Near Infrared Spectroscopy in the Food Industry: a Tool for Quality Management

Teagasc
AGRICULTURE AND FOOD DEVELOPMENT AUTHORITY

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NEAR INFRARED SPECTROSCOPY

IN THE FOOD INDUSTRY:

A TOOL FOR QUALITY

MANAGEMENT

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SUMMARY

Near infrared (NIR) spectroscopy is a rapid, non-destructive analytical technique which has been used in the food and agriculture industries for almost 20 years. Ireland was one of the first countries in the world to adopt this method for national trading purposes and the grain trade has used it for off-farm and in-process analysis since 1981. However, other sectors have been slower to realise its potential and as part of a process of demonstrating the role which it may play in monitoring quality in a range of food industry applications, a programme of research and development has been on-going within Teagasc and its predecessor An Foras Talúntais.

NIR spectroscopy provides the food processor with information. This information may describe how much of a given substance is present in a mixture or how the overall quality of the substance compares to a reference material e.g. a previous batch of raw material, finished goods or a competitor's product. This report provides some examples of pre-competitive R&D on representative qualitative and quantitative problems in a range of foods and food ingredients.

The use of NIR spectra collected within 24 hours of slaughter to predict beef tenderness 14 days later shows considerable promise. Non-destructive monitoring of flesh composition in farmed salmon has paved the way for the efficient use of expensive feed materials while the content of each species in binary mixtures of minced beef and lamb has been accurate enough to suggest the use of NIR spectroscopy as a rapid screening tool by regulatory agencies, food processors and retailers.

Classification of a range of food ingredients (including skim milk powder and flour) into one of a number of functionally-discrete categories has been successfully achieved with levels of accuracy high enough to warrant immediate industry utilisation i.e. greater than 90% for skim milk powders and 97% in the case of flour. Species confirmation in a number of raw minced meats (chicken, turkey, pork, beef and lamb) has been achieved with over 90% accuracy in feasibility studies.

Calibrations transferred from one NIR instrument to another lose accuracy because of differences in instrument construction, sample presentation and other factors. A research effort has recently been applied to this problem of transferability and results are available for both scanning and fixed filter instruments. The success achieved opens the way for using NIR results obtained in different companies or countries as an uncontested basis for trade.

INTRODUCTION

The industrial environment

The Irish food industry operates in an increasingly competitive international business environment. Thus, food processors require faster and more complete information about raw materials and their products at all stages of production. Raw material or finished goods quality has traditionally been assessed by determining the concentration of key constituents; while this indirect approach still has merit, the direct measurement of quality is a goal to which processors aspire. Using the latter approach, comparisons within and between different product batches may be made to confirm consistency and to ensure that material with the correct functional properties is delivered to the appropriate end-user. Finally, there is the issue of authenticity. In a climate of ever more regulation, food processors are required to take all reasonable steps to ensure that the composition of their product is as described on the label. Thus, meat described as fresh should not have been frozen, wild salmon should not be from a fish farm and minced beef should not contain meat from any other species.

To address all these issues in real time, a rapid analytical tool is required. Near infrared spectroscopy possesses the necessary speed, ease of use and robustness to be able to generate critical information either on- or off-line; output from NIR instruments may be included in feedback loops for alteration of ingredient incorporation levels or used at a weighbridge to control product release. This report highlights some of the R&D work which has been performed locally so as to provide an overview of the type of applications for which the technique is suitable.

Near infrared spectroscopy

Among reasons which may explain the suitability of NIR for food analysis may be mentioned its ease of operation, the requirement for only minimal (or even no) sample preparation, and the ability to analyse material which contains significant amounts of water

The near infra-red spectral region is normally understood as encompassing the 1100-2500nm range; of recent times, attention has also focused on the 750-1100nm region which has been variously described as the near near infra-red (NNIR) or the Herschel infra-red (Davies, 1990). Absorptions in these regions arise from overtones and/or combination bands of fundamental absorptions of bonds involving carbon (C), hydrogen (H), oxygen (O) or nitrogen (N). Bonds of this type are common in organic material and the resulting multiplicity of bands gives rise to the typical, relatively ill-defined near infrared spectra shown in Fig. 1. A further consequence of this number of bond types is that no single wavelength may be identified to which only one component in a mixture responds. In practice this means that measurements have to be taken at a number of wavelengths to perform quantitative and qualitative analyses.

A characteristic of the practical application of NIR spectroscopy has always been the wide range of sample presentation options available. Sample accessories are available to handle liquids, slurries, solids, films, wholegrains and whole fibrous material; together with the use of fibre optic accessories (single fibres or fibre bundles), remote reflectance, interactance immersion and surface interactance probes allow almost any type of sample to be conveniently and reproducibly handled.
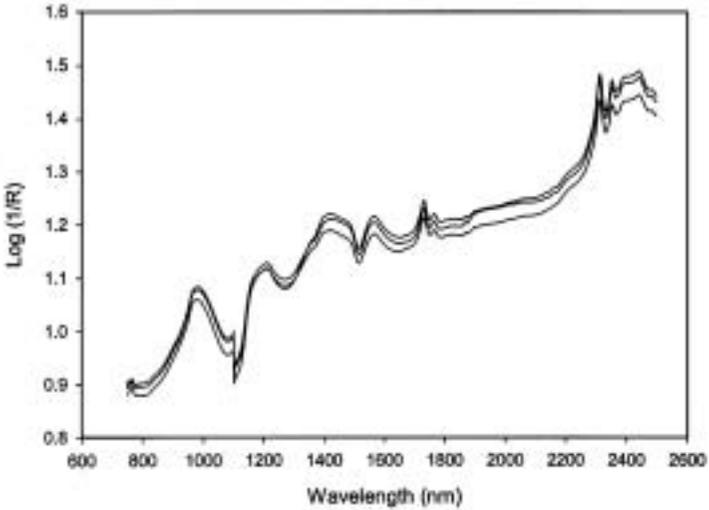
Figure 1. NIR reflectance spectra of raw minced meat samples (chicken, turkey & pork) showing the characteristic sloping baseline and paucity of fine structure

Figure 2. Using a scanning NIR spectrophotometer for direct analysis of intact salmon flesh

Chemometrics

Given the overlapping nature of NIR spectra referred to above, multivariate statistical techniques (chemometrics) are required for the development of calibrations which are then used for the analysis of new samples. These techniques may be used to predict constituent concentration or to compare the spectrum of an unknown to a spectral library of e.g. previously-manufactured products of the desired quality. The main method used for quantitative work is partial least squares regression while a number of methods may be used for discrimination e.g. factorial discriminant analysis, K-nearest neighbours analysis etc. All of these techniques reduce the size of spectral datasets significantly but they also treat them in various ways which enable the extraction of any underlying similarities or differences between materials. A preliminary step called principal component analysis is often applied to datasets prior to discrimination. This procedure modifies the original wavelength data so that all of the information contained in the original spectrum is contained in say 20 new axes or dimensions. Each sample spectrum may then be represented as a single point (score) on each of these new axes. (For a fuller description of the application of these techniques, the reader is referred to Downey, 1994.)

Partial least squares regression uses information from the entire spectrum to predict composition but emphasises wavelengths which exhibit high correlations with the chemical data in the learning sample set.

Discrimination between different functional classes of a given food ingredient and confirmation of the authenticity of another are basically the same analytical problem. What is involved is the comparison of a test material to a set of values established as typical for the reference material. The basic assumption behind the application of spectroscopy to this problem lies with the generation of a "fingerprint" of foods. Given that an individual food ingredient has a characteristic chemical composition, it will also have a characteristic spectrum since this is the result of the absorption of radiation by many (or all) of its chemical constituents. Because the exact composition of any naturally-occurring material will vary somewhat depending on variety, growing location, season etc., there exists a range of

typical spectra for this material. In principle, therefore, what is needed is a library of representative spectra to which the spectrum of a test material may be compared in order to establish its quality or authenticity. Statistical techniques use for this purpose try to group similar materials close together in tight clusters separated from other dissimilar samples e.g. fresh meat and frozen-then-thawed meat.

Transfer of calibrations

Multi-site implementation of NIR in large companies is fast becoming the norm. This means that a calibration developed for a given product at one location should be capable of transfer to instruments at other sites without any significant loss of accuracy. Despite the low manufacturing tolerances used by instrument manufacturers, loss of accuracy has always been a feature

Figure 3. Collecting near infrared spectra of meat samples for confirmation of freshness

of such calibration transfer with this and other spectroscopic techniques. To overcome this practical limitation, a number of mathematical solutions have been reported and work at The National Food Centre on one such option has demonstrated its potential when transfer takes place either between sophisticated monochromator instruments only or between these and simpler fixed filter equipment.

APPLICATIONS

The applications described in this report are representative of a range of practical problems to which NIR has been applied and have been selected to demonstrate the type of physical samples which may be analysed by this procedure.

Quantitative measurements

BEEF TENDERNESS PREDICTION

Perhaps the major challenge facing the Irish beef industry is the consistent production of tender meat. In parallel with research to realise this goal through husbandry practices or technological intervention, tools are required to determine the texture of beef post-slaughter and, perhaps more importantly, to enable the prediction of eventual (14 day) beef tenderness by measurements taken soon after slaughter. As part of one Structural Funds research project (Project ARMIS Number 4248), near infrared spectra were recorded from raw *longissimus dorsi* muscle from 70 heifers at 24 hours, 48 hours, 7 days and 14 days after slaughter. These spectra were used to try to predict established physical (Warner-Bratzler shear force) and organoleptic quality indicators as assessed by sensory analysis (tenderness, texture, flavour and acceptability) after cooking. Spectra were collected from meat pieces by placing a fibre optic probe directly on the surface of the meat; a measurement was made on each side of the meat with the average being stored for statistical analysis. Promising results were achieved for the prediction of Warner-Bratzler shear force (instrumental measurement), tenderness and texture (sensory analysis); correlation coefficients and standard errors of prediction for these parameters are shown in Table 1. These results suggest that NIR measurements on raw meat may be able to predict certain of these parameters with sufficient accuracy for rapid screening of meat samples.

Of perhaps greater interest are the results obtained when spectra collected from meat pieces at various times after slaughter were used to predict these instrumental and sensory properties in longissimus dorsi samples taken from storage after 14 days. Results obtained are shown in Table 2 and reveal the potential of this approach for beef quality prediction. It appears that spectra collected as soon as 24 hours post-slaughter may be able to predict the eventual textural properties of cooked meat after 14 days storage with a level of accuracy sufficient at least for early screening purposes. While the actual levels of accuracy reported may not be sufficient for immediate implementation at factory level, they do suggest the possibilities of this spectroscopic sensor and lay the basis for a further expanded study.

Table 1. Prediction of quality attributes of beef striploins by NIR spectroscopy

Quality attribute	R[1]	SEP[2]	Range[3]	n[4]
WBSF	0.61	2.10	2.4 - 15.3	229
Tenderness	0.53	0.88	2.1 - 7.4	201
Texture	0.54	0.45	1.9 - 4.6	201
Acceptability	0.42	0.56	1.8 - 5.0	201

[1] correlation coefficient   [2] standard error of prediction

[3] range in laboratory results   [4] number of samples

Table 2. Prediction of 14 day quality attributes of beef striploins by spectra collected at various times post-slaughter

Time of spectral collection	Quality attribute	R[1]	SEP[2]	n[3]
24 hour	WBSF[4]	0.69	0.86	70
	Tenderness	0.50	0.62	70
	Texture	0.50	0.31	70
	Acceptability	0.54	0.43	70
48 hour	WBSF	0.59	0.96	70
	Tenderness	0.35	0.66	70
	Texture	0.37	0.33	70
	Acceptability	0.35	0.48	70
7 day	WBSF	0.75	0.82	57
	Tenderness	0.60	0.57	57
	Texture	0.49	0.32	57
	Acceptability	0.46	0.47	57
14 day	WBSF	0.73	0.82	70
	Tenderness	0.49	0.62	70
	Texture	0.60	0.29	70
	Acceptability	0.56	0.43	70

[1]correlation coefficient   [2] standard error of prediction

[3] number of samples   [4] Warner Bratzler shear force

FARMED FISH CARCASS COMPOSITION

Salmon has always been considered as a high-value gourmet fish. Traditionally, the market for this commodity has been supplied by wild fish but now the practice of fish farming has developed as a means of increasing sales and supplying the market at times of the year when wild fish are not normally available.

Flesh composition is of major importance from the processor's perspective. In smoking operations, high flesh oil contents lead to gaping and unpleasant mouthfeel while low oil levels diminish the uptake of smoke flavour. In farming situations, feed formulation and usage rate are the principal regulators of flesh composition. Feed is also the single largest cost input into a commercial salmon farming operation. Thus, the drive to optimise fish oil content arises from the need to meet processor and consumer requirements as well as from considerations of cost-control and, ultimately, profitability.

NIR spectroscopy was investigated as a tool for the routine analysis of fish flesh composition in a rapid and cost-effective manner; funding for this work was obtained under the Operational Programme for Rural Development. Moisture and fat were the proximate constituents selected for investigation because of their pre-eminent importance in salmon processing and marketing. Given the irregular shape and dimensions of the fish, the most practical option for spectral collection was the use of a fibre optic probe. In order to maximise the relevance of this work to commercial enterprises, spectra were collected through the skin and scales of whole, gutted fish.

Several sites were selected and marked on each side of every salmon purchased; normally there were six along the dorsal surface of each side and six along the ventral surface. Sites were approximately equally spaced along each surface and were slightly larger than the surface area of the fibre optic probe head. Spectra were recorded at each site by simply placing the fibre optic probe on the fish carcass (Fig. 2); when the entire set of spectra for each salmon had been acquired, the sites were excised and stored at $-20^{0}C$ prior to chemical analysis by reference methods. Two hundred and ninety four (294) salmon sites were treated in this manner; fish samples were selected to cover a wide variation in fish maturity. Equal numbers of dorsal

and ventral samples are represented in the above dataset and calibrations were separately developed for these locations. Predicted compositional results are shown in Table 3.

Table 3. Prediction of oil and moisture in salmon carcasses by NIR spectroscopy

Constituent	Location	R[1]	SEP[2]	Range[3]
Oil	Dorsal	0.84	2.04	3.1 - 16.7
	Ventral	0.86	2.41	3.3 - 22.8
Moisture	Dorsal	0.83	1.45	62.1 - 73.9
	Ventral	0.88	1.90	57.9 - 73.5

[1] correlation coefficient [2] standard error of prediction [3] range in chemical values

Accuracy levels such as those obtained in the present work should have significant practical possibilities in the salmon-rearing industry. While they may not be appropriate for fine control of fish fat levels, it will be possible by their use to detect fish with very high or very low oil contents. This may permit better consistency of product through the removal of carcasses with extreme compositions. The technique should also be valuable for charting body composition during growth since in principle the measurement could be made on stunned fish which may then be returned to the farm cage unharmed. In this way, expensive feed use may be monitored and its efficiency maximised.

MEAT SPECIES QUANTIFICATION IN RAW MINCED BLENDS

Meat authenticity is of significant concern to consumer and retailer alike (Lumley, 1996). An authentic meat may be defined as one which is what it purports to be and in this regard meat species is an important issue. Partial substitution of an expensive meat with one of lower cost or quality is an economic fraud on the processor and consumer and may pose religious (e.g. pork) or perceived health (bovine spongiform encephalitis) problems. While speciation is not problematic when meat is examined in large pieces, after mincing it becomes difficult to establish species without sophisticated analytical procedures. The potential of NIR spectroscopy to solve this problem was investigated in work supported by Structural Funds (Project ARMIS Number 4248).

Thirty-two beef (*m. semimembranosus*) and 33 lamb (*m. longissimus dorsi*) samples were purchased from local retail outlets. After removal of all sub-cutaneous fat, samples were minced. In addition to these 100% beef and 100% lamb samples, a series of mixed species samples was prepared containing 5% lamb:95% beef, 10% lamb:90% beef and 20% lamb:80% beef (w/w). Once minced, samples were placed in polyethylene bags and scanned (Fig. 3).

The most accurate calibrations obtained predicted lamb content over the range 0 - 100% with a standard error equal to 6.71% (w/w) and with a prediction error of 1.1% over the range 0 - 25%. The regression graph for the latter calibration may be seen in Fig. 4.

These results strongly demonstrate the potential for NIR spectroscopy to act as a screening tool for the examination of minced meat samples when admixture is suspected.
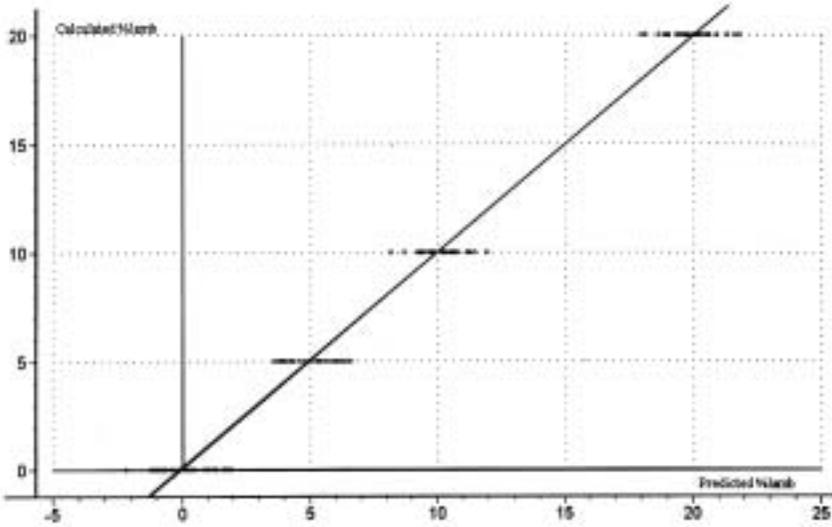
Figure 4. Prediction of percentage lamb in beef + lamb mixtures using NIR spectra in the 1100 - 2498 wavelength range

Qualitative measurements

CLASSIFICATION OF MILK POWDER GRADES

Skim milk powder is a major food ingredient produced by the dairy industry. Powders may be manufactured by the use of a high-, medium- or low-heat pre-treatment of milk to produce three types of product which vary significantly in a number of important functional properties. Methods currently used by the dairy industry to confirm the heat classification of skim milk powders are time-consuming, involving estimation of undenatured whey protein by turbidimetry and dye-binding techniques. A more rapid and inexpensive procedure would facilitate the required level of assurance at reduced cost.

Sixty-six samples of commercially-produced milk powders were obtained from a number of creameries in Ireland: 21 of these were classified by their creamery of origin as high-heat, 25 as medium-heat and 20 as low-heat powders. All samples in each group were produced in a single season and came from more than one creamery.

In order to facilitate discrimination between the three groups of milk powders, a new variable called "class" was created; low-heat (L) samples were arbitrarily ascribed a value of 1, medium-heat (M) samples a value of 2 and high-heat (H) samples the value 3.

The spectral dataset was initially subjected to a principal component analysis but this was unable to effectively separate the three milk powder groups. Following the application of factorial d iscriminant analysis, optimum separation was achieved by a model using 10 principal components. All 34 calibration samples were classified correctly as were 91% of those (n = 32) in the prediction set. Of these latter, all 10 of class 3 (H) and all 9 of class 1 (L) were correctly classified; three of the 13 samples in class 2 (M) were incorrectly classified as belonging to class 3 (Table 4). The separation achieved may be seen in Fig. 5 in which the incorrectly classified samples are clearly marked.

Thus, discriminant function 1 separated the samples in class 1 (L) from the others, while function 2 effected the discrimination between classes 2(M) and 3(H). Some mis-classification of medium heat samples is not unexpected in any three-way classification system; in this case, particular difficulties arise from the somewhat arbitrary definition of the heat-treatment classes, with certain processing plants further dividing the medium group into medium-high and medium-low subgroups. In such cases, classification difficulties may also be experienced with the existing chemical procedures.

Table 4. Predicted classification of milk powder samples using NIR spectroscopy*

		L	M	H
Calibration development sample set	L	11	0	0
	M	0	12	0
	H	0	0	11
Calibration evaluation sample set	L	9	0	0
	M	0	10	3
	H	0	0	10

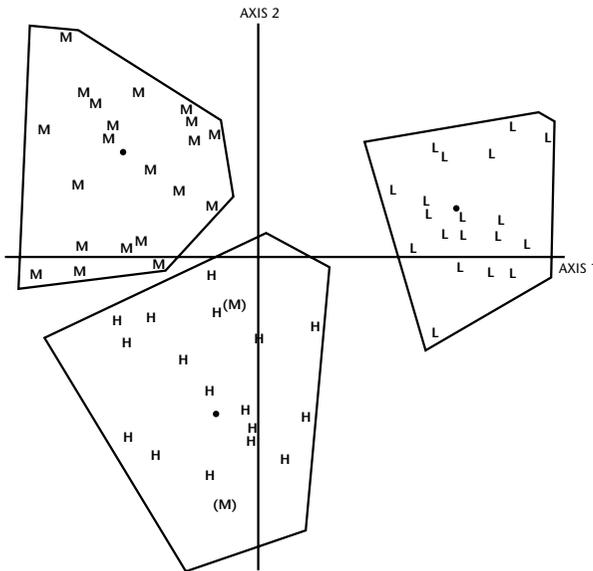* row: actual powder classification; column: classification by NIR model

Figure 5. Discriminant map of commercial skim milk powders using NIR spectroscopy. Samples in parentheses are wrongly classified; (●) represents the cluster centre

The main wavelengths contributing to the two calculated discriminant functions were identified. They indicated that differences in the structure of protein and in the quantity and state of water in powders from the three heat-treatment classes formed the basis for the discrimination achieved.

SEGREGATION OF FLOUR GRADES

Flour mills produce a range of white flours which differ significantly in their functional properties. For example, flours suitable for bread manufacture require a high content of good quality protein while material destined for cake manufacture needs a lower protein content and a smaller average particle size. Low batch-to-batch variation in each of these flour types is required by the plant bakery industry; because of rationalisation and automation, production lines tend to be run continuously with bakers reluctant to alter baking conditions to meet between-batch variations in flour quality. In addition, accidental delivery of the wrong flour type to an industrial bakery can have significant financial implications because of the need to retrieve the flour from a customer's premises, clean the flour silo and pipework, replace the flour with a correct delivery and provide compensation for loss of production. It is therefore desirable that flour mills are in a position to confirm the identity and quality of each consignment of flour before it leaves the mill premises.

Two hundred flour samples were collected over several months from a single milling company. Six different types of white wheatflour were sampled, namely biscuit, self-raising, household, cake, baker's and soda bread mix. Spectra of the entire flour collection were subjected to a principal component analysis after which the sample scores were plotted for visual examination. The results obtained were encouraging especially when components 1 & 4 were plotted as abscissa and ordinate respectively (Fig. 6); in this case, baker's (B), hi-ratio (H), soda-bread mix (M) & biscuit (T) flours seemed to be located into discrete clusters. This plot shows overlap between self-raising (S) and retail (C) flour samples. Four samples were located in inappropriate groups on this plot – 3 retail and 1 self-raising flour.
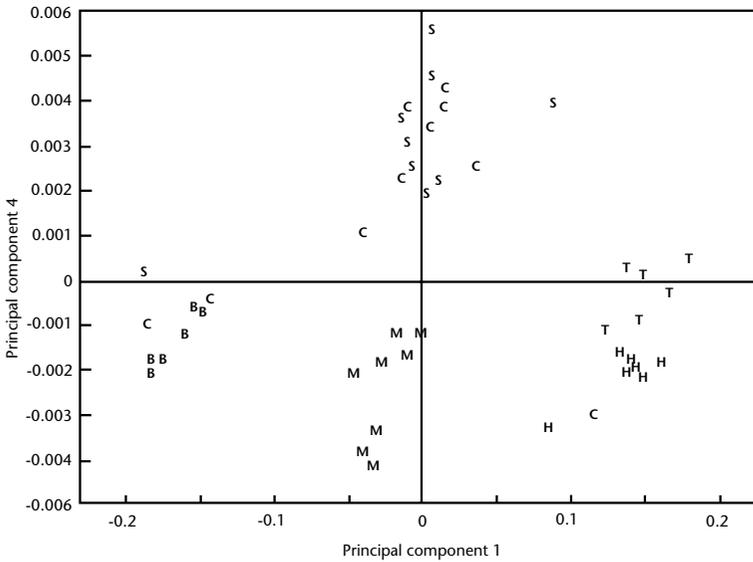
Figure 6. Principal component scores plot for six types of white flour segregated by NIR spectroscopy

Analysis of the protein content of these samples revealed two of the retail flours to contain 12.0 and 11.7% protein - these are values typical of baker's flour while retail flours should contain approximately 8.5% protein. The samples were located in the baker's flour cluster in the scores plot and were therefore removed from the collection on the basis of probable incorrect identification or formulation by the mill. This highlights the ability of the combination of NIR spectroscopy and multivariate statistical analysis to detect unusual sample material without recourse to chemical or other analytical procedures. Analysis of the two remaining samples for protein content produced values within the normal range for these flours; a normal level of raising agent was detected in the self-raising flour. Because no reason to the contrary could be discovered, these samples were retained in the sample collection.

Factorial discriminant models were developed using 36 samples as a calibration set. The optimum model used only 8 components and correctly identified all of the calibration flours. Out of a total of 99 prediction samples, 92 were correctly classified (93%). Following an examination of the main wavelengths contributing to the discrimination models, the basis for this discrimination was identified as differences in particle size and additive incorporation level between the flour types. This interpretation is in accordance with the milling protocol and additive blending procedures in commercial flour mills.

IDENTIFICATION OF MEAT SPECIES

With the ever-increasing consumption of pre-processed foods in Ireland, concern over the issue of food authenticity is rising. In the case of meat and meat products, a number of specific issues arise. These include the partial substitution of meat from a high-value species by meat of a lower value, the extension of meat products using fat or rind in excess of levels normally associated with the particular meat type and the addition of other non-meat proteins or water. An additional factor with regard specifically to pork is its avoidance for religious reasons. In order to guard against fraud or mis-representation, an ability to determine meat species is therefore required by food processors and regulatory agencies alike.

Reports of the ability of NIR spectroscopy to discriminate between fresh and previously-frozen beef have been published (Downey and Beauchêne, 1997a & b) and it was judged appropriate to perform a feasibility study into its potential for distinguishing between visually-similar raw minced meat from a small number of species i.e. pork, chicken and turkey. In this particular study, the use of spectral data in the visible wavelength range in combination with NIR readings was also investigated.

Samples of chicken breast (n= 24), turkey breast (n = 25) and pork loin chops (n = 25) were purchased at a number of local retail outlets over a period of several weeks and stored overnight at 4⁰C. Principal component analysis alone was unable to discriminate between the three meat types; although pork samples were separated from the poultry meats, considerable

overlap was found between chicken and turkey samples. Factorial discriminant models were developed using spectral data in the wavelength ranges 400 - 750nm, 400 - 1100nm, 1100 - 2498nm and 400 - 2498nm respectively. The best results were obtained using the 400 - 1100nm range, with 7 principal components being optimal. All of the calibration samples were correctly classified while a success rate of 91.9% (34 of 37 samples) in the validation set was obtained.

Figure 7. Discriminant scores plot for minced raw meat samples (C = chicken; P = pork; T = turkey) revealing segregation by NIR spectroscopy according to species.

A two-dimensional scores plot for this discriminant model reveals that all of the pork samples were separated while some overlap was apparent between chicken and turkey samples (Fig. 7). Discrimination between pork and poultry is principally achieved along the first axis; the main features

accounting for this separation belong to water, myoglobin and oxymyoglobin. Thus, the pigments/water ratio may be important in the observed discrimination. The second discriminant function separates chicken from turkey; spectral features of importance are again related to myoglobin, oxymyoglobin and traces of erythrocyte haemoglobin. This work has now been extended to include beef and lamb meats. The results obtained for this expanded work support the above findings and suggest the suitability of this approach for confirmation or refutation of the accuracy of a labelled raw minced meat product.

Transfer of calibrations

The initial calibration process for NIR instruments is time-consuming and expensive. In order to maximise the value of this work, companies often seek to develop a single calibration on one instrument and transfer it to a number of others either on the same or in different sites; transfer between trading organisations in separate countries is becoming increasingly demanded. When calibrations are transferred in this manner, significant additional errors arise from a number of sources e.g. differences in instrument manufacture, operator performance, environmental considerations. Until recently, the only way to reduce these extra errors involved either a separate calibration exercise for each instrument or minor modifications to the actual calibration. Both these methods had disadvantages; the first was expensive while the second involved continual adjustments over time.

An alternative approach has been developed in recent years. This involves a mathematical procedure in which the spectral output of each subsidiary or slave instrument is adjusted to be identical to that recorded for the same samples on a pre-selected controlling instrument on which all calibration development work has been performed - a so-called master instrument. The output from the mathematical exercise is a standardisation file which is specific for each instrument. After standardisation, the raw spectra recorded on an instrument may be modified automatically to be identical to spectra of the same samples scanned on the master. Thus, when samples of milk

powder are analysed for moisture content by an instrument in the USA using a calibration developed on the American instrument, those samples provide the same moisture reading when analysed by a standardised instrument in Ireland using the same calibration.

In principle this procedure may be applied to different instrument types (i.e. scanning or fixed-filter instruments) and indeed to models from different manufacturers. No published information regarding its application to anything other than a single instrument model from one manufacturer is however available and a research project was funded by the European Union to verify that widespread transfer was indeed achievable.

STANDARDISATION PROTOCOL

Sixty-five specially-prepared samples sealed in air-tight cells were distributed sequentially to every participant in the project, 16 in all. Duplicate spectral readings on each of these samples were recorded under controlled conditions in participants' laboratories and forwarded to the master instrument laboratory in Belgium for processing. After statistical analysis to ensure that each instrument was performing according to specification, these spectral readings were used to identify the differences between each instrument and the master monochromator. In the final standardisation step, a file was prepared to modify the slave instrument spectra to make them identical to those of the master using both a proprietary and a public domain software package. Results reported below are those for the proprietary package.

In order to demonstrate the effectiveness of the standardisation process, two other pieces of work were undertaken. In the first, European calibrations to predict protein and moisture in ground wheat & barley and oil in maize were developed at The National Food Centre. These were produced using a collection of almost 1200 grain samples from the 1994 and 1995 harvests collected in the main European producing countries. The following low standard errors of prediction were obtained:- wheat protein - 0.19, barley protein - 0.27 and maize oil - 0.33. The second task involved circulating 30 validation samples of each grain type to each participant with an instruction

that they analyse these using the European equations before and after their instruments were standardised. In this way, it would be possible to demonstrate the effect of such standardisation on prediction accuracy.

TRANSFER BETWEEN MONOCHROMATOR INSTRUMENTS

A summary of the prediction errors obtained when the same set of ground grain samples were analysed on 8 different standardised monochromator instruments is shown in Table 5. Each instrument was located in a different country and used the same European calibration for each cereal. (Two instruments developed faults during the project and were unable to provide appropriate data.) What is notable about these results is their consistency from instrument to instrument, particularly in the case of barley. Maize data were complicated by some changes in moisture content observed during the experimental work. However, these results clearly show that this standardisation will permit the utilisation of a single calibration in separate instruments to produce virtually identical results.

Table 5. Ground cereal analysis by standardised NIR monochromator instruments

Instrument No.	Wheat protein	Barley protein	Maize oil
	SEP[1]	SEP	SEP
1	0.15	0.28	0.16
2	0.17	0.26	0.25
3	0.18	0.29	0.17
4	0.24	0.28	0.33
5	0.17	0.27	0.17
6	0.16	0.27	0.31
7	0.21	0.33	0.22
8	0.28	0.28	0.2

[1]SEP = standard error of prediction

TRANSFER BETWEEN MONOCHROMATOR AND FIXED-FILTER INSTRUMENTS

Data revealing the predictive performance of 6 standardised fixed-filter instruments are shown in Table 6. What is most apparent from this table is that even after standardisation, fixed-filter instruments produced large and unacceptable errors from one instrument to the next. It seems therefore that for these instrument types, standardisation alone is incapable of removing all of the sources of variation between instruments and that continual calibration adjustment may be required for them. These variations are most likely to arise from instrument design and construction factors and may therefore not be overcome by any data treatment procedure.

Table 6. Ground cereal analysis by standardised NIR filter instruments

Instrument No.	Wheat protein	Barley protein	Maize oil
	SEP[1]	SEP	SEP
9	0.38	0.44	0.25
10	N/A	0.47	N/A
11	0.36	0.55	0.53
12	0.87	0.75	0.34
13	0.43	0.61	1.37
14	1.98	0.39	1.09

[1]SEP = standard error of prediction : N/A = data not available

CONCLUSIONS

- NIR spectroscopy provides the food processor with information. This information may describe how much of a given substance is present in a mixture or how the overall quality of the substance compares to a reference material. Examples contained in this report reveal the potential of the technique to answer representative quality questions faced by the food processing industry. NIR technology may be used in off-line, near-line or on-line situations

- The use of NIR spectra collected within 24 hours of slaughter to predict beef tenderness after 14 days shows considerable promise for commercial applications.

- Non-destructive monitoring of moisture and oil levels in farmed salmon has paved the way for the more efficient use of expensive feed materials.

- Prediction of the content of each species in binary mixtures of minced beef and lamb has been accurate enough to suggest the use of NIR spectroscopy as a rapid screening tool by regulatory agencies, food processors and retailers.

- Classification of a range of food ingredients (including skim milk powder and flour) on the basis of quality has been successfully achieved with levels of accuracy high enough to warrant immediate industry utilisation i.e. over 90% for skim milk powders and 97% in the case of flour.

- Species identification in a range of raw minced meats (chicken, turkey, pork, beef and lamb) has been achieved with over 90% accuracy in feasibility studies.

- European calibrations for protein in wheat & barley and oil in maize have been developed. Their transfer between monochromator instruments located in eight different countries has been successfully demonstrated. This achievement opens the possibility of using NIR results in different companies or countries as an uncontested basis for trade.

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