Meat Science 115 (2016) 27–33

Contents lists available at ScienceDirect

Meat Science

journal homepage: www.elsevier.com/locate/meatsci

Assessment of tenderness of aged bovine *gluteus medius* muscles using Raman spectroscopy

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ARTICLE INFO

Article history:
Received 13 February 2015
Received in revised form 22 December 2015
Accepted 28 December 2015
Available online 30 December 2015

Keywords:
Meat quality
Shear force
Handheld Raman device
Non-invasive

ABSTRACT

A portable 671 nm Raman system was evaluated as a rapid and non-destructive device for the assessment of beef tenderness using 175 *gluteus medius* muscles (99 for calibration, 76 for validation) aged at −1 °C and 7 °C for fourteen days. Raman and shear force (SF) measurements were performed with the aged beef. The samples stored at −1 °C showed on average only slightly increased SF values. The correlation of Raman spectra with SF using partial least squares regression yielded cross-validated predictions of SF for both storage temperatures with coefficients of determination $R^2_{cv} = 0.33–0.79$. Validation with independent samples resulted in predictions with $R^2_{val} = 0.33$. Using thresholds between 30 and 49 N, tough and tender samples could be discriminated with partial least squares discriminant analysis with 70–88% and 59–80% accuracy during cross-validation and validation, respectively. These results demonstrate the principle feasibility to predict the SF and thus toughness of raw, aged *gluteus* beef cuts with a portable Raman device showing potential for grading beef cuts.

1. Introduction

Flavor, tenderness and juiciness are the most important quality traits for the overall liking of beef (Hunt et al., 2014). Studies show that customers are willing to pay a higher price for beef cuts with guaranteed tenderness (Miller, Carr, Ramsey, Crockett, & Hoover, 2001) and, accordingly, that tenderness is ranked higher than price when it comes to the purchasing decision (Reicks et al., 2011). Consequently, the management of tenderness is an issue especially for the red meat production due to the storage time required for aging. On average, beef is aged for 28 days before retail sale in the U.S. but storage times up to 67 days are used in practice (Guelker et al., 2013). During this time, the tenderness of meat is changed by proteolytic degradation of proteins and by protein oxidation (Huff Lonergan, Zhang, & Lonergan, 2010).

While tenderness can only be directly assessed by human panelists this procedure is expensive and yields subjective results. Therefore, mechanical methods are used as objective measures for tenderness but again these methods are time-consuming, destructive and laborious. The Warner-Bratzler shear force (WBSF) test and the slice shear force test are the most often applied mechanical methods. A Warner-Bratzler apparatus is used to measure the force which is required to cut the cooked sample with a V-shaped blade in relation to the distance the blade has cut into the meat. Conventionally, the maximum force recorded during the cutting process is reported. The results of this WBSF measurement usually show discrepancies with tenderness values estimated by a trained sensory panel with correlation coefficients between −0.2 and −0.9 (Destefanis, Brugiapaglia, Barge, & Dal Molin, 2008; Peachey, Purchas, & Duizer, 2002; Shackelford, Wheeler, & Koohmaraie, 1999). This discrepancy may be explained by the finding that tenderness is mainly evaluated subjectively (Wezemael, Smet, Ueland, & Verbeke, 2014). Despite its disadvantages, the WBSF is still an accepted objective measurement for meat tenderness (Lorenzen et al., 2010).

Several publications were addressing a replacement for the WBSF method (Damez & Clerjon, 2008; Prieto, Roehe, Lavín, Batten, & Andrés, 2009; Xiong, Sun, Zeng, & Xie, 2014). Table 1 shows an overview of – mostly spectroscopic – studies which used beef or lamb cuts and at least 40 samples. On the one hand, good results are reported in some studies using near-infrared (NIR) reflectance spectroscopy with R^2's during calibration ranging from 0.5 to 0.8. On the other hand, only moderate correlations or no useful predictions are reported in other studies. A change from reflectance to transmittance spectra does not significantly improve the correlations (Leroy et al., 2003). For visible-NIR hyperspectral imaging (496–1036 nm) correlations with $R^2 = 0.45$ are shown to predict cooked beef tenderness (Cluff et al., 2008).

As NIR spectra revealed potential for the identification of tender beef according to WBSF (Shackelford, Wheeler, & Koohmaraie, 2004; Shackelford, Wheeler, & Koohmaraie, 2005), a portable near-infrared reflectance (NIR-R) instrument was developed for beef carcasses (Rust

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http://dx.doi.org/10.1016/j.meatsci.2015.12.020
0309-1740/

Table 1
Overview of non-invasive shear force studies with beef and lamb. Methods: NIR = near-infrared, R = reflectance, T = transmission, HI = hyperspectral imaging, EI = electrical impedance, EC = electrical conductivity; muscles: SM = *M. semimembranosus*, LD = *M. longissimus dorsi*, LTL = *M. longissimus thoracis et lumborum*, PM = *M. psoas major*; samples = number of samples, Prep. = with sample preparation, R^2 = coefficient of determination, RMSEC = root mean squared error of calibration and cv = cross-validated.

Method	Muscle	Samples	Prep.	R^2	RMSEC	Reference
NIR-R	LD	80 + 39	No	0.67	12.0	Park, Chen, Hruschka, Shackelford, and Koohmaraie (1998)
NIR-R	LD	127	No	0.47–0.55	14.9–18.1^{cv}	Rødbotten, Nilsen, and Hildrum (2000)
NIR-R	LD	48	No	0.52–0.83		Rødbotten, Mevik, and Hildrum (2001)
NIR-R	LD, SM	75	No	0.26 (LD)		Venel, Mullen, Downey, and Troy (2001)
NIR-R	LD	172–174	No	0.12–0.25^{cv}	7.7–11.2^{cv}	Leroy et al. (2003)
NIR-R	LD	146 + 146	No	0.22–0.38		Shackelford et al. (2005)
NIR-T	LD	167–170	No	0.15–0.41^{cv}	8.0–9.6^{cv}	Leroy et al. (2003)
NIR-HI	LL, PM	44 + 17	Yes	0.45		Cluff et al. (2008)
EI + EC	LD	47	No	0.42–0.46		Byrne et al. (2000)
Raman	Silverside	52	Yes	0.75	6.3^{cv}	Beattie et al. (2004)
Raman	LTL	70 + 70	Yes	0.79–0.83	2.4–3.0	Schmidt et al. (2013)
Raman	SM	80	No	0.27^{cv}	11.5^{cv}	Fowler et al. (2014a)
Raman	LL	80	No	0.06^{cv}	13.6^{cv}	Fowler et al. (2014b)

et al., 2008). This and a similar instrument are shown to discriminate tender from tough beef ribeye (n = 1155) (Shackelford, King, Wheeler, & Koohmaraie, 2012a), strip loin (n = 467) (Shackelford, Wheeler, & Koohmaraie, 2012b) and *longissimus lumborum* (LL) samples (n = 768) (Rust et al., 2008),

Correlations of up to $R^2 = 0.46$ are reported for non-spectroscopic techniques such as electrical impedance and conductivity with WBSF of bovine *longissimus dorsi* (LD) muscles (n = 47) (Byrne, Troy, & Buckley, 2000).

Promising results are reported with Raman spectroscopy. This method gives insight into the molecular composition of a sample by means of vibrational spectroscopy. The scattered light can reveal biochemical and structural information. PLSR (partial least squares regression) correlations of Raman spectra of roasted beef silversides with shear force (SF) are promising with $R^2 = 0.75$ and root mean squared error of cross-validation RMSECV = 6.3 N (Beattie, Bell, Farmer, Moss, & Patterson, 2004). In a more recent study, a portable Raman scanner with potential for the application in processing plants is shown to measure shear force and cooking loss of frozen and thawed sheep muscle (Schmidt, Scheier, & Hopkins, 2013). The Raman spectra of 140 LT (*M. longissimus thoracis*) and LL samples from two different origins correlate well with the SF yielding $R^2 = 0.79$ and 0.83 for the two data sets separated according to origin. However, while freezing/thawing was required for the transport of the samples this is not reflecting the typical process flow of meat production. Therefore, subsequent work is evaluating the ability of Raman spectroscopy for predicting SF from Raman spectra of meat cuts that have never been frozen. A study with *semimembranosus* (SM) muscles, shows only a limited ability for the prediction of SF of aged lamb from Raman measurements performed on day 1 post mortem (p.m.) with cross-validated coefficient of determination $R^2 = 0.27$ whereas no correlation is found for the LL (Fowler, Schmidt, van de Ven, Wynn, & Hopkins, 2014a, 2014b).

While clear potential of Raman spectroscopy for non-destructive and rapid determination of SF has been shown for roasted beef and for frozen and thawed lamb, data on beef that has not been frozen or grilled is lacking. Therefore, the aim of this study is to evaluate whether the Raman spectra measured on wet aged, raw beef samples can predict the measured WBSF.[1]

2. Materials and methods

2.1. Meat samples

In total, samples from 175 young bulls were collected from commercial abattoirs over two periods of 5 months each (one for calibration and one for validation, both separated by two years). Ten samples were measured on average per day every two weeks which required ten sampling days for calibration and eight for validation. The animals had an age of 18–24 months, a slaughter weight of 360–400 kg and they were from different origin (90% Simmentals and 10% mixed origin, mostly brown cattle). Three to four days p.m., the hindquarters were transported to the processing plant where they were cut and boned until day five p.m. The steak meat was vacuum-packed separately for further transport and aging. Of these, 89 (50 and 39 for calibration and validation, respectively) were aged at −1 °C and 86 (49 and 37) at 6–7 °C for a period of further 14 days. Total aging time was 19 days. Left and right side were randomized to avoid a position bias and with a view to the applicability of the method.

2.2. Measurement of Raman spectra

The vacuum packages were removed prior to the Raman measurements. The aged *gluteus medius* (GM) muscles were excised and they were allowed to warm to ambient temperature. A fresh cut was made along the fiber direction to avoid bias of the spectra due to the development of different microbial flora on the samples stored at −1 and 7 °C. Without additional time for blooming, at least 15 Raman spectra were recorded at different positions on the fresh cut perpendicular to the fiber direction with the 671 nm Raman system described earlier (Schmidt, Sowoidnich, & Kronfeldt, 2010; Scheier, Bauer & Schmidt, 2014). Briefly, the system consisted of a Raman sensor head with integrated laser, collection optics and Raman filter (Schmidt et al., 2010) which was connected with an optical fiber to a miniature spectrograph (HORIBA Jobin-Yvon, Longjumeau, France). The optical resolution of this system was 8 cm^{-1} and Raman spectra were recorded in the range from 340 to 2100 cm^{-1}. The system was controlled by a self-written LabVIEW program (National Instruments, Austin, TX, USA). The integration time was 3 s and 5 accumulations were taken per spot (15 s total integration time per spot). The laser power was adjusted to 100 mW. Accidental measurements of fat were detected with an automated routine and saved separately (Scheier, Bauer & Schmidt, 2014). In case of fat detection, the measurement was repeated at a new position. The Raman system was used with the same wavenumber calibration for calibration and validation measurements. The laser power, however, was reduced to 50 mW for the validation. To compensate for this, the integration time was set to 5 s and 6 accumulations per spot were taken to keep a constant energy of excitation (1.5 J).

2.3. Shear force measurements

The shear force was determined with the same sample material that has been used for the Raman scans. The method was modified from (Hopkins, Toohey, Warner, Kerr, & van de Ven, 2010). The GM was cut

[1] Preliminary results of this work were presented at the 59th ICoMST in Izmir (Bauer et al., 2013).