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Study of the Fouling Effect of Antifoam Compounds on the Crossflow Filtration of Yeast Suspensions

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ABSTRACT

The fouling effect of different antifoams and compounds of antifoams on crossflow filtration membranes is studied by on-line measurement of the permeation flow during the concentration of suspensions of baker's yeasts in reverse osmosis water (initial yeast concentration, 9 g/L dry matter; concentration factor, 6). The experiments are carried out on three types of tubular mineral microfiltration membranes, two types of flat-sheet organic microfiltration membranes, and one ultrafiltration mineral membrane. The compounds tested are principally silicone oils, silica + silicone based antifoams, and a modified organic antifoam (modified polyalcoxyester). The presence of 100 ppm of any of these compounds

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in the initial feed suspension does not cause a measurable effect for all membranes compared to the fouling due to yeasts. The fouling of a microfiltration mineral membrane due to the amount of silicone antifoam equivalent to 2 years of industrial operation (that is, cumulated in one run without yeast, 25,000 ppm in 12 L feed solution) does not increase beyond the fouling resulting from filtration of the reference yeast suspension.

INTRODUCTION

The industrial fermentation processes often involve several separation steps allowing the recovery of a cell-free liquid and a concentrate of microorganisms. Crossflow filtration appears to be competitive compared to centrifugation and evaporation for the concentration of metabolites and microbial cells (1–3), and permits the development of integrated fermentation methods by coupling a mineral ultrafiltration membrane unit with a stirred tank reactor (4–7).

The main drawback of crossflow filtration is the fouling of membranes which decreases the flux of permeate and changes the rejection features. Sustained effort of experimental work and modeling is devoted to understanding and overcoming this phenomenon, taking into account adsorption on the membrane, relative diameters of pores and particles, cell concentration, transmembrane pressure and compaction of the cake layer, viscosity of the medium, tangential velocity, and turbulence (8–11). Liberge et al. (12) measured and modeled the permeation flow for microfiltration of yeasts on tubular mineral membranes.

But the role played by many unidentified components present in industrial fermentation broths is still questioned. This is the case for antifoam additives, which may contain surfactant, solid particles, and insoluble oil. A report (13) indicates that a general effect of antifoams is to reduce flux rates through the membrane, but no quantitative data are given. The fouling effect of 11 antifoams on organic membranes, especially polysulfone with a 100,000 nominal molecular weight cutoff, has been investigated (14). When dispersed in clean tap water (at a 0.05% w/v concentration), these antifoams show very contrasting effects, from drastic fouling for some inverted cloud-point defoamers to only small reduction of the flux for 100% silicone compounds, for example.

In order to evaluate the effect of different antifoams on the fouling of industrial membranes, a set of experiments has been carried out with six different membrane types, operated in a standardized procedure of concentrative batch filtration of a suspension of yeasts. Complete antifoams and some constitutive compounds were assayed.

MATERIAL AND METHODS

Yeast Suspension and Additives

All feed solutions consisted of 173 g dehydrated baker's yeasts SAF levure (S. I. Lesaffre, F-59703-MARCQ) dispersed in 12 L reverse osmosis (RO) water; dry weight assays indicated a 9 g/L dry matter concentration.

This survey includes different types of defoamers (cf. Table 1). Silicone products Rhodorsil were supplied by Rhône-Poulenc; organic antifoams (a polypropylene glycol ester from Bevaloid and a modified polyalcoxyester) have also been tested. In test experiments, 1.2 g of an antifoam product (100 ppm) was added to the yeast suspension described above, before filtration.

These initial concentrations of yeast and additive are usual in industrial practice.

Filtration Equipment and Procedures

A schematic diagram of the filtration unit is shown in Fig. 1. A single volumetric pump (single rotor screw-pump type) performed the circulation of the suspension and maintained the transmembrane pressure (ΔP). The hold-up volume of the circulation loop was about 1.7 L.

Membranes

Different tubular mineral membranes and organic flat-sheet membranes from Tech-Sep company (F-01703-MIRIBEL) have been tested. Their characteristics are given in Table 2.

TABLE 1

Commercial name	Nature
Rhodorsil antifoam 416	PDMS ^a + silica + surfactant
Rhodorsil antifoam 10153	Copolymer methylsiloxane-polyether + silica
Rhodorsil oil 47V100	PDMS ^a
Rhodorsil resin 10363	PDMS ^a + resin
Rhodorsil antifoam EP 6703	Silicone compound + modified starch (powder-25% active matter)
Bevaloid A59016	Polypropylene glycol ester
—	Modified polyalcoxyester

^a Polydimethylsiloxane (silicone oil).

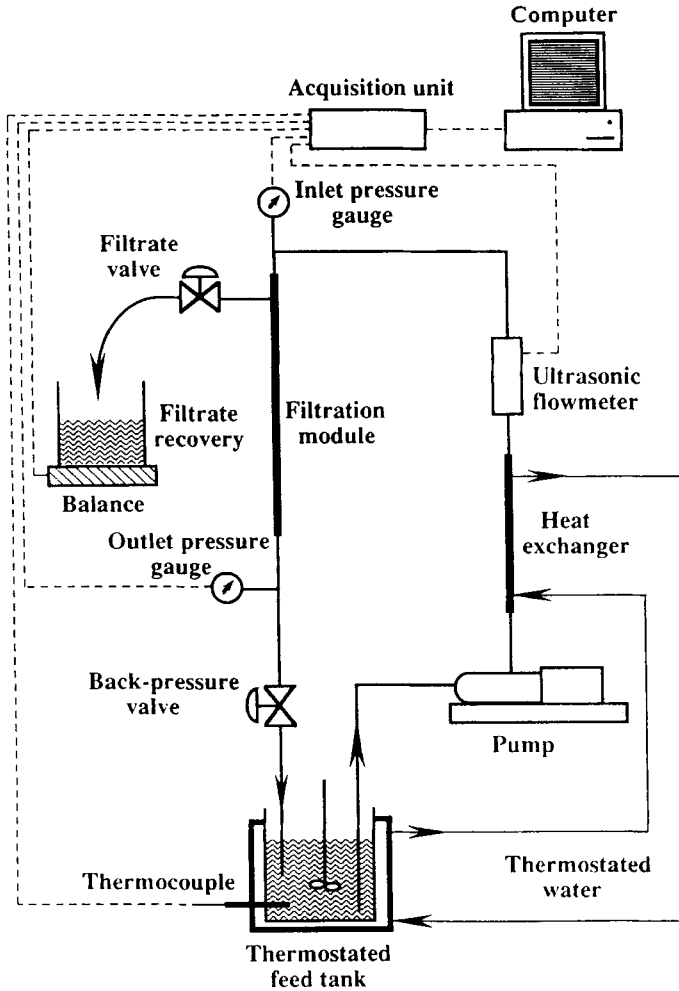


FIG. 1 Schematic diagram of the filtration unit.

Sensors

An ultrasonic flowmeter (model Uniflow System 990 from Controlotron) measured the circulation flow. The inlet and outlet pressures of the membrane were given by pressure gauges (CEMIC 0-4 bar and WIKA 0-10 bar) calibrated with a standard manometer. The temperature of the feed was measured in the feed tank using a thermocouple type K. The effluent

TABLE 2

Commercial name	Mean pore diameter (μm)	Nature	Geometric features surface area operated	Pressure; velocity; temperature
CARBOSEP: M9	Large ultrafiltration, 0.03	Mineral-zirconium oxide on a carbon support	Single tube; 1.2 m \times 6 mm ϕ^a 0.0226 m ²	3.5 bar; 5 m/s; 20°C
M14	Microfiltration, 0.14			
M45	Microfiltration, 0.45			
KERASEP	Microfiltration, 0.45	Mineral alumina	Seven channel element (three channels closed); 0.856 m \times 4.5 mm ϕ^a 0.0484 m ²	3.5 bar; 4.8 m/s; 20°C
IRIS 6502	Microfiltration, 0.2	Organic PVDF	Two flat sheets in series; 17.75 cm \times 8.4 cm; 0.0255 m ² (Ray-Flow system)	1.5 bar; 2 m/s; 20°C
IRIS 6508	Microfiltration, 0.8			

^a Tubular shape: length \times internal diameter.

filtrate was recovered and weighed on an electronic balance (METTLER PM 4600).

Regulation

The temperature of the retentate was maintained at 20°C by a cryothermostat (model HS 50 from HUBER) where the feed tank was immersed and which was also connected to a heat exchanger integrated in the circulation loop. The transmembrane pressure and the crossflow velocity were adjusted by means of the backpressure valve and the speed of the pump.

Data Acquisition Unit and Computer

Experimental data (pressure, temperature, circulation flow, and filtrate weight) were stored on a computer (IBM PC AT) through a data acquisition unit (model Helios 1 from Fluke-Philips) monitored by software (LT-Control from Laboratory Technologies Corp.) as described elsewhere (12). The computer was used to calculate on-line the permeation flow rate from the filtrate weight as a function of time.

Measurements from sensors were collected at a 1 s⁻¹ frequency during the course of all the experiments. In order to smooth the high-frequency variations of parameters (pulsations of pressure and permeate flow), successive measurements were averaged before being stored in the output

file. In addition, three different storage frequencies were defined according to the time of operation: during the first 2 minutes of filtration, when the decrease of the permeation flow is fast, one averaged data was stored per 5 seconds; for the following hour this frequency became 1 min^{-1} and then 5 min^{-1} for the rest of the time of the experiment (cf. Table 3).

Operating Conditions

For Carbosep membranes, the transmembrane pressure ΔP [defined as $(P_{\text{outlet}} + P_{\text{inlet}})/2 - P_{\text{atm}}$] was set to 3.5 bar and the crossflow velocity to 5 m/s. The same pressure was used for the Kerasep membrane but the velocity was set to 4.8 m/s in order to keep the shear-stress τ constant:

$$\tau = (P_{\text{inlet}} - P_{\text{outlet}})D/4L$$

where L and D are the length and the diameter of the tube, respectively.

For the organic membranes the circulation flow was maintained at 800 L/h (corresponding to a velocity of 2 m/s according to the manufacturer), and the transmembrane pressure was 1.5 bar.

All experiments were carried out at 20°C .

Procedure

The successive steps of the experiments were as follows.

1. The filtration unit, equipped with a *new membrane*, was fed with 11 liters RO water, and the water flux through the membrane was measured for 15 minutes under working conditions in the case of Carbosep membranes; the transmembrane pressure was decreased and the duration of observation was shortened for the Kerasep and organic membranes because of the great initial water flux obtained in these cases.
2. The filtrate valve was turned off while maintaining the circulation flow; the predispersed yeast and antifoam compound (1 L volume)

TABLE 3^a

	Stage number		
	1	2	3
Scanning frequency	1 measure/1 second	1 measure/1 second	1 measure/1 second
Stage duration	2 minutes	1 hour	24 hours
Averaging on	5 measures	20 measures	20 measures
Storage frequency	1 point/5 seconds	1 point/1 minute	1 point/5 minutes

^a After Liberge et al. (12).

- were added and rapidly mixed to make up the 12 L of initial feed suspension.
3. The filtrate valve and the acquisition program were turned on simultaneously, and the concentration run was carried on until 10 L filtrate were recovered (concentration factor 6). During the experiment, independent observations of time and filtrate weight were made in order to secure the results of on-line acquisition.
 4. With the filtrate valve turned off, the unit was rinsed with softened water, then with RO water (high speed pumping under minimal pressure and without recirculation of water).
 5. Filtration of RO water was performed under working conditions for 15 minutes with the data acquisition on.

Silicon Analysis

After centrifugation of the sample (if needed), 10 g of the upper liquid phase was extracted with 12.5 mL carbon tetrachloride. Both phases were mixed for 20 minutes in a 100-mL glass bottle. The lower phase was collected and analyzed by atomic absorption. Standard solutions consisted of Rhodorsil 47V100 in CCl_4 .

Operating Variables Measured

The effect of the additives on the fouling of each type of membrane was evaluated through a comparison of the filtration flux decrease during the concentration run with the experimental time spent to concentrate the suspension 6 times (Δt_{c6}).

The operating condition parameters (temperature, pressure, and crossflow velocity) were kept constant throughout each experiment with good accuracy (cf. the example in Fig. 2). However, in order to compare the membranes under theoretically identical conditions, a standardized filtration flux J^{4-25} , expressed in $\text{L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ for a 4-bar transmembrane pressure and at 25°C, was computed from the experimental flow rate of permeation:

$$J^{4-25}(t) = J^{\text{exp}}(t) \times [4/\Delta P(t)] \times [\eta(\theta(t))/\eta(25^\circ\text{C})]$$

where $\Delta P(t)$ and $\theta(t)$ are the transmembrane pressure and temperature at time t , η is the viscosity, and $J^{\text{exp}}(t)$ is the experimental filtration flux at time t .

The temperature correction affects the viscosity of the medium, which was shown to vary like that of water (15).

For mineral membranes, the standardized flux is supposed to closely represent an experimental reality due to the small difference between the

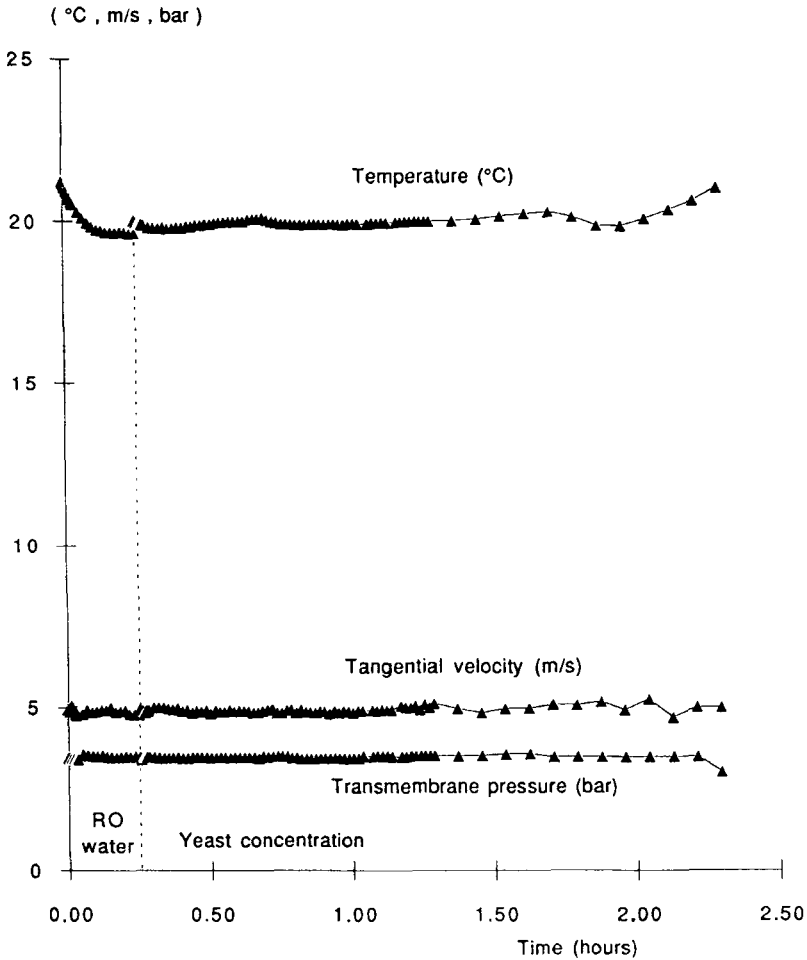


FIG. 2 On-line acquisition of operating parameters for a typical run: temperature, recirculation velocity, and transmembrane pressure as functions of time (experiment of concentration of reference yeast suspension on Carbosep M9 membrane).

actual temperature and pressures of the runs and the theoretical conditions. For organic membranes, the theoretical transmembrane pressure of 4 bar is not realistic and the $J^{4-2.5}$ flux must only be considered as a numerical standardization.

In order to give a simple representation of the filtration runs, the concentration factor (CF) has been plotted versus time. The marks correspond

to direct measurements of time and weight. The continuous curve is deduced from on-line computed flow rates under experimental conditions:

$$CF(t) = V_0/[V_0 - V_f(t)]$$

where V_0 is the initial feed volume and $V_f(t)$ is the total filtrate volume recovered at time t .

$$V_f(t) = \Omega \int_{t_0}^t J^{exp}(t) dt$$

where Ω is the operating membrane area and t_0 is the starting time of the concentration run.

The superposition of the marks and the continuous line indicates the reliability of the computed data.

In addition, a standardized filtrate volume (V_f^{4-25}), corresponding to the filtrate recovered after a 1-hour concentration run on a 1-m² membrane area under standardized conditions, has been computed. This permits comparison of all the pairs tested (feed suspension, membrane) with this common quantitative parameter. A time of 1 hour was chosen because it is shorter than the shortest experiment—and so avoids any extrapolation in time—and longer than the period of the abrupt decrease of the permeation flow at the beginning of the concentration runs.

$$V_f^{4-25} = \int_{t_0}^{t_0+1h} J^{4-25}(t) dt$$

RESULTS

Fouling Effect of the Yeast Suspension in the Presence of Different Additives (100 ppm)

Figures 3–7 show some examples of the evolution of the standardized flux with time for the concentration of yeasts in the presence of silicone additives (Rhodorsil 416 or Rhodorsil 47V100) or their absence (reference suspension: only yeasts). The results obtained with the different types of membranes are presented.

The first evidence is the similarity of the flux decrease profiles during concentration whether an additive was present or not. This qualitative observation is illustrated by the plot of repeated assays with the same feed suspension; for example, the two runs with only yeasts on membrane M9 (Fig. 3) or with the addition of Rhodorsil 416 on membrane M14 (Fig. 4). These expected identical curves cannot be distinguished from the other

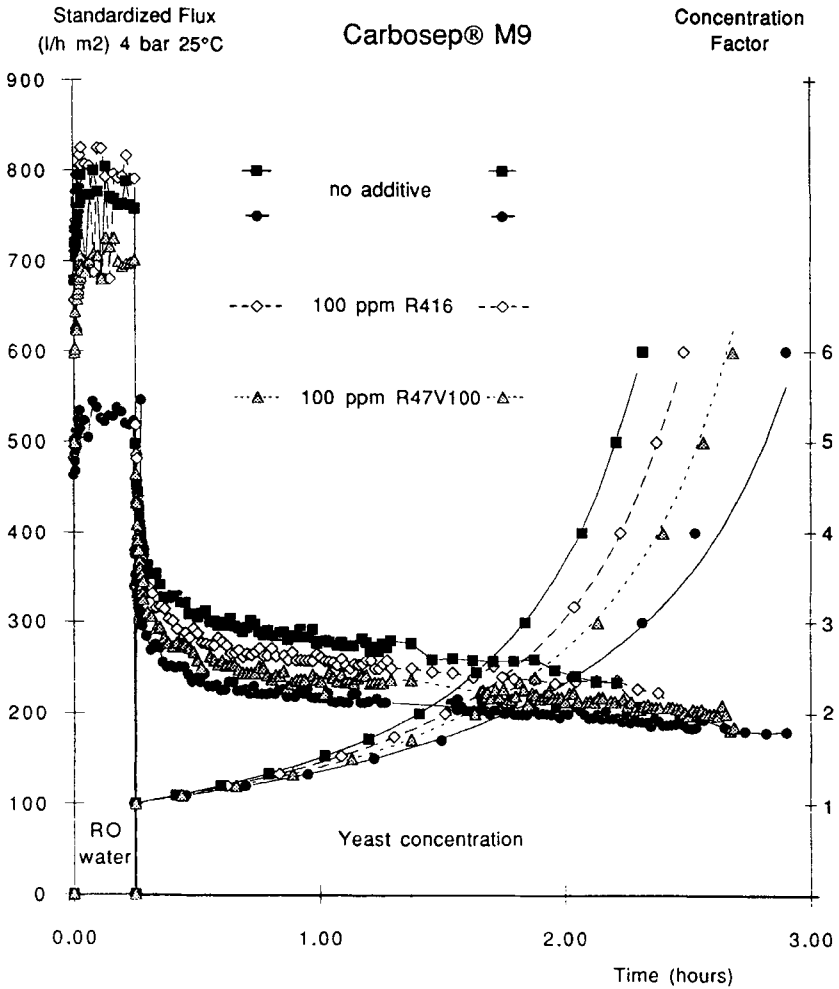


FIG. 3 Standardized flux (J^{4-25}) and concentration factor as functions of time for filtration runs on Carbosep M9 membrane; comparison of the results with and without Rhodorsil 416 and 47V100 additives.

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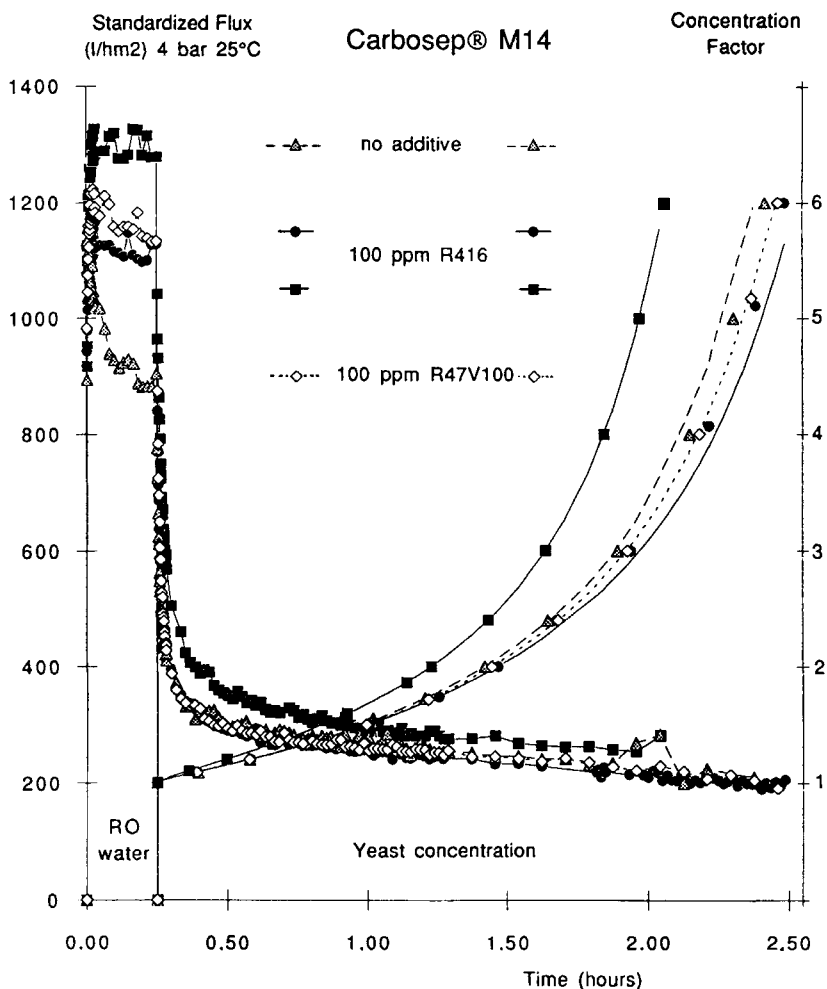


FIG. 4 Standardized flux (J^{4-25}) and concentration factor as functions of time for filtration runs on Carbosep M14 membrane; comparison of the results with and without Rhodorsil 416 and 47V100 additives.

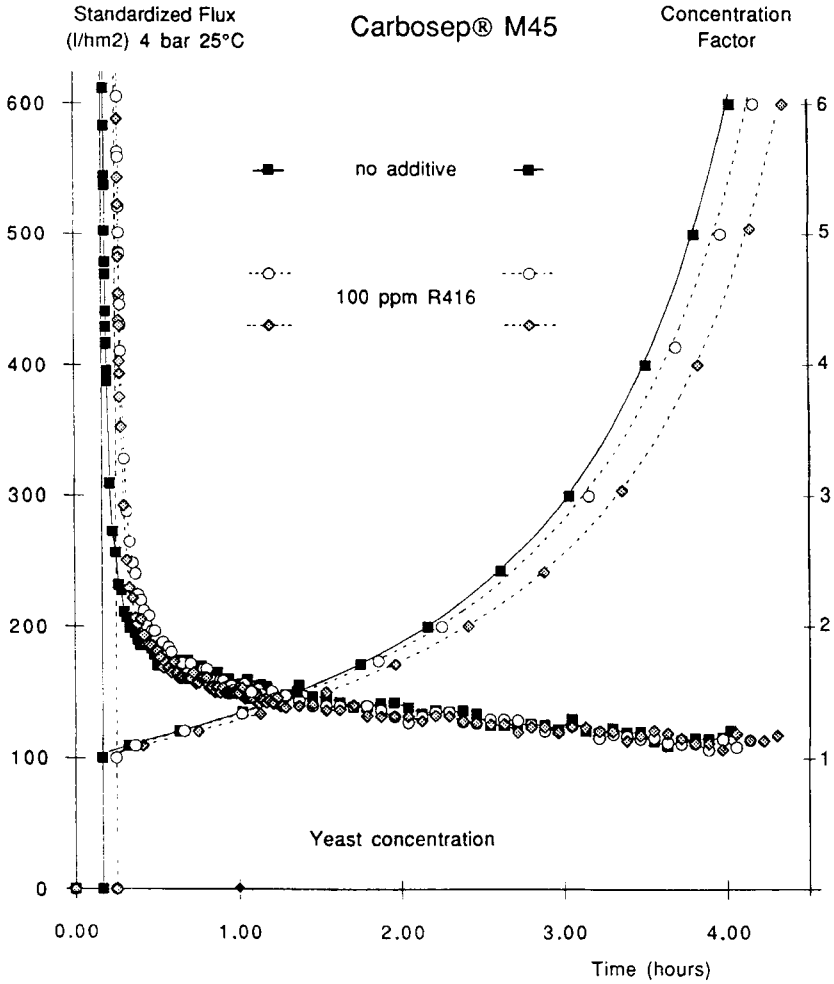


FIG. 5 Standardized flux (J^{4-25}) and concentration factor as functions of time for filtration runs on Carbosep M45 membrane; comparison of the results with and without Rhodorsil 416 additive.

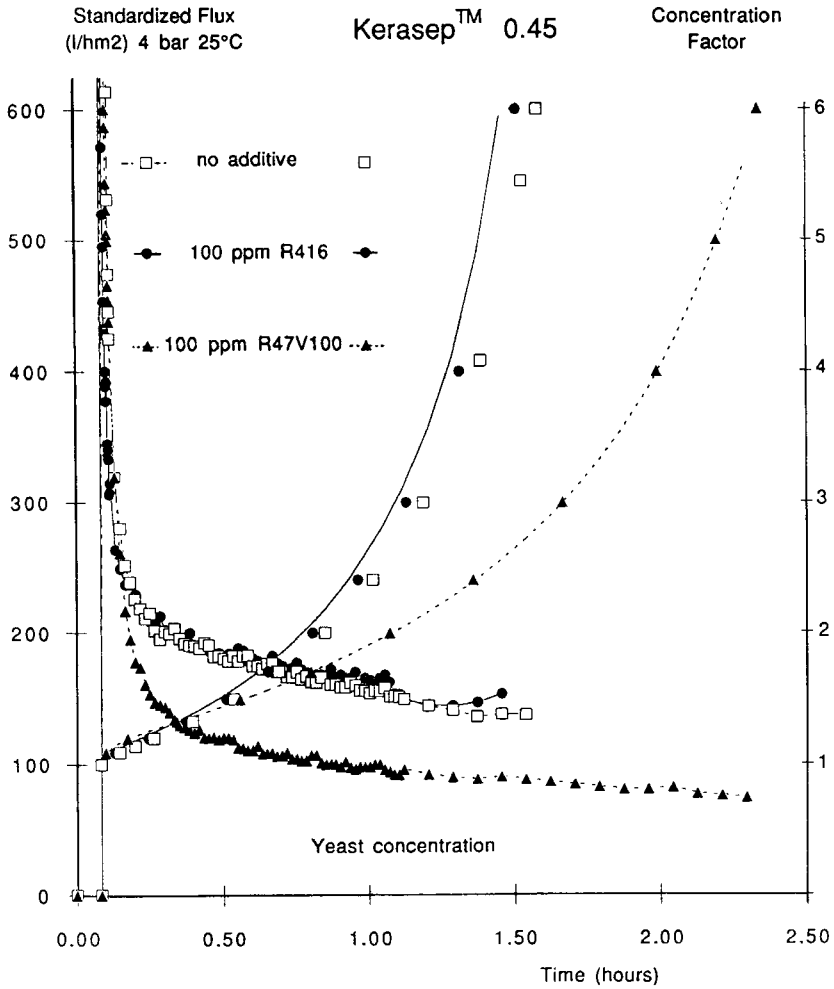


FIG. 6 Standardized flux (J^{4-25}) and concentration factor as functions of time for filtration runs on Kerasep membrane; comparison of the results with and without Rhodorsil 416 and 47V100 additives.

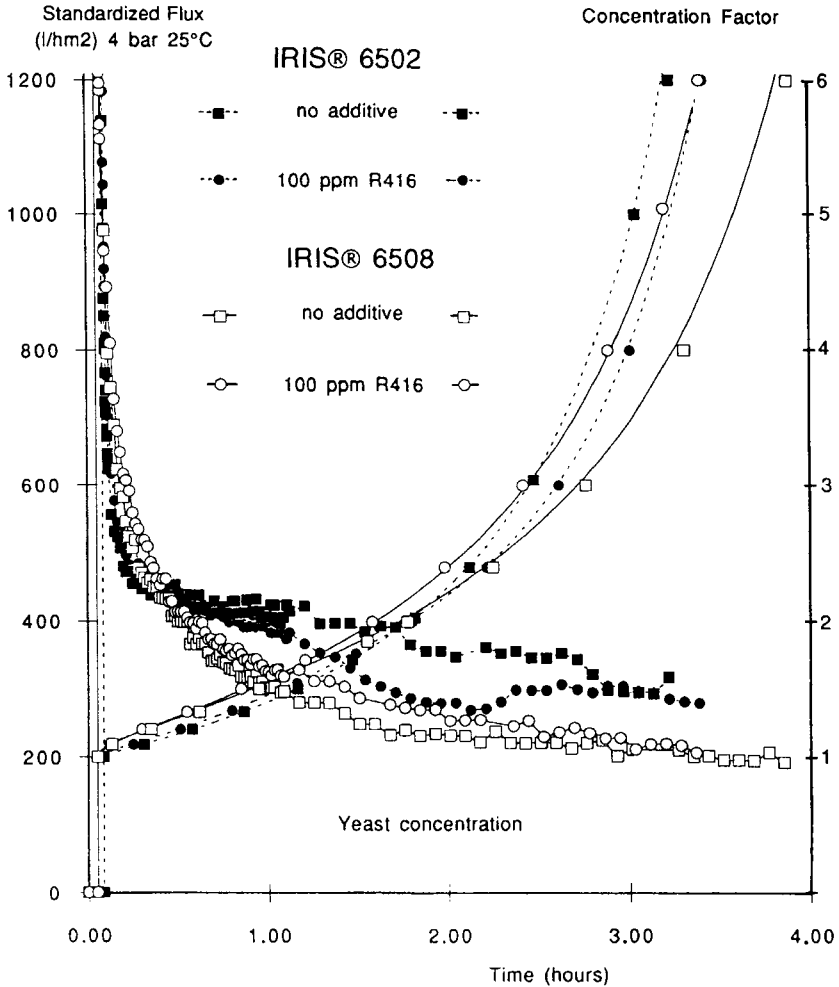


FIG. 7 Standardized flux (J^{4-25}) and concentration factor as functions of time for filtration runs on IRIS 6502 and IRIS 6508 organic membranes; comparison of the results with and without Rhodorsil 416 additive.

plots obtained with different feed suspensions. This result indicates that the differences between flux profiles for the same type of membrane are mainly due to differences between the membrane samples themselves.

The results of the whole set of experiments are reported in Table 4. Some runs with Carbosep membranes were performed 2 or 3 times in

TABLE 4^a

Membrane	Results	Yeast	Yeast + R416	Yeast + R47V100	Yeast + R10153	Yeast + B1590/6	Yeast + R10363	Yeast + REP6/703	Yeast + modified polyalcoxyester
CARBOSEP M9	n	2	2	2	1	1	2	1	2
	Δt_{c6} (minutes)	142	148	138	124	138	138	117	134
	V_f^{4-25} (liters)	268	258	269	298	221	273	317	282
	Variation in V_f^{4-25}	0	-3.4%	0.36%	11%	-17%	2.1%	18%	5.5%
CARBOSEP M14	n	3	3	2	1	2	2	1	2
	Δt_{c6} (minutes)	131	124	128	123	140	140	142	142
	V_f^{4-25} (liters)	296	316	300	297	271	273	276	282
	Variation in V_f^{4-25}	0	6.7%	1.1%	0.27%	-8%	-7.8%	-6.7%	-4.7%
CARBOSEP M45	n	2	2	2	1	1	1	1	1
	Δt_{c6} (minutes)	220	240	240	183	183	183	208	208
	V_f^{4-25} (liters)	196	198	198	183	183	183	253	253
	Variation in V_f^{4-25}	0	0.93%	0	-7%	-7%	29%	1	1
KERASEP	n	1	1	1	1	1	1	1	1
	Δt_{c6} (minutes)	90	86	135	109	132	132	109	109
	V_f^{4-25} (liters)	197	213	160	177	161	161	182	182
	Variation in V_f^{4-25}	0	8.4%	-18%	-10%	-18%	-18%	-7.7%	-7.7%
IRIS 6502	n	1	1	1	1	1	1	1	1
	Δt_{c6} (minutes)	189	200	166	166	166	181	162	185
	V_f^{4-25} (liters)	457	475	558	558	500	500	540	486
	Variation in V_f^{4-25}	0	3.9%	22%	22%	9.4%	18%	6.5%	6.5%
IRIS 6508	n	1	1	1	1	1	1	1	1
	Δt_{c6} (minutes)	230	201	196	196	172	172	208	182
	V_f^{4-25} (liters)	514	514	689	689	575	575	608	651
	Variation in V_f^{4-25}	0	0.39%	35%	35%	12%	12%	18%	27%

^a n = number of runs; Δt_{c6} = experimental duration for concentration factor 6; V_f^{4-25} = computed filtrate volume for 1 hour of filtration (4 bar, 25°C), on a 1-m² membrane area; Variation V_f^{4-25} = $[V_f^{4-25}(\text{yeast})/V_f^{4-25}(\text{yeast})] - V_f^{4-25}(\text{yeast})$; all additives were present at a 100-ppm initial concentration.

order to evaluate the reproducibility of the results. In these cases, only averaged values of Δt_{c6} and V_f^{4-25} are given.

It is remarkable that the relative variations of the averaged V_f^{4-25} values obtained with different feeds, compared with the suspension of yeasts without additive, are within $\pm 8\%$ for each type of membrane, while the relative variation between individual results of repeated runs is within $\pm 13\%$ (data not shown). The same conclusion can be deduced from a comparison of the Δt_{c6} values: the fouling effect of the antifoam compounds is not significant compared to the effect of the yeasts.

For the experiments which were done only once, the scale of variation, relatively to the yeast reference, was larger, but generally less than 20%. But no trend appears which could be correlated with the presence or the absence of an antifoam compound. This result, which is surprising with regard to the literature (13), can be explained in different ways:

The compounds cannot cause fouling because they do not interact with the membrane

The compounds could interact but the quantity present in the feed suspension is too small compared to the amount of yeasts or with regard to the membrane area used

Fouling Effect of High Concentration of Rhodorsil 416 Antifoam on Carbosep M14 Membrane

A single test experiment was carried out using the Carbosep M14 membrane because it appears to be a good choice for yeast concentration. The duration of operation was shorter than what was used with the other mineral membranes tested after comparison of the experimental Δt_{c6} values in the Carbosep series or after comparison of the standardized data.

The following considerations have guided the design of the experiment: One crucial constraint for numerous industrial applications is to be able to maintain an average permeation flux approximately stabilized over a long period of utilization (for example, 2 years) by means of regular cycles of filtration and cleaning operations. An industrial filtration unit in working conditions is supposed to handle 125 m³ of fermentation broth per m² of membrane during a period of 2 years.

If the broth contains 100 ppm antifoam, that would correspond to 280 g of silicone compound with respect to the membrane area operated in our experiments. The same procedure of filtration as described in the Material and Methods Section has been thus performed 1) with only RO water as reference, and 2) with 300 g Rhodorsil 416 antifoam dispersed

in 12 L RO water (without yeasts). The good dispersion of the product was confirmed by examination of the filtration unit after concentration. No significant sedimentation was observed in the usual dead-ends of the circulation loop (manometers) nor in the feed tank.

Table 5 and Fig. 8 compare the results of the above experiments with those of a precedent run with only yeasts. They show that a 250-times increase of Rhodorsil 416 concentration causes membrane fouling lower than fouling due to yeasts. That partly answers the points put forward previously.

1. It seems less surprising that no special effect of fouling was observed when 100 ppm Rhodorsil 416 was added to the suspension, and similarly, that no effect was observed for related compounds.
2. The Rhodorsil 416 antifoam does not permeate freely through the membrane since it causes fouling. This fouling phenomenon is also underlined by the effect of water rinsing: a similar increase of the water flux is obtained after rinsing a yeast-fouled or a Rhodorsil 416-fouled membrane (third part of the J^{4-25} plot). This is in agreement with the preliminary, and semiquantitative, analyses of silicon performed on samples of filtrate and samples of retentate (initial feed and 6-times concentrated retentate). The analyses indicate that less than 1 ppm Si is measured in the filtrates, while an initial concentration of 40 ppm silicon atom is expected in the initial retentate (corresponding to 100 ppm silicone compounds) and while silicon is found to be concentrated in the final retentates (assays done for Rhodorsil 47V100 and 416 antifoam for the different types of mineral membranes tested).
3. The progressive fouling due to the equivalent of 2 years of operation put in one run, even if not representative of the industrial conditions, provides some indication for the possible utilization in crossflow filtration of antifoam-loaded broths but requires further investigation in order to understand the "fouling reputation" of these compounds.

TABLE 5

	RO water	Yeast	Rhodorsil 416 (25 g/L)
Δt_{c6} (minutes)	39	130	67
V_f^{4-25} (liters)	870 ^a	298	529

^a Extrapolation.

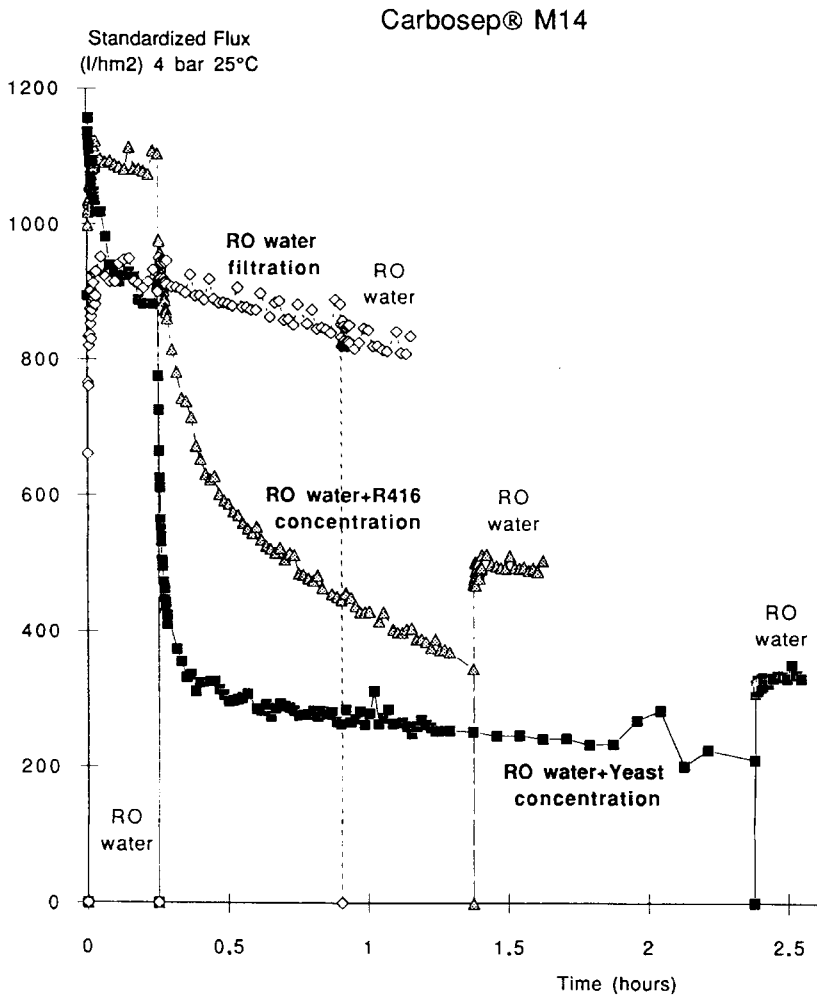


FIG. 8 Comparison of J^{4-25} flux on Carbosep M14 membrane for filtration runs with RO water only and with yeast suspension without additive and high concentration (25,000 ppm) of the Rhodorsil 416 antifoam only. The third part of the plots, following the concentration step, represents the standardized flux obtained after membrane rinsing.

CONCLUSION

Crossflow filtrations carried out with different antifoams do not indicate a specific fouling effect due to these additives when they are dispersed at an initial concentration of 100 ppm in a suspension of yeasts in water. This result was well established for different silicone compounds, particularly for the Rhodorsil 416 antifoam, for Rhodorsil 47V100, and for a modified polyalcoxyester, after repeated filtration runs on Carbosep M9 and M14 membranes. The differences between the filtration features of distinct assays in the same membrane series was lower than 8%. For the other pairs (additive, membrane) submitted to a single assay, the same behavior was observed.

The filtration experiment performed without yeasts but with a high concentration of Rhodorsil 416 antifoam (25,000 ppm) dispersed in RO water showed only a slight fouling. This strengthens the conclusion that under the prevailing conditions (yeasts and 100 ppm additive), membrane fouling was mainly due to yeasts.

However investigations under industrial conditions should be carried out. The present experimental procedure, according to which a new membrane was used for each run, leads to clear conclusions, but long-term fouling series of filtration-cleaning cycles on the same membrane should be studied.

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