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Original article

Cleaning-in-place in the dairy industry: criteria for reuse of caustic (NaOH) solutions

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Abstract – Cleaning-in-place NaOH solutions from a milk standardisation dairy plant are reused for several days before being discharged to the purification station. During reuse the surface tension of the industrial cleaning solutions was reduced from ~70 $mJ \cdot m^{-2}$ for newly prepared NaOH down to ~30 $mJ \cdot m^{-2}$ during the week. The decrease was rapid during the first few reuse cycles and stabilised thereafter, while the polluting load and suspended solids increased inversely. The cleaning efficiency test consisted of an ultrafiltration membrane fouled with whey proteins, which was cleaned using these cleaning solutions, either without any treatment, or following crossflow micro- and nanofiltration. It was found that microfiltrates and nanofiltrates with both low surface tension and suspended solids resulted in higher efficiency and cleanliness, and a much faster cleaning rate. The effects of suspended solids and low surface tension will have to be further studied to determine the role of each parameter and confirmed on stainless steel surfaces and in a milk processing plant.

Cleaning / NaOH / surface tension / dairy industry

Résumé – **Le nettoyage en place dans l'industrie laitière : critères pour la régénération des solutions de soude.** Les solutions de soude caustique de nettoyage en place d'un atelier de réception et standardisation de lait sont réutilisées pendant une semaine (soit environ 400 cycles de nettoyage) avant d'être rejetées vers la station d'épuration. Pendant cette période, la tension superficielle décroît de 70 $mJ \cdot m^{-2}$ pour une solution fraîche à environ 30 $mJ \cdot m^{-2}$. La diminution est rapide pendant les premières dizaines de cycles puis se stabilise ensuite, tandis que la charge polluante et les matières en suspension augmentent. L'efficacité et la vitesse de nettoyage par ces solutions industrielles, sans traitement ou après épuration par microfiltration ou nanofiltration tangentielle, ont été quantifiées à l'aide d'une méthode utilisant une membrane d'ultrafiltration colmatée par des protéines de lactosérum. Les microfiltrats et les nanofiltrats sont plus efficaces que la solution industrielle réutilisée dont ils proviennent et même que la solution de soude caustique fraîche.

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Ainsi, l'absence de matières en suspension et une faible tension superficielle sont favorables à un nettoyage efficace et rapide. Ces résultats doivent été confirmés pour des surfaces et équipements en acier inoxydable. L'expérimentation à entreprendre visera à dissocier les effets des matières en suspension et de la tension superficielle et à les quantifier.

Nettoyage / NaOH / tension superficielle / industrie laitière

1. INTRODUCTION

During processing of milk products two main different types of deposits could be distinguished, each with its own character and composition. For cold operation plants (<50 °C) proteins and fat comprise the soil, as lactose and minerals are easily flushed away during rinsing. For hot operation plants surface deposit composition depends on heating temperature: 60% proteins in the range 70–90 °C; 80% inorganic compounds between 110 and 140 °C [6, 25, 29]. In order to restore the efficiency and performance of the production lines a cleaning process cycle must be carried out with the utmost care and attention, since it is crucial to obtain cleanliness of the equipment for continuous production of microbiologically safe products. The cleaning sequence is a series of steps comprising rinsing, chemical washing and sanitising [20, 31]. The chemical cleaning phase is a multistage operation. The cleaning agent first contacts the soil to be removed, wets and penetrates the deposit, reacts with and breaks down the deposited material, and finally disperses it in the cleaning solution. In general, a cleaning process involves the application of different forms of energy to the soil, i.e., chemical energy imparted by the cleaning solution, heat energy by controlling the temperature of the cleaning solution and mechanical energy produced by the flow and agitation of the cleaning solution. Those forms of energy are involved in the intensification of the kinetics of the limiting phenomena: mass transfer (convection, diffusion) and chemical reactions [13, 14].

Detergents for cleaning processes are complex materials formulated from a wide range of chemicals, each being selected to contribute one or more desired properties to the final product. Alkaline detergents for cleaning of proteinaceous and fatty deposits are usually composed of hydroxide, mainly sodium or potassium, with the optional addition of sequestering and surface active agents [28]. The latter are added to facilitate the detergent wetting, suspending, chelating and rinsing powers etc., which are the weaknesses of the pure hydroxide solution [22].

The most common approach to a cleaning process in large-scale operations is to employ cleaning-in-place, CIP, a procedure which involves the circulation or spraying of cleaning and rinsing solutions without the dismantling of the pipe work or any other equipment. The cleaning solutions are commonly used either as single-use or recycled for reuse and multi-use, depending on the processing practices and load of soiling on the process equipment. The solutions are drained after a few to several hundred cleaning cycles with no reasonable technical or scientific reason: colour, odour, floating material, suspended solids, etc. To prolong the life of detergent solution by reducing its pollution, it is common practice to eliminate the soil particles by sedimentation, centrifugation, or by membrane technology [28]. In recent years membrane filtration processes such as microfiltration, (MF), ultrafiltration, (UF), and nanofiltration, (NF), were proposed for the task of CIP solution regeneration, resulting in significant removal of both suspended solids and soluble pollution (soluble chemical oxygen demand, COD_{sol}) [5].

During studies of CIP cleaning solutions in the dairy industry, it was observed that simultaneous to the increase of pollution load (suspended solids, soluble compounds) the surface tension of the solutions dropped to ~50–30 mJ·m^{-2}, compared to fresh 2% NaOH solution (72 mJ·m^{-2}) [19].

Several procedures for evaluating the cleanliness of milk processing surfaces and equipment and its kinetics are proposed for stainless steel. These include sophisticated chambers inserted into industrial equipment [30]; miniaturisation of heat exchanger plates [27] or stainless steel disks [18] or tubes [3, 17]; and heated radial flow chambers [12]. For membranes, which are used for the separation/concentration of milk components, cleaning consists of a wash cycle and the measurement of pure water flux, Ca ion content and optical density of the retentate and permeate, and the development of hydraulic resistance during cleaning and of the cleaned membrane [8]. The hydraulic resistance was used to study the kinetics of the cleaning process [7] and to evaluate different membrane cleaning agents [16].

The aim of the present work was to study the cleaning performance efficiency of reused NaOH CIP solutions after they had been used for varying numbers of industrial cleaning cycles (up to about 400), and to assess the influence of low surface tension values between 30 and 35 mJ·m^{-2} on the cleaning rate. The methodology used consisted of studying the hydraulic parameters of a cleaning cycle on UF membranes fouled with whey proteins as a model to assess the cleaning performance (efficiency, kinetics).

2. EXPERIMENTAL

2.1. Fluids

The solutions used for the cleaning experiments were newly prepared NaOH, and untreated and regenerated reused CIP NaOH solutions.

Reused cleaning solutions composed of NaOH only (at around 2%) without additives, which were obtained from a local dairy (Lactalis, 35 Retiers, France). The solutions originated from the CIP system of milk standardisation equipment (holding tanks, pipelines, cream separator, pasteuriser). The solutions were reused at the plant for one week (about 400 cleaning cycles) for the CIP process at 70–80 °C before they were discharged to the purification station owing to a dark colour or high amount of organic matter. All samples were from the same batch (same week) but differed in the number of cleaning cycles they had served (Tab. I). The composition of the reused CIP solutions was relatively reproducible week after week as was confirmed by sampling and analysing several different batches.

Reused CIP solutions were submitted to: MF, 0.1 μm membrane; temperature, T = 70 °C; tangential flow velocity; v = 7.0 m·s^{-1}; flux, J = 300 L·h^{-1}·m^{-2}; volume reduction ratio, VRR = 50 at constant transmembrane pressure, ΔP. NF, Sol-gel membrane; T = 70 °C; v = 2.7 m·s^{-1}; $\Delta P = 10 \times 10^5$ Pa; VRR = 36–44 at constant flux, as described by Dresch et al. [10]. One solution (NF-415) was divided into two portions which corresponded to low volume reduction ratio during NF (up to 7 for #1) and high volume reduction ratio (up to 33 for #2) (Tab. I).

2.2. Physico-chemical parameters

Analysis of samples was performed on samples, which were stored at 20 °C, within a week after the experiment. Accuracy of the methods is given in brackets:

Density (<1%): DM A48 density meter (AP Paar, Austria). Dynamic viscosity (<1%): D8 viscometer (Haake, Germany), for Newtonian fluids. The viscosity at 70 °C was extrapolated, assuming the behaviour of water according to Henck [19]. NaOH titration (<2%): by HCl 1N, with

Table I. Composition, characteristics and cleaning parameters of the test solutions.

Solution origin	NaOH $g \cdot L^{-1}$	SS $mg \cdot kg^{-1}$	COD_t $g \cdot L^{-1}$	COD_s $g \cdot L^{-1}$	γ $mJ \cdot m^{-2}$	Efficiency (-)	Cleanliness (-)	$k \times 10^{15}$ $m \cdot min^{-1}$
NaOH-0[1]	20.0	0	0.0	0.0	73	0.91	0.41	1
NaOH-0[1]	20.0	0	0.0	0.0	73	0.94	0.55	15
NaOH-0[2]	20.0	0	0.0	0.0	71	0.95	0.44	60
NaOH-250[2]	17.6	790	4.5	2.8	30	0.88	0.24	50
MF-250[2]	16.6	44	3.3	3.1	30	0.97	0.59	110
NF-12	14.8	0	3.2	3.2	33	0.96	0.61	230
NF-25	19.2	0	3.5	3.5	30	0.98	0.73	500
NF-75	17.4	0	2.5	2.5	35	0.99	0.88	280
NF-80	16.3	0	2.5	2.5	33	0.94	0.57	210
NF-150	16.9	0	3.3	3.3	35	0.96	0.68	230
NF-250[2]	15.8	11	1.9	1.9	35	0.98	0.62	360
NF-400	17.6	0	2.9	2.9	31	0.98	0.77	330
NF-415-1*	16.3	0	2.7	2.7	30	0.98	0.75	280
NF-415-2*	17.3	0	3.3	3.3	34	0.98	0.86	140

[1] From Alvarez (2001) [personal communication].
[2] From Dresch (1998) [9].
MF: microfiltrate;
NF: nanofiltrate;
Number after the solution origin (NaOH-250) denotes the number of cleaning cycles it had performed;
SS: suspended solids;
COD_{tot}: total chemical oxygen demand;
COD_{sol}: soluble chemical oxygen demand;
* Solution NF-415 was divided into two portions which corresponded to a low volume reduction ratio during NF (7 for #1) and high volume reduction ratio (33 for #2).

phenolphthalein. Surface tension, γ: (±2.5 $mJ \cdot m^{-2}$) Krüss K 12e (Palaiseau/Hamburg, France/Germany) at room temperature.

2.3. Pollution parameters

COD was measured by Nanocolor Test 29 cuvettes and a PF 10 pocket filter photometer (Macherey-Nagel, Hoerdt, France). This method was correlated with the results given by the NF T 90-101 AFNOR French standard [2] (accuracy: <5%). COD can either be total, when measured on the whole sample, or soluble, when measured in the supernatant after centrifugation at 3000 *g*, 20 °C for 20 min.

Suspended solids, SS (accuracy: <5%), were determined according to the NF T 90-105 AFNOR French standard [1]: filtration on a 1.2 µm glass-fibre filter and drying at 105 °C overnight.

2.4. Cleaning experiments

The ultrafiltration membrane used was Céram 15 $kg \cdot mol^{-1}$ 3 channels, 1.2 m long, α-alumina with Zr or Ti filtering layer, 0.045 m^2 (Tami Industries, 26 Nyons, France). After a standard cleaning cycle, the clean UF membrane hydraulic resistance, R_m, was recorded using 0.2 μm filtered water [16]. Fouling of the UF membrane was achieved using reconstituted whey proteins in deionised water from a whey protein concentrate powder, WPC, PS-90 (Armor Protéines, 35 St. Brice en Coglès, France) at a concentration of 27.8 $g \cdot L^{-1}$ β-lactoglobulin and 3.2 $g \cdot L^{-1}$ α-lactalbumin, as determined by HPLC [24]. Sodium azide at 0.02% was added to prevent bacterial growth.

The UF membranes were fouled using the WPC solution at the following operating conditions: $T = 50\ ^{o}C$; $v = 4.0\ m \cdot s^{-1}$; $\Delta P = 1.5 \times 10^5$ Pa for 1 h. After a water rinse the irreversible fouled membrane resistance, $R_{if\,WPC}$, was calculated from the pure water flux measurement [15]. The cleaning cycle of the fouled UF membrane was performed at: $T = 50\ ^{o}C$; $v = 4.0\ m \cdot s^{-1}$; $\Delta P = 1.0 \times 10^5$ Pa for 1 h. The development of membrane fouling resistance, R_f, with time during the cleaning test was calculated according to Darcy's law [16]. After rinsing with water, the cleaned membrane water flux was measured to calculate the residual irreversible fouling after the test cleaning solution, $R_{if\,c}$. The residual fouling (if any) after the cleaning test was removed using a standard cleaning cycle (Ultrasil 13 followed by HNO_3) as described for the clean membrane [9], and the cleaned membrane resistance, R_m, was calculated. Our data acquisition system provided the mean value of each parameter (T, v, ΔP, J) during the experiment every 15 s.

The kinetics of the cleaning process was assumed to obey a second-order reaction as presented by Daufin et al. [7] for cleaning of membranes after milk UF. The calculation of the rate constant, k, was performed according to the following equation:

$$\frac{dR_f}{dt} = -k\,R_f^2, \text{ giving } \frac{1}{R_f} = kt + \text{constant}.$$

As a result of a rough analysis it was assumed that the very first part of the curve $R_f(t)$ was the only part which reflected the cleaning phenomenon, then that part was analysed according to a second order kinetics reaction. Thus, in order to calculate the rate constant, each R_f curve was printed and the first 5 min or less relevant experimental data points were assigned and graphed as $\frac{1}{R_f}$ vs. time, and the rate constant for each cleaning test was determined. It should be noted that by working that way, a few data points that are already influenced by the refouling phenomenon were taken into account, which resulted in both a calculated value smaller than the true cleaning rate constant and an error depending on the actual experimental points. This error was in the range 19–370% with one very high value of 1800%.

The cleaning efficiency (error ≃ 6%) which represented the relative part of the fouling removed from the membrane by the cleaning process was expressed as follows:

$$\text{Cleaning efficiency} = 1 - \frac{R_{if\,c}}{R_{if\,WPC}}.$$

The UF membrane hydraulic cleanliness (error ≃ 20%), which represented the relative contribution of residual fouling resistance to clean membrane resistance was calculated as follows:

$$\text{Membrane cleanliness} = \frac{R_m}{R_m + R_{if\,c}}.$$

The criterion is much more discriminating than that for efficiency. In order to better illustrate cleanliness and to show how thin the residual fouling layer was after cleaning, pore narrowing (error ≃ 2%) was estimated according to Poiseuille's equation as the ratio between the diameter of a membrane pore after the cleaning test, d_{cf}, to that of the pore of a clean membrane, d_c.

$$\text{Pore narrowing} = \frac{d_{cf}}{d_c} = \left(\frac{R_m}{R_m + R_{if\,c}} \right)^{1/4} .$$

It should be noted that Poiseuille's equation is an accepted approximation for the determination of mean pore diameter of UF membranes [4], in spite of the large pore size distribution displayed even by ceramic UF membranes.

Replication of cleaning tests

Determination of the efficiency of the various cleaning solutions was conducted in duplicates, which gave a rough evaluation of the reproducibility (difference between the replicates). The cleaning tests of the different reused nanofiltrates were not replicated. The significance of the difference between results (cleaning efficiency, cleanliness, cleaning rate) from different experiments was assessed by taking into account experimental errors and difference between previous replicates.

3. RESULTS AND DISCUSSION

3.1. Surface tension and COD in the course of the CIP cycles

As shown in Figure 1, the surface tension of the reused cleaning solution reduced quickly during the early stages compared to the surface tension of a clean NaOH solution of 2% (~72 mJ·m^{-2}, inserted as an extra point, *, in Fig. 1). Already, after a single use of a new cleaning solution its surface tension is reduced to around 50 mJ·m^{-2} (N. Alvarez, personal communication). It stabilised thereafter at a level of ~30 mJ·m^{-2}. At the same time the COD load increased with the number of cleanings. It should be noted that the procedure at the plant for renewing the cleaning solution at the end of the week consisted of draining the used solution without particular attention to total emptying or rinsing of the tank before introducing the new solution. Therefore, in certain cases the residues of the old (discharged) solution "contaminated" the newly prepared solution, thus leading to high levels of suspended solids and low surface tension of the renewed cleaning solutions.

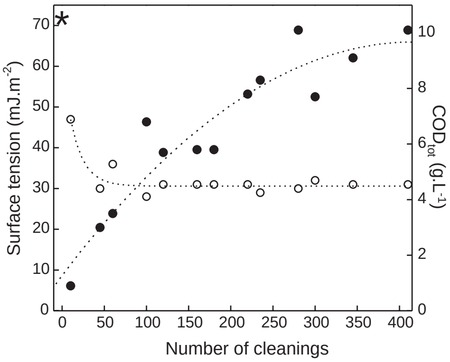

Figure 1. Reduction of surface tension, γ and increase of total COD, COD$_{tot}$, as a function of number of cleaning cycles of the reused caustic cleaning solutions.
○ γ $R^2 = 0.86$
● COD$_{tot}$ $R^2 = 0.90$
* γ of a newly prepared 2% NaOH solution.

The change in surface tension of used cleaning solutions has also been noted by Henck [19] and Dresch et al. [10], while Friis et al. [11] have reported that the surface tension of an enzyme-based cleaning solution is reduced in the course of cleaning. Milk and milk fouling deposits contain several surface-active components such as more or less denatured proteins and derivatives of the milk fat globule membrane such as free fatty acids [23, 26]. Thus, the reduction of the surface tension of the reused CIP solutions could probably be due to the presence of various constituents resulting from soil components and residual milk, hydrolysed by the NaOH, as was also noted for small chain peptides obtained after enzymatic hydrolysis of milk proteins [21]. It should be noted that the reused caustic cleaning solutions attained a low surface tension due to the above-mentioned constituents, which resembles the surface tension of expensive surfactants containing detergents formulated for the dairy industry.

The measure of soluble COD, although seeming to include all the above constituents, did not correlate well to the value of the surface tension, and neither did the soluble nitrogen (results not presented). The phenomenon of the reduction of the surface tension and the species responsible for it, proteins and fat, are now under further investigation.

3.2. Impact of suspended solids and surface tension on cleaning efficiency

The composition of newly prepared NaOH solution, and reused solutions after no treatment or total removal of the suspended solids by MF and NF membranes is presented in Table I. The cleaning efficiency of microfiltrates (0.97 ± 0.01) and nanofiltrates (0.97 ± 0.02) was higher than that of the reference "clean" caustic solution, which did not yield complete cleaning of the membrane (0.93 ± 0.02), while the standard cleaning cycle (Ultrasil 13 + HNO_3) is presented for comparison (Fig. 2). The poor cleaning efficiency of NaOH alone (0.91 – 0.95) has also been reported for membranes after milk UF [7]. The least satisfactory efficiency was with the reused solution, NaOH-250: 0.88 ± 0.02.

The cleanliness of the UF membrane after the cleaning performed with the different solutions confirmed and amplified the conclusion: it ranged from 0.57 to 0.88 for the regenerated (MF, NF) solutions, compared to 0.44 and 0.24 with clean and reused caustic solutions, comparable to inefficient detergents found in other work using UF and MF membranes fouled by milk and whey [7, 8, 16].

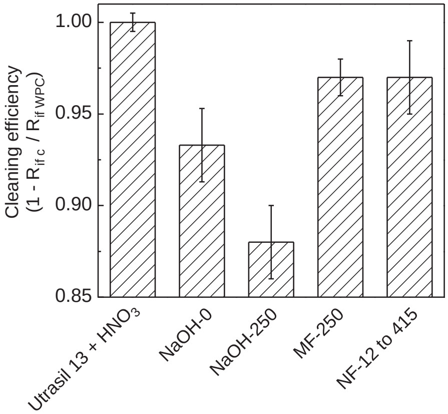

Figure 2. Cleaning efficiency of new and reused CIP solutions.
NaOH-0: newly prepared NaOH;
NaOH-250: industrial solution after 250 cleaning cycles;
MF-250: microfiltered industrial solution after 250 cleaning cycles;
NF-12 to 415: nanofiltered industrial solutions after 12 to 415 cleaning cycles.
Number after MF/NF (i.e., NaOH-250) denotes the number of cleaning cycles the solution had performed.

The contribution of the thickness of the residual fouling was roughly assessed by calculating pore narrowing; it was between 0.80 and 0.86 for pure caustic solutions and 0.88 and 0.97 for reused solutions. Despite cleanliness of 0.57–0.88 for the reused solutions, pore narrowing for the lowest efficiency solution was around 1/20 of the pore diameter, which corresponds to a residual fouling layer of less than 0.3 nm [(1 – 0.88) × 3.5] in the 7 nm pore diameter of the UF membrane used for the test (Tab. I). The latter emphasised the strictness of membrane cleanliness as a cleaning criterion for comparing filtrates of reused CIP solutions.

The nature of the soluble components of the micro- and nanofiltrates, that is, their lowered surface tension (30–35 $mJ{\cdot}m^{-2}$) was likely to explain their higher efficiency (0.94–0.99) and the improved cleanliness

(0.57–0.88) compared to those of the clean NaOH solutions (71–73 mJ·m^{-2} surface tension: 0.91–0.95 efficiency and 0.41–0.55 cleanliness), since lowering the surface tension is known to catalyse the cleaning rate by giving the cleaning solution good wetting properties [28, 31]. The poor results obtained with the untreated reused industrial solution were much as expected, since despite its low surface tension this solution contained a high amount of suspended solids, which could re-foul the UF membrane and alter cleaning performance. This is probably related to the large size of the particles (two populations ranging from 50–500 nm and 5–50 nm) compared to the membrane pore size of 7 nm [9]. This led to the formation of a fouling layer that slowed down permeation flux through the membrane pores during cleaning, and thus resulting in poor cleaning efficiency. Such a detrimental effect of SS has previously been reported on cleaning of stainless steel tubes fouled by heated milk [19].

Finally, a faster cleaning rate was obtained for caustic solutions with low surface tension: despite occasionally high error, the rate constant (2nd order kinetics) was in the range 50–500 × 10^{15} m·min^{-1}, with an order of magnitude higher than 1–60 × 10^{15} m·min^{-1} for clean NaOH (Tab. I). Extra experiments are running so as to quantify and dissociate the role of SS and surface tension on cleaning kinetics with the reused solutions (ex: NaOH-250).

Understanding the influence of the different components present in the reused solutions on the cleaning efficiency will be necessary in order to determine what regeneration treatment should be necessary, if any. Would it be necessary to remove particles and part of the COD, and to what extent? Would it be necessary not to increase surface tension too much, which in turn means which membrane separation, MF, UF, or NF would be the most appropriate? Is there a limit to the life span of regenerated/reused caustic solutions?

4. CONCLUSIONS

Reused CIP solutions demonstrated a high load of SS and COD and low surface tension due to residual milk components and soils that are hydrolysed by the caustic solution. The life span of these solutions at the dairy plant is arbitrarily fixed with no scientific basis. In the present work it is shown that when MF and NF permeates of reused solutions were put to a cleaning test using UF membranes fouled with whey proteins, they achieved better cleaning efficiency compared to newly prepared NaOH solutions. The cleaning kinetic rate assessed from the experimental data according to a second order kinetics shows that faster cleaning was achieved with caustic solutions containing both low surface tension and no suspended solids.

According to the findings of the present study, the effect of low surface tension and high suspended solids has to be dissociated and quantified by using stainless steel surfaces rather than an UF membrane to ascertain the validity of the better cleaning ability of the reused solutions for dairy plants.

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LIST OF ABBREVIATIONS AND SYMBOLS

COD_{sol}: soluble chemical oxygen demand

COD_{tot}: total chemical oxygen demand

SS: suspended solids

d_c: pore diameter of a clean membrane (nm)

d_{cf}: pore diameter of a membrane after a cleaning test (nm)

J: permeate flux ($L \cdot h^{-1} \cdot m^{-2}$)

k: rate constant of the cleaning solution ($m \cdot s^{-1}$)

R_f: overall fouling hydraulic resistance (m^{-1})

R_{if}: irreversible fouling hydraulic resistance (m^{-1})

$R_{if\,c}$: irreversible fouling hydraulic resistance after cleaning test (m^{-1})

R_m: membrane fouling hydraulic resistance (m^{-1})

T: temperature (°C)

v: crossflow velocity ($m \cdot s^{-1}$)

VRR: volume reduction ratio (–)

ΔP: transmembrane pressure (Pa)

γ: surface tension ($mJ \cdot m^{-2}$)

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