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Inline UV-Vis spectroscopy to monitor and optimize cleaning-in-place (CIP) of whey filtration plants

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ABSTRACT

Cleaning in place (CIP) is a necessary, complex and costly process employed all throughout the food industry. CIP of membrane filtration plants is especially delicate as membranes have limited stability towards temperature and can be permanently damaged or gradually changed by cleaning chemicals used for every day. We investigated the capability of inline UV-Vis spectroscopy to elucidate the dynamics of CIP of membrane filtration plants as a gateway to control and optimize the process. For this investigation aged membranes that had been used for industrial ultrafiltration of whey were transferred to a pilot plant equipped with inline UV-Vis spectroscopy on both the retentate and permeate side. Then the dynamics of multiple fouling and cleaning of these membranes were investigated. The results indicate that the first CIP step, caustic cleaning could be shortened and possibly reduced in concentration. The second step of CIP, enzymatic cleaning, seems to be active even longer than the anticipated time. Challenges, first findings and future steps of full scale inline UV-Vis spectroscopy are discussed. We conclude that inline UV-Vis spectroscopy can be used to optimize the processing time, energy and chemicals needed for cleaning in place of membrane filtration plants.

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1. Introduction

Cleaning in Place (CIP) is an integral part of food processing. Especially filtration plants with spiral wound membrane modules are known to be very challenging from a cleaning perspective. Dead-ends and pockets inside the spiral modules e.g. restrict flow and the sensitive membrane material prevents the use of too harsh chemicals (Stanga, 2010). This requires a very well defined multi-step approach, considerably different from e.g. tank cleaning. Another major challenge is the compositional variation in feed streams, even for the same unit operation in the same dairy, and a possible “carryover” effect of the membrane-internal fouling from previous runs. This makes prediction of the degree of fouling at the beginning of each CIP operation and active predictive-control nearly impossible. As a result, CIP of membrane filtration plants is almost exclusively time- and recipe-based, where the recipe is designed for a “worst case” scenario and the dynamics of the different CIP steps in membrane operation are neglected. The dairy industry would benefit from a new measurement based approach to cleaning that enables it to determine endpoints of cleaning steps, minimize the water footprint and (periodically) optimize the concentrations and total amounts of cleaning agents used. The course of cleaning in membrane operations in the dairy industry can be investigated by monitoring the development of absorbance at 280 nm (the ultraviolet range). Around this wavelength the aromatic amino acids tyrosine and tryptophan have their absorption maximum. These amino acids are an important part of the structure of whey proteins and do thus indicate presence of proteins or protein fragments. Whey protein levels in aqueous solutions down to 10–20 ppm could be quantified from Ultraviolet-Visual (UV-Vis) spectra (Lyndgaard, Rasmussen, Engelsen, Thaysen, & van den Berg, 2014). Some components of sweet whey like the glycomacropeptide can potentially be found in the fouling layer but do not contain any aromatic amino acids (Kilara & Chandan, 2011, pp. 317–333). The third aromatic amino acid, phenylalanine, has its absorption maximum around 260 nm, but can be neglected for the purpose of monitoring CIP dynamics because the extinction coefficient is much lower compared to tyrosine and tryptophan (Wetlaufer, 1963). UV-spectroscopy for inline measurements is a relatively recent development. Previously only the visible range of a UV-Vis spectrum could be used for inline analysis because optical fibers

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would solarize quickly, often within minutes, and the light transmitting ability for wavelengths below 280 nm was thereby lost. Additionally, UV-Vis process spectrometers with affordable and reliable array detectors have been developed which allow whole UV-Vis spectra to be recorded within seconds. Using a measurement cell that is implemented in the process line equipped with light fibers allows the spectrophotometer to be placed in safe distance away from process hazards, making process UV-Vis for CIP monitoring a viable option (Herman, 2000; Liauw, Baylor, & O'Rourke, 2010). The objective of this study was to investigate suitability of inline UV-Vis measurements to monitor, describe and optimize the dynamics of CIP for membrane plants used by the dairy industry.

2. Materials and methods

2.1. Aged membranes

Aged spiral wound membrane modules (6338HFK328, Koch membranes, USA; 22 m^2 membrane area per membrane cartridge) were taken from an industrial ultrafiltration plant (Arla Foods Ingredients, Nr. Vium, Denmark) which is used to concentrate whey in four loops (16 tubes with 4 membrane modules in each loop). The plant typically concentrates whey from around 5.4 to 10 °Brix. For each of two experimental weeks one set consisting of two aged membrane modules was taken from the third loop of the production plant. The first set of membranes was taken after a caustic cleaning step, the first stage in a full cleaning cycle, from a position just before the permeate outlet. A second set of membranes was taken directly after product displacement and flushing, again from the third loop. The membrane modules were stored at 5 °C for no more than 48 h before they were transferred to a custom made pilot plant (200 L dead volume; Alfa Laval, Silkeborg, Denmark).

2.2. Simulated fouling and cleaning

In the pilot plant production with intermittent CIP was simulated five days in a row for each of the two experiments/sets of membranes. Production (feed pressure 100 kPa, booster pressure 200 kPa, temperature 10 °C) was simulated by concentrating 1 m^3 of whey (5.4 °brix) to 0.3 m^3 concentrate (7.1 °brix; app. one and a half hour run time, permeate discarded) and by continuing the process in full recirculation of both permeate and retentate to a total filtration time of three hours. CIP was running on time which started after reaching the set-point temperature (50 °C) employing industrial cleaning solutions dosed in concentrations comparable to full scale production (Ecolab, Düsseldorf, Germany). It consisted of either a one-step reduced cleaning or a full three-step CIP (Table 1). The three steps, employed according to industrial practice, were 1) Caustic (15 min, 0.26 ml/100 ml Ultrasil 115; first half of the total volume added at the start of heating, second half added when 50 °C was reached), 2) Enzymatic (40 min, 0.5 ml/100 ml Ultrasil 69 new and 0.33 ml/100 ml Ultrasil 67; added when 50 °C was reached) and 3) Acid (25 min, 0.24 ml/100 ml Ultrasil 78; added at the start of heating). Total time of the three cleaning steps, including intermediate flushing and heating, was around three hours. Caustic cleaning chemicals were added gradually over the course of one min in order to avoid a pH spike caused by concentrated cleaning agent circulating as a wave through the plant, which might potentially damage the membranes or change the observed cleaning dynamics. The heating ramp of the pilot plant to go from the flushing temperature (10 °C) to the cleaning set-point of 50 °C was slowed down to 23 min to mimic industrial dynamics. A feed pressure of 100 kPa and booster pressure 80 kPa were used for all CIP steps and flushings.

Table 1
Fouling and cleaning regime for the five consecutive days, replicated in the two experimental weeks.

Fouling 1	Caustic	Enzymatic	Acid
Fouling 2	Caustic		
Fouling 3	Caustic		
Fouling 4	Caustic	Enzymatic	Acid
Fouling 5	Caustic	Enzymatic	Acid

2.3. Measurements and calculations

UV-Vis measurement cells (10 mm optical path length, Optek, Essen, Germany), connected to a UV-Vis process spectrophotometer (Tec5, Oberursel, Germany) via light fibers were installed in both the retentate and permeate outlet flows. The spectrophotometer has three channels/array detectors, one for each measuring cell and one connected to an internal fiber loop to automatically compensate for intensity fluctuations of the xenon flash lamp light source.

UV-Vis measurements rely on the well-known Lambert-Beer law:

$$A_\lambda = \varepsilon_\lambda \cdot b \cdot c \tag{1}$$

where A is the absorbance at a given wavelength λ, ε is the molar absorption coefficient (L mol^{-1} cm^{-1}) for a particular chemical species, b is the path length (cm) of the measurement and C is the concentration (mol L^{-1}) of the given solute. Strictly speaking, Lambert-Beer's law is only valid for a monochromatic (one wavelength only) light source. The linear dependency between absorbance and molar concentration is valid for a certain range of concentrations. If the concentration is exceeded, absorbing molecules might start to interact with each other (aggregation, dissociation, etc.; Liauw et al., 2010); in this investigation, during cleaning, we expect to operate well within this linear range.

Fig. 1 shows UV-Vis spectra of the cleaning agents in water for the concentrations as applied in the CIP steps from Table 1. Changes in temperature (10–50 °C) had no observable effect on the measurements (data not shown). The background signal used in the computation of absorbance was MilliQ water and the clean water spectrum is thus a flat line at value zero. Various process data (temperature, permeate flow, pressure, conductivity, etc.) as well as UV-Vis spectra from 250 to 750 nm were recorded real-time. Small air bubbles and foam will develop in a dairy membrane system during CIP and this will introduce offset in the inline UV-Vis

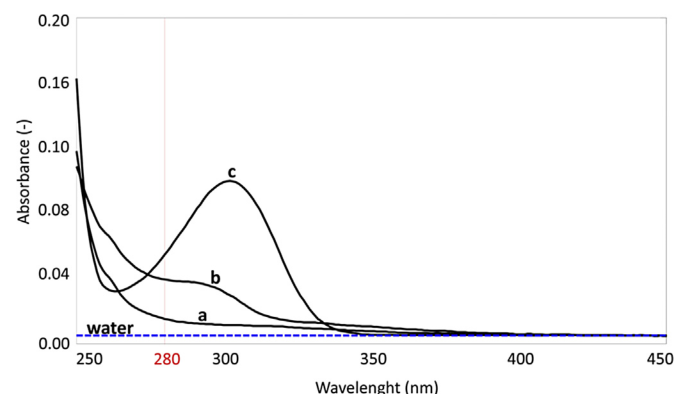

Fig. 1. UV-Vis spectra of cleaning agents in the concentration applied for CIP. a) Caustic, b) buffer plus enzymatic and c) acid.

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