Available online at www.sciencedirect.com

ScienceDirect

Current Opinion in Food Science

Considerations about the gap between research in near-infrared spectroscopy and official methods and recommendations of analysis in foods

Giacomo Squeo[1], Jordi Cruz[2], Davide De Angelis[1], Francesco Caponio[1] and José M Amigo[3,4]

Near-infrared spectroscopy (NIR spectroscopy) is a powerful analytical technology for measuring food characteristics. More and more applications of NIR spectroscopy have been studied, and an increasing number of commercial solutions are available on the market. However, only 25 documents about NIR spectroscopy have been issued by international bodies, while the number of scientific papers published in recent years has always been higher than 60/year. Studies prove that NIR spectroscopy could boost the sustainability of the food system, the food quality, and the optimisation of production by real-time monitoring. Considering the technical, analytical, and environmental advantages of NIR spectroscopy, more efforts should be made to extend the applicability of NIR spectroscopy solutions and promote the development of more official methods based on NIR spectroscopy.

Addresses
[1] Department of Soil Plant and Food Sciences (DiSSPA), University of Bari Aldo Moro, Via Amendola 165/A, 70126 Bari, Italy
[2] Escola Universitària Salesiana de Sarrià, Passeig Sant Joan Bosco 74, 08017 Barcelona, Spain
[3] IKERBASQUE, Basque Society for the Promotion of Science, Plaza Euskadi, 5, Bilbao 48009, Spain
[4] Department of Analytical Chemistry, University of the Basque Country UPV/EHU, Barrio Sarriena S/N, Leioa 48940, Spain

Corresponding authors: Squeo, Giacomo (giacomo.squeo@uniba.it), Cruz, Jordi (jcruz@euss.cat)

Current Opinion in Food Science 2024, **59**:101203

This review comes from a themed issue on **Open Issue**

Edited by **Anderson S Sant'Ana** and **Alejandro Gregorio Marangoni**

Available online 2 August 2024

https://doi.org/10.1016/j.cofs.2024.101203

Introduction

Accurate analyses play a fundamental role in modern food production systems. Depending on the scope, the analysis of food and food ingredients ensures product quality, safety, genuineness, and authenticity. Different types of analysis can be employed to achieve these goals, like chemical, physical, structural, and sensory analyses. At the same time, the parameters to be monitored may be defined by the current legislation or directly by the companies. A paramount case is the extra virgin olive oil, whose quality parameters are well established [1]. Thus, to label an olive oil as 'extra virgin', it is necessary to evaluate whether the characteristics of the product comply with the standards defined by the regulation.

Apart from or in addition to mandatory standards, companies and any food business operator may establish their own set of standards. This allows food operators to guarantee the desired quality level for consumers, to respect their internal specifications, or to establish pricing for incoming or outgoing products with suppliers or buyers. As a matter of example, a company involved in fruit processing would be interested in determining the payment for raw materials based on the maturity level, assessment of infections, appearance, ripening stage, or plausible hits in the fruits. Therefore, the results of food and ingredient analysis represent the objective basis for assessing the compliance of the product with the current and specific regulations and/or with the technical specifications of the company, underscoring the unreplaceable and fundamental role of food analysis within the food industry, carrying legal, economic, and societal implications.

This fact, together with the breakthrough experienced in the field of data analysis, has driven companies to adopt more technological solutions that imply the adaptation of nondestructive, rapid, and green analytical approaches to replace the more common wet chemistry methods. The drivers of this innovation can be found in i) the urgent need to improve the sustainability of human activities; ii) the need to improve the performance in terms of resources and time [2]; iii) the implementation of the analysis step within the manufacturing process, according to the Industry 4.0 and Industry 5.0 paradigms [3]; iv) the need to increase the competitiveness in a more specialised and evolving market; and v) the need to provide well-controlled products to demanding costumers. It is in this framework where particular attention has been gained by near-infrared spectroscopy (NIR spectroscopy), which has passed from a sleeping technique to one of the more promising and applied techniques in the food sector [4].

NIR is the vibrational electromagnetic radiation occurring in the range of 12500–4000 cm^{-1} or 800–2500 nm. Photons at those energies interact with the matter, creating a spectrum characterised by the so-called overtones and combination bands of the fundamental vibrations of heteroatomic bonds. Depending on the matter under consideration, the signal could be quite complex, but, in general, it is highly nonspecific. Although the bands can be associated with specific functional group frequencies [5], the extraction of useful chemical information is usually carried out by using proper chemometric tools [6•]. It is thanks to the great development of Chemometrics (a.k.a. machine learning [7]) and the increased accessibility to hardware, new sensors, and computing power that NIR spectroscopy applications in food and food products have become so widespread, making it one of the dominant analytical technologies in the modern food sector. Its applications span from laboratories (academic, private, and industrial), where it is mainly used as a rapid method for analysing specific food characteristics, industry (as a process analytical tool for fast sorting of raw material and finished products), as well as process monitoring and control, until the open field (portable instruments and airborne vehicles) [3,8,9].

However, despite this rapid expansion, there has not been a parallel evolution of official analysis methods established by recognised international organisations, limiting the broader use of NIR spectroscopy for routine work and creating an asymmetry between the current quality assessment practices and the needs of companies and regulatory bodies of NIR spectroscopy analytical routines generated according to official methods. The aims of this manuscript are i) to present the readers with an overview of the official methods based on NIR spectroscopy available nowadays from recognised international bodies, ii) to provide some considerations about which could be the reasons for the gap in the alignment between the official and nonofficial NIR spectroscopy methods of analysis, and iii) to highlight the benefits and impact that official NIR spectroscopy methods could have on the food system.

Official documents based on near-infrared spectroscopy

Table 1 reports the investigation results concerning the official documents based on NIR spectroscopy issued by different international organisations. At the same time, Figures 1 and 2 depict graphical summaries of the issuing organisations and the type of documents, respectively.

A total of 25 entries have been found (Table 1), most of them published by the American Association of Cereal Chemists (AACC), the American Oil Chemists' Society (AOCS), and the International Standards Organisation (ISO) (Figure 1). Overall, they can be divided into two groups: i) guidelines and recommendations; ii) method and standard procedures (Figure 2). Guidelines serve as valuable resources for scientists, analysts, and laboratories, offering guidance on various aspects of analytical work. They can be used as references to assist analysts in performing experiments, calibrations, and measurements effectively and reliably. It is worth noting that a total of eight guidelines for NIR applications have been identified, emphasising the importance of good practices in sampling [10,11•] and spectra acquisition, two of the aspects giving higher sources of error when dealing with measurement of large volumes of samples.

In contrast, the survey revealed 17 standard analysis methods covering various raw materials and products. Overall, legumes, cereal grains, flour, and oilseeds encompassed 16 distinct documents, while two official methods for evaluating oil and fat quality parameters were found. Official methods have also been developed for milk and dairy products, wine and alcoholic beverages, and meat products.

Regarding the sampling mode, most applications use the reflectance mode (in solid samples), while fewer applications use transmittance and transflectance (in liquid samples) (Table 1). This might be due to historical reasons linked to the first developments of NIR spectroscopy in the 70s, which were mostly directed toward the analysis of solid samples, as well as to the limitations of NIR technology when dealing with the high content of water of specific samples.

The range and diversity of compounds and parameters that official methods cover are noteworthy. Table 1 reveals that many of the official documents serve as viable alternatives to traditional chemical analyses, primarily focusing on the proximate composition of solid samples, including the protein, moisture, and fat content in the samples. Other methods regard the determination of iodine value, fatty acid type, and *trans*-isomers in fats and oils or alcohol content in drinks of vitivinicultural origin and beer. An interesting application is found in the AACC 39-70.02 [12], the assessment of wheat hardness, a physical-structural parameter, which proved how NIR spectroscopy could be applied to measure other critical attributes of ingredients and foods apart from the chemical composition, thus broadening the spectrum of its applications [2].

AACC, AOAC, and ISO released guidelines about instrument management and model development, calibration, maintenance, validation, and transfer. ISO

Table 1

Official documents based on NIR spectroscopy issued by different international organisations.

Organisation	Acronym	Method No.	Title	Type[a]	Sampling mode[a]	Parameter(s)[a]	Collection/reference	Notes
American Oil Chemists' Society	AOCS	Am 1a-09	Near-Infrared Spectroscopy Instrument Management and Prediction Model Development	Guideline			Official Methods and Recommended Practice of the AOCS, 7th Edition	
		Am 1-92 Reapproved 2017	Oil, Moisture and Volatile Matter, and Protein in oilseeds by Near-Infrared Reflectance	Standard procedure	Reflectance	Oil, moisture, volatile matter, protein	Official Methods and Recommended Practice of the AOCS, 7th Edition	
		Cd 1e-01	Iodine Value by Pre-calibrated FT-NIR	Standard procedure		Iodine value	Official Methods and Recommended Practice of the AOCS, 7th Edition	
		Cd 14f-14	Rapid Determination of Total SFA, MUFA, PUFA, and trans-Fatty Acid Content of Edible Fats and Oils by Pre-Calibrated FT-NIR	Standard procedure	Transflectance	Total SFA, MUFA, PUFA, *trans* fatty acid	Official Methods and Recommended Practice of the AOCS, 7th Edition	
American Society of Brewing Chemists	ASBC	Barley 5	Moisture	Method		Moisture	ASBC Methods of Analysis, 14th Edition	
		Beer 4	Alcohol	Method		Alcohol	ASBC Methods of Analysis, 14th Edition	
		Flavored Alcohol Beverages 1	Alcohol and Original Extract content in Flavored Alcohol Beverages by Near-Infared	Method		Alcohol and Original Extract	ASBC Methods of Analysis, 14th Edition	
Association of Official Analytical Chemists	AOAC	2007.04	Fat, Moisture, and Protein in Meat and Meat Products	Method	Transmittance	Fat, moisture, protein	J. AOAC Int. 90, 1073(2007)	
		997.06-2005	Protein (Crude) in Wheat - Whole Grain Analysis Near-Infrared Spectroscopic Method	Method			Official Methods of Analysis of AOAC INTERNATIONAL (OMA), 22nd Edition	
Cereals & Grains Association (formerly, American Association of Cereal Chemists)	AACC	39-00.01	Near-Infrared Methods -Guidelines for Model Development and Maintenance	Guideline			AACC Approved Methods of Analysis, 11th Edition	
		39-01.01	Evaluation of NIR Instrument Calibration	Guideline			AACC Approved Methods of Analysis, 11th Edition	
		39-10.01	Near-Infrared Reflectance Method for	Method	Reflectance	Protein	AACC Approved Methods of Analysis, 11th Edition	

Table 1 (*continued*)

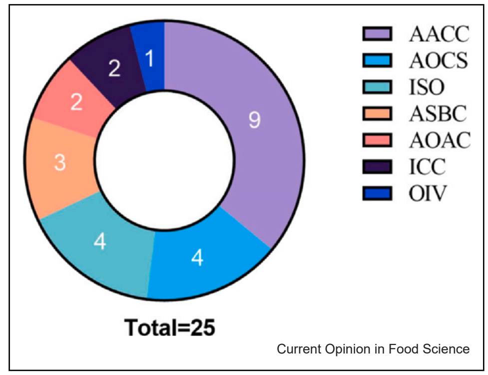
Organisation	Acronym	Method No.	Title	Type[a]	Sampling mode[a]	Parameter(s)[a]	Collection/reference	Notes
			Protein Determination in Small Grains					
		39-11.01	Near-Infrared Reflectance Method for Protein Determination in Wheat Flour	Method	Reflectance	Protein	AACC Approved Methods of Analysis, 11th Edition	
		39-20.01	Near-Infrared Reflectance Method for Protein and Oil Determination in Soybeans	Method	Reflectance	Protein, oil	AACC Approved Methods of Analysis, 11th Edition	
		39-21.01	Near-Infrared Reflectance Method for Whole-Grain Analysis in Soybeans	Method	Reflectance, Transmittance	Protein, oil, moisture	AACC Approved Methods of Analysis, 11th Edition	
		39-25.01	Near-Infrared Reflectance Method for Protein Content in Whole-Grain Wheat	Method	Reflectance	Protein	AACC Approved Methods of Analysis, 11th Edition	
		08-21.01	Prediction of Ash Content in Wheat Flour - Near-Infrared Method	Method		Ash	AACC Approved Methods of Analysis, 11th Edition	
		39-70.02	Near-Infrared Reflectance Method for Hardness Determination in Wheat	Method	Reflectance	Hardness	AACC Approved Methods of Analysis, 11th Edition	
International Association for Cereal Science and Technology	ICC	159	Determination of Protein by Near Infrared Reflectance (NIR) Spectroscopy	Generic Methods		Protein	ICC Standards	
		202	Procedure for near-infrared (NIR) reflectance analysis of ground wheat and milled wheat products	Recommendations	Reflectance	Protein, moisture	ICC Standards	
International Organisation of Vine and Wine	OIV	OIV-MA-BS-08: R2009	ABV by Near-infrared reflectance spectroscopy in spirit drinks of vitivinicultural origin	International methods of analysis	Transflectance or transmittance	Alcohol by volume	Compendium of International Methods of Analysis of Spirituous Beverages of Vitivinicultural Origin	
International Organisation for Standardisation	ISO	ISO 21543:2020 | IDF 201:2020	Milk and milk products - Guidelines for the application of near-infrared spectrometry	Guideline	Transmittance, diffuse reflectance or transflectance		https://www.iso.org/standards.html	
		ISO 23291:2020 | IDF 248:2020	Milk and milk products - Guidelines for the application of in-line	Guideline		Protein, fat and total solids in liquid milk and milk products; protein, fat and moisture in solid	https://www.iso.org/standards.html	Mid infrared included

Table 1 *(continued)*

Organisation	Acronym	Method No.	Title	Type[a]	Sampling mode[a]	Parameter(s)[a]	Collection/reference	Notes
			and on-line infrared spectrometry			or semi-solid products, such as milk powder, and butter and liquid dairy streams		
		ISO 12099:2017	Animal feeding stuffs, cereals and milled cereal products. Guidelines for the application of near-infrared spectrometry	Guideline		Moisture, fat, protein, starch and crude fibre and parameters such as digestibility in animal feeding stuffs, cereals and milled cereal products.	https://www.iso.org/standards.html	
		ISO/CD 18419	Oilseeds – Guidelines for the application of near-infrared spectrometry	Guideline		Constituents such as moisture, fat and protein and some minor parameters such as total glucosinolates in oilseeds and oilseeds meals.	https://www.iso.org/standards.html	Under development (latest access June 24)

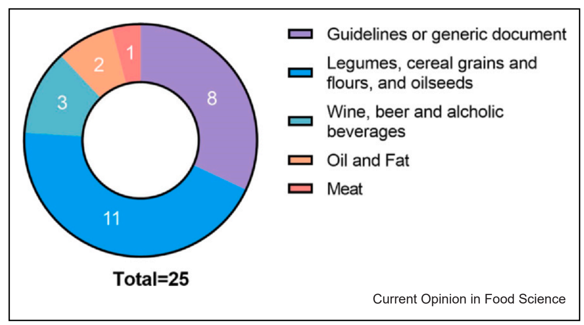
[a] As reported (if reported) in the document description, preview, or abstract.

Figure 1



Pie chart showing the bodies that issued official documents about NIR spectroscopy.

Figure 2



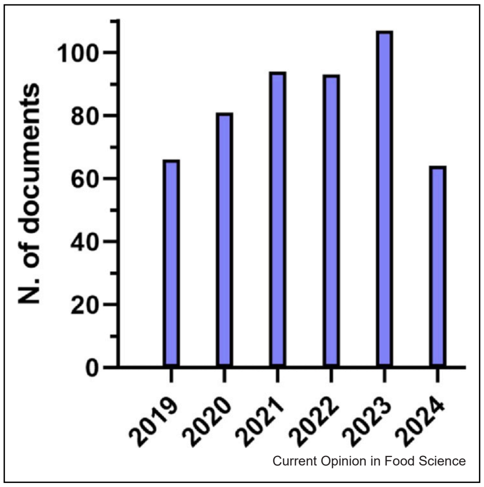
Pie chart showing the food categories covered by official documents about NIR spectroscopy and the type of document. Within the 'guidelines', other food categories are included. See Table 1 for more details.

21543:2020 | IDF 201:2020 and ISO 23291:2020 | IDF 248:2020 address the issues related to the application of NIR spectroscopy in in-line and on-line scenarios specifically designed for milk and milk products [13,14]. The AOAC 2007.04 [15] specifically refers to commercial equipment, the FOSS FoodScan™ device (FOSS A/S, Hillerød, Denmark) for the determination of moisture, protein, and fat in meat and different kinds of meat products.

Current trends in research and industry

The research environment is developing faster than regulations and guidelines. Figure 3 presents the results of the literature search carried out using the keywords 'nir', 'near', 'food', and 'infrared' within the topic (title, abstract, and keywords) in the 'agricultural and biological sciences' subject area of the Scopus database, considering the research work published in the last five years (2019–21 May 2024).

Figure 3



Number of research articles regarding NIR published from 2019 to 2024 within the agricultural and biological sciences category in the Scopus database (data updated until 21 May 2024).

The total number of records found is 505. More than 60 articles/year were published, overcoming the barrier of 100 articles in 2023. This impressive growth can be attributed to several key factors, such as i) an increasing number of applications covering other food products and/or other parameters beyond those historically focused [16–19]; ii) an increasing number of instrumental capabilities (benchtop, handhelds, in-field, and industrial setups) and the combination of NIR spectroscopy with other techniques, such as imaging [20–24]; iii) an increasing number of mathematical approaches to process NIR spectroscopy data [25,26]; iv) a general shift toward untargeted analysis even for qualitative purposes, such as authenticity [27–29], and it highlights the net difference between the scientific articles regarding NIR spectroscopy application in the food sector and the official methods at the disposal of the food system actors (Figure 3).

The increasing application of NIR spectroscopy is also reflected in the high number of commercial equipment nowadays available for the quality assessment of various foods. In fact, NIR instruments with ready-to-use calibrations are increasing its number in the spectroscopy industry. These commercial instruments represent a step forward in the use of this technique because no knowledge about NIR spectroscopy and chemometrics is previously required. On the other hand, every instrument could be arranged for a specific application (one or few), and consequently, these instruments can be limited to other diverse applications.

Also, nowadays, the replacement of benchtop instruments for portable ones or the direct introduction of portable NIR instruments instead of benchtop instruments is observed. This is surely linked to a better performance of these instruments now than some years ago and to the low price if compared to a benchtop one. As previously commented, the increase in the use of portable NIR instruments is one of the reasons for the increasing number of applications that are appearing in the literature [30]. Nonetheless, the authors have no knowledge of portable NIR official standard methods within this increasing number of applications. Several reasons might be involved in this fact, starting from the fact that portability is normally associated with smaller wavelength ranges (normally in the region between 400 and 1700 nm) due to the lower cost of the sensors. Another reason is the lack of standardisation in the measurement of solid samples, together with the variability of the measurements associated with measuring in uncontrolled conditions.

Why extending the number of near-infrared spectroscopy official methods could be welcomed

Improving the range of official applications of NIR spectroscopy could be welcomed from different strongly interconnected perspectives. First, from a sustainability point of view, routinary wet chemistry methods have a higher environmental impact than spectroscopic ones [31–33]. For example, Casson et al. [31] have found an impact average gap of 36 times between chemical and optical methods. This gap would be even larger if multiparametric applications were considered [16,19]. NIR spectroscopy is a naturally green analytical technique [34] that matches by its nature the Green Chemistry principles, allowing to i) reduce the usage of solvents and other chemicals; ii) shorten the analysis duration, which decreases in the range of seconds or few minutes at maximum; iii) reduce the delay in obtaining the analytical results, paving the way to in-line or on-line food monitoring; iv) reduce the occupational exposure to laboratory staff thanks to automatisation and miniaturisation [35]. Recently, Saveliev and colleagues [36•] proved how NIR-based methods, coupled with chemometrics, outperformed in terms of greenness with respect to wet methods.

Second, NIR spectroscopy is a perfect high-throughput analytical method for dealing with a very large volume of samples. This point becomes particularly relevant when considering the need for controlling bodies to ensure high productivity and efficiency, the responsibility of food business operators to ensure food safety, or the significant challenges related to food fraud and authenticity.

These aspects allow us to highlight a third fundamental point, barely stressed by the specific scientific community, which is the powerful combination of NIR spectroscopy with classification algorithms for the development of qualitative assessment methods. To the best of the authors' knowledge, no official methods make use of the NIR fingerprint to ensure food authenticity and/or compliance despite it being one of the more interesting applications of NIR spectroscopy in modern food systems. When coupled with class modelling techniques, such as Soft Independent Modelling of Class Analogy (SIMCA) [37•], NIR spectroscopy emerges as a powerful, rapid, and effective tool to track food genuineness and/or category compliance [38]. Several successful applications can be found in the literature [24,27,38,39]. As far as we know, some companies are now delivering this solution mainly to assess the compliance of food products or ingredients with the desired technical specifications.

NIR spectroscopy and Chemometrics: the irruption of deep learning

Undoubtedly, NIR spectroscopy cannot be understood without Chemometrics. During this manuscript, Chemometrics has been present in an indirect manner of connecting the spectral signatures with the concentrations or classes. Therefore, we will not make an exhaustive review on that topic since several good reviews already exist in the literature dealing with the application of Chemometrics in NIR spectroscopy and the food sector [6•]. Nevertheless, we cannot let pass the opportunity to comment on aspects that are invading the literature but only to a limited extent the official documents reported in Table 1, that is, the strong irruption of the deep learning (DL) scenario.

Classically, NIR spectroscopy can be considered as spectral radiation that works perfectly under the Beer–Lambert law. Therefore, in a close setting, and if the spectra are correctly measured (and not saturated), linear multivariate models have demonstrated their great advantages (especially Partial Least Squares, PLS, and its variations) [40]. Nevertheless, there are more open scenarios where the signals may differ from the ideal linear situation, for example, when different harvesting periods are considered or when models want to be globalised to work in a wide umbrella of similar products, needing the usage of more sophisticated chemometric models, like artificial neural networks (ANN) and their progression to DL structures [26].

ANN models and their sub-class DL are more advanced models capable of handling nonlinear situations that make them look like the perfect alternative to sometimes badly called 'more classical linear approaches'. While there are cases where it has been clearly demonstrated that ANN can really offer a great advantage, it is still not clear i) if such complex model structures are really needed, ii) what is the real gain of using them in terms of predictability and transferability, and iii) how to present them in a normative as perfectly validated models. While this is true for the official documents, the literature is more and more flooded with applications of ANN and DL predicting quality parameters in food using NIR spectroscopy. The curious matter is that most of these works are presented as a main novelty, disregarding the fact that, for instance, ANN has been used in NIR spectroscopy data for more than 25 years [41]. Also, there is a strong trend to compare their performance with so-called 'classical methods' like PLS, but without a correct performance of the PLS model and emphasising the architecture of the network needed to improve the R^2, for instance, from 0.915 to 0.920, without accounting for the fact that the experimental error might be larger than the improvement.

There are some promising movements towards the application of ANN and DL in NIR spectroscopy. Nevertheless, there is still a clear lack of showing real and undisputable improvement from more linear but stable solutions. This might be one of the reasons for not having many official documents approving the use of ANN and DL that, to the best of our knowledge, till now are limited to just one standard [15]. A deeper understanding of the benefits of more complex models through comprehensive validation protocols is strongly needed [7,26,42]. There is nowadays an overabundance of data analytical methods. Most of them are proposed as 'new' methods or variations of already existing methods with a limited number of samples. Consequently, an urgent standardisation of the minimum requirements for an acceptable predictive model must be accomplished and implemented. In this sense, the presence of official guidelines (Table 1, Figure 2) and the further development of new ones are welcomed and would help depict a harmonised and standardised framework for all the involved actors.

The future:opportunities and challenges of near-infrared spectroscopy in the future food sector

The current landscape of applications in the food sector presents promising opportunities and notable challenges for the future use of NIR spectroscopy. Upon examining recent literature, it is apparent that various significant applications still need to be addressed by both experimental research and established official analysis methods. For instance, the food sector is showing a rapid evolution marked by the emergence of alternative protein-based foods, encompassing meat analogues and dairy substitutes, as well as food formulations using by-products and innovative ingredients like microalgae or insects. Within these unique food applications, the use of NIR spectroscopy in experimental research remains limited [16,17,43], while dedicated official methods of analysis established for these specific products are currently non-existent to the best of the author's knowledge. However, it was recently reported that some of the official methods developed for meat can be transferred to meat analogues with minor adjustments [44]. For example, considering that moisture and protein are generally determined with high reliability and reproducibility by NIR spectroscopy, it would be desirable that official methods can be developed for such applications in innovative food products. This evolution would not only facilitate regulatory compliance and ensure the quality and authenticity of these evolving food products but also reduce the environmental impact of the analytical procedures and the time required for the analysis.

Other opportunities for standardisation include continuous process monitoring using NIR spectroscopy. In fact, standardised approaches in continuous monitoring enable the consistent assessment of critical parameters throughout production. In the food sector, this includes monitoring variables like moisture, fat and protein content, and other quality attributes during food production. For example, in the dairy sector, the protein-to-fat ratio is crucial in controlling the coagulation process during cheese manufacturing, and it can be monitored in-line with NIR spectroscopy [45]. Therefore, the integration of NIR spectroscopy allows for real-time analysis, ensuring that products meet defined quality standards.

Moreover, when standardised and automated, continuous monitoring helps identify irregularities or variations early in the process. This early detection allows for immediate corrective actions, reducing downtime, minimising waste, and guaranteeing that critical food safety treatments are applied correctly [8•,46,47]. This proactive approach aids in maintaining optimal production conditions and streamlining operations. In addition, the vast usage of NIR spectroscopy in the context of process analytical technology (PAT) has set the basis for what has been defined as 'the second green analytical revolution of NIR spectroscopy analysis' [6•].

Given the significant benefits and potential offered by NIR spectroscopy technology in process monitoring and control, it becomes highly desirable to make more effort to extend the integration of this technology into standard methods within the food sector. As reported (Table 1), nowadays, only one standard from ISO is available for the dairy sector.

The challenges that NIR spectroscopy has to face within the food sector have been well highlighted in recent papers [48–50], and these challenges basically refer to i) the natural and high variability of complex products such as foods and ingredients; ii) some inherent limitations of the technique, such as the strong absorption caused by water or the limited sensitivity; and iii) its indirect nature and the linked chemometric-related issues, for example, models optimisation, stability, and transfer. Nonetheless, here we want to stress the attention on the aspects of education and knowledge. In fact, even considering some limitations, NIR spectroscopy as an analytical technology is now very mature reaching, from the hardware point of view, outstanding performances (e.g. detectors, resolution, wavelength selection and accuracy, ruggedness, signal-to-noise ratio, etc.). On the other hand, knowledge of NIR spectroscopy and chemometric basis is still a major bottleneck within the food sector. For someone, it's kind of 'magic', able to measure almost everything, from minor compounds in ppm or even ppb concentration to any kind of properties. The side effect of this poor knowledge is that, when negative results are obtained, these are thought of as a failure of the technology itself and not a misunderstanding and improper use of it. Also, lack of knowledge about the basic concepts of multivariate analysis of NIR data undermines the rational and responsible use of NIR spectroscopy, as well as the full understanding of its potentiality and limitations.

Final remarks

The benefits of a vast application of NIR spectroscopy within the food sector (production, research, quality control, process monitoring) are clear and include both the 'mere' technical aspects and the 'public' ones. However, without official documents issued by recognised bodies, that are fundamental tools and guarantees for all the food operators, those advantages would not be fully exploited.

As final remarks, we would suggest some points and challenges that deserve special consideration to further implement NIR spectroscopy technology in official regulations:

1) Researchers must handle the fact that for quantitative purposes, the existing regulations regarding the concentration thresholds of specific compounds must be complied with and always fulfilled.
2) There must be full disclosure and a comprehensive evaluation of the analytical figures of merit (e.g. limit of detection) that any analytical method requires to be declared as valid by the regulatory bodies.
3) When multivariate/machine learning/chemometrics is used, it is especially relevant to provide a proper, exhaustive, and accurate validation procedure that demonstrates the predictability and reliability of the measurements and the results obtained.
4) There must be a clear physicochemical explanation of the correlation found between the NIR spectra and the measured compounds or properties. As it is well known, causality is not correlation. Direct or indirect correlations will be perfectly valid in a predictive

model only when the cause of that correlation is understood and it is demonstrated to be reproducible and stable enough.

Upon the fulfilment of these points, we believe that an NIR/Chemometric method could become attractive enough for regulatory bodies to consider it for official development within the food sector.

CRediT authorship contribution statement

Giacomo Squeo: Conceptualization, Investigation, Data curation, Writing – original draft, Writing – review & editing, Visualization. **Jordi Cruz:** Investigation, Data curation, Writing – original draft, Writing – review & editing, Visualization. **Davide De Angelis:** Investigation, Data curation, Writing – original draft, Writing – review & editing, Visualization. **Francesco Caponio:** Resources, Writing – review & editing, Visualization. **José M Amigo:** Conceptualization, Investigation, Writing – review & editing, Supervision.

Data Availability

No data were used for the research described in the article.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

Giacomo Squeo, Davide De Angelis, and Francesco Caponio acknowledge that this research was carried out within the Agritech National Research Center and received funding from the European Union NextGenerationEU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR) — MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.4 — D.D. 1032 17/06/2022, CN00000022). This manuscript reflects only the authors' views and opinions, neither the European Union nor the European Commission can be considered responsible for them.

This work was also supported in part by collaboration with REDTPI4.0-320RT0006 CYTED program. José M Amigo thanks the Innovation Fund Denmark for supporting this research under 'HyperSort — Hyper-spectral imaging for advanced textile sorting and food control' project. Besides, this research has been supported by the Elkartek Programme, Basque Government (Spain) (SMART-EYE, KK-2023/00021).

References and recommended reading

Papers of particular interest, published within the period of review, have been highlighted as:

- • of special interest
- •• of outstanding interest

1. IOC: Trade standard on olive oils and olive-pomace oils. *Standard COI/T.15/NC No 3/ Rev.19/2022*; 2022. Available online: ⟨https://www.internationaloliveoil.org/wp-content/uploads/2022/12/Norme-comerciale-REV-19_ENK.pdf⟩ [Accessed 3 May 2024].

2. Cozzolino D: **The ability of near infrared (NIR) spectroscopy to predict functional properties in foods: challenges and opportunities**. *Molecules* 2021, **26**:6981, https://doi.org/10.3390/molecules26226981

3. Grassi S, Casiraghi E: **Advances in NIR spectroscopy analytical technology in food industries**. *Foods* 2022, **11**:1250, https://doi.org/10.3390/foods11091250

4. Aouadi B, Zaukuu JLZ, Vitális F, Bodor Z, Fehér O, Gillay Z, Bazar G, Kovacs Z: **Historical evolution and food control achievements of near infrared spectroscopy, electronic nose, and electronic tongue — critical overview**. *Sensors* 2020, **20**:5479, https://doi.org/10.3390/s20195479

5. Ozaki Y, Morisawa Y: **Principles and characteristics of NIR spectroscopy**. In *Near-Infrared Spectroscopy*. Edited by Ozaki Y, Huck C, Tsuchikawa S, Engelsen SB. Springer Nature Singapore Pte Ltd.; 2021:11-35.

6. • Sørensen KM, van den Berg F, Engelsen SB: **NIR data exploration and regression by chemometrics — a primer**. In *Near-Infrared Spectroscopy*. Edited by Ozaki Y, Huck C, Tsuchikawa S, Engelsen SB. Springer Nature Singapore Pte Ltd.; 2021:127-189.
This chapter offers a very useful overview about the data structure, the data preprocessing, and some of the possible supervised and unsupervised multivariate techniques that could be applied to NIR spectroscopy data.

7. Amigo JM: **Data mining, machine learning, deep learning, chemometrics: definitions, common points and trends (Spoiler Alert: VALIDATE your models!)**. *Braz J Anal Chem* 2021, **8**:45-61, https://doi.org/10.30744/brjac.2179-3425.AR-38-2021

8. • Solberg LE, Wold JP, Dankel K, Øyaas J, Måge I: **In-Line Near-Infrared spectroscopy gives rapid and precise assessment of product quality and reveals unknown sources of variation — a case study from commercial cheese production**. *Foods* 2023, **12**:1026, https://doi.org/10.3390/foods12051026.
The work presented a very interesting application of NIR spectroscopy for food process monitoring and control.

9. Tonolini M, van den Berg FW, Skou PB, Sørensen KM, Engelsen SB: **Near-infrared spectroscopy as a process analytical technology tool for monitoring performance of membrane filtration in a whey protein fractionation process**. *J Food Eng* 2023, **350**:111487, https://doi.org/10.1016/j.jfoodeng.2023.111487

10. Esbensen KH, Abu-Khalaf N: **Before reliable near infrared spectroscopic analysis — the critical sampling proviso. Part 1: Generalised theory of sampling**. *J Infrared Spectrosc* 2022, **30**:291-310, https://doi.org/10.1177/09670335221124612

11. • Esbensen KH, Abu-Khalaf N: **Before reliable near infrared spectroscopic analysis — the critical sampling proviso. Part 2: Particular requirements for near infrared spectroscopy**. *J Infrared Spectroc* 2022, **30**:311-321, https://doi.org/10.1177/09670335221124611.
The work highlights the fundamental importance of proper sampling procedures when dealing with NIR spectroscopy.

12. AACC 39-70.02: Near-infrared reflectance method for hardness determination in wheat. *AACC Approved Methods of Analysis (11th ed.)*. ⟨https://www.cerealsgrains.org/resources/Methods/Pages/39InfraredAnalysis.aspx⟩ [Accessed April 15, 2024].

13. ISO 21543:2020 | IDF 201:2020: Milk and milk products. *Guidelines for the Application of Near Infrared Spectrometry*. ⟨https://www.iso.org/standard/77606.html⟩ [Accessed May 6, 2024].

14. ISO 23291:2020 | IDF 248:2020. Milk and milk products. *Guidelines for the Application of In-line and On-line Infrared Spectrometry*. ⟨https://www.iso.org/standard/75145.html⟩ [Accessed May 6, 2024].

15. AOAC 2007.04. Fat, Moisture, and Protein in Meat and Meat Products. ⟨https://irp-cdn.multiscreensite.com/f96e2517/files/uploaded/Parico-FoodScan-AOAC-Summary-of-test-results.pdf⟩ [Accessed April 30, 2024].

16. Squeo G, De Angelis D, Summo C, Pasqualone A, Caponio F, Amigo JM: **Assessment of macronutrients and alpha-galactosides of texturized vegetable proteins by near infrared hyperspectral imaging**. *J Food Compos Anal* 2022, **108**:104459, https://doi.org/10.1016/j.jfca.2022.104459

17. Cruz-Tirado JP, dos Santos Vieira MS, Amigo JM, Siche R, Barbin DF: **Prediction of protein and lipid content in black soldier fly (*Hermetia illucens* L.) larvae flour using portable NIR spectrometers and chemometrics**. *Food Control* 2023, **153**:109969, https://doi.org/10.1016/j.foodcont.2023.109969

18. Chapman J, Elbourne A, Truong VK, Newman L, Gangadoo S, Pathirannahalage PR, Cheeseman S, Cozzolino D: **Sensomics-from conventional to functional NIR spectroscopy-shining light over the aroma and taste of foods**. *Trends Food Sci Technol* 2019, **91**:274-281, https://doi.org/10.1016/j.tifs.2019.07.013

19. Sun D, Cruz J, Alcalà M, Romero del Castillo R, Sans S, Casals J: **Near infrared spectroscopy determination of chemical and sensory properties in tomato**. *J Infrared Spectrosc* 2021, **29**:289-300, https://doi.org/10.1177/09670335211018759

20. Huck CW: **New trend in instrumentation of NIR spectroscopy — miniaturization**. In *Near-Infrared Spectroscopy*. Edited by Ozaki Y, Huck C, Tsuchikawa S, Engelsen SB. Springer Nature Singapore Pte Ltd.; 2021:193-210.

21. da Silva Medeiros ML, Cruz-Tirado JP, Lima AF, de Souza Netto JM, Ribeiro APB, Bassegio D, Godoy HT, Barbin DF: **Assessment oil composition and species discrimination of *Brassicas* seeds based on hyperspectral imaging and portable near infrared (NIR) spectroscopy tools and chemometrics**. *J Food Compos Anal* 2022, **107**:104403, https://doi.org/10.1016/j.jfca.2022.104403

22. Kappacher C, Trübenbacher B, Losso K, Rainer M, Bonn GK, Huck CW: **Portable vs. benchtop NIR-sensor technology for classification and quality evaluation of black truffle**. *Molecules* 2022, **27**:589, https://doi.org/10.3390/molecules27030589

23. Fulladosa E, Barnés-Calle C, Cruz J, Martínez B, Giró-Candanedo M, Comaposada J, Font-i-Furnols M, Gou P: **Near infrared sensors for the precise characterization of salt content in canned tuna fish**. *Spectrochim Acta Pt A Mol Biomol Spectrosc* 2023, **303**:123217, https://doi.org/10.1016/j.saa.2023.123217

24. Ríos-Reina R, Callejón RM, Amigo JM: **Feasibility of a rapid and non-destructive methodology for the study and discrimination of pine nuts using near-infrared hyperspectral analysis and chemometrics**. *Food Control* 2021, **130**:108365, https://doi.org/10.1016/j.foodcont.2021.108365

25. Houhou R, Bocklitz T: **Trends in artificial intelligence, machine learning, and chemometrics applied to chemical data**. *Anal Sci Adv* 2021, **2**:128-141, https://doi.org/10.1002/ansa.202000162

26. Mishra P, Passos D, Marini F, Xu J, Amigo JM, Gowen AA, Jansen JJ, Biancolillo A, Roger JM, Rutledge DN, Nordon A: **Deep learning for near-infrared spectral data modelling: hypes and benefits**. *TrAC Trends Anal Chem* 2022, **157**:116804, https://doi.org/10.1016/j.trac.2022.116804

27. Totaro MP, Squeo G, De Angelis D, Pasqualone A, Caponio F, Summo C: **Application of NIR spectroscopy coupled with DD-SIMCA class modelling for the authentication of pork meat**. *J Food Compos Anal* 2023, **118**:105211, https://doi.org/10.1016/j.jfca.2023.105211

28. Nagy MM, Wang S, Farag MA: **Quality analysis and authentication of nutraceuticals using near IR (NIR) spectroscopy: a comprehensive review of novel trends and applications**. *Trends Food Sci Technol* 2022, **123**:290-309, https://doi.org/10.1016/j.tifs.2022.03.005

29. Moghaddam HN, Tamiji Z, Lakeh MA, Khoshayand MR, Mahmoodi MH: **Multivariate analysis of food fraud: a review of NIR based instruments in tandem with chemometrics**. *J Food Compos Anal* 2022, **107**:104343, https://doi.org/10.1016/j.jfca.2021.104343

30. Yan H, De M, Géa, Neves I, Noda GM, Guedes ACS, Ferreira F, Pfeifer X, Chen HW, Siesler: **Handheld near-infrared spectroscopy: state-of-the-art instrumentation and applications in material identification, food authentication, and environmental investigations**. *Chemosensors* 2023, **11**:272, https://doi.org/10.3390/chemosensors11050272

31. Casson A, Beghi R, Giovenzana V, Fiorindo I, Tugnolo A, Guidetti R: **Visible near infrared spectroscopy as a green technology: an environmental impact comparative study on olive oil analyses**. *Sustainability* 2019, **11**:2611, https://doi.org/10.3390/su11092611

32. Grassi S, Jolayemi OS, Giovenzana V, Tugnolo A, Squeo G, Conte P, De Bruno A, Flamminii F, Casiraghi E, Alamprese C: **Near infrared spectroscopy as a green technology for the quality prediction of intact olives**. *Foods* 2021, **10**:1042, https://doi.org/10.3390/foods10051042

33. Simanjuntak LE, Soesilo TEB, Herdiansyah H: **Analysis of near infra red (NIR) impact as the control process of a palm oil mill and refinery plant for environmental sustainability**. *Pol J Environ Stud* 2020, **29**:3013-3021, https://doi.org/10.15244/pjoes/115210

34. Gullifa G, Barone L, Papa E, Giuffrida A, Materazzi S, Risoluti R: **Portable NIR spectroscopy: the route to green analytical chemistry**. *Front Chem* 2023, **11**:1214825, https://doi.org/10.3389/fchem.2023.1214825

35. Shi M, Zheng X, Zhang N, Guo Y, Liu M, Yin L: **Overview of sixteen green analytical chemistry metrics for evaluation of the greenness of analytical methods**. *TrAC Trends Anal Chem* 2023, **166**:117211, https://doi.org/10.1016/j.trac.2023.117211

36. • Saveliev M, Panchuk V, Kirsanov D: **Math is greener than chemistry: assessing green chemistry impact of chemometrics**. *TrAC Trends Anal Chem* 2024, **172**:117556, https://doi.org/10.1016/j.trac.2024.117556.

This work, in a very useful way, highlights and measures how NIR spectroscopy and chemometrics are greener than common wet chemistry methods.

37. • Vitale R, Cocchi M, Biancolillo A, Ruckebusch C, Marini F: **Class modelling by soft independent modelling of class analogy: why, when, how? A tutorial**. *Anal Chim Acta* 2023, **1270**:341304, https://doi.org/10.1016/j.aca.2023.341304.

This work provides an excellent theoretical and practical background for the application of SIMCA, one of the simplest although efficient multivariate methods for food authentication.

38. De Angelis D, Summo C, Pasqualone A, Faccia M, Squeo G: **Advancements in food authentication using soft independent modelling of class analogy (SIMCA): a review** (in press.), *Food Qual Saf* 2024,fyae032, https://doi.org/10.1093/fqsafe/fyae032

39. Mendez J, Mendoza L, Cruz-Tirado JP, Quevedo R, Siche R: **Trends in application of NIR and hyperspectral imaging for food authentication**. *Sci Agropecu* 2019, **10**:143-161.

40. Allegrini F, Olivieri AC: **Linear or non-linear multivariate calibration models? That is the question**. *Anal Chim Acta* 2022, **1226**:340248, https://doi.org/10.1016/j.aca.2022.340248

41. Walsh J, Neupane A, Koirala A, Li M, Anderson N: **Review: the evolution of chemometrics coupled with near infrared spectroscopy for fruit quality evaluation. II. The rise of convolutional neural networks**. *J Infrared Spectrosc* 2023, **31**:109-125, https://doi.org/10.1177/09670335231173140

42. Lopez E, Etxebarria-Elezgarai J, Amigo JM, Seifert A: **The importance of choosing a proper validation strategy in predictive models. A tutorial with real examples**. *Anal Chim Acta* 2023, **1275**:341532, https://doi.org/10.1016/j.aca.2023.341532

43. Dhanapal L, Erkinbaev C: **Portable hyperspectral imaging coupled with multivariate analysis for real-time prediction of plant-based meat analogues quality**. *J Food Compos Anal* 2024, **126**:105840, https://doi.org/10.1016/j.jfca.2023.105840

44. McClements DJ, Weiss J, Kinchla AJ, Nolden AA, Grossmann L: **Methods for testing the quality attributes of plant-based foods: meat-and processed-meat analogs**. *Foods* 2021, **10**:260, https://doi.org/10.3390/foods10020260

45. Pu YY, O'Donnell C, Tobin JT, O'Shea N: **Review of near-infrared spectroscopy as a process analytical technology for real-time product monitoring in dairy processing**. *Int Dairy J* 2020, **103**:104623〈https://doi.org/10.1016/j.idairyj.2019.104623〉.

46. Wold JP, O'Farrell M, Tschudi J, Eskildsen CE, Andersen PV, Ottestad S: **In-line and non-destructive monitoring of core temperature in sausages during industrial heat treatment by NIR interaction spectroscopy**. *J Food Eng* 2020, **277**:109921, https://doi.org/10.1016/j.jfoodeng.2020.109921

47. Grassi S, Giraudo A, Novara C, Cavallini N, Geobaldo F, Casiraghi E, Savorani F: **Monitoring chemical changes of coffee beans during roasting using real-time NIR spectroscopy and**

chemometrics. *Food Anal Methods* 2023, **16**:947-960, https://doi.org/10.1007/s12161-023-02473-w

48. Biswas A, Chaudhari SR: **Exploring the role of NIR spectroscopy in quantifying and verifying honey authenticity: a review**. *Food Chem* 2024, **445**:138712, https://doi.org/10.1016/j.foodchem.2024.138712

49. Shawky E, Nahar L, Nassief SM, Sarker SD, Ibrahim RS: **Spice authentication by near-infrared spectroscopy: current advances, limitations, and future perspectives**. *Trends Food Sci Technol* 2024, **148**:104522, https://doi.org/10.1016/j.tifs.2024.104522

50. Jia W, Georgouli K, Martinez-Del Rincon J, Koidis A: **Challenges in the use of AI-driven non-destructive spectroscopic tools for rapid food analysis**. *Foods* 2024, **13**:846, https://doi.org/10.3390/foods13060846