



Application of Raman spectroscopy and chemometric techniques to assess sensory characteristics of young dairy bull beef



Ming Zhao^a, Yingqun Nian^{b,c}, Paul Allen^b, Gerard Downey^a, Joseph P. Kerry^c,
Colm P. O'Donnell^{a,*}

^a School of Biosystems and Food Engineering, University College Dublin, Belfield Dublin 4, Ireland

^b Department of Food Quality and Sensory Science, Teagasc Food Research Centre, Ashtown Dublin 15, Ireland

^c School of Food and Nutritional Sciences, University College Cork, Cork, Ireland

ARTICLE INFO

Keywords:

Beef
Chemometrics
Eating quality
Raman spectroscopy
Sensory attributes

ABSTRACT

This work aims to develop a rapid analytical technique to predict beef sensory attributes using Raman spectroscopy (RS) and to investigate correlations between sensory attributes using chemometric analysis. Beef samples ($n = 72$) were obtained from young dairy bulls (Holstein-Friesian and Jersey \times Holstein-Friesian) slaughtered at 15 and 19 months old. Trained sensory panel evaluation and Raman spectral data acquisition were both carried out on the same *longissimus thoracis* muscles after ageing for 21 days. The best prediction results were obtained using a Raman frequency range of 1300–2800 cm^{-1} . Prediction performance of partial least squares regression (PLSR) models developed using all samples were moderate to high for all sensory attributes ($R^2\text{CV}$ values of 0.50–0.84 and RMSECV values of 1.31–9.07) and were particularly high for desirable flavour attributes ($R^2\text{CV}$ s of 0.80–0.84, RMSECVs of 4.21–4.65). For PLSR models developed on subsets of beef samples i.e. beef of an identical age or breed type, significant improvements on prediction performances were achieved for overall sensory attributes ($R^2\text{CV}$ s of 0.63–0.89 and RMSECVs of 0.38–6.88 for each breed type; $R^2\text{CV}$ s of 0.52–0.89 and RMSECVs of 0.96–6.36 for each age group). Chemometric analysis revealed strong correlations between sensory attributes. Raman spectroscopy combined with chemometric analysis was demonstrated to have high potential as a rapid and non-destructive technique to predict the sensory quality traits of young dairy bull beef.

1. Introduction

Beef eating quality perception consists of both expected and experienced quality dimensions. Expected eating quality is positively related to perceived colour, marbling, brand and origin. Experienced eating quality is a combination of taste, texture and juiciness, which dominates consumer satisfaction and purchase intentions for beef (Banovic, Grunert, Barreira, & Fontes, 2009). Tenderness has been cited as the most important factor determining beef eating satisfaction (Savell et al., 1987). However, it has been shown that when tenderness reaches a consistent level, flavour becomes the next most important factor affecting beef palatability (Killinger, Calkins, Umberger, Feuz, & Eskridge, 2004). Most consumers would be willing to pay a premium for meat cuts with a superior tenderness combined with a juicy property, without off-flavours (Aaslyng et al., 2007; Miller, Carr, Ramsey, Crockett, & Hoover, 2001). It should be noted that these eating quality traits are non-visible and highly subjective. A number of direct and

indirect methodologies have been developed to assess consistent beef eating quality.

In the absence of reliable objective instrumental measurements, sensory panel evaluation has been used as the most direct or traditional method to determine beef eating quality. Sensory profiling is a method that is able to establish relationships between descriptive sensory and instrumental or consumer preference measurements (O'Sullivan & Kerry, 2009). However, the reliability of sensory analysis largely depends on the quality of the sensory training carried out prior to profiling and the actual food choice behaviour (Dijksterhuis & Byrne, 2005; O'Sullivan & Kerry, 2009). Moreover, sensory panel evaluation is costly and time-consuming.

Accordingly, rapid, robust, accurate and non-destructive techniques to predict beef sensory properties are required by the meat industry. The Meat Standards Australia (MSA) grading scheme was developed as an indirect tool, using a multiple regression approach with production and processing variables, to build a model to predict palatability of

* Corresponding author.

E-mail address: colm.odonnell@ucd.ie (C.P. O'Donnell).

individual muscles for different cooking techniques (Thompson, 2002). Computer vision technology and digital imaging systems have been shown to have high accuracy for the prediction of overall sensory acceptability ($R^2 = 0.95$; Jackman, Sun, & Allen, 2010), flavour ($R^2 = 0.84$), juiciness ($R^2 = 0.71$) and tenderness ($R^2 = 0.64$) of beef (Jackman, Sun, & Allen, 2009).

The use of spectroscopy techniques for prediction of sensory characteristics of raw beef has been investigated in recent studies. Near infrared (NIR) spectroscopy has been reported as a method to rapidly assess sensory properties of raw meat (Brøndum, Byrne, Bak, Bertelsen, & Engelsen, 2000; Liu et al., 2003; Ripoll, Albertí, Panea, Olleta, & Sañudo, 2008; Rodbotten, Nilsen, & Hildrum, 2000; Venel, Mullen, Downey, & Troy, 2001). Overall, low or moderate prediction accuracies ($R^2CV < 0.6$) were obtained probably due to C–H, O–H and N–H overtones of NIR which cannot provide spectral information on specific molecular functional groups, thereby limiting the accuracy of the biochemical profiling of meat (Wang, Lonergan, & Yu, 2012). Visible NIR hyperspectral imaging has been shown to have good potential to evaluate beef quality traits including pH, colour, marbling, water holding capacity and texture (ElMasry, Barbin, Sun, & Allen, 2012; ElMasry, Sun, & Allen, 2011; Wu et al., 2012). Kamruzzaman, ElMasry, Sun, and Allen (2013) reported prediction results (R^2CV of 0.69) for lamb sensory tenderness using NIR hyperspectral imaging, however use of NIR hyperspectral imaging for the prediction of beef sensory properties has not been widely reported. Raman spectroscopy (RS) is a non-invasive vibrational spectroscopic technique that has considerable advantages compared to other techniques as it is relatively insensitive to water unlike mid-infrared spectroscopy. In addition, RS can provide high resolution spectral information on chemical composition in situ (Wang et al., 2012). Preliminary studies have demonstrated the potential of RS (R^2CV s of 0.19–0.71) to predict sensory attributes of cooked beef rounds (Beattie, Bell, Farmer, Moss, & Patterson, 2004). Another RS study on intact samples reported a relatively lower coefficient of determination (R^2CV s of 0.17–0.44) for the prediction of sensory traits of raw beef loins (Fowler, Schmidt, & Hopkins, 2016). However, limited information is available on the prediction of raw beef sensory quality using Raman spectroscopy.

Beef palatability is significantly affected by on-farm production factors, such as animal breed, slaughter age, sex, feeding system, etc. (Frylinck, Strydom, Webb, & du Toit, 2013). Holstein-Friesian (HF) is the predominant Irish dairy breed. There is also a current interest in the Jersey breed as it has been shown to have good potential for cross-breeding under Irish conditions. Cattle age is also an important factor in determining meat tenderness and palatability (Schönfeldt & Strydom, 2011). The effects of breed and age on sensory quality of young dairy bull beef has been recently investigated (Nian, Allen, et al., 2017). However, sensory quality prediction for bull beef of specified breeds or ages has not been reported to date.

The aims of this study were to (1) establish correlations between sensory attributes of bull beef using chemometric approaches; (2) demonstrate the potential of RS to predict sensory traits of *longissimus thoracis* (LT) muscle of dairy bulls; (3) demonstrate the potential of RS ($1300\text{--}2800\text{ cm}^{-1}$) and chemometrics for the prediction of sensory attributes of a specified cattle age (e.g. 15 or 19-month) or breed (e.g. JEX (Jersey \times Holstein-Friesian) or HF).

2. Materials and methods

2.1. Source of materials

Young dairy bulls ($n = 72$) were slaughtered in 2012 and 2013 from two breeds (HF ($n = 46$) and JEX ($n = 26$)) at two slaughter ages (15 months old ($n = 34$) and 19 months old ($n = 38$)). Bulls were slaughtered in a commercial abattoir and the *longissimus thoracis* (LT) muscle was removed from the cube roll (ribs 6 to 10) on the left side of each carcass at 48 h post-mortem. After holding until 72 h post-mortem,

muscles were cut into individual slices (ca. 25 mm thick) and vacuum-packed. Samples were aged for 21 days at 4 °C and then stored at –20 °C before sensory analysis and Raman measurement.

2.2. Trained sensory panel evaluation

For sensory analysis, frozen steaks were thawed in a circulating water bath at 10 to 15 °C for approximately 45 min. Steaks were cooked on a double contact electric grill (Velox CG-3, Velox Grills, UK) set at 230 °C, to an internal temperature of 70 °C according to the AMSA Guidelines (AMSA, 1978). Temperature was monitored with a probe (Eurolec TH103TC, Technology House, Ireland) inserted into the centre of each steak for the duration of cooking. Steaks were grilled for 1 min on one side, then turned over for 1 min, and then turned twice more for 2 min followed by continuous turning each min until done. All external fat and major connective tissue was then trimmed from cooked samples prior to cutting into cubes about $20 \times 15 \times 25$ mm. These samples were wrapped in aluminum foil and held for about 3 min before serving to eight trained sensory panellists who were seated in individual booths with red fluorescent lighting. Each panel member was trained according to AMSA (1995) standards, and received six samples in randomised order (each panellist tasted the samples in a different order within each session) in two sets with three samples in each set and approximately 3-min intervals between each set. Panel members were provided with a salt-free cracker and water for cleansing the palate between samples.

Panellists were required to score each sample for 16 attributes. These attributes were defined and rated during different phases of eating. Roast beef aroma intensity (aroma) was evaluated before eating, and initial tenderness was the texture of the first bite. During further mastication, juiciness, cohesiveness, ease of disintegration, chewiness, fattiness, stringy, astringency and some flavour terms including roast beef flavour (beef flavour), metallic and rancid were evaluated. Residual roast beef flavour length (flavour length), residual metallic (res-metallic), residual fattiness (res-fattiness) and residual dryness (dryness) were the sensations left in the mouth 12 s after swallowing the sample; and thus they were described as residual or after effects. These sensory attributes were categorised into four different terms including 1) texture term with its relevant attributes, i.e. initial tenderness, ease of disintegration, cohesiveness, chewiness and stringy; 2) juiciness term with juiciness, astringency and dryness; 3) flavour term with aroma, beef flavour, metallic, rancid, flavour length and res-metallic; 4) fattiness term with fattiness and res-fattiness (Nian, Allen, et al., 2017). Each attribute was rated using 'Compusense® five' sensory evaluation software (Guelph, Ontario, Canada) on station computers using a 100 mm unstructured line scale with 0 mm being equivalent to no attribute intensity and 100 mm being equivalent to the highest intensity of the attribute (Corbin et al., 2015; Resconi, Campo, Font i Furnols, Montossi, & Sañudo, 2010). Descriptive statistics of the sensory data were summarised to define the range of each sensory attribute.

2.3. Statistical analysis of sensory data

Pearson correlations between sensory attributes were calculated by the CORR procedure using SAS Version 9.3 (SAS Institute Inc., Cary, NC, USA, 2002) with a significance level of $P < 0.05$.

Furthermore, in order to define correlations between sensory attributes and sample types, partial least squares regression models with multiple y-variables (PLS-2) were developed using sensory attribute results from 72 samples (X data) and biological sample type, i.e. HF and JEX; 15- and 19-month old (Y data). Dummy values of 0 and 1 were given to 19-month old JEX breed and 15-month old HF breed respectively to form a binomial metric of sample types. Leave-one-out full cross-validation was performed. Correlation loadings of X and Y were plotted out in 2-D form together with two of the Hotelling T^2 ellipses for determining outliers at 75% and 95% confidence limits, respectively.

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