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Passive microrheology as a useful tool for milk gel analyses

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Passive microrheology as a useful tool for milk gel analyses

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www.formulaction.com

STABILITY & SIZE
MICRORHEOLOGY
RHEOLOGY ON CHIP

FORMULACTION
An overview

A full range to characterize dispersions:

- *Without denaturation*
- *With easy operation*
- *From Formulation to Application*

TURBISCAN
STABILITY & SIZE
DISPERSION STABILITY & SIZE BY MULTIPLE LIGHT SCATTERING

FLUIDICAM
RHEOLOGY ON CHIP
FLOW RHEOLOGY BY MICROFLUIDICS

RHEOLASER
MICRORHEOLOGY
ZERO SHEAR MICRORHEOLOGY
THERMAL ANALYSIS BY MICRORHEOLOGY

DISPERSION STATE

SIZE
Dispersibility, aggregation..

STABILITY
Size variation, migration…

RHEOLOGY

FLOW BEHAVIOR
Injectability, sprayability…

STRUCTURE AT REST
Gelling, stability…

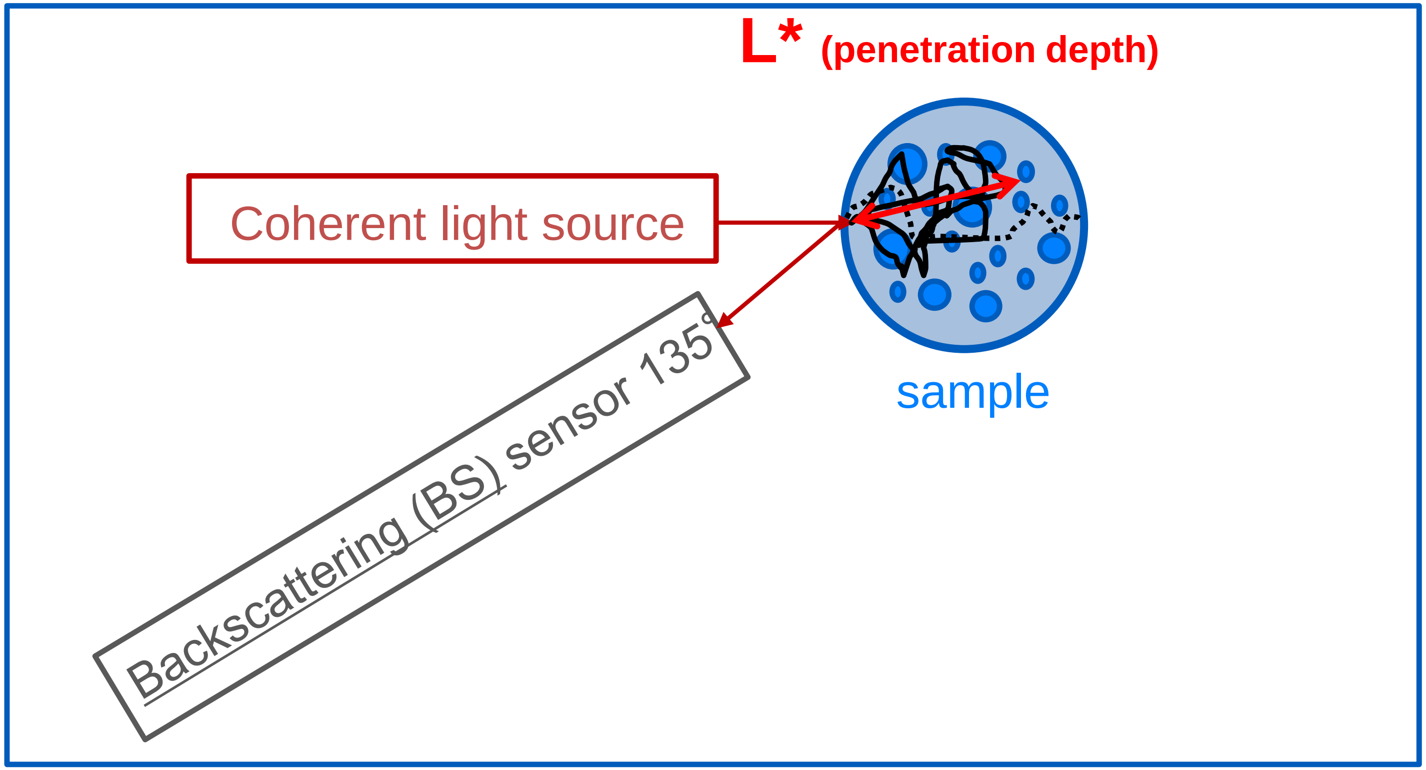
THERMAL ANALYSIS

PHASE TRANSITION
Crystallization, melting…

FROM FORMULATION TO APPLICATION

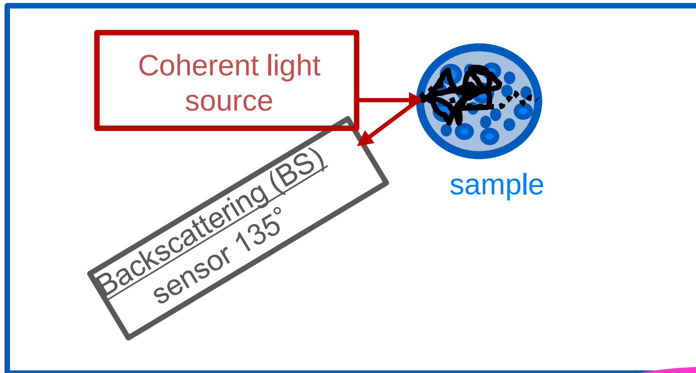
Core technologie

Multiple light scattering



Core technologie

Multiple light scattering



Turbiscan

Static Multiple Light Scattering

- Stability analysis
- Size determination

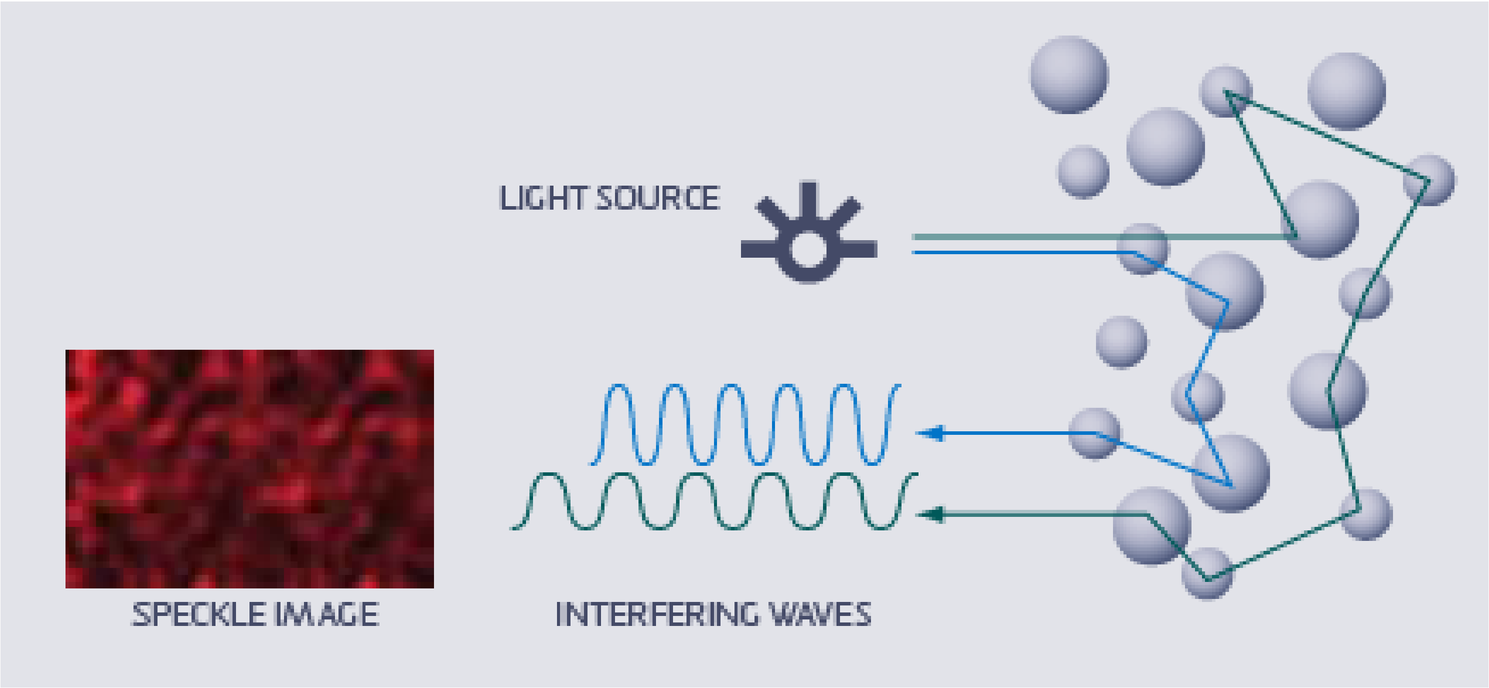
Rheolaser range

Diffusing Wave Spectroscopy
–
passive microrheology

- Bulk rheological properties
- Gel time

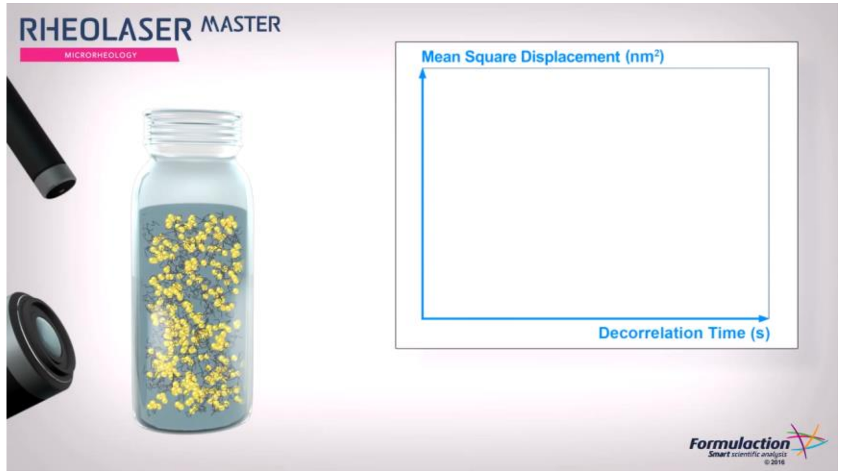
STABILITY & SIZE
MICRORHEOLOGY
RHEOLOGY ON CHIP

Diffusing Wave Spectroscopy
Principle



Diffusing Wave Spectroscopy

Principle



Analysis of backscatterd light in dynamic mode

=

Mean Square Displacement curves give information about bulk rheology

Gel point determination is quite complicated.

Usually people do it the easy way :

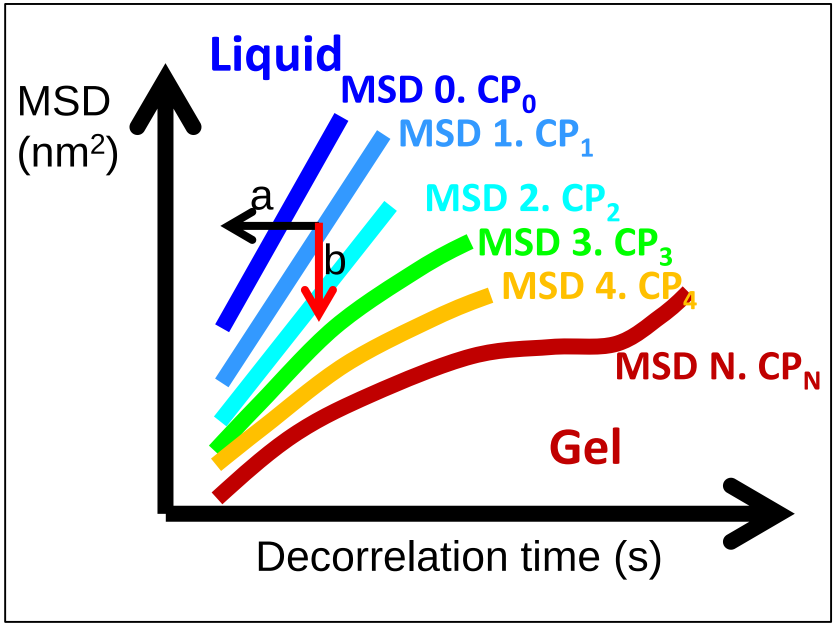
gel point is G' = G'' (at one frequency)

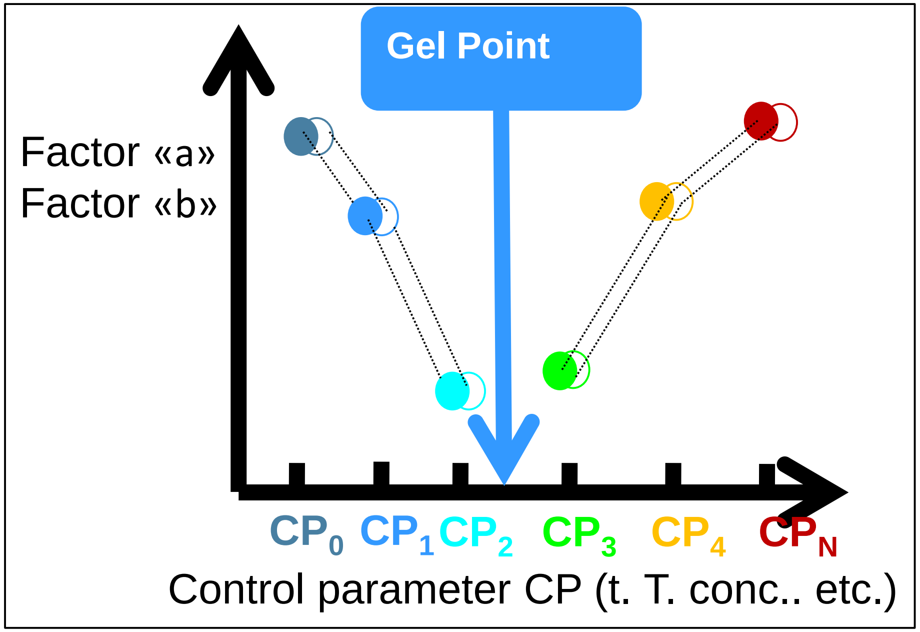
Actually:

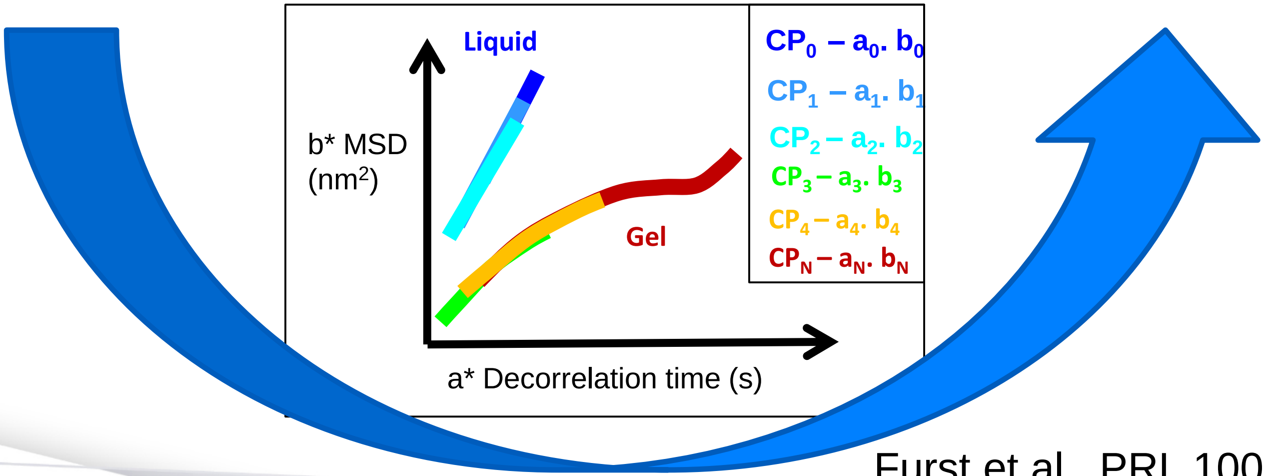
$G' \sim G'' \sim \omega^n$ Definiton according to Winter and Chambon

Rheolaser Master

Gel point determination







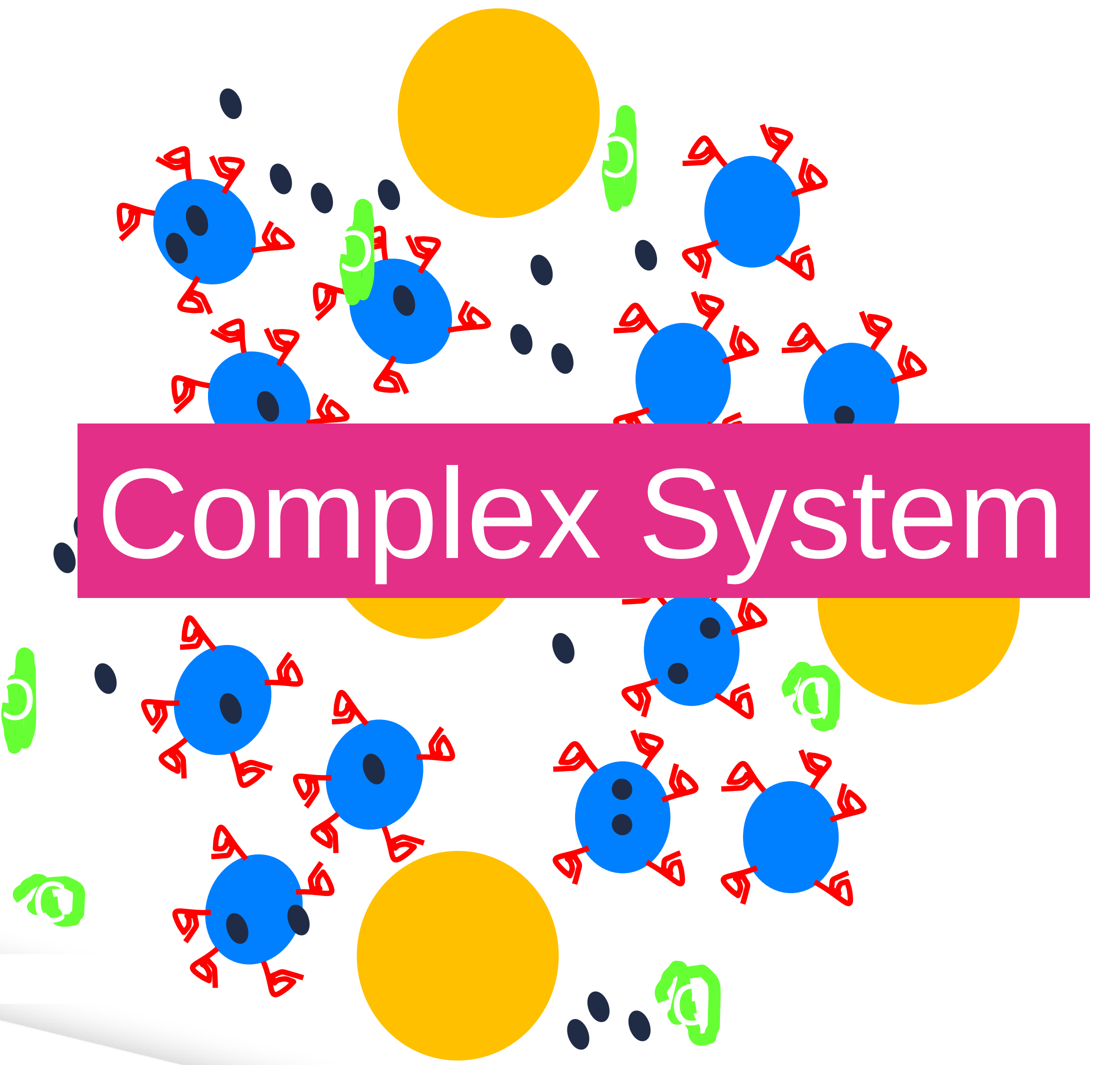
Furst et al., PRL 100, 146001 (2008)

Objective:

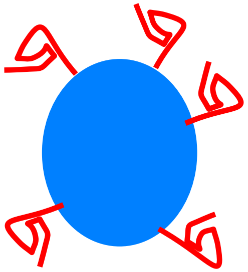
Studying milk gels (cheese and yogurt) with Rheolaser (DWS)

Milk

An introduction

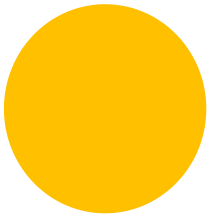


Complex System



3% Proteins
80% caseins
Hydrophilic casein
Hydrophobic casein

20 %
Whey proteins



Fat globules

• Minerals

Milk
An introduction

Composition of cow milk

- Water (87.5 %)
- Fat (3.7 %)
- Proteins (3.3 %)
- Glucides (4.7 %)
- Minerals (0.7 %)
- Others: enzymes, vitamines, pigments (0.1 %)

0.5-4 µm, composition depends of season, origin

0.5-4 µm, composition depends of season, origin

Changes during lactation !

Formulaction Smart scientific analysis

Introduction

–

Cheese

Cheese making

Introduction

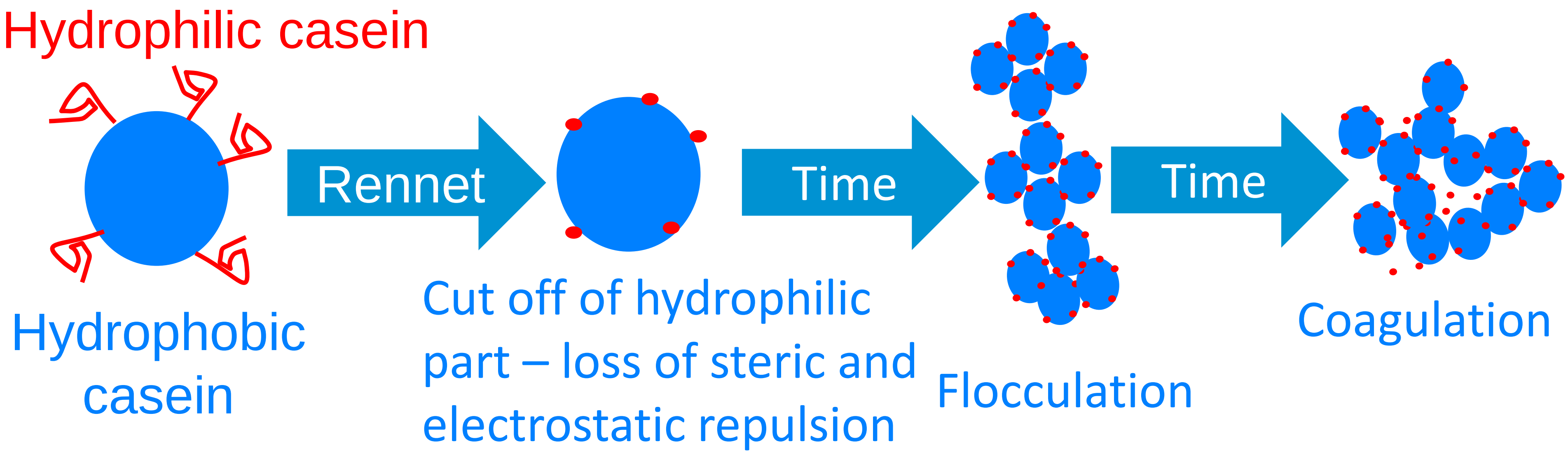
Milk	MILK PREPARATION	CURDLING	CURD PROCESSING	RIPING	Cheese
	Mechanical. thermal processing. Milk ajustment	Rennet (ferment) addition	Cutting. salting. washing. etc.	Several days to several months	

Influence of milk preparation and study of the curdling step with diffusive wave spectroscopy

1. Milk preparation

- Homogeneization of fat droplet size
- Pasteurization of milk
- Adjusting milk properties by adding cream, proteins, pH

2. Curdling



Flocculation : gel point (percolation point)
Coagulation : somewhere during gel curing

Results

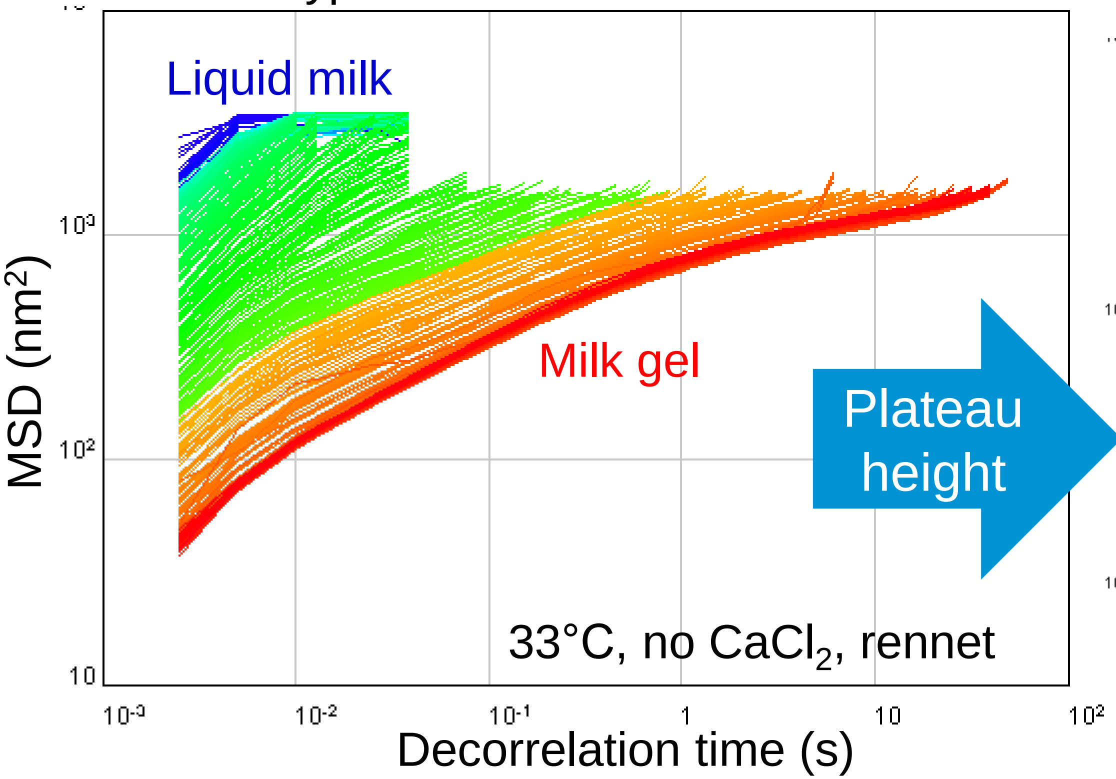
in collaboration with

INP TOULOUSE PURPAN Learning for the future
Ecole d'ingénieurs

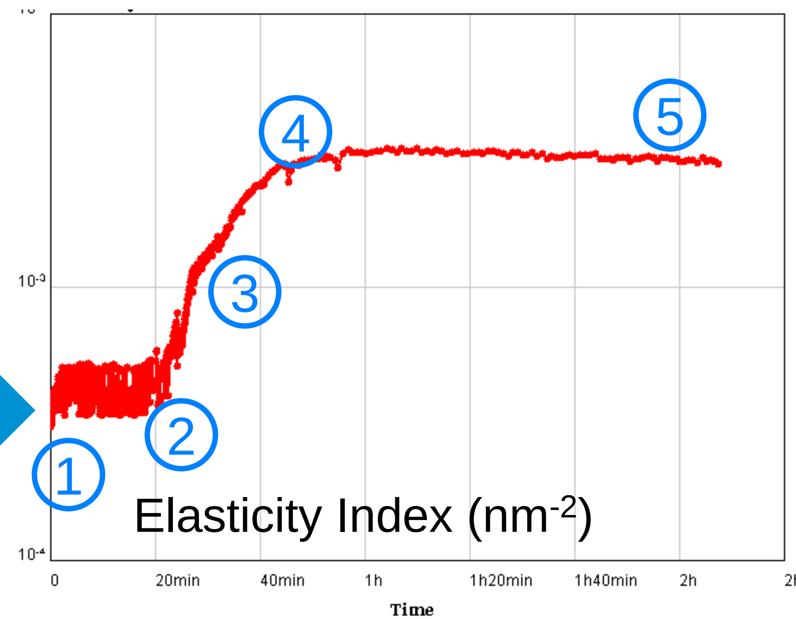
Cheese making

General results

Typical evolution of MSD curves



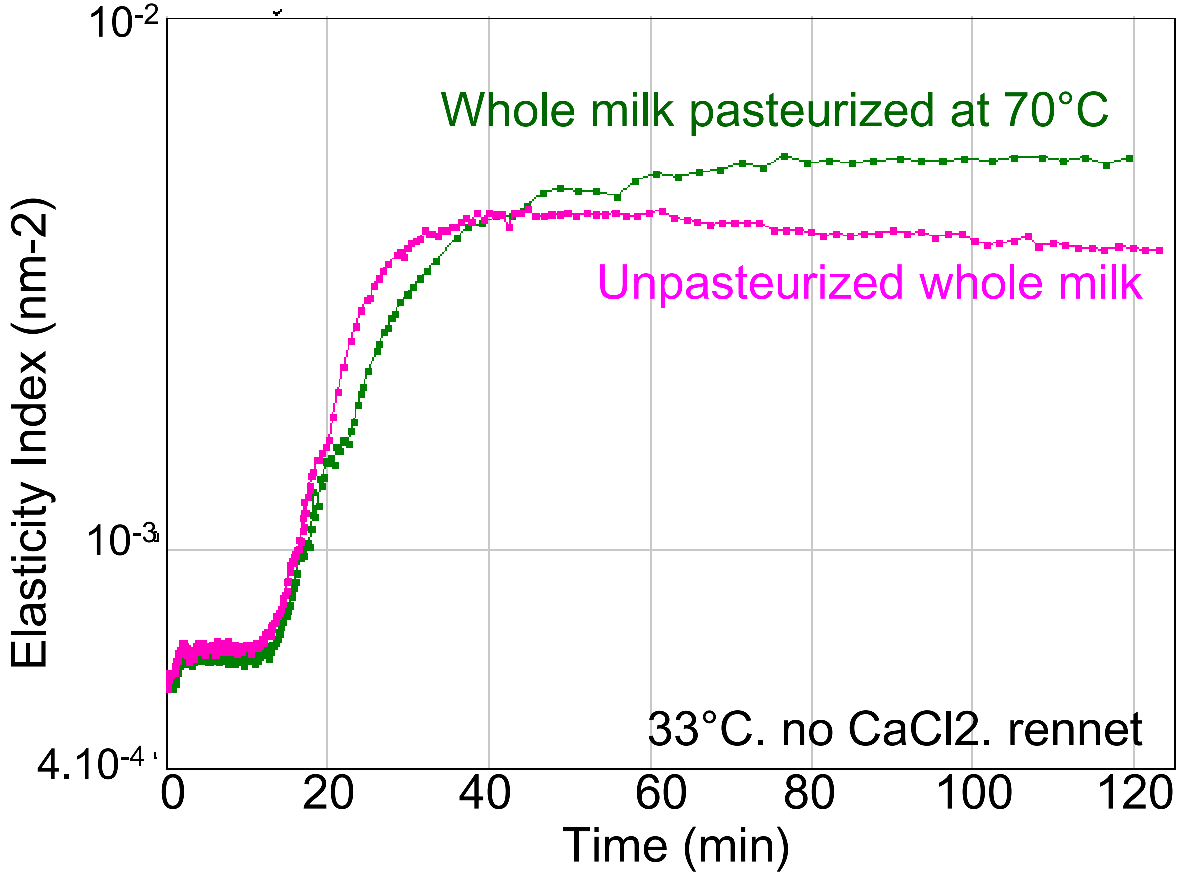
Elasticity Index (nm^{-2})



1. Enzymatic reaction
2. Flocculation
3. Formation of coagulation
4. Maximum elasticity of coagulum
5. Syneresis

Cheese making

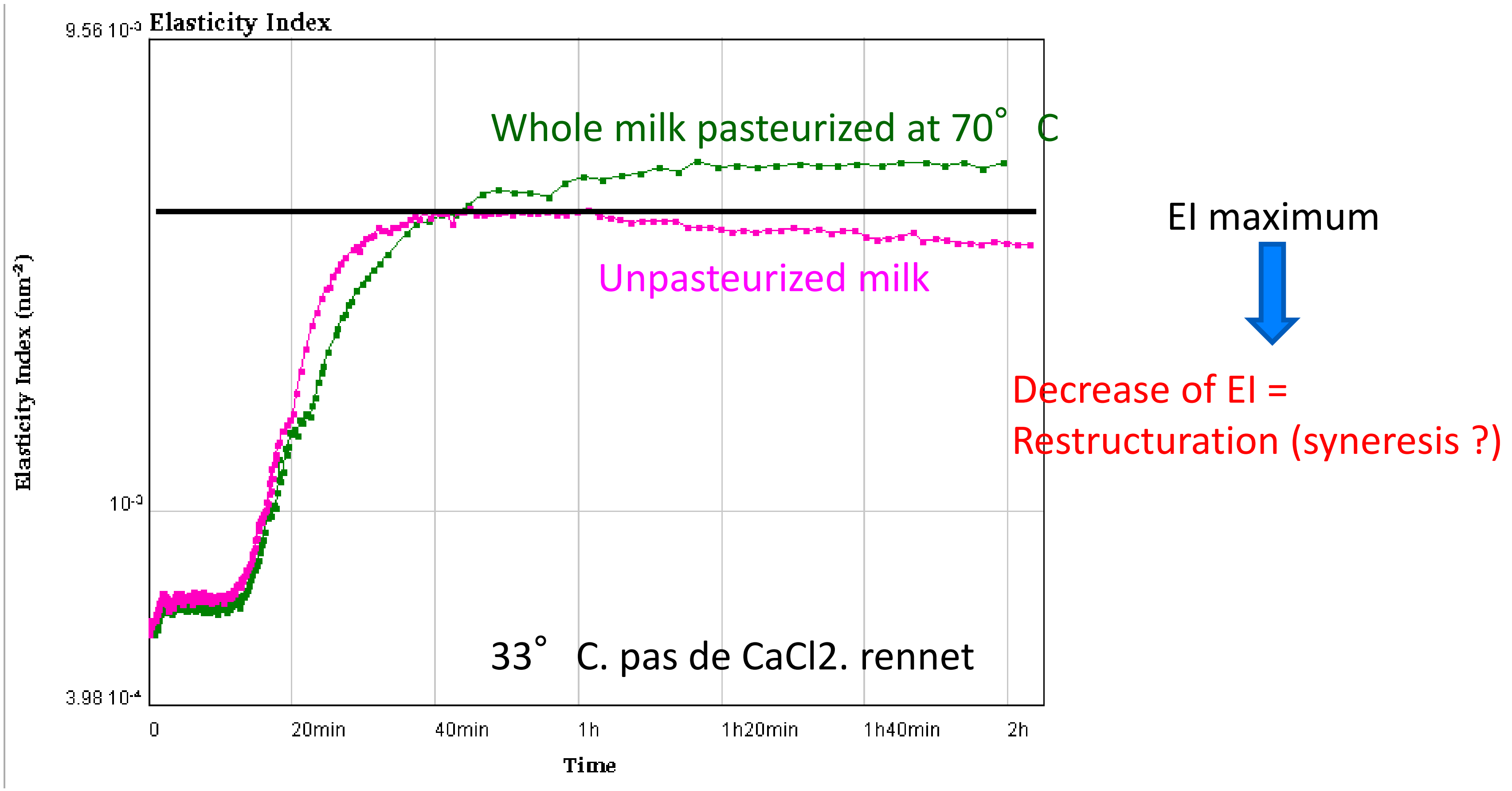
Influence of pasteurization



The pasteurized milk forms a stronger milk gel

Cheese making

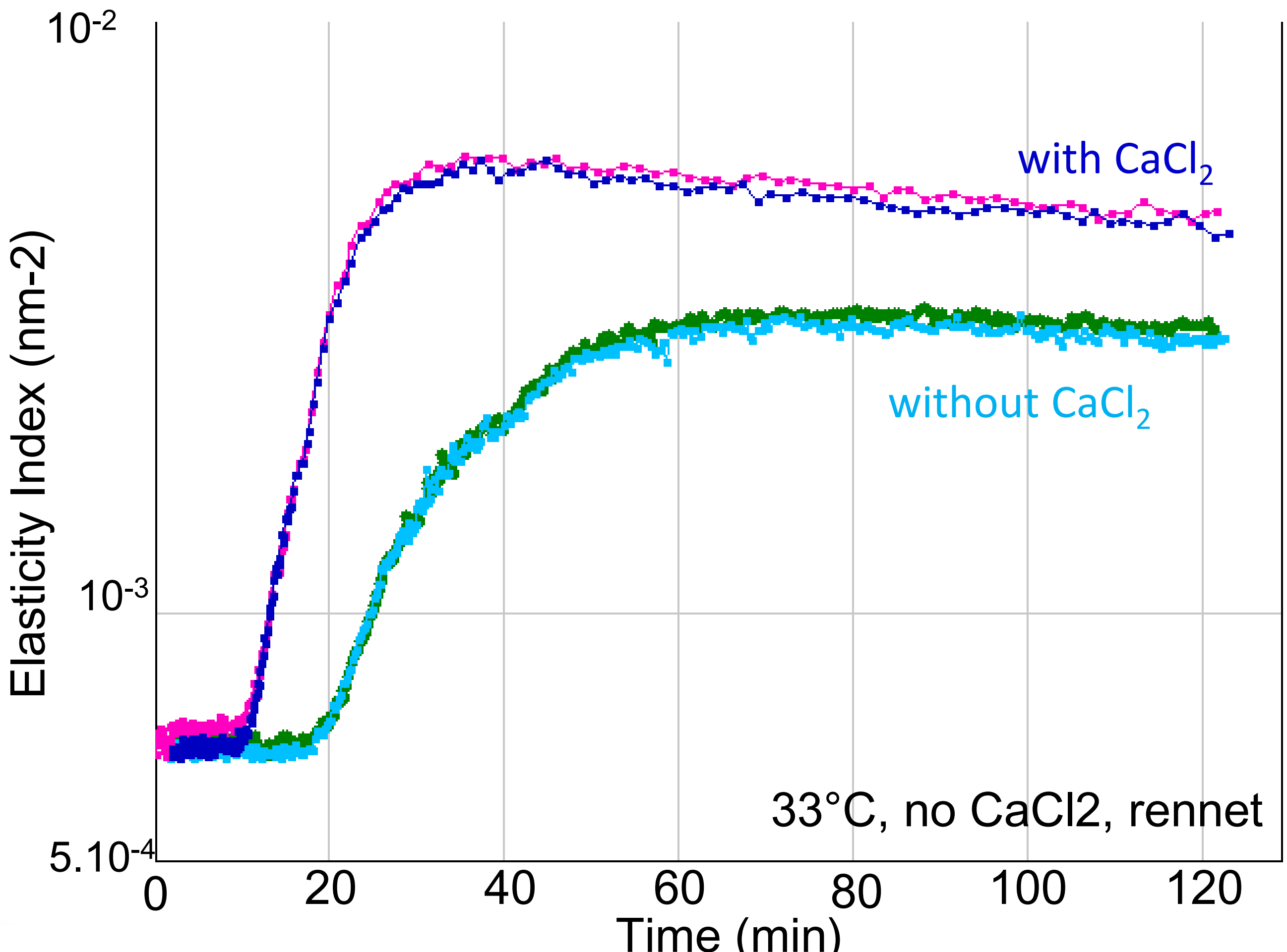
Observation of synergesis



Rheolaser allows the observation of syneresis

Cheese making

Influence of $CaCl_2$

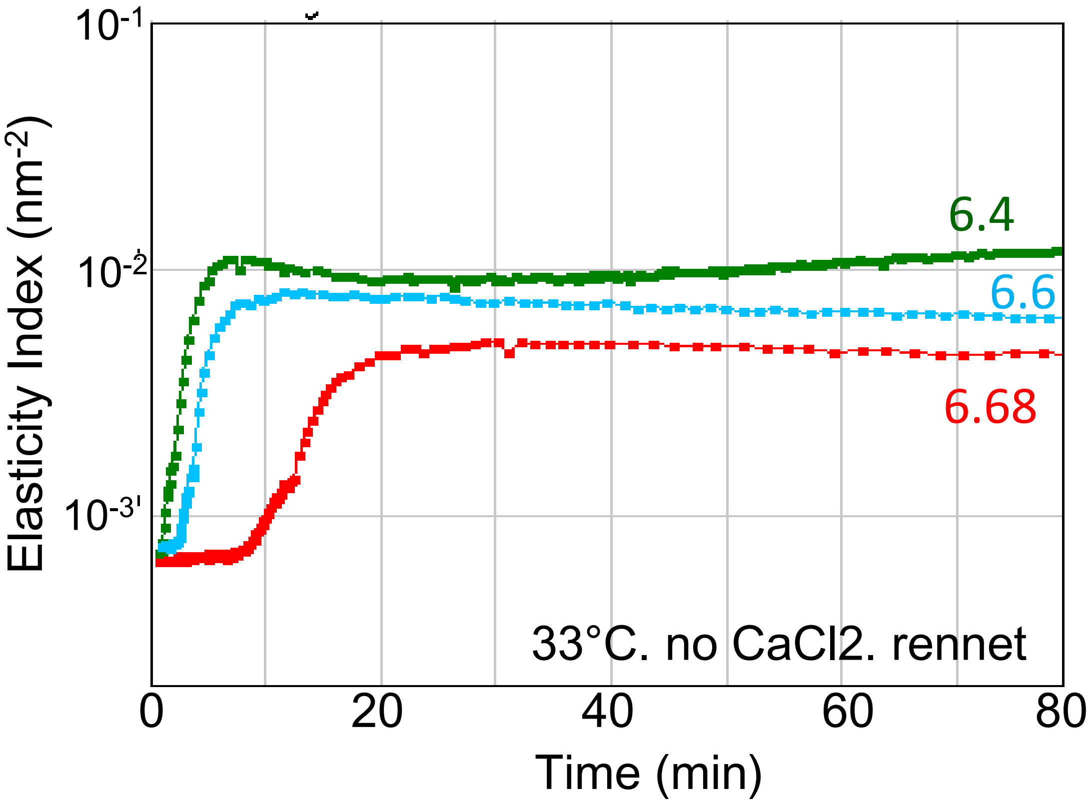


1. Milk with $CaCl_2$ addition forms gel earlier and with a higher gel strength
2. Milk with $CaCl_2$ undergoes syneresis earlier and more strongly → curd cutting has to be done earlier

Cheese making

Influence of pH

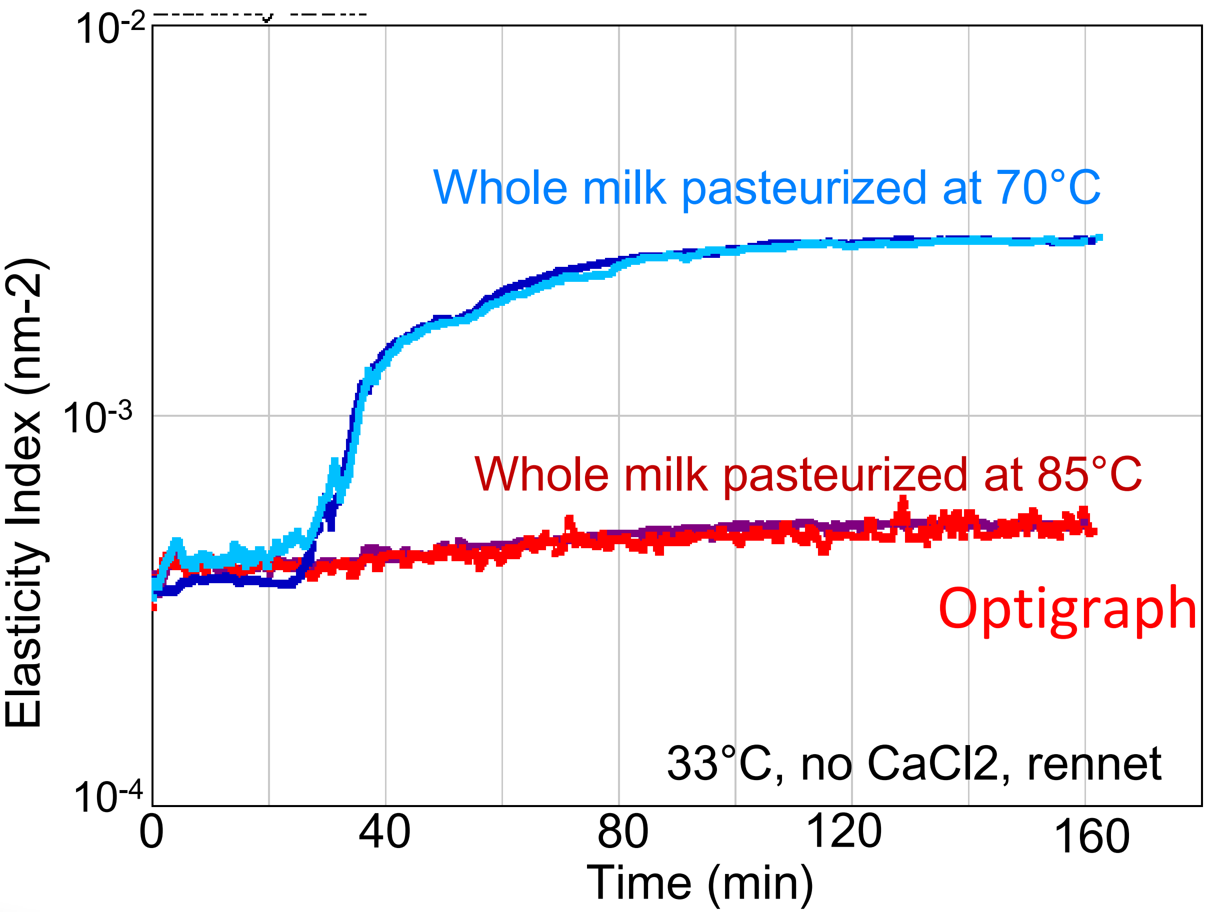
pH ajusted with GDL (glucono-δ-lactone)



1. The lower the pH, the better works the enzyme, the faster is gel formation

Cheese making

Influence of high temperature



Optigraph indicates « gel formation »

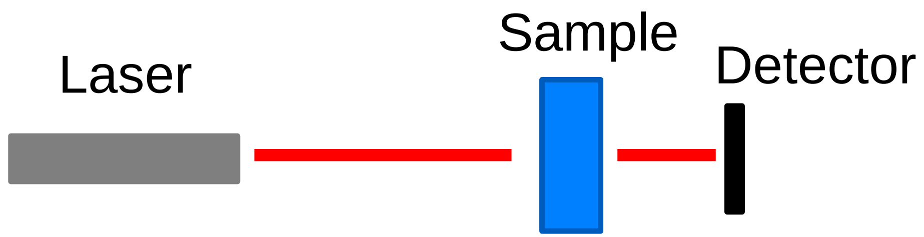
At 70° C → milk gel formation

At 85° C → only slight flocculation observed

Cheese making

Flocculation time – Rheolaser vs. Optigraph

Optigraph



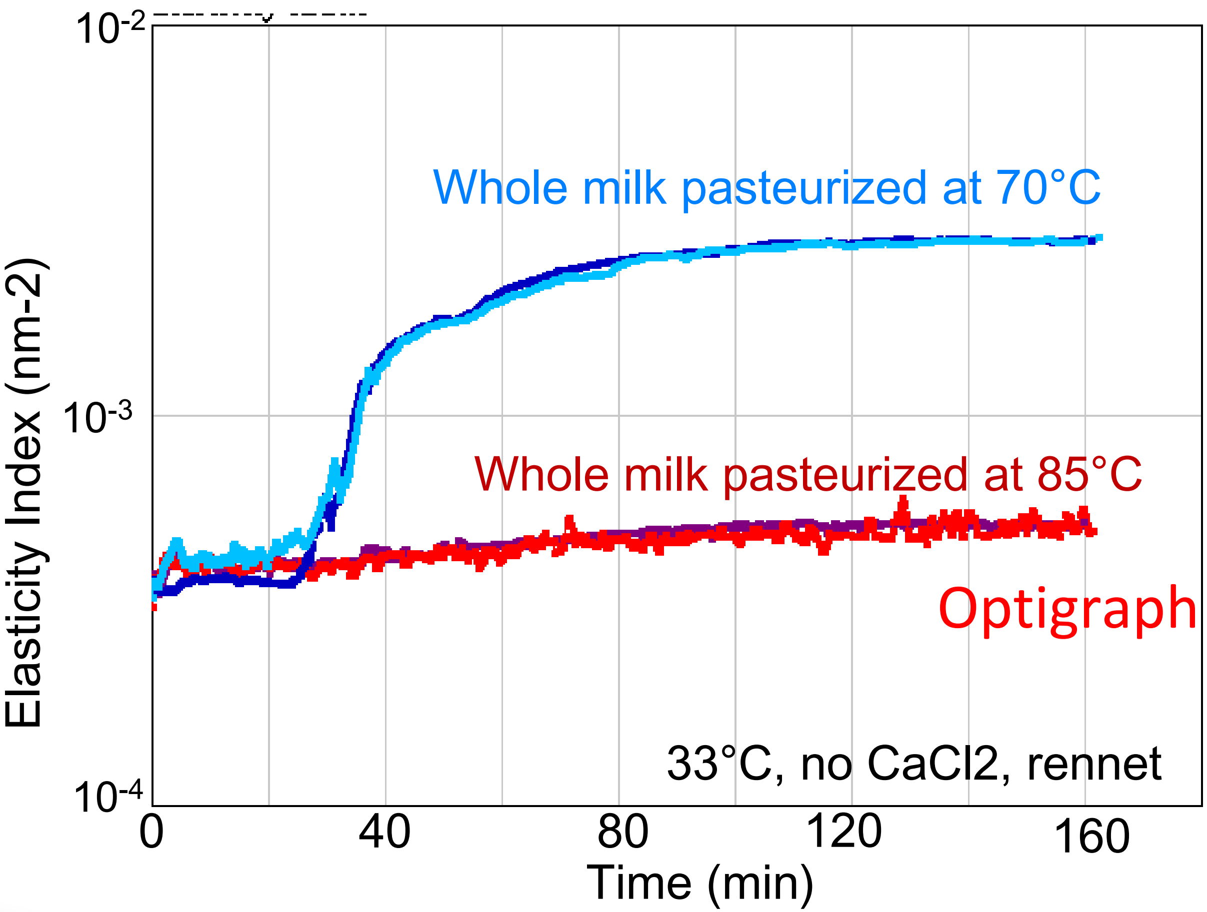
Measures the increase of laser power to maintain 4 mV at the detector

Purely optical method,
No correlation with rheological properties

Formagraph – not anymore produced

Cheese making

Influence of high temperature



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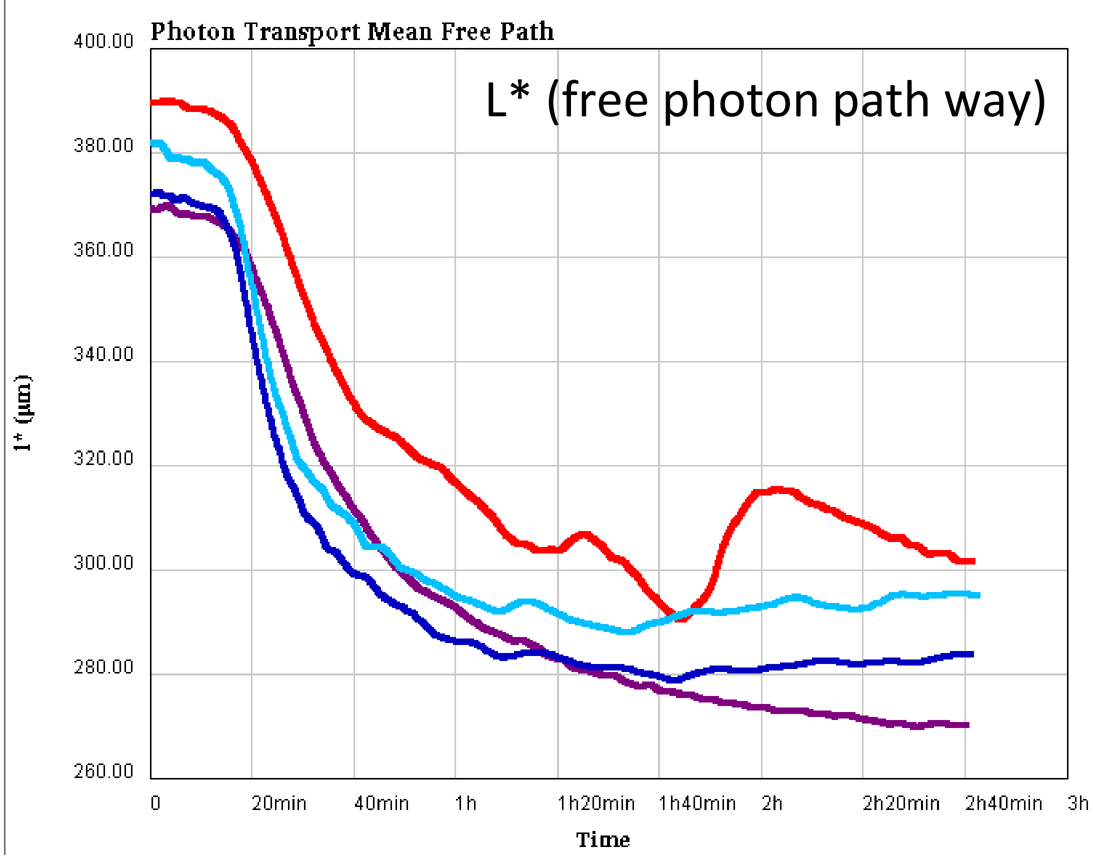
Cheese making

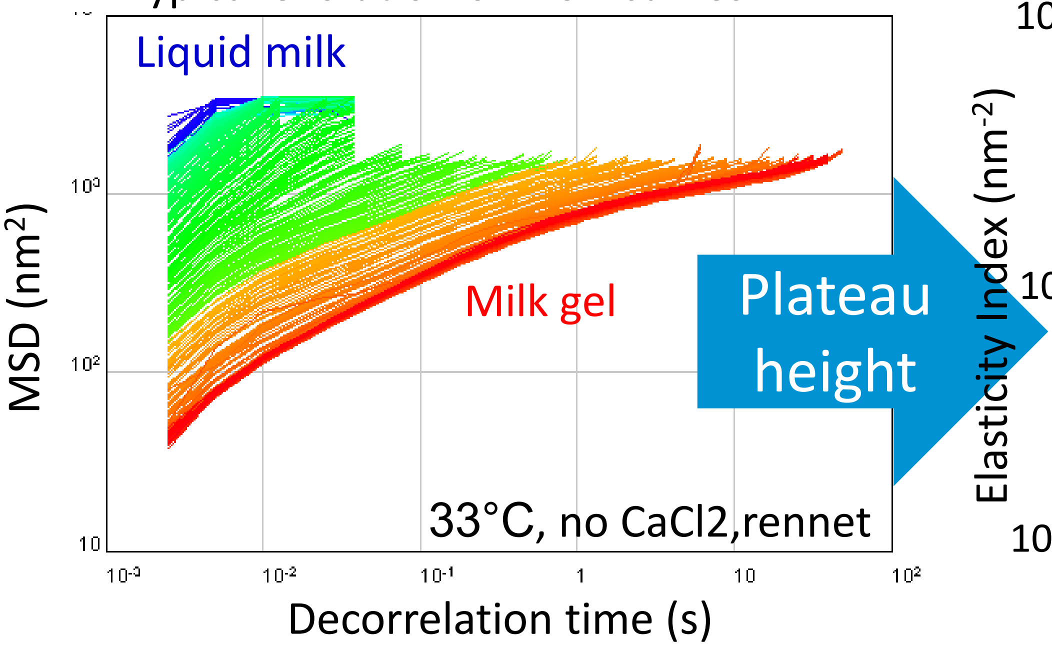
Influence of high temperature

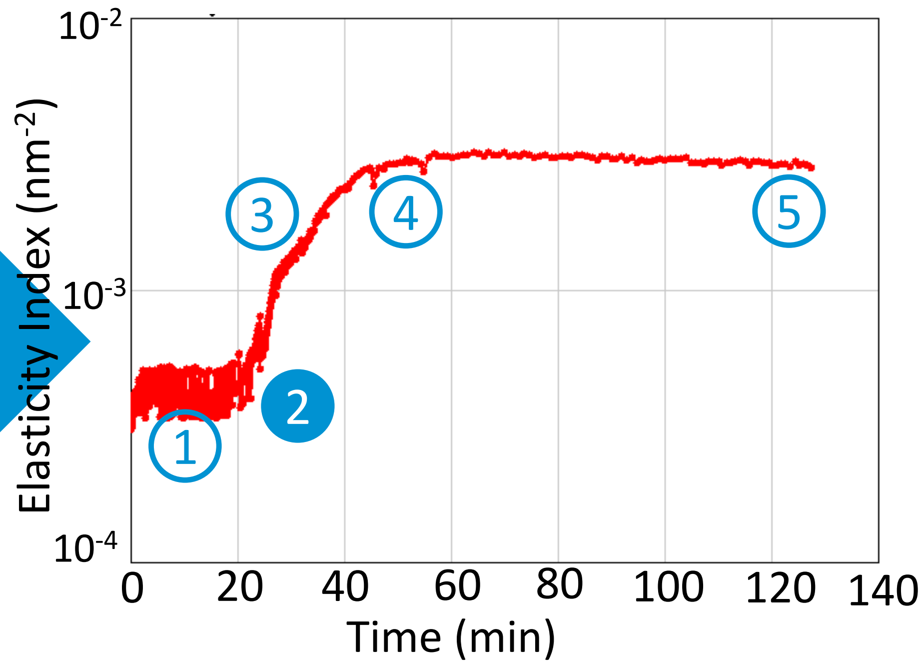


Cheese making

General resultsc

Typical evolution of MSD curves

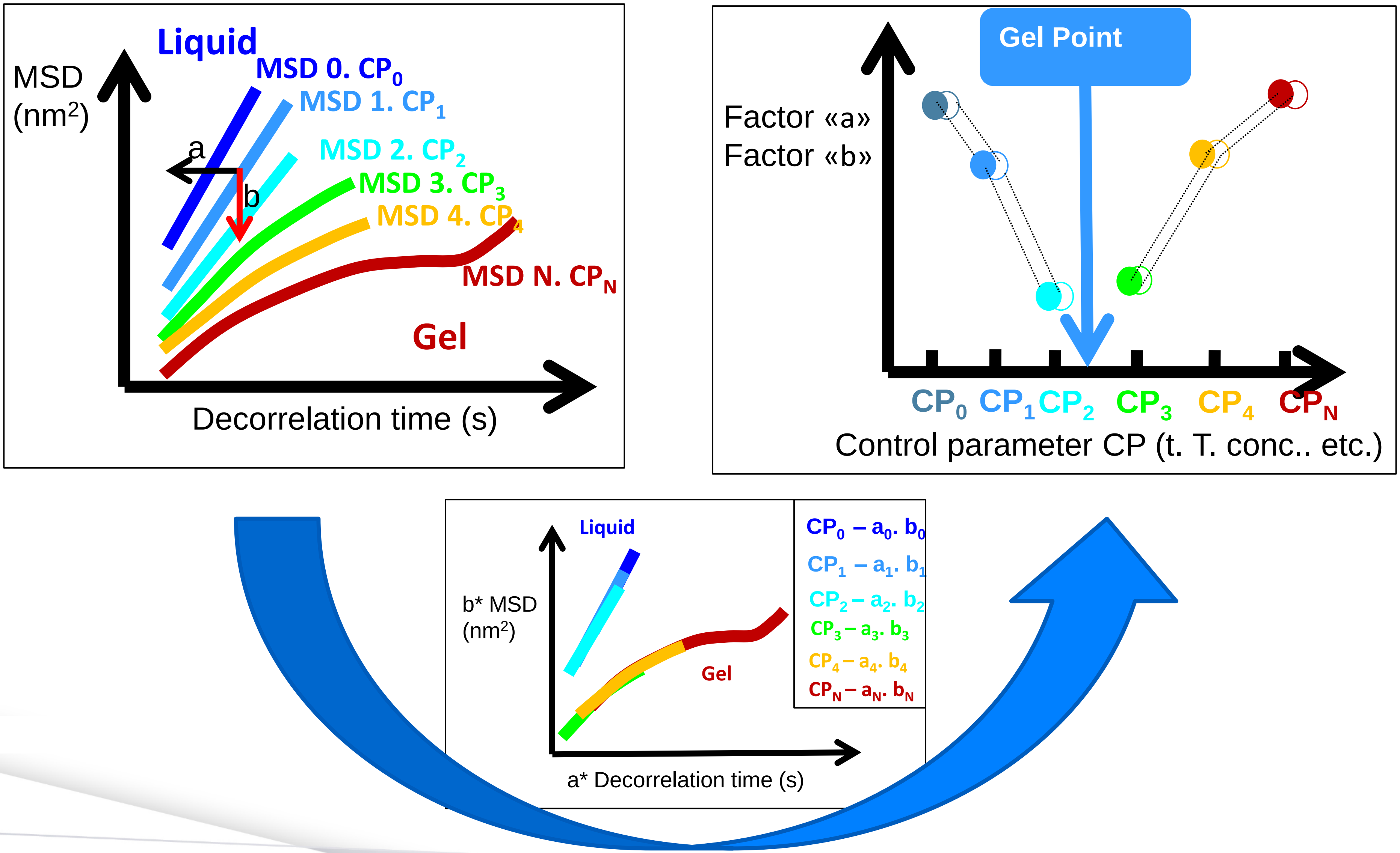




1. Enzymatic reaction
2. Flocculation
3. Formation of coagulation
4. Maximum elasticity of coagulum
5. Syneresis

Cheese making

Time cure superposition



Cheese making

Flocculation time – Rheolaser vs. Optigraph

	Rheolaser (min)
w/o $CaCl_2$	24.2 ± 0.2
with $CaCl_2$	12.9 ± 0.4
Milk. 3.3% proteins	9.2 ± 0.4
Milk. 4% proteins	11.6 ± 0.1
Milk. 5% proteins	14.2 ± 0.1
Unpasteurized Milk. 5% proteins	16.3 ±0.4
Pasteurized Milk. 5% proteins	14.5 ± 0.6

Cheese making

Flocculation time – Rheolaser vs. Optigraph

	Rheolaser (min)	Optigraph (min)
w/o $CaCl_2$	24.2 ± 0.2	23.8 ± 0.1
with $CaCl_2$	12.9 ± 0.4	15.7 ± 0.3
Milk. 3.3% proteins	9.2 ± 0.4	10.6 ± 0.1
Milk. 4% proteins	11.6 ± 0.1	13.7 ± 0.7
Milk. 5% proteins	14.2 ± 0.1	17.6 ± 1.9
Unpasteurized Milk. 5% proteins	16.3 ±0.4	16.6 ± 0.4
Pasteurized Milk. 5% proteins	14.5 ± 0.6	16.7 ± 0.3

Rheolaser and Optigraph have similar results.

In all cases, visual observation with spoon test are closer to Rheolaser

Cheese making
Conclusions

Milk gels for cheese preparations were studied

- Rheolaser determines similar values for flocculation time in comparison to Optigraph
- Rheolaser detects also « gel strength » and gel formation
- Rheolaser observes syneresis
- Rheolaser can use 6 independent positions
- What else ?

3. Curd cutting

Cutting is a very important step:

- Is it done too early – network is too loose and whey protein is loss

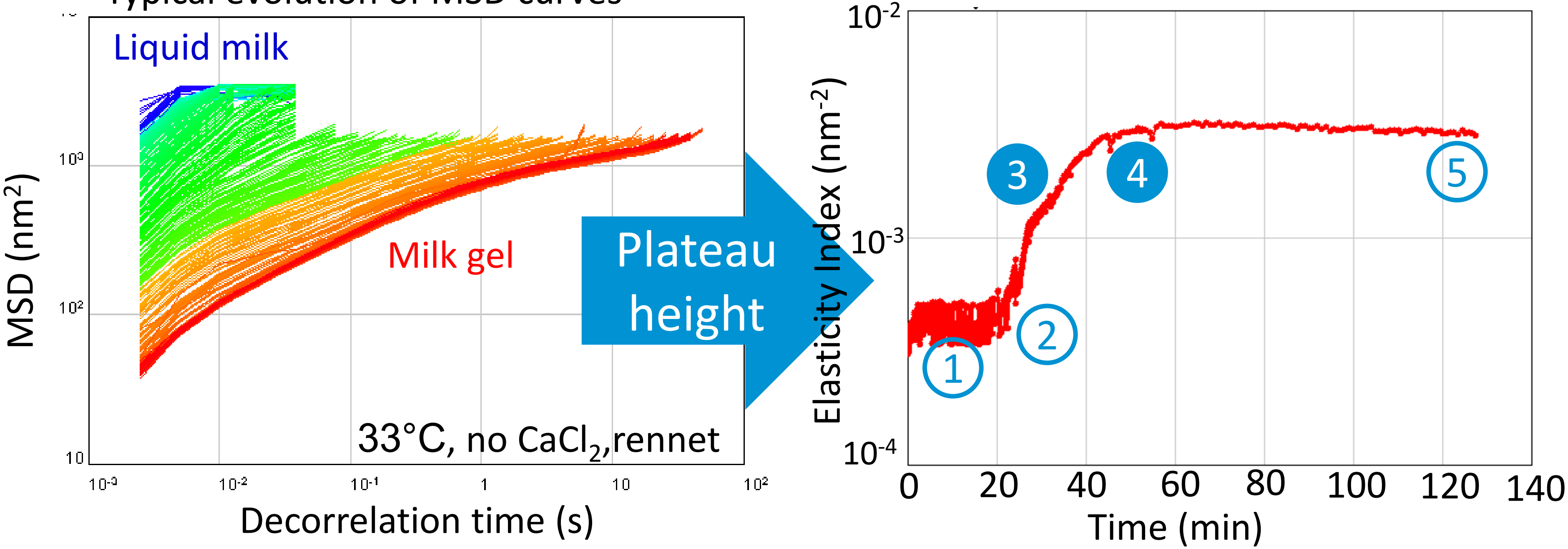
- Is it done too late – network is too strong and humidity is enclosed

Quality problems (taste, shape, etc) and yield loss !

Cheese making

Where is the ideal curd cutting time

Typical evolution of MSD curves



1. Enzymatic reaction
2. Flocculation
3. Formation of coagulation
4. Maximum elasticity of coagulum
5. Syneresis

Formulaction
Smart scientific analysis

Cheese making

General results

New project on curd cutting time determination in collaboration with the

Agriculture Engineering School Toulouse (INPT) and several companies in France.

INP TOULOUSE PURPAN Learning for the future

Ecole d'ingénieurs

Thank you for your attention !

INP Purapn:
Hélène Tormo
Loubnah Belahcen

Interns:
Fernando Egea
Chloé Berthau