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Predicting pork water-holding capacity with NIR spectroscopy in relation to different reference methods

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ABSTRACT

The objective of the study was to evaluate the ability of near infrared (NIR) spectroscopy to predict pork water-holding capacity (WHC) in relation to different methods of WHC determination. The study comprised 228 *longissimus dorsi* muscle samples (147 for calibration, 81 for external validation). Calibration models were developed using WinISI II on visible (λ = 400–1100 nm), NIR (λ = 1100–2500 nm) or the whole spectral range (λ = 400–2500 nm). Models' quality was assessed by means of the coefficient of determination of cross-validation (R^2_{cv}) or prediction (R^2_p) and the error of cross-validation (se_{cv}) or prediction (se_p). The R^2_{cv} ranged from 0.28 to 0.62, while se_{cv} was estimated between 1.0% and 1.4% for EZ drip loss (meat juice container procedure), 2.3% and 2.5% for cooking loss, 1.9% and 2.3% for centrifuge force and 0.9% and 1.0% for tray drip loss, and was similar to the repeatability (s_r) of the method (1.1%, 1.8%, 2.0%, 0.4%, respectively). The results of the external validation confirmed those obtained by cross-validation ($R^2_p = 0.22$—0.44; $se_p = 1.2$—1.3%, 2.2–2.4%, 2.1–2.3% and 1.1% for EZ drip loss, cooking loss, centrifuge force and tray drip loss, respectively). The best calibration results were obtained for EZ drip loss ($R^2_{cv} = 0.62,\ se_{cv} = 1.0\%$) and tray drip loss ($R^2_{cv} = 0.49,\ se_{cv} = 0.9\%$) on visible spectral range. NIR spectroscopy offers great advantages for the *on-line* application, however, its prediction ability is limited by the reference methods and this should be taken into account.

1. Introduction

Quality assurance and control are among the main tasks in production and processing of all kinds of food. For fresh pork, its quality to a large extent relates to water-holding capacity (WHC) which is technologically and financially important for food-processing industry and also for consumers as an attribute when purchasing meat. Many diverse methods for the determination of WHC have been used such as drip loss, cooking/heating loss, centrifuge force method, thawing loss, processing loss, Napole yield, technological yield, *etc.* (Aaslyng et al., 2003; Allison et al., 2002; Bertram et al., 2001, 2003; Merour et al., 2007; Otto et al., 2004, 2006; Penny, 1975; Trout, 1988; Zhang et al., 1995). Moreover, within the same methodological approach there exist several modifications; *e.g.* drip loss which presents losses of water owing to the gravity could be performed as bag method (Honikel, 1998), meat juice container procedure (EZ drip loss; Christensen, 2003), tray drip loss (Allison et al., 2002; Lundström and Malmfors, 1985) or filter paper method (Kaufmann et al., 1986). The problem of WHC evaluating is that there is a variety of methods which differ in many factors (like sampling site, size and shape of meat samples, type and duration of treatments and physical principle of water release) and are often not well correlated to each other (Allison et al., 2002; Merour et al., 2007). Consequently, the results differ considerably among studies. Beside the methodological variability a critical point or a limitation of WHC determination is that methods are lengthy and destructive and thus unsuitable for *on-line* application. Difficulties in measuring and controlling WHC under industrial conditions caused exploring and introducing novel rapid analytical methods, among which near infrared (NIR) spectroscopy is particularly interesting. In contrast to conventional methods NIR spectroscopy offers fast and simple determination of many parameters; its downside is that it needs a calibration for every single purpose. Studies on this subject are quite numerous and show large variability of results in terms of prediction ability of NIR spectroscopy methods (for review see Prieto et al., 2009). According to Monin (1998) the limitation of NIR spectroscopy could be related to the precision of the reference methods, therefore the reference method must be carefully chosen depending on the particular needs in each case and a wide range of reference values reached to maximise the predictability of NIR spectroscopy (Prieto et al., 2009). In view of these aspects, it was the aim of the present study to compare prediction ability of NIR spectroscopy in relation to different WHC reference methods within the same study.

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2. Materials and methods

2.1. Animals

The experiment comprised samples of meat collected from 228 fattening pigs of similar age (6 months) slaughtered during spring (from March to May 2008) in six series. Animals used in the study were the progeny of Landrace × Large white dams and Pietrain × Duroc or Pietrain sires. Gilts and barrows were nearly equally represented within both crossbreeds and all series of slaughter. All pigs were reared on the same farm and slaughtered in one commercial abattoir according to their standard procedure.

2.2. Sampling procedure and WHC measurements

Samples of *longissimus dorsi* muscle were taken in the abattoir 1 day after the slaughter and stored at 4 °C until the next day, when WHC analysis was performed. In the laboratory approximately 15 cm long piece of *longissimus dorsi* muscle was taken between last rib and 3rd or 4th lumbar *vertebra* and split to six slices. From the caudal to cranial direction the slices were used for (Fig. 1): centrifuge force method (2.5 cm), cooking loss (2.5 cm), tray drip loss method (2 × 2.0 cm), NIR spectroscopy (2.0 cm) and EZ drip loss method (2.5 cm).

The WHC of meat was measured using four different procedures. Meat pieces were weighed prior to and after the treatment (centrifugation, cooking, *etc.*) and the loss of weight was expressed as a percentage of initial sample weight. EZ drip loss method was performed as described by Christensen (2003). Shortly, two cylindrical pieces with a diameter of 2.5 cm cut from the central area of 2.5 cm thick steak were weighted, placed in plastic sealable cups (Sarstedt AG & Co. meat extract collector) and stored at 4 °C for 24 h and reweighed. Centrifuge force method was performed as described by Allison et al. (2002). Two cylindrical pieces with a diameter of 2.5 cm cut from the central area of 2.5 cm thick steak were weighted, placed in a plastic centrifuge tubes and centrifuged 30 min with 8400 × *g* at 4 °C and reweighed. For cooking loss two cylindrical pieces with a diameter of 2.5 cm cut from the central area of 2.5 cm thick steak were weighted, placed in plastic tubes and cooked in a water bath at 85 °C for about 10 min resulting in a core temperature of 75 °C. Subsequently the meat samples were lightly dabbed and reweighed. Cooking loss was performed as described by Bertram et al. (2003) with a slight modification (sample size). Tray drip loss (called also retail display) was performed as described by Allison et al. (2002), with modification related to slice thickness (2.0 cm) and storage time (5 days). Samples were placed on plastic trays, covered with a moisture permeable plastic overwrap, and stored at 4 °C for 5 days. All WHC methods were carried out in duplicates. For further analysis both results were averaged.

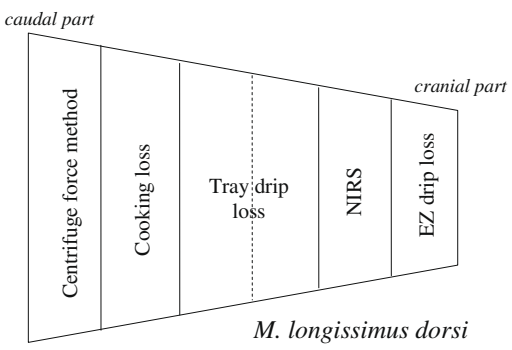

Fig. 1. Sampling for WHC and NIR measurements.

2.3. NIR spectroscopy measurements

A slice of *longissimus dorsi* muscle used for NIR analysis was cut at 2nd lumbar vertebra (Fig. 1). The meat samples were scanned intact 48 h *post-mortem* using the laboratory spectrophotometer NIR Systems model 6500 (Silver Springs, MD, USA). Two slices of each sample were separately placed into a quartz cup (47 mm × 57 mm) and covered with a paper disc. Meat scans were taken over the wavelength range from 400 to 2500 nm. Absorbance data were collected every 2 nm as log 1/*R*, where *R* represents reflectance. For subsequent analysis both spectra of the same sample were averaged.

2.4. Statistical analysis

The statistical analysis for meat quality traits was performed using SAS (SAS, 2002). Basic statistic parameters for WHC measurements were calculated using the UNIVARIATE procedure. Relationships among WHC methods were calculated using the CORR procedure on raw experimental data (phenotypic correlations) and after the adjustment for the effects in the model (residual correlations). To calculate residual correlation coefficients WHC measurements were adjusted using the GLM procedure for the effects of sex, crossbreed, slaughter series within crossbreed and sample weight. The precision of WHC methods was evaluated according to ISO 5725 (1994) as standard deviation of the difference (s_r) between the replicates (*i.e.* repeatability). Since the absolute values of s_r are not suitable for the comparison of variables which span different variation ranges (like different WHC methods used in this study), the variables were standardized using a normalisation in a continuous space ($x_{NEW} = (x_{OLD} - \bar{x})/SD$, where $\bar{x}$ and *SD* represent the mean and standard deviation, respectively). The repeatability was recalculated on the standardized values (standardized repeatability, Ns_r; Lohninger, 1999).

2.5. Spectral data processing

Spectral data processing was performed using WinISI II (WinISI II Manual, 2000). The samples were first divided into calibration and validation subsets. The samples for calibration set were selected by WinISI II option Make and use scores. Spectra were first reduced to independent sources of variation (scores) to replace the spectra using PL2 option (an algorithm that reduces spectral data to scores and fine-tunes them with reference values) on the basis of global *H*, which was set to 3. After that the redundant spectral information was removed by the selection of samples with the neighbourhood concept (neighbourhood *H* set to 0.5). Using described procedure 147 samples (65%) were placed into the calibration set and the remaining 81 samples were used as an independent (prediction) set for validation.

Calibration models for each reference method were prepared separately for visible (λ = 400–1100 nm, which covers visible and Herschel infrared spectral range), for NIR (λ = 1100–2500 nm) and for the whole spectrum (λ = 400–2500 nm). All models for the prediction of WHC were developed using modified partial least squares regression with internal cross-validation. Samples for which the difference between actual and predicted values exceeded three standard deviations were considered as outliers. The mathematical treatment applied was 1 4 4 1, where the first number indicates the order of the derivative (1 is the first derivative of the log 1/*R*), the second number is the gap in nm over which the derivative is calculated, the third and fourth number refer to the first and the second smoothing. The "SNV and Detrend" option was used to correct scatter effects in the spectra. The number of PLS factors was limited to 10. The actual number of PLS factors was defined for every single calibration model respecting the fall