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NMR and texture measurements to characterize the evolution of apple microstructure during thermal treatment

A. Leca, S. Clerjon, A. Traore, J.M. Bonny, C.M.G.C. Renard



INTRODUCTION



Joint Research Unit (UMR) for the Safety and Quality of Plant Products

Team Quality & Process Engineering

Our main research questions :

Understand how fruits & vegetables interact with unit operations to build the quality of processed products

→ Fruits & vegetables products texture and rheology

→ Cell wall evolution along process

→ Analytical development to characterize fruits & vegetables heterogeneity

→ Fast analytical methods (Visible-NIR-MIR spectroscopy)

→ Tissue structure and texture analysis

→ Modeling the process and quality build-up



INTRODUCTION



CEPIA



NMR Platform for agronomy, food science & nutrition

NMR/MRI tools :

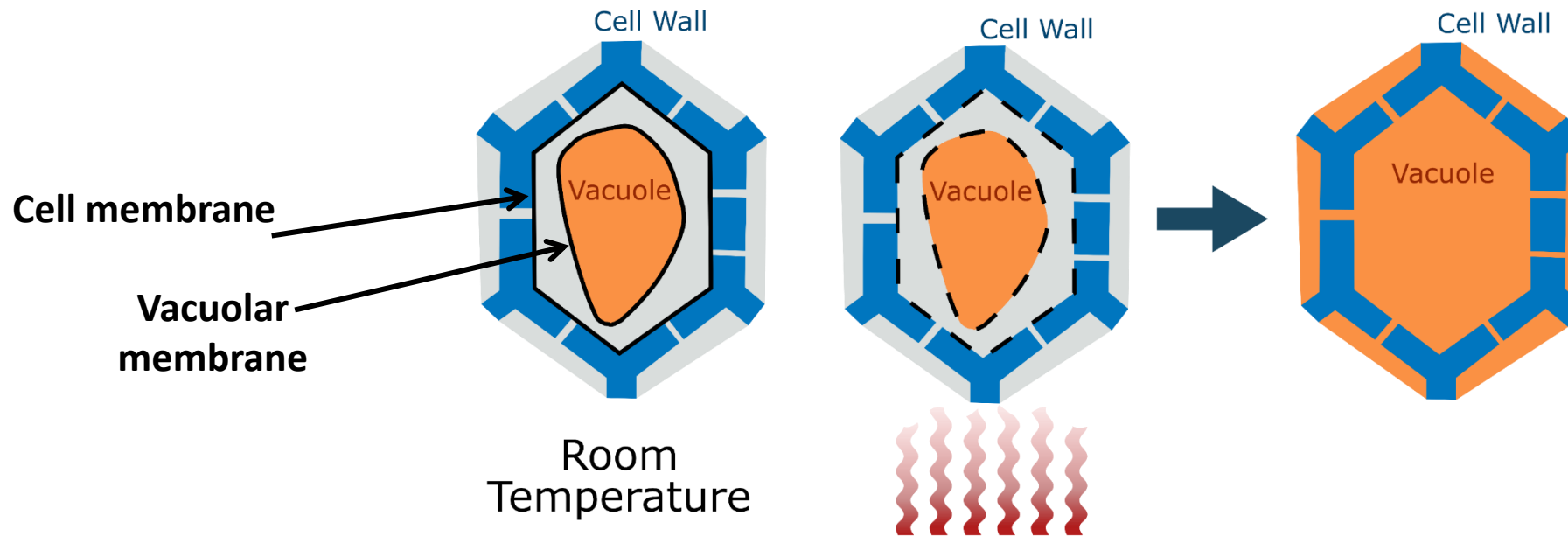
- Highly skilled researchers and engineers
- In-vitro and in-vivo experiments
- Dedicated building with 3 specific equipments :
 - 4.7 T horizontal imager
 - 11.7 T horizontal imager and spectrometer
 - 9.4 T vertical imager and spectrometer



IT'S ALL ABOUT TEXTURE

**Problematic : Apple texture drops during cooking.
But what really happens and at which temperature and rate?**

90% of water in fresh apples is located in intracellular space...
What happens during cooking, how does microstructure evolve?
Is the "leak" of vacuolar fluid simultaneous with the loss in texture?



The study : follow texture and vacuolar water in apples

Objective :

- Understand and quantify the effect of thermal treatment on apple microstructure

How :

- By cooking pieces of apple flesh at different temperatures and measure :
- Their texture with a reference mechanical test
 - The water mobility in flesh with NMR imaging and spectroscopy

Materials & Methods : Fruits & cooking

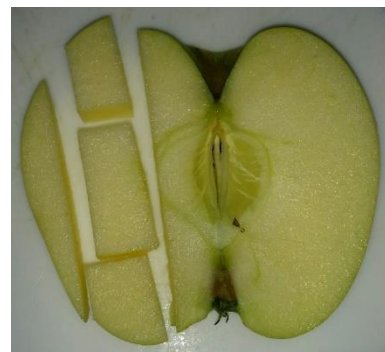
Apples



cv. Golden Delicious



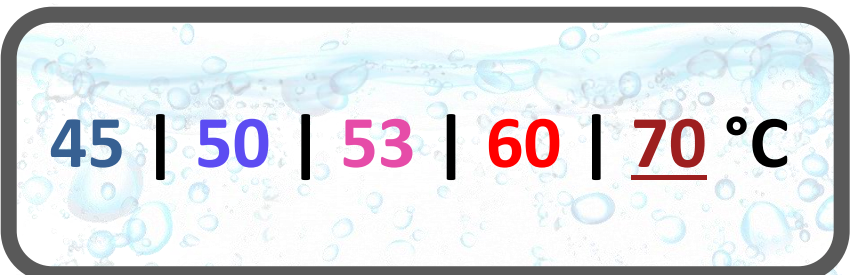
Slabs cut into the parenchyma



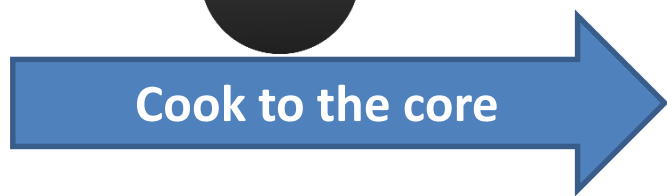
Sealed in vacuum bags

Cooking Conditions

Raw



Bain-marie



24 hours rest at room temperature

Texture Analysis

Puncture test



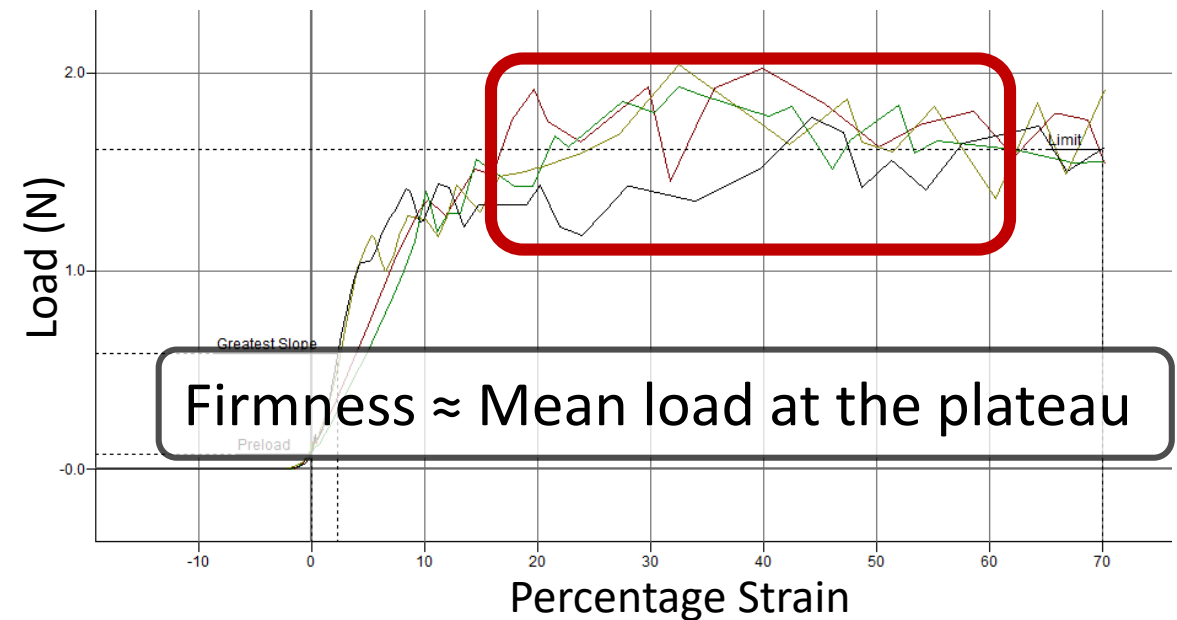
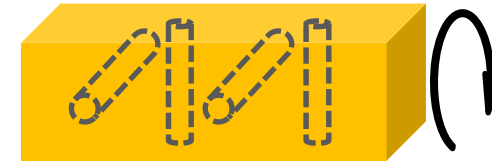
50 N Load Cell



2 mm

Cylindrical punch : $\varnothing = 2 \text{ mm}$, $L = 17 \text{ mm}$
 Descending speed : $100 \text{ mm} \cdot \text{min}^{-1}$
 Test limit : percentage strain $\varepsilon = 70\%$

2 punches on 2 orthogonal sides of slabs
 6 repetitions for each temperature



Magnetic Resonance Imaging

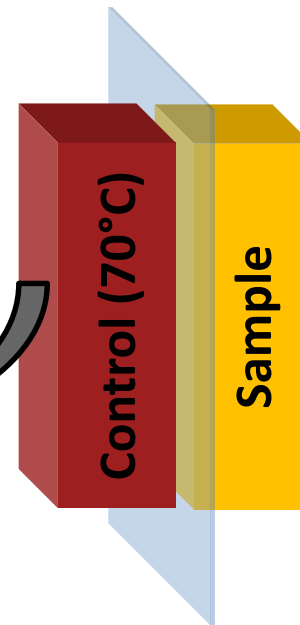
T2 MRI



Spatial resolution : 1 mm

Time to produce MRI image for 1 slab : 32 min

9 echo times from 6.5 to 200 ms combined to produce a T2 map



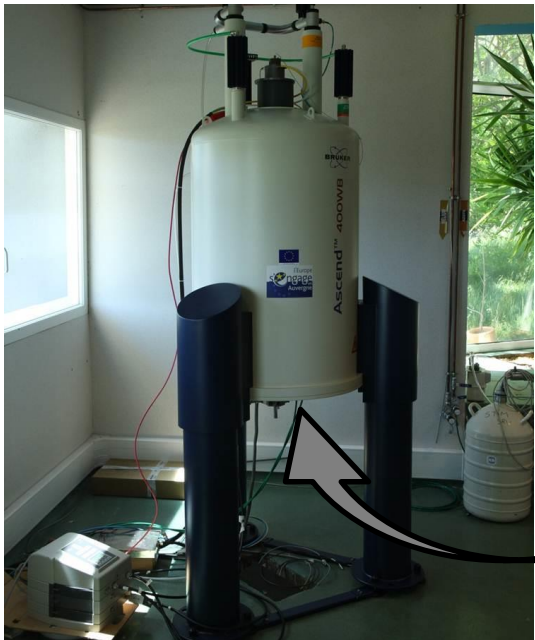
- Each sampled slab placed next to a control slab cooked at 70°C, separated by a plastic film
- 6 repetitions for each temperature (raw, 45, 50, 53, 60 °C)

9.4T Bruker Ascend 400WB
Ø 30mm ¹H volume coil

NMR Spectroscopy

T2 with CPMG (Carr-Purcell-Meiboom-Gill) sequence

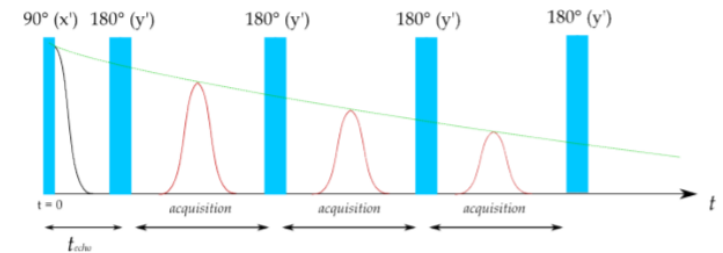
- Quantitative NMR to study water dynamics in a product by following the echo decay signal by repeating impulsions
- Well suited to avoid signal diffusion



9.4T Bruker Ascend 400WB
Ø 30mm ^1H volume coil



$t_{\text{echo}} = 500\mu\text{s}$
 $n = 128$ data points/rep.



Source : A. Comment, 2014, EPFL

- Single slabs placed into the detector, always oriented along the tangential axis
- 6 repetitions for each temperature (raw, 45, 50, 53, 60 °C)

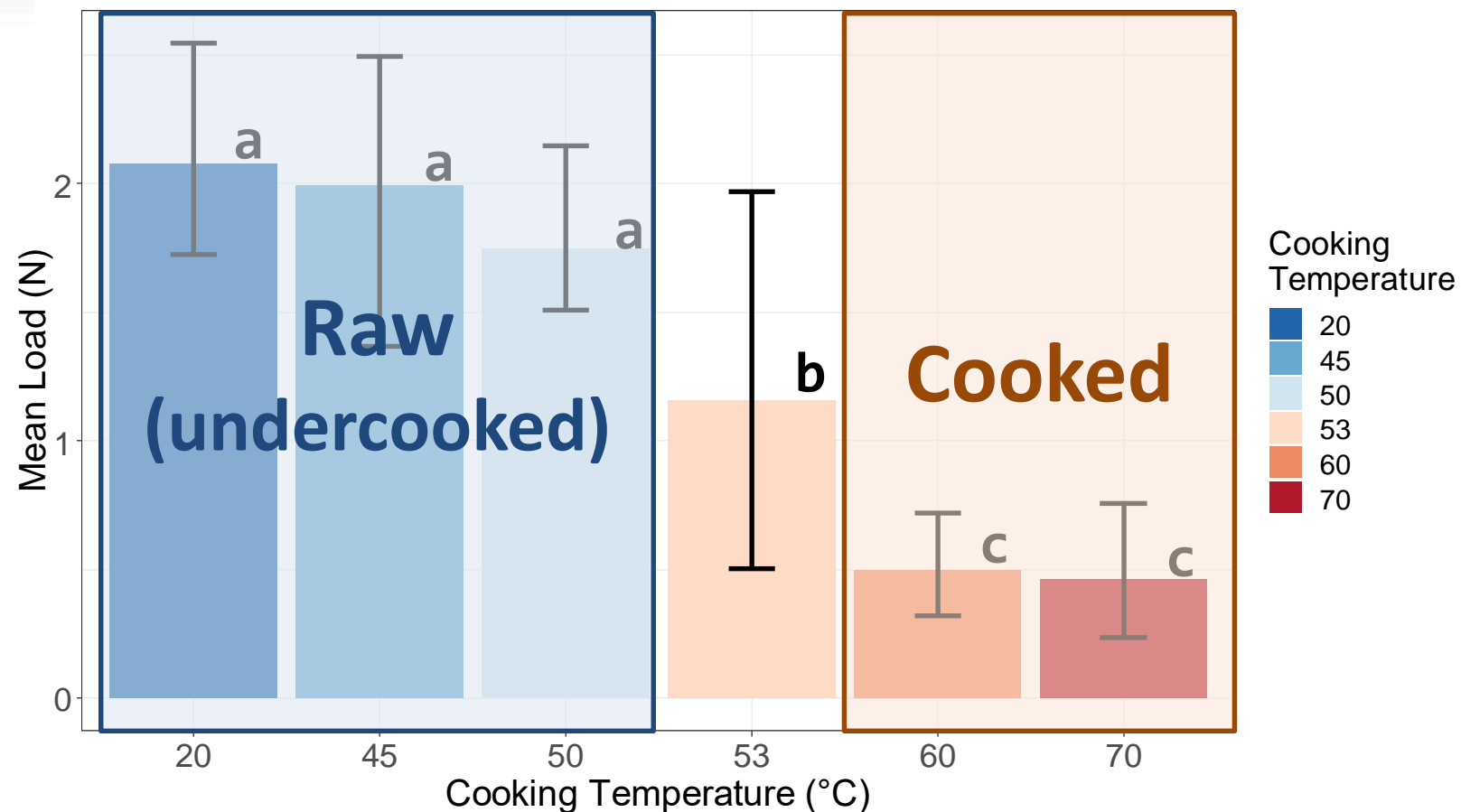
Texture analysis results : firmness

Three homogeneous groups
(Tukey test, $\alpha=0.05$)

Two main groups :

- Cooked
- Undercooked

Transitional state at 53°C



MRI Results : T2 maps



Dark : 'bound water'
Light : 'free mobile water'



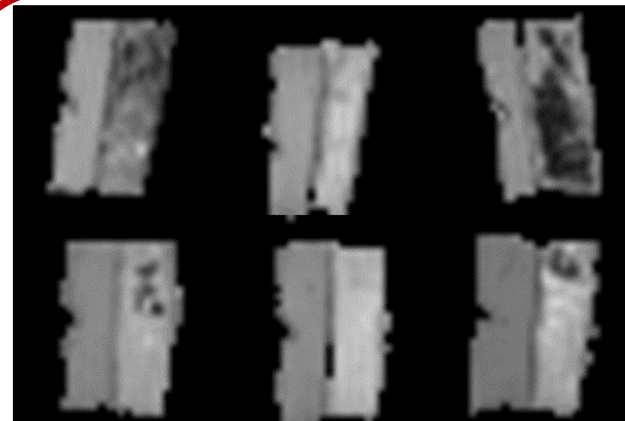
RAW



45°C



50°C

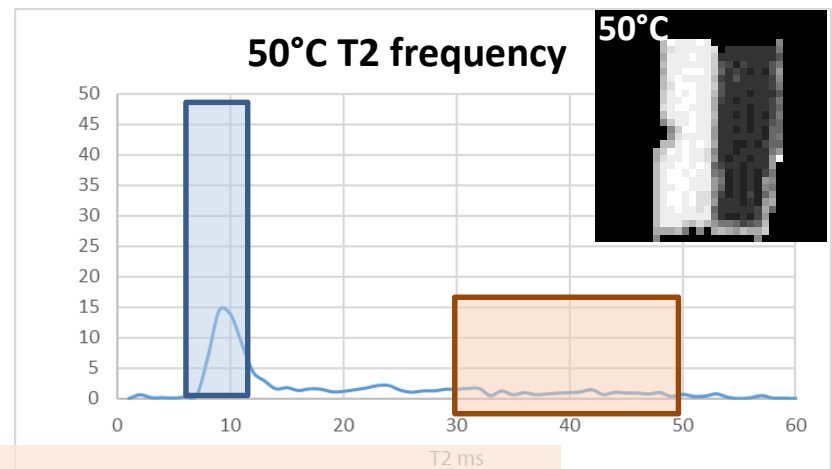
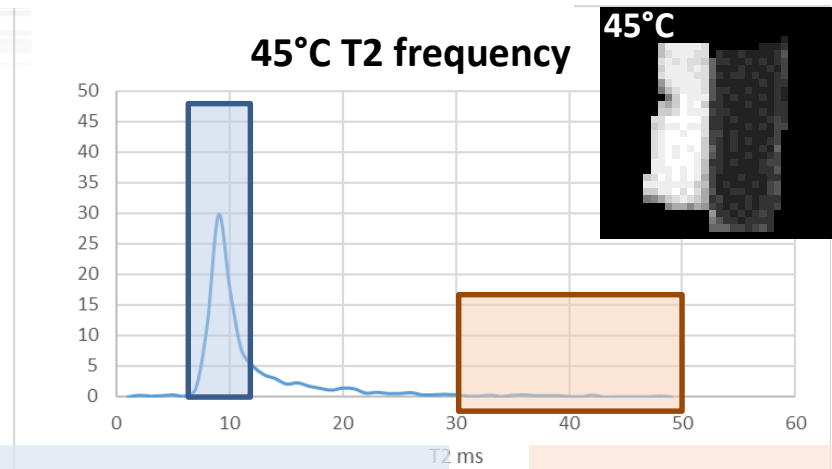
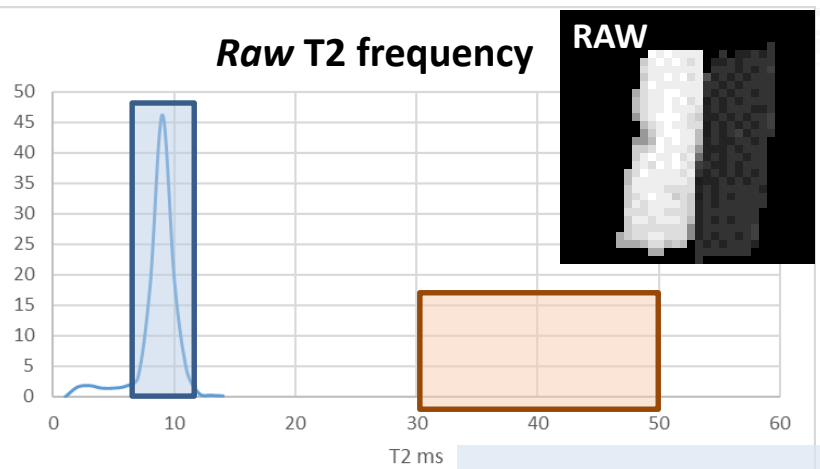


53°C



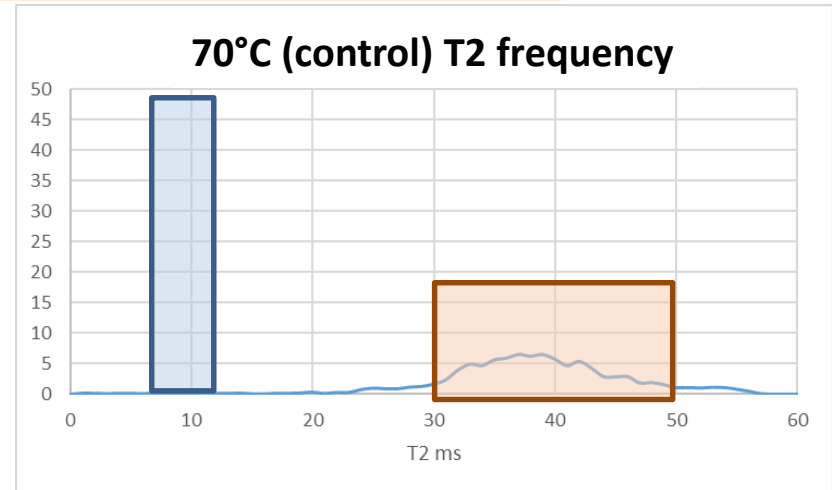
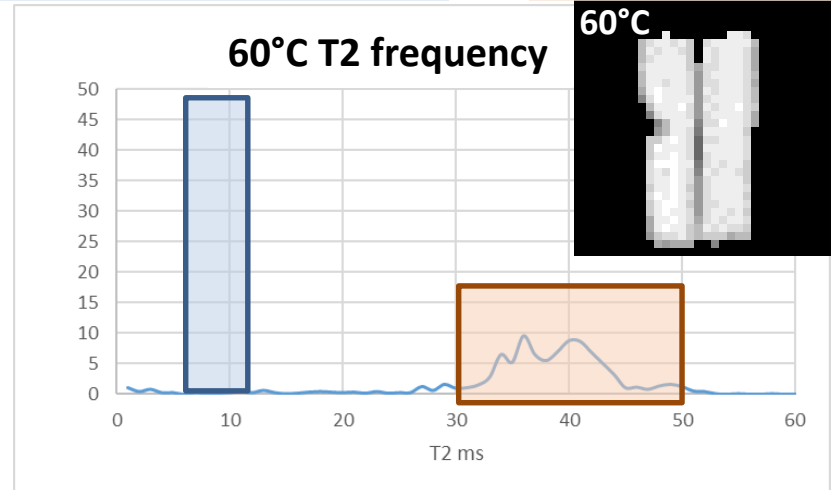
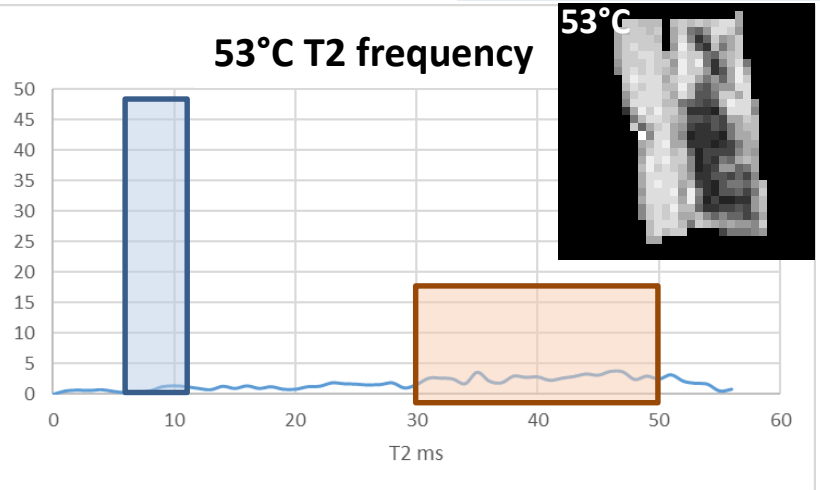
60°C

NMR Results : MRI T2 frequencies



Lower T2 range : 7-11 ms

Higher T2 range : 30-50 ms



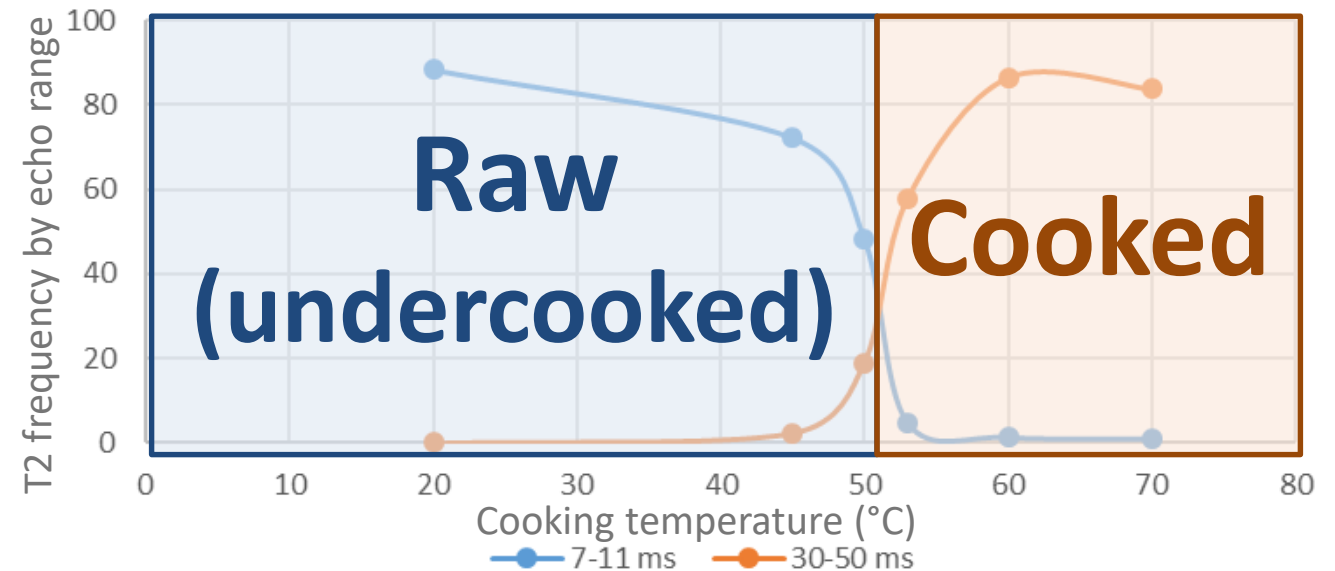
NMR Results : MRI T2 synthesized

Two main T2 populations

Drastic increase in mean T2 when cooked warmer than 50°C

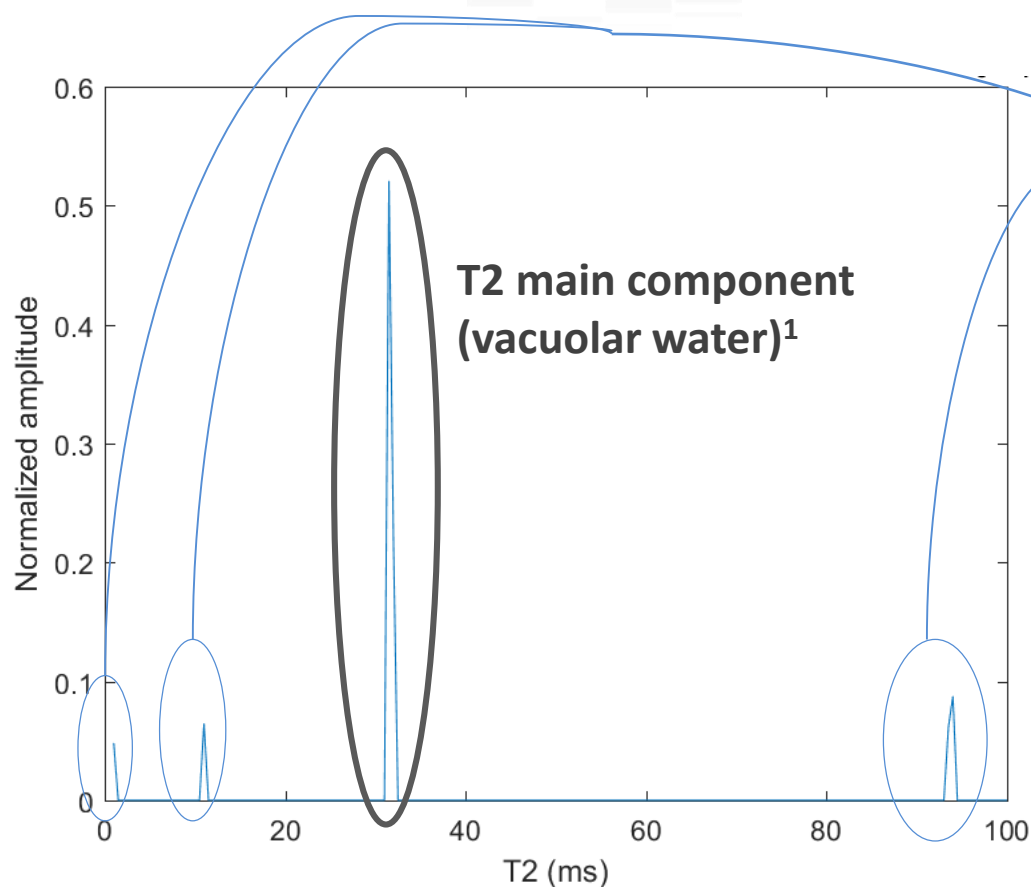
Summarizing the T2 mapping frequency reveals a clear change of water state around 50°C

Evolution of *raw* and *cooked* T2 as a function of temperature



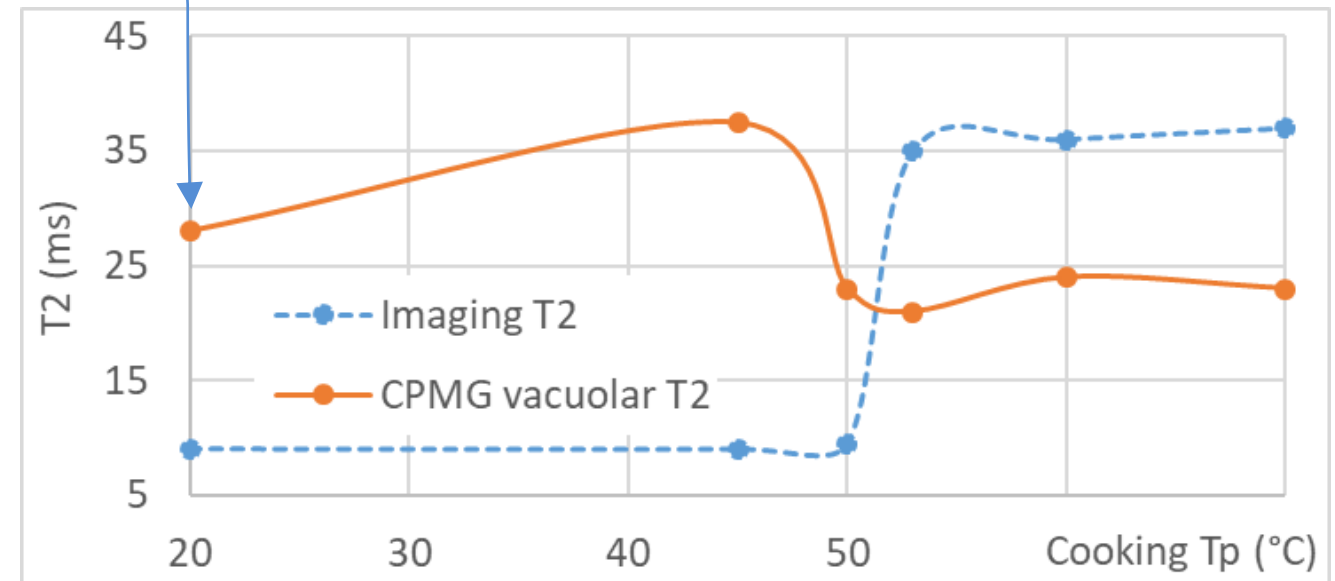
20°C = raw apple slabs

NMR Results : Spectroscopy T2



Example of spectrum analysis on a raw slab

CPMG curve = weighed average of all T2 peaks

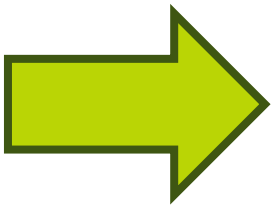


Quantitative NMR shows a drastic change at 50°C

1. Hills & Remigereau, 1997

Results Synthesis

Texture and NMR show almost similar results

- Firmness drops around 53 °C
 - MRI shows a shift in mean T2 between 50 and 53 °C
 - Quantitative NMR shows a drastic drop in T2 weighed average at 50°C
-
- 
- The microstructural and textural change occur in the 50-53 °C range
 - Mechanical evolution (loss in firmness) occurs at higher temperature than release of vacuolar water

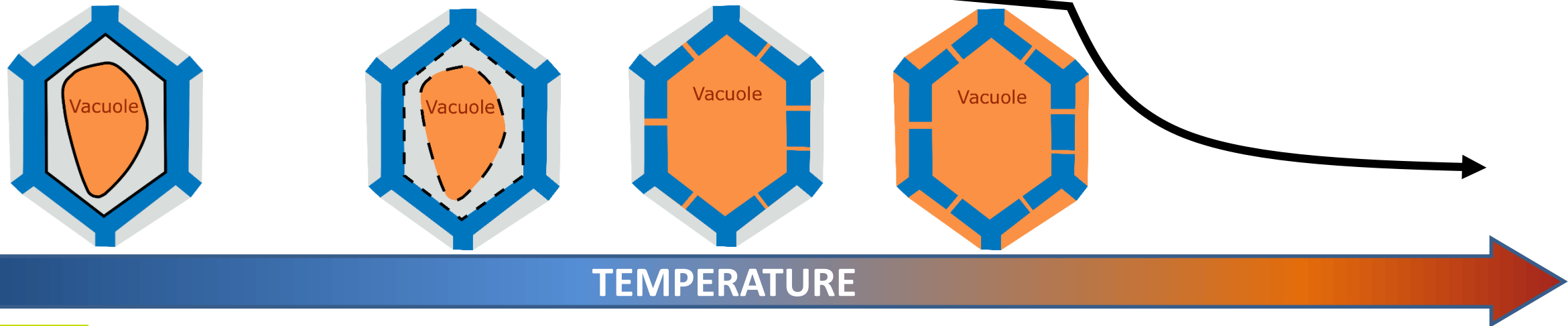
Results Synthesis

Results interpretation : the mechanism is complex and multi-scale

Thermal treatment impact occurs in steps :

1. $<50^{\circ}\text{C}$: cell and plasmalemma resist to heat
2. 50°C : plasmalemma starts to be destroyed and begins to « leak »
3. $\approx 53^{\circ}\text{C}$: structure is significantly affected, causing loss in firmness
4. 53 to 60°C : firmness drops to a floor value

FIRMNESS



Conclusion & where we go from now

The thermal denaturation and its effect on apple firmness has been quantified

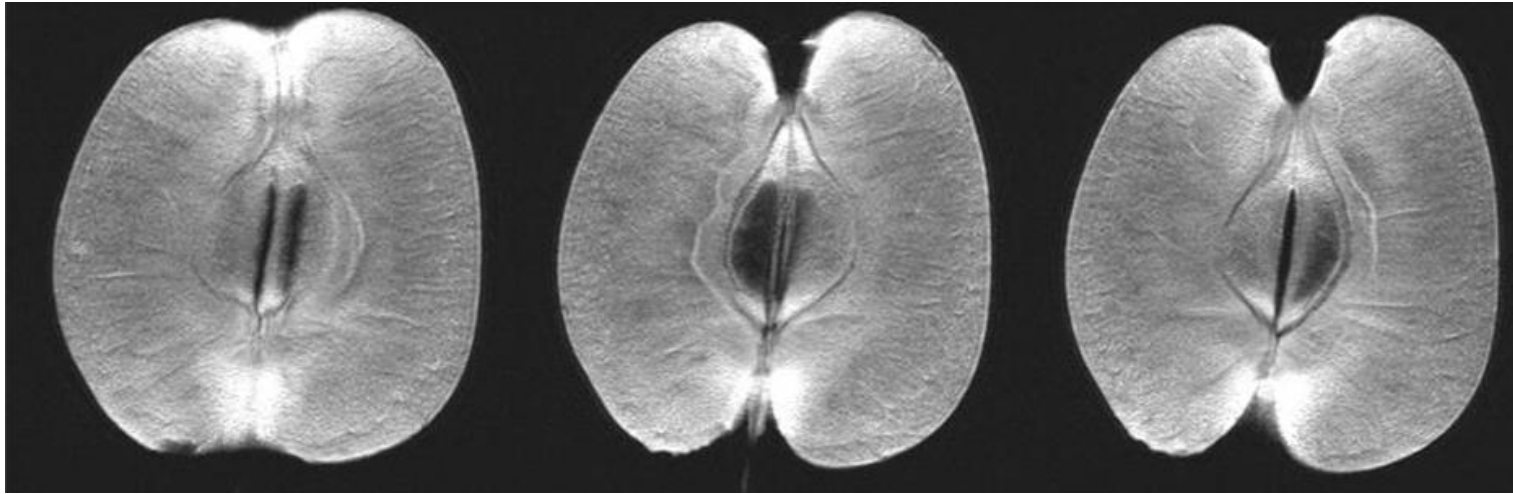
NMR is an efficient method to characterize the evolution of microstructure and understand the underlying mechanisms

NMR is an efficient method to anticipate a loss of texture

NMR spectroscopy is a self-sufficient method to describe such mechanism (no more valuable information with MRI)

Go further :

- Test on more varieties/species which underwent thermal treatment
- Extend study on the porosity of cell wall heated over 50°C



Thank you for your attention!