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Current Opinion in Food Science

Advances in NIR spectroscopy applied to process analytical technology in food industries

Silvia Grassi and Cristina Alamprese

Process analytical technology (PAT) in food industries can improve process efficiency and final product quality by enhancing understanding and control of the manufacturing processes. Near infrared spectroscopy (NIRS) is one of the predominant e-sensing technologies used in PAT, thanks to its ability in fingerprinting materials and simultaneously analyzing different food-related phenomena. Recent advances have shown good potentials of NIRS in real-time monitoring and modeling of different food processes. However, most studies have been carried out at a lab scale, while applications at industrial levels are still few. To bridge the gap between NIRS potentials and its actual implementation in PAT, more efforts are requested to both researchers and industries in order to close the control loop for an efficient and automated processing management.

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Introduction

This paper discusses recent advances (published in 2015–2017) in near infrared spectroscopy (NIRS) applied to process analytical technology (PAT) in food industries. Prominent applications at lab and industrial scale are reported.

Real-time evaluation and assurance of the process efficiency and final product quality based on real-time process data can represent a great benefit for food industries and PAT implementation can facilitate this approach. The advantages for the company's business, meant as growth in profit margins and production efficiency, are a clear key driver of PAT application in food industries together with consumers' demand for high and consistent quality of the final products, as well as requirements by control bodies for food safety and traceability [1]. Recently, the attention for a higher environmental sustainability of food processes has been individuated as the fourth driver involved in PAT application; PAT can actually be recognized as a green production strategy, optimizing the efficient use of resources [2].

PAT approach was firstly introduced by FDA for pharmaceutical industries, as 'a system for designing, analyzing, and controlling manufacturing through timely measurements (i.e., during processing) of critical quality and performance attributes of raw and in-process materials and processes, with the goal of ensuring final product quality. The goal of PAT is to enhance understanding and control the manufacturing process' [3]. As reported in the interesting review by van den Berg *et al.* [4], PAT can be considered 'a silent revolution in industrial quality control in food processing'. Within PAT, quality control turns from a feedback approach to a model-predictive approach based on real-time process adjustment during manufacturing. Besides the clear advantages in product quality assurance and process management, a successful implementation of PAT enables also a deep understanding and a continuous learning about food materials and process dynamics, paving the way for innovations through a Quality by Design (QbD) approach. QbD can be considered a systematic way of food development based on the pre-definition of critical quality characteristics that can be designed by an accurate and sound process understanding and control. Taking into account the strict regulatory environment in which the food industry acts as well as the consumers' requirements, the effective use of modeling and control strategies can also help in ensuring food safety, authenticity, and quality, while lowering production costs and increasing energy efficiency. However, the extremely heterogeneous and varying properties of raw materials, the complex transformations that can occur during the processing chain, and the perishable nature of the products increase the challenges in this scenario.

The PAT system has four main components (Figure 1): initial understanding of relevant factors affecting the process dynamics and the final product quality, process analysis, multivariate data analysis, and process control. This review will focus on recent NIRS applications for the monitoring of critical process parameters and quality attributes, moving progress in multivariate data analysis to the background. Chemometrics is of course of

Figure 1

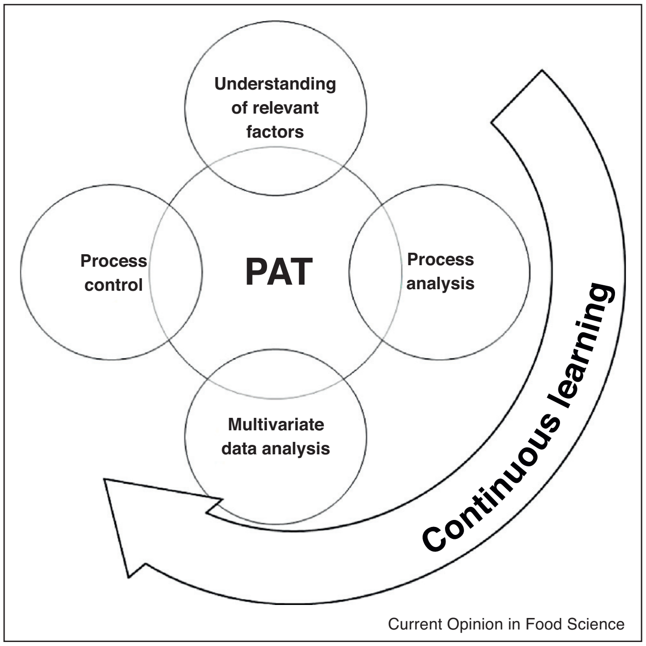

Illustration of the four main components of process analytical technology, aimed at enabling a deep understanding and a continuous learning of food materials and process dynamics.

fundamental importance for PAT, but a detailed survey of the advances in data analysis science is out of the scope of this review. Some of the most important univariate and bivariate parametric and non-parametric statistical techniques are reviewed by Granato *et al.* [5], whereas useful qualitative and quantitative multivariate approaches are reported in several recent reviews [6–10]. Chemometrics has improved the whole control process by reducing the time of analysis and providing more informative results [11]. It can be applied to PAT at different levels: for the design of experiments in order to screen and optimize the critical parameters to be considered in the process control [8,12,13]; for process control [14–16]; for both regression and classification modeling in order to predict simultaneously several critical quality attributes from the real-time collected data [8,13]; for handling data structures from multiple analytical platforms [8,11,17].

Spectroscopic sensors are optimal instruments for real time analysis during manufacturing, being rapid, non-invasive, very flexible, and rugged. NIRS, in particular, with its ability to fingerprint food materials and to simultaneously analyze different phenomena, is one of the predominant e-sensing technologies used in PAT. Its spreading is also favored by the possibility to transport radiation through optical fiber probes and by the growing availability of low-cost portable devices, which can be more easily implemented into the processing line.

Process analysis

Most of the published researches deal with the use of NIRS in process analysis, including characterization of raw materials, as well as intermediate and final products. The complexity and high variability of food systems and the dynamic nature of food processing together with the large number of interconnected factors affecting the outcomes are main challenges for PAT implementation in food industries.

Recent developments of NIRS in the field of liquid foods are covered by Wang *et al.* review [18•], focusing on the detection of quality attributes and adulterations of alcoholic beverages (red wines, rice wines, and beer), nonalcoholic beverages (juice, fruit vinegars, coffee beverages, and cola beverages), oils (vegetable, camellia, peanut, and virgin olive oils and frying oil), and dairy products (milk and yogurt). Dairy industry is the object also of the review by Munir *et al.* [19••], with a focus on milk powder, for which not only composition is important, but also technological performance (e.g., particle size and dispersibility), sensory and microbiological attributes. A more comprehensive review about QbD for food processing has been published by Rathore and Kapoor [20], considering case studies in the field of both vegetable and animal products.

As regards the dairy products, a representative work has been published by Melenteva *et al.* [21], who proposed a global model for the spectrophotometric (400–1100 nm) determination of fat and total protein content in raw cow milk. A very large set of milk samples (>1000) collected during a whole year were analyzed, taking into account also geographical, genetic, and breeding management factors, as well as a milk storage period up to 24 h (at $5 \pm 1\,^{\circ}C$). Moreover, the authors proposed some approaches for the model transfer between two different instruments.

At a lab scale, NIRS has been largely used for the monitoring of fermentation processes, because it can give simultaneously information about chemical composition, textural properties and microbial growth. Some good recently published reviews report results about the application of NIRS in wine and brewing industries [22•,23••]. In these fields, the control of raw material quality (e.g., compositional, phytosanitary, genetic), processing operations (e.g., mashing and fermentation) and final product quality can be successfully achieved by NIRS and multivariate data analysis. A recent paper by Svendsen *et al.* [24] applied a NIR fiber optic reflectance probe for in-line control of yoghurt fermentation in a large lab scale (15 L fermenter). By means of principal component analysis (PCA) and kinetic modeling, they were able to model both texture changes due to the gel formation and chemical information related to the sugar conversion into lactic acid performed by microbial starters.

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The usefulness of PAT in process optimization has been proved also for fruits and vegetables drying, one of the most energy consuming unit operation in postharvest processing. Raponi *et al.* [25] reviewed literature from 1999 onwards exploring NIRS, multi-spectral and hyper-spectral vision systems application in quality control of fruit and vegetable drying processes. Recently, Moscetti *et al.* [26] reported the benefits of NIRS applied to carrot drying process monitoring. They focused on physico-chemical changes of carrot slices during hot-air drying forerun by hot-water blanching. They demonstrated the efficacy of the spectroscopic system (1100–2300 nm) in predicting water activity, moisture content, soluble solid content, total carotenoids and color changes during drying through partial least squares (PLS) regression models optimized by variable selection through an interval PLS (iPLS) algorithm. Furthermore they applied PLS-Discriminant Analysis (PLS-DA) for the classification of carrot slices according to the drying phases, obtaining models with good accuracy in prediction (>75%). However, the industrial transfer of the presented approach requires a more robust validation together with the increment of the instrumentation performances in terms of light-beam intensity and wavelength range covered.

Some interesting applications have been suggested also for coffee roasting [27], honey refining [28], dispersed ground oilseed concentration in diluted solid–liquid dispersions [29], and meat composition and grading [30,31]. However, it is worth noting that the majority of the cited works were carried out at a lab scale, and, even if they show a great potential of NIRS applied to PAT for food processing, a real implementation in food industries can present additional challenges.

From process analysis to process control

Despite the abundant scientific literature demonstrating the great potential of NIRS applied to process analysis in view of PAT implementation, real case study reports about on-line/at-line process analysis at industrial scale are still few. It should be considered that the requirements for on-line technology application in industrial production differ from laboratory-based analysis. Indeed, the NIR instruments should ensure not only the ability in measuring the parameter(s) of interest, but should also enable an appropriate feedback speed, a non-invasive character and a cleaning-in-place compatibility.

Porep *et al.* [32] and Beghi *et al.* [33] described the application of visible/near infrared spectroscopy for an on-line evaluation of crushed grapes and phytosanitary status at the receipt station of wineries. Calibration PLS-DA and PLS models for different relevant parameters (e.g ., diseased bunches, density, fructose, glucose, and organic acid content, acidity, pH) were developed, which may improve payment systems and quality management. However, due to the coarse qualitative or semi-quantitative prediction achieved, further studies are necessary in order to optimize and validate the models. Moreover, in view of a final on-line application, the best operating conditions and the engineering phases to perform the measurements directly at the grape receipt should have been assessed.

An industrial scale-up of NIRS applications is more evident in the monitoring of fermentation processes. Among the published papers, Vann *et al.* [34••] evaluated the potential of NIRS for on-line monitoring of beer fermentation firstly at lab scale (26.5 L fermenter) and then in a 300 L pilot-scale plant for validation. The models developed for sugar consumption rate, ethanol production rate, yield of ethanol on total sugars and fermentation lag-time were then incorporated into a feed-forward control strategy for yeast management. This strategy was able to early detect shifts in fermentation performances and consequently adjust yeast re-pitching rates in order to improve batch-to-batch consistency.

A large-scale (approximately 1000 kg) experimental mill was used by Allouche *et al.* [35] for monitoring olive malaxation by an Acousto-Optic Tunable Filter (AOTF)-NIR equipment. In particular, real-time characterization of olives (pulp/stone ratio, extractability index, moisture and oil contents) and the potential characteristics of the extracted oil (free fatty acids, peroxide value, UV parameters, pigments and polyphenols) were evaluated, considering different months and years of olive harvesting, as well as different processing conditions (i.e., types of hammer mill, sieve diameter, hammer rotation speed, and temperatures). The use of an artificial neural network (SS-ANN) allowed reaching good predictive capability, showing the possibility to obtain almost instantaneously the information needed to optimize process conditions in terms of both productivity and oil quality, despite the high variability of the raw material and the dynamic conditions of the spectra collection. The acquired knowledge can be used for the implementation of a process-automation system able to regulate different processing variables with minimal loss of time and costs.

The advantages of real-time measurements, modeling and control in food processing are well illustrated by Glassey *et al.* [36] through the discussion of different industrial case studies. In particular, the authors demonstrated that NIRS might be used to improve the consistency of dry ingredient mixing in food industries. Considering bread and confectionery powder mixtures with different particle size distributions, experiments were performed using two conical screw mixers of 4000 L capacity, each equipped with a NIR diffuse reflectance fiber-optic probe. Both powder homogeneity and composition were successfully predicted by applying a suitable NIR data elaboration. The obtained results were then validated using a tumble blender with a nominal capacity

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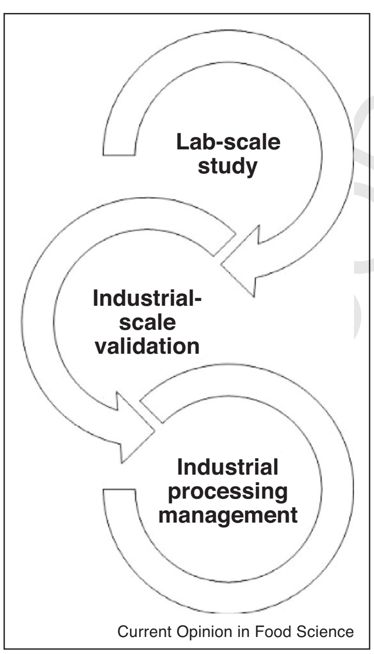
of 2000 L. Being the plant in motion, a Wi-Fi and battery powered portable device (MicroNIR) was used for spectra acquisition. The validation produced good results, allowing the identification of the homogeneity point. A further experiment was carried out for caramel and custard processing, where mixing of mainly water and sugar at high temperatures leads to high density and high viscosity products. A 2000 L mixing and cooking vessel was used, applying to the recirculation pipe a NIR probe connected to a MicroNIR. Good PLS calibration models with high coefficients of determination and low errors were obtained for color, moisture, and water activity. However, the predictions for an unknown batch were not very accurate. In any case, since only three batches were used for calibration, the models can be improved by analyzing more production batches.

Conclusions

Process-automation systems can really help food industries in improving process efficiency, meanwhile satisfying law requirements and consumers' needs. The complexity of a food process represents a challenge for PAT implementation, but NIRS can be successfully exploited in this field. However, the recent scientific literature is still too much focused on studies carried out at a laboratory level, just demonstrating the potential of NIRS in understanding, modeling and monitoring food-related phenomena. Some manufacturers are integrating spectroscopic sensors into processes, but only as substitutes for traditional off-line analytical procedures. This can lead to a confused perception of PAT.

To bridge the gap between NIRS potentials and its actual implementation in PAT tools, more efforts are requested to both researchers and industries. A good dissemination and a closer collaboration are needed in order to transfer the process analysis carried out at a lab-scale into an industrial process control, closing the loop for an efficient and automated processing management (Figure 2). Resistance to change must be overcome by food industries, as well as deeper statistical knowledges and management skills must be transferred to the future generation of food technologists. Only in this way the 'PAT silent revolution' might be accomplished, with important implications for food producers and consumers.

Figure 2



Scheme for the development of an efficient and automated processing management tool.

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Conflict of interest

The authors declare no conflict of interest.

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- • of special interest
- •• of outstanding interest

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