Preliminary study of a prototype benchtop NMR instrument as a device for shear force and drip loss measurement in *post rigor* meat

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Abstract

Nuclear magnetic resonance (NMR) studies have contributed successfully to the basic understanding of the physical and chemical water properties within meat. However, to utilise the full potential of NMR to measure meat quality attributes – drip loss and shear force, it should be implemented as an on-line rapid, non-invasive and non-destructive measurement technique. The aim of this experiment was to examine NMR data from a prototype NMR benchtop instrument (Magritek, NZ) with shear force and drip loss in lamb loins. Electrical stimulation and wrapping was used to create a sample set of lamb loins (n = 40) that varied in meat tenderness and drip loss. Shear force and drip loss measured day 1 to 4 post slaughter were compared to NMR data. Relaxation data changed significantly with ageing when the same sample was monitored day 1 to day 4 post slaughter. The T_{21} time constant decreased, the T_{21} population increased and the T_{22} population decreased over the ageing period. The overall correlation between shear force and NMR relaxation measurements was 0.62 (explaining 69% of the variation). The prediction for drip loss gave a lower R^2 value opf 0.42 (explaining 50% of the variation).

Introduction

Nuclear Magnetic Resonance (NMR) has been extensively used in meat science allowing the evaluation of water population and mobility within muscle microstructure (Bertram and Andersen 2004). The NMR T_2 relaxation of the water in muscle and meat can be separated into the relaxation decay of two or three exponential populations which represents two to three major water compartments in meat. The major population, T_{21}, is characterized by a fast time constant of 30-50 ms and contributes 80-95% of the relaxation of T_2. The second and slower population, T_{22}, has a time constant of 100-250 ms and accounts for 5-15% (Bertram and Andersen 2004). The T_{21} time constant corresponds to water located within highly organized protein structures, e.g. water in protein structures and between the myofibrillar protein structure actin and myosin filament (intramyofibrillar), while the T_{22} time constant corresponds to water located outside the myofibrillar network (extra-myofibrillar). Within meat, any change in the T_{21} time constant implies alterations in the structural organisation of myofibrillar water, whereas changes in the T_{22} time constant represents the *post mortem* reorganization of water closely associated with changes in membrane properties (Bertram *et al.* 2002). In addition, K_{21} and K_{22} represent the proportion of water represented by either the T_{21} or T_{22} time constants. An increased proportion reflects an increase in the size of the population or number of protons (essentially water).

The aim of this study is to investigate the potential for a prototype NMR benchtop instrument developed by NZ based NMR company Magritek to predict meat tenderness and drip loss through advanced chemometrics modelling.

Materials and methods

To test the Halbach NMR instrument a sample set of lamb *M. Longissimus dorsi* (LD) (total of 40 LD from 20 lambs) with variation in tenderness was created through electrical stimulation (stimulation (SS)/no stimulation (US)), wrapping (WW/UW) and ageing time (1 to 4 days post slaughter) using a factorial design. Within 20 mins of slaughter, the LDs were stimulated and/or tightly wrapped in four layers of cling film and placed in a waterbath at 35°C. When the LDs reached a pH of 5.6, the two LDs per animal were cut in half and allocated as sample A, B, C and D. Four sub-samples of approximately 1 x 1 x 4 cm^3 were excised from each sample. Two of the sub-samples were cut in parallel (sub-sample 1 and 2) and two were cut perpendicular to fibre direction (sub-sample 3 and 4). Each sub-sample was measured in replicate = 8 readings per loin sample. The same sub-samples from sample A were measured every day (1 to 4 days post slaughter) using the same sub-samples every day. The four sub-samples from B, C and D samples were measured only on days 2, 3 and 4, respectively. Samples were stored and measured at room temperature

from day 1 to day 4. LF-NMR measurements were carried out in a probe with magnetic field strength of 0.29 T, with a corresponding average resonant frequency for protons of 11.80 MHz using the Carr–Purcell–Meiboom–Gill (CPMG) sequence. This used an echo time (i.e. the time between 90 and 180° pulse) of 200 μs; and an RF pulse of 15 μs. Data from 512 echoes were acquired using 4 scan repetitions. The repetition time between two succeeding scans was 1.5 s, which allowed the longitudinal magnitisation to return to equilibrium. A duplicate vegetable oil measurement was taken prior to the start and regularly throughout the trial as a control. Shear force and drip loss was measured on all loin samples using a MIRINZ tenderometer and the press drip method (Rosenvold *et al.* 2008). The chemometrics modelling used to estimate the relationship between shear force or drip and NMR parameters was based in generalized additive models 'GAM' (Reis *et al.* 2007).

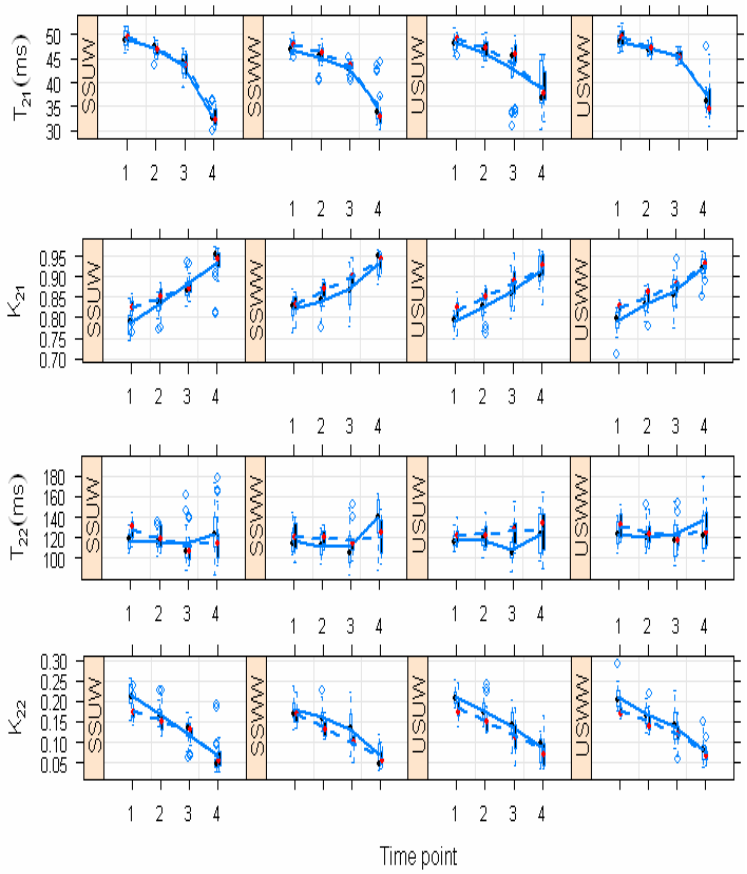

Figure 1. T_{21} time constants and population for sample A measured day 1 to day 4 post slaughter. Samples were either electrically stimulated and wrapped (USWW), electrically stimulated and unwrapped (SSUW) – or non stimulatated and unwrapped (USWW). Open bars corresponds to samples cut parallel to the fibre direction and full bars to samples cut perpendicular to the fibre direction. The relaxation time for T_{22} are in ms (10 seconds) and the populationsand K_{21} and K_{22} are a presentage of 100 %.

Results

The shear force decreased from day 1 to day 4: day 1 average and standard deviation 9.8 kgF ± 3.5, day 2 was 7.7 kgF ± 4.1, day 3 6.8 kgF ± 3.0 and day 4 6.0 kgF ± 3.1. Drip loss at day 1: 20.7 % ± 0.9, day 2: 20.6 % ± 0.9, day 3 % 20.3 ± 1.5 and day 4 21.0 % ± 1.2, and didn't change significantly over time. The NMR results showed a significant decrease in the fast T_{21} time constant (30-50 ms), T_{22} time constant remaining constant (100-140 ms), K_{21} significantly increased and K_{22} significantly decreased (Figure 1) from day 1 to day 4. The overall correlation between shear force and NMR relaxation measurements was 0.62 explaining 69% of the variation (range 0.40-0.61) (Figure 2). The prediction for drip loss gave a lower value of R^2 (0.42 explaining 50% of the variation) (not shown).

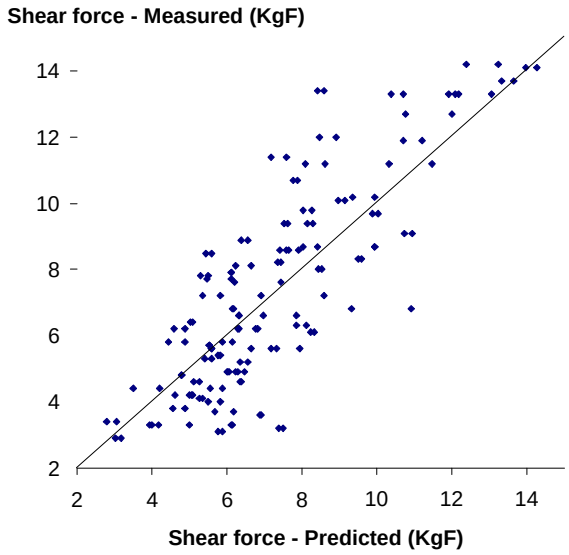

Figure 2. Predictions of shear force by GAM model for samples measured by 1 to 4 post slaughter (R2:0.62. Explainded variance: 69 %).

Discussion

Relaxation measurements were significantly affected by ageing. Ageing of meat is defined by the process of proteolysis. The proteolytic degradation of cytoskeletal proteins subsequently enables swelling of the myofibrils and an uptake of water in the intramyofibrillar space. An increased K_{21} over the ageing period indicates an increase in the number of protons in the intramyofibrillar space over the ageing period. A decrease in the T_{21} time constant is occurring because there is an increase in the number of relaxation sinks within the myofibrils, i.e. protons. In addition there is a decrease in K_{22} indicating a decrease in water in the extramyofibrillar space. Combined these results indicate either an increasing uptake of water into the intramyofibrillar space or alternatively/in addition to the release of water from bound proteins during the proteolysis process (Pearce *et al.* 2007).

Conclusions

The overall correlation between shear force and NMR relaxation measurements was 0.62 explaining 69% of the variation. The correlation may be improved if the effect of within muscle variability is reduced by using the same sample for both measurements. The correlation between shear force and the NMR relaxation parameters is a positive result, providing further evidence to support ongoing research using NMR to predict tenderness online in *post rigor* meat. The prediction for drip loss gave a lower value of R^2 (0.42 explaining 50% of the variation).

This research indicates that an increase in the concentration of water with a fast relaxation time (K_{21}) and a decreasing T_{21} time constant is associated with more tender meat. The opposite result has been observed in pork. This result needs to be further confirmed with red meats.

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