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Fast determination of beef quality parameters with time-domain nuclear magnetic resonance spectroscopy and chemometrics

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ABSTRACT

The noteworthy of this study is to predict seven quality parameters for beef samples using time-domain nuclear magnetic resonance (TD-NMR) relaxometry data and multivariate models. Samples from 61 Bonsmara heifers were separated into five groups based on genetic (breeding composition) and feed system (grain and grass feed). Seven sample parameters were analyzed by reference methods; among them, three sensorial parameters, flavor, juiciness and tenderness and four physicochemical parameters, cooking loss, fat and moisture content and instrumental tenderness using Warner Bratzler shear force (WBSF). The raw beef samples of the same animals were analyzed by TD-NMR relaxometry using Carr-Purcell-Meiboom-Gill (CPMG) and Continuous Wave-Free Precession (CWFP) sequences. Regression models computed by partial least squares (PLS) chemometric technique using CPMG and CWFP data and the results of the classical analysis were constructed. The results allowed for the prediction of aforementioned seven properties. The predictive ability of the method was evaluated using the root mean square error (RMSE) for the calibration (RMSEC) and validation (RMSEP) data sets. The reference and predicted values showed no significant differences at a 95% confidence level.

1. Introduction

For most consumers, a sensory response reflects their first impression about the quality of foods [1,2]. In general, the flavor, juiciness, and tenderness are the main properties used to evaluate the quality of meat on the consumer's plate [3].

The sensory analysis of food is determined by trained assessors who award scores for different attributes. These scores have a specific point scale to describe the tested property [4]. However, the problem with this procedure is the inherent subjectivity in the results [5]. Additionally, these panel evaluations are time-consuming and expensive [6].

Several studies have shown that meat sensory attributes such as juiciness and tenderness depend on the water content and its distribution. Bertram et al. [7] showed the relationship between water mobility and its distribution using transverse relaxation time (T_2) measured by the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence (T_2 relaxometry) and the sensory attributes in pork slaughtered between the ages of 90 and 180 days. Time-domain nuclear magnetic resonance (TD-NMR) relaxometry showed three different water environments in the meat linked to (i) proteins, (ii) myofibrils, and (iii) the myofibrillar lattice. Each region has a distinct range of relaxation times based on its aforementioned water distribution [7–9]. The meat with the highest juiciness score was from the 90-day old pork, and it had a longer relaxation time from the extramyofibrilar water corresponding to more mobile water than that observed in the older pigs [7].

Properties such as cooking loss, fat and moisture content, and instrumental tenderness are the main physicochemical parameters commonly used to evaluate beef quality [10,11]. However, the drawback is that the content of fat and/or moisture is estimated by taking an average value for one carcass. In other words, the reported value does not belong to the product purchased by the consumer [12].

The practice of displaying the nutrients or chemical properties on the product's label could be improved by utilizing the results from analytical laboratory tests and other techniques [13]. The specific characteristics for each package should be displayed on the product's label to help the consumer's choice and should not be displayed as an average estimation [12].

Therefore, the goal of this study was to show the possibility of predicting sensory traits, such as beef flavor, juiciness, and tenderness, as well as the physicochemical parameters, such as

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cooking loss, fat and moisture content, and instrumental tenderness using Warner Bratzler shear force, and TD-NMR relaxometry, which depends on fat and water content and their distributions.

The relaxometry studies were performed using a standard CPMG sequence to measure the T_2 values and the sequence known as Continuous Wave-Free Precession (CWFP) to measure longitudinal relaxation time, T_1 and T_2, in a single and fast experiment [14]. In our study, the entire TD-NMR signals obtained from the sequences were investigated to develop models to predict these seven properties. The main objective was not to replace the human opinion but to speed up the sensory tests and help the manufacturer to improve quality control.

2. Materials and methods

Animal Care and Use Committee approval was not obtained for this study because samples were taken from federally inspected slaughter facilities.

2.1. Procurement of samples

Sixty-one Bonsmara heifers were separated into five groups according to genetic (breeding composition) and feed system (grain and grass feed). After harvest and chilling, a portion of each left side strip loin (*Longissimus dorsi* muscle) was collected, vacuum packaged and sent to the Meat Lab at the State University of Campinas (UNICAMP, Campinas, São Paulo state, Brazil).

After 14 days of aging, for each portion, 4 steaks (2.5 cm thick) were prepared, overwrapped with parchment paper and frozen (−20 °C) before being used for sensory analysis, shear force and proximate analysis at Meat Laboratory at UNICAMP (Campinas, São Paulo state, Brazil) and, for TD-NMR tests at Embrapa —Instrumentation (São Carlos, São Paulo state, Brazil). For cooking and TD-NMR measurements, each steak was first trimmed of any external fat and connective tissue.

2.2. Sensory analysis

Steaks were thawed (4 °C for 24 h) and cooked in a conventional electrical oven—FEC (Imequi, series 8–4000 W, São Paulo, Brazil) equipped with upper and lower electrical resistances. The oven was pre-heated with the thermostat adjusted to 170 °C, and the steaks' internal temperatures were individually monitored.

Each steak was placed on a metal rack over an aluminum tray and was turned over after reaching an internal temperature of 40 °C. After this point, only the upper resistance was left on. Steaks were removed from the oven when they reached an internal temperature of 71 °C.

Grilled steaks were immediately cut into 1 cm cubes, which were placed in glass flasks with metal lids. For sampling, cubes with apparently no internal connective tissue were used.

A yogurt maker with the thermostat adjusted to 40 °C was used to keep the samples warm until evaluation, which was conducted in individual light- and temperature-controlled booths [15].

A trained sensory panel of eight members evaluated the tenderness, juiciness and flavor intensity of the samples on an 8-point scale (8=extremely tender, juicy and intense and 1=extremely tough, dry and bland) [16]. A total of 444 evaluations were performed, and they were reported by means of individual scores.

2.3. Cooking loss

Before cooking, steaks were trimmed to remove external fat and the thawed weight was recorded. Once steaks exited the oven, the cooked weight was recorded immediately. Cooking loss (%) was calculated using the following formula:

$$\text{Cookingloss}(\%) = \left[\frac{(\text{thawedweight(g)} - \text{cookedweight(g)})}{\text{thawedweight(g)}}\right] \times 100$$

2.4. Proximate analysis

Steaks were trimmed of fat and connective tissue, ground and oven dried to a constant weight (12 h). The moisture content was determined by weight difference [17]. The extraction of intramuscular lipids was performed using the Bligh and Dyer method [18], which is a recommended method for determining total lipid content in biological tissues [19].

2.5. Warner Bratzler shear force (WBSF)

After cooking, following the same procedure as performed for the sensory analysis, the steaks were placed on trays, covered with plastic film, and stored overnight at 4 °C for WBSF analysis.

In the next day, the cores from the lateral, middle, and medial portions (for a total of 6 cores, 1.3 cm in diameter) of each steak were removed parallel to the longitudinal orientation of the muscle fibers. The cores were sheared using a texture meter TA-XT 2i (Texture Technologies Corp./Stable Micro Systems, Godalming, Surrey, UK) equipped with a 1 mm thick Warner–Bratzler blade, and the peak shear force was recorded, and the average was determined. The peak load (kg) for all 6 cores was averaged, and the mean peak load (kg) was analyzed for each sample [16].

2.6. Time-domain nuclear magnetic resonance measurements

After thawing, each raw sample was separated into three cylindrical slices using a cylinder cutter with a diameter of 2 cm. For measurements, a benchtop SLK 100 TD-NMR spectrometer (Spinlock Magnetic Resonance Solution, Cordoba, Argentina) equipped with a 0.23 T permanent magnet (8.9 MHz for ^{1}H) and a 13 × 30 mm probe head was applied to collect CPMG and CWFP decay signals. The CPMG sequence was executed using $\pi/2$ and π pulses of 6.28 and 12.56 μs, respectively, and echo times of τ=300 μs with a total of 1500 echoes. The dead time was approximately 50 μs. The CWFP [14] sequence also used $\pi/2$ and π pulses of 6.28 and 10.6 μs, echo times of τ=141.56 μs and 1501 echoes. The frequency offset was 5 KHz. Each signal for both sequences was the result of an average of 4 scans. The room temperature was held constant at 23 °C.

2.7. Data set evaluations

For the construction of PLS multivariate regression models, two separate data matrices were organized into 61 lines, which correspond to the samples, and 1500 and 1501 columns for variables (time) of CPMG and, of CWFP, respectively. TD-NMR signals are related to the independent variables (**X** matrix). The flavor, juiciness, tenderness, cooking loss, fat content (total fat), moisture content and instrumental tenderness (Warner Bratzler shear force) were the dependent variables (**Y** matrix) used to compute the regression models. In the calibration data set, 49 samples were included, and the validation data (randomly chosen) set was comprised of 12 samples. The PLS chemometric technique is available on the Pirouette 4.0 rev. 2 software (Infometrix, Bothell, Washington, USA). An electronic spreadsheet