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Near infrared spectroscopy for food and feed: a mature technique

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The technique has gained pride of place in the food and feed sectors

ndoubtedly, for food and feed specialists, near infrared (NIR) spectroscopy is considered an essential analytical tool that has contributed greatly to the quality and safety enhancement of their products. Moreover, for many years it has been implemented with success at different stages of the food and feed production chains, allowing not only gains in speed of analysis but also larger analytical throughputs. For example, NIR spectroscopy is used to analyse and segregate raw materials entering the factory, to prevent unsatisfactory loads from entering the food or feed chain. to redirect raw material batches to more profitable process options and for payment of agro-food products based on quality parameters (e.g., in the cereal sector). In addition, NIR spectroscopy assists engineers in different production processes by providing suitable in-line, on-line and at-line solutions that can help to maximise company profits by detecting problems and reacting as fast as possible to adjust the process if required.¹

NIR spectroscopy techniques are also used in many other areas such as chemistry, petrochemistry, pharmacy, environmental and spatial sciences, biology, mineralogy, archaeology, and art. All these applications have contributed to create an energetic community, involving specialists from different areas, proposing innovative technologies and plug-and-play analytical solutions for specific challenges to help producers, processors, distributors, retailers, controllers and consumers.

In the last 35 years, NIR spectroscopy has become a technique of choice for food and feed analysts and also for researchers. For the scientific community, for instance, NIR spectroscopy is used for the non-destructive monitoring of complex reactions as well as in projects where the analytical issue is not the final goal but the technique is implemented in order to maximise the quantity of information collected by unit of time (or let us say by euro) and to try to address appropriately the sampling issues. An indicator of the success of NIR spectroscopy technology is the continuously increasing number of companies proposing NIR spectroscopy solutions at any level (in a recent survey more than 60 of them have been identified and we know that we are not at all exhaustive).

Great progress has been made in 25 years

In order to illustrate the progress made by the NIR community in the food and feed areas, it is interesting to take the example of the history of the determination of protein content by NIR spectroscopy. This determination is maybe one of the first performed in the history of NIR applications and today one of the parameters (with moisture content) which is most widely determined by NIR spectroscopy. Since then, NIR spectroscopy has been expanded and become the "reference" method for very diverse products. However, and before having a look at some of the achievements made in the last decades, it is worth mentioning that the main principle of NIR spectroscopy technology remains unchanged during these decades!!! This means the interaction between photons and matter. When comparing a spectrum taken today with a spectrum collected 25 years ago, it is amazing to observe that we are still playing with the same information. It is true that the photon



Figure 1. On-line NIR probe and sampling device installed at a feed reception stage (courtesy of Provimi, France. Q-Saffe European project).

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Figure 2. On-line NIR sensor installed in feed process (Courtesy of Dumoulin, Belgium. Projet FIRST Dumoulin-CRA-W).

information is collected faster and easier now, but no tremendous improvements in terms of signal-to-noise ratio and sensitivity in the NIR spectral information have been made for decades.

Over the last 25 years, the most tremendous developments and innovations have been seen in the construction of rugged and compact instruments equipped with adequate and easy-to-use sample presentation devices, internal automatic control systems as well as friendly interfaces both to collect and to manipulate the data. The generalisation of NIR networks (including instruments from different generations and manufacturers) and the availability of large databases for the determination of composition is a matter of fact. It would be interesting to calculate the positive impact in terms of carbon emission reduction of the use of NIR spectroscopy. Innovations can be found also in the field of sensor technology through miniaturisation and portability placing the instrument closer and closer to the sample. The concept of bringing the laboratory to the sample is replacing the classical concept of bringing the sample to the laboratory. Today, satellites, combine harvesters and production lines are already built with embedded NIR sensors. Tomorrow, farmers will have unmanned vehicles equipped with NIR and other sensors to detect any stress or disease in their crops and to allow interventions locally; processors will have at their disposal robotic equipment equipped with NIR sensor(s) to adequately (in terms of sampling issues) check internally the quality of cereal

cargoes in boats, trucks, silos or process lines; and consumers will have at their disposal NIR smartphones equipped with an NIR imaging system to assess the composition and energy intake of their daily dishes. ... Unrealistic? ... Maybe not!

Another recent achievement of the NIR spectroscopy community in the field of protein content determination is the development of international standards and guidelines specific for the determination of such a parameter by NIR spectroscopy (i.e. 15048 and 12099). These documents, in combination with the fact that more and more laboratories are making the determination of protein content by NIR spectroscopy under accreditation (ISO 17025), are contributing greatly to the acceptance of this technology by official bodies. This is a key point since recent crises, such as melamine contamination in feed and in food products, have highlighted that classical methods used in official controls have serious limitations when it comes to properly quantifying protein content in foods or food ingredients. The official methods in place are principally indirect methods (based on determination of N content and multiplication by a factor in order to estimate the protein content) and were fooled by unscrupulous people; NIR protein content determination is based on the absorption of NIR photons by the sample constituents (including proteins). Because of the use of a suitable NIR method in an environment under the supervision of an effective quality system, it seems that the melamine crisis has not had a major impact in the feed sector.² In a similar way, the use of NIR spectroscopy for the detection of untargeted mislabelling or contaminants at the port of entry of raw material is being realised.^{3,4} Other examples could be chosen to illustrate the progress of NIR spectroscopy for protein analysis, such as the determination of protein at the single kernel level by NIR imaging, the application of this technique for protein content determination through the packaging in cheese or for the selection of bone fragments rich in collagen in archaeological samples in order to rationalise ¹⁴C and DNA analysis.⁵ Of course, these examples are biased by our ongoing projects, but give an idea of the NIR evolution in the protein content determination.

What about the future?

For the next 25 years, food specialists have some expectations in terms of NIR

development. To answer the forthcoming challenges and the increasing level of sophistication of food, integrated NIR systems combined with spatial analysis techniques (microscope, camera), sampling systems and other confirmatory techniques (PCR, immunosensor etc.) will improve quality, safety, authenticity and processing issues in the food and feed sectors. It is also expected that innovative technology will be used in order to get smaller and low-cost handheld instruments (including imaging systems) with enhanced signal-to-noise ratios and with reduced spectral response differences between units of instruments from the same manufacturer. As an example, a fully integrated solution has been recently launched by Aunir (http://www.nir4farm. com) based on a VIAVI® spectrometer weighing 60 grams and large spectral databases. The device offers an analytical tool to farmers and farm advisers to improve feed efficiency.

As we are witnessing the emergence of new sensors based on alternative spectroscopic techniques (mid-infrared, Raman, terahertz, nuclear magnetric resonance etc.), it could be interesting to define the right places and the complementarity of NIR spectroscopy to these newcomers. There is also a great interest in the development of NIR-based sensors for gas analysis. NIR spectroscopy is quite restrictively used to analyse solid, slurries and liquids. However, the development of a gas NIR sensor to control the status of food and feed products during storage, or the follow-up of the gas phase during processing could be of great importance.

In terms of software, it would be great if all software would include uncertainty in the expression of prediction results and would be flexible in term of exportation and importation of data (this is not something new in the wish list of NIR spectroscopy users!). In terms of data treatment, there are several justifications in terms of cost, reduction of volume of toxic reagent used and reduction in the number of reference values to calibrate the spectrometers.^{6,7}

We have a dramatic need for more basic research studies allowing us to understand better the interaction between NIR radiation and material. We know that the difficulty is to get financial support for such studies. A similar trend appears with sampling studies involving NIR techniques.... However, we know that the future complete endorsement

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of NIR spectroscopy methods is dependent on these studies.

Finally, as education is the keystone in the education-research-innovation triangle, we have to reinforce the presence of NIR spectroscopy in graduate and master degree courses. There is a lag between the ubiquitous use of NIR spectroscopy in the food and feed industries and its optional inclusion in High-School and University programmes.

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