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Are low-cost, hand-held NIR sensors suitable to detect adulterations of halal meat?

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Abstract The demand of halal meat products is growing globally. Therefore, it is important to detect adulterations and food fraud attempts in a fast, non-invasive manner for example by using hand-held near-infrared (NIR) devices. In this study, samples of pork, lamb, beef and chicken were measured pure and in mixtures of 2, 5, 10, 25 and 50% pork in the non-pork meat samples, respectively. Five sensors were tested with varying wavelength range: Scio (740-1070 nm), Linksquare (400-1000 nm), Tellspec (900-1700 nm), MicroNIR (900-1650 nm), ASD Labspec 4 High-Res (350-1700 nm). A one-class-classification approach was used for data analysis, applying pork as the target group. For comparison, thresholds of the models were chosen to correctly identify 100% of the pork samples and 75% of all mixtures. Comparing the sensors upon the correct detection of all halal meat samples, i.e., no-pork containing ones, the Scio and the ASD Labspec performed best with an outcome of 34% and 32%, respectively. The Linksquare, MicroNIR and Tellspec were able to correctly identify 27%, 27%, and 10%, respectively, of the halal products. Concluding, the application of these five NIR devices are challenging when it comes to the detection of meat products from different species. Nonetheless, the usage of this application in combination with suitable chemometric approaches may contribute to the detection of food fraud in halal products.

Keywords Near-infrared sensors, pork, lamb, beef, chicken meat, one-class-classification

1 Introduction

The market for halal-certified products increases within Western societies. While halal products have been intended for Muslim consumers, Jewish consumers as well as vegetarians/vegans, and people with various types of allergies or dietary restrictions purchase halal-certified products [1,2]. When it comes to halal meat, several differences are found to the commercial meat that is available in Western countries. Halal meat may only contain meat from ruminant species like cows or birds like chicken. Horse and pig meat are not considered halal. Besides the species also the feed that is fed to the animals plays an important role. Animals fed with additions of biosolids or animal protein concentrates must undergo a quarantine period with other feed before slaughtering. Moreover, halal meat may only be retrieved from a slaughtering process that renders animals immobile or unconscious, without killing it, prior to the blood drainage [2]. These differences in animal species, feed and slaughtering process have been hard to detect and trace. Hence, several cases of fraud occurred as listed by [3] or illustrated in detail by [4]. Both authors conclude that the main enabling factor of halal meat food fraud is the challenging detection of halal meat authenticity. One possible solution to overcome the issue of halal meat authenticity detection is applying spectroscopy. Spectral data may be collected for example by near-infrared (NIR) sensors. [5], [6], and [7] showed that the discrimination of animal species is possible when using a portable FT-IR or benchtop NIR sensor with a wavelength range reaching between 1000 and 2000 nm. although spectral data acquisition is fast and easy to conduct, the machines trialled in the past were very costly, heavy and bulky [8]. In this paper, we show the application of a one-class-classification (OCC) chemometric approach on data obtained by using several hand-held NIR sensors. OCC describes one specific class as the target class and returns predictions of samples being out or in the respective target class. In the case of halal meat detection, in particular the speciation issue, the target class was set as pork meat, i.e., non-halal meat. That means in that all samples that are "in", do contain pork and are therefore not halal. On the other hand, all samples that are "out" do not contain pork and may be labelled as halal.

2 Materials and methods

2.1 Materials and sample preparation

Pork, beef, lamb and chicken meat was purchased at local butchers in Wageningen, the Netherlands. 40 samples of each species were purchased in 20 days, being two different meat parts per day per species, i.e., shoulder and leg of lamb, pig and cow or breast and drumstick from chicken. For reference purposes, all purchased, pure samples underwent a real-time PCR assay to validate the species identity. The method used has been described previously by [9]. All samples were purchased as intact meat and minced with a meat mincer Tristar VM-4310 (Smartwares Europe, Tilburg, Nederland). Mixtures of pork with beef, lamb or chicken, respectively, were prepared in the concentrations 2, 5, 10, 25, and 50% pork/ other meat (w/w). A randomised approach was used to make almost every day six mixtures using the two parts of lamb, beef and chicken mixed with pork, leading to 117 sample mixtures. In total 277 samples were measured, being 117 sample mixtures and 160 pure samples. To ensure that all samples have a similar storage period, all freshly prepared samples and mixtures were frozen, stored at -18 °C and thawed for 12h prior to the measurements.

2.2 NIR spetroscopy and data acquisition

Five different hand-held sensors (Fig. 2.1) were studied as follows:

- Scio (Consumer Physics, Herzliya, Israel), wavelength range 740-1070 nm, size 6.8 x 4.0 x 1.9 cm
- Linksquare (Stratio Inc, Paolo Alto, CA, USA), wavelength range 400-1000 nm, size 2.4 x 11.4 x 2.4 cm
- Tellspec (Tellspec Inc., Toronto, Ontario, Canada), wavelength range 900-1700 nm, size 8.2 x 6.6 x 4.5 cm
- MicroNIR (Viavi Solutions Inc, Santa Rosa, CA, USA (former JDSU)), wavelength range 900-1650 nm, size 4.5 x 4.4 x 4.0 cm
- ASD Labspec 4 HighRes (Malvern Panalytica, Almelo, the Netherlands (former ASD)), wavelength range 350-1700 nm, size 12.7 x 36.8 x 29.2 cm

The NIR hardware was calibrated according to the manufacturer instruction with a 99% diffuse reflectance standard. Measurements were done in diffuse reflectance mode by slightly pressing the optical part of the sensor to the sample or by slightly hoovering the optical part above the sample in a distance of about 1 cm. For the Scio, Tellspec, Linksquare and ASD Labspec sensors the integration time was set automatically by the manufacturer, for MicroNIR the integration time was set at 8 ms with 200 scans. Spectral measurements were conducted at room temperature when the meat had a temperature between 19 and 21 °C. The measurements were repeated four times.



Figure 2.1: Scio, Linksquare, Tellspec, MicroNIR, and ASDLabspec (pictures courtesy of the respective hardware manufacturers).

2.3 Data analysis

Outliers were excluded manually using principal component analysis after standard normal variate (SNV) pre-processing in Unscrambler X 10.5 (Camo Analytics AS, Oslo, Norway). Out of the 1108 acquired spectra, 12 of Scio, 10 of Linksquare, 144 of Tellspec, 16 of MicroNIR, and 13 of ASD Labspec were excluded, resulting in 1096, 1098, 964, 1092, and 1095 spectra for Scio, Linksquare, Tellspec, MicroNIR and ASD Labspec, respectively. It is worth mentioning that the outliers are probably measurement errors, as no sample was found to be an outlier for all sensors. The OCC chemometric approach was conducted in R 3.6.1 (R Core Team, 2018) as described in detail by [10] and [11]. The same R-packages were used as previously reported. Among commonly used pre-processing methods and algorithms the most suited combination was picked according to (area under the receiver operator characteristic) AUROCs of the target class (pork) against the individual other classes, namely beef, lamb and chicken. For each sensor, three models were picked manually. Averaged class distances of four

repetitive measurements (in a row on one occasion) were used for further calculations. Classification results of each model were fused into a final classification using a decision tree, i.e., if more than one out of the three models classified a sample as 'out-of-class' it was classified as 'not containing pork'.

3 Results and discussion

3.1 Raw NIR data

The five sensors used in this study resulted in five very different groups of NIR spectra (Fig. 3.1). Next to the different measurement range of the sensors, also the hardware technology used differs for each sensor, resulting in different responses at each wavelength.



Figure 3.1: Raw data of all pure samples averaged according to meat species (Wavelength ranges were Scio 740-1070 nm, Linksquare 400-1000 nm, Tellspec 900-1700nm, MicroNIR 900-1650 nm and ASD Labspec 4 HighRes 350-1700nm).

3.2 Models picked for the different sensors

- For the Scio sensor all models only used the fourth quarter section from the four sections with equal lengths sorted by increasing wavelength. The first model included SNV as pre-processing and used Soft Independent Modelling of Class Analogies (SIMCA) with three principal components (PCs) as selected by a 5-fold (inner loop) cross-validation. The second model used the 1rst derivative (Savitzky-Golay) an 11-point filter length and Principal Components Analysis (PCA) residual, calculating the sample residuals (Q residuals) with three principal components as selected using a 5-fold (inner loop) cross-validation. The third model included the 2nd derivative (Savitzky-Golay, 11-point filter length) and One-Class Support Vector Machine (OCSVM) with radial basis kernel and automatic parameter estimation.
- The Linksquare sensor models included in the first model SNV followed by baseline correction (SNV-DT, Detrend). It used only the fourth section of the spectra and a SIMCA (3PCs). The second model combined the 1st derivative (Savitzky-Golay, 11-point filter length), used only the fourth section of the spectra and calculated the distance to the k-Nearest Neighbor (kNN), with two neighbours as selected using a 5-fold (inner loop) cross-validation. The third model used the 2nd section of the spectra, SNV-DT and kNN as the algorithm.
- All Tellspec models were based on the fourth section of the spectra. The first one included the 1st derivative (Savitzky-Golay, 11-point filter length) and PCA (3PCs), the second the 2nd derivative (Savitzky-Golay, 11-point filter length) and kNN (2 neighbors) and the third SNV and PCA (3 PCs).
- MicroNIR spectra were analysed using only the fourth section of the spectra. The first model included the 2nd derivative (Savitzky-Golay, 11-point filter length), and PCA (3 PCs), the second the same 2nd derivative processing and kNN (2 neighbors) and the third the 1rst derivative (Savitzky-Golay, 11-point filter length), and PC (3 PCs).

• The first ASD Labspec model used the fourth section of the spectra, SNV-DT and SIMCA (3PCs). The second SNV, the second section of the spectra and SIMCE (3PCs), and the third the 2nd derivative (Savitzky-Golay, 11-point filter length), the fourth section of the spectra and OCSVM.



Figure 3.2: Correct classification in % for pork, mixtures (2 to 50% pork in lamb, beef and chicken (w/w)), lamb, beef and chicken samples deriving from the fused classification results of three models per sensor, respectively. Thresholds were set to achieve a correct identification of 100% pork and 75% mixture samples.

3.3 Determination of thresholds after fusion of classification results

The thresholds of the fused classification results were set in the way that 100% of all samples containing 100% pork were correctly identified as "pork" (Fig. 3.2). In that case, with no false negatives, the correct classification of pure beef, chicken, and mutton samples, as well as mixtures, was lower than 100%. The correct classification of mixtures was set by adjusting the thresholds at 75%, whereby the ones containing less than 10% pork where wrongly identified in most cases. In summary, the thresholds were chosen in a way that some halal products, i.e., pure lamb, beef and chicken, were wrongly identified as containing pork but no pork was undetected. In an industrial setting, this approach should

be beneficial as it reduces the number of samples that must be identified by more complex, costly and time-intensive experiments such as polymerase chain reactions (PCR).

3.4 Comparing sensors

Overall the Scio and the ASD Labspec performed best as still 34% or 32%, respectively, of the halal products were classified correctly (Fig. 3.2). Nevertheless, the Scio sensor was only able to discriminate beef samples as 'non-pork' but none of the other meat species such as chicken and lamb. Here, the ASD labspec sensor performed better identifying correctly, 15% of lamb, 51% of beef and 29% of chicken samples. In contrast to the two mentioned sensors, The Linksquare and MicroNIR identified 27% of the halal-products correctly and the Tellspec only 10%. Both the Linksquare and the ASD Labspec performed well on the identification of chicken with 53% and 29% correctly identified. This may be caused by the wider wavelength range that includes part of the visible spectrum 400-1000 nm and 350-1700 nm for Linksquare and ASD Labspec, respectively. Chicken meat has a lighter colour than the other meat species and may be discriminated visually. Also, both sensors included models that used the second section of the spectrum where the visible wavelengths may be found. In literature, the discrimination of meat according to its protein, moisture and fat content was successful using the Scio and another miniaturized NIR device. [12] showed that samples containing fat from 5% to 43%, protein from 12% to 23% and a moisture content of 35% to 69% may be correctly classified. In contrast to the study of [12], the samples used in this study are probably too close to each other concerning their composition. According to the USDA database, raw pork contains 72.6-72.9 g/100 g water, 19.6-20.5 g/100 g protein, 5.4-7.1 g/100 g fat, whereas chicken may contain 73.9-76.8 g /100 g water, 19.4-22.5 g/100 g protein, 2.6-3.7 g/100 g protein fat, lamb 72.5-74.4 g/100 g water, 19.7-21.1 g/100 g protein, 6.5-8.3 g/100 g fat and beef 73.5-73.6 g/100 g water, 20.4-20.5 g/100 g protein, 5.4-6.7 g/100 g fat. These might be the reason why, so few samples of the pure, halal-meat products were correctly identified when the threshold was chosen to correctly detect mixtures in a 75% rate.

4 Conclusion and outlook

The application of fast, cheap and hand-held devices is challenging when it comes to the detection of meat products from different species. Nevertheless, the presented OCC approach enables the application of these devices to contribute to species admixture detection for halalcertified products. Five different devices were tested. The Scio and the ASD Labspec performed best followed by the Linksquare, MicroNIR and Tellspec sensor. Further research is conducted using hyperspectral imaging cameras that cover a wavelength range in the VIS-NIR from 400-1700 nm and include spatial information for classification attempts. Moreover, different slaughtering techniques and feeding will be included.

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