Feasibility study of measuring cheese through the plastic of the packaging using a fiber optic probe with FT instrument - transferring predicted values from a dispersive monochromator instrument

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Introduction

Nowadays, in more and more countries, weight intake has become one of the major public health issues. Indeed, obesity is responsible for many diseases and increases the risk of early mortality. The obesity rate, which is linked to the quantity of fat consumption, could be reduced if a more clear and reliable information was available for the consumers. For this reason, one important point concerns the quality of the information displayed on the packaging of the agro-food products regarding their content, which becomes a major issue, not only from a legal point of view, but also in terms of public health and security. Regulations require a complete and clear labeling of all the food products. For this reason, having rapid, easy and non destructive measurement techniques for producers and inspection authorities becomes a priority in order to avoid fraud and supply consumers with reliable information.

Important fat based products need to be correctly labeled. Products like cheese have an average annual consumption by inhabitant in Europe of 17.77 kg (in 2010). The diversity of cheeses found in the market shows the importance of this kind of products in the EU dairy food diet.

The aim of this work is to show that NIR spectroscopy coupled with a fiber optic probe can be particularly well suited to quantify important characteristic properties of cheese as protein, fat and moisture contents. The novelty is that the measurements are directly performed on samples throughout the plastic of the packaging and using models developed using only few samples with reference values.

Materials and methods

In this study, 163 samples of “hard paste” cheeses were purchased in different supermarkets in Belgium. These samples were divided into two groups: one group of 35 samples and another one of 128 samples. The group of 35 samples was analyzed by reference methods for fat, protein and dry matter. The reference method used was Rose Gottlieb for the determination of the fat content, Kjeldahl for protein and the steaming at 102°C for dry matter.

Nineteen samples were used to build the models, whereas that the rest (16 samples) were used as a validation set for all the calibration models developed. This validation set was chosen to cover all the chemical range for the three parameters (protein, fat and dry matter) and to cover the whole spectral dispersion range. For the 128 remaining samples, just the spectra were collected and no reference values were available. Due to the limited number of samples available, Multiple Linear Regression (MLR) models were developed.

Results and discussion

Three different approaches were tested in this work. The first approach consisted to measure all the samples of cheese with a Matrix F from Bruker equipped with a solid probe directly throughout the plastic of the packaging. Each cheese was measured 10 times and the averaged spectrum was used. To avoid over-fitting, the number of wavelengths used was limited.

The results obtained by this first approach are shown in Table 1. Regarding to SEC and RPD the calibrations seem to be of good quality but the predictions on an independent set of validation show a lack of accuracy. The performances are around two times worse on the validation set than on the calibration set. Probably the quality of the spectrum measured with a solid probe and the influence of the plastic explain, partially, these results. The results obtained by this first approach were, then, not enough satisfactory.

Table 1: Results of MLR calibrations on the Matrix F

MLR _MATRIX	Calibration						Validation						
	N	SD	R^2	SEC	RPD	λ	N	SD	R^2	SEP	Bias	SEPC	RPD
Protein	19	3.39	0.84	1.32	2.57	3	16	2.91	0.48	2.56	-0.65	2.45	1.14
Fat	19	6.11	0.96	1.21	5.05	3	16	5.40	0.90	1.96	0.99	1.74	2.76
Dry matter	19	5.26	0.92	1.49	3.53	2	16	4.73	0.79	2.61	1.53	2.19	1.82

The second approach consisted on measuring all the samples (163) without the plastic on a Foss XDS instrument. This instrument was used due to its better acquisition spectral characteristics compared to a solid probe (less noise in the region 2300-2500 nm). Other advantages are the larger area of samples scanned and the possibility to measure in ring cups. As for the Matrix F instrument, MLR calibrations were performed with the same reduced (19) set of samples. In that case, the results obtained on the validation set (16) were very good as indicated in Table 2.

Table 2: Results of MLR calibration on XDS

MLR _XDS	Calibration						Validation						
	N	SD	R^2	SEC	RPD	λ	N	SD	R^2	SEP	Bias	SEPC	RPD
Protein	19	3.39	0.96	0.70	4.84	3	16	2.91	0.93	0.82	-0.35	0.77	3.54
Fat	19	6.11	0.99	0.67	9.12	4	16	5.40	0.98	0.71	-0.08	0.73	7.57
Dry matter	19	5.26	0.97	0.89	5.91	1	16	4.73	0.95	1.06	0.01	1.10	4.47

The third and last approach consisted of using the MLR models validated from the Foss XDS to predict the content of the 128 samples of cheese for which no reference values were available. These predicted values, combined with the reference values available for the 19 samples of the calibration set are grouped together as a complete 'reference data' library. The next step consisted to associate the spectra of all the samples measured with the Matrix F instrument through the plastic and that library. This step was used then, to develop a complete PLS model, which has been validated using the 16 samples from the validation set as indicated in table 3.As it can be observed, the results obtained by this technique are satisfactory because it allows an estimation of the content of the three parameters with reasonable error.

Table 3: Results of PLS calibrations on the Matrix F

PLS_MATRIX	Calibration						Validation						
	N*	SD	R²	SECV	RPD	Terms	N	SD	R²	SEP	Bias	SEPC	RPD
Protein	143	3.30	0.92	0.91	3.63	11	16	2.91	0.93	0.97	-0.23	0.97	2.99
Fat	143	5.01	0.92	1.45	3.46	5	16	5.40	0.96	1.27	0.77	1.04	4.25
Dry matter	142	4.91	0.91	1.43	3.43	5	16	4.73	0.93	1.45	0.82	1.25	3.26

Conclusion

As shown in this study, it is not necessary to have many samples with reference values determined by wet chemistry to develop accurate PLS calibrations. Measurements by optical solid probe seem to be a good method to control the information mentioned on the label of the packaging as they allow to measure directly throughout the plastic.

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