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PROXIMATE ANALYSIS OF BEEF SAMPLES BY NEAR-INFRARED REFLECTANCE SPECTROSCOPY

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SUMMARY

Near infrared reflectance spectroscopy, using a fixed-filter instrument (Technicon InfraAlyzer 400R), was evaluated for the determination of moisture, protein and fat in beef samples. 3-rib cuts of beef and the M. longissimus dorsi were the samples on which the technique was evaluated by comparison with chemically-determined moisture (oven-drying), protein (Kjeldahl) and fat (Foss-let). An all-possible-combinations wavelength search routine (using log 1/R data at 19 wavelengths) was used to determine the optimum wavelength combinations for predicting each constituent.

For the 3-rib beef cuts (moisture, 42-64%; protein, 13-20%; fat, 15-42%) correlation coefficients were 0.97-0.99 for the three constituents. The narrow ranges of values for the constituents in M. longissimus dorsi samples (moisture, 70-76%; protein, 21-24%; fat, 1-5%) made calibration development difficult. No appreciable difference was found for M. longissimus dorsi samples prepared using a coffee-grinder or Ultra-turrax. The near infrared reflectance technique is suitable for prediction of moisture, protein and fat in 3-rib beef cut samples used for carcase evaluation studies.

INTRODUCTION

Near infrared (NIR) reflectance spectroscopy has been used for the analysis of agricultural products and foods. This technique has been applied to the analysis of meat and meat products with varying success.[1-4] In summary, the NIR technique has been found to give good results for moisture and fat but poorer results for protein. Some of the problems associated with the application of NIR to meat analysis are sample homogenisation and its presentation in the instrument and the effects of temperature, pH and the quantities and types of protein and fat in the meat.[1]

NIR reflectance spectroscopy has been found to be an effective method for carcase composition determination of poultry, based on the measurement of fat and moisture.[5] The objective of this study was to determine the accuracy of the NIR technique for measuring moisture, protein and fat in beef samples used for carcase evaluation. Two very different sample types were used in terms of muscle and fat content and the ranges of these constituents in the samples. Different sample homogenisation systems were evaluated and the temperature of the samples was closely controlled.

MATERIALS AND METHODS

Samples were taken from steer beef carcases during boning-out for carcase evaluation. A 3-rib (10th to 12th) cut and a sample of M. longissimus dorsi were taken from one side of each carcase and the 3-rib cut was deboned. The samples were minced sequentially through 10 mm and 5 mm sieves with mixing of samples by quartering between mincings. 5-600 g of each sample was frozen and then minced through a 2mm sieve. Subsamples were refrozen and stored for analysis.

Chemical analyses for moisture, protein, fat and ash were carried out on thawed samples. Moisture and ash were determined on samples (3g) in ovens at 103°C and 500°C, respectively, for 16 h. Protein (Kjeldahl) was determined on samples (1g) using sulphuric acid digestion with copper catalyst and quantitation by automated titration (Tecator 1030 Analyser). Fat was determined by the Foss-let technique using 30g sample[6]. All analyses were in duplicate.

Samples of frozen rib cut and M. longissimus dorsi were ground in coffee-grinders (Moulinex Super Junior S) for 30 seconds. Samples of the M. longissimus dorsi were homogenised, also, in an Ultra-turrax, after thawing, for 90 seconds, with the samples being held in an ice-bath during homogenisation to prevent over-heating. All samples were stored in a refrigerator overnight and then placed in an incubator (23°C) for 3 h before reading on the NIR spectrophotometer. Samples were read on an InfraAlyzer 400R (Technicon Instruments) at 19 wavelengths in the region 1400-2400 nm. Samples were read as duplicate packings in open cups, with each packing being read twice after rotation of the cup through 180 degrees. Reflectance data were collected as log 1/R values for each of the 19 wavelengths and stored on magnetic tape using an interfaced HP-85B computer (Hewlett-Packard) and dedicated software (Technicon Instruments).

A total of 69 3-rib cut samples and 41 M. longissimus dorsi samples were available for this study. Sample sets of 39 (calibration) and 30 (prediction) were developed by random selection from the 69 3-rib cut samples. Calibration development was performed using an all-possible-combinations search routine for groups of 3 to 5 wavelengths. The best 15 calibrations for each group, on the basis of the multiple correlation coefficent (R), were evaluated on the prediction set by simple linear regression analysis of predicted versus chemical data. Calibration development was performed, as described above, for the total 41 M. longissimus dorsi samples and the derived calibrations used to compare the two sample homogenisation techniques.

RESULTS AND DISCUSSION

The results of the duplicate chemical analyses for percentage moisture, protein and fat are contained in Table 1. The ranges of moisture, protein and fat for the 3-rib cut samples are sufficently wide to be suitable for development of a NIR calibration, but the ranges of these constituents in the M. longissimus dorsi samples are relatively narrow. The range of ash values for both sample types (3-rib cut samples: 0.8 - 1.2%, M. longissimus dorsi samples: 0.8 - 1.5%) are too narrow for development of a NIR calibration for this constituent. The sum of moisture, protein, fat and ash for the 3-rib cut samples and the M. longissimus dorsi samples have mean values, standard deviations and ranges of 100.3, 0.73, 98.2-101.5% and 100.1, 0.95, 98.6-101.8%, respectively. The precision of the chemical analyses is given by the standard deviation of differences between duplicates in Table 1.

Preparation of 3-rib cut samples by homogenising the frozen, minced material in a coffee-grinder for 30 seconds yielded a suitable material for packing in the open cup of the InfraAlyzer. An even surface was achieved without obvious loss of moisture from the surface of the meat. However, suitable samples for NIR analysis were not obtained with this technique for the high moisture/low fat samples of M. longissimus dorsi. These samples were difficult to pack, yielding a surface which broke rapidly and moisture was removed from the sample surface during packing. An alternative sample homogenisation technique, using an Ultra-turrax,

was evaluated for the M. longissimus dorsi samples. A limit of 90 seconds homogenisation was set because of the heat generated in the process. The samples produced with this technique had a different consistency to those produced with the coffee-grinder but were not homogenous. Sample homogenisation is an important problem for the application of NIR analysis to meat samples, especially those with relatively low fat content (<10%). The use of a food processor/bowl cutter may yield a more suitable sample for NIR analysis. [2,7]

Table 1. Chemical analyses of meat samples

Sample set (n)	Constituent	Range(%)	Mean	SD[a]	SDD[b]
3-rib cut, calibration (39)	moisture	44.2-64.5	55.2	5.6	0.33
	protein	14.2-19.9	17.2	1.6	0.24
	fat	15.1-41.4	27.0	7.0	0.36
3-rib cut, prediction (30)	moisture	41.6-64.6	55.1	5.6	0.29
	protein	13.2-19.5	17.1	1.6	0.24
	fat	16.3-42.7	27.2	6.8	0.41
M. l. dorsi, calibration (41)	moisture	70.6-76.2	73.7	1.2	0.23
	protein	21.6-24.4	23.1	0.7	0.28
	fat	0.6-5.7	2.1	1.2	0.22

[a] Standard deviation for values in the sample set.

[b] Standard deviation for differences between duplicate chemical measurements.

The calibrations for moisture, protein and fat in 3-rib cut samples (Table 2) are chosen on the basis of their performance on the prediction set. Calibrations with the least number of wavelength terms are chosen where addition of further wavelength terms does not appreciably improve the correlation coefficient(r) and the standard error of prediction (SEP). The particular wavelength combinations shown for moisture, protein and fat were selected as they contain the wavelengths 1940 for moisture, 2180 for protein and 1759 for fat, respectively. The three constituents, moisture, protein and fat, can be determined with acceptable accuracy by NIR spectroscopy on 3-rib cuts of beef.

Table 2. Calibration and prediction statistics for the near infrared reflectance determination of moisture, protein and fat in 3-rib cuts of beef.

Constituent	Wavelengths (nm)	Calibration set (n=39)		Prediction set (n=30)	
		R[a]	SEC[b]	r[c]	SEP[d]
Moisture	1940, 2100, 2139, 2208	0.99	0.92	0.98	1.13
Protein	1680, 1818, 2180	0.97	0.41	0.99	0.26
Fat	1680, 1759, 2190	0.99	1.03	0.99	0.98

[a] Multiple correlation coefficient

[b] Standard error of calibration

[c] Correlation coefficient

[d] Standard error of prediction

Calibrations for moisture, protein and fat in M. longissimus dorsi samples are shown in Table 3. The particular wavelength combinations shown have the same number of wavelengths as are used for each constituent in the 3-rib cut samples (Table 2) and are the "best set" in each case. The multiple correlation coefficients for moisture and protein are very low and the standard error for protein is too high for practical use. The calibration for fat is somewhat better and may be sufficiently accurate to replace the chemical method. Without the availability of sufficient samples for a prediction set, it is not possible to properly assess these calibrations. No differences are found between the two sample homogenisation techniques used.

Table 3. Calibration statistics for the near infrared reflectance determination of moisture, protein and fat in M. longissimus dorsi samples (n=41) of beef prepared by two homogenisation techniques.

Constituent	Homogeniser	Wavelengths (nm)	Calibration set	
			R[a]	SEC[b]
Moisture	Coffee -grinder	1680, 1722, 2100, 2336	0.87	0.60
	Ultra -turrax	1680, 1734, 1940, 1818	0.72	0.82
Protein	Coffee -grinder	2139, 2190 2208	0.49	0.66
	Ultra -turrax	1722, 1734 1778	0.64	0.57
Fat	Coffee -grinder	1680, 1759 2100	0.93	0.44
	Ultra -turrax	2270, 2310 2336	0.92	0.48

[a] Multiple correlation coefficient

[b] Standard error of calibration

CONCLUSIONS

NIR reflectance spectroscopy provides a rapid technique for the analysis of moisture, protein and fat in 3-rib cut samples of beef which are used in carcase evaluation studies. The technique needs to be evaluated further to establish its usefulness for the determination of these constituents in M. longissimus dorsi samples.

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REFERENCES

1. Kruggel, W.G., Field, R.A., Riley, M.L., Radlaff, H.D. and Horton, K.M. Near-infrared reflectance determination of fat, protein and moisture in fresh meat. J. Assoc. Off. Anal. Chem. 1981, 64 (3), 692-696.

2. Lanza, E. Determination of moisture, protein, fat and calories in raw pork and beef by near infrared spectroscopy. J. Food Sci. 1983, 48, 471-474.

3. Mifek, K. and Fruhling, R. Untersuchung zur Analyse tierischer lebensmittel mittels IR-reflexionsanalyse. Ernahrung 1985, 9 (3), 201-208.

4. Kopp, J., Bonnet, M. and Renou, J.P. Controle non-destructif: application des techniques utilisant l'infrarouge et la RMN au controle de la viande et des produits carnes. Spectra 1986, 14 (110), 37-43.

5. Renden, J.A., Oates, S.S. and Reed, R.B. Determination of body fat and moisture in dwarf hens with near-infrared reflectance spectroscopy. Poultry Sci. 1986, 65. 1539-1541.

6. Official Methods of Analysis (Horwitz, W. Ed.) AOAC, Arlington, VA, 1980, 13th edn, sec. 24.006, p. 376.

7. Hildrum, K.I., Martens, H. and Lea, P. Detection of errors in the NIR-analysis of meat components, in Proceedings of the 30th European Meeting of Meat Research Workers, Bristol, England, 1984, pp. 399-400.