Development of a Low-cost NIR Instrument for Minced Meat Analysis: Spectrophotometer and Sample Presentation

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Abstract

The feasibility of using a compact, economical NIR instrument to predict moisture content (MC) and total fat content (TFC) in minced pork was demonstrated. The instrument was evaluated using two different interactance geometries of the measuring head. Results were compared with those from the NIRS6500, which is commonly used as a standard NIR research instrument. Minced pork samples were prepared from loin, ham, bacon and shank muscles. PLS calibration indicated that using spectra from the short wavelength region from 700 nm to 1050 nm provided similar results between instruments. The standard error of cross validation (SECV) for MC and TFC were 2.4 % W/W and 2.5% W/W dry weight. The ratio of the standard deviation of the reference data to the standard error of cross validation (RPD) was 3.8 for MC and 4.3 for TFC, indicating that the compact NIR instrument is suitable for screening applications. NIR instruments are routinely used in the food industry to determine quality characteristics such as MC and TFC. However, the equipment is typically expensive. This research demonstrates that compact, economical NIR instruments have the potential for alleviating the issue.

Keywords: Near infrared; Minced meat; Pork; Total fat content; Moisture content

1. Introduction

Near infrared (NIR) spectroscopy is a powerful tool for quality evaluation of meat products, especially at the research level (Ozaki et al., 2007; Prieto et al., 2009; Weeranantanaphan et al.,
2011). However, due to industry scepticism regarding high equipment cost, the implementation of NIR technology in the meat industry, both for off-line and in-line quality control, lags behind other industries such as wheat and fruit. Although the technology itself has been academically accepted and is the registered AOAC method for fat, protein and moisture content analysis (AOAC, 2007), implementation has been limited to large manufacturers.

Considering past meat scandals such as false labelling of ground beef by mixing mutton and chicken by the Meat Hope Co. in Japan in 2007, or the horse meat scandal in Europe in 2013, the potential benefits of a low cost system to measure quality indices such as fat content and to verify product labels has become clear. The use of such equipment by local food suppliers is needed to regain consumer trust. In a separate study, we found that beef, pork and chicken had different fatty acid ratios (Noguchi et al, 2012). Here the objective is to develop a system to measure such ratios in ground meat and verify their origins. Specifically, as a prelude to full scale label authentication, the objective is to verify that a compact and low cost NIR instrument, initially developed for measuring fatty acid groups in subcutaneous fat of swine carcasses (Cyranoski, 2008), would be sufficiently accurate for minced meat evaluation compared with a standard NIR instrument.

2. Materials and Method

2.1 Samples
Samples were acquired from four muscles (bacon, ham, loin and shank) of twenty swine carcasses, for a total of eighty samples. Sampling was performed after allowing the carcasses to age for five days at the Zennoh slaughter house in Tsukuba city, Japan. A hand meat mincer with a 3.2-mm diameter sieve size was used to mince the samples, which were placed in standard zip-lock bags, packed in cooler boxes, and transported to the National Food Research Institute (NFRI), also in Tsukuba city. The samples were subsequently stored in a 5 °C storage room for 48 hours before spectral acquisition. After 24 hours of storage, the samples were kneaded by hand to remove cracks between minced pork particles. Samples for S/N analysis were purchased from a retail supermarket and transferred to ziplock bags prior to hand kneading and spectral acquisition.

2.2 Spectral acquisition
NIR spectra were acquired using two configurations of the compact NIR instrument, as well as with the standard research instrument as described below:

i) A compact silicon diode array grating spectrophotometer model S-2930 (Soma Optics Ltd., Tokyo, Japan), (hereafter referred to as SOMA-A), equipped with a measuring head with five lamps aligned at an angle of 60 degrees towards the sample from the sample surface plane (Figure 1 (A)). The instrument had a fixed resolution of 10 nm and its weight and dimension were 12.5 kg and 425(W)x310(H)x325(D) mm (including touch screen computer). Spectra were acquired in the interactance mode in the region from 700 nm to 1050 nm, at 1 nm intervals with 100 times averaging. The detector integration (or gate) time was 100 ms.

ii) The same instrument (hereafter referred to as SOMA-B) with the five lamps aligned perpendicular to the sample (Figure 1 (B)). The resulting acquisition area was the same for both the A and B configurations. Again, spectra were acquired using interactance mode, with the sample
(still in a zip-lock bag) placed on top of the measuring head. The measuring conditions, i.e. wavelength region, intervals and scan times were similar to those of the SOMA-A.

![Diagram of measuring heads](image)

**Fig 1.** Schematic diagram of the measuring heads for the experimental instruments of SOMA-A (left), SOMA-B (center) and NIRS6500 (right).

iii) A research type grating spectrophotometer model NIRSystems 6500 (Foss, Høganäs, Denmark), (hereafter referred to as NIRS6500) equipped with a fiber optic probe in interactance mode (Figure 1) configured as previously reported (Saranwong et al., 2003). The NIRS6500 was used as a standard NIR instrument using the commercially available interactance probe in the 700-1100 nm region. Spectra were acquired in the wavelength region from 400 nm to 1100 nm, at 2 nm intervals with 50 times averaging. Samples in ziplock bags were placed in direct contact with the probe.

Duplicate measurements were performed for the SOMA-A, SOMA-B and NIRS6500 to reduce the effect of sample positioning. In order to simulate the environment of a meat processing facility, NIR measurements were conducted in a 15 °C room. Samples were transferred from the 5 °C storage room to a 10 °C room for a few hours prior to NIR measurements.

The S/N for each instrument was evaluated by measuring the spectra of the same minced pork samples ten times without sample movement at room temperature (25 °C). Sample temperature was maintained at 25 °C using a water bath.

2.3 Chemical analyses

After spectral acquisition, samples were kept at -40 °C and transferred to Zennoh Laboratory for chemical analyses by conventional methods. Two grams of each sample were taken for MC analysis by oven drying at 135 °C for two hours (Weeranantananaphan et al., 2011). Another two grams were taken for TFC analysis by Soxhlet extraction with diethyl ether (Ministry of Education, Culture, Sports, Science and Technology, 2005). Single measurements were performed for MC, while duplicate measurements were performed for TFC analysis. For TFC, the laboratory error (LE),
defined as the standard deviation of the absolute residue between duplicate measurements, was calculated (Williams and Norris, 2001). Samples with absolute residue value greater than twice the LE value (p<0.05) were not used for the calibrations.

3. Data Analysis

Average spectra derived from duplicate measurements were used for each instrument. The spectra were pretreated using a Savitzky-Golay second derivative (28 nm averaging for left and right side, and second order polynomial) Partial least squares (PLS) regression was used to develop the calibration equations. The full NIR wavelength regions acquired by each instrument were used for the calibration. Full cross validation was used due to the complexity of the sample mixture.

4. Results and Discussion

4.1 NIR spectra of minced pork samples

NIR spectra with base line correction of minced pork (bacon) samples acquired by each instrument are shown in Figure 2. Water and fat bands in the spectra from all instruments are clearly observed around 970 nm and 926 nm, respectively. This similarity of observed peaks between the test and standard instruments indicates that spectra derived from the compact instrument are suitable for the calibrations that follow.

![NIR spectra of minced pork samples](image)

Fig 2. Typical spectra of minced pork (bacon) samples obtained from each instrument.

<table>
<thead>
<tr>
<th>Items</th>
<th>Moisture (% W/W)</th>
<th>Fat (% W/W Dry Weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>67.2</td>
<td>10.5</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>9.07</td>
<td>10.80</td>
</tr>
<tr>
<td>Minimum</td>
<td>43.3</td>
<td>2.3</td>
</tr>
<tr>
<td>Maximum</td>
<td>75.0</td>
<td>43.5</td>
</tr>
<tr>
<td>Number of samples</td>
<td>79</td>
<td>64</td>
</tr>
</tbody>
</table>
4.2 Calibration and validation for moisture and TFCs

Due to an error in spectral recording, one spectrum was missing, leaving 79 samples for MC calibrations. For TFC, the LE calculated was 0.49 %W/W dry weight (d.w.) resulting in the removal of twelve samples having absolute residue more than 0.98 %W/W d.w. Three additional samples identified as outliers in calibrations were also removed, leaving a total of 64 samples for TFC calibrations. Chemical characteristics of the samples used for developing the calibration equations are shown in Table 1. The PLS calibration results for each instrument are shown in Table 2. Calibration results of the SOMA-A instrument were superior to those of SOMA-B. Since the same model of spectrophotometer is used in both cases, the difference in results was presumably due to the differences in sample presentation. For the SOMA-A instrument, incident light is concentrated towards the middle portion of sample at a depth of approximately 15 mm (Figure 1). This configuration reduces loss of NIR light due to internal diffuse reflection, as compared to the configuration for SOMA-B in which light is conducted into the sample at a 90 degree angle. The standard error of cross validation for calibration equations of MC and TFC for SOMA-A data were 2.4%WW and 2.5%WW d.w. respectively, similar to the results obtained for data from the NIRS6500. The RPD values, for SOMA-A calibrations were 3.8 for MC and 4.3 for TFC, indicating the instrument is suitable for screening applications.

Table 2 PLS calibration results for moisture content (MC) and total fat content (TFC)

<table>
<thead>
<tr>
<th>Spectrophotometer</th>
<th>MC (%W/W)</th>
<th>TFC (% W/W Dry Weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$F$</td>
<td>$R^2_c$</td>
</tr>
<tr>
<td>Soma-A</td>
<td>3</td>
<td>0.94</td>
</tr>
<tr>
<td>Soma-B</td>
<td>3</td>
<td>0.93</td>
</tr>
<tr>
<td>NIR6500</td>
<td>5</td>
<td>0.96</td>
</tr>
</tbody>
</table>

$F$: The number of factors used in the PLS calibration equation; $R^2_c$: determination coefficient for calibration set; SEC: standard error of calibration; SECV: standard error of cross validation; RPD: ratio of standard deviation of reference data to SECV.

4.3 Scatter plots of MC and TFC for the “SOMA-A”

Fig 3. Scatter plots for (a) moisture and (b) TFC predictions using the calibration models calculated from the spectra acquired with SOMA-A.
Scatter plots of the cross-validation predictions vs. the chemical values for MC and TFC are shown in Figure 3. The predicted values were obtained from the calibration equations shown in Table 2 for the SOMA-A. Samples were unevenly distributed along the target line (the 45 degree line) because TFC varied widely depending on muscle type (bacon, ham, loin or shank). While this uneven sample distribution is not ideal for purposes of developing calibrations, it is not expected to be a factor in comparing performance between instruments.

4.4 Discussion of results

Figure 4 shows the calibration structure for the NIRS6500 and SOMA-A. For the NIRS6500 regression coefficient plots for MC, peaks were found in the vicinity of 950 nm and 776 nm, while, for SOMA-A only one peak in the vicinity of 950 nm was prominent. For TFC, both the NIRS6500 and SOMA-A used only the fat band in the vicinity of 925 nm. The 776 nm and the 950 nm peaks for the MC calibration corresponds to the water band at 758 nm and 964 nm (Williams and Norris, 2001), while the 925 nm peak for the TFC corresponds to the fat absorption band at 926 nm reported earlier (Chen et al., 2002).

Figure 5 shows standard deviations calculated from the ten spectra of the same minced pork samples plotted against wavelengths for the SOMA-A and the NIRS6500. Since the standard deviation is a measure of noise, or "repeatability," this explains why the NIRS6500 results for MC were superior to those for SOMA-A, but similar TFC results were obtained for both instruments. The plots indicate the spectral noise of the instrument, without the effect of sample movement (Williams and Norris, 2001). The 925 nm peak used by both instruments showed similar noise levels. However, the 776 nm peak was used by only the NIRS6500, and in this vicinity, the noise level was three times lower than that of SOMA-A. Even though the noise level of SOMA-A in the vicinity of 950 nm was slightly lower than that of the "NIRS6500", the results in Table 2 indicate that it was not enough to compensate the inability to use information in the 776 nm area. Nevertheless, the RPD obtained for MC by SOMA-A, indicates that this compact instrument is sufficiently accurate for screening applications and perhaps for fatty acid ratio measurement that would lead to label authentication in the future. Development of calibration equations for routine screening applications of SOMA instruments would require larger sample sets and validation by a separate test set over time, as is standard practice for development of NIR applications.
5. Conclusion

A compact, low-cost NIR instrument equipped with a measuring head in which the lamps are configured to direct the incident light towards the centre of sample was demonstrated as the basis for an instrument specialised for quality analysis of various minced meat. The calibration results obtained from the compact instrument were at an acceptable level as compared with the standard NIR instrument.

Acknowledgement

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