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CAN NEAR INFRARED SPECTROSCOPY BE APPLIED IN THE INDUSTRIAL SETTING TO VERIFY THE NUTRITION DECLARATION OF PROCESSED FOODS? – STUDY OF SELECTED POLISH DELI PRODUCTS

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ABSTRACT

Background. The aim of this study is to evaluate near infrared spectroscopy (NIR) for the verification of nutrition declaration on food labels of selected Polish deli products. The accurate declaration of nutritional information is very important for food manufacturers due to legal requirements and for maintaining the image of a trustworthy business. Laboratory analysis is the golden standard, but it is also time consuming and requires infrastructure to run the processing of the samples safely. The alternative solution, NIR, is a rapid, cost-effective, and safe analytical technique which is already used in food industry, however, so far, there is no information on the performance of this method in relation to the verification of nutritional declarations on the labels of processed foods.
Materials and methods. In this work, the performance of previously developed NIR calibrations for three groups of Polish deli foods, containing meat (DM), cheese (DC), and pasta (DP), is evaluated. In each product group, between 29 and 50 products have been analysed. Samples were analysed simultaneously using NIR as well as traditional laboratory techniques. Then the differences of the measurements between the NIR prediction and the analytical results were compared with the label declaration tolerance levels set by the European Commission.
Results. The developed NIR calibrations allow the fibre and carbohydrate contents to be predicted in compliance with tolerance levels in all three product groups for 100% of the analysed samples. Any remaining nutrients which need to be declared on the label are predicted correctly in 100% of the samples within at least one group of analysed Polish deli foods. The only exception is the content of saturates which is predicted correctly in 83–97% of the samples. These results indicate that NIR could aid food manufacturers in reducing the number of non-compliant nutrition declarations by 9 to 27%.
Conclusions. The NIR method is a rapid solution that can effectively support food manufacturers in reducing the number of incorrectly labelled food products, controlling compliance with the recipe, and demonstrating due diligence to ensure that the information about the nutritional value of the purchased food is appropriate. Further research should focus on including a wider variety of products into calibration sets to ensure even better performance of NIR for prediction of the nutritional value of processed foods.

Keywords: near infrared spectroscopy, nutrition value, food label information compliance, in-situ food analysis, accuracy, deli products

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INTRODUCTION

A declaration of the nutritional value on the label of food products is required in the European Union, as well as in Poland from December 13, 2016, in accordance with Regulation No. 1169/2011 (Regulation…, 2018; Sałata, 2015). The rationale behind the legal requirement for the nutritional value declaration is to fight against the spreading epidemic of obesity which, with the current trend, by 2030, will affect every third European and Pole (Krzysztoszek et al., 2019). Food labelling and consumer information is the leading strategy of the European Union and Poland in the fight against this epidemic (Obesity prevention…, 2021; Rębiś, 2018). Previous studies indicate that this strategy may bring some positive effects. For example, it has been shown that women who read the nutritional information have on average a Body Mass Index which is 1.49 lower than women who do not read this information (Loureiro et al., 2012). In addition, recent studies have shown that high-energy, high-fat, and sodium warnings can result in a reduction in the prevalence of obesity by 4.98 percentage points over five years (Basto-Abreu et al., 2020).

However, for such satisfactory results to be achieved, the nutritional information on the packaging must be accurate. In 2012, the European Commission published a guide for competent authorities to control the compliance of the information on food labels, in which tolerance values for nutrient components were established (Guidance…, 2012). Do foods offered on the European market comply with the nutritional values declared on the label according to the guide's criteria? Information in recent publications shows that inconsistencies in nutrition labelling are very common. For example, a study of yoghurts available on the Irish market showed that the sugar content reported on the label of as many as 19% of the products was not compliant with the tolerance values set by the European Commission (O'Mahony et al., 2020). Furthermore, tests of ready-to-eat foods showed that more than half of those products with a salt content ≥1.25 g/100 g were not correctly labelled, and 27% did not match the declared content of saturated fat (Albuquerque et al., 2020).

In Poland, the authority controlling the compliance of food products with the label is the Agricultural and Food Quality Inspection (AFQI). Information on products that do not comply with the label is published in the Public Information Bulletin by the regional AFQI. The information provided also includes financial penalties. Providing inaccurate information about the nutritional value of food by producers is against Polish law. The Act on the commercial quality of agri-food products states that whoever "places on the market agri-food products that do not correspond to the commercial quality specified in the provisions of commercial quality or declared by the manufacturer on the label of these products shall be subject to a financial penalty of up to five times the value of the material benefit obtained or which could be obtained by introducing these agri-food products for trade, but not less than PLN 500" (Obwieszczenie…, 2021: pos. 630, art. 40a). In 2020, AFQI issued 836 decisions imposing fines due to the violation of the Act on the commercial quality of agri-food products for a total value of PLN 1,631,000 (Agricultural and Food Quality Inspection, 2020).

In addition to fines, food producers also have to worry about losing their status as a trustworthy business among their customers and consumers. The consequences of inaccurate consumer information about nutritional value may therefore be very severe. It is important for food producers to have access to tools that can quickly and easily verify nutritional information.

Currently, the "golden standard" that allows the accurate determination of nutritional value is chemical analysis using classical and instrumental techniques (Albuquerque et al., 2020). However, due to the cost and time it takes to perform this analysis (up to 2 weeks), it is used very sparingly. In the worst case, the information that appears on the nutrition label may be based on a single laboratory assessment following the development of a new product or even calculated based on published data for the nutrition of the product's ingredients. Large food businesses typically not only test the product after the development stage, but have a schedule of periodic, nutritional laboratory tests. However, due to the time needed to obtain results, many products arrive on the market before the nutritional information is available. In such cases, the label contains data obtained from analyses of previous batches of the product. Without a verification tool that allows quick assessment of the nutritional value, there is no room for batch-to-batch variation.

In order to be able to monitor the accuracy of the nutritional value, a method that allows analysis to be carried out on site in a timely manner is required. One such method is near infrared spectroscopy (NIR). Predicting the nutrient content of homogeneous food samples using this method takes under a minute and does not require the use of toxic and environmentally harmful reagents. This method is already used in food industry for the routine analysis of the nutrients in products such as milk and meat, but its application to more complex matrices such as deli products is not practiced due to the lack of availability of calibration databases. In a publication from 2020, such calibrations for deli products with meat (DM), cheese (DS), and pasta and noodles (DP), together with validation data were presented (Dudkiewicz et al., 2020). In this short communication, we would like to present the usefulness of the developed calibrations in terms of verifying the compliance of the nutritional value of the mentioned deli products according to the guidelines of the European Commission (Guidance…, 2012).

MATERIALS AND METHODS

The development of the calibration for the deli products used in this study was described in a previous publication (Dudkiewicz et al., 2020). Briefly, a set of deli products selected to cover the variability in each of the three product groups (DM, DC, and DP) from different retailers was purchased. Calibrations were based on 60 samples for each product group, while another 29 DP, 38 DM, and 50 DC samples were used for validation and presentation of the results in this publication (number of samples provided by the analytical laboratory). The samples used for validation were different and independent of those used for calibration. The content of the nutritional components for the set of samples used for validation is given in Table 1.

Table 1. The content of nutritional components in the validation sample set, % mean content ±standard deviation

Component	DM	DC	DP
Protein	8.2 ±2.7	8.0 ±1.7	4.1 ±1.0
Carbohydrates	26 ±3	30 ±3	33 ±5
Sugars	1.8 ±1.1	4.7 ±3.0	2.5 ±3.4
Fibre	2.2 ±0.5	1.6 ±0.2	2.6 ±0.4
Fat	7.7 ±2.9	2.1 ±0.7	1.7 ±1.0
Saturates	2.51 ±0.94	0.91 ±0.69	0.61 ±1.25
Salt	0.84 ±0.16	0.72 ±0.06	0.66 ±0.05

The DM group included meat dumplings, meat and potato dumplings, and tortilla with meat. The DC group included cheese dumplings, sweet fromage dumplings, and cheese tortellini, while the DP group included dumplings, pancakes, cooked pasta, gnocchi, and steamed dumplings.

All samples were prepared using homogenization with a laboratory mill (GRINDOMIX GM 200, Retsch, Germany). The homogenization of samples prior to the nutrition value assessment is the only sample preparation required in NIR analysis. However, it is an essential step. Similar to in routine laboratory assessment, introduction of non-homogenized samples into the NIR instrument will likely result in inaccurate prediction of the nutritional value. Nevertheless, based on our experience, even coarse mincing will give samples of adequate homogeneity for the NIR instrument used in this work, FoodScan™ Lab (FOSS, Denmark), which is also popular within the industry. The size of the sample placed in the dish of this instrument was about 120 cm^3. During a single reading, the sample dish was rotated and sixteen subscans were taken from different areas. The subscans were then merged together to give the result. This practice was aimed at minimizing the measurement error due to sample inhomogeneity when compared to traditional laboratory approaches.

The nutritional value of all samples of the deli products was simultaneously characterized by classical and instrumental laboratory methods (Megazyme, 2019; PN-A-04018:1975; PN-A-82100:1985; PN-EN 13804:2013-06; PN-EN ISO 12966-2:2011; PN-ISO 1444:2000; Regulation…, 2018).

The NIR instrument was controlled by ISIscan™ software (FOSS, Denmark). The instrument operated in the transmission mode, and the wavelength resolution was 2 nm.

For the tests, cuvettes compatible with the instrument with a glass window of 14 cm in diameter were used. A range of wavelengths of 850–1050 nm was applied to acquire spectra. Within this range of wavelengths, the spectrum contains 2^{nd} overtone for N-H and O-H bonds, as well as 3rd overtone for C-H bonds (Osborne, 2006).

The spectral responses to NIR are weak, which in practice means that the raw spectra for different deli product groups look very similar shape-wise (Fig. 1a) and hence, it is not possible to find regions which change with a change in nutritional composition without chemometrics.

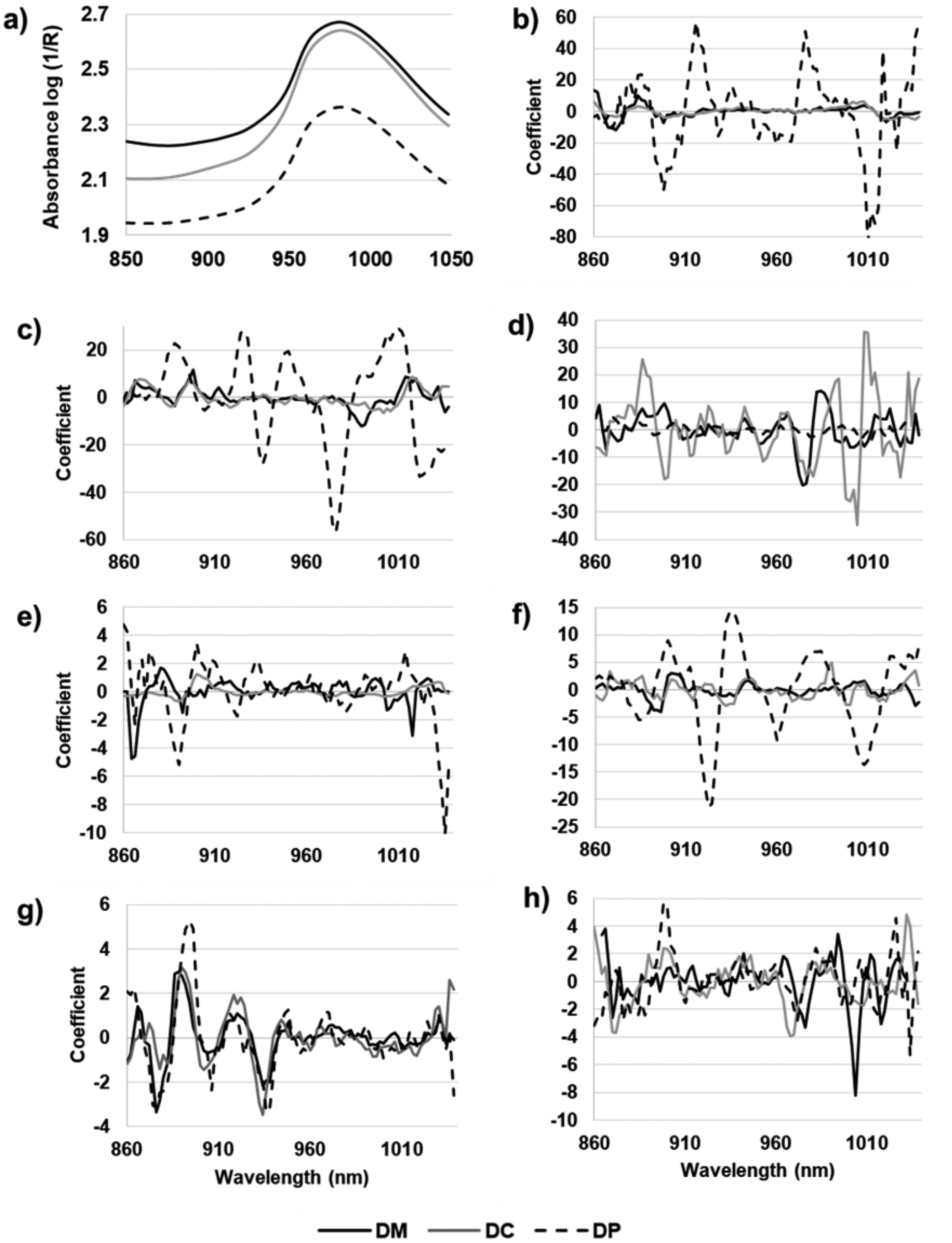

Fig. 1. Output from NIR calibration. Averaged, unprocessed NIR spectra for samples used in calibration (a) and beta coefficients calculated by means of modified PLS for: protein (b), carbohydrates (c), sugars (d), fibre (e), fat (f), saturates (g), salt (h)

To develop calibrations, nutritional value data were paired with NIR spectra. The chemometric software used to obtain calibration equations was WinISI 4 (FOSS, Denmark). The mathematical approach applied was Modified PLS (Partial Least Squares Regression), available within the software.

Quality parameters for the developed calibrations, such as coefficients of determination (R^2), root mean square errors of cross validation (RMSECV), and ratios of performance to deviation (RPD), as well as nutritional value of samples in the calibration dataset, were given in a publication by Dudkiewicz et al. (2020). Here, to visualize which spectral areas were important for the prediction of nutritional compounds, we have presented values of calibration coefficients for protein, carbohydrates, sugars, fibre, fat, saturates, and salt (Fig. 1b–1h). The Modified PLS assigns coefficients across the full spectrum based on the variability of the nutritional compound in the samples used for the calibration, with 2 nm increments. The greater the absolute value of the coefficient, the more important this region is for calibration. Based on the results presented here, it can be noticed that important NIR absorbance bands were located in different areas of the spectrum, depending on the deli product group. The only exception seemed to be saturates (Fig. 1g), where the most important absorption bands span 860–950 nm and overlap between the product groups.

The differences in spectral responses indicate that for accurate predictions, appropriate division of deli products into groups is necessary.

The usefulness of NIR for verification of the compliance of the nutritional value with the declaration on the product label was assessed according to the relationship in Equation 1:

$$\sigma \leq \text{tolerance} \qquad (1)$$

where:

σ – absolute difference between the result from the laboratory test and the result from the NIR prediction.

Tolerance – acceptable difference between the result of the laboratory test and the value stated on the label recommended by the European Commission (Guidance…, 2012).

RESULTS

The σ values for the seven nutrient components that need to be measured or calculated in order to provide nutritional information compliant with European legislation are summarized in Figure 2.

The tolerance values are referenced in Table 2, while Figure 2 uses only the most conservative tolerance value.

The median values of σ were all lower than the minimum value for tolerance (Table 2). In addition, for all nutritional components except carbohydrates, more than 75% (third quartile) of the results met the lowest tolerance criterion. The NIR method showed the greatest compliance with the reference methods for fibre and fat as for these components in two of the three product groups, all the prediction results complied with the lowest tolerance level.

Table 3 summarizes the number of samples for which σ values compliant with criterion in Equation 1 were recorded. These results take into account all tolerance levels given in Table 2, and not only the most conservative one (applied in Figure 2). In this summary, only the results which fell within the analytical range for NIR and the reference methods were used. Therefore, the number of qualified results is different in the same food group depending on the nutrient tested. The analytical ranges for the methods were given in a previous publication (Dudkiewicz et al., 2020).

The two components of the nutritional value for which σ satisfied the condition in Equation 1 for all samples regardless of the product group were carbohydrates and fibre (Table 3). For all other nutrients, except for saturates in at least one product group, the NIR prediction gave acceptable σ for 100% of the samples within the analytical range.

The best quality of the overall nutritional value prediction was achieved for the DC group, where the contents of carbohydrates, fibre, fat, and salt were predicted using NIR with satisfactory accuracy (σ) for all the samples. For the remaining components, the NIR prediction allowed the content to be estimated correctly in >90% of the samples (results marked in yellow). The only problem in this group was the prediction of sugar content in sweet products, where σ satisfied the Equation 1 condition for only 34% of the samples. These samples were excluded from the summary of

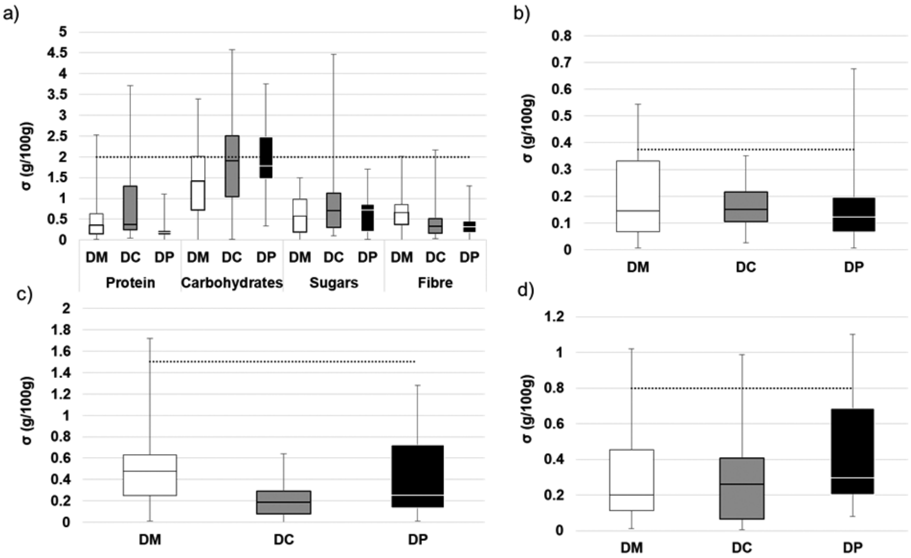

Fig. 2. Box and whiskers plot of σ values for the content of protein, carbohydrates, sugars and fibre (a), salt (b), fat (c), and saturates (d) in the three analysed groups of foods – DM, DC, and DP. The dotted line on the graph is the most conservative tolerance level established by the European Commission (Table 2). The results for sugars in DC include only deli products which were not sweetened

Table 2. European Commission's guidance criteria for the evaluation of the nutritional value declaration compliance

Component	Concentration in the food product g/100 g	Tolerance (±)
Carbohydrates, sugars, protein, and fibre	<10	**2 g**
	10–40	20%
	>40	8 g
Fat	<10	**1.5 g**
	10–40	20%
	>40	8 g
Saturates	<4	**0.8 g**
	≥4	20%
Salt	<1.25	**0.375 g**
	≥1.25	20%

In bold are the tolerance values which were referred to in Figure 2.

the results for sugar content in Table 3. The remaining nutrient components were predicted with an acceptable σ in 94–100% of the DC samples, regardless of whether they were sweetened or unsweetened.

DISCUSSION

The literature does not provide much information on the compliance of NIR predictions with the requirements of the European Commission's guide with regards to the accuracy of the nutritional declaration. However, there are several papers where the authors provided the results of NIR predictions and reference values (Bagchi et al., 2016; Deng et al., 2018; Marrubini et al., 2017; Neves et al., 2019; Wójcicki, 2018). For example, when determining the nutritional value of ready meals, pasta with sauce products, the authors obtained slightly worse σ values for protein (median 1.10, in this study from 0.18 to 0.37 depending on the group), carbohydrates (median 2.30, in this study from 1.40 to 1.90), fibre (median 0.90, in this study from 0.32 to 0.67), and fat (median 0.50, in this study from 0.19 to 0.48) compared to the results presented here (Neves

Table 3. The number of NIR results analysed for compliance with the tolerance set by the European Commission and the number of compliant results

Component	Number of qualified results			Percentage of samples with σ ≤ tolerance		
	DM	DC	DP	DM	DC	DP
Protein	43	50	22	95	94	100
Carbohydrates	33	32	29	100	100	100
Sugars	34	24*	17	100	96*	100
Fibre	36	17	29	100	100	100
Fat	44	48	9	93	100	100
Saturates	40	36	6	95	97	83
Salt	39	16	13	82	100	92

The percentage of acceptable σ values was colour: coded from green – highest percentage of compliance (100) to amber – lowest percentage of compliance (<90).
*Results include only deli products which were not sweetened.

et al., 2019). On the other hand, these authors obtained a similar σ median for the prediction of sugars (0.60 and in this paper 0.58 to 0.72).

Similarly, in a study on the determination of the nutritional value of medical foods, greater σ values were calculated for protein (median 0.55) and fat (median 0.47) but similar ones for carbohydrates (median 1.45) compared with the σ values obtained in this study (Deng et al., 2018).

Thus, it can be said that, for complex foods, the calibrations obtained in this study allowed the nutritional value to be predicted with a comparable or smaller σ error in comparison to the literature data available so far.

Many authors emphasize that NIR predictions work best for matrices where the calibration is based on very similar products, unlike the deli foods studied here. In other studies on rice bran, the median σ was 0.29 for protein and 0.33 for fat (Bagchi et al., 2016). These values were within the range of the differences obtained for the deli products studied here. However, the authors obtained much better results for fibre (median σ = 0.05), which may be due to the high content of this component in the bran.

Slightly lower values of σ compared with our results were also obtained in a study of sugar content in plant milks (median 0.50; Marrubini et al., 2017). On the other hand, the median σ for the protein content in our deli products was lower than in the protein supplements in another study (σ at the level of 1.06; Wójcicki, 2018).

The NIR calibrations in this study were performed on a very diverse groups of products, however, the prediction allowed their nutritional value to be verified with similar accuracy to the NIR predictions developed by other authors for less variable matrices.

An important question to address is whether NIR could be used to reduce the proportion of food products where the nutritional value is not correctly stated on the label. For the most crucial nutrients for diet-related diseases – salt, fat, saturated fat, and sugars – the NIR predictions obtained in this study allowed the nutritional value to be verified with sufficient accuracy (Eq. 1) for a proportionally larger number of samples (82–100%) compared to the number of correctly labelled examples of food products available on the European market, as reported in the literature (73–88%; Albuquerque et al., 2020; O'Mahony et al., 2020). The use of NIR in nutritional labelling compliance checks could therefore reduce the percentage of errors by 9 to as much as 27%.

CONCLUSIONS

This study indicated the potential of NIR to verify the nutritional value of deli products available on the Polish market in accordance with EU regulation 1169/2011. The method can effectively support food manufacturers in reducing the number of incorrectly labelled food products, controlling compliance with the recipe, and demonstrating due diligence with regards to consumer information about the nutritional value of purchased food.

Further research should be aimed at improving NIR calibrations by extending their range to a larger number of more differentiated samples to ensure even greater consistency of NIR predicted values with laboratory test results. Furthermore, the research presented here has shown that identifying multimodal data distributions (such as bimodal distribution of sugar content in sweetened and unsweetened dumplings) may be crucial for NIR calibration performance. Such calibrations should be split where possible to ensure an accurate prediction for each of the modal tendencies, e.g., high and low sugar content.

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