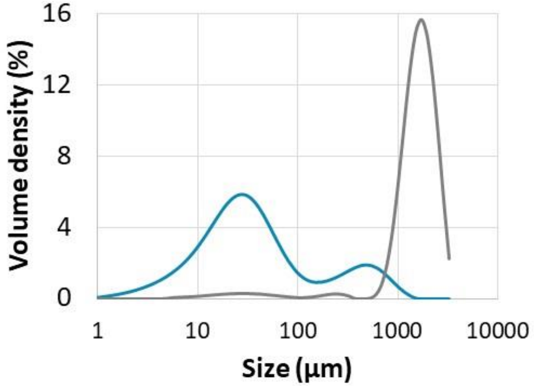


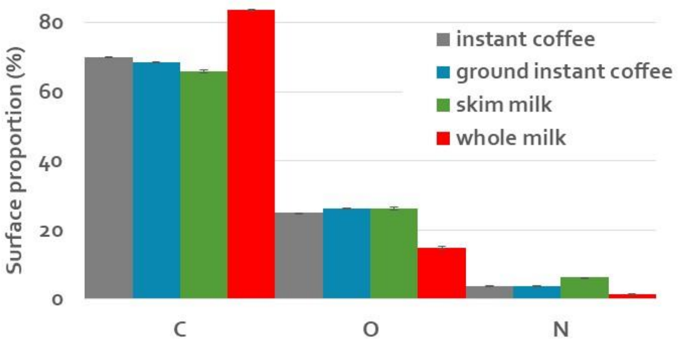
Impact of fat content on reconstitutability

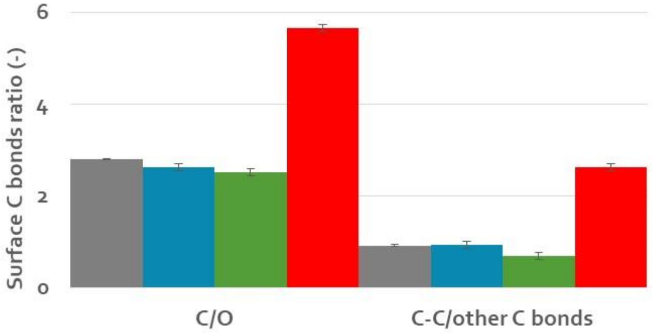


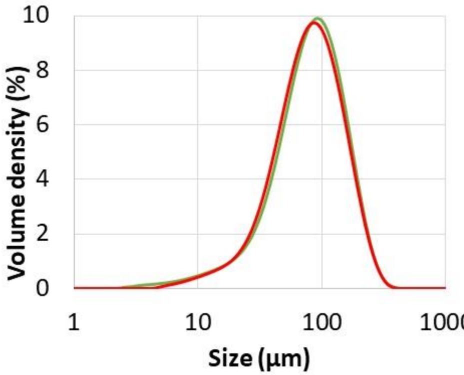
Whole milk powder

Skim milk powder









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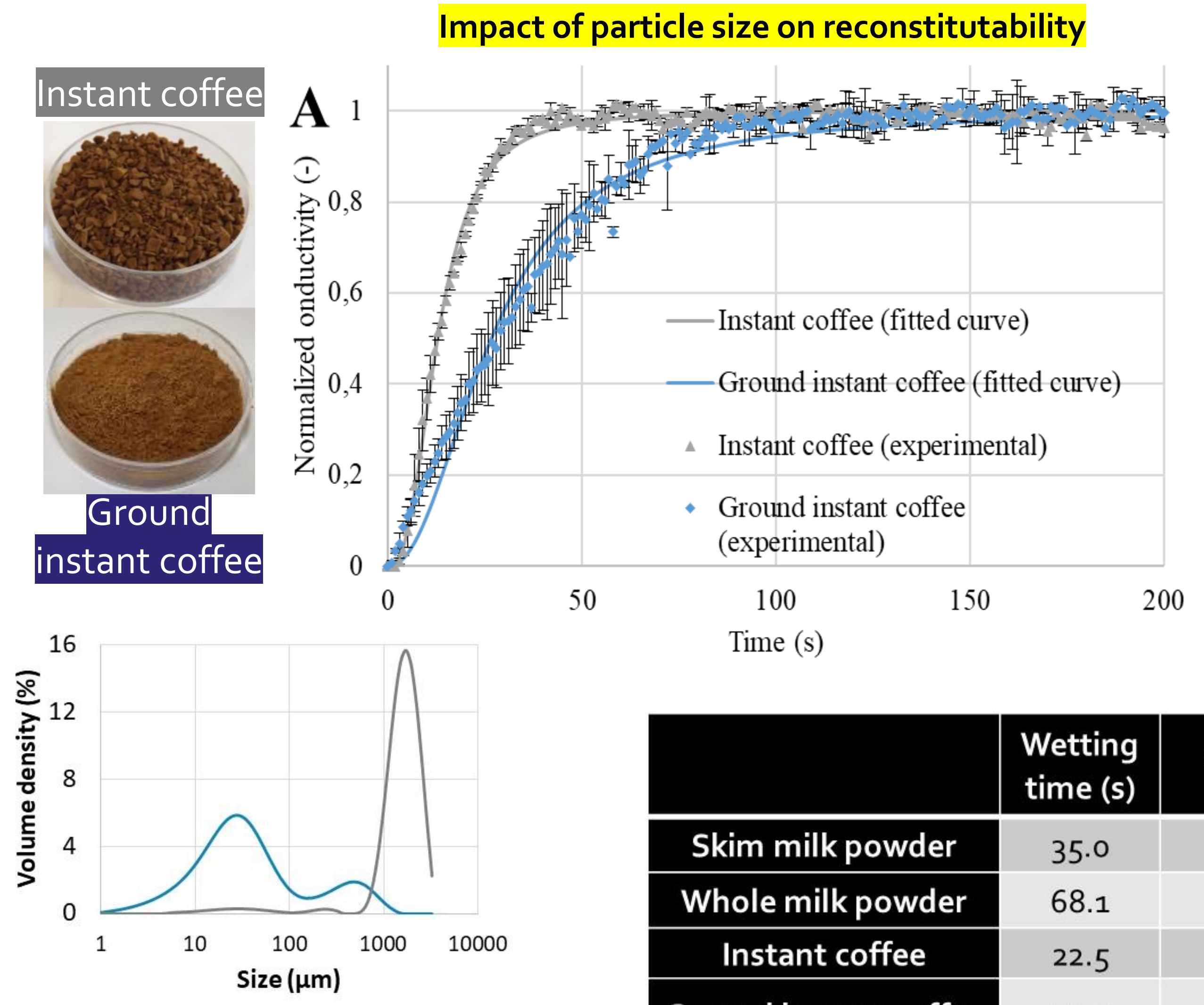
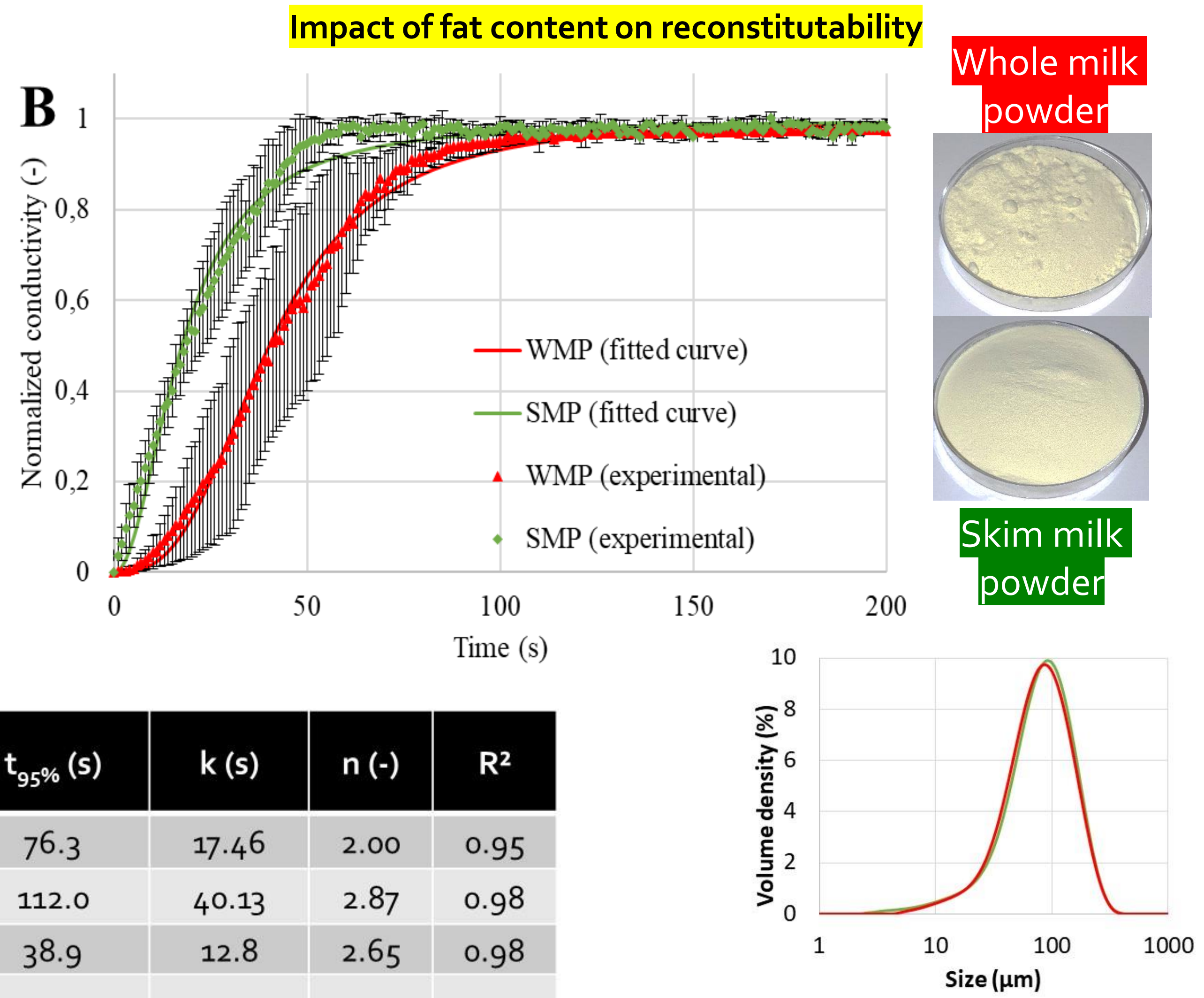
Reconstitutability followed by conductimetry





	Wetting time (s)	$t_{95\%}$ (s)	k (s)	n (-)	R^2
Skim milk powder	35.0	76.3	17.46	2.00	0.95
Whole milk powder	68.1	112.0	40.13	2.87	0.98
Instant coffee	22.5	38.9	12.8	2.65	0.98
Ground instant coffee	51.5	105.2	26.64	2.14	0.98

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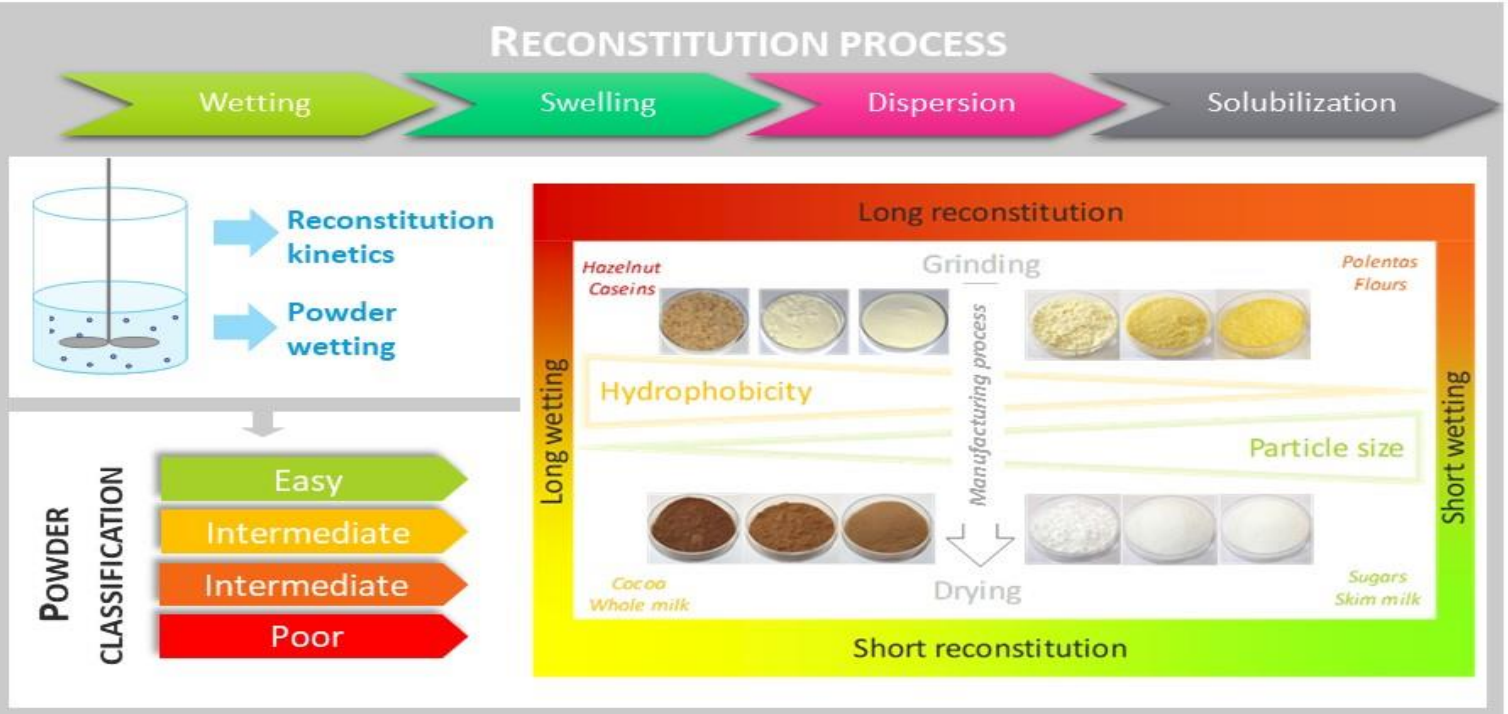
TAKE-HOME MESSAGE FROM YEAR #1

Physicochemical characteristics of powders were correlated with the **wetting** and **reconstitution times** of fifty food powders by principal component analysis (PCA). Four powder categories were identified based on wetting and reconstitution times.

Green group: short wetting and reconstitution times,
Yellow group: long wetting time and short reconstitution time,
Orange group: short wetting time and long reconstitution time,
Red group: long wetting and reconstitution times.



Powder classification according to their reconstitution behavior :

- **Long wetting times** associated to high particle surface hydrophobicity, small median particle size, as well as high protein and fat contents in the powder bulk.

Horizontal axis (PC1)

- **Long reconstitution times** linked to the powder manufacturing process (powders obtained by grinding of solid materials led to poorer reconstitution behavior than spray-dried powders) and low sugar content in the powder bulk.

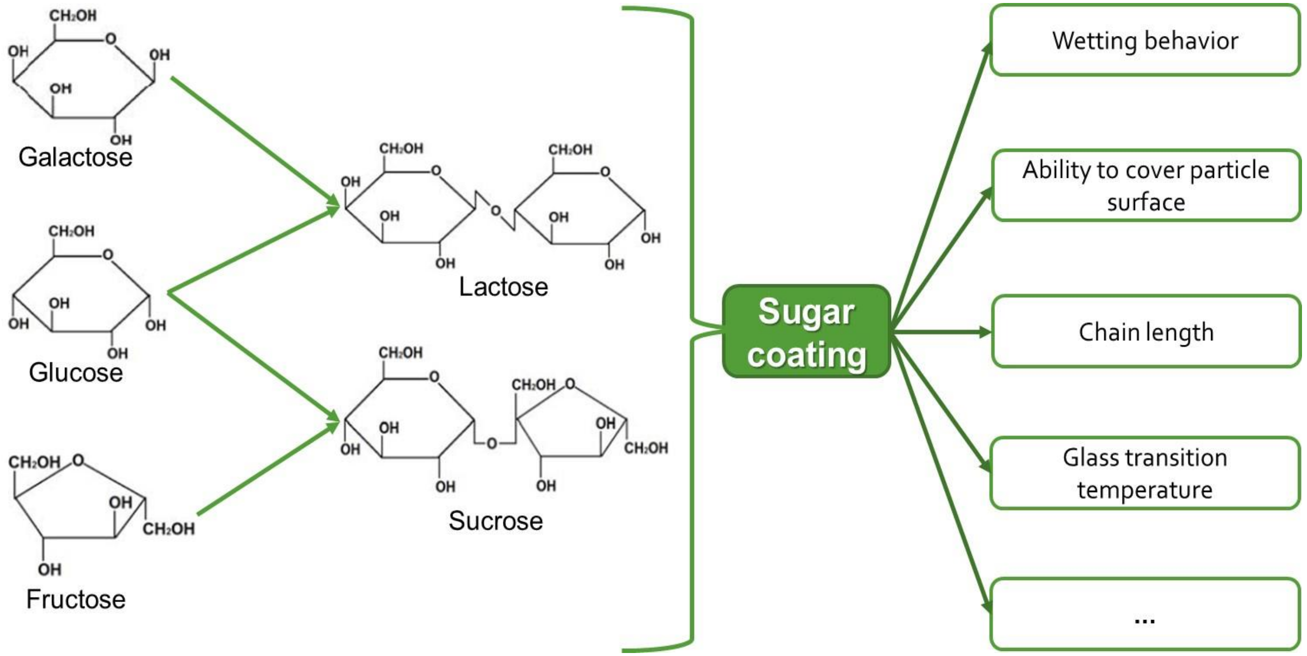
Vertical axis (PC2)

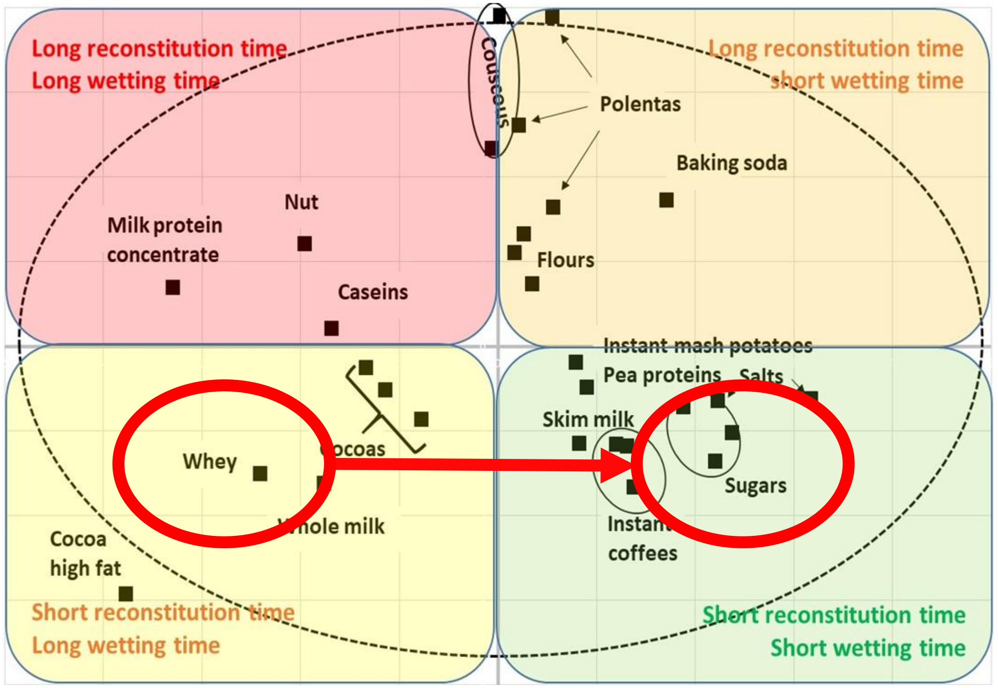
3 NEW KNOWLEDGE ABOUT POWDER RECONSTITUTABILITY THANKS TO A FOCUS ON PARTICLE SURFACE (YEAR #2: 2020)

⚠ Laboratory closure from mid march until mid may 2020…
The remote working was focused on data treatment and paper writting.

Focus on some powders from each class of reconstitutability

Whey powder coating with sugar solutions (validated with IFPRI)





Whey powder
Non-wettable

Sugar powders
Easily wettable

- Determination of the minimal sugar proportion necessary to improve whey powder wettability
- Impact of sugar nature
- Impact of powder surface (stiffness, rugosity, surface coverage…)

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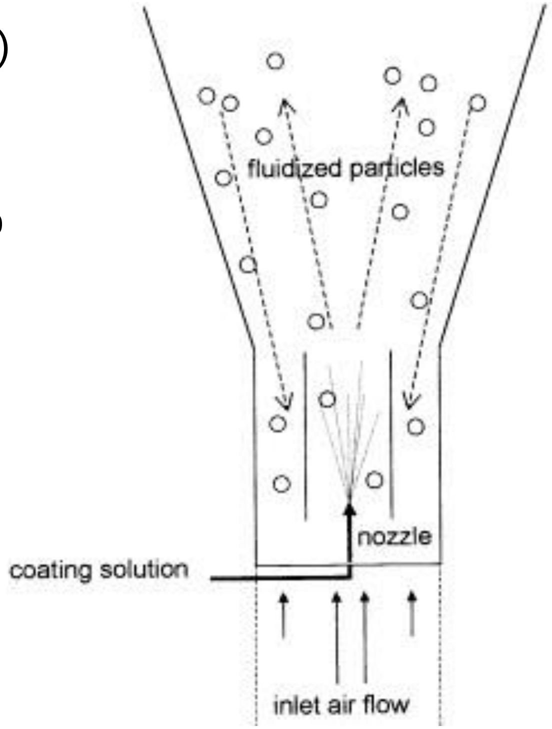
FIRST SUGAR COATINGS

- One day course training with Glatt application specialist (in February)
- Various process conditions and formulations were tested:
  - Coating duration from 5 to 15 min
  - Coating with sucrose solutions at concentrations from 0 to 45 %
  - Flow rate of 0.80 $g.s^{-1}$

Experiment code	Time (min)		
Sucrose (%)	5	10	15
0	#1	#2	#3
15	#1-1	#2-1	x
30	x	#2-2	x
45	x	x	x

First line: Reference powders = whey powder + process impact (attrition)



Mini-Glatt fluid bed coater (Würster design)

Quantification of sugar content in coated powders

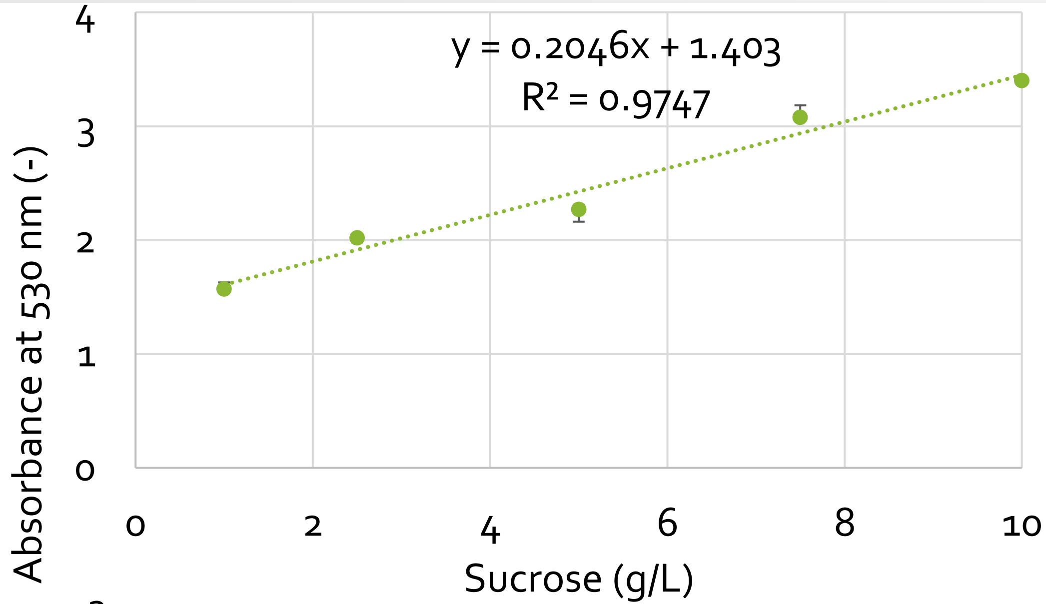
Example for sucrose coating

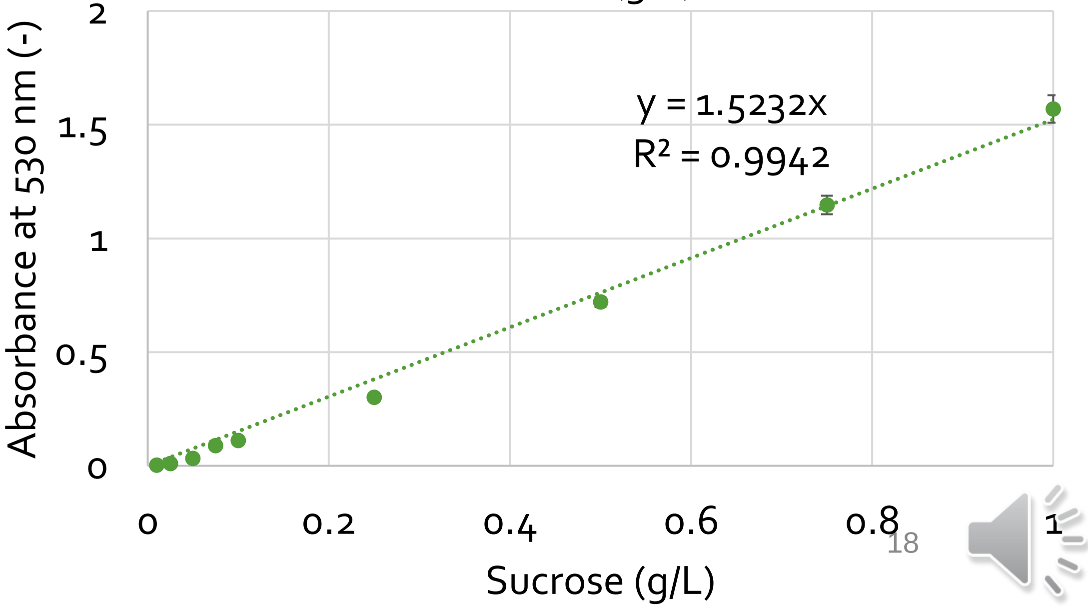
Sucrose quantification

Sample acid hydrolysis → Reaction with DNS ← Sucrose standards

Reaction with DNS → Absorbance at 530 nm

Samples	Sucrose g / 100 g
Whey powder	0
15 % sucrose; 5 min (#1-1)	1.11 ± 0.42
15 % sucrose; 10 min (#2-1)	1.97 ± 0.51
30 % sucrose; 10 min (#2-2)	3.84 ± 0.65
Sucrose powder	100

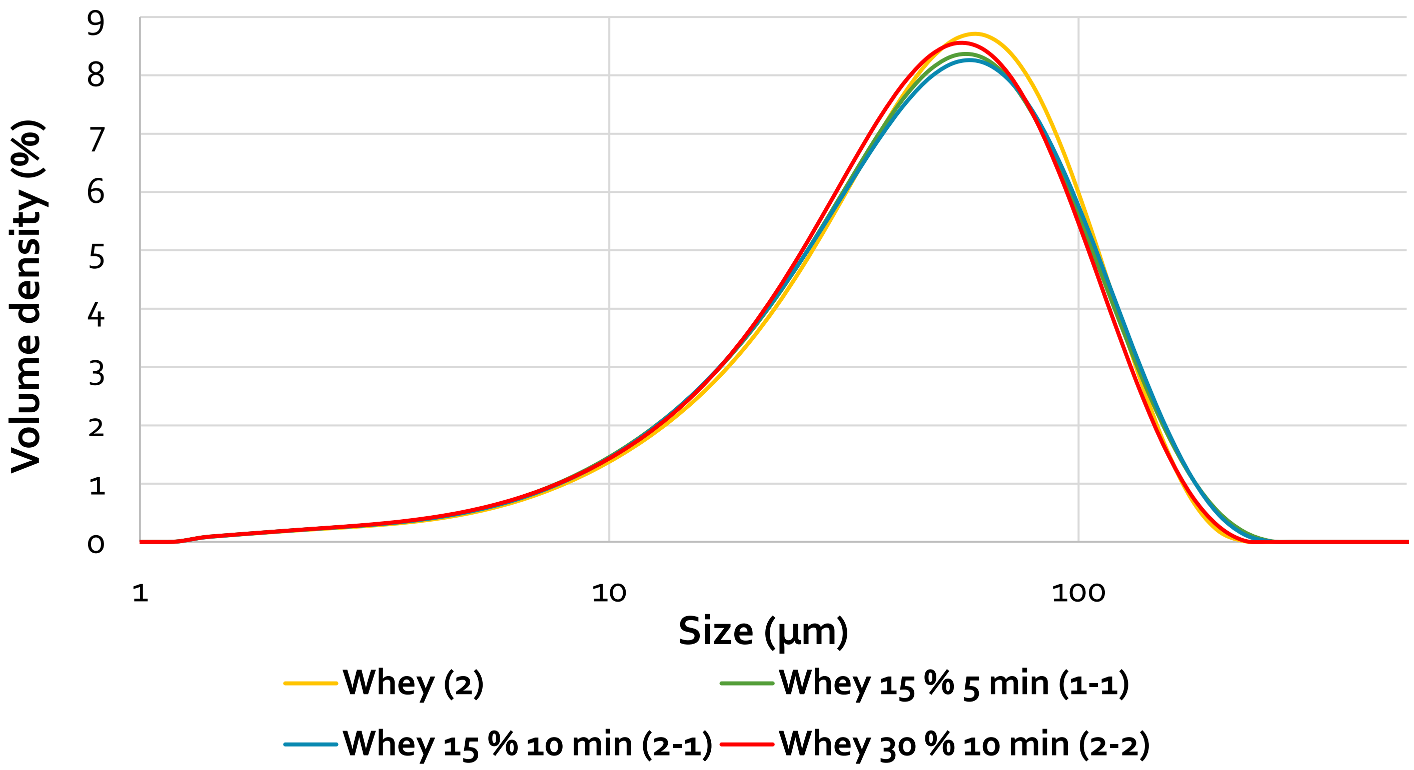




Physicochemical characterization

Particle size distribution

- No significant differences between uncoated and sucrose-coated whey powders
- No agglomeration induced by coating
- Fine and regular sucrose coverage

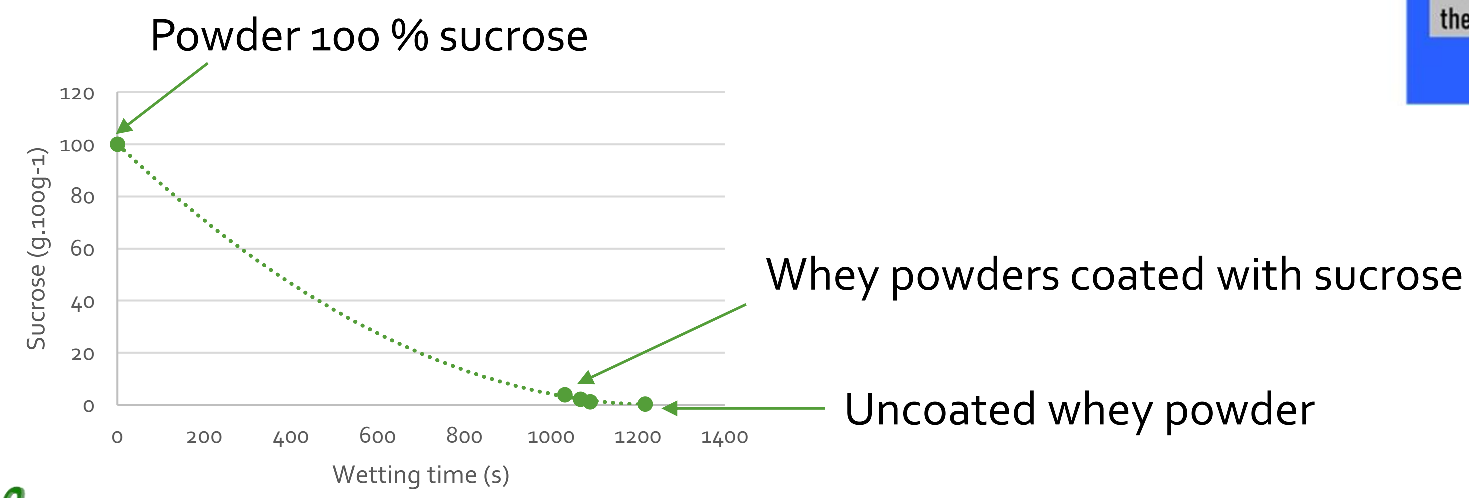


	D_{10} (µm)	D_{50} (µm)	D_{90} (µm)	Span (-)
#2	14.1 ± 0.0	48.3 ± 0.1	105.5 ± 0.7	1.9 ± 0.0
#1-1	13.6 ± 0.1	47.2 ± 0.1	107.5 ± 0.7	2.0 ± 0.0
#2-1	13.6 ± 0.0	47.5 ± 0.1	108.5 ± 0.7	2.0 ± 0.0
#2-2	13.6 ± 0.0	46.3 ± 0.4	103.5 ± 0.7	1.9 ± 0.0

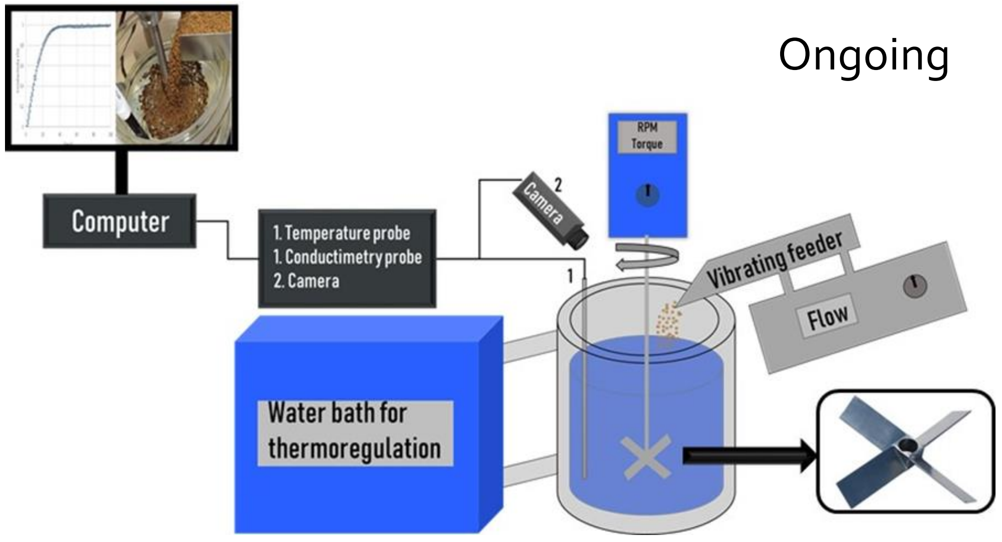
RECONSITUTABILITY

- Wetting time (IDF standard)

Samples	Wetting time (s)	Sucrose (g/100 g)
Whey	1 217 ± 11	0
#1-1	1 090 ± 4	1.1
#2-1	1 068 ± 17	2.0
#2-2	1 032 ± 8	3.8



- Reconstitution monitored by conductimetry



Ongoing

Only slight powder wetting improvement; in further experiments, longer coating durations and higher sugar concentrations will be used.

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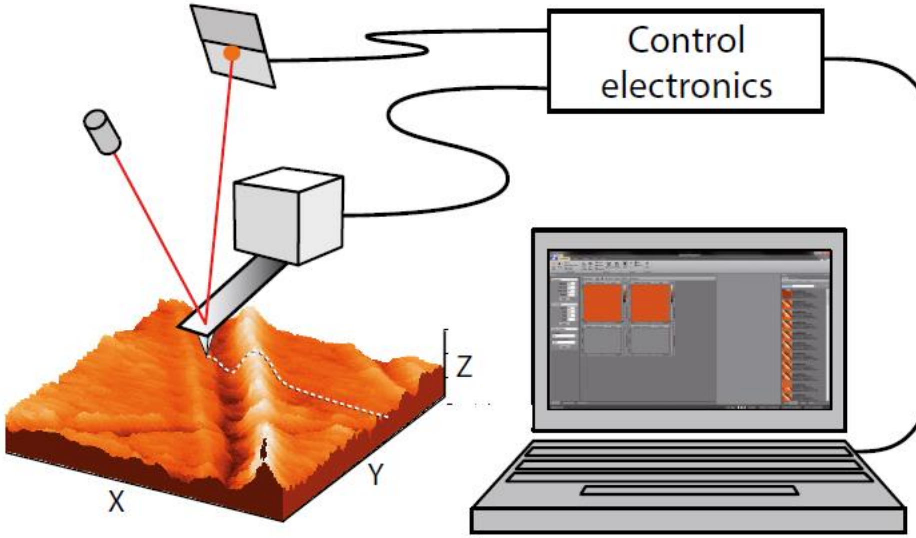
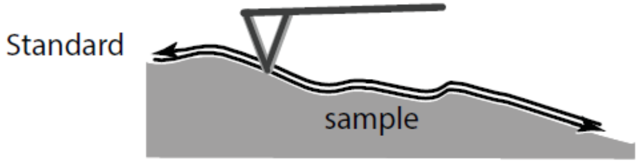
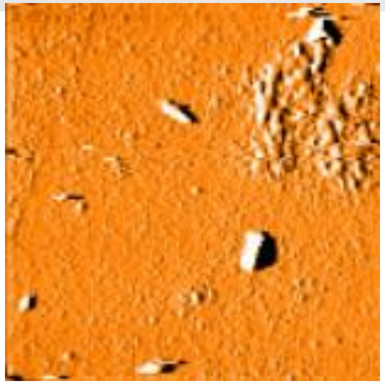
SURFACE CHARACTERIZATION

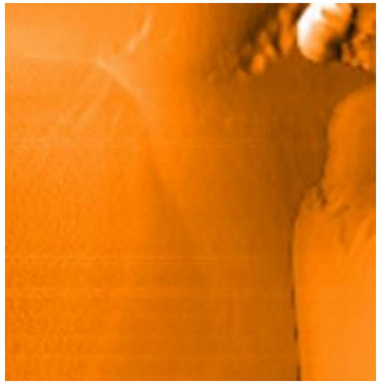
- Surface topography and roughness







Powder 2 (5 x 5 µm²)
Reference whey powder



Powder #2-1 (5 x 5 µm²)
Whey powder coated during 10 min with 15 % sucrose solution

AFM Nanosurf Flex-Axiom

ONGOING - Roughness

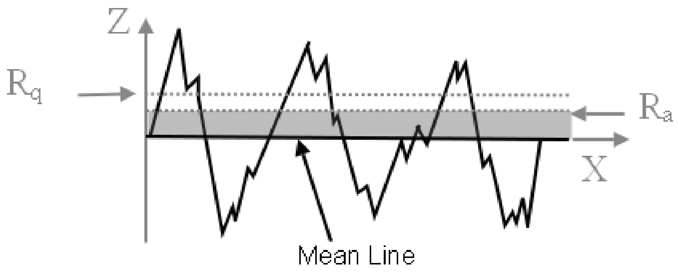


Image analysis for roughness calculation: Rq & Ra

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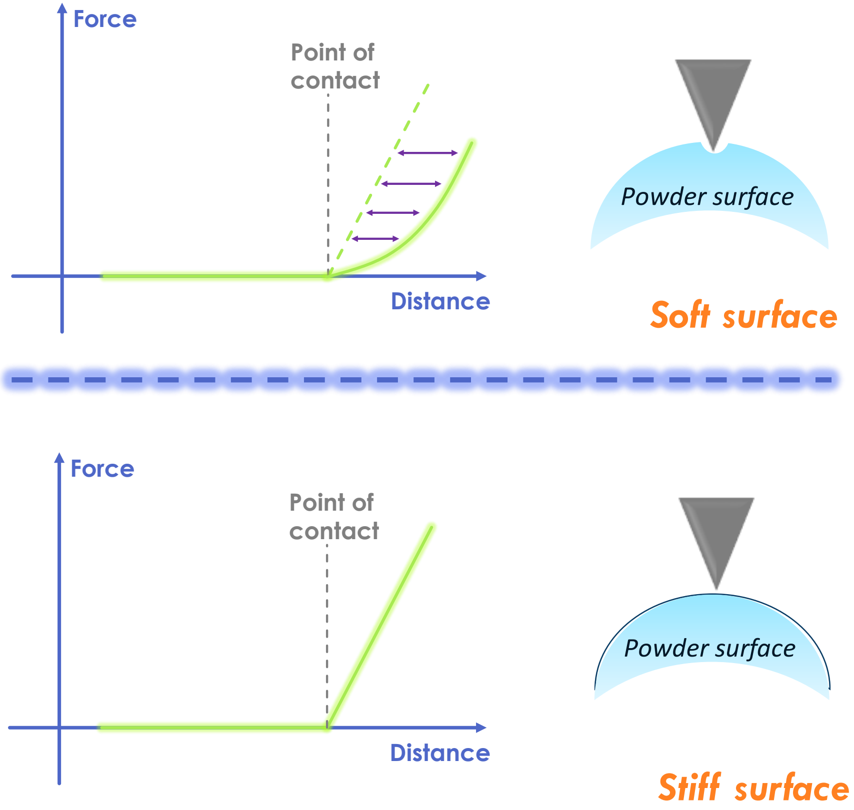
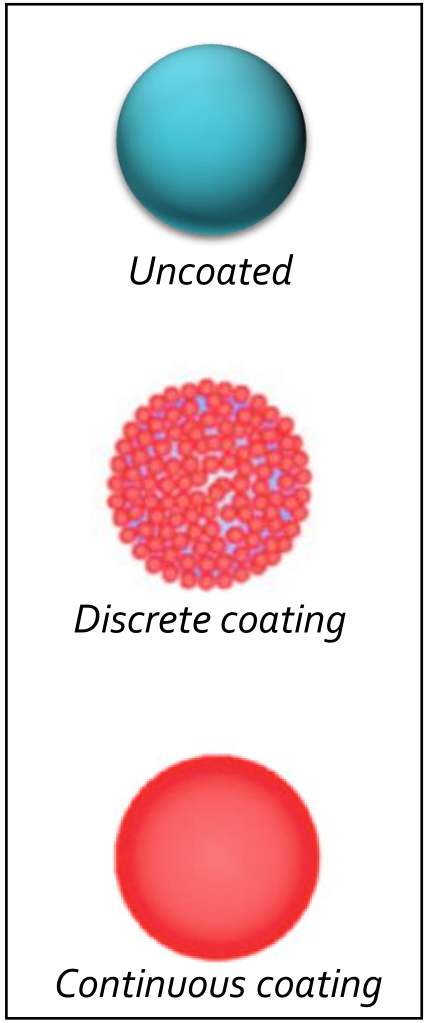
SURFACE CHARACTERIZATION

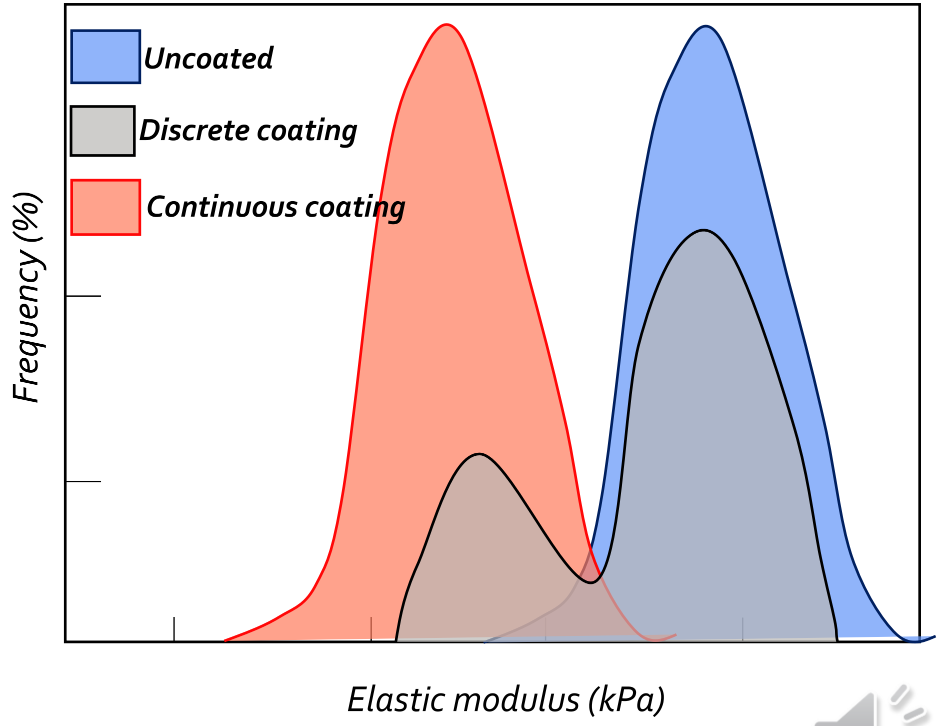
- Surface mechanical properties (nanoindentation)

Expected results based on the results of other studies already performed by the laboratory





AFM Nanosurf Flex-Axiom



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4 PERSPECTIVES

Year #2 ongoing…

- Coating with various sugars
- Surface characterization
- Functional properties

Laboratoire d'Ingénierie des Biomolécules

FIRST SCIENTIFIC PAPER FROM YEAR 1

Main physicochemical powder characteristics influencing their reconstitution behavior

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Abstract

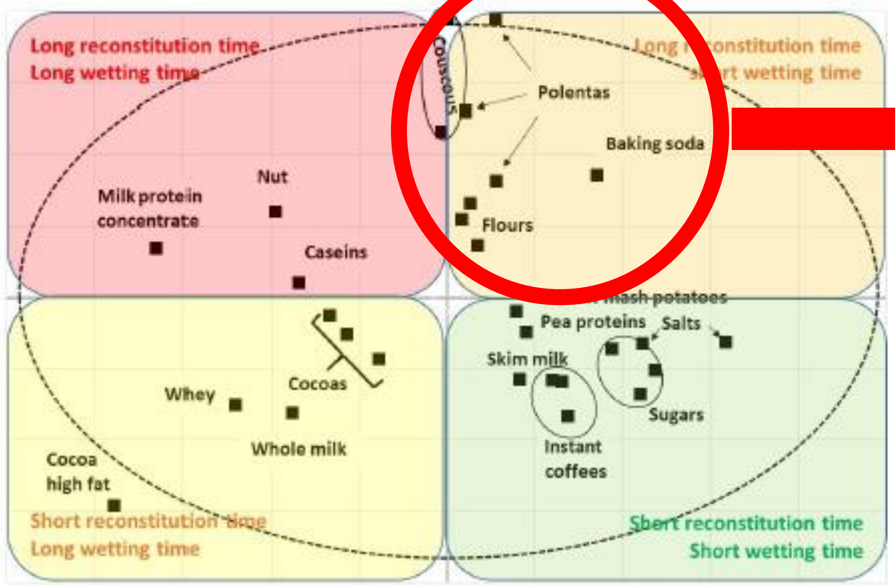
Sixty food powders corresponding to a wide range of physicochemical characteristics (chemical bulk and surface composition, particle size, span) and manufacturing processes (grinding, freeze-drying, spray-drying, crystallization) were investigated. A wide range of reconstitution profiles were measured by conductivity measurements and analyzed thought data fitting. Physicochemical characteristics of powders were correlated with the wetting and reconstitution of sixty food powders by principal component analysis (PCA). Four powder categories were identified based on wetting and reconstitution times; i.e. *Green group*: short wetting and

Draft sent to industrial project members
Waiting for comments/corrections from IFPRI members

Possible journals :

- Journal of Food Engineering
- Food Hydrocolloids

SECOND SCIENTIFIC PAPER FROM YEAR 1

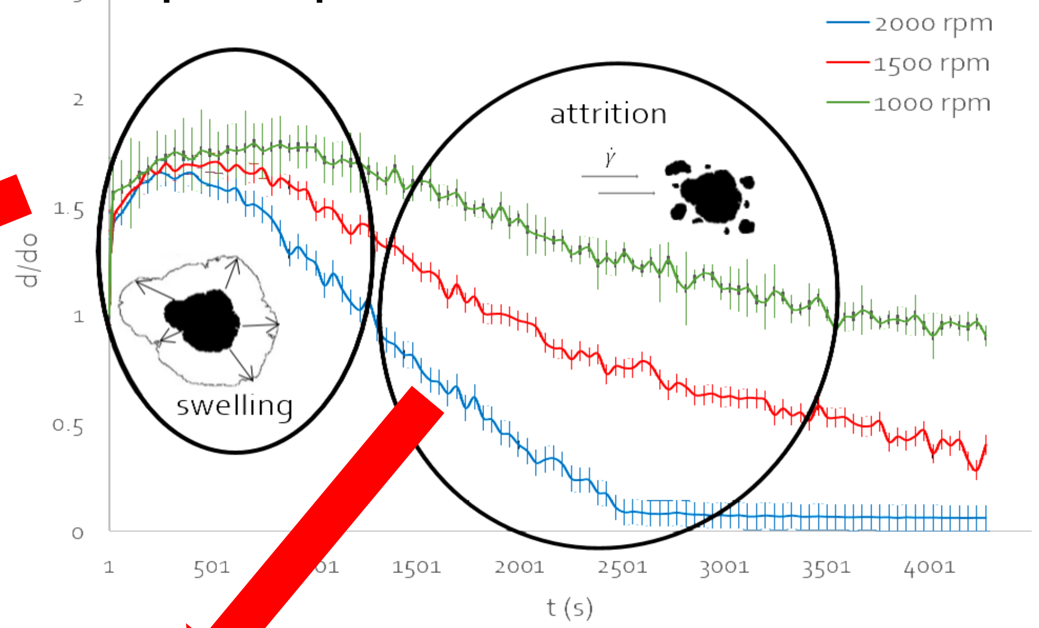


Naïma GAUDEL – 3 months Post doc focused on cereal powders reconstitution (couscous, flours, etc.):

- Swelling step
- Attrition step

Reconstitution kinetics at different stirring rates

Impact of particle size



Swelling rates vs stirring rate for two mean particle sizes

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- Food Hydrocolloids
- Powder Technology

Attrition rates (µm.s^{-1}) vs shear (for 2 grain sizes)

	2000 rpm	1500 rpm	1000 rpm
Attrition rate for fine powder	0.77 ± 0.01	0.48 ± 0.04	0.26 ± 0.01
Attrition rate for medium powder	2.19 ± 0.16	1.14 ± 0.04	0.52 ± 0.03

THANK YOU FOR YOUR ATTENTION !

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