

# Filtration of milk and whey in detail

## Appropriate research plant design helps solving scientific tasks concerning the filtration of milk and whey Sima-tec supports German Technical University of Munich

During long term cooperation SIMA-tec has supported research activities executed at TUM for developing processes to recover pure fractions from milk and whey.

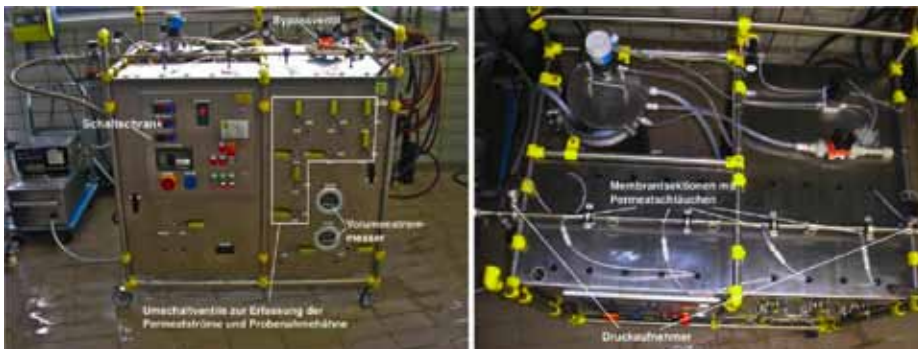


Figure 1: Lab scale system for protein fractionation by means of microfiltration. The right picture shows the equipment for segment-wise permeate collection (photographs: Chair for Food and Bioprocess Engineering at the Technical University of Munich (Weihenstephan site), s. also 4, legends according to 4)

Milk is a mixture of water, fat, diverse proteins, lactose, and minerals, and consequently it is a high-value food. In Germany about 31.4 tons of raw milk were produced in 2016 1, the total amount throughout Europe was about 153 million tons<sup>2</sup>. Raw milk is processed to obtain drinking milk, cheese, butter, a large variety of fresh products as well as powdered milk and whey. In addition, ultrapure isolated fractions or single milk ingredients are recovered as food supplements (e. g. whey proteins), baby food or components of pharmaceutical products (e. g. pure lactose). Besides the use as food, food supplement or pharmaceutical ingredient, there is also the option to ferment liquid waste streams to

produce biogas, lactic acid as basis for biopolymers and to recover purified wastewater as process water. A large number of process steps on the way from skimmed milk to finished products are designed as membrane filtration steps. Although many membrane processes are well established in the dairy industry, new applications in milk processing are still being identified. Furthermore, there is a strong demand to make established processes more energy efficient and sustainable.

All membrane processes that are implemented to concentrate or fractionate casein or whey proteins are limited with respect to their performance and economics due to the tendency of proteins to build up fouling layers on the membrane. Many research projects on the filtration of milk and whey, which were carried out in the last ten years, especially focussed on these fouling

layers depending on the particular ingredients to be retained. At the Chair for Food and Bioprocess Engineering at the Technical University of Munich (TUM), Prof. Ulrich Kulozik and his team have analysed the mechanisms of building fouling layers since many years, and they develop strategies to make certain separation processes more efficient or even to make them possible. To this they use mature and partly highly specialized test facilities, which were developed in cooperation with SIMA-tec GmbH and built by this company that specializes in system engineering for research purposes.

### Small, but not simple

Research facilities for developing and testing membrane technologies for milk and whey processing either in laboratory or in pilot scale must offer technical opportunities that are adapted to the complexity of the respective scientific task. Simple filter tests for the determination of retention and flux, as they are usually carried out in stirred cells, are probably sufficient for primary material screenings. To enter the next level of development, some membrane manufacturers offer standardized test cells to be used in the cross-flow mode. However, there are scientific tasks that may only be solved by using lab scale and pilot plants, which allow adjusting hydraulic conditions and operational parameters in the same range that exists in the industrial plant. Furthermore, the pilot plants should offer a large variety of parameter settings as well as the opportunity to install different membrane products. Setting and measurement of

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figure 2: Pilot test plant PSta06 in the lab in Weiherstephan (photograph: Chair for Food and Bioprocess Engineering at the Technical University of Munich (Weiherstephan site))

relevant parameters should be very precise, even if some pipes have very small diameters, which mean that small deviations in measured values of volume flow or velocity mean high percentage deviations from average values. Measuring devices installed in these pipes with nominal diameters down to 1 mm need to be equipped with high value, highly dynamic sensors.

To achieve the best fit of the requirements of a particular research project and its available budget all opportunities and limits of the system should be precisely defined during preliminary communication between the system builder and the operator. However, besides any specialization, it often makes sense to design systems to be multi-functional. Thus, they can be used in different research projects. They may possibly be adapted or retrofitted with acceptable effort to achieve a good cost-benefit ratio. Semi- or fully-automatic operation via PLC as well as the opportunity to record, visualize, and store all process data and forward them to a connected PC respectively, are demanded more and more.

### Hygienic requirements

Milk and milk fractions are highly perishable food. Thus, one must take care that after operational interruptions or if products are to be changed no product residues are present in the system, when it is restarted. This is valid for industrial systems as well as for lab scale or pilot plants. However, small systems with small pipe diameters and small cross-sectional areas of valves and sensors pose high challenges on cleaning all components that get into contact with the

medium. Product residues like coagulating proteins are likely to block narrow regions. Appropriate cleaning procedures must be implemented in research systems to prevent from measuring errors due to product residues.

### Project examples from the TUM Fractionation of proteins by using ceramic membranes

Piry et al. 3, 4 studied the influence of the distance from the inlet of a membrane module on the characteristics of the fouling layer built up by proteins during microfiltration of skim milk. During the planning of the experimental setup, they faced the challenge to make the dependence of passage distance of characteristic parameters clearly measurable. First a lab scale system was developed that can be equipped with a ceramic single channel

membrane of 1.2 m length and about 6 mm diameter. Such a membrane obeys industrial standards and may be operated under operational conditions that are typical for industrial applications. The membrane was divided into single segments of different numbers and lengths. The permeate areas of these segments were separated from each other by intermediate pieces, in order to collect the individual permeates of each segment and to determine the protein retention of the respective membrane section separately. The filtration resistances of the particular length sections were calculated from the measured permeate fluxes and trans-membrane pressure differences. The particular flow resistances of the membrane material and of the fouling layer were obtained by comparing the total resistances measured for the filtration of pure water and of milk. In doing so, regions with transport resistance dominated by the membrane material could be clearly differentiated from regions with permeate transport controlled by the fouling layer.

Sima-tec supported the research by constructing and building the lab scale system as well as by retrofitting an existing pilot plant for cross-flow filtration. The lab scale system is shown in figure 1. Several commercial membrane and module types (tubular modules with polymer or ceramic membranes or spiral wound modules with polymer membranes) may be implemented in the system. It is highly flexible and easy to modify. Volume flows from 200 to 2,000 l/h and operational pressures of up to 5bars may be set. The system may be operated in batch mode with recirculating retentate and permeate as well as in continuous feed & bleed mode.



figure 3: Laboratory test unit Cube 80-VA (left) in the lab, with measurement box (middle) for acquisition and visualization of data and thermostat (right) (photograph: Chair for Food and Bioprocess Engineering at the Technical University of Munich (Weiherstephan site))

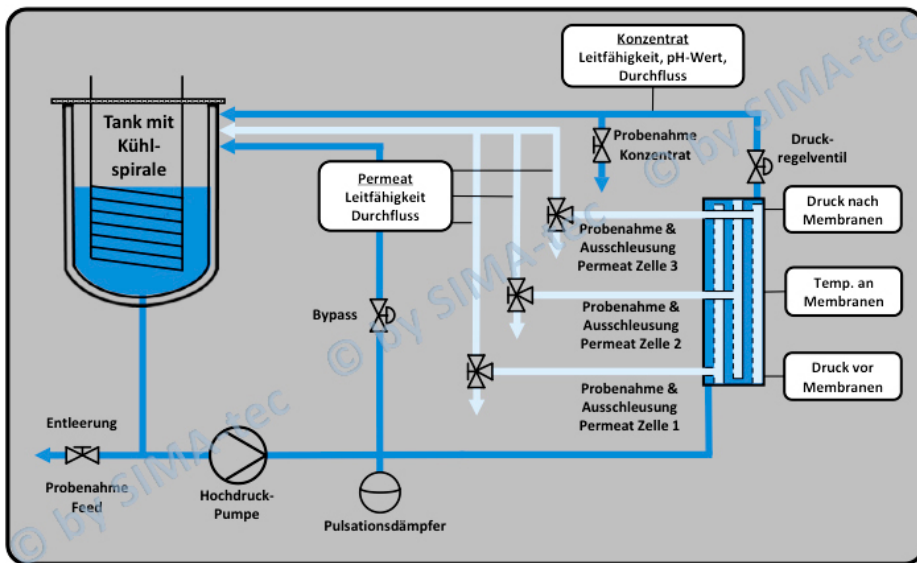


figure 4: Process scheme of the laboratory test unit CUBE containing three membranes (photograph: SIMA-tec GmbH)

## Ultrafiltration of sweet whey

In a subsequent project the lab scale system shown in figure 1 was used to investigate the influence of temperature and pre-treatment measures on the flux and fouling intensity occurring with the ultrafiltration of sweet whey 5. As pre-treatment processes, classic pasteurization and microfiltration by using a ceramic membrane with a mean pore diameter of about 0.5  $\mu\text{m}$  were compared. The ultrafiltration and the preceding microfiltration were carried out at temperatures of 20 and 50  $^{\circ}\text{C}$  respectively. If the influences of single parameters and conditions on the separation performance are to be identified, the exact, reproducible, and constant setting of all other parameters must be made sure. As an example, this is valid for the pressure difference along the flow path of the membrane and thus for the flow velocity along the membrane surface as well as for the medium temperature.

## The role of milk and whey proteins in building up fouling layers

Further research activities focussed on elucidating the molecular processes that lead to the formation of fouling layers during the fractionation and concentration of whey proteins. Filtration experiments were carried out with casein-free whey (so-called "ideal" whey), with model suspensions containing isolated, native whey protein  $\beta$ -lactoglobulin

( $\beta$ -Lg) or with native  $\beta$ -Lg together with a defined amount of heat-treated, aggregated  $\beta$ -Lg 6. The experiments were performed either in lab scale systems for dead-end filtration or in a pilot plant for cross-flow filtration. Different work packages addressed the influence of the protein condition, the filtration process (micro- or ultrafiltration), the process conditions (trans-membrane pressure difference and temperature) as well as the milieu conditions (e.g. pH, ionic strength) on the mass transport through different membranes.

It was observed that with micro- and ultrafiltration basically different mechanisms are effective in building up cover layers by whey proteins. During microfiltration, the proteins primarily adsorb to the membrane surface and are cross-linked in a subsequent reaction. With ultrafiltration, colloidal interactions between particles were identified as main reason. In addition, reactive protein aggregates may contribute to fouling during ultrafiltration as well.

Another part of research activities aimed at clarifying the structural characteristics of casein layers that build up during micro- and ultrafiltration of skimmed milk. These filtration processes are carried out to selectively retain the casein and to recover the whey proteins with the permeate to the largest possible extent. During the experiments it was shown that the compressibility of casein layers, which can be interpreted as a measure for the pressure dependency of the filtrations resistance, decreases in the presence of whey proteins. This is due to the fact, that casein and whey

proteins build up a porous gel, which exhibits a lower flow resistance than the pure, compressed casein layer. All in all, the knowledge gained through these experiments helps to predict the filtration behaviour and to develop strategies avoiding severe fouling that are adapted to the particular processes.

The pilot plant used in this project is PSta06, a universal test system for micro- and ultrafiltration of aqueous media in the pressure range of up to 6 bars (see figure 2). Since the system contains a 150 l-collection container, which may be refilled during operation, and a pressure vessel, in which a 6" spiral-wound module may be installed, operation under industrial conditions is possible. For implying other module types the pressure vessel can be exchanged. The standard measuring devices comprise a feed level sensor, pressure sensors at the feed entry and permeate discharge of the module, as well as a temperature sensor in front of the pressure pump. The rotational speed of the pump is controlled via a frequency converter. A heat exchanger serves for temperature conditioning of the medium, and it may be operated with ice water as well as with hot steam, which allows for testing within a large temperature range. The pilot plant is equipped with a programmable logic controller (PLC), which is connected to an external computer for collection and analysis of generated data. This equipment enables a safe and reproducible implementation and documentation of different measuring tasks. The pilot plant PSta06 is excellently suited for testing cleaning strategies with industrial wound modules. However, if one intends to inspect visually how fouling layers are built along the flow path over a membrane surface or if a cleaning has been successful over the entire membrane area, one has to damage the module to get access to the membrane surface.

Test cells, in which membranes and spacers are put one above the other in the same manner as they are arranged in a spiral-wound module, are much cheaper. The height of the flow channel is determined by the spacer height and is similar to the height of the flow channel between two membrane layers in a wound module. SIMA-tec offers its portable laboratory test unit Cube for performing trials with flat membranes for micro-, ultra-, nanofiltration, and reverse osmosis. The test unit is available in different designs for simple basic measurements as well as for specialized measuring campaigns. At the Chair for Food and Bioprocess



Engineering at TU Munich they use the type Cube 80-VA, which is completely made from stainless steel (see figures 3 and 4). It is possible to install several flat membrane sheets in the cell, thus the entire flow path that the feed takes from the inlet to the concentrate discharge in a wound module can be emulated. The comparison of pressure dependent permeation rates of casein and whey protein, as shown in figure 5, reveals that the test cell is able to reproduce the filtration behaviour of a wound module very well. The membrane sheets are easily accessible for subsequent visual inspections or even further optical analysis of fouling layers.

## Summary

During long term cooperation SIMA-tec has supported research activities executed at TUM for developing processes to recover pure fractions from milk and whey. Different laboratory and pilot test units, which were designed and built in close consultation with the customer, and which fulfil the particular demands of the scientists, helped and still help gaining results that are accepted worldwide as advancement in dairy technology. Despite any specification the systems exhibit high flexibility and the

possibility to be modified, so that they can be adapted to new scientific tasks. Thus, they offer a high quality as well as an economic solution for experimental studies. ▲

## References:

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<sup>2</sup>EDA (European Dairy Association): Annual Report 2016/2017, s. eda.euromilch.org

<sup>3</sup>A. Piry, W. Kühnl, T. Grein, A. Tolkach, S. Ripperger, U. Kulozik: Length dependency of flux and protein permeation in crossflow microfiltration of skimmed milk; Journal of Membrane Science 325 (2008), 887 - 894

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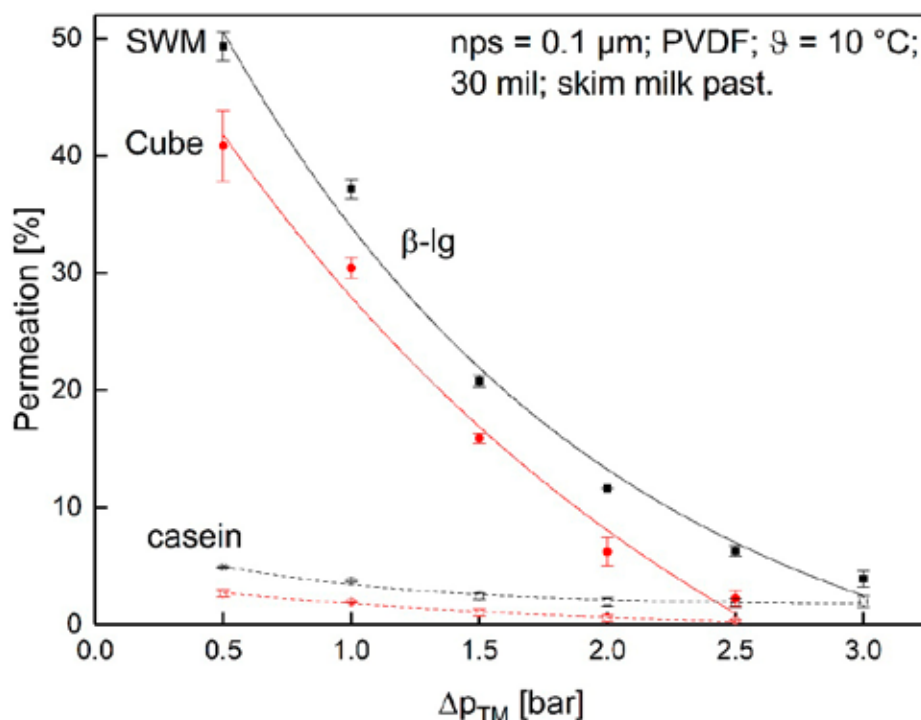


figure 5: Permeation of casein and whey protein during the filtration of skimmed milk by using the Cube 80 test cell and a commercial spiral-wound module (SWM) as function of the trans-membrane pressure (nps: nominal pore size; mil: milli-inch) [photograph: Chair for Food and Bioprocess Engineering Technical University of Munich (Weihenstephan site)]

## Tub retains signature shine

RPC Superfos's CombiPac tub is being used to pack a new range from the UK's favourite butter and spreadable brand. The line extension for Lurpak Spreadables offers tasty new infusions, but the pack retains the well-known silver shine from the parent brand. Packaging plays a major role in distinguishing a brand on shelf and at home, with colour an inherent part of this. For this reason, Arla Foods UK wanted to keep the silver base in the packaging for its new Lurpak Spreadables Infusions. Globally, the silver colour is recognised as a characteristic for Lurpak butter, so the new pack for the new spreadable butter variants has to match the existing packaging for Lurpak butter and spreadable butter.



Suitable for butter, yellow fats and dairy products, the CombiPac tub offers a combination of plastic and cardboard. The outer part is made of cardboard, providing light barrier protection and ensuring a lightweight pack. The inner part consists of a thermoformed plastic sheet, protecting the edible content and making the pack grease resistant.

To meet the precise needs of Arla Foods, the regular pack was slightly adapted so that both the lid and tub are thermoformed and the size made slightly smaller, so that it is perfect for picnics. ▲

## Mouvex integrates Finder Triplex Series Pumps into product offering

Mouvex, part of PSG, a Dover company and one of the leading manufacturer of positive displacement pumps, is pleased to announce the integration of Finder Triplex Series pumps into its growing portfolio of transfer solutions for the energy and industrial markets. Launched in 1952 under the original name of PMH, Finder Triplex Series pumps are highly reliable reciprocating plunger pumps specifically designed for a wide variety of critical applications found in oil & gas (onshore and offshore), nuclear and general industrial industries. These pumps are also compliant with API 674 to provide the best in reliability and safety. Triplex Series pumps are available in seven models – TD18, NF50, NH77, NJ116, NL171, TN 260 and TP420 – with power rating ranging from 13 to 310 kW (18 to 420 HP). Typical applications include water jetting, methanol injection, glycol recirculation, descaling, boiler feeding, and others. ▲

Finder Triplex Series (skid)

