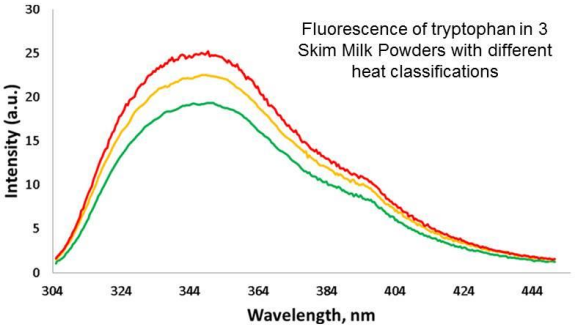
Project number: 6463
Funding source: DAFM 11/F/052

Date: Jan 2018
Project dates: Nov 2012- Jan 2018

Fluorescence spectroscopy for quality control in dairy ingredient and infant formula manufacture



Multivariate data analysis allows manufacturers to use the large quantities of information present in fluorescence spectra for monitoring important quality parameters.

Key external stakeholders:

Dairy ingredient manufacturers, infant formula manufacturers, dairy ingredient end-users

Practical implications for stakeholders:

Quality control is a key concern for manufacturers and end-users of dairy ingredients. This project has demonstrated the feasibility of using fluorescence spectra in combination with chemometric modelling for:

- Product specification control – spectral data can be collected and analysed instantaneously instead of relying on time-consuming, lab based analytical methods
- Quality control of received ingredients– spectral data can discriminate between ingredients which provides dairy ingredient end users with validation that the correct ingredient has been received or, used in a recipe
- Distribution chain control: spectral data is sensitive to storage temperature and can potentially be used to determine if temperature abuses have occurred during transportation

Main results:

- Dairy Ingredients:
  - Fluorescence spectra is capable of discriminating between skim milk powder (SMP) , demineralized whey powder (DWP) and whey protein concentrate (WPC) powder with 100% accuracy
  - Soluble protein content can be predicted in SMP and WPC 35 with an accuracy of 2.9g/100g powder
- Infant formula:
  - Models developed using tryptophan emission spectra for infant formula powder predicted storage time, pre-drying heat treatment temperature and soluble protein content with RMSECV values of 0.3 months, 8.3 °C and 1.01g protein /100 g powder, respectively

Opportunity / Benefit:

Process analytical tools are of growing interest to the Irish dairy processing industry. The results of this study show that fluorescence spectroscopy has strong potential as the basis for a successful process and quality analytical tool. The project consisted of spectral and physicochemical analysis of large quantities of dairy materials and the significant expertise generated is available for interested parties.

Collaborating Institutions:

UCD

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External collaborators: Prof. Colm O'Donnell, UCD

1. Project background:

Dairy ingredients and infant milk formula (IMF) manufacturers constantly strive to improve the quality and nutritional composition of their products. As a result, quality specifications are extremely high and meeting these demands can be challenging due to variability in compositional, environmental and processing parameters which can alter underlying physicochemical properties thereby affecting product quality. Typically, dairy manufacturers use time-consuming analytical techniques to determine effect of certain parameters on product quality. However, the timeframe in which results become available does not allow important process interventions to be made in real time. Therefore, in-line process analytical technology (PAT) which can rapidly capture data and allow for real-time control of critical process parameters is of extreme interest to processors. PAT tools are already extensively employed in the pharmaceutical sector; they allow manufacturers to produce materials of consistent quality and also help to reduce waste & overall costs. Recent developments in vibrational spectroscopy and data analysis have significant potential in food process applications and it is the goal of this project was to evaluate fluorescence spectroscopy and in particular front facing fluorescence spectroscopy (FFFS) for use as a PAT tool in dairy ingredient and infant formula manufacture.

2. Questions addressed by the project:

- Can FFFS be applied as a PAT tool in the infant formula distribution chain i.e. from manufacture of primary dairy ingredients all the way through to the distributor/consumer of finished infant formula products?
- What key product characteristics can be measured by FFFS?
- How do these key characteristics relate to processing decisions?
- Can fluorescence based analysis be employed as a rapid at-line tool in the manufacture of dairy ingredients and infant formula?

3. The experimental studies:

The project consisted of 5 distinct experimental phases, which investigated the use of the following PAT tools:

1. FFFS in dairy ingredient manufacture
2. FFFS in model infant formula manufacture
3. FFFS for discrimination between brands and storage conditions in commercial infant formula products
4. FFFS for compositional control in commercial infant formula
5. Rapid fluorescence based analyser for determination of soluble protein content in infant formula and dairy ingredients

4. Main results:

- The potential of FFFS coupled with chemometrics as a PAT tool was successfully applied to discriminate between dairy ingredients during manufacture (skim milk powder, whey protein concentrate and demineralised). It was possible to use spectral data to distinguish between different process conditions (e.g. heat treatment temperatures) and to monitor protein denaturation and Maillard browning
- FFFS simultaneously estimated pre-drying heat treatment temperature and storage time while also predicting soluble protein and surface free fat content during manufacture of model infant formula powders. Partial least squares – discriminant analysis (PLS-DA) was successfully applied to identify powders as a function of temperature applied during storage.
- Models developed using tryptophan emission spectra for infant formula powder predicted storage time, pre-drying heat treatment temperature and soluble protein content with RMSECV values of 0.3 months, 8.3 °C and 1.01g protein /100 g powder, respectively
- Successful models were developed which allowed for prediction of fat content and pH in commercial

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infant formula powders.

- Rapid at-line fluorescence analysis was demonstrated to be an accurate technique to measure key product and quality attributes such as whey protein nitrogen index and soluble protein. Results were highly correlated (R^2 > 99 %) with the more time consuming reference methods indicating the suitability of the method.

5. Opportunity/Benefit:

The project demonstrated the suitability FFFS for IMF and dairy ingredient quality control. For manufacturers this represents an opportunity to improve product quality and specification control thereby avoiding costly out-of-specification batches. In the case of IMF, this is of extreme importance due to potential impacts of poor quality products on highly sensitive end-users. For exporters/importers of products, the potential use of FFFS PAT techniques to identify and discriminate between products can be used to verify product authenticity and remove counterfeit product from the market. Ultimately, use of the PAT methodologies developed in this project will be beneficial to end-users/consumers as the cumulative effect across the distribution chain will result in better, safer products.

6. Dissemination:

Presentations & workshops:

1. Henihan, L., O'Donnell, C. and O'Callaghan, D. The effects of heat treatment on Model Infant Formula powder using front-face fluorescence spectroscopy 43rd Annual Food Research Conference, UCD, Dublin Ireland (10 – 11 December 2014)
2. Henihan, L., O'Donnell, C. and O'Callaghan, D. Use of front-face fluorescence spectroscopy for analysing the effects of heat treatment on rehydrated skim milk powder 9th NIZO Dairy Conference Milk Protein Functionality. Papendal, Netherlands. (30 September 2015 – 02 October 2015)
3. Henihan, L., O'Donnell, C. and O'Callaghan, D. Analysing the effects of heat treatment on dairy powders & infant formula. IDF Concentrated & Dried Milk Products Symposium, Dublin (11-13 April 2016)
4. Henihan, L., O'Donnell, C. and O'Callaghan, D. Predictive modelling of pre-heat treatment, protein denaturation and Maillard browning of skim milk powders using front-face fluorescence spectroscopy. Society of Dairy Technology Conference - UCC, Cork (10-12 April 2017)
5. A workshop was organised at Teagasc, Moorepark, on 12/3/15 for dissemination of FIRM-funded projects in conjunction with the Society of Dairy Technology. Two presentations were made by members of the project team, and there was a demonstration of a novel fluorescence based analyser.

Main publications:

1. Henihan, L. E., O'Donnell, C. P., Esquerre, C., Murphy, E. G., & O'Callaghan, D. J. (2018). Quality assurance of model infant milk formula using a front-face fluorescence process analytical tool. Food and bioprocess technology, 11(7), 1402-1411.
2. Henihan, L. E., O'Donnell, C. P., Esquerre, C., Murphy, E. G., & O'Callaghan, D. J. (2019). Fluorescence-based analyser as a rapid tool for determining soluble protein content in dairy ingredients and infant milk formula. Innovative food science & emerging technologies, 52, 75-79.
3. Henihan, L. E., O'Donnell, C. P., Esquerre, C., Murphy, E. G., & O'Callaghan, D. J. (2018). Comparison of front-face fluorescence spectroscopy and fourier transform infrared spectroscopy as process analytical tools in dairy ingredient and infant formula manufacture. Biosystems and food engineering research review 23, 57.

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