ROUNDTABLE

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Moderator: **Dr Ir Vincent Baeten**, Head of the Food and Feed Quality Unit, Walloon Agricultural Research Center

What are the perspectives of vibrational spectroscopy techniques in the untargeted detection of mislabelling and contaminants in food and feed chains? Are we able to detect the next melamine crisis?

Piotrowski: NIRS is a valuable tool in the detection of organic substances. If a suitable calibration is available for the substance, NIRS is capable of detecting it. The main challenge is the speed at which a new calibration can be set up. Creating a suitable database can take time. Close links between the food/feed supply chains and quality control partners should be maintained to identify and highlight critical control points in processing so that monitoring steps can be put in place to avoid such crises.

Caneda: Vibrational spectroscopies have a host of advantages for the control of mislabelling and contaminants in food. They are fast, require little/no sample preparation and are hygienic. The biggest challenge is that with contaminants the detection levels are very low. For example the FDA has established a 1ppm limit for melamine in milk formula. NIR is best where speed is important, although very robust calibration models are required to detect contaminants at trace levels. Raman can be used for detection down to ppb and ppt levels using SERS but requires sample preparation. In conclusion, these techniques are ideal to deal with high volume of samples accurately and quickly. They do however require a serious and rigorous calibration procedure with strong field validation.

Nørgaard: In the complex food supply chain there are three main areas to be concerned with: adulteration of incoming raw material, process deviations and mishaps, and deviations from end-product quality. Vibrational spectroscopy – with mid-IR and NIR targeting different sample matrices – matches the requirements to an analytical platform which is capable of detecting the yet unknown adulterants or contaminants in a high sample-throughput industrial setting. Mid-IR-based global untargeted models for raw milk analysis are examples of already commercially available global applications. The untargeted mid-IR or NIR approach should always be considered a screening tool which alerts to the need for further investigations to determine the nature of the abnormality. So yes, we have the methods to screen for the next melamine crisis!

Do you think that vibrational spectroscopy is the right method for official controls? What is the place of such technique in official lab?

Caneda: The demands placed on testing labs are only going to increase as more food with stricter controls require ever more testing. This means that the throughput of samples in official labs will go on increasing. With traditional techniques, the sample preparation is often very time consuming and requires costly reagents and consumables, this is a bottleneck. It is also an environmental issue to use ever more chemicals. Vibrational techniques like NIR and Raman can take raw samples and test them in an unadulterated state. Currently, specific chemical lab tests are the gold standard for this kind of testing but we strongly believe that the advantages of sampling speed and cost enabled by modern techniques like NIR and Raman, will bring a sea change for how this testing is approached in the years to come. Chemical will become the way to validate and calibrate higher throughput instruments using vibrational spectroscopy.

Nørgaard: Vibrational spectroscopy can be used for screening of adulterants or contaminants in feed and food products – also in the official control laboratory. Due to the non-invasive property and very short analysis time it is a powerful screening technique with an excellent cost-benefit level compared to other techniques. However, it is important to recognise that a positive result provided by any screening method always has to be verified by primary reference methods – and such methods are often readily available in the official control laboratory. The Food and Drug Administration has introduced a regulatory framework for Process Analytical Technology implementation boosting vibrational spectroscopy for control in the pharmaceutical industry, and the same routines are now seen in the food segment.

Piotrowski: There are numerous advantages to using NIRS. As long

as the material being measured is organic and the levels present in the sample are high enough to overcome the noise of the instrument, NIRS can be used in an official capacity. Wet chemistry will always be necessary but NIRS allows more frequent analysis which can help to identify changes to expected results quicker and more cost-effectively than with wet chemistry alone.

What is your strategy to set up the right sampling for heterogeneous samples?

Nørgaard: For liquid samples, where mid-IR spectroscopy based methods are a natural choice, the analytical sampling is of course much easier to perform than for powder or solid samples. In a new solution for wheat and barley whole grain grading we utilise the power of vision based analysis, both in the visual and NIR spectral range, to overcome the sampling problem by analysing every single seed in the sample. In this solution it is possible to classify 10,000 single seeds for 10-15 defects in around three minutes – a truly impressive system for analytical sampling of heterogeneous samples.

Piotrowski: There are three factors to consider with sampling; the frequency, the technique used and the ability to replicate the sample. It is very important to be as consistent as possible when sampling. At Aunir, we have standard sampling procedures we recommend for our clients to ensure that sampling is carried out as frequently as is efficient, and that any changes in personnel do not effect results.

Caneda: Food and feed samples are almost invariably heterogeneous so the sampling is always a priority. In the real world, users need an accurate and representative result of the sample and inhomogeneous samples are the big challenge. Our strategy is to use dynamic techniques that allow the spectrometer to collect many spectra over a large sample (the speed of NIR being key here). Then using averaging you can produce a result that is accurate, representative and much closer to the real product or process you are trying to control. This is also a critical part of producing high quality calibrations for NIR. Different standard accessories can be used for the sample whether it is liquid, solid, or slurry. Alternatively we can develop custom solutions.

What are the key criteria of good spectroscopy software?

Piotrowski: Aunir is a leader in the development and supply of NIRS solutions. We pride ourselves on having a large dataset with multiple

mathematical transformations, plus good varietal, seasonal and geographical spread of the samples in it. Aunir is dedicated to providing calibrations so we are constantly updating our database and adjusting the software to ensure the most accurate calibrations are on offer to customers.

Caneda: Software is the hardware integrator. Without a user friendly design, all the advantages of the sensing and sampling hardware are lost. The first level is to bring it all together and display clean and clear spectra that can control the measurement set up and allow users to quickly verify the result. Then you need functionality to help automate the sampling and repeatability of the process. The next step is to integrate the modelling and calibration routines. Finally you need fast and efficient saving and storing of current and historical data. Ideally we'd like to see the industry adopt common formats that can be used more interchangeably, helping to build up more data to create better and more accurate calibration models.

Nørgaard: Good spectroscopy software is essential for realising the solution's full potential to create value to the users. The software must be dedicated with respect to application development and offer seamless integration with databases, and instrument and networking software. The software should provide options to identify, to qualify and to quantify – IQ^2 – the sample in question in a logical way with efficient cutting-edge chemometric algorithms. As an example, an identification model can determine if we have skimmed milk powder or whole milk powder in the process pipe or if the sample is deviating from these groups. The qualification model zooms into the actual product – often through a tighter model – and finally quantification of composition can be performed confidently.


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5,000 scans per second. Use of this technology is predominantly in the beverage and oil industry at present but this will be used increasingly for solids on fast moving production lines in the near future.

About the participants

Chris Piotrowski has worked in the NIR industry since 1978. His early years were spent gaining knowledge and skills as an analytical chemist. By the late 1980s he was tasked with introducing an NIR network to the 10 feed mills of J Bibby & Sons. Following this successful implementation he returned to running both the analytical and NIR divisions of Central Laboratories. In 1995 Central Laboratories was set up as a commercial laboratory serving the food and feed industries. The laboratory grew rapidly covering a diverse range of techniques, including analytical chemistry, microbiology and NIR spectroscopy. In 2008 the decision was made to sell the analytical division of Central Laboratories to AB Agri allowing Chris and the team to focus on the core activity of NIR under the new name of Aunir.

Argentinian **Gustavo Caneda**, a chemical engineer, spent his early career working in industrial processing plants. In 1991 he discovered the exciting possibilities afforded by the power of NIR spectroscopy. This led him to invest his time developing strong experience in chemometric modelling and NIR quality and process control, including as a trainer and consultant to companies worldwide. In 2001 he founded TecnoCientifica, creating his own line of bench top and On-Line NIR instruments using Ocean Optics technology. In 2014 Gustavo joined Ocean Optics to add his experience and help the company to further develop dedicated Industrial Solutions using NIR, UV-Vis and Raman spectroscopies.

Lars Nørgaard is Senior Manager, PhD, Affiliate Professor at FOSS and Head of Team Chemometric Development at FOSS Product Innovation. He holds a Master's of Science in Chemical Engineering and a PhD in chemometrics and analytical chemistry. He has more than 20 years of research and teaching experience within chemometrics and spectroscopy for food-agri and pharmaceutical applications. Before joining FOSS in 2010 Lars was head of Department of Food Science at University of Copenhagen, and he was appointed affiliated professor at University of Copenhagen in 2011.

What will be the next revolution in NIR spectroscopy?

Nørgaard: FOSS believes that the next revolution in NIR spectroscopy, among other things, will address the difficulties in analysing heterogonous samples. With, for example, hyperspectral NIR cameras it will be possible to predict the chemical composition for a large number of small subsamples without sacrificing speed and in addition decrease the limit of detection for adulterants and contaminants. Also the next generation of process analysers will make it possible to provide much more confidence into feed and food manufacturing.

Caneda: In terms of the technology, there is definitely a drive for smaller spectrometers that can be integrated into more handheld, consumer and medical devices. For the food industry this is a tremendous opportunity for the identification and authentication of ingredients and quality control from the finished goods warehouse to the supermarket aisle. The idea that anyone could have a handheld NIR spectrometer to check the quality of their food in restaurants or at home is getting closer to reality than many might think.

Piotrowski: There are several revolutions on the NIRS horizon that we can see. Everyone is constantly striving for improved accuracy – this will continue to improve in the coming months and years. The use of NIRS will increase as industries realise the value that it can deliver. Hand-held NIR devices are coming onto the market which enables NIRS to be used in more and more portable situations. In conjunction with this, automatic scanning on production lines will become standard for a wider range of measurable factors. Acoustic Optical Turnable Spectra (AOTS) technology is being developed that allows