INRAE Université de Lille institut Chevreul

FCFP 2022

Fouling and Cleaning in Food Processing

28 - 30 March 2022 in ville de Lille

Preface

More than 40 years have passed since the very first in this series of symposia on "Fouling and Cleaning in Food Processing" was initiated and held in Lund, Sweden in 1981. For the first time ever, this event will be held in France in Lille and we hope it will be as enjoyable as the previous ones. Lille is a typical city in the North of France and we hope that you will find time to enjoy the beauty of the region to a greater extent than the short tour of the old town proposed.

As with the previous successful conferences in this series, we hope that this "FCFP 2022" event will achieve its objective of bringing together researchers, students and industrialists interested in fouling and cleaning in the food sector, which differs greatly from other fields since hygiene requirements cannot be underestimated or even ignored. Indeed, the generation of unwanted deposits or layers in food processing facilities compromises the hygienic quality of processing, leads to the need for regular cleaning and reduces the possibilities for microbiological growth or survival and cross-contamination of products. The additional energy consumption and investment caused by fouling affects the costs and environmental performance of these food systems, and environmentally friendly manufacturing methods are now under intense public concern requiring both responsible practices (life cycle analysis, circular economy) and new policy regulations. Limited resources e.g. potable water and the growing population of our planet are forcing us to change our current practices to ensure sustainability and to offer food processes and cleaning sequences based on renewable sources (e.g. plant proteins versus animal proteins, green chemistry for detergents).

During the conference, there will be approximately attend 40 oral presentations and a poster session. Accordingly, we hope that creative, stimulating exchanges of valuable knowledge and ideas will take place among scientists, expert, industrial and students

This event has been organized, with the help of Lille University by UMET joint research Unit (Unité Matériaux Et Transformations) hosting a large portion of the research in Materials Science of Lille university), specially the PIHM's team (Processus aux interfaces et Hygiène des matériaux) of UMET who carried out a scientific project arround food processing hygien issues for INRAE (French National Research Institute for Agriculture, Food and Environment).

We would like to thank all the participants for the confidence of registered in such uncertain period (Covid sanitary crisis, Ukraine conflict) bringing their financial support which contributed to the success of this event. We would like also thank our sponsors namely the fédération de recherche Michel-Eugène Chevreul freely hosting us in their building, lille University and MICA Division of INRAE developing research in fundamental and applied microbiology focused on food, animal and human health and biotechnologies department dedicated to microbiology subventioning this event.

The organisers wish to thank the back office, namely Aline Waquet (Communications officer) and Christophe Dufourmantelle (Financial Unit manager) helping to solve website and payment issues and in advance PIHM's students facilitating registration and sessions happening.

Following the conferences, some of the proposed articles will be invited for submission to a special issue of the IChemE/EFCE Journal: *Food & Bioproducts Processing*. which for inclusion of an issue which will be partly dedicated to this conference

Finally, we encourage you to make the most of this event to interact with each other, discussing topics of common interest and generating many informal discussions during the breaks, the gala dinner or the evening events. During the COVID health crisis, we all realised that the lack of face-to-face congresses could lead to frustration, as nothing beats the informal exchanges that often lead to successful research projects or new collaborations. We hope you will make new acquaintances, renew

old friendships, networking is so important for the success of our research. We have the opportunity here to meet and exchange with each other, so please take advantage of it!

Best wishes

Thierry Benezech, Christine Faille, Maude Jimenez, Guillaume Delaplace

Villeneuve-d'Ascq

March 2022

PROGRAMME FCFP-2022

Monday 28/03/2022

8:00 **Registration**

8:30 **Conference introduction**
Catherine Renard (Deputy head of division Transform INRAE, F) and Ian Wilson (University of Cambridge, UK)

SESSION 1: Sustainability in operation and design

9:00: **Lessons to learn from Roadmapping in Cleaning and Decontamination**
Ian Wilson (University of Cambridge)

9:30 • **Systematic cleaning investigations in micro structured equipment building on Sinners circle**
Felicitas Aselmeyer (Technische Universität Braunschweig)

• **Cyber Physical Cleaning Systems: A three level approach**
Marc Mauermann (Fraunhofer Institute for Process Engineering and Packaging IVV, Dresden)

• **Foam flow in biofilm removal to improve hygiene and energy saving in food industry**
Heni Dallagi (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

10:30 Coffee break & putting up posters

11:00 • **CFD-based three-dimensional Modeling of an Adhesively Detaching Soil Layer in a Channel Flow with Sudden Expansion**
Christian Golla (Institute of Fluid Mechanics, Technische Universität, Dresden)

SESSION 2: Monitoring and analysing of fouling and cleaning

11:30 **Sustainability and cleaning: preparing for the future**
Hein Timmerman (Diversey)

12:00 • **Cleaning of cohesive soil layers in a radial flow cell**
Karthikeya P. Deshmukh (University of Cambridge)

• **Aspects of modelling the Cleaning of a Chocolate with Yield Stress in a pipe using CFD**
Vera Liebmann (Institute of Fluid Mechanics, Technische Universität, Dresden)

12:40 Lunch

13:40 • **Cleaning of thick viscoplastic soil layers by impinging water jets**
Melissa Chee (University of Cambridge)

• **Experimental study on the cleaning effect of pulsating sprays**
Matthias Joppa (Fraunhofer Institute for Process Engineering and Packaging IVV, Dresden)

• **Supervised cleaning-in-place processes enabled by a fiber-optical fluorescence sensor**

Vivien Behrendt (Fraunhofer-Institute for Physical Measurement Techniques IPM)

- **Structural degradation along the cleaning front in cleaning a whey protein gel**

Hanna Wiese (Technische Universität Braunschweig)

- **Design and development of a milking machine pilot: monitoring fouling and cleaning under real conditions – PiloTraite research program**

Alice Hubert (Institut de l'élevage)

- **Direct measurement of the cohesive strength of whey protein gel in contact with NaOH by wire cutting experiments**

Manuel Helbig (Institute of Natural Materials Technology, Technische Universität, Dresden)

- **Hygienic design aspects for tank and vessel cleaning in the food industry**

Bo BB Jensen (Alfa Laval)

16:00 Coffee break

POSTER SESSION

16:30

- **Elucidating studies of purging viscoplastic fluid from pipes**

Rubens Fernandes (University of Cambridge)

- **Impact of the physiological state of mold spores on their resistance to chlorinated disinfectant**

Aurélie Hanin (ACTALIA)

- **Antimicrobial activity of free and encapsulated carvacrol against *Pseudomonas aeruginosa* biofilms**

Samah Mechmechani (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

- **Methodology to select commercial enzyme incorporated in formulated detergents used in the cleaning of ultrafiltration PES/PVP membrane fouled by skim milk**

Sophie Kavugho-Mission (Univ. Rennes, CNRS, ISCR)

- **The drying dynamics and resistance to cleaning of droplets containing Bacillus spores on various materials**

Maureen Deleplace (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

- **How to release a sponge: relaxation of casein micelles fouling layers during ultrafiltration, a SAXS study**

Maksym Loginov (INRAE, STLO, Rennes)

- **Fouling mechanisms of coconut milk foulants formed during pasteurization**

Phanida Saikhwan (Faculty of Engineering, Thammasat University)

- **Use of patch-clamp for the investigation of the interaction force between particles and substrate**

Anna Ipatova (Univ. Lille, CNRS, UMR 8520, IEMN)

17:30 **End of the day 1**

19:00 – 20:00 Guided tour of old Lille

Tuesday 29/03/2022

SESSION 3: Surfaces, interfaces and modifications

9:00: **Cleanability of Laser Etched Biomimetic Surfaces with Repeated *Staphylococcus aureus* and Milk Fouling**
Kathryn Whitehead (Manchester Metropolitan University)

9:30 • **Surface engineering of stainless steel to mitigate dairy fouling adhesion**
Manon Saget (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

• **Biomimetic replication of brassica leaves and their efficacy to prevent biofouling of *Escherichia coli* and *Staphylococcus aureus***
Luciana Gomez (Manchester Metropolitan University)

• **Bacterial contamination at the air-liquid-wall interface. Relative roles of physico-chemical vs bacterial phenomena**
Christine Faille (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

10:30 Coffee break

11:00 • **The hygiene factor as an improved description of the hygienic quality of food contact surfaces**
Alan Friis (FORCE technology)

SESSION 4: Heat transfer fouling and cleaning

11:30 • **Thermal treatment of liquid foods under fouling: whole system analysis**
Sandro Macchietto (Department of Chemical Engineering, Imperial College London)

• **Influence of steam-induced wetting and soaking of food- and cosmetic-based contaminants on the efficiency of Cleaning in Place processes of containers**
Siegfried Beckmann (Fraunhofer Institute for Process Engineering and Packaging IVV)

• **Effects of casein and carrageenan on whey fouling during pasteurization**
Jen-Hi Huang (Department of Food Science, Purdue University)

12:30 Lunch

13:30 • **Effect of micellar casein decalcification on the fouling the UHT plant and on the heat stability of high protein beverages**
Marwan Abdallah (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

• **Insolubilisation of mineral salts during vacuum concentration of whey in relation to fouling of dairy evaporators**
Gaëlle Tanguy (STLO, INRAE, INSTITUT AGRO)

• **Effect of calcium on the thermal denaturation of whey proteins and subsequent fouling in a bench-scale fouling rig: couple experimental studies and numerical modeling**
Weiji Liu (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

• **Whey protein fouling prediction in plate heat exchanger**
Sakhr Alhuthali (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

SESSION 5: Membrane fouling and cleaning

15:00 **<u>Sustainability in the food industry : how membrane technologies can contribute ?</u>**
Geneviève Gésan-Guiziou (INRAE, STLO, Rennes)

15:30 Coffee break

16:00 • **Evidencing strengths and weaknesses of alkaline detergents to formulate efficient mixtures useful for polymer membranes' cleaning**
Murielle Rabiller-Baudry (Univ Rennes, CNRS, ISCR)
• **Fouling of polyethersulphone ultrafiltration membranes during the decaffeination of ground coffee brews**
Triantafyllos K. Manios (CASE, Department of Chemical Engineering, University of Bath)
• **Development of in-situ and ex-situ tests for the characterization of the aging of organic membranes in wine microfiltration**
Maxime Pontie (Univ. Angers)
• **Enhanced cleaning of oily foulant on microfiltration membrane using microbubbles**
Monique Mi Song Chung (Department of Food Science, Purdue University)
• **Reducing concentration polarisation in nanofiltration by using 3D printed composite membranes.**
John Chew (Bath University)

17:40 **End of the day 2**

19:30 Gala diner

Wednesday 30/03/2022

SESSION 6: Hygiene : cleaning and disinfection methods

9:00 • **Air-water Interfacial Flows for Removal of Bacterial Fouling**
Sepideh Khodaparast (University Academic Fellow, School of Mechanical Engineering, University of Leeds)

• **Influence of temperature, material, slope and strain on *Listeria monocytogenes* biofilm formation**
Tessa Tuytschaever (Research group VEG-i-TEC, Department of Food Technology, Safety and Health, Faculty of Bioscience Engineering, Ghent University)

• **Effect of heavy water incorporation on the viability of *Listeria innocua***
Sylvain Trigueros (French Agency for Food, Environmental and Occupational Health & Safety (ANSES)

• **Impact of environmental and application conditions on the inactivation of dry fungal spores by disinfectants**
Aurélie Hanin (ACTALIA)

10:20 Coffee break

10:50 • **The use of bio-sourced antimicrobials for the disinfection of food contact surfaces**
Jina Yammine (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

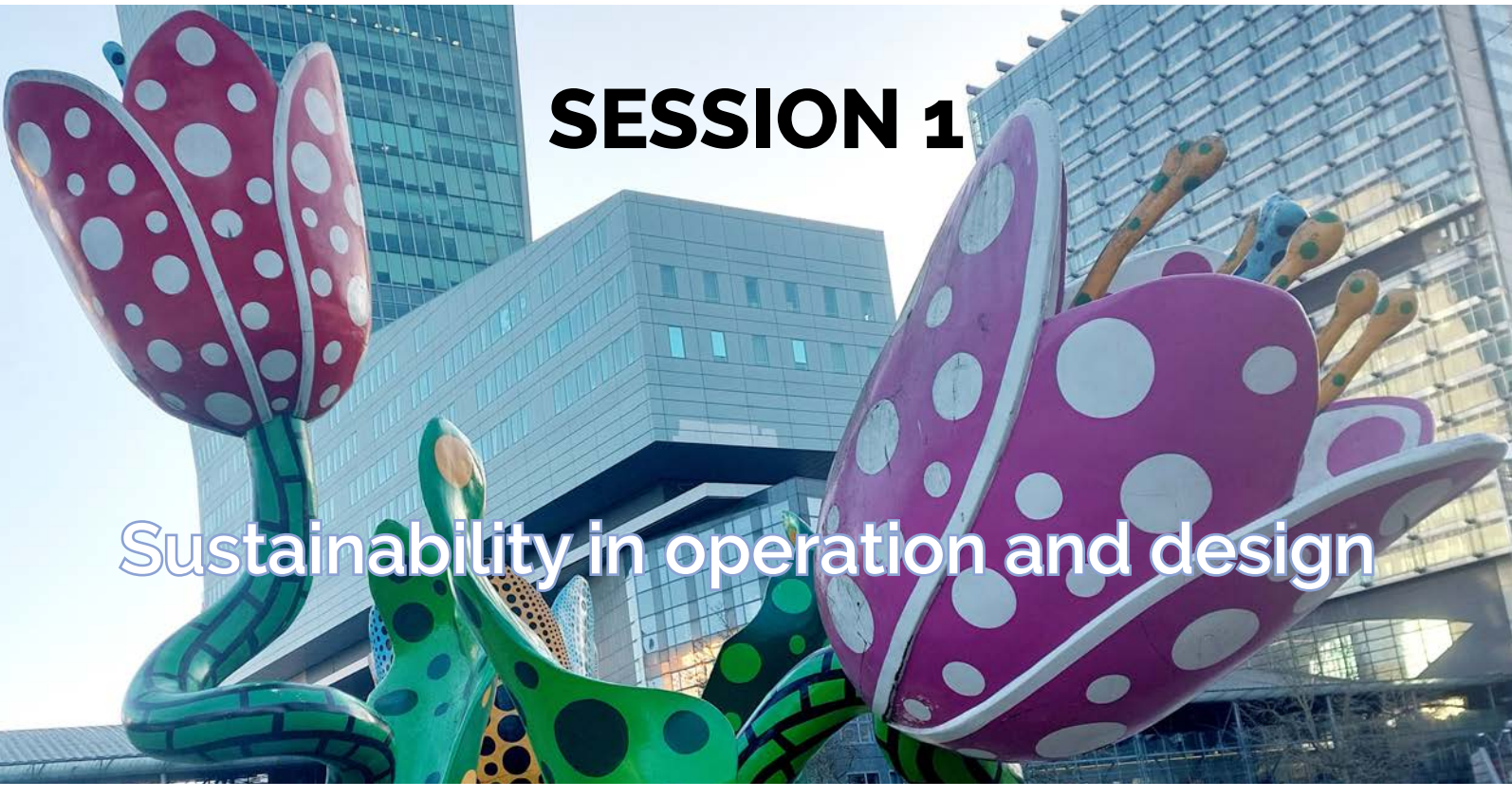
• **Microencapsulation of a water soluble ruthenium (II) complex derived from optically pure limonene as an efficient tools against bacterial food pathogens biofilms**
Yousra EL Fannassi (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

• ***Listeria monocytogenes* in the vegetable/potato processing industry: a case study**
Tessa Tuytschaever (Research group VEG-i-TEC, Department of Food Technology,

11:50 **Conference Summary**
Guillaume Delaplace (Univ. Lille, CNRS, INRAE, Centrale Lille, UMET)

12:00 Lunch

13:00 **Close**



SESSION 1

Sustainability in operation and design

Lessons to learn from Roadmapping in Cleaning and Decontamination

Ian WILSON[1], Graham CHRISTIE, Peter FRYER, Ian HALL, Julien LANDEL and Kath WHITEHEAD

[1] *Department of Chemical Engineering and Biotechnology, University of Cambridge, Philippa Fawcett Drive, Cambridge, CB3 0AS, UK*

The UK's Engineering and Physical Sciences Research Council supported a series of meetings over the period 2019-2021 to develop a roadmap for future research in Quantitative Modelling in Cleaning and Decontamination. Quantitative modelling in this context is the development of numerical and predictive tools, based on scientific principles, which can support the design, operation and decision making. The activity involved identifying past and current activities on this topic across a range of different fields, including food and drink, consumer goods, healthcare, pharmaceuticals, nuclear, civil defence and biofilms. Input was received from operators, manufacturing companies, government agencies and researchers.

A number of factors emerged as common to many cleaning and decontamination applications. These included the understanding and communication of risk as applied to cleaning and decontamination operations; the need to link models of cleaning performance to resource availability and decision making processes; the advances and gaps in sensors and data technology; and the need to develop a common taxonomy that will facilitate transfer of expertise between sectors.

In short, Sinner's circle, often used to frame discussions of cleaning, needs an upgrade.

Systematic cleaning investigations in micro structured equipment building on Sinner's circle

Felicitas ASELMEYER, Christoph SPIEGEL, Wolfgang AUGUSTIN* and Stephan SCHOLL

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Micro structured equipment, i.e. components with at least one characteristic dimension smaller than 1 mm, are an option to develop novel, highly optimized production processes especially for the food industry. The small characteristic dimensions lead to high surface-to-volume ratios, resulting in an intensified heat and mass transfer as well as higher process controllability. Thus, micro structured equipment enables new opportunities for production routes in food processing while at the same time avoid unfavorable side or consecutive reactions. Despite all advantages outlined, micro structured components are prone to fouling phenomena especially while processing food ingredients, which yet limits their industrial application, due to the exceedingly high food safety requirements. A systematic investigation of the cleaning behavior of micro structed equipment is a core step to overcome these barriers.

To conduct reliable cleaning experiments, especially in micro structured equipment, comparable and well-defined initial conditions, such as deposit height, distribution and composition, are essential. Unfortunately, soil deposits generated under flow conditions even with constant process parameters, tend to deviate noticeably. Therefore, the development of the preparation procedure for model soil deposits based on a defined soil system, is mandatory for comparable cleaning investigations.

The experimental investigation of cleaning processes in micro structured equipment is carried out in a rectangular, modular and optical accessible flow channel with a height of 1 mm, a width of 4.5 – 18 mm and a length in flow direction of 80 mm, resulting in a micro component in one dimension, which, nevertheless, allows the investigation of microscale effects. The corresponding test rig is operated in once-through mode ("lost cleaning"). Dyed whey protein-based hydrogels with a defined layer height of 0.5 mm are used as model soil system. Consequently, a remaining flow channel height of 0.5 mm is obtained at the beginning of each cleaning experiment. Depending on the preparation procedure the width of the model soil deposit is adjustable between 18 and 4.5 mm to vary the aspect ratio and thus the hydraulic diameter to determine the influence of further miniaturization. To monitor the cleaning process the test rig is equipped with two absolute pressure sensors measuring the pressure drop, as well as an optical observation of the flow channel. Based on the optical observation, additional information like surface coverage and layer thickness can be calculated with temporal and spatial resolution. Furthermore, samples of the cleaning solution are collected continuously over the course of an experiment to determine the protein content of the solution via Bradford protein assay.

This contribution investigated the applicability of Sinner's circle, which describes the dependency of the four cleaning parameters (i) chemistry, (ii) temperature, (iii) mechanical action and (iv) cleaning time, on the cleaning behavior of micro structured equipment by varying cleaning parameters such as fluid temperature, mass flow rate and cleaning agent concentration. Furthermore, the influence of different aspect ratios as well as the corresponding time-resolved shear stress during cleaning was evaluated. First results could prove the applicability of Sinner's circle for the cleaning in micro structured equipment. As in the macroscale, the cleaning time decreased with increasing temperature and the cleaning agent concentration of caustic soda had the same optimal concentration between 0.5 and 0.7 wt.-%. Finally, the applicability of macroscale cleaning models for protein-based contaminants on micro structured equipment is discussed.

Cyber Physical Cleaning Systems: A three level approach

Marc MAUERMANN[1], Andre BOYE[1], Max HESSE[1], Tilman KLAEGER[1], Jialiang YIN[1]

[1]*Fraunhofer Institute for Process Engineering and Packaging IVV, Dresden, Germany*

Consumer and regulatory guidelines expect that cleaning processes are scientifically developed, using risk-based principles, so that they can be routinely and consistently reproduced. Through the underlying principles of cleaning validation and verification commercial cleaning processes are developed based on worst case scenarios. This means that hard to clean equipment areas and hard to remove soiling determine the cleaning effort for the whole machinery, as most industrial cleaning processes use cleaning devices and process controls that are not able to adapt to an uneven distribution of soiling or the required cleaning effort. Another issue is the lack of effective online measurement methods which can be used for process verification and to identify the cleaning endpoint.

For adaptive cleaning processes the cleaning devices are not the only critical element. Control systems of cleaning processes currently used do not offer the necessary degree of flexibility and are not prepared for the integration of innovative cleaning equipment and the associated online sensor technology. The fusion of hardware, software, and sensor technology in the form of smart cleaning processes offers the opportunity for highly adaptive, efficient, and safe cleaning processes. A common architectural approach for such smart systems is setting up cyber-physical systems with a fusion of IoT-enabled (cleaning) hardware and sensor technology driven by a service-oriented software. Though the overall complexity of such systems is higher than a classical mechatronic approach, this provides more flexibility e. g. by utilizing modern software development patterns. The implementation of cyber-physical cleaning systems depends on the present or intended degree of automation, on the accessibility of the soiling for monitoring and on the technology readiness level of the basic elements, that fit for purpose.

We are presenting a three-level approach for cyber-physical cleaning systems with current R&D results and further demands for research and development. Level 1: Hybrid-adaptive cleaning process: knowledge-based, data input but no feedback loop. Level 2: Hybrid-adaptive cleaning process: Based on inline measurements, closed-loop feedback. Level 3: Fully adaptive cleaning process: Based on inline measurements, self-learning, continuously improving.

Foam flow in biofilm removal to improve hygiene and energy saving in food industry

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[2] *LAMIH CNRS UMR 8201, Polytechnic University Hauts-de-France, INSA Hauts-de-France, Campus Mont-Houy, F-59313 Valenciennes Cedex 9, France*

In the food industry, the adoption of new strategies for the cleaning of contaminated surfaces is of crucial concern to ensure food safety while reducing the consumption of energy and potable water for environmental issues. Cleaning using wet foam flow (50% of air and 50% of SDS surfactant dissolved in osmosed water (0.15% w/w)) shows promising results in removing *Bacillus* spores' contamination, due to the interesting mechanical properties of the foam at the walls such as fluctuation of wall shear stress, liquid drainage, and capillary imbibition.

This study, aimed at testing the ability of this method to remove more complex contamination patterns, such as *Bacillus cereus* biofilms developed on stainless steel surfaces of 2B finish. Coupons were immersed horizontally in a soil suspension containing 10^7 CFU/ml and held at 30°C for 24 h. Then, half of the coupons were directly analysed after rinsing (count of cultivable cells, microscopic observation). The other coupons were subjected to different time cleaning procedures (wet foam cleaning at different average shear stresses between 2 and 13 Pa and cleaning in place (CIP) condition using the same surfactant at 10 Pa).

Observation of the foam detachment curves showed a detachment kinetics comprising two phases, the first is characterised by a very fast detachment (about 1.5 log CFU in <35 sec), the second by a slower detachment where the percentage of residual adherent bacteria after 20 min exceeds 2 log CFU. Increasing the wall shear stress improved the cleaning efficiency of the biofilms. But in the same time, the increase in frequency or the decrease in amplitude of the local wall shear stress could probably better explain this efficiency. When a CIP procedure was applied, the first phase, corresponding to a rapid but shorter detachment with a relatively inefficient second phase (1.2 log CFU of decimal reduction) inducing a higher number of residual adherent bacteria. Next, a life cycle assessment (LCA) of the cleaning procedures, SDS surfactant-containing foam, SDS surfactant-containing CIP, and NaOH-containing CIP, using SimaPro software, was performed. It was found that the CIP steps containing NaOH contribute the most to the environmental impacts, with the highest water and electricity consumption. While the foam flow impacts on energy and wastewater, emissions showed significant benefits.

CFD-based three-dimensional Modeling of an Adhesively Detaching Soil Layer in a Channel Flow with Sudden Expansion

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Cleaning is an important process step in the food industry, especially to avoid contamination during increasingly frequent product changes. The equipment is cleaned almost daily causing high ecological and economic expenses. Predicting the cleaning time required to remove a thin soil layer can prevent too many resources from being used. The authors recently presented a model for the cleaning mechanisms of adhesive detachment [1] and a model for cohesive separation. Both were successfully validated in a fully developed, predominantly two-dimensional channel flow with dried ketchup and starch, respectively. These two mechanisms are representative for a wide range of soils, so that the models developed are likely to be applicable to a large number of practical situations.

The purpose of the present contribution is to apply the model for an adhesively detaching soil at the next level of geometrical complexity. To this end, a square duct with a stepwise expansion of each wall, as depicted in Fig. 1, top, is considered. The Reynolds numbers of flow investigated are between 13.000 and 39.000, based on the hydraulic diameter of the upstream duct.

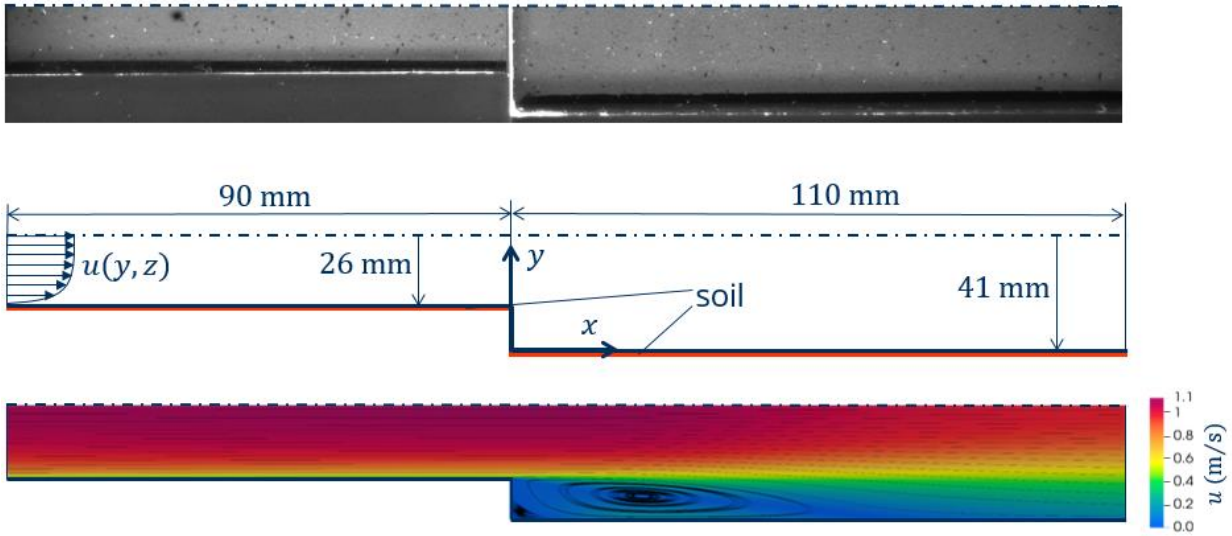
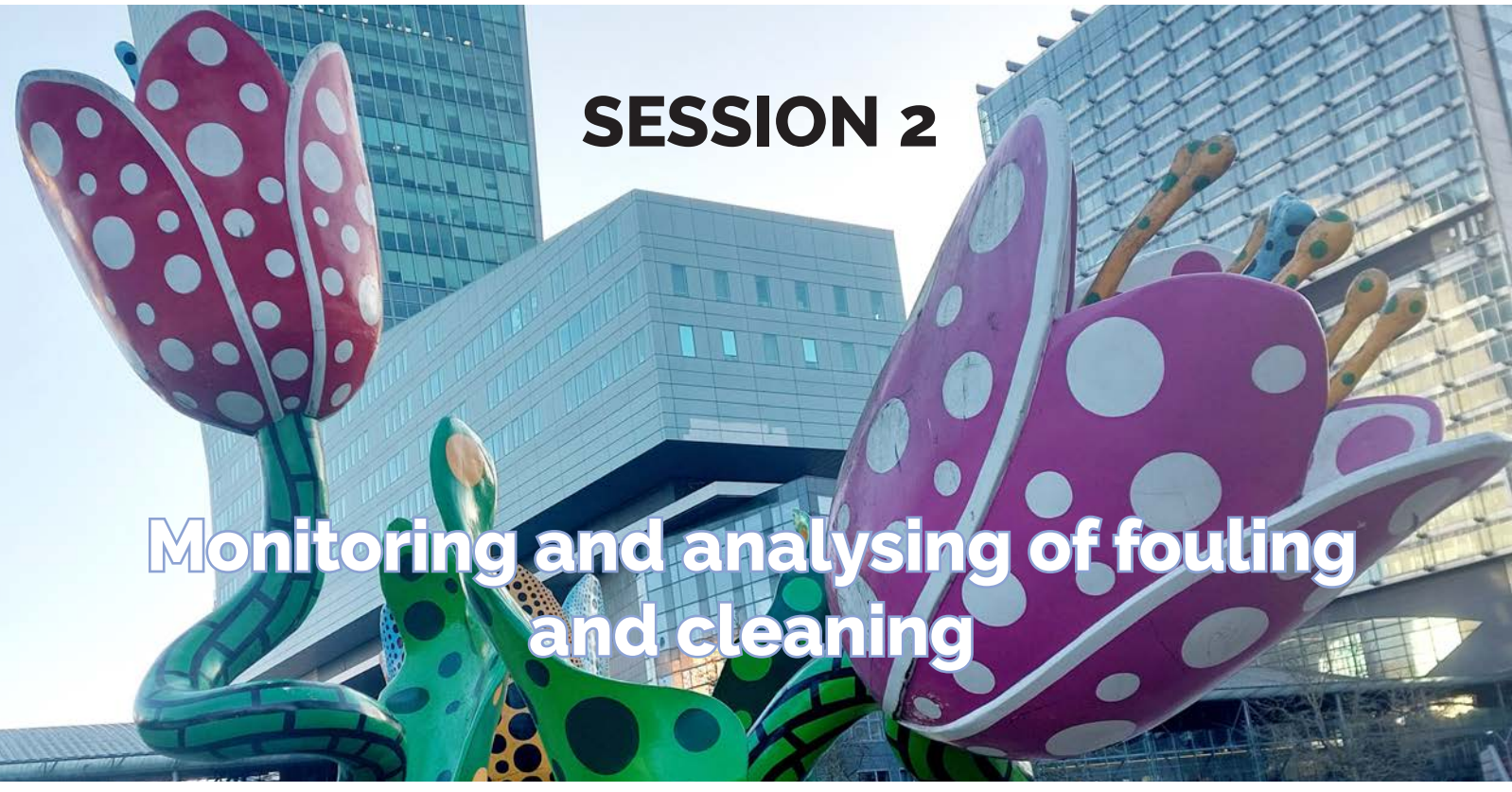


Figure 1: Investigated configuration of a channel flow with a sudden expansion. Top: photo of the experiment, with a top view on the soil. Center: sketch of the side view with relevant dimensions. Bottom: Preliminary simulation result showing the streamwise velocity u and the streamlines of the average flow.

In a square duct, the flow is three-dimensional due to secondary flows of the second kind, and the existing model for two-dimensional flow needs to be extended in the first step. The model is validated using cleaning experiments in a fully developed channel flow without step. After that, it is applied to the channel flow with sudden expansion. Various cleaning parameters, such as the soil mass coverage and the flow velocity, are varied and the quality of the model predictions is investigated. The results are compared with experiments in a channel flow with sudden expansion.

[1]. Köhler, H., Liebmann, V., Golla, C., Fröhlich, J. and Rüdiger, F., (2021). Modeling and CFD-simulation of cleaning process for adhesively detaching film-like soils with respect to industrial application. *Food and Bioproducts Processing*, 129, pp. 157-167.



SESSION 2

Monitoring and analysing of fouling and cleaning

Sustainability and cleaning: preparing for the future

Hein TIMMERMAN

Global Sector Specialist F&B at Diversey and EHEDG EXCO Chair SCPP, Belgium

The implications of the climate crisis and increased resource scarcity are felt across the planet, and create new, complex challenges for business and society. In this dynamic world, all food businesses must ensure they operate in a way that continually minimizes their environmental impact and delivers value to society. Besides the prerequisite demand of cleaning and hygiene to assure food safety, all products and services that enable cleaning should on top minimize their environmental footprint and operate more efficiently. All stakeholders from cleaning chemical suppliers, to OEM's that provide the process equipment to the end user have engaged in environmental, social and governance (ESG) priorities and diverse actions, with as goals and near-term targets which will further reduce our environmental footprint, address social inequality and deliver solutions to help reach overall sustainability goals.

Assuring a consistent cleaning result with the less environmental impact starts with the hygienic design: cleaning of food processing equipment also is a critical operation from an environmental point of view, since it is one of the most water -and energy-consuming operations as well as a wastewater generator. Minimising the environmental impact of the cleaning and disinfection operations, while maintaining hygienic standards, is a major challenge for the food industry. It has been proven with recent studies that hygienic design reduces environmental impacts related to sanitation of equipment and installations—from water, energy and chemical products, to wastewater and CO2 emissions—and consequently, can positively contribute to a cost reduction in the industrial activity. Overall, a 48 percent water savings was obtained when cleaning the hygienically designed equipment.

A second major impact is the route towards so called green cleaning: it can be defined as the use of cleaning products, tools, equipment and methods that protect the health of the end user, lower the total cost of cleaning, and prevent environmental damage. For cleaning to be 'green', the products must effectively remove pollutants and pathogens from the food surfaces without introducing new risks to occupants. Green cleaning methods are extremely important to ensure the products, tools, and equipment are used properly. Finding the right balance to tackle the various food soils with the right chemistry whilst mitigating the food safety risk is part of a well-balanced sustainable future.

Cleaning cohesive soil layers in a radial flow cell

Karthikeya P. DESHMUKH[1], Dragana ARLOV[2], Stewart CANT[3], Anders GORANSSON[2], Fredrik INNINGS[2,4] and Ian WILSON[1]

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[2]*Tetra Pak Processing Systems, Research & Technology, Ruben Rausings gata, 221 86 Lund, Sweden.*

[3]*Department of Engineering, Trumpington Street, Cambridge, CB2 1PZ, UK.*

[4]*Lund University, Food Technology and Engineering, Lund, Sweden.*

Efficient and reliable cleaning is essential for the manufacture of food, beverage, and pharmaceutical products. Successful prediction of 'cleanability' in cleaning-in-place operations requires (*i*) a good understanding of the flow behaviour and (*ii*) reliable knowledge of the factors determining the removal of a given soil. Computational fluid dynamics (CFD) codes can now provide high-quality quantitative information about industrially relevant flows, e.g. (*i*). The challenge is identifying (*ii*).

The radial flow cell (RFC) has been used by many workers to study the removal of thin films and particulates from flat, rigid surfaces. It has several attractive features, including a small footprint, straightforward sample preparation requirements, and 2-D (axisymmetric) geometry. The RFC provides well-defined but nevertheless complex flow characteristics when operated under steady flow at moderate Reynolds numbers. In this work, an RFC is used to investigate the cleaning behaviour of two different, thin soil layers under similar flow conditions. The soils tested were (*a*) dried layers of instant coffee, used to model removal by diffusive processes, and (*b*) dried layers of an abrasive cleaning suspension where the cleaning mechanism is dominated by particulate removal.

The RFC was fabricated from clear PMMA so that the soiled surface could be monitored during cleaning. The aspect ratio (gap height/inlet pipe diameter) could be adjusted from 0.5 to 4.2. A steady flow of water was provided by gravity, giving inlet Reynolds numbers in the range 500-1500. The flow behaviour in the RFC was simulated using the commercial CFD code Fluent™. The effect of aspect ratio on the transition from steady to unsteady flow, the formation and location of recirculation zones, and surface parameters such as local shear stress and convective mass transfer coefficient was studied, and the results were compared with experimental observations and the published literature.

The removal of each soil type from glass and 304 stainless steel surfaces was investigated for a range of aspect ratios, soil layer thicknesses and inlet Reynolds numbers. The primary removal measure was the appearance and growth of a circular region clear of soil. With the particulate soil, this was accompanied by a ring of translated particulates, while with the coffee, other cleared regions appeared. The average rates of removal are compared with simple models based on mass transfer.

Cleaning of thick viscoplastic soil layers by impinging water jets

Melissa W.L. CHEE, Ghadir GHASEMI, Mohammad RASHID, Rubens R. FERNANDES and D. Ian WILSON

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Impinging liquid jets are widely used in cleaning-in-place operations in the food and bioprocessing sectors to clean the internal surfaces of tanks and other process vessels. The jets can be created by spray balls, static or rotating nozzles, or spinning heads. The flow of liquid serves to (i) wet the residual product, soil layer or fouling deposit on the walls, promoting reactions or changes which facilitate removal, and (ii) generate hydraulic forces which drive removal. The hydraulic forces can be associated with impact, e.g. where a droplet or jet strikes the wall, or with flow – either as a fast moving film flowing radially outwards from the point of impingement or as a falling film.

Removal of viscoplastic soil layers is challenging as the presence of a yield stress requires higher levels of hydraulic force than many other substances. The removal of viscoplastic soil layers by impinging liquid jets has been studied by several workers recently, and workers such as Tuck et al. (2020) have reported a noticeable effect of layer thickness, where thick layers (which we define those as ones where the layer thickness is much greater the depth of the liquid film). They observed the formation of blisters as the jet penetrated the layer initially, which grew and eventually burst.

This paper reports a systematic study of blister formation and the effect of layer thickness on layer behaviour and the rate of removal. Layers of viscoplastic material of known thickness were prepared on transparent target plates and subjected to the impact of a coherent, turbulent water jet. Cameras mounted above and below the target plate allowed the size and shape of the region deformed by the jet to be quantified over time. Nozzle diameters of 1, 2 and 3 mm, and layer thicknesses up to 20 mm were studied. Layers were generated from Carbopol® gels, toothpaste, a hand cream (Nivea Soft®) and a range of food products whose rheology was determined separately.

For thin layers, the impinging jet created craters which grew steadily larger over time. For some materials, a transition to blister formation was not observed. For others, the ratio of layer thickness to jet diameter determined the onset of blister formation. Where blister formation was observed, the rate of removal was noticeably faster before the blister ruptured or burst: after this event, the rate of removal could be described by existing kinetic models. The factors determining blister formation and rupture are discussed.

Tuck, J.P. et al. (2020) Cleaning of thick viscoplastic surface deposits using an impinging jet: Effect of process variables. J. Food Eng., 266*, 109699.*

Experimental study on the cleaning effect of pulsating sprays

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The reproducible cleaning of open geometries and vessels in the food industry is usually ensured by automated Cleaning-in-Place systems, which spray the cleaning fluid onto the surfaces to be cleaned. The pulsating operation of nozzles is one, so far little investigated, possibility to improve the efficiency of these operations and thus to reduce the ecological footprint and the costs of cleaning.

Therefore, the influence of various operating parameters and other influencing factors on the temporal development of the cleaned area is investigated in an experimental study for the simplified configuration of a vertical water spray impinging on a horizontal stainless steel surface in a distance of 150 mm. The industrial full-cone and flat fan nozzles used in this research deliver volume flow rates of up to 8 l/min at operating pressures between 2 and 6 bar and pulsation frequencies of up to 10 Hz. Pulsation is generated by switching a pivoted-armature valve upstream of the nozzle. Vanilla pudding and rice pudding were chosen as food model soiling to study the cleaning properties of a homogeneous soil layer and a soil layer with particulate defects, respectively.

The results of the cleaning experiments indicate that the use of pulsating sprays is beneficial for certain configurations. Two phases of the cleaning process were observed during the investigations on the removal of vanilla pudding by means of a full-cone nozzle. During the transient start-up process, when the full-cone spray is not fully developed yet and resembles a large lump of liquid instead, pulsation had a positive effect. Compared to continuous operation, pulsation resulted in an up to 38% higher cleaning rate. At a later stage, when the cleaned area extends far beyond the area of impact of the liquid lump, continuous operation outperformed pulsation. In this stage, the cleaned area primarily depends on the relative on-time of the valve, which determines, for example, the position of the hydraulic jump.

When a flat fan nozzle was used to remove vanilla pudding, no positive effect of pulsation was observed for the influencing parameters investigated. The cleaning rate decreased almost linearly with the on-time of the valve. The reason for this is most likely that the fan builds up very quickly without changing the characteristics of the flow, indicated by high-speed recordings.

However, the effect of pulsation not only depends on the type of nozzle, but also on the soil. For the cleaning of rice pudding by means of a flat fan nozzle, pulsation increased the cleaning rate significantly. One possible reason for this is that grains in the rice pudding protrude from the dried layer and are thus subjected to very strong stresses by a spreading film flow. The influence of pulsation frequency, valve on-time, nozzle distance and soil properties is subject to further studies.

Supervised cleaning-in-place processes enabled by a fiber-optical fluorescence sensor

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The ongoing efforts to make food production sustainable and efficient require a considerable use of resources. Especially cleaning processes are consuming significant amounts of energy, cleaning agents and water. Since the determination of cleanliness inside a closed vessel is almost impossible, standard cleaning procedures are designed for the worst case contamination to ensure the required quality. However, in many cases, the requirements can be accomplished with less energy, time and resources given appropriate measures for the cleanliness inside.

To meet this demand, we have developed a fiber-optics-based sensor, which is able to detect fouling inside vessels using laser-excited fluorescence of the fouling deposit. The sensor can be integrated permanently into any pipeline. Since the tiny fiber tip is flush with the surrounding stainless steel surface, it imposes no significant perturbation of the fluidic system. As a result, fouling deposits accumulate in similar ways on top of the sensor and its surroundings during production. When the cleaning process is started, the sensor irradiates these deposits with a violet laser light and records its fluorescence emission. Thereby, even very thin layers of fouling can be detected by this highly sensitive device.

We have validated the technique using model deposits such as tomato paste, whey protein concentrate, milk powder and beer. The tests were performed in realistic and small-scale cleaning systems where the sensor result, i.e. the recognition of the clean state, was validated by visual inspection of the surface. Even small organic layers of about 1-5 µm whey protein are detected very reliably. The measurement frequency can be set up to 20 Hz, thus providing a real time assessment of cleanliness. Overall, the determination of the clean state is excellent, making the fiber-optical fluorescence sensor a promising solution for the monitoring of cleaning-in-place processes in food production.

Structural degradation along the cleaning front in cleaning a whey protein gel

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In order to meet the requirements of hygienically safe products, cleaning procedures in food processing plants are frequently oversized. This is disadvantageous for economic and ecological reasons and a reduction of deployed resources can only be realized if the cleaning process is adapted to the soil – and thus be understood. Milk processing is a characteristic example of a cleaning intense sector in the food industry and whey protein-based soils are being used extensively to study the underlying mechanisms of fouling and cleaning.

Whey protein-based deposits typically form a polymeric, three-dimensional network. Cleaning of such soils can be simplified as a multistep process where the cleaning agent diffuses into the soil, may cause physical swelling and chemical reactions degrade inter and intra molecular bonds. The soil network is weakened and finally network molecules or fragments diffuse back into the cleaning agent or are detached by the flow forces.
In this study the relation of the first diffusion step and the subsequent network degradation which form a cleaning front within the material was investigated. Therefor a whey protein gel was used with a height of 3 mm. The material strength of the gel was adapted to the strength of raw milk fouling layer as presented in [1, 2]. The diffusion front of sodium hydroxide into the gel is determined optically by applying a pH indicator as presented in [3]. Cleaning experiments were conducted in a transparent flow channel made of acrylic glass to enable a cross-sectional observation of the diffusion front. Experiments show that the diffusion front is reduced with increasing temperature as the removal is accelerated. The removability is investigated by inline Fluid Dynamic Gauging (iFDG) with a constant shear stress applied on the soil material by a siphon nozzle causing the detachment of weakened material. The portion of the soil which was withdrawn by suction is in good agreement with the diffusion rate. This suggests a fast weakening by degradation of the soil network.
Beyond that, the network degradation of the soil is analyzed visually by digital microscopy. First results show a defibering of network walls which serves as an explanation for the change in mechanical properties. The latter is characterized by the change of viscosity as a measure for the material stability.

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Design and development of a milking machine pilot : monitoring fouling and cleaning under real conditions – PiloTraite research program

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The partnership of this research program is composed of Actalia, AgroParisTech, BioDyMia-Univ Lyon1, Bourgogne-Franche-Comté, Pays-de-la-Loire and Pyrénées Atlantique Chambers of Agriculture, Comtois cheese technical center, French Livestock Institut, Lemma, Olivier de Serres agriculture school and Faculty of Agriculture and Life Sciences of Purpan. PiloTraite has received funding from the French Ministry of Agriculture, Agri-food and Forest and from Kersia.

During food processing, controlling microbiological quality of surfaces in contact with food remains essential. In a milking machine, milk and ambient air (which co-circulate) participate to fouling of surfaces and biofilm formation. In this context, milking machine conception, maintenance and especially cleaning/disinfection have to deal, in the same time, with sanitary, technological and environmental matters. The design and construction of a milking machine pilot was motivated by the need to study in real conditions fouling and cleaning/disinfection, at an intermediary scale between farm and laboratory.

In the PiloTraite research project, a milking machine pilot was designed in real size, including every standard constitutive elements, from clusters to the delivery line. Several specific materials (stainless steel, glass and polymers like rubber and silicon) were used. To our knowledge this pilot is unique. A sophisticated device carrying removable stainless steel coupons was included in the system. This device enables to proceed to physico-chemical and microbiological characterization after extraction. Moreover, a transparent part of this device allows a direct vision of the fouling inside the pilot.

To investigate biofilm formation, a methodology was developed including different protocols : *i)* contamination of the pilot (for example, a complex biofilm collected from a farm milking machine), ii) cleaning and disinfection and iii) sampling for analyzes (physico-chemical and microbiological characterizations).

This unique pilot will be further used to understand the parameters involved in the fouling (milk properties, temperature, flow rate…) as well as to improve the performance of cleaning and disinfection procedures.

Direct measurement of the cohesive strength of whey protein gel in contact with NaOH by wire cutting experiments

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Keywords: cohesive strength, binding forces, fracture toughness, swelling, wire cutting, whey protein

In the food or pharmaceutical sector, fouling deposits in heat exchangers or other machine areas reduce the efficiency and endanger the quality and safety of products. For the design of cleaning processes in general and for the parameterization of process models in particular, it is necessary to determine cleaning-relevant material properties of the soils and their change depending on the cleaning liquid and the soaking time. The dominant cleaning mechanism depends on the balance of cohesive strength within the soil and the adhesive strength to the substrate.

This paper describes a method for the direct measurement of the cohesive strength of whey protein gel (WPG) with a protein content of 15 % and a treatment temperature of 80 °C.

The forces required to separate the protein gel with a wire are determined by varying the wire diameter. By approximating the cutting force as a function of the wire diameter, interactions of the wire (geometry, friction) on the cutting force can be compensated to determine the cohesive strength. The method developed for this purpose deals with the control of the wire pretension based on the acoustic measurement of the first natural frequency of the vibrating wire. To define the cutting position of the wire within the gel, the local movement of the phase boundary between soil and cleaning fluid as a function of the soaking time for the considered cleaning fluids was optically determined. The cohesive strength of the virgin and the two states (transparent, opaque) of the WPG as a result of interactions with sodium hydroxide were determined.

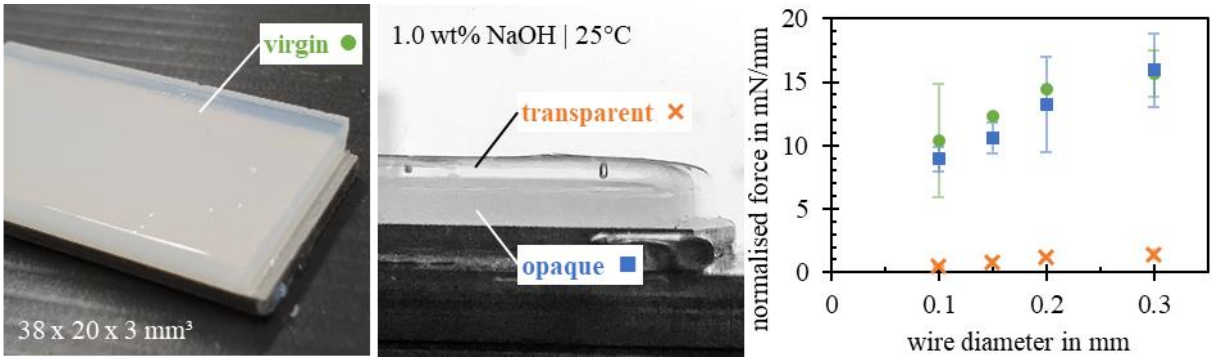

Fig 1: sample plate with virgin WPG (left), transparent and opaque phase of WPG after 990 s in contact with NaOH (middle), normalized forces for the three states of WPG for different wire diameters (right)

Hygienic design aspects for tank and vessel cleaning in the food industry

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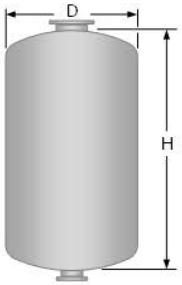
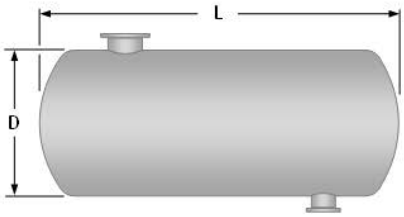
Tank cleaning is an important process in the food industry, not only to manage food safety and quality aspects of the products to be produced but also the key to do this in cost effective and in a sustainable manner. To provide a basic understanding of the cleaning and hygienic design of tank cleaning devices and the tank they intend to clean, a new EHEDG (European Hygienic Engineering and Design Group) guideline has been developed. The EHEDG workgroup consist of experts from the leading equipment manufacturers, hygienic design specialists, end-users and universities combining practical knowledge with leading-edge research in vessel cleaning and soil behaviour.

The guideline presents a tool for the initial selection of tank cleaning device technology and the background information needed for all interest groups to gain a better understanding of the important issues and parameters related to vessel cleaning.

The guideline provides an overview of different tank cleaning technologies, their cleaning mechanisms, and associated hygienic design requirements. In addition, important installation requirements for the cleaning devices are provided. Hygienic design aspects to manage food safety are covered, as well as important aspects that influence sustainability aspects (e.g., water usage) and total cost of ownership. Moreover, the hygienic design of the vessels to be cleaned are explained, which is an important pre-requisite to facilitate an effective cleaning process.





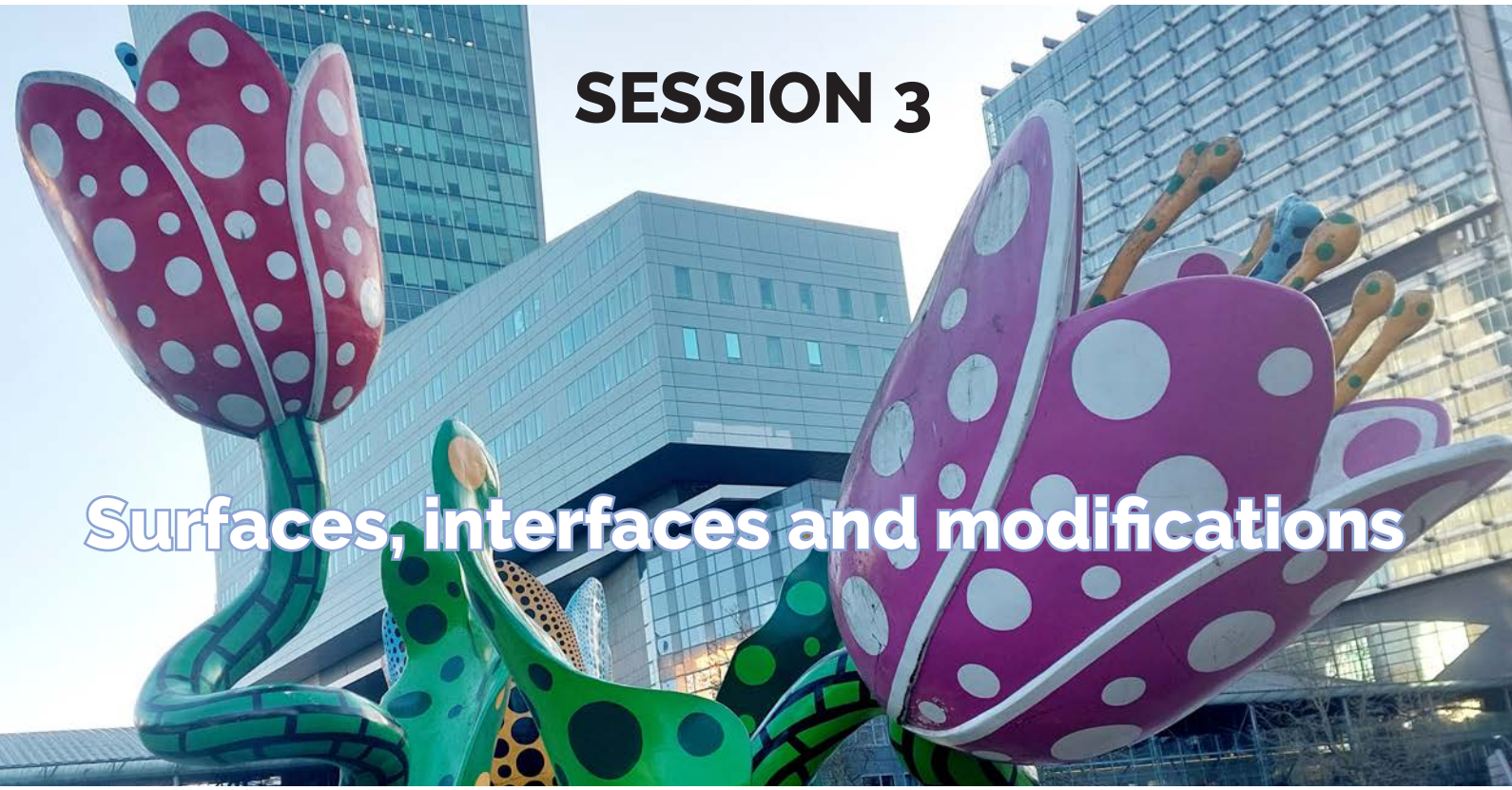
Tank size & orientation	Soil*	Cost - investment				Cost - Operational				Liquid usage				Cleaning time				Pressure			
		Static	Single-Axis	C.Single-Axis	Multi-Axis	Static	Single-Axis	C.Single-Axis	Multi-Axis	Static	Single-Axis	C.Single-Axis	Multi-Axis	Static	Single-Axis	C.Single-Axis	Multi-Axis	Static	Single-Axis	C.Single-Axis	Multi-Axis
D < 2 m; H < 2-3xD L < 2 m; L < 2-3xD	light, easy to rinse			NR	NR			NR	NR			NR	NR			NR	NR			NR	NR
	medium soil				NR				NR				NR								NR
	high soil, mechanical action required	NR				NR				NR				NR				NR			
2 < D < 4 m; H < 2-3xD 2 < L < 4 m; L < 2-3xD	light, easy to rinse				NR				NR				NR				NR				NR
	medium soil	NR				NR				NR				NR				NR			
	high soil, mechanical action required	NR				NR				NR				NR				NR			
D > 4 m; H < 2-3xD L > 4 m; L < 2-3xD	light, easy to rinse	NR				NR				NR				NR				NR			
	medium soil	NR				NR				NR				NR				NR			
	high soil, mechanical action required	NR	NR			NR	NR			NR	NR			NR	NR			NR	NR		

*For indications of what is light, medium and high soil see Table 2.

Figure 1: Decision support tool for selection of tank cleaning device (from the upcoming EHEDG guideline 51 Hygienic design aspects for tank and vessel cleaning in the food industry)

The EHEDG guideline on hygienic design aspects for tank and vessel cleaning in the food industry is an important supplement to the recently published EHEDG guidelines on other aspects of cleaning in the food industry.

This presentation will highlight some important aspects of tank cleaning in the food industry.



SESSION 3

Surfaces, interfaces and modifications

Cleanability of Laser Etched Biomimetic Surfaces with Repeated *Staphylococcus aureus* and Milk Fouling

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The accumulation of microorganisms and/or organic material can result in surface biofouling, and this is a particular problem in the food industry. It has been suggested that one way to control microbial/organic material adhesion and subsequent biofouling is by using biomimetic surfaces. A laser surface texturing process was used to produce six differently patterned topographies (three rippled and three spiked). The surfaces were analysed for their topography (optical profilometry) and wettability (contact angle). The surfaces were spray inoculated with *Staphylococcus aureus* suspended in either sterile distilled water or whole milk, then spray cleaned with a chlorinated, alkaline cleaner. The surfaces were spray cleaned 1, 5, 10, 15, and 20 times and were analysed for changes in changes in topography, wettability and biofouling (epifluorescence microscopy).

Analysis of variance (ANOVA) was conducted to assess the effect of the surface factors on biofouling, whilst principal component analysis (PCA) was used to discern underlying relationships. The results demonstrated that of the six surfaces with different topographical patterns, three had much rougher surface features (spikes (S_a 15322µm, 12172µm, 7018µm) than the rippled featured surfaces (26µm, 19µm, 15µm). The rippled surfaces were all fouled by 20 cleans, regardless of surface type or clean, had higher levels of biofouling in milk (98.16 %) than the spiked surfaces (57.80 %), and demonstrated an increase in biofouling as the number of cleaning cycles (and refouling) increased.

There were no significant differences in the overall level of biofouling between the different rippled sub-textures. The number of cleans particularly affected the properties of the rippled surfaces, predominantly the wettability and biofouling. Surface texture sub-type did not influence biofouling of the rippled surfaces. However, the spiked surfaces showed no overall increase in biofouling and the number of cleans was not inducive to a change in surface properties. In addition, biofouling of the surfaces with spike topographies were predominantly influenced by the texture sub-type.

This work demonstrates that biomimetic surfaces can be used to reduce biofouling, but particular attention should be afforded to the careful selection of surface design.

Surface engineering of stainless steel to mitigate dairy fouling adhesion

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Unwanted fouling deposits are formed on heat exchangers' surface during mandatory thermal treatments (pasteurization, sterilization) of dairy products in food processing industries. These deposits can contaminate dairy products to pasteurize and also impair heat transfer mechanisms by creating a thermal resistance, thus leading to regular shut down of the processes. Therefore, periodic and drastic cleaning-in-place (CIP) procedures are implemented. These CIP involve the use of chemicals and high amount of water, thus increasing environmental burden. It has been estimated that 80% of production costs are owed to dairy fouling deposit.[1] To reduce dairy fouling, stainless steel surface is modified to either inhibit attachment of depositing species or to ease their removal during cleaning. Here, we focus on this latter approach, by developing specific coatings (Slippery Liquid-Infused Surfaces (SLIS) [2] and atmospheric plasma coatings [3]) of low contact angle hysteresis to limit fouling adhesion onto stainless steel surfaces. First, SLIS are inspired by *Nepenthes* plant by designing slippery interface between the substrate and the fouling providing very efficient fouling-release surfaces.

Slippery surfaces were elaborated in three steps: (i) femtosecond laser surface structuring, (ii) silanization and (iii) lubricant impregnation. In order to maximize lubricant retention and manufacturing speed, laser manufacturing parameters were optimized. To develop food compatible SLIS, coconut oil was used as a lubricant and compared to a fluorinated oil.

Second, plasma nano-structured coatings intend to mimic lotus leave surfaces, by creating a dual-scale roughness preventing adhesion of denatured dairy proteins. Superhydrophobic double layers based on fluoro and silane coatings were sprayed by atmospheric pressure plasma (Lab-Scan, Axcys Technologies) and conditions were optimized depending on the fouling test results obtained.

Finally, both processes (femtosecond laser surface structuring and atmospheric pressure plasma) were combined to elaborate SLIS and lotus-like surfaces. Promising fouling-release performances have been obtained for both SLIS and plasma nano-structured coatings.

Fig.1: SEM images of hydrophobic plasma coating (left) and laser textured surface (right)

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The authors thank Region Hauts-de-France for its financial support through the ECONETIAA project, the French National Agency (ANR) for its financial support (ECONOMICS Project ANR-17-CE08-0032) and the french Renatech network (Micro Nano Fabrication Plateform and NearField Platform)

Biomimetic replication of brassica leaves and their efficacy to prevent biofouling of *Escherichia coli* and *Staphylococcus aureus*

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Biofouling in the food industry is a huge issue that results in costly cleaning procedures and considerable use of cleaning agents. One way to reduce the amount of cleaning is to design naturally cleaning surfaces based on biomimetic designs.

Four self-cleaning leaves (Tenderheart cabbage, Cauliflower leave, White cabbage, and Leek) were analysed for their surface properties. The leaves were subjected to attachment, adhesion and retention assays using *Listeria monocytogenes* and *Escherichia coli*. Using water contact angle, it was determined that all the surfaces were non-wettable, with the Tenderheart cabbage demonstrating the most non-wettable surface (147°). However, when the hydrophobicity of the surfaces was determined, the White cabbage was demonstrated to be the most hydrophobic surface (ΔG_{iwi} = -88.7 mJ m^{-2}), which was due to high γ_s^{LW} (17.4 mJ m^{-2}) and low γ_s^{AB} (0.2 mJ m^{-2}) values.

The White cabbage demonstrated the lowest S_a (3.0 µm), S_q (3.5 µm) and S_{pv} (29.0 µm) values, being the least rough surface. However, the White cabbage also showed the widest, rounded surface features of all the leaves. Radar graphs were used to represent the physicochemical parameter values. All the leaves had non-polar adaxial surfaces demonstrated by the higher value of the γ_s^{LW} than their corresponding γ_s^{AB} values. The thin asymmetric diamond shapes with a long upward pointing arm were indicative of high γ_s^{LW} values and lower γ_s^{AB}, γ_s^{+}, and γ_s^{-} values. This demonstrated that the White cabbage was the most hydrophobic leaf, followed by the Cauliflower leaf.

The less hydrophobic surfaces of the Tenderheart and Leek leaves had higher γ_s^{-} values, which led to graphs with wider bases and various triangular shapes. The FTIR spectra showed that although all the leaves had some of the same peaks (3300 cm^{-1}, 2848 cm^{-1}, 1736 cm^{-1} and 1018 cm^{-1}), each leaf also demonstrated individual peaks. *L. monocytogenes* were generally bound to the surfaces in lower numbers than *E. coli.* The leek leaves retained more *E. coli* cells than the other cabbages. The Tenderheart cabbage promoted *E. coli* attachment and adhesion. Hence, for *E. coli,* surface hydrophobicity was not a controlling factor of the bacterial retention, attachment and adhesion. The Leek leaves (the least hydrophobic surface) retained more culturable *L. monocytogenes* cells than the other cabbage leaves, suggesting a correlation between surface hydrophobicity and the retention and attachment of *L. monocytogenes.*

This work demonstrates that the biological factors and environment need to be taken into consideration when designing self-cleaning surfaces based on biomimetic principles.

Bacterial contamination at the air-liquid-wall interface. Relative roles of physico-chemical vs bacterial phenomena.

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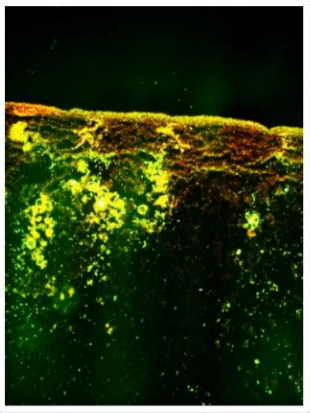

Introduction. Many data available in the literature on biofilms in the food industries relate to fully submerged biofilms. Conversely, the role of air-liquid-wall (ALW) interfaces on surface contamination has been the subject of relatively few publications, in spite of the warnings issued by some authors about the risk due to these areas on the control of surface hygiene procedure. Studies available in the literature have indeed shown that pathogenic bacteria (e.g. *Salmonella spp*. or *Bacillus spp*.), or spoilage bacteria (*Pseudomonas spp.*) are able to form biofilms at ALW interfaces. The aim of the current study was to investigate the role of material hydrophobic/hydrophilic properties on the adhesion of bacterial spores (simple adhesion) and on the formation of biofilms at the ALW interface, to identify the relative role of physico-chemical and microbiological phenomena in the formation of these structures.

Materials and methods. Three *Bacillus* spores, were used in this study: *B. anthracis* 9131 (hydrophobic, surrounded by a flexible membrane called exosporium) and *B. subtilis* PY79 spsI and yfnH (hydrophobic vs hydrophilic, without exosporium). The formation of deposits at ALW interfaces was monitored on 3 materials: stainless steel with a 2R surface finish (SS-2R), glass and polypropylene (PP). For the adhesion tests, coupons were partially immersed in an aqueous suspension of spores (dormant bacterial form). For biofilm formation, three bacterial strains were used: *B. cereus* 98/4, *E. coli* SS2, and *P. fluorescens* 1, and the soiling suspension was composed of vegetative bacteria in a growth medium (TSB). The bacterial deposits at ALW interfaces (distribution on the surface, 3-D organization) were observed at different times, ranging from 1 to 24 h, by epifluorescence and confocal laser scanning microscopy (CLSM, Zeiss, LSM780).

Results. We first observed that the amount of spores adhering to the ALW interface increased with time and then sometimes stabilised. The maximum was reached relatively quickly for hydrophobic spores [between 2 and 4 h for Ba 9131, at 8 h for Bs PY79-spsI] and at 24 h (or more) for the hydrophilic spores of Bs PY79-yfnH. In contrast, the influence of the material is the same regardless of the properties of the spores. Both hydrophilic materials are highly contaminated at the interfaces, while PP is only little or extremely little contaminated depending on the spores. Furthermore, the architecture of the spore deposit also depended on the material. First, some 3D-structures were observed by CLSM on glass and SS-2R, not on PP. Moreover, the deposition line was larger on the hydrophilic materials. One explanation could be that spore adhesion is promoted at the meniscus, which was more marked on hydrophilic materials (2.5 mm, 1.5 mm, and -0.5 mm on glass, SS-2B and PP, respectively). Similar trends were observed on biofilm formation. Despite the different abilities of the three bacteria to form biofilms at interfaces, PP was constantly the least contaminated material by a 24 h biofilm and the biofilms were thinner on this material.

Significance. This work highlights the importance of physicochemical phenomena in the formation of biofilms at ALW interfaces.

The hygiene factor as an improved description of the hygienic quality of food contact surfaces

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Research articles and studies in literature have highlighted that the intrinsic surface characteristics of a food contact product has a great impact on the cleanability of the surrounding materials and foodstuff. However, a clear correlation between surface topography and cleanability has not yet been scientifically proven and established.

With this aim, we have developed a hygiene factor based on the surface roughness profile and correlated it to practical hygiene testing.

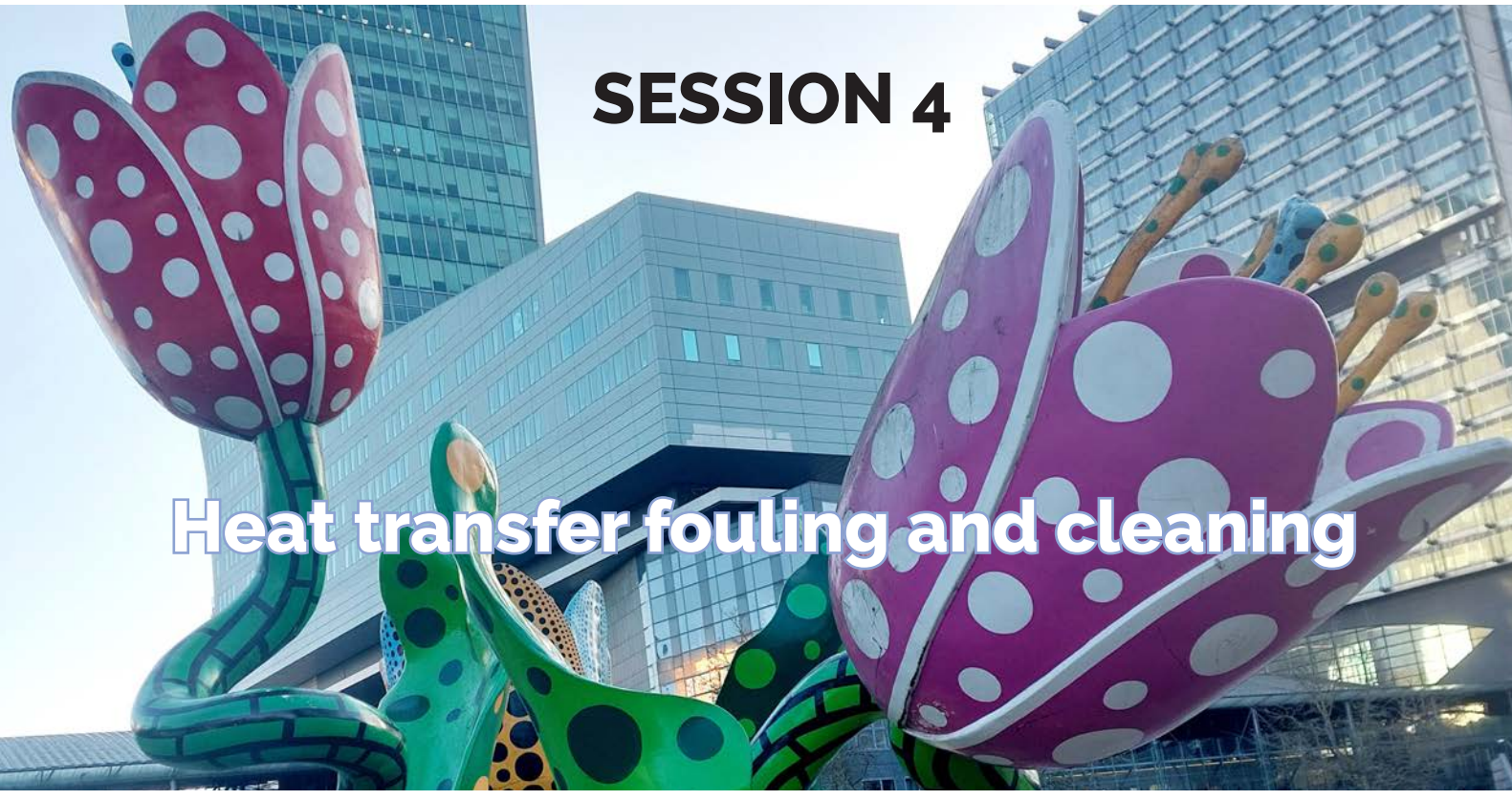
The current industrial guidelines and rules of thumb are based on the characterization of surface roughness given by the R_a value, which is often measured only in one direction across the surface. One of the traditional measurement methods employ moving a physical pickup across the surface, however characterisation is evolving to methods based on microscopy. This characterization of the surface characteristics by only one value is a major simplification, and it is evident by simply observing a typical surface topography.

Therefore, the hygiene factor we have developed also includes the number of peaks on the surface, giving a more adequate description of its topography. Moreover, the entire measurement is carried out by using an optical 3D microscope, thus avoiding physical contact with the surface.

The hygiene factor is defined as the inverse product of R_a (the geometric mean distance from the mean line of roughness profile) and R_{pd} (the peak density i.e., the number of peaks per cm of the roughness profile).

The validity of the hygiene factor has been evaluated and verified with both stainless steel and plastic plates. The tested stainless-steel surfaces had different finishing (grinding, polishing, bead blasting and ViwaTeq®), and the tested plastic surfaces were obtained by injection moulding using mould with different surface roughness.

The overall goal is to provide the industry a tool for the characterization of materials in terms of hygienic quality by using 3D optical microscopy and the calculated hygiene factor. In addition, the hygiene factor can have high practical relevance for industry, e.g. in case of comparing the hygienic functionality between new surfaces and surfaces in use.



SESSION 4

Heat transfer fouling and cleaning

Thermal treatment of liquid foods under fouling: whole system analysis

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The heat treatment (pasteurisation, sterilization) of liquid food products, typically conducted in an energy integrated system of three plate heat exchangers (PHEs) and a holding tube, presents many challenges. The key process requirement (that the fluid be held above a target temperature for a minimum time) is not easy to measure and monitor. Due to fouling, operations involve heating-cleaning cycles, i.e. are done in a batch mode, with production interrupted during cleaning. The process is energy, water and wastes intensive. Highly complex interactions arise between equipment, thermal, hydraulic and fouling behaviour, their evolution over time, resources used, wastes produced and overall economic performance.

A detailed distributed dynamic model is used for each of the process equipment components: individual PHEs and connecting tubes. The model of each component consists in a set of partial differential equations, accounting for the key underlying phenomena at each point along the flow path (assumed 2-dimensional): thermal behaviour (for heat transfer and temperatures), hydraulic behaviour (for pressure drops), thermophysical properties of all streams, fouling behaviour (based on a typical protein reaction model) and deposit growth models (Guan and Macchietto, 2018, Sharma and Macchietto, 2021). The PDAEs solution gives at each point (of each side in each channel for PHEs, each axial length point for tubes) the temperatures, protein compositions in the strems, reaction rates, deposition rates and deposit thickness. The models of all units are linked together (according to the process flowsheet) in a dynamic model of the integrated process. The integrated process model can be used to calculate its evolution over time, for example as a function of changes in operating conditions, fouling characteristics, control scheme (Zhu et al, 2020), or scheduling of operations (Tseng and Macchietto, 2021). All modelling and simulations are done using gPROMS 7.0.7 (Process Systems Enterprise).

In this paper, based on the above overall dynamic process model, a new visualisation method is presented, which allows us to much better understand and monitor the overall process. A case study for a typical high temperature, short time (HTST) heat treatment of milk shows that pasteurisation (which happens when the fluid is held above a target temperature) occurs in two of the PHEs and in the holding tube, while the actual pasteurization time changes over time due to fouling. The model-based method presented allows it calculation, visualisation and monitoring, accounting for the key aspects: equipment geometry, flow configuration in the PHEs, fluid properties, fouling and deposition characteristics, and operating conditions (inlet flow rates and temperatures, heating and cooling control). The new diagram is also used to depict the local distribution in the entire system, at a given time, of important quantities such as heat transfer rates, shear rates, deposition rates and deposit thickness.

The diagram gives new insights into the operation of the system overall and its interacting elements. First, it highlights a clear, strong relationship between the relative flow directions within a PHEs, heat flux pattern and corresponding effects on fouling rates and deposit thickness distribution along the equipment. PHEs are characterised by various combinations of co-current or counter-current flows in adjacent channels. These depend on both the PHE configuration (as single or multi-pass on the hot and cold sides) and the PHE utilisation (as pre-heater or main heater). The analysis evidences a very complex heat transfer picture, which is different for the various PHEs in the system.

Such detailed understanding is used to devise an alternative pasteurisation process. It is shown that adding an exchanger and holding tube to the traditional scheme and using them in a suitable rotating

schedule of preheating, heating, and cleaning, the integrated system can achieve uninterrupted milk production in a semi-continuous operation, where milk production can be maintained indefinitely.

A case study for a typical HTST heat treatment of milk demonstrates the use of the novel visualisation and the development of a new semi-continuous process with significantly higher throughput (+23%) and lower cost (-47%) than the traditional (batch) operation. The reduction in energy and cleaning resources used, and wastes produced, hence improved process sustainability, are discussed.

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Influence of steam-induced wetting and soaking of food- and cosmetic based contaminants on the efficiency of cleaning-in-place processes of containers

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The cleaning of tanks or containers in the food and cosmetics industry with conventional tank cleaning systems requires a large amount of cleanser. Especially for swellable soiling, complete wetting of the surfaces and sufficient soaking of the soiling is advantageous for an efficient cleaning process.

For soaking, only a small amount of cleanser needs to get in contact with the soil. Using conventional tank cleaning systems, significantly more cleanser is used during the soaking step than required. An aspect that can have a disadvantageous effect on the cleaning process is that tank installations and a complex tank geometry may hinder the complete wetting of the surfaces to be cleaned. Flooding the tank with wet steam prior to cleaning to initiate the soaking process in a resource-saving manner addresses these issues.

In this work, steam of varying saturation was injected into a test container to cause soaking and swelling of different soils prior to spray cleaning. The test setup consisted of a rectangular stainless-steel container with a volume of 100 L, a steam generator, a CIP-System and an UV-camera sensor for monitoring of the cleaning process. Using this test set-up, the influence of a first soaking step on the duration of the subsequent spray cleaning was investigated and compared with a conventional spray cleaning process. Flooding the tank with wet steam prior to cleaning reduced the cleaning time for most of the soiling. As a result, a reduction of up to 46 % of the used cleanser was achieved.

Effects of Casein and Carrageenan on Whey Fouling during Pasteurization

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Sweetened dairy products such as flavored milk, ice cream, yogurt, and milk shake are important foods on the market nowadays. However, thermal treatments on these products result in more severe fouling than milk due to their complex composition. While most of current research in dairy fouling focuses on whey, the effects of casein (CS), the most abundant protein in milk, and non-milk ingredients like carrageenan (CRG; used as texturizer, stabilizer and fat replacer in dairy products) are rarely studied, hence only little is known about the fouling mechanisms of complex dairy products.

This study aims to provide fundamental understanding of the interactions of CS and CRG with whey under pasteurization conditions and how they affect protein fouling. The fouling behaviors of different model dairy solutions composed of whey protein isolate (WPI; 2 wt%), CS (0.5 wt%) and CRG (0.03 wt%) were characterized under well-controlled temperature and flow using a spinning disc apparatus. Adding CS to WPI solution increased the fouling tendency because CS reduced the denaturation temperature of WPI. However, CS-WPI deposit featured a more porous structure that resulted in a lower dry mass compared to WPI deposit. The addition of CRG increased both the fouling resistance and deposit mass owing to the formation of more compact deposits. Fluorescence and Raman spectroscopy analyses on protein structure and tryptophan residues in β-lactoglobulin (β-lg) proved that CS can make β-lg less heat-stable and promote its denaturation, leading to increased fouling.

This study can serve as the groundwork for investigating the fouling of complex dairy products, and ultimately helping manufacturers select proper ingredients to mitigate dairy fouling.

Effect of micellar casein decalcification on the fouling the UHT plant and on the heat stability of high protein beverages

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The increase in demand for nutritional food products has motivated the development of new milk protein-based nutraceutical products that may play a role in weight management, meal replacement and the treatment of people with acute illnesses or suffering from nutrient deficiencies. In this context, our work aimed to study the impact of the calcium-reduction level (0, 10 and 40%) of micellar caseins and the presence of other ingredients, such as sugar and fat, on the heat stability and fouling behavior of neutral high-protein (14%) drinks after UHT treatment at 143°C with 5 s holding time. The assessment of heat stability was done using UHT pilot-scale tubular heat exchanger and a run-time of 120 min. The volumetric flow rate was kept at 200 l/min. Our goal is to identify the suitable modified-structure of micellar caseins for the development of high protein beverages.

Our results showed that when the decalcification degree of micellar caseins has increased from 0 to 40%, the fouling rate drastically decreased by 10-fold for casein dispersions without sugar and fat. In addition, the results of SDS-Page, HPLC and ICP-MS revealed that the increase of decalcification degree resulted in deposits with a high content of minerals. In fact, the increase of decalcification degree from 0 to 40% promoted an increase of the mineral % from 13 to 70 % and a decrease of the protein % from 77 to 7 %. On the other, our results underlined that the addition of sugar and fat induced an increase of the fouling deposition for all drinks and this increase was mostly linked to the increase of casein protein deposition. Furthermore, the increase of the calcium-reduction from 0 to 40% resulted in a 6-fold increase in the apparent viscosity. The addition of fat and sugar also resulted in an increase of the apparent viscosity whatever the calcium-reduced level.

Overall, our results underlined that decalcification, which reduces the concentration of calcium ions, seems to be a good option in order to improve to heat stability of micellar caseins at high protein concentrations and to reduce the fouling of the UHT plant.

Insolubilisation of mineral salts during vacuum concentration of whey in relation to fouling of dairy evaporators

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Ultrafiltration permeate is a co-product of cheese and casein manufacture in the dairy industry. It consists mainly of water, lactose, whey proteins and minerals. In order to reduce storage and transportation costs or for further processing, ultrafiltration permeate is concentrated using vacuum evaporation. During ultrafiltration permeate concentration in falling-film evaporators, the mineral fraction undergoes strong modifications due to the exceeding of the solubility limits of some salts and their subsequent precipitation. It may lead to the deposition of some matter onto the hot surfaces of the evaporation tubes, which reduces the performances of equipment. The mass deposition depends mainly on the mineral composition of the ultrafiltration permeate but this latter is highly variable and is function of the processes used to obtain the ultrafiltration permeate.

In this study, we investigated to what extent mineral composition and initial pH influenced the nature and the quantity of precipitate formed when concentrating different ultrafiltration permeates. Model ultrafiltration permeates with variable mineral compositions and pH were produced by acidification of milk to pH values between 4.6 to 6.3 using different acids (hydrochloric, citric and lactic) followed by ultrafiltration. They were representative of the diversity of ultrafiltration permeates encountered in the dairy industry.

The results showed either no precipitation or the precipitation of calcium salts in the concentrates. The nature and the quantity of precipitated salts depend on the process used to obtain ultrafiltration permeate. In ultrafiltration permeate concentrates, the main anions phosphate, citrate and lactate are in competition to associate with calcium. When the solubility limits of the least soluble calcium salts (calcium phosphate and calcium citrate) are exceeded, salt precipitation takes place. The behaviour of these ions during concentration could be estimated by considering: (i) the nature and amounts of cations and anions in the concentrates, (ii) the anionic ionic forms present at the pH of the concentrates and their association constants with calcium, (iii) the solubility limit of calcium salts.

Effect of calcium on the thermal denaturation of whey proteins and subsequent fouling in a bench-scale fouling rig: couple experimental studies and numerical modeling

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The unwanted formation of deposits formed upon the stainless steel surface of plate heat exchanger (PHE) during thermal processing is an unresolved issue in the dairy industry. This fouled layer reduces heat transfer efficiency, therefore deteriorates the quality of the product. After decades of study, it has long been recognized that the thermal denaturation of the main component of the whey protein (*i.e*. beta-lactoglobulin, BLG) is the key that drives fouling. Generally, BLG denaturation can be described using a two-step consecutive reaction as N→U→A where the native BLG (N) unfolded its tertiary structure and expose a free thiol group. The unfolded BLG (U) becomes activated and can interact with other whey proteins or with κ-casein on the casein micelle surface to form aggregates (A) through thiol/disulfide exchanger reactions.

It is widely accepted that the unfolded BLG generated in the bulk is the precursor for fouling growth. For this and other reasons, the denaturation kinetics of BLG has been extensively studied as well as several important factors that affect it. Among them, calcium ion content has been recently noted to have a dramatic impact both on the denaturation and the deposition processes. Nevertheless, most of attention has been paid to the calcium effect concerning thermal denaturation of BLG, how exactly calcium affects the deposition reaction has never been studied exhaustively. Conceptually, dairy fouling is typically considered to comprise mass transfer, bulk reactions and surface reactions. The first two can nowadays be well calculated for a given geometry using computational fluid dynamics (CFD) combined with thermal denaturation kinetic models. However, it is difficult to obtain deposition kinetics as most studies are performed in industry-like systems such as those in pilot plant PHEs. The complexities of the fluid mechanics and the configuration itself in the PHEs make it hard to investigate the surface reaction kinetics concerning the fouling growth.

To this end, the first step of this study is to develop a bench-scale fouling rig that could provide simple configuration and thus simple fluid mechanics (*i.e*. laminar regime). Model fouling fluids containing 0.5 wt.-% whey proteins with elevated ionic calcium concentrations were applied to investigate the effect of calcium on the deposition kinetics. The benchtop-scale fouling rig was designed to provide a rectangular microchannel with a dimension of 2 mm width, 3.5 mm height as shown in Figure 1(a). A schematic of the fouling set-up is displayed in Figure 1(b), where the device was mounted upon a plate heater so as to heat the solution gradually from inlet to outlet. A flow rate of 2 $ml \cdot min^{-1}$ with a constant surface temperature of the plate heater set at 90 °C resulted in a temperature profile of the solution ranging from

60 to 83 °C (in u-shape tunnel). The next step of this study is to perform a realistic 3D simulation on the whey protein denaturation and subsequent deposition based on the geometry of the fouling rig. The thermal denaturation of proteins was numerically simulated concerning the effect of calcium effect, after which, the deposition reaction kinetics can be calculated with the combination of experimental fouling deposition rates. The numerical model was built using the same dimension of the benchtop device.

For deposition reaction, we assume a surface reaction that occurs only on the bottom surface of the channel (upon stainless steel) with a reaction order of one:

$$\frac{\partial C\mathrm{D}}{\partial t} = k\mathrm{D}C\mathrm{U} \quad \text{(eq. 1)}$$

The temperature dependence of the deposition reaction constant $k\mathrm{D}$ can also be expressed by the Arrhenius equation:

$$k\mathrm{D} = k\mathrm{D}0exp\left(\frac{-E\mathrm{aD}}{RT}\right) \text{ (eq. 2)}$$

where $k\mathrm{D}0$ is the deposition frequency factor (m·s^{-1}) and $E\mathrm{aD}$ is the activation energy (J·mol^{-1}) for the deposition. The surface reaction directly determines the mass flux of unfolded BLG across the liquid-solid interface. The deposition kinetic constant $k\mathrm{D}$ can be calculated by combining the simulated unfolded BLG concentration on the surface $C\mathrm{U}$ with the experimental deposition rates (average value for the whole deposition area).

Figure 1(c) presents simulated concentrations of different BLG species during heating process along the microchannel. Satisfactory agreements can be found between the experimental values of soluble BLG concentrations at the outlet of silicone tube with simulated values as shown in Figure 2(a) which consolidates the simulation methodology. Figure 2(b) shows a quasi-linear relationship of fouling rate at elevated ionic calcium concentrations. These fouling rates are comparable to those performed in large scale fouling experiments in real heat exchangers in pilot plant. For example, our recent study with 0.5 wt% WPI at an ionic calcium of 38.7 ppm report an overall dry deposits of 17.1 g in a 10-passes PHE (temperature ranges from 65 to 85 °C), resulting in mean fouling rates of ~1.6 mg·m^{-2}·s^{-1}).

As the calculation of surface reaction kinetics needs the localized information of unfolded BLG species, only the fouling rates obtained at four specific calcium concentrations (*i.e*. 40, 50, 60 and 70 ppm) were used to calculate the corresponding deposition frequency factor $k\mathrm{D}0$. The dependence of $k\mathrm{D}0$ against various calcium concentrations is plotted in the inset of Figure 2(b). A quasi-linear dependence of $k\mathrm{D}0$ on the calcium content can be found, implying that the deposition rate of unfolded BLG is proportional to the calcium concentrations (reaction order of one). These results might suggest that only one calcium ion is involved in the deposition reaction. It is, to our knowledge, the first study to reveal the role of ionic calcium on whey protein deposition reaction coupling thermal denaturation of proteins.

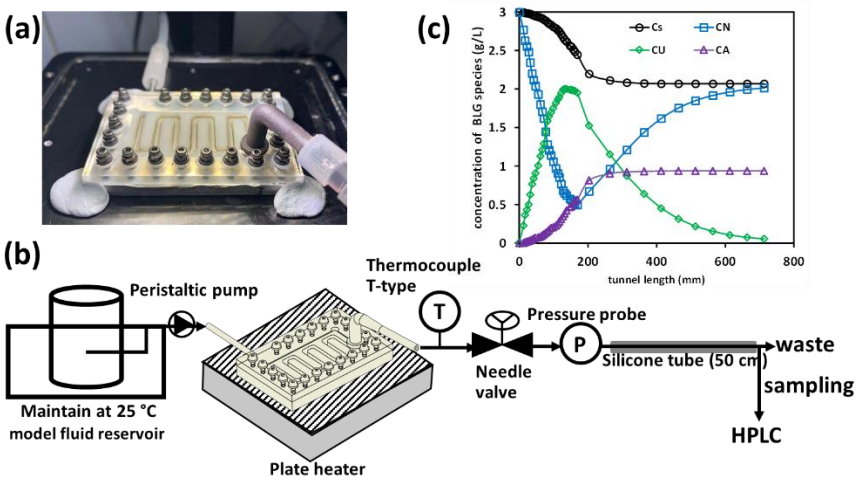

Figure 1. (a) The picture of the fouling rig mounted upon a plate heater. (b) experimental set-up for fouling runs. (c) Simulated concentrations of different BLG species along the tunnel. C_s, C_N, C_U and C_A represent soluble, native, unfolded and aggregated BLG, respectively. C_s refers to BLG concentration as detected in a HPLC system, where soluble BLG content consist of native and unfolded that refolds back to its native state after cooling. The model fluid contains 0.5 wt% whey proteins with addition of 40 ppm Ca^{2+}.

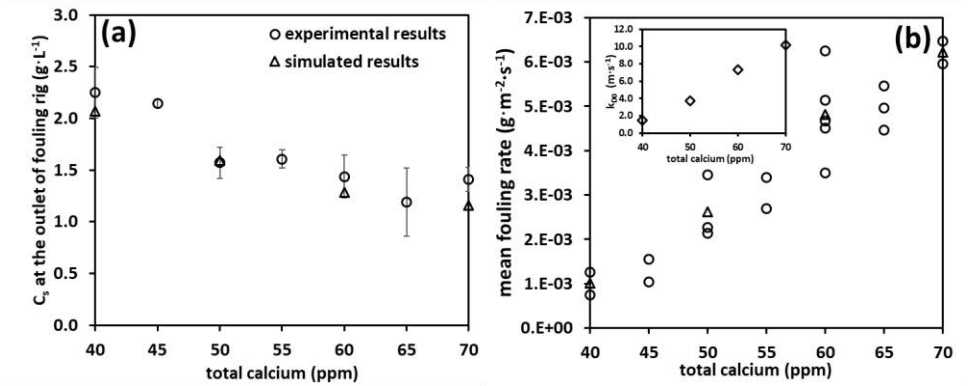

Figure 2. (a) Soluble BLG concentrations obtained at the outlet of fouling system (outlet of silicone tube). Symbols in circle represent experimental data while the ones in triangle denote simulated results. Error bars show standard deviation of at least two replicates. (b) Mean fouling rate at various calcium levels. Note that symbols in triangle show average values at specific calcium level which was used for simulation. Inset figure shows the values of $k\mathrm{D}0$ for deposition simulation.

Whey protein fouling prediction in plate heat exchanger

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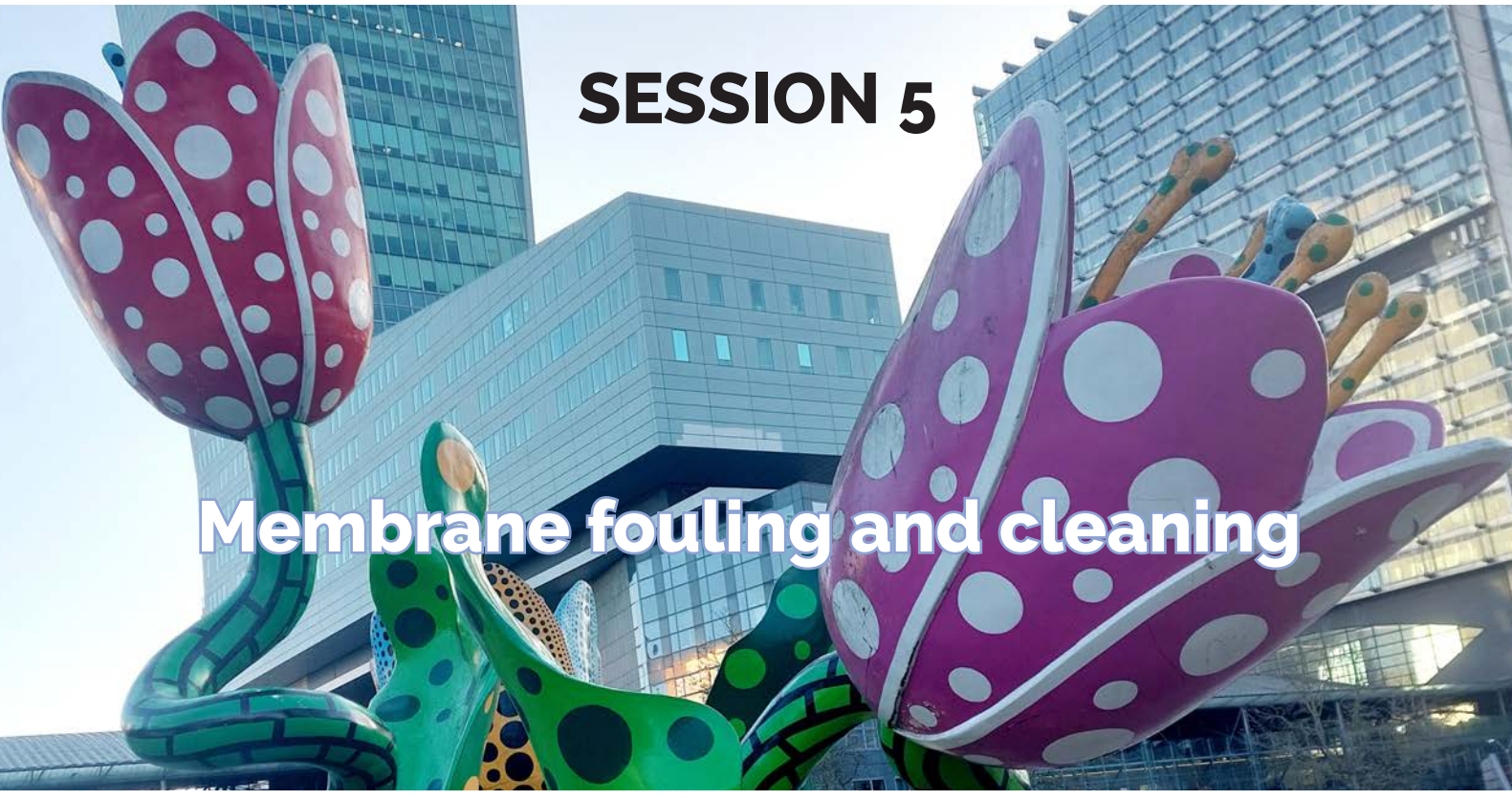
Plate heat exchanger (PHE) is commonly used equipment to thermally treat industrial products for different purposes. The heating process is frequently ceased for cleaning cycles that intended to remove deposit from the heating surface.

Despite decades of research, a detailed and accurate model for simulating, monitoring, and control of fouling is still not available. Computational Fluid Dynamics (CFD) models can predict fouling for a single product and configuration, but they are computationally expensive. Other statistical based models can predict total fouling mass at a fixed operating time depending on the training data set. Whey protein fouling prediction in PHE remains a challenge because most of the models fails to predict outside narrow operating conditions.

The 2D PHE dynamic model presented here contains i) various protein denaturation reaction kinetics; ii) deposit growth spatial distribution model; iii) a dynamic model of mass and heat transfer in a single PHE channel; iv) flexible assembly of channels into a variety of PHE configurations. Unfolded protein was considered as the only precursor of fouling growth. The PHE model was trained to capture deposit mass after 2 hours operation for 1% whey protein solution and 100 ppm calcium concentration in 5 heating channels PHE. The experimental data for model fitting show four different thermal profiles. The model was then used to predict deposit mass of the same protein solution for three different runs in 10 channels PHE. The proposed model shows good predictive capability with minimum computational burden. Our model can predict the local and total deposit mass well for varying whey protein and calcium concentrations.

This study is an attempt of using a combination a first-principles modelling approach with other engineering tools such as symbolic regression to improve fouling prediction. It also illustrates the methodology required for obtaining physicochemical parameters used in the model.

The computation time is lower than typical CFD models, less than a minute. Then effective use of the model to optimize the operation of a complete PHE, including typical heating and fouling policies is possible. The ability to predict deposit distribution on the plates is also useful for monitoring, diagnosis, and control purposes from an industrial point of view.



SESSION 5

Membrane fouling and cleaning

Sustainability in the food industry: how membrane technologies can contribute?

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In the whole food production chain, from the farm to the fork, food processing steps have a large environmental impact. In the few last decades, much research has focused on the development of alternative non-thermal technologies, such as membrane separation processes, to optimize heat recovery or water consumption. Membrane separation processes are playing a vital role in the sustainability of the food processing steps.

In one sense, they can ensure eco-friendly extraction and valorization of bioresources as well as non-thermal stabilization by reducing energy consumption. They can also efficiently reclaim effluents and wastewaters to augment natural water supplies and recovery of compounds of interest. However, a largely unasked question is how well the membrane technologies we use and the way we operate them stand up to scrutiny based on 'sustainable development' criteria. At the technical and operational level, we have the age-old problems of fouling and cleaning efficiency and their impact on 'sustainable' long-term operation. At a more general level, the dilemma is that current economic frameworks may not necessarily lead to systems or modes of operation that satisfy environmental criteria leading to inevitable compromises.

This talk addresses the different levels of sustainability in the context of membrane technology in food processing. It illustrates how filtration technologies could play a decisive role for sustainable food preservation or valorization of raw materials, by-products and water. It presents also previous environmental design attempts of food membrane separation processes on the one hand and emerging, promising approaches on the other. These attempts are illustrated with examples taken from the dairy sector, and explicitly discussed with respect to the potential rewards and challenges of their respective application.

Evidencing strengths and weaknesses of alkaline detergents to formulate efficient mixtures useful for polymer membranes' cleaning

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The cleaning efficiency of a detergent is generally checked by the mean of a set of long-term filtrations with the detergent under evaluation and based on the membrane flux recovery after cleaning. For 20 years, in our team we continuously develop an original methodology able (1) to rapidly evaluate if a formulated detergent is really efficient or not and (ii) to drive the development of efficient detergent formulation. This approach is based on the quantification of residual fouling by ATR-FTIR as a better information than water flux recovery that can be sometimes misleading as already reported in [**1,2**].

One step beyond is to use this approach to formulate new detergents and to rapidly evaluate the efficiency of a wide selection of prototypes in order to select the promising ones and understand the possible origins of failure of the others. Progressively, the methodology has also integrated a way for estimation of short & long-term chemical degradation of the membrane, moreover realistic when compared to those reached at industrial scale [**3**]. This approach was first successfully applied to the development of a non-oxidizing biocide useful for RO membrane that was proved to be harmless toward a (spiral) polyamide membrane [**4**].

In this study we illustrate this overall methodology to develop new alkaline detergents to clean PES/PVP membranes used in skim milk ultrafiltration. The irreversible fouling at cleaning start being made of proteins, in the selected pH range (11.0 – 12.4), the heart of the detergent efficiency is the surfactant system. A single ATR-FTIR analysis, able to (i) evaluate the cleaning efficiency, (ii) identify the (irreversible) adsorption of detergent components on the membrane and (iii) evidence the membrane short/long term degradation is reliable for decision making on formulation whereas microwave activation is relevant for long term ageing evaluation [**3,4**]. We have tested 15 surfactants either single or in mixture in alkaline matrix. Long term filtration (here 50 h in the detergent at target concentration of use) was only conducted on formulated detergents pre-validated during the first steps minimising the development cost associated with long-term filtration with inappropriate prototypes. Surprisingly, some very efficient surfactants with respect to the membrane cleaning were able to induce unexpected membrane degradation and had to be rejected. Nevertheless, the development of an alkaline detergent efficient for PES/PVP membrane cleaning was successful. Finally, the methodology was extended to the evaluation of (i) the risk of the surfactant leakage from the membrane to the processed fluid and (ii) the impact of the storage of a spiral membrane in diluted detergent (12-18 months) on its performances.

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Fouling of polyethersulphone ultrafiltration membranes during the decaffeination of ground coffee brews

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The production of reduced caffeine coffee beverages (so called 'half-decaf') has recently gained increased commercial attention. Ultrafiltration (UF) can offer a viable alternative to the use of solvents in the decaffeination process, also operating at ambient temperatures, and a low operational cost. This paper reports the development and evaluation of the fouling occurring during the selective reduction of caffeine from coffee brews using (i) commercially available synthetic tight ultrafiltration (TUF) polyethersulphone (PES) membranes (GP95PP – Alfa Laval) and (ii) Self-made mixed matrix PES membranes (MMMs) fabricated in-house.

Performance is reported in terms of fluxes, hydraulic resistances, and component rejection ratios. A cross-flow rig was operated at transmembrane pressures of 2-9 bar and cross-flow velocities of 0.04 - 0.1 m/s at temperatures in the range 25-50^{o}C. The process is viable: a retentate is produced with a much reduced caffeine concentration whilst still rich in higher molecular weight bioactives. Commercial PES membranes (GP95PP – Alfa Laval) had a permeate flux of 6.5 L m^{-2} h^{-1} and a fouling index (FI) of 35%. Rejection ratios were ca 30% for caffeine, > 90% for both polyphenols & proteins, & > 80% for melanoidins, over a 29hr filtration period. Membrane surface modification due to fouling takes place, altering the hydrophobicity and surface roughness.

An effective cleaning protocol is reported comprised of 0.5 wt% NaOH at 50^{0}C. The flux decline and recovery along with changes in the key component rejection are reported for multiple fouling and cleaning cycles. The effectiveness of PES MMMs developed in-house are benchmarked against commercial 2kDa PES membranes (GP95PP – Alfa Laval). Differences in filtration performance and cleanability between the two classes of membranes are linked to variations in surface properties.

Keywords: Fouling, Cleaning, Ultrafiltration, Coffee, Caffeine

Acknowledgements: We thank our industrial collaborators: Andrew Downie of *Bettys* & *Taylors Group* for the supply of coffee, and David Carr of *Kerry Ingredients*, Cam Gloucestershire for the supply of dead-end prefiltration cartridges.

Development of *in-situ* and *ex-situ* tests for the characterization of the ageing of organic membranes in wine tangential microfiltration

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Objectives: The objective of this work is to develop a characterization method of fouled/ageed organic microfiltration membranes in winemaking. In this work, we mainly interested in the *in-situ* measurements to assess more accurately the onset of ageing induced by repeated chemical cleaning cycles. In our case, the chemical cleaning cycles applied is quite frequent which can accelerate membrane ageing. According to Yadaf *et al.* in 2009, the use of sodium hypochlorite in a chemical cleaning of PES membrane can cause polymer chain breaks. Because of the latter, the characteristics of the material will change, and hence, reduce the performance of the membrane. The originality of this work is to associate several analysis to distinguish the phenomenon of fouling and ageing.

Materials and methods: Today we have analytical methods to assess the cleanliness of the membrane such as permeability analysis etc. However, there is no tool or *in-situ* analysis method that gives us information on the state of the membrane (clean, fouled, and aged). To complete that we have developed a transmembrane streaming potential box to achieve such aim.

Results and outlook: We found that the analysis of the hydraulic permeability of the two mini-modules is not consistent with the physical state of the membrane (permeability is too high). This increase in hydraulic permeability can be translated by reduction of the membrane resistance. This is due to the pore diameter enlargement which means ageing. The microscopy analysis has confirmed this hypothesis on ageing by the presence of micro cracks in the surface of the membranes. In addition, we also found a homogeneous cake on the membrane which is bonded to the fouling phenomenon. The analysis of the transmembrane streaming potential and the isoelectric point shows an evolution of the charge of the membrane which is due to the remaining cake residue. Finally, we concluded that the used industrial membrane is not only aged but also fouled. In addition, we studied the influence of the location of the fibres in the module between 2000 fibers on the phenomenon of fouling and ageing. It appears that the fibers which is located outside the module has suffered the ageing phenomenon more than the fouling phenomenon. In perspective, it would be interesting to verify the feasibility of the streaming potential measures with an industrial pilot (work in progress).

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Keywords : Organic Membrane – Wine – Microfiltration – Fouling – Cleaning – Ageing

Enhanced Cleaning of Microfiltration Membrane Fouled by Vegetable Oil using Microbubbles

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Oily wastewater (OW) is an oil-in-water emulsion which can be produced throughout the food supply chain (e.g., edible oil refineries, slaughterhouses, dairy processing plants, restaurants, etc.) and cause significant environmental and human health hazards. Membrane technology is a promising approach to OW treatment; however, membrane fouling by oil droplets is a severe problem that results in reduced permeate flux and separation efficiency. Membrane cleaning can largely increase the operating cost by the uses of extra energy, chemicals, and water, and the impact of processing downtime. Microbubbles (MBs) feature long residence time in liquid, larger surface to volume ratio, and hydrophobic gas-liquid interface. These unique properties enable MBs to adsorb water-insoluble organic molecules and thus be used as cleaning and separation agent.

This study aims to introduce MBs into cleaning liquids and evaluate the performance of their cleaning of vegetable oily foulants on filtration membrane. Palm oil-in-water emulsions were used as model OW and filtered by crossflow flat-sheet microfiltration membrane at bench scale for fouling formation. Water and NaOH solution (0.05%) containing MBs with the average size of 2.32 μm and density of 1430 bubbles/mL were generated by a centrifugal pump and used for membrane cleaning at different crossflow velocities (0.65−1.2 m/s). Recovery of permeate flux after cleaning was measured to determine the cleaning performance. While incorporating of MBs into water did not enhance the removal of oily foulant, cleaning with MB-infused NaOH solution for 20 min showed 2−3-fold increase in the flux recovery compared to NaOH solution without MBs. Furthermore, MB-assisted cleaning proved more effective against oily foulant that formed a cake layer on membrane surface than blocked membrane pores.

Reducing concentration polarization in nanofiltration by using 3D printed composite membranes

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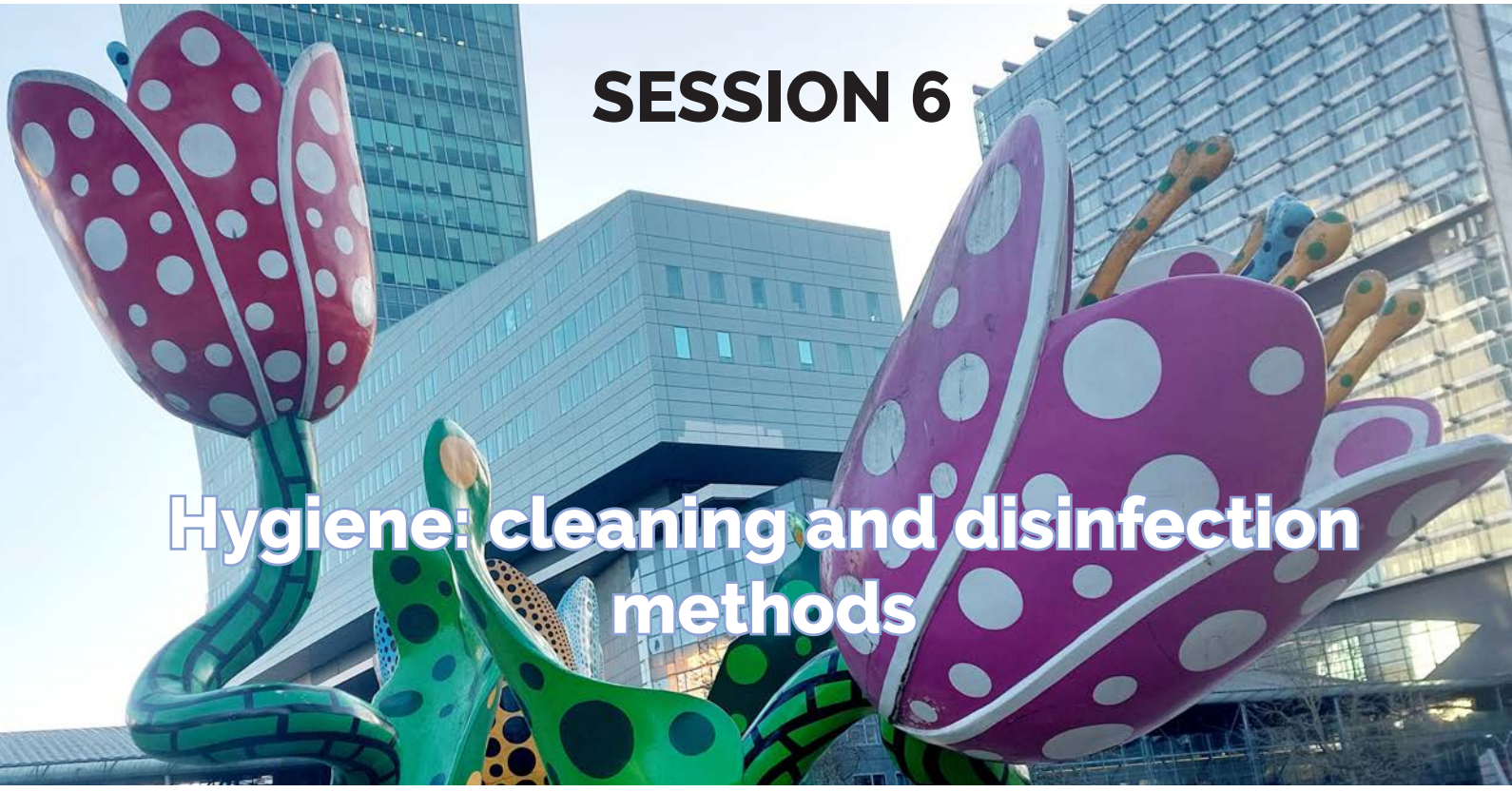
3D printed composite membranes with wavy patterns were used to minimise the impact of concentration polarisation (CP) in nanofiltration (NF). The membranes consist of a polyvinylidene fluoride (PVDF) selective layer coated with polydopamine (PDA) deposited on a 3D printed asymmetric wavy support. The molecular cut-off of the membrane was approximately 550 Da, determined using a crossflow NF setup at a transmembrane pressure of 2 bar, using a range of dyes. The CP behaviour of the composite membranes was assessed by filtration of Congo red (CR) dye solution (0.01 g L^{-1}) showing that the wavy pattern significantly reduced the impact of CP compared to the flat membranes, with a nearly tripling the solute mass transfer coefficient and a 57% decline of the CP factor. Computational fluid dynamics showed that these significant performance improvements were due to improved hydrodynamics near the membrane surface, with the maximum surface shear stress induced by the wavy structure (1.35 Pa) an order of magnitude higher than that of the flat membranes (0.18 Pa) at Re = 700.

These results demonstrate that 3D printing is a viable technology route to reducing CP in membrane nanofiltration applications.



SESSION 6

Hygiene: cleaning and disinfection methods

Air-water Interfacial Flows for Removal of Bacterial Fouling

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Biofouling refers to the deposition of undesired biological substance onto a surface. Synthetic objects in natural environments are often irreversibly inhabited by microorganisms, especially bacterial cells within minutes of immersion, which then colonize connected by a web of extracellular polymeric substances (EPS) to form Biofilms. Here, we demonstrate the effectiveness of air-water flows for removal of bacterial fouling at their early-stage colloidal deposition as well as their later-stage biofilms, through experimental analysis. Time-resolved micro- and macro-imaging is performed to measure the cleaning efficiency of interfacial flows for various strains of pathogenic bacteria. We discuss the impact of flow parameters, especially the propagation speed of the air-water interface, on the efficacy of the proposed method. In confined microgeometries, air-water interfaces reach up to 90% cleaning efficiency. Air-water interfaces exhibit close to 100% efficiency in biofilm removal on open surfaces of various materials, namely soft agar gels, woven fabrics, and solid metallic surfaces. The cleaning efficiency of the approach is, however, dramatically reduced at higher interfacial velocities. Here, we discuss the physical mechanism responsible for this phenomenon by offering side-by-side comparisons with experiments performed on synthetic models. Fundamental efficacy analysis, such as those discussed here, are essential for the development of novel physical anti-biofouling solutions that demand use of minimal bio-chemicals.

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Influence of temperature, material, slope and strain on Listeria monocytogenes biofilm formation

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Despite the efforts, the persistence of *Listeria monocytogenes* in the food industry remains a problem. Biofilms can be the cause for this and their growth and structure are influenced by a whole range of factors. A better understanding of these factors can help in developing elimination/prevention strategies. In this research, the influence of temperature, material, slope, and strain on the biofilm formation of *L. monocytogenes* was investigated.

A standard protocol for the formation of mono-species biofilms was developed. The following parameters were fixed for all experimental setups: incubation time (24h), medium (10% TSB), static and inoculation level (5 log CFU/ml). The potential influential parameters were four different materials (stainless steel 304L type R and type B, glass and polypropylene), incubation temperatures (4°C, 21°C, 30°C), slope in which the materials were hung (angle 0° or horizontal/90° or vertical) and strains used (2 *L. monocytogenes* strains: LFMFP 207 and LFMFP 1049 and one strain *P. aeruginosa:* LMG 28185; which were cultivated in TSB for 24h at 37°C). After incubation (24h) the coupons were hung/laid to dry for 5 minutes and rinsed with demineralised water to remove non-adhered cells. Flocked nylon swabs were used to swab the coupons for enumeration. For the vertical setup, two areas were swabbed, namely the air-liquid interface area and a fully submerged area to look for higher cell counts due to elevated oxygen levels. For the horizontal setup only the submerged area was swabbed. Microscopy was done on separately incubated coupons to examine the biofilm structure. The microscopy coupons were dried for one hour after rinsing and stained with acridine orange for 15 minutes in a dark environment.

Little to no attachment was observed at 4°C for all the strains.Significantly higher cell counts of *P. aeruginosa* were observed in comparison with the *L. monocytogenes* strains at 21 °C and 30°C. No differences in cell count and microscopy were noticed between the used *L. monocytogenes* strains. Only for *P. aeruginosa* increasing cell counts by increasing temperatures (max. 7.49 and 8.64 log CFU/cm^2 for 21 and 30°C) were found. The surface material did not influence the biofilm formation for the studied strains in both angles (horizontal and vertical), nor did the oxygen availability (interface vs. submerged area) in the vertical setup. The slope did not influence the *P. aeruginosa* biofilms in contrast to the *L. monocytogenes* biofilms where a horizontal setup (max. 6.63 and 6.52 log CFU/cm^2 for strain LFMFP 1049 and LFMFP 207 at 30 °C) resulted in higher cell counts compared to the vertical setup (max. 5.07 and 5.40 log CFU/cm^2 for strain LFMFP 1049 and LFMFP 207 at 30°C) and visually more structure (e.g. cellclusters). Microscopy revealed complex biofilm structures with different structures between interface and submerged for *P. aeruginosa* at 21 and 30°C for both angles on all materials.

For *L. monocytogenes* monospecies biofilms, a horizontal setup increased the cellcount. Microscopically, the start of a real biofilm could be seen by the formation of cell clusters. Keeping this hygienic design aspect in mind can help identify critical locations more prone to contamination/biofilm formaton which are indepentend of temperature and material.

Effect of heavy water incorporation on the viability of *Listeria innocua*

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Listeria innocua is a Gram-positive ubiquitous bacterium, widely distributed in a range of environments (vegetation, water, soil) and in food-processing environments. *L. innocua* has the phenotypic characteristic close of *Listeria monocytogenes* specie, an important foodborne pathogen and the etiological agent of human listeriosis, a rare but frequently fatal disease. *L. innocua* is a non-pathogenic bacterium although excessively rare cases of *L. innocua* septicemia and meningitis infections have been reported in human and ruminants. Different environmental stresses can induce the viable but non-culturable (VBNC) state during food processing, such as cleaning and disinfection procedures. Bacteria in the VBNC state have very low metabolic activity and do not divide. Consequently, VBNC cells do not grow on standard microbiological media but retain the ability to recover and become culturable under favourable conditions (ie. resuscitation).

An innovative approach is to use mass spectrometry, Raman spectroscopy coupled with heavy water labelling of metabolism appears to be an innovative technique for the study of bacterial metabolism and spectral changes. We evaluated the impact of heavy water incorporation on the viability state of *L. innocua* cells (Viable Culturable (VC) or VBNC) by Raman spectroscopy. We exposed the *L. innocua* bacterial suspension to different heavy water concentrations (0%, 25%, 50% and 75%) during different times of incubation (0h30, 1h00, 1h30, 2h, 4h, 6h, 24h, 48h, 72h and 96h). For each heavy water concentrations, total, viable (VC and VBNC) and VC populations were quantified by qPCR, PMA-qPCR and plate count agar respectively. In parallel, we analyzed heavy water absorption by Raman spectroscopy. The results of the quantification showed that exposure to deuterium does not affect the viability of *L. innocua* cells. The incorporation of D2O is linked to the concentration and the time exposure.

Impact of environmental and application conditions on the inactivation of dry fungal spores by disinfectants

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Fungi are a major source of contamination of agri-food environments because of their ability to survive and grow on foodstuff, but also because of their sporulation ability, which eases their dissemination in production environments. Biocidal products are key tools for reducing contamination risks, and their efficacy can be evaluated through normative assays (ex. NF EN 17272 (2020) standard). Though the fungicidal effectiveness of biocide products can be assessed with several standards, few studies were carried out to assess the effectiveness of disinfection procedures under near-reality conditions. This work aims to investigate the impact on dry fungal spores of disinfection procedures used in the field.

The dry harvest of fungal spores after cultivation under a moderate water stress increases the intrinsic resistance to biocides and is assumed to better mimic the actual physiological state of spores in food production environments. Dry spores of *A. flavus, C. cladosporioides, M. circinelloides and P. commune* were then adhered to stainless steel surfaces and submitted in pilot-plant conditions to four commercial disinfectants containing chlorine, hydrogen peroxide, triamine or glycolic acid. Different application modes were used, *ie.* fumigation, nebulization and foam gun. Inactivation of spores was assessed according to the temperature, the humidity and the combination of biocide concentration and contact time for instance, in order to identify the most impacting factor and leading to the best results.

According to the disinfection process, results were highly variable, with a mean inactivation ranged from 1,6 to >7 log reduction with the highest inactivation using fumigation. The target fungal species, the contact time, the biocide concentration but also the ambient temperature and foam density can affect the disinfection effectiveness depending on the tested procedure. The results will help foodborne operators adapt and optimize their C&D procedures in order to reduce the occurrence of food spoilage by molds.

The use of bio-sourced antimicrobials for the disinfection of food contact surfaces

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Keywords: disinfection, biofilms, essential oils, food contact surfaces.

Biofilm formation on food contact surfaces results in an increased occurrence of food-borne diseases, outbreaks, reduced shelf-life of food products along with costly socio-economical losses. To overcome these contaminations, several control measures including the use of synthetic antimicrobials have been adopted. Nevertheless, these agents have become much less effective due to the microbial resistance developed towards them, their side toxic and environmental effects, along with negative consumers' perceptions. Therefore, increased efforts are urgently needed to find effective alternatives to control biofilms. In this project, bio-sourced antimicrobials and particularly essential oils (EOs) active components were used as convenient natural alternatives for synthetic antimicrobials. EOs are derived from plant sources, they are environmentally friendly, biodegradable into non-toxic products with potent antimicrobial activities against biofilms. Our findings showed that carvacrol and thymol had a significant effect on *Salmonella enteriditis* biofilms developed on stainless steel coupons. In addition, the antibiofilm effect of both EOs active components was observed under scanning electron microscopy. It was shown that carvacrol and thymol induced morphological alterations to the bacterial cells. They seemed deformed and lysed with holes in their cell walls. Moreover, the mechanism of antimicrobial activity of both carvacrol and thymol was investigated by exerting cell membrane damage with subsequent leakage of potassium ions and green fluorescent proteins.

Microencapsulation of a water soluble ruthenium (II) complex derived from optically pure limonene as an efficient tool against bacterial food pathogens biofilms

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Keywords: Ruthenium (II), limonene, complexation, microencapsulation, biofilm, antibacterial agent

In the present work bioactive aminooxime ligands based on optically pure (R)-Limonene have been synthesized. Their Ruthenium (II) cationic water-soluble complex was prepared by a reaction between dichloro (para-cymene) Ruthenium (II) dimers and aminooxime ligands in a 1:2 molar ratio. Antibacterial and antibiofilm activities of the synthetized complex were assessed against *Escherichia coli*, *Staphylococcus aureus*, *Listeria monocytogenes* and *Enterococcus faecalis.* The results revealed that the Ruthenium (II) complex has higher antibacterial and antibiofilm activities in comparison to free ligands or the enantiopure (R)-Limonene. Moreover, microencapsulation of this complex reduced its cytotoxicity and improved their minimum inhibitory concentration and antibiofilm activity towards the considered bacteria. The Ruthenium (II) complex targets the bacterial cell membrane which leads to rapid leakage of intracellular potassium and proteins. Our study suggests that the developed Ruthenium (II) complexes could be useful as an alternative to conventional disinfectants.

Listeria monocytogenes in the vegetable/potato processing industry: a case study

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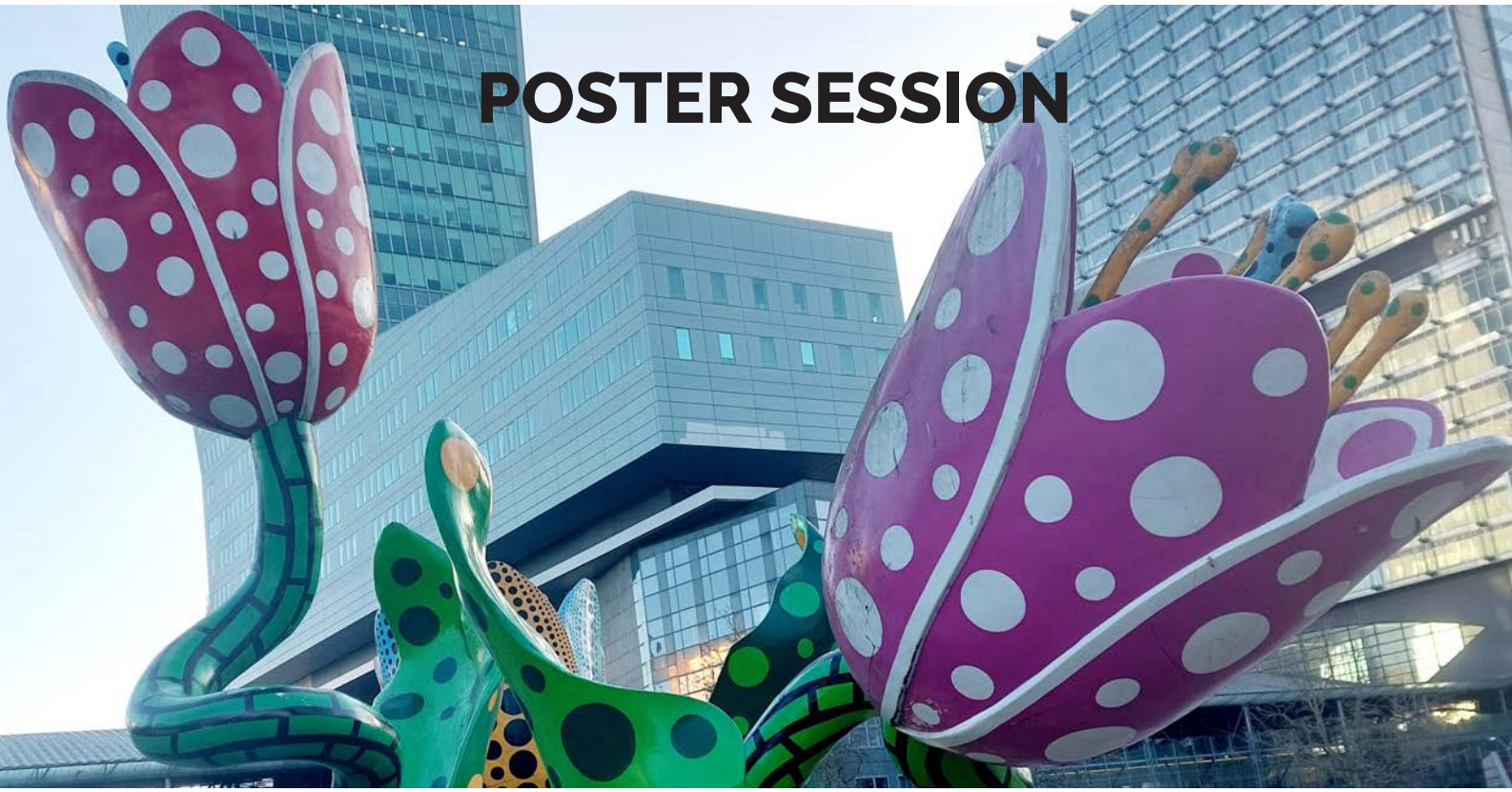
Despite the efforts, the persistence of *Listeria monocytogenes* in the food industry remains a problem. Many studies already sampled production areas but often lack in-depth research in the surrounding environment/microbiology and limited as such the development of specific prevention and intervention strategies. In this study, a sampling of the environment of the freezing tunnel in a food company was performed to study the presence and build-up of *L. monocytogenes.* Several aspects were investigated such as the influence of cleaning and disinfection (C&D), evolution of microbiota between C&D cycles, hygiene status of the cleaning equipment, during technical maintenance and of the personnel. To reveal possible contamination scenarios extra microbial as well as environmental parameters are measured to develop more fitted prevention and intervention strategies.

Environmental sampling was done around the freezing tunnel of a food company that produces products that underwent heat treatments enough to eliminate *L. monocytogene*s before freezing. Sampling took place during production (three weeks production, 10 sampling occasions) or before/after C&D (two sampling occasions). Sampling locations included food contact surfaces (e.g. transport belt), non-food contact surfaces (e.g. floor beneath transport belt), surrounding (e.g. gutter), staff and cleaning utensils. Surface characteristics (angle, material) and visual presence of water/food particles were noted during every sampling occasion. Also, the surrounding temperature and relative humidity were measured. Swabs (Flocked nylon swabs) were taken in duplo, one swab was put in ¼ strength Ringers solution (enumeration of surrounding microbiology and *L. monocytogenes*) and one swab in half Fraser broth (for the detection of *L. monocytogenes*) stored at 4°C and analyzed within 24h. Sulfite-reducing *Clostridia*, *E. coli*, coliforms, *Pseudomonas* spp., lactic acid bacteria, thermophiles, and anaerobic spores were determined on selective plates. For the anaerobic spores, heat treatment of 10 minutes 80°C was done to eliminate the vegetative cells. *L. monocytogenes* positives were isolated and verified with the carbohydrate utilization test, for both the enumeration on selective plates and on half Fraser broth.

In a period from February 2021 until October 2021, 358 samples were analyzed. Overall, in the production area, 43.1% of L. monocytogenes positive samples were found before C&D (n=51), this decreased to 11.8% after C&D (n=51). Positive results were mainly situated near the transport belt, mainly on food contact surfaces. A build-up in positive L. monocytogenes samples was seen, going from 26.5% to 45.1% to 50.0% for respectively weeks 1 (n= 49), 2 (n= 51) and 3 (n= 60) of production. Again the samples were mainly situated near the transport belt, i.e. on the food contact surfaces. This area was for the majority of the samples positive in the three production weeks. The increase in positive L. monocytogenes samples was caused by an increase in positive results for the surrounding (e.g. gutter, floors not situated under the transport belt) and outside of closed equipment (e.g. walls of equipment not situated beneath the transport belt). 100% of the shoes of the staff (n=6) were found positive after C&D, this was 16.7% for the hands (n=6) and 25% for the cleaning utensils (n=6). A build-up during production in positive samples was seen on the hands and shoes of the staff going on average from 50.0% (n=6) to 33.3% (n=6) to 91.7% (n=6) in weeks 1, 2, and 3 of production. Overall, positive samples were often accompanied with coliforms, sulfite-reducing Clostridia, and lactic acid bacteria.

Overall, the area of the transport belt showed the highest contamination rate with L. monocytogenes and could cause contamination of the product stream by e.g. formation of aerosols during intermitted rinsing, re-entrance of contaminated food particles in the food stream. The staff and cleaning utensils showed high potential for the spreading of L. monocytogenes during production and the re-entrance of L. monocytogenes after C&D. Keeping this in mind, specified intervention strategies can be developed to decrease the L. monocytogenes contamination.

POSTER SESSION



Elucidating studies of purging viscoplastic fluid from pipes

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Many products in the fast-moving consumer goods (FMCG) exhibit viscoplastic behaviour, or behaviour which is often characterised as viscoplastic. They are often manufactured in batches on multi-product lines, leading to a need for regular cleaning operations to purge one product from the system before processing another and to avoid cross-contamination. This is usually achieved by recirculation of water by automated cleaning-in-place systems.

Recent studies of water-based purging of a range of FMCG products by the group at Birmingham (Palibiyik *et al*., 2018, *AIChEJ*, **64**, 1517-27, doi 10.1002/aic.16105) present a series of intriguing results where water caused displacement of a core of material from an initially filled pipe and then eroded the annulus of residual material on the wall. They reported that the time to clean the pipe fitted a general dependency on wall shear stress when the wall shear stress was expressed as □* = yield stress/wall shear stress, with □* > 1. This is an unexpected result as the material is not expected to flow in this case.

This poster reports a series of experimental and modelling investigations aimed at elucidating these observations. The rheology of a number of toothpastes, reflecting one of the groups of products studied by Palibiyik *et al*., was studied. The results indicate that simple viscoplastic descriptions are unable to capture the observed behaviour. Wall slip was measured using a simple pipe system. Purging, and particularly the initial breakthrough of water to form a 'core', was performed using a syringe pump, monitoring the motion of the initial plug of toothpaste and the pressure drop across the plug. The change of paste rheology on contact with water was also characterised. The findings provide an alternative framework for explaining the Palibiyik *et al*. results.

Impact of the physiological state of mold spores on their resistance to chlorinated disinfectant

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Introduction

Fungi are a major source of contamination of agri-food environments because of their ability to survive and grow on foods, particularly through the production of spores that also facilitate their dissemination. They can directly or indirectly cause the contamination of food products. To reduce the risks of contamination, manufacturers use a wide variety of chemical disinfectants. Their fungicidal efficacy is evaluated according to the protocols described in many standards, including the standard NF EN 17272 (2020) for the specific evaluation of process-product couples for airborne room disinfections. However, these standards may have some limitations. Indeed, the chosen species, the physiological conditions of fungi during sporulation and the presence of a soiling substance can have an impact on the survival rate of conidia in response to a treatment with a disinfectant. The impact of these parameters was then investigated in this study.

Material and methods

Two strains of *Penicillium commune* isolated from spoiled cheeses were used. Spores were produced on reduced aw (aw 0.95) or non-reduced aw (aw 0.99) media. Then, spores were collected without being rehydrated or being rehydrated in different interfering substances (whole or skim milk, BSA 0.3 g/l or 1/20th skim milk) and subjected to a 0.1% chlorine treatment for 12.5 min after adhesion on stainless steel surfaces.

Results and conclusion

Where spores were in contact with skim or whole milk, the activity of the chlorine solution is nil or greatly reduced. In contrary, when spores were suspended in 0.3 g/L BSA or 20-fold diluted skim milk (standard conditions), no surviving spores were counted in almost all cases with a log reduction greater than or equal to 3-3.5 log. Dry spores produced on a medium with reduced aw displayed intermediate phenotypes.

This study then showed that the use of spores rehydrated in 1/20th skim milk or in BSA at 0.3 g/l could cause an overestimation of the efficiency of chlorine towards dry spores produced under suboptimal growth conditions and could call into question the normative conditions used to validate the fungicidal effect of biocides. Besides, these results demonstrate once again the importance of cleaning prior to disinfection.

Antimicrobial activity of free and encapsulated carvacrol against *Pseudomonas aeruginosa* biofilms

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Pathogenic bacterial contaminations of abiotic surfaces in the food and health sectors represents a serious public health concern. The high resistance of biofilms to antimicrobial agents makes the removal of these microbial communities a difficult task. Essential oils, such as carvacrol, are bio-based antimicrobials that have broad anti-biofilm activity provided by the removal and prevention of biofilm formation. However, the application of EOs faces many challenges such as their low stability, high volatility, and low water solubility that can decrease their activity. Microencapsulation of EOs is a very effective tool to overcome these limitations, control their release and improve their penetration into biofilm deep layers.

In this work, spray drying was used to develop microcapsules containing carvacrol, in order to improve its antimicrobial activity against *Pseudomanas aeruginosa* biofilms adhered to stainless steel surfaces (SS). Microscopic morphology of the developed capsules and treated biofilm bacterial cells were observed using scanning electron microscopy. Results showed that treatment with free carvacrol (F-C) and encapsulated carvacrol (E-C) induce several morphological changes, damages and deformations to the treated bacterial cell structures (Figure 1).

The minimum inhibitory concentrations (MICs) of F-C and E-C was assessed against planktonic cells of *P. aeruginosa*. The MIC of E-C (5 mg ml^{-1}) was 4- times lower than that of F-C (1.25 mg ml^{-1}).

Enumeration of *P. aeruginosa* biofilms exposed to carvacrol (F-C and E-C) at MIC levels showed significant reduction of biofilm biomass after different exposure times (1, 5 and 15 min). Whatever the exposure time, E-C was significantly more effective in reducing *P. aeruginosa* biofilm biomass than F-C and was able to reduce biofilm biomass below the detection limit after 15 min of treatment.

In addition, our findings showed that when bacterial cells were exposed to carvacrol, cell envelope damage was substantially strong. This damage resulted in K+ and protein leakage, as demonstrated on the *P. aeruginosa* GFP strain. Results demonstrated that both F-C and E-C induced altered cytoplasmic cell membrane permeability and cell death, and that E-C (1.25 mg l^{-1}) was more effective than F-C (5 mg l^{-1}) even at a 4-fold lower concentration.

As a conclusion, carvacrol showed a strong and significant antimicrobial effect against *P. aeruginosa* biofilms. In addition, spray-drying microencapsulation could be used as an effective tool to enhance the antibiofilm activity of carvacrol, while reducing the used concentrations.

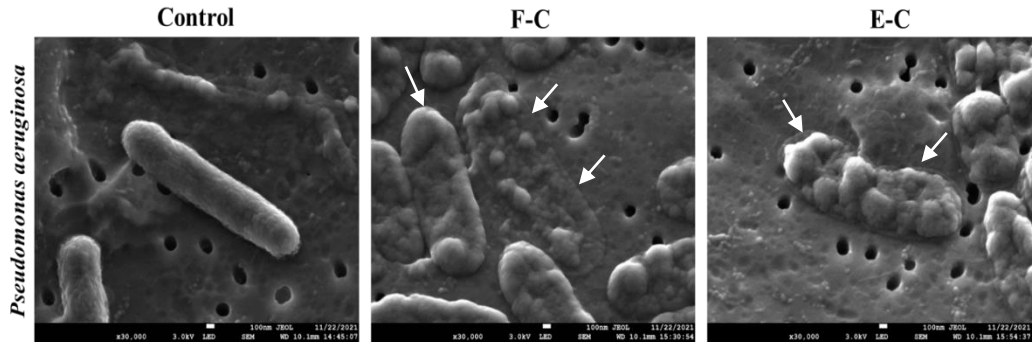

Figure 1: Scanning electron micrographs of *Pseudomonas aeruginosa* biofilm cells, after treatment with free and microencapsulated carvacrol at MIC. The control represents cells treated with potassium phosphate buffer.

Methodology to select commercial enzyme incorporated in formulated detergents used in the cleaning of ultrafiltration PES/PVP membrane fouled by skim milk

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PES/PVP polymer membranes are widely used at industrial scale for skim milk ultrafiltration (UF) aiming at protein content standardization before the cheese making process. Membranes are systematically fouled by milk proteins that are the main cleaning target. Either alkaline or enzymatic detergents are suitable for protein removing from membranes. Detergents' suppliers develop formulations from ingredients they have to select. In the case of enzymatic detergents, they also need to select a source of enzyme. Subtilisin is a protease family, among which Subtilisin A is widely used in detergents. However, they are commercialized with stabilizing components which are more or less identified and could have an impact on formulated detergents performances.

In this study we propose a methodology to facilitate the selection of the enzyme in mixture with its stabilizing components ("stabilized enzyme") based on two criteria: (i) Enzymatic activity based on common test using azocasein; (ii) Cleaning efficiency validation in UF condition which include skim milk fouling, cleaning in place (CIP) and ATR-FTIR characterization of membranes to quantify residual proteins. To illustrate the methodology, we used a formulated mixture (proprietary composition of Hypred - KERSIA group) in which three different sources of stabilized Subtilisin were used. For confidentiality purpose they will be referred to as source X, B and C. The last one has been used at two concentrations: C-1 and C-2 with concentration C-2 > C-1. For this first selection, no inactivation of the enzyme was achieved after the CIP step. Membranes were characterized with ATR-FTIR after sufficient drying in a desiccator. The results (**Table 1**) show that (1) the water flux recovery was systematically higher than the initial flux. This is probably due to the hydrophilization of the membrane surface by adsorption of hydrophilic detergent compounds; (2) Residual enzymes on pristine membrane remained after CIP; (3) With respect to residual proteins, cleaning performances were similar. Considering all criteria including the residual enzymes and proteins on skim milk fouled membranes, the enzyme source X seems best adapted along with C. The source X corresponds to commercial DEPTA UF 305 L®.

Table 1: UF PES/PVP membranes (HFK-131, Koch, MWCO 5-10 kg.mol^{-1}) cleaned with different subtilisin sources incorporated in the same formulated detergent used 60 min at 5 g.L^{-1}, 50°C, pH 10.5

	Cleaning of Pristine Membrane			**Cleaning of Skim Milk Fouled Membrane**		
	Number of Membranes	**Water flux Recovery**	**Residual Enzymes (µg. cm^{-2})**	**Number of Membranes**	**Water Flux Recovery**	**Residual Enzymes + Proteins (µg. cm^{-2})**
Fouled Membrane after water rinsing	-	-	-	15	0.2 ± 0.1	40 ± 19
Source of Enzyme						
X	1	3.3	7 ± 4	2	2.5 ± 0.2	7 ± 5
B	1	2.3	5 ± 4	2	3.0 ± 0.8	11 ± 7
C-1	1	1.6	3 ± 2	2	1.8 ± 0.5	9 ± 4
C-2	2	4.0 ± 1.1	6 ± 4	2	2.3 ± 0.5	9 ± 4

The drying dynamics and resistance to cleaning of droplets containing *Bacillus* spores on various materials

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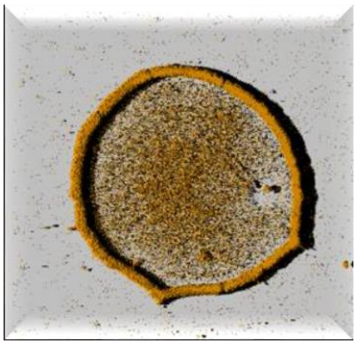
Introduction. In the food industry, a major source of surface contamination is the presence of bioaerosols (aerosols containing particles of biological origin) which settle on surfaces and equipment and subsequently contribute to the contamination of food during processing. The formation of these deposits is of particular concern as it has been shown that some bacteria survive in aerosols and after drying on surfaces, and that the structures thereby formed are sometimes very resistant to the hygienic procedures implemented to control surface hygiene. The aim of this study was to identify the role of the physicochemical properties of materials and particles on the formation of deposits and their subsequent resistance to a cleaning procedure.



Materials and methods. 1-µl droplets containing *Bacillus* spores (hydrophilic/hydrophobic, presence/absence of an external membrane called exosporium) were placed on various materials and left to dry. The observation of the dried deposits (distribution on the surface, 3-D organization) and of the droplets during evaporation (kinetics of the droplet shape, spore movement within the droplet) was carried out with an inverted confocal laser scanning microscope (Zeiss, LSM780). The resistance of the dried deposits to cleaning was evaluated as follows. Contaminated coupons were placed in rectangular test tubes, connected to a cleaning-in-place (CIP) pilot rig and subjected to a cleaning procedure involving NaOH 0.5% et 60°C. The number of the residual adherent spores was evaluated by enumeration on agar.

Results. We first observed that the deposit architecture after evaporation of the droplets was strongly affected by the material (deposit size) but also by the particle properties (presence and structure of a peripheral ring, spore cluster). Multiple two-dimensional images (Z-stacks) were then taken of droplets at different times during evaporation to determine the kinetics of spore distribution within these droplets. This representation made it possible to observe that certain phenomena were affected by the properties of the spores and the materials. Among these phenomena, we can note the sedimentation of spore clusters, the accumulation of certain spores at the air-liquid interface, or the rapid formation of a peripheral ring (net vs. diffuse). Image sequences recorded at the material level confirmed the previous observations and made it possible to follow the particle movements. First, the expected outward motion was clearly observed, mainly with hydrophilic spores. Conversely, many hydrophobic spores (and spore clusters) remain stationary, probably as a result of strong interactions with the material.

We then established that the least resistant spores were still the hydrophilic ones, while the detachment was 3–100 times more effective on glass than on other materials. Moreover, the peripheral ring is partially or completely detached, which would indicate a more pronounced fragility with respect to a cleaning procedure.

Significance. This work confirms that bioaerosols represent a major risk in terms of surface hygiene in the food industry

How to release a sponge: relaxation of casein micelles fouling layers during ultrafiltration, a SAXS study

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Abstract

This work is devoted to the role of local fouling layer properties in membrane filtration and to importance of relaxation (transmembrane pressure release) for membrane rinsing. The fouling layer is described as a soft matter with local properties (compressibility, permeability, yield stress) depending on its local concentration. It is suggested that concentration of local fouling layer at different steps of membrane filtration cycle can be described using filtration-consolidation approach (during the layer formation at constant applied pressure and relaxation at zero transmembrane pressure) and simple force balance (during the layer removal via membrane rinsing). The fouling layer behavior was demonstrated using a model compressible colloid – casein micelle. Local fouling layer concentration on the membrane surface was accessed via *in situ* small-angle X-ray scattering (SAXS) measurement during ultrafiltration of casein micelle suspension. It was demonstrated that relaxation step results in swelling of fouling layer (local concentration decrease near membrane surface), that was important for efficient removal of fouling layer at the following rinsing step.

Keywords. Membrane fouling – membrane rinsing – relaxation – polarization layer structure – rheological properties – SAXS.

INTRODUCTION

We assume that reader of this abstract is familiar with the problem of membrane fouling during micro- and ultrafiltration of colloids; so, there is no need to introduce the idea: “fouling reduces filtration efficiency; let’s reduce the fouling”.

Our current work is focused on the external membrane fouling, i.e. formation of colloidal deposit (gel, filter cake) on membrane surface, during filtration of colloids. Solid deposit is formed at the membrane surface, when a critical concentration of sol-gel transition is exceeded in a concentration polarization layer during filtration. We are interested in properties and behavior of this deposit during following membrane rinsing. Such a deposit can be removed via membrane rinsing at sufficiently intense hydrodynamic conditions, which are required in order to erode the deposit or “unstuck” it from membrane surface. The hydrodynamic force required for efficient membrane rinsing (it can be expressed as a critical wall shear stress in filtration cell) must increase with the deposit concentration (consolidation), because increasing of a gel concentration must increase its resistance to erosion (cohesion) and its adhesion to membrane surface.

Hence, it can be hypothesized that decreasing of the deposit concentration (for example, during its relaxation/swelling at zero transmembrane pressure) must soften the deposit, reduce its cohesion and its adhesion to membrane surface, reduce the critical wall shear stress required to erode it and, thus,

facilitate the membrane rinsing. Our objective was to analyze (both theoretically and experimentally) the variation of local concentration of colloidal deposit on filtration membrane surface during the relaxation step of filtration and study the influence of deposit relaxation of following membrane rinsing.

METHODS

Suspension of casein micelles (6 wt. %, 25°C) in milk ultrafiltrate (their natural environment) was used as a model colloidal system. Casein micelles powder was purchased from Ingredia (Promilk 802B, Ingredia, France). Casein micelles are well characterized object: highly compressible spherical particles, stable against aggregation at neutral pH, having permeability, compressibility and rheological properties strongly depending on concentration, and experiencing reversible sol-gel transition at osmotic pressure of 10-30 kPa.

Filtration experiments were carried out in specially developed SAXS-filtration cell, which allows *in situ* observation of concentration polarization and deposit layers on the membrane surface during cross-flow or dead-end filtration. Fully retentive polyethersulfone ultrafiltration membrane with molecular weight cut off 100 kDa (Pleiade, Orelis Environnement, France) was used in experiments.

The deposit was observed during every stage of filtration with the help of SAXS (beamline SWING, synchrotron SOLEIL, France). The full width at half maximum of the used X-ray beam was equal to 25 µm, which allowed observation of local concentration distribution even in close vicinity to the membrane surface ("skin layer").

Filtration experiments were performed in three stages: deposit formation at constant transmembrane pressure (1.2 bar) in dead-end filtration (2.5 h); deposit relaxation at zero transmembrane pressure in the absence of cross-flow (45 min); deposit erosion (membrane rinsing) at zero transmembrane pressure under gentle cross-flow (15 min).

Formation and relaxation of casein micelles deposit on the membrane surface was described in frame of conventional filtration-consolidation theory.

RESULTS AND CONCLUSIONS

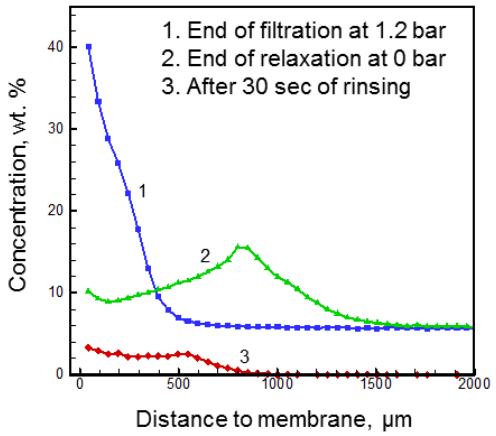

Figure 1. Casein micelle concentration versus distance to membrane surface at different stages of filtration.

It was observed that filtration at 1.2 bar (which is above the critical osmotic pressure of sol-gel transition for casein micelles) resulted in formation of thick (500 µm) and dense deposit of casein micelles (concentration at membrane surface about 40 wt. %). At such a high concentration casein micelles behave as elastic gel with high yield stress. Relaxation resulted in significant swelling of the deposit with strong reduction of concentration on the membrane surface (it must be noted that this swelling was expected according to consolidation theory). Decrease of the deposit concentration at the membrane surface down to 10% (which is below the gel point of casein micelles) resulted in decrease of its adhesion to membrane surface. Therefore, the swelled deposit was easily removed from the membrane surface on the very beginning of rinsing with the help of dispersion medium (milk ultrafiltrate recirculation).

ACKNOWLEDGMENT

SAXS measurements were performed using the Beamline SWING of Synchrotron SOLEIL in the frame of proposal 20170238. SOLEIL is acknowledged for invaluable technical assistance and financial support.

Use of patch-clamp for the investigation of the interaction force between particles and substrate

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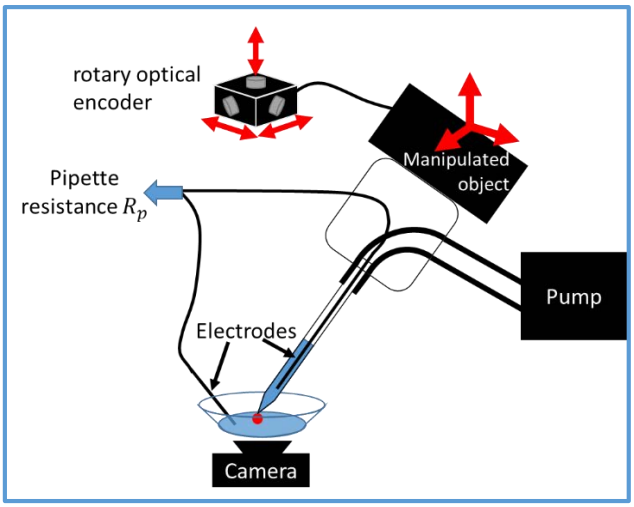
Introduction. The force of interaction between bacteria and materials is an essential factor in the understanding of phenomena at interfaces and has been studied experimentally for years. In the current work, we present an original method deriving from the patch-clamp technique to measure this interaction force.

Materials and methods. Briefly, a micropipette is brought into contact with an adherent particle under the optical microscope. A given aspiration pressure is then applied and the pipette is pulled away from the particle at a given rate. If the particle does not detach, a higher aspiration pressure is applied and the experiment is repeated until the particle detach. The minimum pressure, allowing the detachment is used to calculate the interaction force between the particle and the substrate. This technique was applied to hydrophilic and hydrophobic microspheres with diameter of about 2 µm placed on an hydrophilic substrates Same technique was also used to measure the interaction force of *Bacillus* spores placed on the hydrophilic substrate: B. cereus 98/4 spores (hydrophobic, surrounded by a soft membrane called "exosporium"), *B. subtilis* PY79 (hydrophilic, surrounded by a mucous layer called "crust") and *B. subtilis* PY79 *ΔspsA* (a recombinant strain deleted in *spsA*, modified at the "crust" level, which makes the spores hydrophobic).



Results. A huge variability was first observed on the force required to detach the two microspheres from the substrate. Therefore, our results demonstrated that the hydrophilic microspheres were less adherent (average interaction force = 0.07 nN) on the hydrophilic substrate than the hydrophobic microspheres (4.62 nN), which suggests a major role of the hydrophilic/hydrophobic character of the particle on the interaction strength. A series of measurements was then performed with *Bacillus* spores. The hydrophilic *B. subtilis* PY79 spores placed on hydrophilic substrate were much less adherent (0.21 nN) than the hydrophobic *B. cereus* 98/4 (48.79 nN) or *B. subtilis* PY79 *ΔspsA* (43.86 nN). Again, it seems that the preponderant parameter is the hydrophilic/hydrophobic character of the particle while the nature and structure of the spore surface did not seem to play a significant role on the interaction strength between spores and material.

Significance. The presented technique is a part of single-cell manipulation methods. It will allow to investigate the role of different parameters that can affect the interaction forces (material and particle properties, drying time...), but also to determine the dispersion of these forces within a population.

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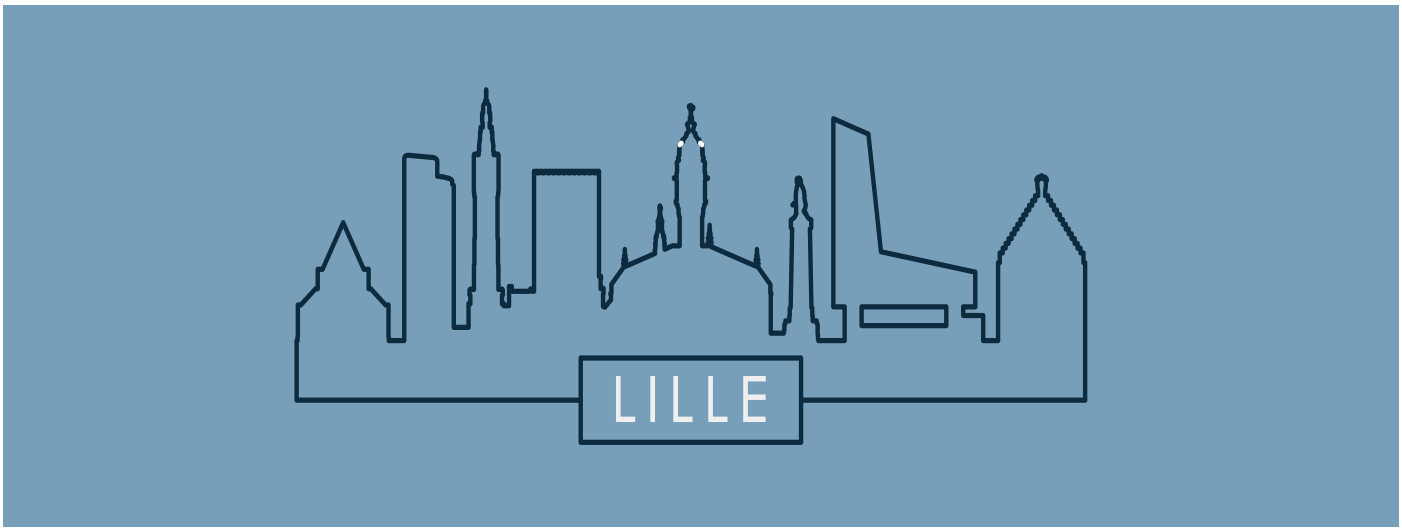

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