Project number: 6728
Funding source: Enterprise Ireland
Date: June 2020
Project dates: Jan 2015 – Dec 2019

Process analytical technologies (PAT) for control and monitoring of dairy concentrates in dairy ingredient manufacture.

Key external stakeholders:
Dairy industry,
Dairy ingredient manufacturers,
Infant formula manufacturers,
Food manufacturers.

Practical implications for stakeholders:
Process analytical technologies (PAT) allow inline, real-time monitoring of process variables (e.g. pressure, flow, temperature and viscosity) and provide dairy processors with accurate and instantaneous information about their process which can ultimately aid in optimisation of the process. Viscosity of dairy concentrates in particular, influences both process performance and quality of the final dairy powder. Current lab based rheological methodologies used to measure viscosity do not always generate results that represent the inline (in-pipe) viscosity of a concentrate. These off-linerheological results can vary depending temperature, flowrate, shear history, storage of the concentrate, concentrate composition, heat load applied before concentration and physico-chemical behaviour of the material (age thickening). Therefore, this highlights the importance of adoption and installation of inline PAT tools for measuring inline process viscosity in real time.

Main results:

Three inline PAT instruments were evaluated for monitoring process viscosity of a range of dairy concentrates i.e. skim milk concentrate (SMC), milk protein concentrate (MPC) and fat filled concentrate (FFC).

Inline Coriolis flowmeter: Non-linear regression was applied to the experimental data to successfully transform the dynamic viscosity output into commonly-used reference viscosity values using a hygienic multivariate flowmeter for measuring process viscosity of skim milk concentrate (with a total solid (TS) content ranging from 10-40%) at 25^{o}C.

Acoustic PAT instruments were also evaluated for measuring process viscosity:

Inline acoustic flowmeter: was evaluated for inline monitoring of MPC under the following conditions, 10-21% TS at 45^{o}C and FFC at pilot scale.

Inline acoustic sensor: was investigated for monitoring process viscosity of vegetable oil at 25^{o}C as well as SMC at laboratory scale at a range of different TS (10-42%) and under an operating temperature of 25^{o}C.

Opportunity / Benefit:

Outputs from this project include significant experience and knowledge of commercially available inline process instruments for measuring viscosity that can assist the Irish Dairy Industry in selecting suitable PAT for measuring dairy concentrate viscosity, which will aid in ensuring final powder quality. The results of this project highlighted the importance of measuring process viscosity inline and in real time.

Collaborating Institutions:

UCD, DPTC

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1. Project background:

The objective of this project was to review developments in process analytical technology (PAT) which have relevance for product quality in the manufacture of dairy powders. The role of process analytics in process optimisation and best manufacturing practice was piloted in the manufacture of dairy powders. The project had the following objectives:

- Assessment of the commercial and emerging process viscometers used for process monitoring in the production spray-dried dairy ingredients.
- Laboratory evaluation of a process monitoring technology for industry implementation to facilitate improved consistency and process efficiencies.
- Laboratory evaluation of a second process monitoring technology, which implemented in industry, will facilitate improved consistency and process efficiencies.
- Demonstration/case study at pilot/plant level in partnership with industry of second developed/evaluated process monitoring technology.

2. Questions addressed by the project:

- What process analytical technologies are commercially available that can be used for measuring and monitoring process viscosity and in particular have the following:
  - Hygienic design,
  - Appropriately pressure rated,
  - Have accreditation e.g. EHEDG / 3A design,
  - Cost effective,
  - Antifouling,
  - Highly sensitive for detecting changes in the process parameter,
  - Rapid and continuous output signal of the process parameter,
  - Can operate independent of the process environment e.g. independent of plant vibration / high process temperatures and are can be cleaned in place (CIP).
- Identification of PAT used for measuring process viscosity in other industries e.g. paint / lubricant and ink industries.
- What emerging PAT are available that can be used for measuring and monitoring process viscosity.

3. The experimental studies:

1. Laboratory evaluation of a low-cost bulk acoustic wave (BAW) sensor for measuring process viscosity of skim milk concentrate.
2. The lab and pilot scale evaluation of a torsional Coriolis flowmeter for measuring inline viscosity of skim milk concentrate.
3. The lab scale evaluation of a surface acoustic wave (SAW) multivariate flowmeter for measuring process viscosity and total solids content of milk protein concentrates.
4. Installation and evaluation of a SAW flowmeter operating at pilot scale (which operated full time in Moorepark Technology Limited for 11 months) for the evaluation of a fat filled concentrate.

4. Main results:

Study 1 major findings:

- A solid-state bulk acoustic wave sensor was used to measure the viscosity and temperature of skim milk concentrate under static and flow conditions.
- Reference viscosity (mPa.s) derived from a flow curve (generated from a rotational rheometer) is

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most commonly used in industry therefore; a mathematical relationship between reference viscosity and acoustic viscosity was developed, so that viscosity can be converted from one unit to the other.

- Non-linear regression was also applied to the reference and acoustic viscosity data and the regression model had an R^2 (coefficient of determination) of 0.99, indicating that acoustic viscosity readings acquired from the acoustic wave sensor can be converted accurately into reference viscosity values.

Study 2 major findings:

- An inline Coriolis flowmeter was used to measure the dynamic viscosity of both a Newtonian fluid (10-60% w/w sucrose solutions) and a non-Newtonian fluid (10-40% w/w skim milk concentrate) at laboratory scale at 25°C.
- Validation of the instrument for measurement of a non-Newtonian fluid was completed at pilot-scale under similar operating conditions.
- Calibration equations developed for sucrose solutions and skim milk concentrates viscosity at laboratory scale demonstrated a good fit to predict with reference viscosity values (R^2 = 0.99) and (R^2 = 0.99) respectively

Study 3 major findings:

- An acoustic flowmeter was evaluated for monitoring viscosity and total solids (TS) content of milk protein concentrate (MPC85) indirectly by correlating with inline parameters i.e. acoustic transmission (AT) and acoustic impedance (AI) respectively.
- A strong negative correlation (r = -0.985) was obtained between inline acoustic transmission and offline apparent viscosity (adjusted R^2 value = 0.97 and standard error of prediction = 1.86 mPa.s).
- A positive correlation (r = 0.97) was observed between acoustic impedance measured using the inline flowmeter and TS measured offline.

Study 4 major findings:

- This study gave an insight into the performance of a hygienic acoustic flowmeter to measure concentrate flow behaviour i.e. solids, viscosity, changes in flow behaviour, as a result of potential changes downstream in real time, at pilot scale.
- No installation issues were observed in the year it was installed in MTL.
- The flowmeter was easy to clean making it CIP able and robust.
- During the entire year of installation, there were no issues with vibration from the process effecting the measurement and stable readings were observed.
- The data obtained was repeatable under similar processing conditions demonstrating good overall repeatability of the instrument.

5. Opportunity/Benefit:

- The real time measurement of process parameters i.e. viscosity and total solid content using these PAT tools allows for optimised and efficient dairy powder manufacturing.
- All instruments and sensors discussed in this technology update are available to be used as part of a collaborative research projects.
    - In particular, the ability of these PAT tools to measure viscosity of additional dairy concentrates e.g. whey protein concentrate and infant formula, would be of interest to investigate.
- The PAT tools are also available to be hired as part of research / company trials, this will allow companies to understand the flow behaviour of their material in real-time and under process conditions.
- These PAT tools have applications in measuring process viscosity of other dairy products e.g. yogurt, UHT milk.

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6. Dissemination:

Main publications:

- O'Shea, N., O'Callaghan, T. F., & Tobin, J. T. (2019). The application of process analytical technologies (PAT) to the dairy industry for real time product characterization-process viscometry. *Innovative Food Science & Emerging Technologies*, *55*, 48-56.
- Bista, A., Hogan, S. A., O'Donnell, C. P., Tobin, J. T., & O'Shea, N. (2019). Evaluation and validation of an inline Coriolis flowmeter to measure dynamic viscosity during laboratory and pilot-scale food processing. *Innovative Food Science & Emerging Technologies*, *54*, 211-218.
- Pu, Y., O'Shea, N., Hogan, S. A., & Tobin, J. T. (2020). Assessment of a solid-state bulk acoustic wave sensor to measure viscosity of Newtonian and Non-Newtonian fluids under static and flow conditions. *Journal of Food Engineering*, *277*, 109917.

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