



15F683 - Future proofing the Irish cheese-whey industry – a step change process to move Irish dairy commodities up the value chain.

Final Report

SUMMARY

The STEPUP project focused on optimisation of serum protein removal from milk using ceramic and polymeric microfiltration approaches. The project identified discrepancies in the reported literature relative to the efficiency of microfiltration of milk and subsequent serum protein depletion which actually falls within the range of 50-70% on a total serum protein basis. The project has provided end users such as the dairy industry and others skilled in the art with a comprehensive mass and energy balance providing a quantitative benchmark of microfiltration performance and serum protein partition efficiency. The STEPUP project has provided a robust mass and energy balance for optimal separation of serum proteins (whey) from milk which can be scaled to industrial installations.

The project provides insights into strategies for improving the efficiency of cheddar and swiss type cheese manufacture by standardization of both the protein content and profile by microfiltration without negatively impacting the flavour profile of the subsequent cheese produced. Overall, the research has shown that there is no impediment to manufacture of cheeses from micellar casein concentrates when standardised with cream and concentrated milk permeates in comparison to cheese milk standardised in a conventional way. This facilitates the removal of much of the native whey from milk in advance of cheese manufacture while retaining a conventional cheese manufacture and ripening process.

The nutritional quality of the whey produced by microfiltration and from cheese making using whey depleted recombined milks has also been assessed whereby altered protein profiles and amino acid contents have been observed related in particular to altered ratios of threonine linked to either absence or enrichment of casein macropeptide, which is of interest to whey ingredient suppliers and nutritional formulators.

KEYWORDS

Cheese, Dairy, Microfiltration

ACRONYM

STEPUP

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November 2022

Section 1 - Research Approach & Results

Start Date

01 February 2017

End Date

31 January 2022

Research Programme

Food Institutional Research Measure

TRL Scale

TRL 5: Technology validated in relevant environment

NRPE Priority area

Sustainable Food Production and Processing

Total DAFM Award

€599,000.00

Total Project Expenditure

€473,181.78

Rationale for undertaking the Research

With the expansion of the Irish milk pool post quota abolition in 2015 the dairy processors in Ireland needed to future proof their operations against price volatilities in dairy commodities, particularly based on the challenges presented by Brexit for the cheese industry. The STEPUP project focused on the development of novel valorisation technologies, such as new fractionation designs, based on microfiltration approaches, which could be retrofit to existing milk processing and cheese making process plants. The creation of high added value in existing product portfolios, particularly cheese and whey, will help buffer the Irish dairy industry against international market volatilities.

The STEPUP project has redesigning cheese and whey processing technologies for the Irish dairy industry by reversing the cheese to whey relationship in both Cheddar and Swiss type commercial processes. The STEPUP project has developed, ceramic/polymeric scalable, cascade microfiltration (MF) and ultrafiltration (UF) processes to create whey depleted recombined milks suitable for cheese making. The soluble or serum proteins removed prior to cheese making have provided dairy processors with a native or ideal whey source which is extremely desirable for sports/nutritional products and in particular for infant formula manufacturers due to its improved nutritional quality over typical commercial protein concentrates derived from cheese whey. The conversion of the whey depleted recombined milks into custom cheeses suitable for manufacture in typical Irish commercial cheese plants, created opportunities for cost effective stabilisation of caseins as new cheese varieties, focused on development of Cheddar, Swiss and Quark style cheeses.

Methodology

This project focused on development of size-based membrane separation technologies to alter the casein to whey ratio in milk prior to cheesemaking. Microfiltration experiments were carried out at large pilot plant scale under continuous operational conditions reflecting commercial scale. The approach focused firstly on optimisation of the separation of serum proteins from milk by both ceramic and polymeric microfiltration approaches. The project has established and validated the process and has met the target serum protein yields while generating the recombined milk for subsequent cheese making studies. A key focus of the optimisation of

the microfiltration process was both quantitative and qualitative analysis of the protein profile in fractionated streams by a combination of nitrogen fraction determination by wet chemistry techniques coupled with gel electrophoresis and chromatographic approaches.

Rennet and acid coagulation properties of whey protein-depleted and whey protein/ β -casein-depleted recombined milk produced by a cascade of microfiltration, ultrafiltration and reverse osmosis, were characterised prior to pilot scale cheese manufacture. Manufacture of cheddar, Swiss and Quark style cheeses were carried out at small pilot scale with subsequent physico-chemical analysis performed throughout the ripening period. Organoleptic quality was monitored from a flavour chemistry perspective by gas chromatography techniques, while rheological characterisation was used to monitor structural changes during and post ripening.

Project Results

The STEPUP project has challenged the scientific domain surrounding the efficiency of depletion of serum proteins by polymeric membranes. Our studies have focused on optimisation of membrane type, plant configuration, operational settings and energy and water usage to define the optimal serum protein depletion approach prior to cheese manufacture. The STEPUP project has established a baseline or seminal position relative to achievable serum protein depletion dynamics from a mass and energy perspective for ceramic and polymeric microfiltration processes. The peer reviewed publications generated by the project team are of great interest to the Irish dairy industry as a whole as they challenge some of the current choices surrounding membrane plant installations from an efficiency/sustainability perspective. Highlighting the relationship between plant sizing water and energy usage per kg protein separated and subsequent operational costs which can be considered relative to the different lifespans of ceramic and polymeric membranes. The project manufactured cheddar and swiss style cheeses from whey protein-depleted and whey protein/ β -casein-depleted recombined milk, which was directly compared to cheese milk standardised in a conventional way. This approach facilitated the removal of much of the native whey from milk in advance of cheese manufacture while retaining a conventional cheese manufacture and ripening process. The filtration process also allowed for standardisation of the recombined milk to a higher casein content (1.5x) resulting in faster coagulation and lower moisture content and higher cheese pH but not to a degree which would be unusual for cheese. From a cheese manufacturers perspective, the capacity to remove a large proportion of the whey protein in advance of cheese manufacture also offers the capacity to add colourant during cheese manufacture targeting expanding markets for coloured cheese without compromising higher value add opportunities for whey products, which is a unique output of the project.

From a flavour chemistry perspective cheddar cheese made from whey depleted recombined milks had higher levels of alcohols, aldehydes, esters, acids, terpenes, benzene and lactone compounds as well as lower levels of sulphur and furan compounds. Due to their higher levels of volatile compounds, it was postulated these samples may be more aromatic or have a more intense flavour profile compared to control cheese. Sensory analysis undertaken by experienced cheese graders provided supplementary information to the volatile profiling, with cheese manufactured from whey depleted recombined milk having a typical Cheddar cheese flavour. While Swiss style cheese made from whey depleted recombined milk had increased levels of aldehydes, ketones and sulphur compounds in resulting cheeses. Removing β -casein from milk by 4.25 %, before cheesemaking, increased the levels of ester and ethanol as well as decreasing the levels of isopropyl alcohol and propionic acid in the cheese.

The nutritional quality of the whey produced from whey depleted recombined milks has also been assessed whereby altered amino acid contents have been observed related in particular to altered ratios of threonine linked to higher levels of casein macropeptide on a protein basis.

Section 2 - Research Outputs

Summary of Project Findings

The year STEPUP project has established robust optimized ceramic/polymeric MF processes to create whey/whey and beta casein depleted recombined milk which has been utilized for cheddar, Swiss and quark style cheese manufacture. The successful cheese production and subsequent ripening has been completed with the cheese produced subjected to full physicochemical and sensory/flavour evaluation. The subsequent whey streams produced from renneting of these recombined milks have the potential to deliver unique nutritional properties in terms of protein profile and amino acid sequence for use in nutritional formulations.

Three PhD students supervised at Teagasc/UCC have established robust cascaded filtration/cheese making solutions at scale utilizing whey depleted recombined milks to deliver industry relevant processes in the following areas:

1. Ceramic/polymeric microfiltration technologies have been assessed relative to whey separation efficiency with maximum efficiencies of 80% achieved. This is at odds with almost all reported literature whereby our studies have highlighted a major issue with previous studies.
2. Cheddar cheese manufactured from whey depleted recombined milks have been successfully manufactured based on ceramic microfiltration demonstrating great potential for optimization of the cheese process especially from a yield/efficiency perspective.
3. The application of cold microfiltration before cheese manufacture can produce β -casein enriched/native serum protein ingredients without compromising rennet coagulability of the cheese milk or the composition and yield of subsequent cheeses.
4. Quark style cheeses were successfully manufactured from whey protein-depleted and whey protein/ β -casein-depleted recombined milk, with significantly higher protein content and significantly lower moisture and higher yield.
5. The nutritional quality of the cheese whey produced from whey depleted recombined milks has also been assessed whereby altered amino acid contents have been observed related in particular to altered ratios of threonine linked to higher levels of casein macropeptide on a protein basis

Summary of Staff Outputs

| Research Output | Male | Female | Total Number |
|-----------------|------|--------|--------------|
| PhD Students | 1 | 2 | 3 |

Summary of Academic Outputs

| Research Outputs | Total Number | Details |
|------------------|--------------|--|
| PhD Theses | 3 | <ol style="list-style-type: none">1. Effect of high heat treatment and β-casein-reduction on the rennet coagulation and ripening of Cheddar and Emmental cheeses manufactured from micellar casein concentrate. Xiaofeng Xia - Teagasc - (2021).2. Evaluation of the Manufacture of Cheese from Micellar Casein Concentrate using Novel Coagulants. Bozhua Li - UCC - (2022).3. Microfiltration of skim milk for serum protein removal and associated mass and energy balance. Surabhi Subhir - Teagasc - (2022). |

1. Subhir, S., McSweeney, P.L., Fenelon, M.A. and Tobin, J.T., 2022. Low temperature microfiltration of skim milk: impact of membrane type, configuration and concentration factor on serum protein permeation efficiency. *International Dairy Journal*, p.105500.
2. Xia, X., Tobin, J.T., Fenelon, M.A., McSweeney, P.L. and Sheehan, J.J., 2022. Production, composition and preservation of micellar casein concentrate and its application in cheesemaking: A review. *International Journal of Dairy Technology*, 75(1), pp.46-58.
3. Subhir, S., McSweeney, P.L., Fenelon, M.A., Magan, J.B. and Tobin, J.T., 2022. Suitability of nitrogen fractions determination to assess serum protein separation efficiency from a mass balance perspective during microfiltration of skim milk. *International dairy journal*, p.105319.
4. Xia, X., Kelly, A.L., Tobin, J.T., Meng, F., Fenelon, M.A., Li, B., McSweeney, P.L., Kilcawley, K.N. and Sheehan, J.J., 2022. Effect of heat treatment on whey protein reduced micellar casein concentrate: A study of texture, proteolysis levels and volatile profiles of Cheddar cheeses produced therefrom. *International Dairy Journal*, 129, p.105280.
5. Xia, X., Tobin, J.T., Subhir, S., Fenelon, M.A., Corrigan, B.M., McSweeney, P.L. and Sheehan, J.J., 2022. Effect of β -casein reduction and high heat treatment of micellar casein concentrate on the rennet coagulation properties, composition and yield of Emmental cheese made therefrom. *International Dairy Journal*, 126, p.105240.
6. Xia, X., Tobin, J.T., Subhir, S., Fenelon, M.A., McSweeney, P.L. and Sheehan, J.J., 2021. Effect of thermal treatment on serum protein reduced micellar casein concentrate: An evaluation of rennet coagulability, cheese composition and yield. *International Dairy Journal*, 114, p.104902.
7. Li, B., Waldron, D.S., Tobin, J.T., Subhir, S., Kelly, A.L. and McSweeney, P.L., 2020. Evaluation of production of Cheddar cheese from micellar casein concentrate. *International Dairy Journal*, 107, p.104711.
8. Xia, X., Tobin, J.T., Sharma, P., Fenelon, M., McSweeney, P.L. and Sheehan, J.J., 2020. Application of a cascade membrane filtration process to standardise serum protein depleted cheese milk for cheddar cheese manufacture. *International Dairy Journal*, 110, p.104796.
9. Li, B., Waldron, D.S., Drake, M., Lyne, J., Kelly, A.L. and McSweeney, P.L., 2022. Suitability of a novel camel (*Camelus dromedarius*) chymosin as a coagulant for Cheddar cheese manufacture. *International Dairy Journal*, 129, p.105346.

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| Other | 1 | Subhir, S., Fenelon, M.A. and Tobin, J.T., 2021. Plant, Equipment and Utilities: Membranes and Membrane Processing (Reverse Osmosis and Nano/ultra/micro Filtration) Plants. Reference Module in Food Science, Elsevier. |
| Other | 5 | <ol style="list-style-type: none"> 1. Subhir et al. (Oct 2021). A mass and energy balance for fractionation of skim milk proteins by ceramic and polymeric microfiltration process (Poster presentation). 12th NIZO Dairy Conference, Netherlands 2. X. Xia, P.L.H. McSweeney, J. Tobin and J.J. Sheehan. Application of microfiltration in cheesemaking: A review. Poster presentation at the 10th Cheese symposium, Rennes, France, April 4th -6th, 2018.nd Technology Conference, Dublin, Dec 6th -7th, 2018. 3. X. Xia, P.L.H. McSweeney, J. Tobin and J.J. Sheehan. Application of microfiltration in cheesemaking. Poster presentation at the 46th Annual Food Science and Technology Conference, Dublin, Dec 6th -7th, 2018. 4. Bozhao Li, David S. Waldron, John T. Tobin, Surabhi Subhir, Alan L. Kelly, Paul L. H. McSweeney. Evaluation of Production of Cheddar Cheese from Micellar Casein Concentrate Poster presented at The 48th Annual Food Science and Technology Conference, 16th December, 2019, University of Limerick, Ireland 5. Xia, X., Tobin. J. T., Sharma. P., Fenelon. M., McSweeney. P. L. H., and J. J. Sheehan. Application of a cascade filtration process for preparation of standardised cheese milk, The 48th Annual Food Science and Technology Conference, 16th December, 2019, University of Limerick, Ireland. |

Intellectual Property

No invention disclosures have been filed for this project, the results of which have been disseminated primarily via peer reviewed publication in relevant technical and scientific journals

Summary of other Project Outputs

| Project | Details | Total No. |
|---------------|---|-----------|
| New Processes | Two optimized processes for depletion of serum protein by ceramic/polymeric membranes prior to cheese making have been successfully developed whereby the cheese making elements within the process have been exploited to both stabilize the micellar casein stream while also generating new whey ingredients | 2 |

Potential Impact related to Policy, Practice and Other Impacts

| Impact | Details |
|----------|---|
| Industry | The StepUp project has demonstrated industrially feasible approaches for standardisation of milk prior to cheese making, where microfiltration has been utilised to create whey depleted recombined milks, novel swiss and cheddar style cheeses and subsequent whey products with altered protein and amino acid profiles. |

Dissemination Activities

| Activity | Details |
|---|---|
| Workshops at which results were presented | The results of the optimization of MF processes for separation of serum proteins was presented in the Virtual Symposium “Membrane Process Innovations in Dairy” held by Friesland Campina in June 2020. |

Section 3 – Leveraging, Future Strategies & Reference

Leveraging Metrics

| Type of Funding Resource | Funding € | Summary |
|--------------------------|-------------|---|
| Other | €254,000.00 | Additional funding has been leveraged with a commercial partner focused on optimisation of microfiltration processes for enrichment of high value dairy fractions |

Future Strategies

Core fundamental dairy science projects focused on separation technology are challenging from a funding perspective. The likelihood is that future program initiatives will be funded from a combination of Teagasc internal grant in aid funding and industry funded contract research projects.

Project Publications

STEPUP List of publications:

1. Subhir, S., McSweeney, P.L., Fenelon, M.A. and Tobin, J.T., 2022. Low temperature microfiltration of skim milk: impact of membrane type, configuration, and concentration factor on serum protein permeation efficiency. *International Dairy Journal*, p.105500.
2. Xia, X., Tobin, J.T., Fenelon, M.A., McSweeney, P.L. and Sheehan, J.J., 2022. Production, composition and preservation of micellar casein concentrate and its application in cheesemaking: A review. *International Journal of Dairy Technology*, 75(1), pp.46-58.
3. Subhir, S., McSweeney, P.L., Fenelon, M.A., Magan, J.B. and Tobin, J.T., 2022. Suitability of nitrogen fractions determination to assess serum protein separation efficiency from a mass balance perspective during microfiltration of skim milk. *International dairy journal*, p.105319.
4. Xia, X., Kelly, A.L., Tobin, J.T., Meng, F., Fenelon, M.A., Li, B., McSweeney, P.L., Kilcawley, K.N. and Sheehan, J.J., 2022. Effect of heat treatment on whey protein-reduced micellar casein concentrate: A study of texture, proteolysis levels and volatile profiles of Cheddar cheeses produced therefrom. *International Dairy Journal*, 129, p.105280.
5. Xia, X., Tobin, J.T., Subhir, S., Fenelon, M.A., Corrigan, B.M., McSweeney, P.L. and Sheehan, J.J., 2022. Effect of β -casein reduction and high heat treatment of micellar casein concentrate on the rennet coagulation properties, composition and yield of Emmental cheese made therefrom. *International Dairy Journal*, 126, p.105240.
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7. Xia, X., Tobin, J.T., Subhir, S., Fenelon, M.A., McSweeney, P.L. and Sheehan, J.J., 2021. Effect of thermal treatment on serum protein reduced micellar casein concentrate: An evaluation of rennet coagulability, cheese composition and yield. *International Dairy Journal*, 114, p.104902.
8. Li, B., Waldron, D.S., Tobin, J.T., Subhir, S., Kelly, A.L. and McSweeney, P.L., 2020. Evaluation of production of Cheddar cheese from micellar casein concentrate. *International Dairy Journal*, 107, p.104711.
9. Xia, X., Tobin, J.T., Sharma, P., Fenelon, M., McSweeney, P.L. and Sheehan, J.J., 2020. Application of a cascade membrane filtration process to standardise serum protein depleted cheese milk for cheddar cheese manufacture. *International Dairy Journal*, 110, p.104796.
10. Li, B., Waldron, D.S., Drake, M., Lyne, J., Kelly, A.L. and McSweeney, P.L., 2022. Suitability of a novel camel (*Camelus dromedarius*) chymosin as a coagulant for Cheddar cheese manufacture. *International Dairy Journal*, 129, p.105346.