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Data Article

Puncture, MRI and NMR relaxometry data for multiscale analysis of the degradation of apple structure due to thermal treatment

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ABSTRACT

The data presented here are related to the research paper entitled "Multiscale NMR analysis of the degradation of apple structure due to thermal treatment" whose aim was to investigate the critical temperature at which the cell membranes of a Golden Delicious apple is highly damaged. Apple sticks were analyzed raw and cooked at 45, 50, 53, 60 °C and 70 °C. The firmness data refers to the puncture tests that were done using a Ta-Plus texturometer. The nuclear magnetic resonance (NMR) relaxometry and imaging data were both acquired with a 9.4 T 400WB instrument. For these three raw data collections, analysis results are also provided. These data are complementary as they cover the different scales from molecular to nearly the whole food system to enlighten the process of membrane degradation during thermal processing of apple. Our NMR data could be reused to optimize inversion algorithms dealing with ill-posed inverse problems. Both firmness and NMR data could be added to databases on food structure studies, either in physico-chemical data handbooks or review studies. Finally, these data could also be reused for the optimization of food thermal processing control.

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Specifications Table

Subject	Food Science
Specific subject area	Apple thermal processing
Type of data	Table Image Graph Figure CPMG decay NNLS outputs Matlab code R code Documentation
How data were acquired	Food Texture Analysis: The puncture tests using a Ta-Plus texturometer (Lloyd Instruments Ltd., Bognor Regis, UK) equipped with a 50 N load cell and a punch 2 mm in diameter and 17 mm long. Software: Nexygen (Ametek, Lloyd Instruments, UK), version 4.5 NMR: High resolution MRI, multi-echo MRI and spectroscopy CPMG using a 9.4 T Bruker Ascend 400WB instrument (Bruker, Ettlingen, Germany) equipped with a microimaging accessory and using a 32 mm diameter birdcage radiofrequency coil used for both excitation and signal reception, at 20 °C. Softwares: Topspin (Bruker), version: 3.2 (NMR data), ParaVision (Bruker) version: PV6.0.1 (MRI data)
Data format	Raw (NIFTI, CSV, TXT) Analyzed (NIFTI, JSON)
Parameters for data collection	Golden Delicious apples were cut in sticks ($12 \times 8 \times 30$ mm^3) vacuum-sealed in food-grade plastic bags. Data were acquired for raw (25 °C) and five thermal treatments, namely at 45, 50, 53 °C, and 60 °C along with the reference one which was cooked at 70 °C. All data were acquired at room temperature.
Description of data collection	1. Firmness: Puncture tests were performed on each individual sample and conditions, data reduction, data averaging 2. MRI: High resolution anatomical MRI acquisitions, Spin Echo imaging at variable echo time acquisitions, T2 relaxation maps were constructed by pixel wise monoexponential fitting of signal intensity decay 3. NMR relaxometry: Acquisitions of T2 [transverse relaxation time, Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence] decays, analysis of T2 data decays with regularized NNLS algorithm to obtain T2 distributions
Data source location	Golden Delicious apples were purchased at a local supermarket in Avignon (FR). NMR and MRI raw data were collected in Saint-Genès-Champanelle (FR, coordinates: 45.71166, 3.01411) and are securely stored in INRAE Data Center located in Toulouse (FR) with a replication in the Data Center of Jouy-en-Josas (FR). Firmness data were collected at INRAE PACA research center in Avignon (FR, coordinates: 43.91539, 4.87846) and are securely stored in INRAE data storage service in Avignon (FR) and in INRAE Data Center in Toulouse (FR).
Data accessibility	Repository name: Data INRAE Data identification number: doi.org/10.15454/HHAFZF; doi.org/10.15454/MMBJFF; doi.org/10.15454/AHLT8G Direct URL to data: https://data.inrae.fr/dataverse/pomCuite
Related research article	Leca, A., Clerjon, S., Bonny, JM., Renard, C., and Traore A. "Multiscale NMR analysis of the degradation of apple structure due to thermal treatment." Journal of Food Engineering 294: 110413, 2021 https://doi.org/10.1016/j.jfoodeng.2020.110413

Value of the Data

- These data complementarily cover the different scales from molecular to the whole food system (object) to enlighten the process of membrane degradation during thermal processing of apple.
- These data would benefit scientists and industrials interested in food process optimization, and data treatment scientists using inversion algorithms optimization.
- CPMG and MRI data could be used to optimize inversion algorithms dealing with ill-posed inverse problems. All data could be added to databases on food structure studies (other apple varieties or other species), either in physico-chemical data handbooks or review studies. Data could also be used in food thermal processing control.

1. Data Description

The dataset containing the spectroscopic CPMG data named “CPMG data of cooked apple” can be found at datainrae repository (https://doi.org/10.15454/AHLT8G). It contains 30 text files named PX_Y_T2.txt, where X is just a sample number from 1 to 31; *Y* represent the temperature at which the simple is cooked with "Cru" for non cooked sample. Each file contains two colons, the first one is time (echo time) in ms and the second one is the normalized integral of the spectrum recorded at the corresponding echo time. The dataset also contain a json file, AppleFitsResults.json with the full output of the data inversion results using a regularized NNLS. The fitting process and parameters are detailed in the related research article. The last file is the 3D plot representing the distribution of the relaxation time for each temperature.

The dataset containing the morphological HR MRI and the relaxation MRI can be found at datainrae repository (https://doi.org/10.15454/MMBJFF). It consists in two sets of not processed resonance magnetic images (MRI) and one set of processed images. All images are NifTI files. The Matlab code used to convert images from Bruker to NifTI files is also provided (BrukerToNifti_PomCuite.m).

The first set consists in the not processed MSME images used to calculate de T2 relaxation maps in the associated paper. There are 270 images corresponding to: 5 temperatures (raw, 45, 50, 53 and 60 °C) × 9 Echo Times (6.5, 8.5, 10.5, 15, 20, 40, 70, 100 and 200 ms) x 6 repetitions. The image name nomenclature is the following: T2_*temperature_scannumber_EchoTime*ms.nii. These MSME images are 2D images, matrice size is 32 × 32 pixels. Spatial resolution is 1 × 1 mm² and slice thickness is 1 mm. On all images, the left sample is a reference sample cooked 18 min at 70 °C for a complete thermal denaturation and the right sample is the sample cooked at the specified temperature (raw, 7 min at 45 °C, 10 min at 50 °C, 10 min at 53 °C, 14 min at 60 °C).

The second set consists in not processed high-resolution (HR) MSME images. Four of them (scan numbers 344, 406, 182 and 268) have been used as illustration of air pores disappearance with heating in the associated paper. There are 12 HR images corresponding to: 4 temperatures (raw, 50, 53 and 60 °C) × 3 repetitions. The HR image name nomenclature is the following: HR_*temperature_scannumber*.nii. These HR MSME images are 2D images, matrice size is 160 × 300 pixels. Spatial resolution is 0.1 × 0.1 mm² and slice thickness is 0.5 mm. On all images, the left sample is a reference sample cooked 18 min at 70 °C for a complete thermal denaturation and the right sample is the sample cooked at the specified temperature (raw, 10 min at 50 °C, 10 min at 53 °C, 14 min at 60 °C).

The third set consists in constructed T2 maps obtained from the first set. There are 30 images (T2 maps) corresponding to 5 temperatures (raw, 45, 50, 53 and 60 °C) × 6 repetitions. The image name nomenclature is the following: t2 *Psamplecode_temperature*.nii. These images are 2D images; matrice size is 32 × 32 pixels. Spatial resolution is 1 × 1 mm² and slice thickness is 1 mm. The signal in each pixel is the t2 value (transversal relaxation) in ms. On all images, the left sample is a reference sample cooked 18 min at 70 °C for a complete thermal denaturation

and the right sample is the sample cooked at the specified temperature (raw, 7 min at 45 °C, 10 min at 50 °C, 10 min at 53 °C, 14 min at 60 °C).

The data corresponding to the texture analysis of apple sticks can be found in the "Firmness Data" dataset at datainrae repository (https://doi.org/10.15454/HHAFZF). It contains all the raw data points extracted from the puncture tests curves acquired by software Nexygen proprietary software of the Lloyd Ta-plus texturometer. These raw data are all stored in a table (CSV file) named *Puncture_raw.tab*, composed of 12 columns and 204,338 rows. The raw data acquisition details and R code statistical treatment to reduce and average firmness values are combined in the R Markdown file *Puncture_Data.Rmd*, along with the compiled PDF file *Puncture_Data.pdf*.

2. Experimental Design, Materials and Methods

2.1. Plant material

Golden Delicious apples (*Malus domestica* Borkh. var. Golden Delicious) were purchased at a local store (Auchan, Avignon, France) at consumable ripeness. They were stored under normal atmosphere in a cold chamber at +4 °C for less than 8 days. For samples to reach room temperature, they were taken out of approximately 4 h before the experiments. For each cooking condition, apples were cut into 12 × 8 × 30 mm^3 sticks, the longer side being cut along the radial axis of the apple. Apart from those kept raw, sticks were vacuum-sealed in food-grade plastic bags (PE-LD 30 μm , RAJA SA, Tremblay-en-France, France). For the NMR measurements, six apple sticks were used for each condition, each one from a different apple. For the firmness analysis, one to four sticks were cut from both sides of an apple, depending on the apple size.

2.2. Thermal treatments

Each thermal treatment were performed by placing three apple sticks in a water bath (Lauda RML6, Königshofen, Germany for the NMR measurements and Julabo ED-19, Seelbach, Germany for the firmness measurements). A preliminary study consisting in placing thermocouples at the center of two apple sticks per cooking temperature, was performed to estimate the time required to reach each cooking temperature at the heart of the samples, giving: 7 min at 45 °C, 10 min at 50 °C, 10 min at 53 °C, 14 min at 60 °C and as a reference for a complete thermal denaturation, 18 min at 70 °C. These times were used for all subsequent experiments at the corresponding temperatures. After thermal treatment, samples were immediately placed in melting ice to cool for 8–15 min. Samples were then maintained at room temperature for 24 h before measurement. Such rest was applied to prevent an unwanted evolution of the water status inside the apple sticks, observed in a preliminary study for at least 6 h following the treatment and cooling steps.

2.3. Firmness measurements

The firmness of the samples was estimated as usually done to determine fruit flesh firmness, *i.e.* by measuring the mean load obtained from puncture tests [1]. One to four sticks were cut from one of the 21 apples used in the firmness experiment. Each sample was punctured on two faces: one along the radial axis of the fruit and the other perpendicular to the radial axis, with two repetitions for each face. The tests were performed using a Ta-Plus texturometer (Lloyd Instruments Ltd., Bognor Regis, UK) equipped with a 50 N load cell and a punch 2 mm in diameter and 17 mm in length, at a penetration rate of 100 mm min^{-1}. Puncture stopped once a 70% strain was reached. Texturometer control and data acquisition were handled with NEXYGEN software (version 4.5, Lloyd Instruments). The load-displacement curves are exported

as CSV files by manually choosing the number of data points, set to 1000 points in the plateau region. Data were then treated with RStudio (version 1.2.5033, R language version 3.6.0) as detailed in the *Puncture_Data.Rmd* and *Puncture_Data.pdf* files (https://doi.org/10.15454/HHAFZF).

After data averaging (as described in the aforementioned documentation), statistical analysis of the data was performed by analysis of variance (ANOVA, α=0.05) on the firmness as a function of the temperature, followed by a post-hoc Tukey test in order to rank the statistically homogeneous groups.

2.4. NMR measurements

All data for *NMR relaxometry* were acquired on a 9.4 T Bruker Ascend 400WB instrument (Bruker, Ettlingen, Germany). This instrument is equipped with a microimaging gradient system and a 32 mm diameter birdcage radiofrequency coil is used for both excitation and signal reception, at 20 °C.

2.5. MRI T2 measurement

Each of the 30 samples (5 temperatures: raw, 45 °C, 50 °C, 53 °C and 60 °C × 6 repetitions) was placed in a 25 mm NMR tube with a reference stick (an apple stick cooked for 18 min at 70 °C).

Nine single spin echo images were acquired in a longitudinal plan intercepting both the reference and the cooked sample, at nine different echo times (6.5, 8.5, 10.5, 15, 20, 40, 70, 100 and 200 ms). The relaxation time is 3000 ms, and the total acquisition duration is 32 min. These 270 MSME images (30 samples x 9 echo times) have been acquired in the purpose to built the T2 maps presented below. The image name nomenclature is the following:

T2_temperature_scannumber_EchoTimems.nii. These MSME images are 2D images, matrice size is 32 × 32 pixels. Spatial resolution is 1 × 1 mm² and slice thickness is 1 mm.

These previous 270 MSME images have been acquired in the purpose to build the T2 maps by fitting the nine echo magnitudes voxelwise, assuming mono-exponential decrease. These raw MSME images are provided in https://doi.org/10.15454/MMBJFF.

The 30 calculated T2 maps correspond to 5 temperatures (raw, 45, 50, 53 and 60 °C) × 6 repetitions. The image name nomenclature is the following: t2 *Psamplecode_temperature*.nii. These images are 2D images; matrice size is 32 × 32 pixels. Spatial resolution is 1 × 1 mm^2 and slice thickness is 1 mm. The signal in each pixel is the t2 value (transversal relaxation) in ms. Fig. 1 gives an example of such T2 maps.

The purpose of this experimental study was to indirectly determine the cell membrane thermal fusion by the T2 measurement. Inter individual variability is taken into account thanks to 6 repetitions on 6 different sticks coming from 6 different apples. On each T2 images (Fig. 1), almost 100 pixels correspond to the T2 in the cooked stick and report on the intra sample spatial variability. Mixed inter and intra sample variability is significantly lower (T.test p = 0.01 between 45 °C and 60 °C T2 (almost 600) values) than the strength of the effect (T2 variation with heating) as demonstrated on the Fig. 3 of the related research article (https://doi.org/10.1016/j.jfoodeng.2020.110413).

Morphological high resolution MSME MRIs were acquired in the same longitudinal plan. We used a multi-slice mono-echo image with an echo time TE=5 ms and a repetition time TR=3000 ms. For each high resolution image the total acquisition duration is 32 min. The 12 HR images presented here correspond to: 4 temperatures (raw, 50, 53 and 60 °C) × 3 repetitions. The HR image name nomenclature is the following: HR_temperature_scannumber.nii. These HR MSME images are 2D images, matrix size is 160 × 300 pixels. Spatial resolution is 0.1 × 0.1 mm² and slice thickness is 0.5 mm. The whole HR images set is provided in https://doi.org/10.15454/MMBJFF.

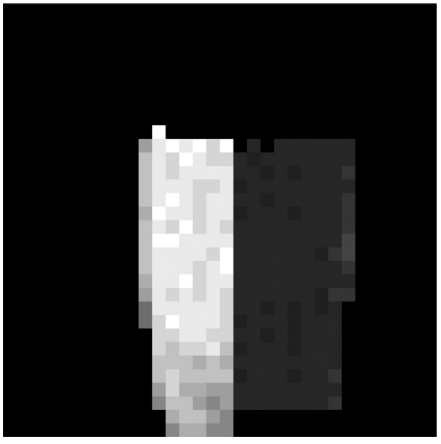

Fig. 1. T2 map for a raw (right) and a reference sample cooked 18 min at 70 °C for a complete thermal denaturation (left). This image (t2 P01_raw.nii) and the whole T2 maps set are available in https://doi.org/10.15454/MMBJFF.

All spin echo and morphological images were converted from Bruker to Nifti format with a Matlab code (BrukerToNifti_PomCuite.m provided in https://doi.org/10.15454/MMBJFF).

Spectroscopic T2 measurements were performed using the Carr- Purcell-Meiboom-Gill (CPMG) pulse sequence, 90x°-τ-[180y°- τ-(*echo*)]n, with an echo time τ of 500 μs and an 90° pulse of 40 μs. The repetition time was set to 2 s and $n = 256$ echoes spectra were recorded to describe the transversal echo decay curve for a total acquisition time of 45 min. Six different samples were analyzed for each time/temperature treatment, except for that of the reference cooked at 70 °C.

Each 2D dataset is Fourier-transformed and the integral of each spectrum is then taken to describe the transversal relaxation decay curve (PX_Y_T2.txt files described above). Each transversal decay is then fitted to a sum of exponentials using regularized NNLS algorithm, to provide a distribution of T2 relaxation times [2]. The full output of the analysis is provided in the Apple-Fitresults.json file in the dataset https://doi.org/10.15454/AHLT8G.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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