Raman spectroscopy for measurement of solid dairy powders

Benefits at a glance

- Ensuring consistent quality of dairy solids during processing has importance in consumer goods and nutritional supplements
- Composition measurements needed to ensure product quality are primarily performed in the laboratory
- Raman spectroscopy provides a simultaneous, rapid, and non-contact measurement of fats, proteins, and carbohydrates in dairy solid products
- Raman-based measurements are used in laboratory, at-line quality assurance, or in-line process control applications

Introduction

Milk powders are an important food. The final dried milk powder can either be consumed on its own or as an ingredient in other foods or beverages. The global milk powder production reached over 11 million tons in 2020 from increased demand of traditional and new milk powder products. Two consumer trends impact production of milk powder: changing consumer demands and intense interest in more personalized nutrition. Growing consumer interest in healthy foods with a tailored nutritional profile has spurned intense growth in the production of milk powders. There is also increased demand for a variety of nutritional profiles such as low lactose powders, high protein powders, or hybrid milk/plant-based products. Higher consumer demand and increased number of products has put a strain on traditional manufacturing and testing capabilities in order to keep up with the number of products as well as the high volume of milk powders.

Benefits of Raman spectroscopy

Many food manufacturers are adopting Industry 4.0 and automation principles to achieve manufacturing flexibility and gains in plant efficiency. Integration of at-line or in-line process analyses are a critical component in achieving these gains because the real-time information provides 24/7 monitoring, enables in-process corrections, and rapidly brings process knowledge building on new manufacturing approaches. These process analyses typically include physical parameters such as flow, pressure, and temperature as well as chemical parameters such as pH and moisture.

Traditionally, more advanced composition measurements were performed in the laboratory. However, this approach has drawbacks including the turnaround time, increased contamination risk, and non-representative sampling. Raman spectroscopy is an optical analysis tool that can address the specificity and representative sampling requirements for milk solids analysis, providing a fast (<2 minute) and non-contact reading of fat, protein, and carbohydrates in milk powders with a single measurement. A large volume sampling probe is ideal for focus-free measurements of solids during processing, and is proven in process operations such as drying, crystallization, and blending.

Results and discussion

Solid milk powders with various concentrations of fats, carbohydrates, and proteins, as shown in Table 1, were obtained and analyzed as received. All milk powder samples were analyzed in a laboratory using a solids-optimized Raman Rxn embedded analyzer (λ=785 nm). The analyzer was equipped with a large volumetric Raman probe, providing a 6 mm spot size. The probe was positioned 25 cm from the samples, providing a non-contact, focus-free, and representative sampling with a single measurement. Raman measurements of the milk powders were collected for 2 minutes directly from open containers or loosely packed in a plastic container.

ⓘ All Raman analyzers and probes referenced in this application note are Endress+Hauser products powered by Kaiser Raman technology.

Sample ID	Fat (mg/100g)	Carbohydrates) (mg/100g)	Proteins (mg/100g)
Powder 1	0.3	2.5	90
Powder 2	1.7	27	57
Powder 3	25.1	53.7	9.8
Powder 4	30	41	12

Table 1. Nutritional overview of macronutrients found in four milk powders as provided on the consumer label.

Figure 1 shows representative Raman spectra from four different milk powders. Bands on the spectra arising from lipids (or fats), proteins, and carbohydrates were identified using known literature values.[1,2] Importantly, bands from the individual components are well resolved and enable robust univariate or multivariate models for quantification. While there is spectral overlap of the constituents, the noted areas in the spectra can be used to monitor changes in macronutrients. In the example of the 1620-1800 cm^{-1} region, a more narrow shape of the carbonyl stretch envelope at ~ 1650 cm^{-1} and the emergence of relatively narrow bands at ~ 1740 and 1301 cm^{-1} indicates a preponderance of lipids in Powders 3 and 4. More intense and broader bands in the 1040-1120 cm^{-1} region is indicative of a higher carbohydrate amount in Powders 3 and 4. By contrast, a broader carbonyl stretch envelope at ~ 1650 cm^{-1} and a narrow ring breathing band at ~ 1005 cm^{-1} indicates a higher presence of proteins in Powders 1 and 2.

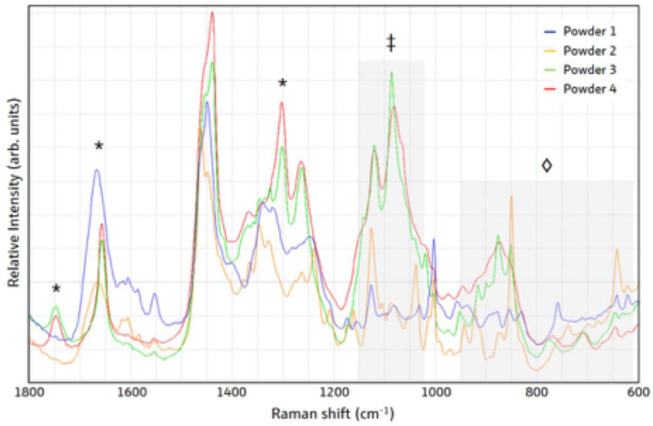

Figure 1. Raman spectra of milk powder samples 1-4 have contributions from lipids (*), carbohydrates (‡), and protein amino acids (◊).

Conclusions

Milk powders provide convenient and tailored nutrition to support the needs of infant formulas, sport supplements, and geriatric supplements. The ability to rapidly measure macronutrients at-line or in-line supports the industry's need for increased plant efficiency and production of diverse products. Raman spectroscopy is well suited for measuring milk powders because it provides a highly specific, multi-component measurement in a process or laboratory environment. The specificity of Raman spectroscopy enables simple visual inspection or spectral library comparison for qualitative insight into the relative amounts of macronutrients. Raman's specificity also enables a robust univariate or multivariate analytical model for lab-to-process quantification without needing major scale-specific model updates. The large volume sampled by the Raman probe ensures a representative sampling of heterogenous solids without needing to refocus the probe on various spots of the sample. These technological aspects of our Raman technology, in addition to our 30+ years of experience in lab-to-process Raman spectroscopy, provide the benefit of real-time and around-the-clock monitoring of milk solids production.

References

1. Koenig, J. L. Raman Spectroscopy of Biological Molecules: A Review. J. Polym. Sci. Macromol. Rev. **1972**, 6 (1), 59–177.
2. De Gelder, J.; De Gussem, K.; Vandenabeele, P.; Moens, L. Reference Database of Raman Spectra of Biological Molecules. J. Raman Spectrosc. **2007**, 38 (9), 1133–1147. https://doi.org/10.1002/jrs.1734.