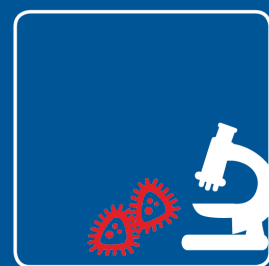


Anni Bygvrå Hougaard:
Effektiv tørring og rekonstituering af
yoghurtpulver

Efficient yoghurt powder drying and reconstitution



Final report

for collaborative projects funded via the Danish Dairy Research Foundation (DDRF)

1. Title of the project

Danish: Effektiv tørring og rekonstituering af yoghurtpulver

English: Efficient yoghurt powder drying and reconstitution

2. Project manager

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3. Other project staff

PhD student Dolores Vickovic, GEA Process Engineering A/S

Post doc Tomasz Pawel Czaja, Dept. Food Science, University of Copenhagen (KU FOOD)

Professor Lilia Ahrné, Dept. Food Science, University of Copenhagen (KU FOOD).

Professor Mogens L. Andersen, Dept. Food Science, University of Copenhagen (KU FOOD).

Jakob Sloth Overgaard, GEA Process Engineering A/S (Replaced by Søren Juhl Pedersen, February 2020)

Søren Juhl Pedersen, GEA Process Engineering A/S (No longer at GEA Process Engineering A/S)

4. Sources of funding

Milk Levy Fund

GEA Process Engineering A/S

5. Project period

Project period with DDRF funding:

January 2018 – December 2021

Revised, if necessary:

January 2018 – December 2022

6. Project summary

Danish:

Produktion af yoghurtpulver indebærer en række udfordringer både under produktionen og ved rekonstituering af pulveret. Effektiviteten af tørreprocessen er ofte lav på grund af problemer med klæbrighed (stickiness) i pulveret, som menes at være relateret til yoghurts indhold af mælkesyre. Projektets formål var at forbedre effektiviteten af tørringsprocessen gennem øget forståelse af laktoses og mælkesyres egenskaber og roller før, under og efter tørring, og derved forbedre mulighederne for industriel produktion af yoghurtpulver i stor skala. Derudover at øge forståelsen af rehydreringsmekanismerne med henblik på opnåelse af forbedrede egenskaber for rekonstitueret yoghurtpulver, tættere på frisk, ikke-tørret yoghurt.

Spraytørring i pilotskala af et modelsystem af rekonstitueret skummetmælkspulver tilsat mælkesyre i forskellige koncentrationer førte til produktion af pulver med højere vandindhold og vandaktivitet og med højere tilbøjelighed til at klæbe fast til væggene af tørretårnet under tørringen. Den ventede sammenhæng mellem klæbrigheden under tørring og en reduktion af pulverets glasovergangstemperatur blev dog ikke observeret, hvilket blev tilskrevet det høje indhold af laktose i modelsystemet, samt tilstedeværelsen af protein.

Det efterfølgende studie blev udført på skyr med tilsætning af ekstra laktose og/eller mælkesyre, hvilket bekræftede at tilsætning af mælkesyre øger klæbrigheden og sænker glasovergangstemperaturen, mens tilsætning af laktose har den modsatte effekt, og at ratioen mellem laktose og mælkesukker derfor er af betydning, ikke udelukkende koncentrationen af de enkelte komponenter. Således kan pulver med samme glasovergangstemperatur godt have forskellig tendens til at klæbe til tørretårnets vægge. Dette blev foreslået forårsaget af forskelle i pulverpartiklernes overflade, hvor en øget koncentration af laktose og/eller mælkesyre blev vist at føre til et skift i overfladesammensætningen fra stærk dominans af protein og mod større tilstedeværelse af laktose/mælkesyre.

Et præliminært studie af rekonstituering af yoghurtpulver viste, at der skal anvendes op til tre gange så meget tørstof som i kommerciel, frisk yoghurt for at opnå lignende viskositet.

Projektet har givet øget forståelse af den klæbrighed der observeres under spraytørring af yoghurt, hvilket potentielt kan overføres til andre produkter indeholdende mælkesyre. Klæbrigheden er relateret til mælkesyre, men også til ratioen mellem laktose og mælkesyre i produktet samt formodentlig også til overfladesammensætningen af pulverpartiklerne.

English:

Production of yoghurt powder involves a number of challenges both during production and in the reconstitution of the powder. The drying efficiency is often low due to problems with stickiness in the powder, which is often linked to the content of lactic acid in yoghurt. The aim of the project was to improve the efficiency of the drying process through increased understanding of the properties and roles of lactose and lactic acid before, during, and after drying, thereby enhancing the possibilities for industrial production of yoghurt powder on a large scale. Additionally, to increase the understanding of rehydration mechanisms to achieve improved properties for reconstituted yoghurt powder, closer to fresh, non-dried yoghurt.

Pilot-scale spray drying of a model system of reconstituted skim milk powder with added lactic acid in various concentrations led to the production of powder with increased moisture content and water activity and with a higher tendency to adhere to the walls of the spray dryer during drying. However, the expected correlation between stickiness during drying and a reduction in the glass transition temperature of the powder was not observed, which was attributed to the high lactose content in the model system, as well as the presence of protein.

The following study was conducted on skyr with the addition of extra lactose and/or lactic acid, which confirmed that the addition of lactic acid increases the susceptibility to stickiness and lowers the glass transition

temperature, while the addition of lactose has the opposite effect, and that the ratio between lactose and lactic acid is therefore significant, not only the concentration of the individual components. Hence, powder with equivalent glass transition temperature can have different susceptibility for adherence to the walls of the spray dryer. This was suggested to be caused by differences in the surface composition of the powder particles, where an increased concentration of lactose and/or lactic acid was shown to lead to a shift in surface composition from being completely dominated by protein towards greater presence of lactose/lactic acid.

A preliminary study of the reconstitution of yoghurt powder showed that up to three times as much dry matter as found in commercial, fresh yoghurt is required to achieve similar viscosity.

The project has provided increased understanding of the stickiness observed during spray drying of yoghurt, which can potentially be transferred to other products containing lactic acid. The stickiness is related to lactic acid, but also to the ratio between lactose and lactic acid in the product and presumably also to the surface composition of the powder particles

7. Project aim

Danish:

- Projektet skal gøre det muligt på industriel skala effektivt at producere yoghurtpulver, som kan rekonstitueres til et produkt med tekstur og mikrobiologisk kvalitet svarende til ikke-tørret yoghurt.
- Problemer med yoghurtpulvers klæbrighed skal løses gennem forøget forståelse af laktose og mælkesyres egenskaber og roller før, under og efter tørring.
- Tekstur og reologiske egenskaber af rekonstitueret yoghurtpulver skal forbedres.

English:

- The project shall enable efficient industrial production of yoghurt powder that can be reconstituted into a product with similar texture and microbiological quality as non-dried yoghurt.
- Problems related to the stickiness of yoghurt powder will be solved by improved understanding of the properties of lactose and lactic acid before, during and after drying.
- Texture and rheological properties of reconstituted yoghurt powder will be improved.

8. Background for the project

Production of yoghurt powder presents a number of challenges both during production and in reconstitution. The drying efficiency is often low due to problems with stickiness of the powder, which is linked to the high levels of lactic acid present in yoghurt. Furthermore, reconstituted yoghurt powder generally show low viscosity and poorer texture as compared to fresh yoghurt. This project hypothesise that the stickiness problem is highly related to the surface composition and properties of the powder particles as well as to the oligomerisation behaviour of lactic acid during and after powder production. By understanding these factors, it is possible to reduce the stickiness and thereby increase the drying efficiency and improve the reconstitution. It is further hypothesised that reconstitution properties of yoghurt powder can be improved by understanding the mechanisms of rehydration and the effects of drying technology and possible additions of texturizing agent on formation of protein-protein and protein-polysaccharide interactions.

9. Sub-activities in the entire project period

Work package	Milestones	Status and comments
	Recruitment PhD	PhD student 1 recruited from September 2018, resigned February 2019. Restart of project with PhD student Dolores Vickovic from May 2019.
WP1	M1.1 Methods linking bulk powder stickiness to microscopic particle surface chemical and physical characteristics established. M1.2 Important yoghurt properties and drying conditions that affect powder stickiness are identified and evaluated. M1.3 Yoghurt powders produced with different technologies under conditions ensuring survival of lactic acid bacteria are characterised.	Completed with minor changes. In M1.3 different technologies for drying were expected, but the project has solely focused on spray drying in small pilot scale.
WP2	M2.1 Oligomerisation behaviour of lactic acid described in model system. M2.2 Conditions for oligomerisation of lactic acid during drying of yoghurt and in the final powder described. M2.3 Storage stability of lactic acid oligomers in yoghurt powder characterised.	Work initiated in student project work in 2018 and 2020, but due to unexpected difficulties in finishing WP1 only limited activities have been done in this WP.
WP3	M3.1 Methods for laboratory reconstitution and rheological characterisation of reconstituted yoghurt powder established. M3.2 Effects of processing conditions, dry matter and additional ingredients on reconstitution properties of yoghurt powder described. M3.3 Optimized methodology for reconstitution of yoghurt powder established.	Work initiated in student project work in 2020, but due to unexpected difficulties in finishing WP1 only limited activities have been done in this WP.
WP4	M4.1 Pilot scale production of yoghurt powder with low stickiness and good reconstitution properties.	No larger pilot scale productions were done
	PhD Thesis and final manuscripts PhD defence	Project period extended, primarily due late start of PhD student and corona-related delays, though no extension of DDRF funding period. PhD thesis handed in October 2022. PhD thesis defended in January 2023.

10. Deviations

Scientific

Handling and understanding of the challenges in relation to production of yoghurt powder related to the composition of the feed for drying and the stickiness observed during the drying process proved to be much more time-consuming and problematic than expected. Therefore, the project was modified to focus solely on spray drying and only in small pilot plant scale (GEA Mobile Minor), and only very limited work has been done within WP2-4 described in the original project outline.

Timeline and economy

The first PhD student recruited for the project started later than expected (September 2018) and resigned again already in February 2019. The project was restarted with Dolores Vickovic as PhD student from May 2019. No changes in the funding from DDRF were needed in this regard. Due to corona lockdowns and related delays the project period was extended to December 2022, also without changing the funding

Jakob Sloth Overgaard changed position internally in GEA and was replaced by Søren Juhl Pedersen as GEA partner and PhD co-supervisor from February 2020. Søren Juhl Pedersen left GEA just before the PhD thesis was submitted by Dolores Vickovic, and the administrative finalization of the project within GEA was returned to Jakob Sloth Overgaard.

11. Project results

Work package 1 (WP1):

The overall aim of WP1 was to establish methodology for production and analysis of yoghurt powder, allowing controlled changes in feed composition and processing parameters for understanding of the effects on powder properties, with a special focus on stickiness during drying, leading to powder deposits inside the drying equipment.

The first set of experiments were performed on a model system consisting of reconstituted low-heat skim milk powder acidified by addition of different concentrations of lactic acid, to address the link between powder stickiness and lactic acid in a relatively simple system. The model system allowed a higher feed dry matter content without the complications of concentrating yoghurt and better possibilities of viscosity control. Spray drying was performed in small pilot scale (GEA Mobile Minor) under varying conditions. The process yield, based on the amount of powder obtained relative to feed introduced, as well as the wall deposits inside the spray dryer was measured for each run. The powders were characterized in terms of moisture content, water activity, particle sizes and glass transition temperature (T_g). The results showed that increasing the lactic acid concentration in acidified skim milk solutions lead to higher moisture content and water activity values of the acidified skim milk powders and higher wall deposition during spray drying, as was expected. High inlet air temperature also negatively influenced wall deposition.

Powder stickiness is considered to be correlated with T_g , i.e. the temperature where a material is transformed from a solid-like, glassy state to a rubbery, viscous state, and often the sticky point is found to be at a temperature 10-20°C above T_g . However, no significant effect of lactic acid concentration on glass transition temperature was observed in this study, which was suggested to be related to the presence of high concentrations of lactose, as well as proteins in the studied model systems. Furthermore, the question of the importance of particle surface composition compared to bulk powder composition was raised.

The results of this study are published in:

Dolores Vickovic, Søren Juhl Pedersen, Lilia Ahn , Anni Bygvr  Hougaard (2023). Effects of lactic acid concentration and spray drying conditions on stickiness of acidified skim milk powder. International Journal of Dairy Technology 76, 2, 266-275. DOI:10.1111/1471-0307.12932

The following studies were designed to address the questions raised in the first set of experiments. On one side, a study to evaluate the potential of combining vibrational spectroscopy techniques, such as near infrared (NIR) and attenuated total reflection mid-infrared (ATR IR), with Raman imaging, as a tool for analyzing the composition and structure of acidified milk powders was initiated. Furthermore, a study to investigate the effect of feed composition, in terms of concentration of lactose and lactic acid, on wall deposition was performed, including obtaining a better understanding of the distribution of components on the surface of individual powder particles, and consequently, the influence on powder stickiness during spray drying.

For these studies acidified milk (yoghurt) powders were produced by spray drying of feeds based on skyr mixed with additional lactose and/or lactic acid to modify the ratio between these, and with final feed dry matter in the range 9.4-13.4%. Powder samples were collected for all formulations from the exit of the dryer as well as from the wall deposits built during each drying run and analyzed separately.

The results revealed that both IR techniques were able to distinguish between the two fractions of powder, i.e. from the dryer exit and from the wall deposits. NIR has potential to be implemented as a sensor to monitor the composition of powder during the spray drying process, and ATR as at-line quality control to describe molecular structural changes in powders, for example, molecular changes caused by prolonged exposure of powder to high drying temperature. and may be useful in characterization of powder production processes.

A method using confocal Raman microscopy was developed to analyze the internal and surface composition of acidified milk powders. Assigning Raman modes makes it possible to mark and color images by dominant component (protein or lactose/lactic acid). It was not possible to distinguish between lactose and lactic acid in this analysis. Increasing the spectral intensity of the selected modes provides differences in color intensity, which correlates directly to the concentration of molecules in the analyzed pixel, and it is possible to color the pixels by their dominant components/molecules.

Analyses of the powders showed that addition of lactic acid, as expected, led to a decrease in Tg and an increase in wall deposits, whereas lactose addition caused an increase in Tg. However, interestingly the addition of lactic acid and lactose to the feed led to no change in Tg compared to no addition of either component, but still an increase in wall deposition was observed, indicating that powder stickiness is not only correlated to the glass transition temperature. Proton mobility (interpreted as mainly water) was analyzed using low field nuclear magnetic resonance spectroscopy (LF-NMR) over a range of temperatures from 25°C to 80°C, revealing significant changes in mobility and abundance of the water populations dependent on the powder composition, which could be correlated to susceptibility to stickiness and wall deposition. Particle surface composition was analyzed using X-ray photoelectron spectroscopy as well as confocal Raman spectroscopy showing that when lactose and/or lactic acid was added the overrepresentation of protein on the particle surface was reduced (figure 1), which was suggested to cause the powder to be more susceptible to stick to the dryer walls during the spray drying process.

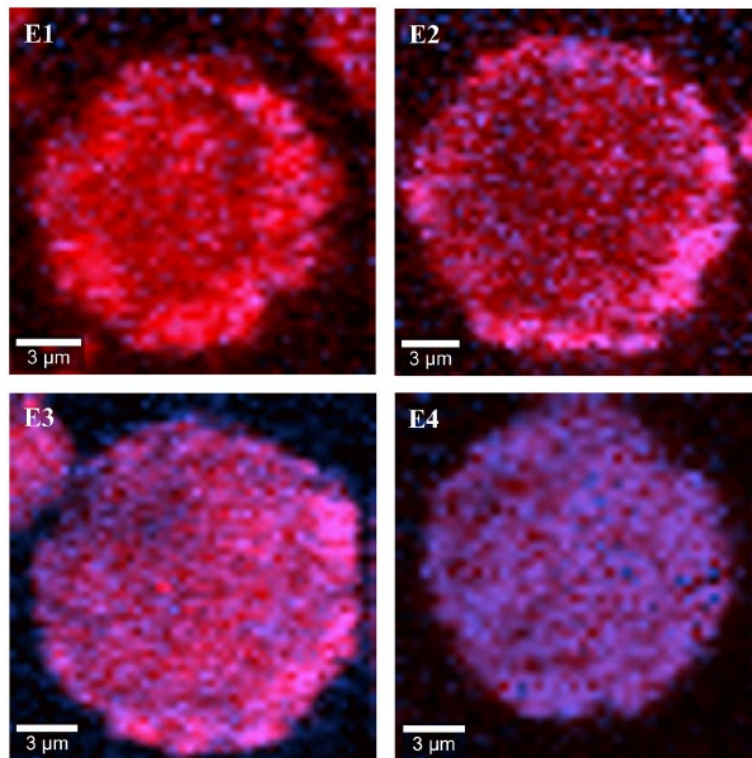


Fig. 4. Confocal Raman images of surface (x,y) scans showing the distribution of milk proteins (red) and lactose/lactic acid (blue) of the studied powder particles. The added concentration of each component (lactose: Lac; lactic acid: LA) in the feed formulations before spray drying: E1: 0% Lac, 0% LA; E2: 2% Lac, 0% LA; E3: 0% Lac, 2% LA; E4: 2% Lac, 2% LA. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Figure 1: Confocal Raman microscopy images of powder particle surfaces, colored according to dominant component in each pixel (red: protein, blue: lactose/lactic acid). *From: Dolores Vickovic, Tomasz Pawel Czaja, Claire Gaiani, Søren Juhl Pedersen, Lilia Ahrné, Anni Bygvrå Hougaard (2023). The effect of feed formulation on surface composition of powders and wall deposition during spray drying of acidified dairy products. Powder Technology 418, 118297*

In order to study the difference between powder particles adhering to the dryer walls and powder particles exiting the dryer immediately, plates were installed inside the dryer, enabling confocal Raman imaging of the adhering particles directly from the plates. No difference in surface composition between adhering particles and collected powder particles were observed, though the adhering particles showed larger particle size (Figure 2).

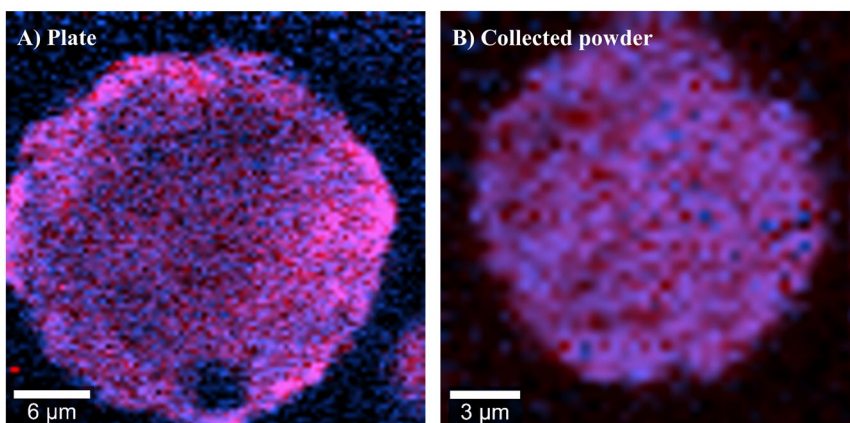


Figure 2: Confocal Raman microscopy image colored according to dominant component in each pixel (red: protein, blue: lactose/lactic acid). Right: Particle from plate installed on dryer wall. Left: Particle from collected final powder. Unpublished results.

These results are published in:

Dolores Vickovic, Tomasz Pawel Czaja, Claire Gaiani, Søren Juhl Pedersen, Lilia Ahrné, Anni Bygvrå Hougaard (2023). The effect of feed formulation on surface composition of powders and wall deposition during spray drying of acidified dairy products. Powder Technology 418, 118297.

DOI:10.1016/j.powtec.2023.118297

Tomasz Pawel Czaja, Dolores Vickovic, Søren Juhl Pedersen, Anni Bygvrå Hougaard, Lilia Ahrné (2023). Spectroscopic characterisation of acidified milk powders. International Dairy Journal 142, 105664.

<https://doi.org/10.1016/j.idairyj.2023.105664>

As part of an MSc thesis related to WP1, a study was conducted aiming at producing concentrated yoghurt for spray drying in pilot scale to enable compositional modifications and increasing the total dry matter of the feed for drying beyond the level of standard yoghurt. These experiments turned out to be generally unsuccessful in obtaining a satisfactory fermentation and a manageable viscosity of the product. Hence the remaining part of the MSc thesis as well as the studies described above was performed on commercial fermented milk products used either as is, or modified to obtain the desired composition.

Work package 2 (WP2):

This work package was aimed at studying the polymerization behavior of lactic acid, but due to the difficulties experienced in WP1 of actually producing powder reproducibly, the only work done in this relation was a preliminary study in an MSc thesis, showing that lactic acid is prone to show instable, dynamic behavior, highly dependent on the environment and conditions, though it was not possible to study the polymerization behavior directly in yoghurt during the MSc project.

Work package 3 (WP3):

This work package was aimed at studying the reconstitution behavior of yoghurt powder and optimize the process for the intended powders from WP1 and WP4. However, due to the difficulties experienced in WP1 of producing yoghurt powders in a reproducible way, the only work done in this relation was a preliminary study in an MSc thesis. This project applied commercially available yoghurt powder as well as a set of experimental yoghurt powder samples obtained from GEA A/S, and hence not much information was available about the samples, in terms of processing details. The results showed that a significantly increased amount of dry matter, up to 35% compared to 11.4% in the commercial yoghurt applied as control, was needed to obtain viscosities similar to commercial fresh yoghurt, and still the rheological properties were different, with the reconstituted product behaving more like a dense solution rather than a weak gel. Reconstitution time and temperature did not have significant impact on the properties of the reconstituted product.

Work package 4 (WP4):

The aim of the work package was to verify the results obtained in lab and small pilot scale in larger scale pilot trials. However, no work was done in larger scale, due to the difficulties experienced in the small scale pilot drying.

Conclusion

The results of the project have increased the understanding of the challenges regarding powder stickiness experienced when drying materials containing lactic acid. The project has focused on the stickiness observed during the drying process leading to powder adhering to the dryer walls, and it was shown that the stickiness is not only related to the glass transition temperature of the powder, but also to the feed composition, e.g. the ratio between lactose and lactic acid, and potentially the surface composition of the particles.

The correlation between stickiness/wall deposition and process yield was on the other hand not straight forward, and more studies are needed to enable efficient drying of yoghurt into a powder with low stickiness and good reconstitution properties.

12. The relevance of the results, including relevance for the dairy industry

The results are mostly relevant for scientists and product/process developers at equipment manufacturers and dairy companies as basis for further studies on the potential of producing yoghurt powder efficiently, with low stickiness during drying and consistent yield of high-quality powder. The results may also be of relevance to other product matrices containing lactic acid (and lactose). As the work in the current project ended up being focused on the ability to produce yoghurt powder reproducibly in small scale, there is a question regarding scaling, which was intended to be studied in the final WP, still unaddressed and expectedly not trivial. Direct implementation in the dairy industry is therefore not immediately awaiting.

The scientific understanding of stickiness and the correlation between powder composition and properties and observed susceptibility to sticky behavior is not yet complete either, e.g. the observed difference in stickiness (measured as powder adherence to the dryer walls during drying) for powders with comparable glass transition temperature would be of interest for further studies.

13. Communication and knowledge sharing about the project

Papers in international journals: 3

Easily read papers: 1 (+1 upcoming)

Student theses: 3 MSc theses and 1 PhD thesis

Oral presentations at scientific conferences, symposiums etc.: 0

Oral presentations at meetings: 0

Other:

Poster presentations at scientific conferences: 2

Scientific papers in peer-reviewed journals:

1: Dolores Vickovic, Søren Juhl Pedersen, Lilia Ahrné, Anni Bygvrå Hougaard (2023). Effects of lactic acid concentration and spray drying conditions on stickiness of acidified skim milk powder. *International Journal of Dairy Technology* 76, 2, 266-275. DOI:10.1111/1471-0307.12932

2: Dolores Vickovic, Tomasz Pawel Czaja, Claire Gaiani, Søren Juhl Pedersen, Lilia Ahrné, Anni Bygvrå Hougaard (2023). The effect of feed formulation on surface composition of powders and wall deposition during spray drying of acidified dairy products. *Powder Technology* 418, 118297. DOI:10.1016/j.powtec.2023.118297

3: Tomasz Pawel Czaja, Dolores Vickovic, Søren Juhl Pedersen, Anni Bygvrå Hougaard, Lilia Ahrné (2023). Spectroscopic characterisation of acidified milk powders. *International Dairy Journal* 142, 105664. <https://doi.org/10.1016/j.idairyj.2023.105664>

Easily read paper:

Anni Bygvrå Hougaard, Mogens Larsen Andersen, Jakob Sloth Overgaard, Lilia Ahrné (2018). Effektiv fremstilling af yoghurtpulver. Mælkeritidende 9, 6-7

14. Contribution to master and PhD education

Education of PhD student Dolores Vickovic.

PhD thesis: Efficient yoghurt powder production – The effect of feed composition and spray drying conditions on stickiness. October 2022, University of Copenhagen.

Education of MSc students – MSc thesis projects:

Hanna Burfeind: Investigating the stability of dried yogurt with focus on lactic acid behaviour. Submitted March 2018, before actual project start, but with direct connection to the objectives of the project.

Mathias Søgaard-Andersen: Preliminary quantification of process variables on properties of yoghurt powder. Submitted December 2020.

Xiaodong Li: Rheological behaviours of reconstituted yoghurt. Submitted December 2020.

15. New contacts/projects

No specific new contacts.