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Membrane technology for the recovery of contaminated single-phase acidic detergents

Izabela Kowalska *

Wroclaw University of Technology, Institute of Environment Protection Engineering, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland

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ABSTRACT

Pressure-driven membrane processes were investigated for the recovery of contaminated cleaning solutions (an acidic single-phase detergent). The experiments were performed in a semi-pilot membrane installation in cross-flow regime with the use of UF and NF differing in their configuration, MWCO and membrane material. The performance of the modules was characterised in terms of their retention characteristics, hydraulic efficiency and tendency to fouling. The influence of long-term filtration time and process conditions (transmembrane pressure, cross-flow velocity and temperature) on these parameters was evaluated. The results revealed that the recovery of spent acidic single-phase detergent is possible with the use of membrane processes. The permeates maintained their basic cleaning properties. The retention of protein from spent detergent amounted to 98.4–100%; however, the separation effectiveness of lactose was more varied (49.5–99.1%) and depended to a greater extent on MWCO and module material.

1. Introduction

In the food industry, cleaning operations are essential for ensuring high standards of hygiene and constant product quality. The cleaning is usually performed by using cleaning-in-place (CIP) systems, which allow the processing equipment to be simultaneously cleaned and sterilized [1,2]. To meet the imposed quality standards, CIP operations consume large amounts of chemicals (caustic soda, acids, surfactants) and water [3]. In production plants connected to the dairy industry, the cleaning system is usually initially flushed with water. Later, chemical cleaning is carried out using alkalis, acids and disinfecting agents. Finally, the equipment is rinsed with water and dried. Depending on the operating mode of the CIP systems (single-use, multi-use or re-use), the volume of alkaline or acidic effluents from the dairy industry is 0.2–2.0 L per 1 L treated milk; and 54–98% of the volume is generated in the CIP system [4].

The need to protect the environment from the harmful effects of aggressive chemicals and the need to reduce water consumption during the washing process have brought about a change in the pattern of cleaning production lines. Conventional methods for cleaning with the help of alkalis and acids are gradually being replaced by novel formulations (single-phase detergents), which effectively simplify the cleaning process. The composition of such a washing detergent contains alkalis or acids, surfactants, complexing and disinfecting agents, and defoamers, which integrates the acid, alkaline and disinfection stages [5].

Pressure-driven membrane processes are widely employed in the dairy industry and the range of application of membranes has escalated in the last 30 years. Membrane processes are primarily used for microbial load reduction, fat reduction in whey protein concentrates, protein concentration or fractionation, and partial demineralisation of the feedstock [6–9]. However, with the use of membrane techniques, which have acquired the name of 'green technologies', the recovery and reuse of a large volume of cleaning solutions within the food industry can also be realised. This leads to a reduction of spent chemicals discharged to the environment, thus saving chemicals, water and energy.

The research published in the past was mainly focused on the recovery of sodium hydroxide and nitric acid and mainly involved nanofiltration [4,10–13], as well as ultrafiltration [14,15] and microfiltration [16]. A critical review of these methods is presented in the work of Suárez et al. [17].

In the literature, there are a very limited number of studies regarding the recovery of single-phase detergent formulation from spent CIP solutions. Research by Fernández et al. [5] revealed the usefulness of spiral-wound NF module with an MWCO of 200 Da for the purification of spent alkaline single-phase detergent from CIP in a yogurt plant. As shown, the purified detergent was successfully reused as a cleaner in real industrial conditions and effectively reduced the consumption of detergent. The applied module (KOCH MPS-34) showed stable permeate flux (45 L h^{-1} m^{-2}) and during the experiment (1800 h) needed only short rinsing steps.

* Tel.: +48 71 320 3639; fax: +48 71 328 2980.
E-mail address: izabela.kowalska@pwr.wroc.pl

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Nomenclature

Symbols

A	membrane area (m^2)
AEC	alkyl ether carboxylate
C_f	concentration of pollution in the feed ($g\ m^{-3}$)
CIP	cleaning in place
CMC	critical micelle concentration
COD	chemical oxygen demand ($gO_2\ m^{-3}$)
CFV	cross-flow velocity ($m\ s^{-1}$)
C_p	concentration of pollution in the permeate ($g\ m^{-3}$)
EON	ethylene oxide units
FR	flux recovery
J	permeate flux ($m^3\ m^{-2}\ s^{-1}$)
J_f	deionised water flux of fouled membrane ($m^3\ m^{-2}\ s^{-1}$)
J_w	deionised water flux of clean membrane ($m^3\ m^{-2}\ s^{-1}$)
MWCO	molecular weight cut-off (Da)
NF	nanofiltration
R	retention coefficient (%)
RF	relative flux (%)
t	filtration time (s)
TDS	total dissolved solids ($g\ m^{-3}$)
TMP	transmembrane pressure (bar)
UF	ultrafiltration
V	volume of the permeate (m^3)

Also, my previous research [18,19] has shown the usefulness of membrane filtration using flat polymeric membranes for the recovery of the single-phase detergents for the purpose of their reuse in cleaning systems.

Taking into account the literature reports, it is advisable to undertake detailed research into the treatment of single-phase detergents and their recycling into the CIP systems. In the present paper, the results of the purification of contaminated cleaning solutions (a single-phase detergent) are reported. For the tests, a liquid acidic single-phase detergent tailor-made for CIP systems in the dairy industry was selected. The effect of the long-term filtration time and process conditions (TMP, CFV and temperature) on the transport and separation properties of UF and NF modules was evaluated. The effect of the process parameters and module type on impurities retention (milk compounds) and the detergency properties of the treated solutions was examined.

2. Experimental

A highly concentrated acidic cleaner recommended for CIP applications in the dairy industry was used. The single-phase detergent was mainly formulated with nitric acid and peroxyacetic acid, with the addition of low-foaming anionic surfactants (alkyl ether carboxylic acid surfactants) and complexing agents. The filtration experiments were conducted for the following model solutions: (A) 0.75% w/v aqueous solution of milk with a fat content of 3.2% w/v, (B) 2% w/v aqueous solution of a single-phase detergent, and (C) 2% w/v aqueous solution of a single-phase detergent to which was added 0.75% w/v of milk to simulate industrial conditions. Solutions A and B are considered as reference solutions. The properties of the feed solutions are presented in Table 1.

The experiments were conducted in a semi-pilot installation made of stainless steel (Fig. 1). Membrane filtration was conducted in an open loop system where the retentate from the membrane was recycled back to the feed tank. The feed solution was pumped to the membrane module by the circulation pump (Grundfos). The studies used commercially available UF and NF modules differing in their configuration, MWCO and membrane material (Table 2). Due to the low pH value of the single-phase detergent solutions, modules with high resistance to the chemical agents were chosen for the tests.

During the experiments, the following process parameters were controlled: the temperature of the feed solution (19–41 °C), the transmembrane pressure (1–4 bar), the recycle flow rate (and cross-flow velocity (0.5–10.5 $m\ s^{-1}$)). In the first stage of experiments, filtration was carried out for 120 min for defined process parameters. The permeate flows were measured every 15 min

Table 1
Physicochemical properties of the feed solutions.

Parameter	Solution A	Solution B	Solution C
pH	7.09	1.96	2.18
Acidity, mmol L^{-1}	–	33.6	32.6
Conductivity, mS cm^{-1}	83.9×10^{-3}	13.46	13.47
Surfactant, %	–	0.029	0.029
Surface tension, mN m^{-1}	47.7	26.7	26.5
COD, $mgO_2\ L^{-1}$	1261	2814	3608
Protein, mg L^{-1}	237	–	237
Lactose, mg L^{-1}	327	–	327
TDS, mg L^{-1}	836	1090	1912

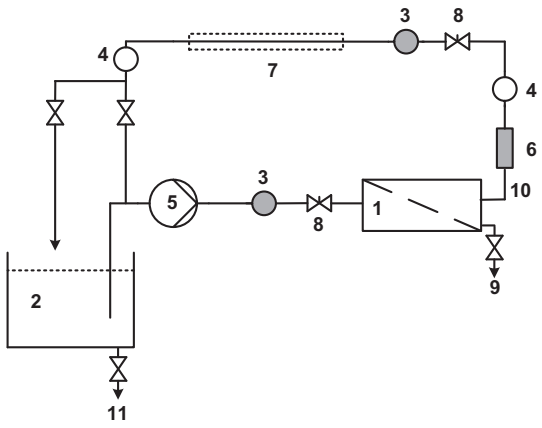

Fig. 1. Schematic diagram of the membrane installation: (1) membrane module, (2) feeding tank, (3) manometer, (4) thermometer, (5) pump, (6) rotameter, (7) cooler, (8) pressure regulation valve, (9) permeate, (10) retentate, (11) drain valve.

and the samples were collected for physicochemical analysis. In the next stage of the experiments, where the effects of the operating conditions were evaluated, filtration was carried out for 60 min and after the allotted time the samples were collected for physicochemical analysis.

The conductivities and pH were measured with a CC-411 conductometer (Elmetron, Poland) (with an EC60 sensor designed for measuring the conductivity in the range of 10 μS cm^{-1}–100 mS cm^{-1}) and a CP-315 M pH meter (Elmetron, Poland), respectively. The acidity was analysed by an acid–based titration method, with the use of 0.1 M NaOH and methyl orange as an indicator. Surface tension measurements of the solutions were taken with an automatic EasyDyne tensiometer (Krüss, Germany) at 20 °C. The concentrations of surfactants in aqueous solutions were determined with the appara-