Close-up of a sample tube Toebes is placing in the NMR machine.

FrieslandCampina Innovation Centre recently commissioned a new Nuclear Magnetic Resonance machine to analyze the structure of 'bound', 'entrapped' and 'free' water in dairy products. The manufacturer originally designed this NMR machine for the petrochemical industry, but modified it for use in the food industry.

By Vincent Hentzepeter | Photos by Foodnote | Translation by Word's Worth

Even before FrieslandCampina consolidated all R&D activities in its innovation center in Wageningen, FrieslandCampina Research in Deventer already had a low-resolution NMR machine. It was used as a measuring instrument to determine the fat content in cheese, solid fat content and water droplet size distribution in butter. Before the move to Wageningen, Antoinette Toebes, Research Specialist | R&D Food Structuring at FrieslandCampina Innovation Centre, had gained several years' experience working with this piece of equipment. When manufacturer Oxford Instruments announced it would stop servicing the machine, it was clear that the R&D department had to start looking for a replacement. This was far from easy, Toebes recalls. "We had a whole list of requirements, but couldn't find a system that even came close to meeting them. Most systems are designed for a specific application, while we were looking for an all-round machine. Fortunately, Oxford Instruments was prepared to modify an existing system they had designed for the oil industry to fit our needs. In 2017, FrieslandCampina became the first food manufacturer to use this machine."

Antoinette Toebes, Research Specialist | R&D Food Structuring at FrieslandCampina Innovation Centre:

'We can now carry out 2D NMR'

2D NMR

FrieslandCampina invested in Oxford Instruments' MQR NMR, a low-resolution, high-performance Time Domain (TD) NMR, sold and serviced in the Netherlands by Salm en Kipp. This system is the manufacturer's first-generation MQR NMR with a 23 MHz magnet, says Toebes. "One of the things that makes this system special is the optional imaging software supplied by Green Imaging Technology, which helps us interpret our NMR data. Their GIT App Builder, which allows you to build your pulse sequences and set your parameters, has been expanded with 2D maps and saturation profile add-ons. This enables us to carry out 2D NMR, which teaches us so much more, particularly about water in our systems. For example, how is water distributed in a system or product? What happens to the water during processing? Is the way in which water is distributed in the product linked to certain functionalities? Our new machine is very much research-oriented. Oxford Instruments and Green Imaging Technology are more than willing to cooperate when we're researching new applications, so we can keep finding new applications."

'2D NMR teaches us so much more, particularly about water in our systems'

Antoinette Toebes, Research Specialist | R&D Food Structuring at FrieslandCampina Innovation Centre, at Oxford Instruments' high-performance Time Domain (TD) NMR, sold by Salm en Kipp.

Modeling

One example of the type of research FrieslandCampina conducts is its modeling of the micelle of lactic acid casein. Casein is present in milk in the form of micelles, submicroscopic particles that consist of a collection of large molecules. These micelles are made up of submicelles, which in turn are made up of an aggregate of several casein protein chains. The diameter of a micelle is variable, but averages about 100 nanometers. Toebes' research department took the studies published by Huppertz et al, 2017 (Huppertz, T., Gazi, I., Luyten, H., Nieuwenhui se, H., Alting, A., & Schokker, E. (2017). Hydration of casein micelles and caseinates: Implications for casein micelle structure. *International Dairy Journal*, 74, 1-11) as its guiding principle for testing. "This model was put to the test using NMR by making bound, entrapped and free water measurable. We want to apply the same principle to cheese (coagulation), yogurt and milk powders."

Development

Although most TD-NMR research work characterises water properties, studying fat is also of interest. Toebes' department goes beyond the well-known standard determinations of bulk fat and solid fat content. "We're increasingly able to determine the solid fat content of products that also contain disruptive ingredients like water and non-fatty solids." Over the past year, the opportunities the new NMR system offers have enabled the researchers to cross over into development as well, Toebes says. The development appli-cations include the determination of solid fat content in dairy toppings, the water distribution in cheeses and monitoring the cheese coagulation process. "We've learned so much. More and more people are embracing technology and are no longer daunted by the complicated terminology associated with proton spins. And we're also learning more about how to interpret the data and the processes behind them. My advice would be: if you're facing an issue involving water in systems, consider using low-resolution NMR. It provides so many new, valuable insights when dealing with research questions!"

Benchtop NMR

Benchtop Nuclear Magnetic Resonance (NMR) is increasingly being used for both applied research and routine applications. The benchtop models are affordable and user-friendly, as opposed to classic NMR equipment with huge magnets used only in high-tech labs and requiring a PhD to operate. Low-resolution NMR enables labs to screen large numbers of samples for certain characteristics, without the need for much sample preparation. The technology is non-destructive, so other—chemical—analyses can follow NMR. For example, it is possible set up an experiment with temperature profiles, in which the probe is either heated or cooled to imitate processing conditions and to study how structure and composition change.