DETECTING PREVIOUS FROZEN BEEF BY NEAR INFRARED SPECTROSCOPY

Kari Thyholt and Tomas Isaksson

MATFORSK, Norwegian Food Research Institute, Osloveien 1, N-1430 Ås, Norway and
Dep. of Food Science, Agric. Univ. of Norway, P.O. Box 5003, N-1432 Ås, Norway

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Introduction

Freezing affects the quality of meat by freezer burn, decreased juiciness, increased drip loss and microbial growth (1-5). According to European Union regulations, labelling should state that the meat has been frozen and must not be refrozen. Consequently, there is need for a fast method to detect if the beef has been frozen.

DESIR (Dry Extract Spectroscopy by Infrared Reflection) has been used to examine solutions (6-13). This technique rapidly removes the water, which is strongly absorbing in the near infrared (NIR) spectroscopy region. The spectral noise is then reduced, and compounds of small concentrations can be measured more accurately (8). DESIR has been reported to give good results in quantitative determining mono- and disaccharide solutions (9), free amino-N in beer (10), spores of pathogen fungus on wheat (11), and authentication of fruit juices (12). Thus, release of compounds in meat juice due to thawing (14-18) could be detected and used to differentiate unfrozen and previous frozen meat. The present preliminary study focuses on using diffuse NIR reflectance spectroscopy (19) directly on intact beef and DESIR on beef juices.

Experimental

M. longissimus dorsi with adhering bones from 6 Norwegian Red Cattle were excised from the carcasses 45 min *post mortem*. The beef was stored at 15°C for 24 h, and two cross section slices of 1.5 cm thickness were cut from each muscle. The rest was divided in two samples of 2 x 2 x 2 cm^3 cubes. The four samples from each muscle were slightly vacuum-packed in polyethylene bags, and then packed in polyamide bags. One slice and one sample of cubes were frozen and stored at -20°C for 24 h, and thawed at 4°C for 24 h. It was frozen again at -20°C for 72 h, thawed at 4°C for 48 h and analysed. This slow, repeated freezing was done to accelerate freezing damages in the beef. The second slice and sample of cubes were stored at 4°C for 48 h and analysed.

The intact beef slices were measured in 10 replicates with the light parallel to the muscle fibre direction. A NIR instrument scanning from 1100-2500 nm (InfraAlyzer 500, Braun & Luebbe GmbH, Germany) was used, as described by Hildrum *et al* (20).

DESIR was performed on juice from the cube samples. The drip juice was collected, and 0.5 ml was pipetted to the centre of glass microfibre filters (GF/A, Whatman International Ltd., England) of 55 mm diameter. Triplicate filters were dried for 2.5-4.5 minutes, with a distance of 37 mm under an infrared (IR) lamp (Siccatherm, 250 W, Osram, Germany). The lamp was connected to a gravimetric equipment (Thermo Control YTCO1L, Sartorius, Germany). Additional, triplicate filters were dried for 15 minutes at 60°C in a cabinet with air circulation (B1112V, Termax, Norway). DESIR was also performed, as described, on centrifuged meat juice. The cubes were centrifuged for 1.5 min at 745 x g (2900 rpm, type MP32, Braun AG, Germany). The filters were stored in exsiccator with silica gel until analysis. The DESIR measurements were done in 3 replicates from 400-2500 nm in a NIR spectrophotometer (Model 6500, NIRSystems Inc., USA). A Standard Sample Cup was used and rotated manually in 3 steps. The NIR reflectance spectra were recorded in 2 nm steps.

The DESIR data sets consisted of 1050 and the intact beef data set of 700 wavelength variables. The mean spectra of the 12 samples, expressed as apparent absorbance (log 1/reflectance), were used. The spectra were centred before the analyses. Partial least squares (PLS) regressions (21) were performed with a dependent dummy variable, 0 for unfrozen and 1 for frozen beef. Predicted, full crossvalidated values above 0.5 were assigned to frozen, and values below 0.5 to unfrozen beef. The computations were performed on The Unscrambler (Version 5.5, Camo, Norway).

Results and discussion

Table 1 shows the results of correct classifications into unfrozen or frozen and thawed beef. Two of the sample preparations gave 100 % correct classification. This indicates that NIR could be used to detect previous frozen meat.
NIR measurements of intact beef was the most rapid method tested, giving one result in a few minutes. Measuring centrifuged juice is more time consuming, but requires a less amount of material. Using drip juice is non-destructive and could be done within 5-7 minutes using the IR lamp. This equipment was the most rapid drying method. A cabinet with circulation of warm air is also reasonable, because this is a more common equipment. It is also rapid when several filters are dried together. Microwave oven

Table 1. The number of correct classified samples to unfrozen or frozen and thawed beef, from a total of 12 samples. In parenthesis are the numbers of PLS factors used.

DESIR				Intact beef
Air drying, 60°C		IR lamp		
Centrifuged	Drip	Centrifuged	Drip	
11 (10)	10 (4)	8 (2)	12 (4)	12 (6)

(8) or freeze drying (13) have not been recommended as drying methods for DESIR. Vacuum drying could be used, but is more time consuming, unless special equipment (13) is made for this.

In this study the results were obtained by few samples and a high number of factors. Therefore a further study is in progress, with a larger number of samples and larger variation among the samples. Preliminary results from this expanded study indicates good classification results.

Conclusion

This preliminary study indicated that near infrared spectroscopy might be used as a method for classification of beef into unfrozen or frozen and thawed.

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References

1. Paul P and Child A M, *Food Res* **2** 339-347 (1937).
2. Kaess G and Weidemann J F, *J Food Sci* **32** 14-19 (1967).
3. Isogai O, *Refrigeration* **47** 435-450 (1972).
4. Judge M, Aberle E, Forrest J, Hedrick H and Merkel R, *Principles of meat science*, Kendall/Hunt Publishing Company, Iowa (1988).
5. Daudin J D, *Technology of meat and meat products*, ed Girard J P. Ellis Horwood Limited, England, pp 7-31 (1992).
6. Alfaro G, Meurens M and Birth G S, *Appl Spectrosc* **44** 979-986 (1990).
7. Isaksson T, *CEC FLAIR/QUEST, Scientist Exchanges, 1991-1994*. ISBN: 0952345528, pp 4-5 (1994).
8. Meurens M, *NIR news* **3** (5) 12-13 (1992).
9. Alfaro G, Meurens M and Vanbelle M, *Proc Anal Appl Spectrosc Conf 2, Spectroscopy Across The Spectrum*, pp 53-58 (1991).
10. Chandley P, *J. Near Infrared Spectrosc.* **1** 133-139 (1993).
11. Asher M J C, Cowe I A, Thomas C E and Cuthbertson D C, *Plant Pathology* **31** 363-371 (1982).
12. Scotter C N G, Hall M N, Day L and Evans D G, *Proc 5th Int Near Infrared Spectrosc Conf*, Haugesund, eds Hildrum KI, Isaksson T, Naes T and Tandberg A, Ellis Horwood, New York, pp 309-314 (1992).
13. Scotter C N G and Day L Z, *Proc 4th Int Near Infrared Spectrosc Conf*, Aberdeen 1991, eds Murray I and Cowe I A, VCH, Weinheim, pp 394-398 (1992).
14. Tappel A L, *Cryobiology*, ed. Meryman H T, Academic Press, London, pp 163-177 (1966).
15. Hamm R and Körmendy L, *J Food Sci* **34** 452-457 (1969).
16. Calvelo A, *Developments in meat science - 2*, ed Lawrie R, Applied Sci. Publ., London, pp 125-158 (1981).
17. Hamm R and Gottesmann P, *Proc of the 28th Eur Meet Meat Res Workers*, Madrid, vol 1, pp 152-155 (1982).
18. Hamm R and El-Badawi A A, *Fleischwirtsch* **64** 471-473 (1984).
19. Hildrum K I, Ellekjær MR and Isaksson T, *Meat Focus International*, April, pp 156-160 (1995).
20. Hildrum K I, Nilsen B N, Mielnik M and Næs T, *Meat Sci* **38** 67-80 (1994).
21. Martens H and Naes T, *Multivariate calibration*, John Wiley & Sons, New York (1991).