PREDICTION OF AGEING TIME OF BEEF STEAKS USING VISIBLE AND NEAR INFRARED REFLECTANCE SPECTROSCOPY

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Abstract – The use of visible/ near infrared reflectance (VIS 400-800 nm and NIR 1100-2500 nm) spectroscopy has been suggested as a food authentication tool. To assess if this technology can be used to authenticate the ageing time of premium beef steaks, *longissimus thoracis* beef steaks were wet-aged (3ºC ± 1ºC) and spectra collected (after 1h oxygenation) at 3, 7, 14 and 21 days of storage (n=112). Principal component analysis (PCA) and partial least-squares (PLS) models were used to investigate if NIR spectroscopy could identify and predict the ageing time of beef. Preliminary results indicate that PCA of spectral data was able to group the samples per ageing time, this grouping being significantly better in extreme samples (3 days and 21 days of ageing). The prediction cross-validation coefficients (r^2) were 0.80 and 0.82 for the NIR and VIS ranges respectively, with the prediction of extreme samples being more accurate compared with 7 and 14 days of ageing. In conclusion, the results showed the potential of both VIS and NIR spectra as objective and rapid methods to predict ageing in beef steaks.

Key Words – Near infrared reflectance spectroscopy, ageing, beef.

I. INTRODUCTION

Authentication to avoid fraud and ensure consumer satisfaction is a key issue for the food industry [1].

In many countries beef steaks aged for a long time are considered to be premium quality products, and priced considerably higher than standard steaks [2] (unaged or aged for a shorter time). For these reasons the meat industry and consumers have an urgent need for methods that can ensure that they are getting what they are paying for.

Analytical methods to verify ageing are lengthy, complex and destructive and are therefore not suitable for application in quality control *per se* [3]. Visible/near infrared reflectance spectroscopy (Vis/NIRS) has potential for this application as it is a non-destructive technique that is quick and relatively easy to use. NIRS research has been mainly focused on the calibration of this technology to predict meat quality [4] and there are few studies using it as an authentication tool in meat. These studies report that NIRS can be used to discriminate between fresh and frozen beef [5], chicken meat muscles [6], and meat of different animal species [7].

During ageing of meat, many biochemical changes occur , the most interesting being the activity of proteases which increase the tenderness [8]. These biochemical changes will produce a characteristic pattern of NIR absorbance known as a fingerprint [9]. The aim of this study was to investigate the reliability and accuracy of the VIS and NIRS spectra to detect the specific fingerprint of premium beef samples differing in ageing time without depending on chemical information.

II. MATERIALS AND METHODS

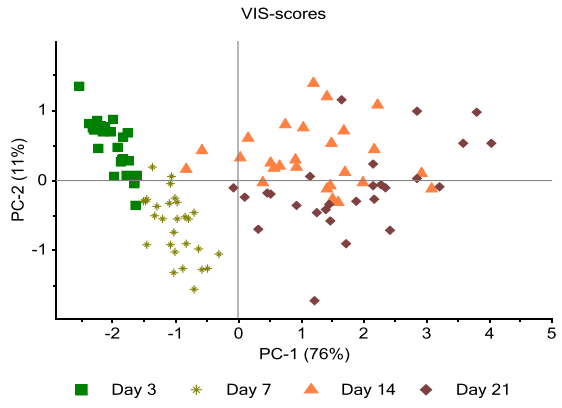
Twenty four month-old steers reared under the same production conditions were slaughtered in a commercial plant by captive bolt stunning followed by exsanguination from the jugular vein. After 48 h in the chill at 0°C the *longissimus thoracis* (LT) was removed, vacuum packed and transported to Teagasc, Food Research Centre Ashtown, Dublin. At 3 days post-mortem, 25 mm thick rib-eye steaks (n=28) were cut from the 10th rib and after 1h of oxygenation each steak was scanned (350-2500 nm) in reflectance mode (10ºC) using a portable ASD Labspec 5000 (ASD Inc., Boulder Colorado, USA) Vis/NIR spectrometer fitted with a high-intensity contact probe with a 10 mm spot size and using the Indico Pro program (ASD Inc.). The 3 days post mortem steaks were considered as unaged. Additional steaks were vacuum packed, and scanned after ageing for 7, 14 or 21 days. At each time, the scanning conditions were as described above (1h

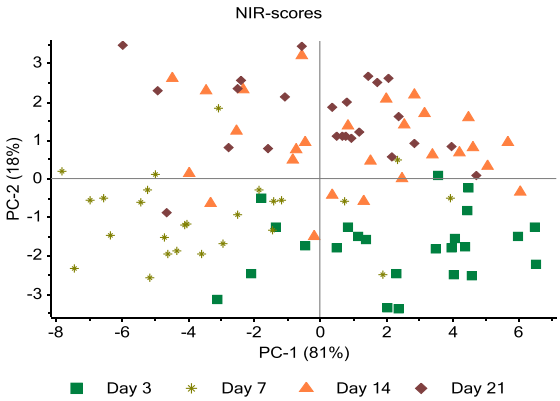
of oxygenation, 10^0C). Vis/NIR spectra (350-2500 nm at 1 nm intervals) were recorded in duplicate (two different spots per steak) by removing and replacing the scanning probe head on the meat surface between scans. Spectra were recorded as log (1/Reflectance) and exported as JCAMP to The Unscrambler X version 10.3 (CAMO ASA, Oslo, Norway) for chemometric analysis. Duplicate spectra were averaged and standard normal variate (SNV) correction was applied for principal component analysis (PCA). PCA was performed using full cross-validation, with uncertainty test using the optimal number of PC, the algorithm used was NIPALS. Both PCA analyses were applied for 10 principal components independently, for the VIS and NIR wavelength ranges to examine the natural groupings of the samples. Discriminant analysis was performed using dummy partial least squares regression (PLS) without any correction using Kernel algorithm. Eighteen spectra of each ageing time (3, 7, 14 and 21 days) were randomly selected from the dataset to form the calibration set while the remaining spectra were used as a validation set (n=40) for regression prediction for VIS and NIR regions independently.

I. RESULTS AND DISCUSSION

Figure 1 shows the discriminant plots for the different ageing times analyzed using the VIS and NIR spectral ranges separately. VIS PC1 explained 76% while PC2 explained 11% of the total variance. For the NIR spectrum PC1 and PC2 explained 81% and 18% of the total variance, respectively.

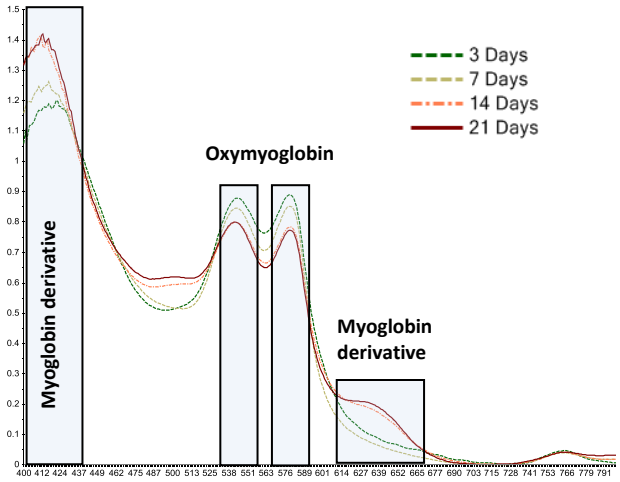
Figure 1. PC1 and PC2 discrimination between ageing days using visible or near-infrared spectra





The highest loadings for the VIS range (PC1 to PC3) were found around the 400-420, 530-590 and 624-650 nm regions. These spectral regions have been related previously with heme-pigment [4]. Figure 2 shows that samples with shorter ageing times (average) had higher peak intensity in the range associated with oxymyoglobin, while with increased ageing time the peak intensity in areas related with other oxidative forms of myoglobin was higher in line with Lyon et al. [4].

Figure 2. Average of the visible spectra at 3, 7, 14 and 21 ageing days.



NIR results (Figure 3) are less clear but, in line with [10] and [11], in general the absorption decreased with increased ageing, with the decrease being particularly noticeable between 1100-1300 nm. The absorbance value in this region has been shown to be negatively correlated with tenderness (i.e. higher absorbance relates to less tender meat) [11]. There was a clear separation between samples aged for 3 or 7 days versus those aged for 14 or 21 days in this region. It is also important to

indicate that the wavelengths related with water (1410-1460) and (1900-2000) increased considerably with ageing time (higher loadings). This increase might be related to the changes in water distribution and mobility during ageing, and probably with the water holding capacity of the samples [12].

Figure 3. Average of the NIR spectra at different 3, 7, 14 and 21 ageing days.

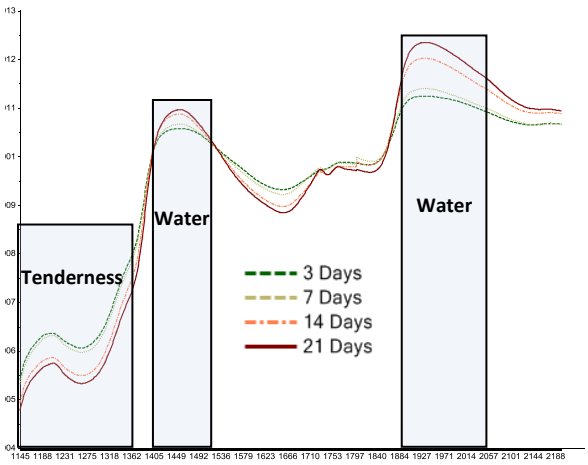


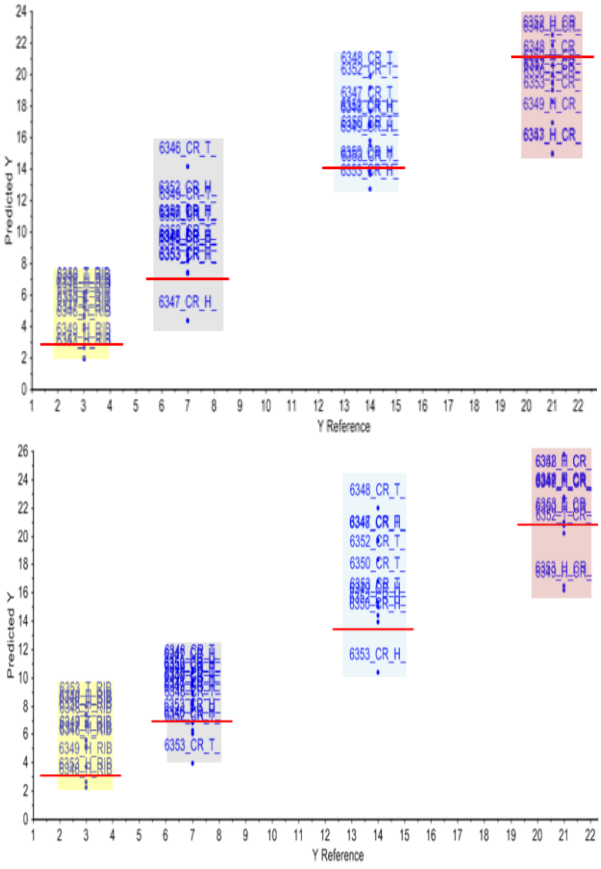
Table 1 shows the calibration statistics for ageing using PLS on untreated spectral data. All the models had a coefficient of determination in calibration (R^2_{cal}) higher than 0.75. Taking account of the relatively small number of samples and the absence of any pretreatment on the spectra these results seem very promising.

Table 1. Visible and near infrared prediction statistics using PLS for ageing time classification.

Item	Slope	RMSE	R-square
VIS PLS	0.814	3.012	0.804
NIR PLS	0.841	3.312	0.756

Regression was applied using 3 factors for VIS and 6 factors for NIR spectra on the validation sample set. Both NIR and VIS gave robust models to predict the ageing time (NIR $r^2 = 0.80$ and VIS $r^2 = 0.82$). Figure 4 represents how samples with known ageing time (reference X axis) are predicted by the model (prediction Y axis). The red line indicates the real value for Y and the coloured areas the dispersion of the samples from the reference value. In general both VIS and NIR are able to classify perfectly between unaged and 21 days aged samples (coloured areas do not overlap). Conversely, in both models, samples with 7 days of ageing can be easily misclassified, mainly as unaged samples, and samples with 14 days can be easily misclassified as 21 days aged samples. These problems with the prediction may be due to the higher variability in tenderness at intermediate days of ageing. This variability could be related with the ageing process itself as it is well known that meat ageing is a very variable process, depending on a high number of biological factors [8]. For these reasons some samples may have reached the plateau of tenderness earlier than 21 days, while other samples needed more time than the average to reach the maximum tenderness.

Figure 4. Visible and near infrared predicted & reference classification using regression.



III. CONCLUSION

In conclusion, the use of VIS/NIR as a tool to authenticate the ageing time of intact steaks seems promising. It allows us to discern perfectly between unaged and 14 or 21 days aged steaks, but further studies are needed to improve the

discrimination between the intermediate ageing classes.

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