Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Bhattacharyya, D. , Jumawan, A. B. , Grieves, R. B. and Harris, L. R.(1979) 'Ultrafiltration Characteristics of Oil-Detergent-Water Systems: Membrane Fouling Mechanisms', Separation Science and Technology, 14: 6, 529 — 549

To link to this Article: DOI: 10.1080/01496397908068474

URL: http://dx.doi.org/10.1080/01496397908068474

SEPARATION SCIENCE AND TECHNOLOGY, 14(6), pp. 529–549, 1979

Ultrafiltration Characteristics of Oil–Detergent–Water Systems: Membrane Fouling Mechanisms

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Abstract

The ultrafiltration characteristics of oil (bilge oil and synthetic based lubricating oil)–nonionic detergent–water (river water and distilled water) systems are evaluated with noncellulosic, tubular membranes. The water flux behavior (membrane fouling) is dictated by the membrane resistance increase due to detergent–membrane interaction and due to surface fouling in the presence of oil–detergent emulsions and suspended solids. Membrane fouling and cleaning requirements depend on the type of oily water systems. Flux drop can be minimized by operating at temperatures above 35 °C and/or with short-term membrane depressurization. In all cases the steady-state water flux is a function of the initial membrane water flux. Depending on the oil water systems, water fluxes of 8 to 52 × 10^{-4} cm/sec are obtained. Excellent oil rejections are observed in all cases: even with oil–detergent systems, an ultrafiltrate oil concentration of less than 10 mg/l can be achieved.

INTRODUCTION

Membrane ultrafiltration with noncellulosic membranes is a promising technique for the simultaneous removal of various soluble organic com-

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pounds, chemical and mechanical emulsions, and particulates present in wastewaters. Ultrafiltration is generally carried out at low pressures (0.7 to 7.0 atm) and offers an attractive alternative in many processing areas. This process produces a dilute ultrafiltrate (permeate) stream and a concentrate stream only 5 to 10% of the raw feed stream. Depending on the type of waste (size of organic molecules) and the nature of the membrane (polymer type and pore size), the ultrafiltrate stream can meet water quality guidelines for direct discharge (*1*, *2*) and/or water reuse (*3*, *4*). However, performance limitations (flux drop) may be encountered during ultrafiltration due to the high-flux characteristics of the membranes, which result in the rapid convection of retained solutes to the membrane surface and the well-documented phenomenon of concentration polarization or gel formation (membrane fouling). Thus, for the successful operation of a membrane ultrafiltration unit, the water flux drop (loss) must be minimized by establishing the proper operating conditions.

The treatment of oily wastes (free oil and emulsified oil and detergents) by ultrafiltration for the purpose of meeting proposed 1980 marine discharge standards (oil less than 15 mg/l) is a promising application. This study is directed toward the processing of shipboard "bilge water" containing fuel oils, lubricating oils, hydraulic oils, detergents, etc. Bilge oil characteristics and generation rates depend on ship types and ship operating modes. Smookler and Harden (*5*) have investigated bilge wastes aboard various classes of Navy ships.

Oil separation techniques have included gravity separation for free oil removal and combination gravity separators with coalescing plates for free and dispersed oil droplets (*6*, *7*). Effluent oil concentrations from these separators are dependent on the specific gravity of the oil, extent of emulsification, oil droplet size, and inlet oil concentration. The presence of detergents (such as bilge cleaners) and/or high suspended solids causes chemical emulsification of oil, and inadequate oil removal (*7*) would be expected with gravity or coalescer systems. In the absence of detergents, bilge water treatment with tight coalescer elements has provided effluents containing less than 20 mg/l oil (*7*).

The use of ultrafiltration for the treatment of various types of oily wastewaters has been reported in the literature (*1*, *8–12*). Harris et al. (*1*), in their studies with turbine lubricating (synthetic) oil and bilge oil, showed that ultrafiltrates containing less than 10 mg/l oil could be consistently obtained even in the presence of detergents. The ultrafiltrate concentration was found to be independent of the feed oil concentration (100 to 5000 mg/l oil) and of the oil specific gravity. In their studies with tubular noncellulosic membranes, extensive flux drops were reported with oil–water systems containing detergents. The cumulative flux drop behavior was also found to be dependent on the type of water (tap, river, or seawater).

The objectives of this study are to investigate experimentally the water flux and fouling mechanisms of tubular noncellulosic membranes with specific oily waters (with and without detergents), and to identify the membrane operating conditions necessary to minimize flux decline and membrane cleaning requirements. The water flux characteristics and fouling behavior are explained in terms of mathematical models.

EXPERIMENTAL

All continuous-flow ultrafiltration experiments were conducted in a commercially available, noncellulosic, tubular unit of 2.5 cm diameter and 1022 cm^2 membrane area. The membrane characteristics are shown in Table 1. Experiments were conducted for 6 to 8 hr (approximate steady-state), and in a few experiments the ultrafiltration time was extended to 15 to 90 hr. A schematic diagram of the experimental unit is shown in Fig. 1. Most experiments were conducted at negligible water recovery, and the feed concentration was maintained constant by the recycle of both the concentrate stream and the ultrafiltrate stream to the feed tank.

The average transmembrane pressure, Δp, was varied between 0.48 and 2.6 atm. The flow regime was always in the turbulent region, and most experiments were conducted at a linear velocity (tube) of 427 cm/sec (102 l/min). The feed solution temperature was maintained constant during the course of an experiment.

TABLE 1

Tubular Membrane Characteristics

Parameter	Membrane
Composition	Noncellulosic
Diameter, cm	2.5
Length, cm	152.4
Membrane area, cm^2	1022
Apparent pore size, Å	50
Maximum operating temperature, °C	60
pH range	2–13
Solute-free water flux (initial water flux) at 1.4 atm; cm/sec × 10^4	75–150

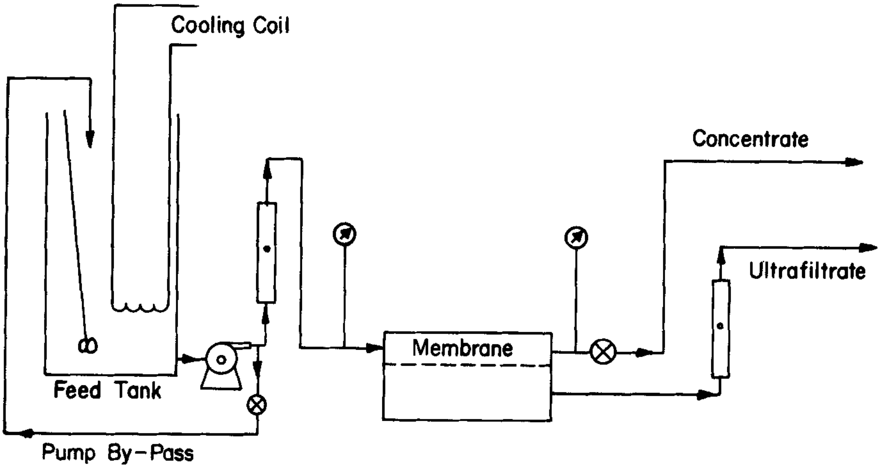

FIG. 1. Ultrafiltration test system.

The feed solutions (to the ultrafiltration unit) investigated in this study were detergent–distilled water, oil–distilled water, oil–detergent–distilled water, river water, oil–river water, and oil–detergent–river water. Bilge oil (oil layer from shipboard bilge) was used in most experiments. The bilge oil used was a mixture of fuel oil, lubricating oils, and hydraulic oils. Some experiments were conducted with synthetic base, turbine engine lubricating oil (Military Specification MIL-L-23699B). The detergent used in this study was nonionic in nature (Military Specification MIL-D-16791E) and contained 99% isooctyl aryl polyether alcohol. Several studies were also conducted with commercial nonionic surfactants (Triton X-100 and Neodol 25–9). Experiments with river water were conducted with water obtained from the Severn River, Annapolis, Maryland (Table 2). A series of ultrafiltration experiments was also conducted with shipboard bilge water.

TABLE 2

Analysis of River Water Used in the Ultrafiltration Studies

Parameter	Concentration
Total solids	11,156 mg/l
Suspended solids	594 mg/l
Conductivity	10,800 μmho/cm
Total organic carbon	3.1 mg/l
Iron	5.6 mg/l
pH	7.8

The membrane rejection behavior was primarily monitored by total organic carbon analysis. In some experiments the oil content in the ultrafiltrate was determined by a solvent extraction–infrared spectrophotometry technique (*1*).

WATER FLUX BEHAVIOR: FOULING MODEL

Water transport through ultrafiltration membranes is by viscous flow, and in the absence of any concentration polarization, surface fouling, and membrane–solute interaction, the ultrafiltrate flux, ("initial flux," with solute-free water) J'_w, is dependent only on transmembrane pressure (Δp) and intrinsic membrane resistance (R_m),

$$J'_w = \Delta p / R_m \tag{1}$$

R_m is a function of temperature only, and because water viscosity (μ) decreases with temperature, J'_w is inversely proportional to μ.

The observed water flux with wastewaters containing particulates, emulsions, and organic molecules may be considerably lower due to membrane–solute interaction (adsorption in the membrane pores) and/or surface fouling,

$$J_w(t) = \frac{\Delta p}{R_m + R_i + R_f} \tag{2}$$

The additional resistance (R_i) due to membrane–solute interaction (particularly by long-chain, polar, nonionic surfactants) may be due to physical adsorption in the membrane pores and/or surfactant micelle formation inside the pores. The adsorption behavior is dependent on the hydrophilic nature of the membrane. Membrane–surfactant interaction is possible even with zero rejection membranes. R_i is expected to be a function of solute concentration and temperature only. Thus ($R_m + R_i$) is an effective membrane resistance in the presence of an interacting solute.

The fouling layer resistance (R_f) is a function of concentration and type of macromolecules and/or suspended solids, gel-layer compressibility characteristics, transmembrane pressure, channel velocity, and operating time. With oil–detergent–water systems, surface fouling is due to suspended solids, free oil, and emulsified oil droplets. In all cases, R_f [and thus $J_w(t)$] reaches an asymptotic value with operating time. The asymptotic surface fouling layer (gel layer) formed by macromolecular solutes will have a resistance considerably larger than gel layers formed by dispersions containing relatively large particles.

Membrane fouling is a very complex phenomenon, and the rate of fouling is dependent on both the type of feed wastewater and operating conditions (pressure, velocity, and temperature). The fouling mechanism can be compared to scaling problems (*13*) in heat exchangers (heat flux versus water flux). The transient resistance is related to the asymptotic (steady-state) resistance (R_f^*) by

$$R_f = R_f^*(1 - e^{-K_1 t}) \tag{3}$$

R_f is related to the rate of build-up of the fouling layer. The rate of change of fouling layer thickness (y_f) or mass of foulant per unit area (m) can be related by

$$\begin{aligned}\frac{dy_f}{dt} = \frac{dm}{dt} &= \text{rate of deposition on membrane surface} - \text{rate} \\ &\quad \text{of removal from membrane surface} \\ &= K_2 C_b J_w - K_3 \Upsilon y_f \qquad (4)\end{aligned}$$

in which C_b is the concentration of fouling component at bulk liquid conditions and Υ is the shear rate at the surface of the deposited gel layer. Υ is related to the mass transfer coefficient, K_S, where $K_S \propto U^{0.8}$ in the turbulent flow region. Kimura and Nakao (*14*) have used a similar equation for surface fouling (no membrane–solute interaction) under unsteady-state conditions with tubular reverse osmosis modules by replacing the removal term in Eq. (4) by $K_S C_b \ln (C_g/C_b)$, in which C_g is the concentration at which gel formation occurs.

Gutman (*15*) described membrane fouling by relating the rate of removal of deposited material on the membrane surface to the occurrence of random turbulent bursts, and showed that $J_w(t)$, and thus R_f, always approach the steady-state (asymptotic) value J_w (corresponding to R_f^*):

$$\frac{J_w}{J_w'} = \frac{1}{1 + \dfrac{K_f C_b}{A_b U R_m}} \tag{5}$$

in which A_b is the area of the fouling layer removed by each turbulent burst, U is the linear tube velocity, and K_f is the resistance of the fouling layer per unit thickness. From Eq. (5), the percent flux loss $[100(J_w' - J_w)/J_w']$ is much larger with membranes of high J_w' (low R_m), in agreement with experimental results (*2*, *14*, *15*).

RESULTS AND DISCUSSION

Various oil-detergent-water systems were utilized for the ultrafiltration experiments. Preliminary experiments showed that ultrafiltration results with feed suspensions prepared in distilled water were similar to those for suspensions prepared with seawater. Suspensions prepared with river water showed enhanced membrane fouling. The analysis of the river water used in this study is shown in Table 2. Most experiments were conducted at a linear tube velocity of 427 to 457 cm/sec and at a transmembrane pressure less than 1.7 atm.

Nonionic Detergent-Distilled Water System

The membrane-detergent interaction was initially studied with three different nonionic detergents, and Fig. 2 shows that a significant flux drop occurred with the three detergents during a very short period and reached a steady-state value of $100(J_w' - J_w)/J_w' = 76\%$ at less than 1 hr operating time. The effects of transmembrane pressure (0.48 to 2.6 atm) and varia-

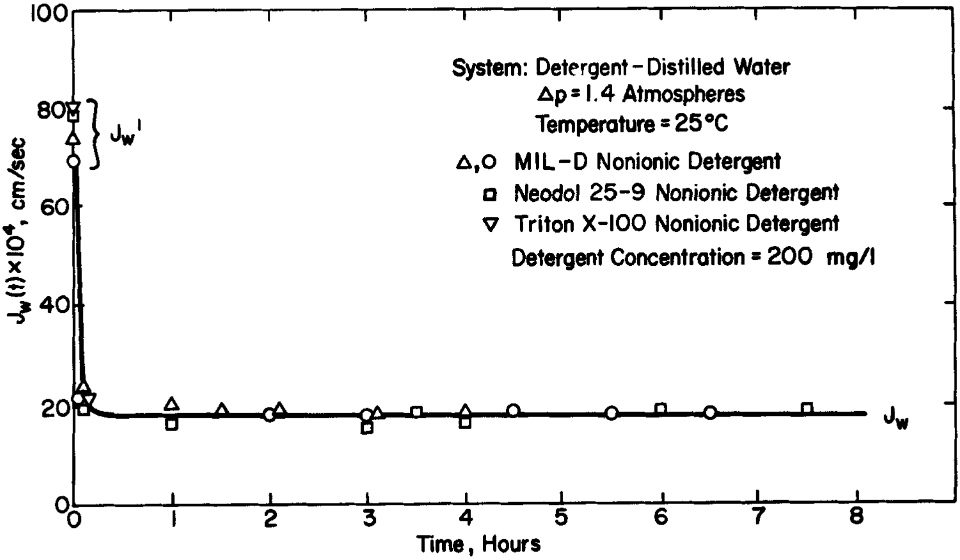

FIG. 2. Variation of water flux behavior with operational time for detergent-distilled water system.

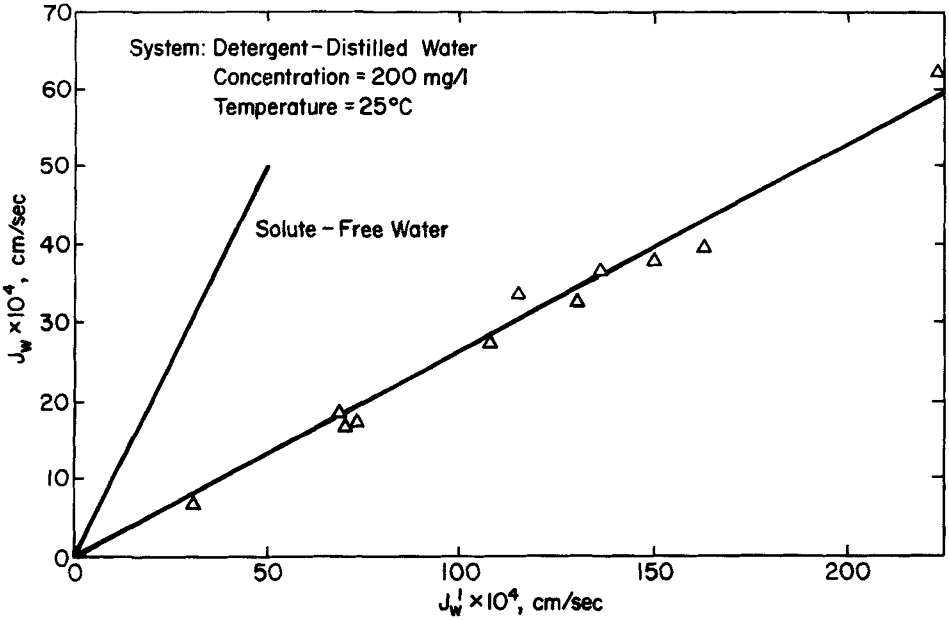

FIG. 3. Dependence of steady-state water flux on initial water flux for detergent-distilled water system.

bility in membrane resistance on steady-state flux (J_w) could be taken into account by the initial water flux, J_w' (solute-free water). Figure 3 shows that J_w (at 200 mg/l detergent concentration) increased linearly with the initial water flux, J_w', indicating a constant flux drop and that the flux drop was not due to surface fouling. The effect of detergent concentration on J_w at a constant Δp (that is, constant J_w') is shown in Fig. 4 for two operating temperatures. J_w approached a constant value above a 100-mg/l detergent concentration for both temperatures. The concentration at which J_w reached a constant value is related to the critical micelle concentration of the surfactant (critical micelle concentration of MIL-D nonionic detergent is approximately 150 mg/l). At 40°C, flux enhancement was due to the lower viscosity of the solution and to the reduction of surfactant adsorption in the pores.

The linear increase of J_w with J_w' (Fig. 3) and the decrease of J_w with detergent concentration (Fig. 4) can be taken into account by an effective

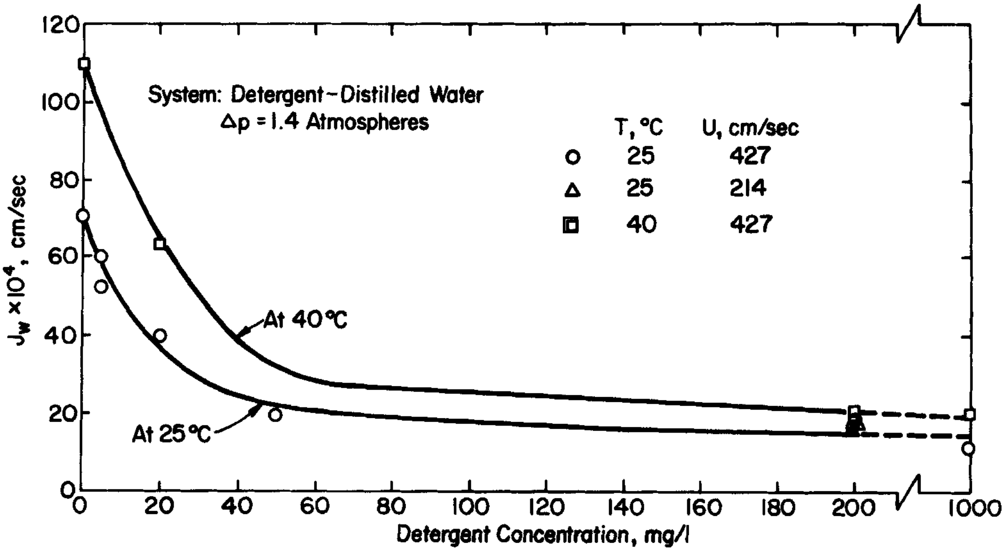

FIG. 4. Effect of detergent concentration on steady-state water flux.

membrane resistance in Eq. (2),

$$J_w = \frac{\Delta p}{R_m + R_i} = \frac{\Delta p}{R_m(e^{KC_b/C_m})} \tag{6}$$

or in terms of $J'_w = \Delta p/R_m$ (Eq. 1),

$$J_w = J'_w/e^{KC_b/C_m} \tag{7}$$

in which the constant K is ≤ 0, C_b is defined for Eq. (4), and C_m is the detergent concentration at which J_w becomes constant. In Eq. (7) the value of C_b/C_m is ≤ 1.0. The assumption of R_f in Eq. (2) to be zero is validated in Fig. 4 where J_w was found to be independent of the tube velocity, U. The membrane–detergent interaction was due to physical adsorption and/or to micelle formation in the membrane pores, and this was validated by the fact that complete flux recovery was obtained with short (less than 1 min) distilled water flushing.

The rejection of detergents by the membrane, defined as

$$100\left(1-\frac{\text{ultrafiltrate concentration}}{\text{feed concentration}}\right)$$

was quite small, which again indicates that the flux drop was not due to surface fouling. The rejections with three nonionic detergents are shown below:

Detergent	Rejection (%)
MIL-D	35
Neodol 25-9	25
Triton X-100	<10

Oil-Water Systems

Oil-water mixtures were studied with both distilled and river water. Most experiments were conducted with 500 mg/l bilge oil (total organic carbon 320 mg/l). This concentration is 5 to 10 times higher than those observed in actual bilge waters. Synthetic based oil was also used in some experiments. Figures 5 and 6 show that the flux drop with river water was considerably faster than that obtained with distilled water. With river water, gel polarization occurred in a short time period because of the cumulative effect of surface fouling due to both oil and fine suspended solids. Flux enhancement (43% increase) was obtained by increasing the temperature from 25 to 40°C (Fig. 5). Figure 7 shows that steady-state J_w with river water became independent of the initial water flux for J_w' values greater than 50×10^{-4} cm/sec. With distilled water, J_w continuously increased with J_w', indicating that complete gel polarization was not achieved. The flux behavior trend shown in Fig. 7 is consistent with Eq. (5). With membranes operating at an initial water flux of 50×10^{-4} cm/sec, the flux drop was approximately 72% with river water and only 23% with distilled water. The oil rejection was found to be independent of operating temperature, water type, and J_w'. Oil rejections were consistently between 98 and 99%, indicating ultrafiltrate oil concentrations to be less than 10 mg/l.

Oil-Detergent-Water Systems

All experiments were conducted with a mixture of 500 mg/l oil and 100 mg/l MIL-D detergent. The total organic carbon of the feed wastes was

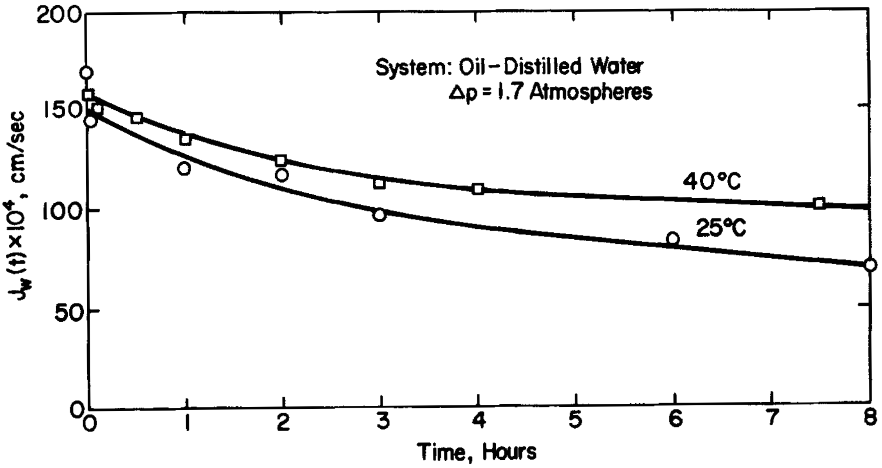

FIG. 5. Time dependence of water flux for oil–distilled water system.

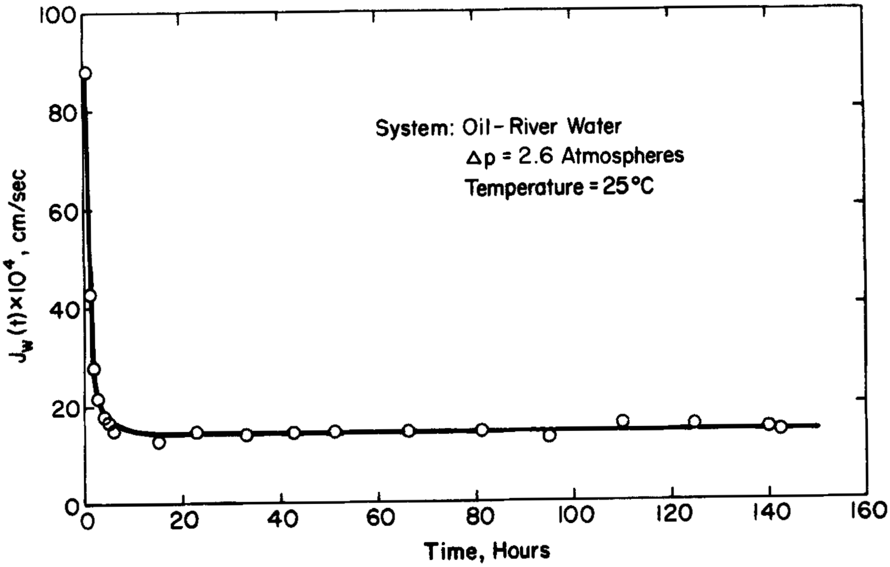

FIG. 6. Time dependence of water flux for oil–river water system.

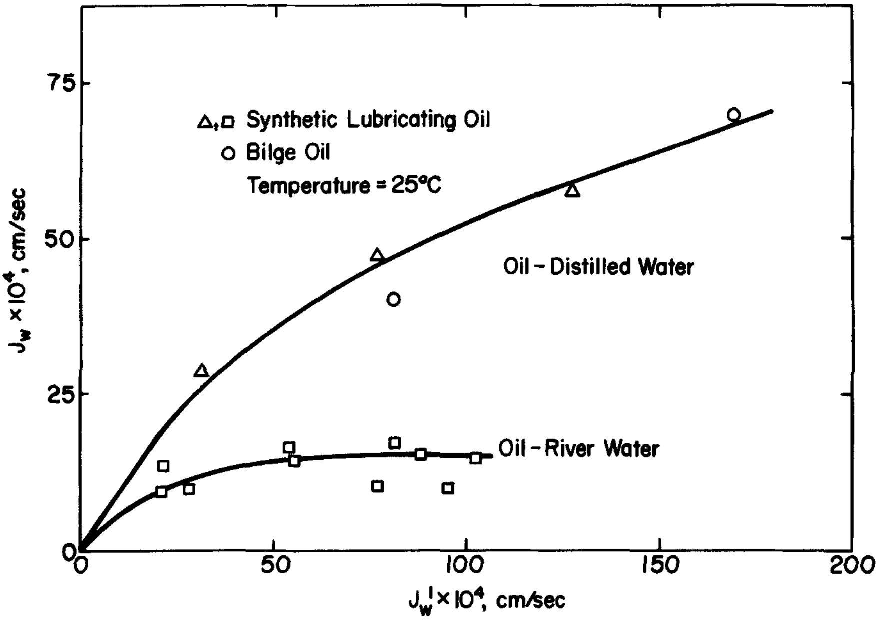

FIG. 7. Dependence of steady-state water flux on initial water flux for oil–water systems.

384 mg/l. Figure 8 shows that the presence of detergent caused considerable flux drops with both distilled and river water due to the presence of oil–detergent–emulsions and free detergent molecules. Figures 9 and 10 show that gelpolarization (maximum R_f) with the distilled water system occurred for membrane operation at >1.4 atm or for membranes of $J_w' \geq 100 \times 10^{-4}$ cm/sec. With river water (Fig. 11) the flux drop $[100(J_w' - J_w)/J_w']$ was quite severe at 25 °C and no flux improvement was observed with membranes of initial water flux greater than 25×10^{-4} cm/sec. At 40 °C a maximum water flux of 21×10^{-4} cm/sec, which corresponds to a flux drop of 59% at 50×10^{-4} cm/sec initial flux, could be achieved with the river water system.

The dependence of the steady-state water flux, J_w, on the initial water flux, J_w', for the oil–detergent–water system is compared with other systems and the results are summarized in Figs. 12 and 13. It is interesting to observe from Fig. 12 that for J_w' less that 100×10^{-4} cm/sec, membrane-detergent interaction (R_i in Eq. 2) rather than surface fouling (R_f in Eq. 2) determined the value of J_w. In the lower initial water flux range,

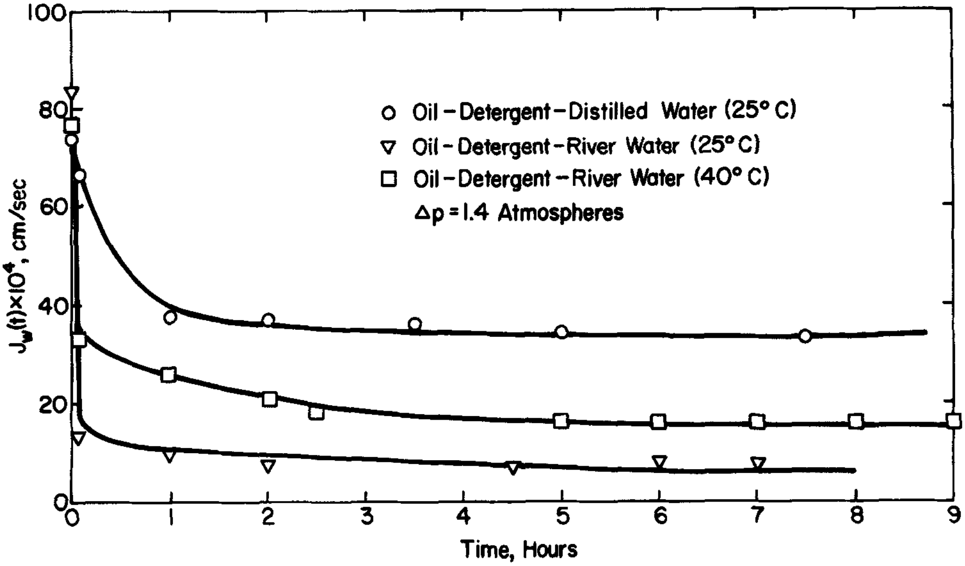

FIG. 8. Time dependence of water flux for oil–detergent–water systems.

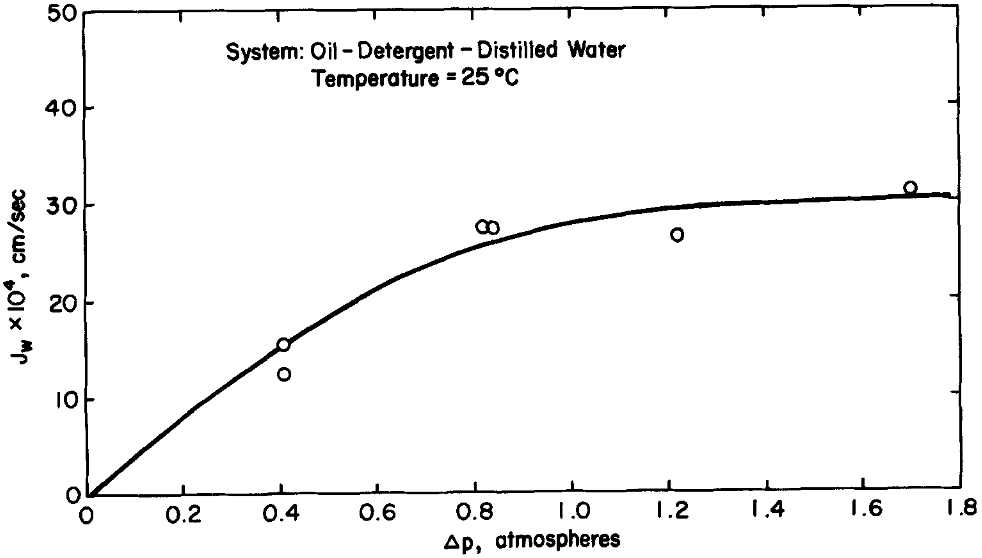

FIG. 9. Transmembrane pressure effect on steady-state water flux for oil–detergent–distilled water system.

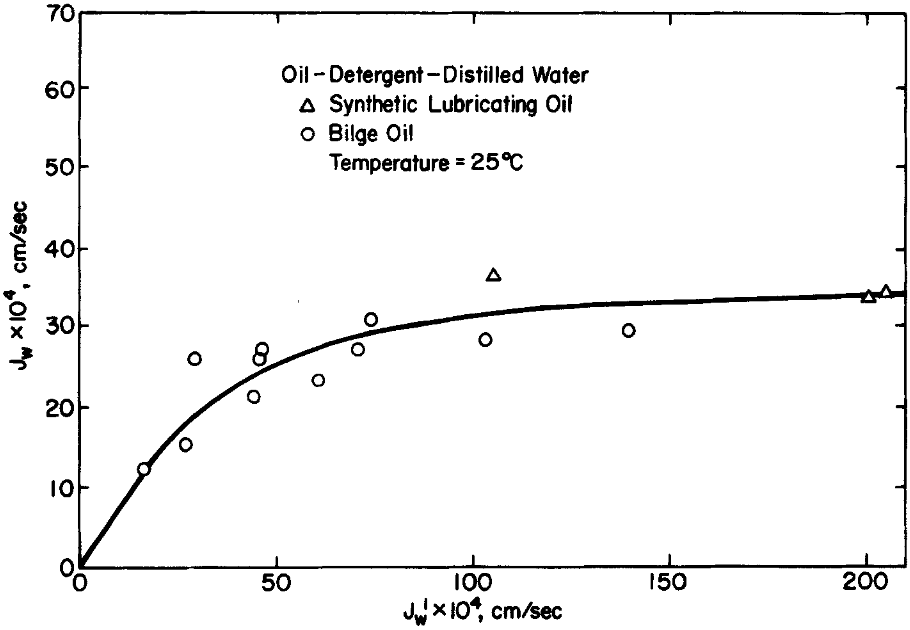

FIG. 10. Dependence of steady-state water flux on initial water flux for oil–detergent–distilled water system.

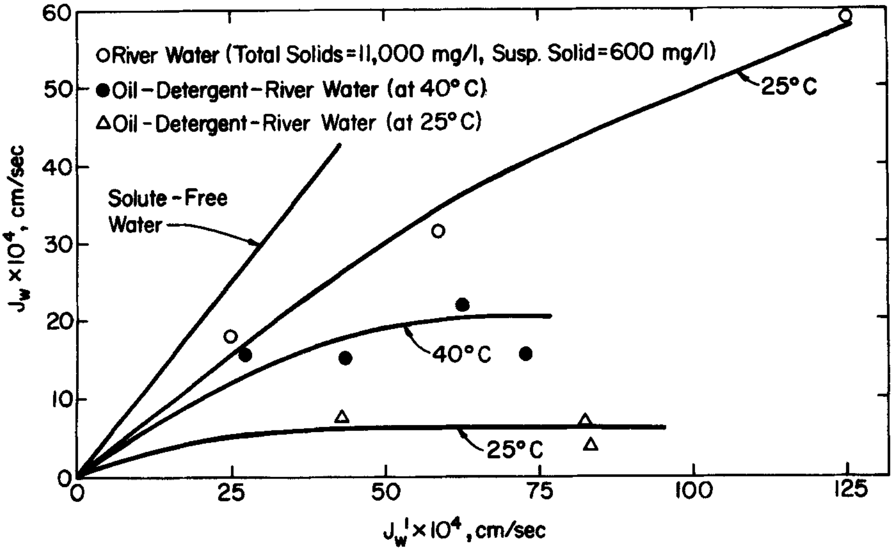

FIG. 11. Dependence of steady-state water flux on initial water flux for oil–detergent–river water system.

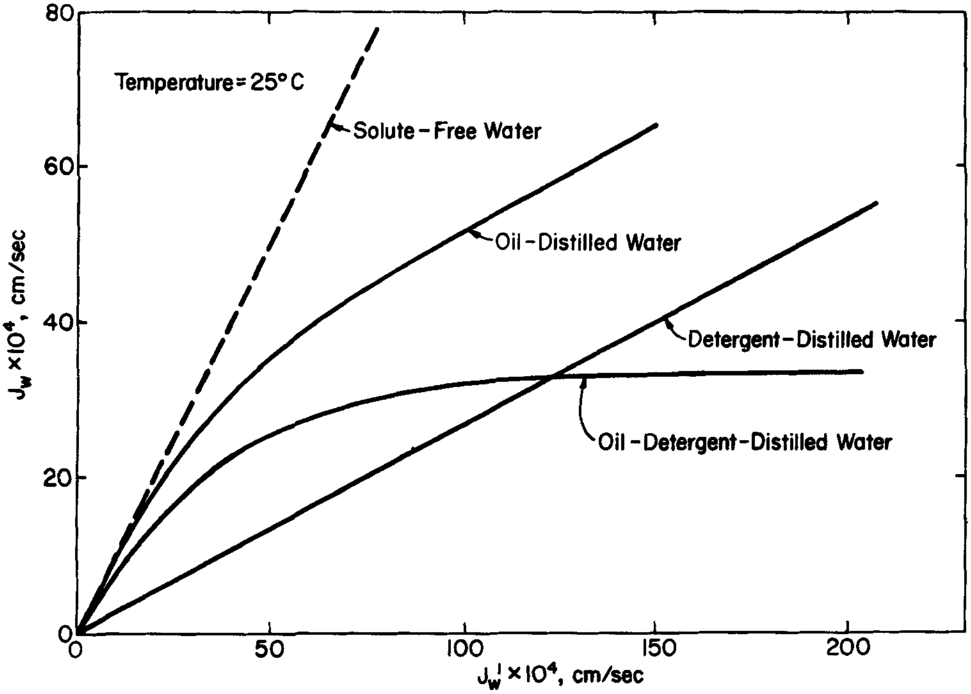

FIG. 12. Comparison of steady-state water fluxes for various distilled water systems.

because the free detergent concentration was lower in the presence of oil (because of emulsification), the water flux was higher than for the detergent–distilled water system. For river water, J_w (Fig. 13) was dictated by both membrane–detergent interaction and surface fouling; the surface fouling could be reduced by operating at higher temperatures. The increase in percent flux loss (with both oil–water and oil–detergent–water systems) with initial water flux was consistent with the surface fouling theory (Eq. 5).

The overall organic carbon rejection was 96 to 98% with the oil–detergent–water system. Surfactant rejection (because of emulsification) was enhanced in the presence of oil. The oil concentration in the ultrafiltrate was always less than 10 mg/l.

Bilge Water System

A series of ultrafiltration experiments was also conducted with actual bilge water; Table 3 shows the analysis of the bilge water. Figure 14 shows

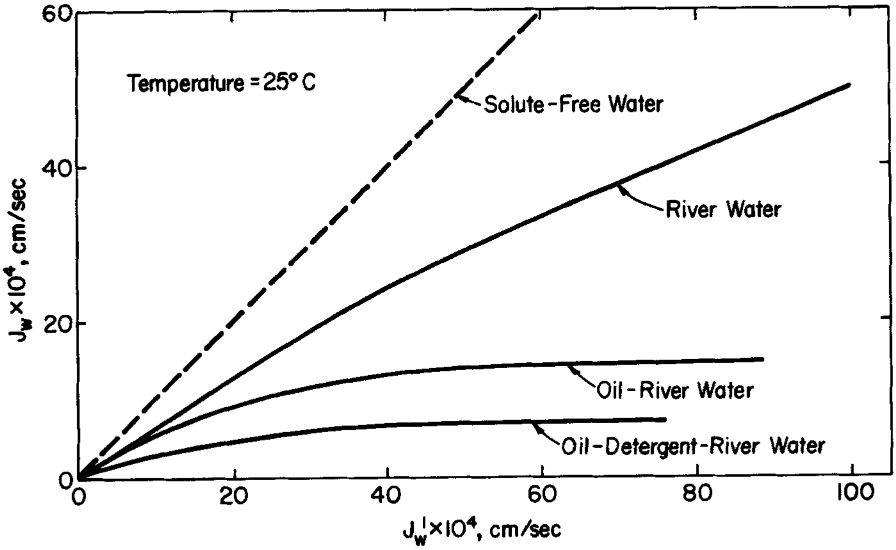

FIG. 13. Comparison of steady-state water fluxes for various river water systems.

TABLE 3

Analysis of Bilge Water

Parameter	Concentration
Total solids	11,135 mg/l
Suspended solids	575 mg/l
Conductivity	6,171 μmho/cm
Total organic carbon	214 mg/l
Oil concentration (approx)	280 mg/l
Iron	9.4 mg/l
pH	8.9

long-term flux behavior (without any membrane cleaning) with the bilge water and bilge water plus added MIL-D detergent. The membrane tube was allowed to depressurize at the end of each 8 hr run for at least 1 hr. In the absence of detergent, excellent flux behavior was obtained; the flux stayed constant after 10 hr and the flux drop was only 13%. An abrupt flux drop occurred (Fig. 14) after detergent addition. Figure 15 shows the effect of detergent concentration on the steady-state water flux. It is

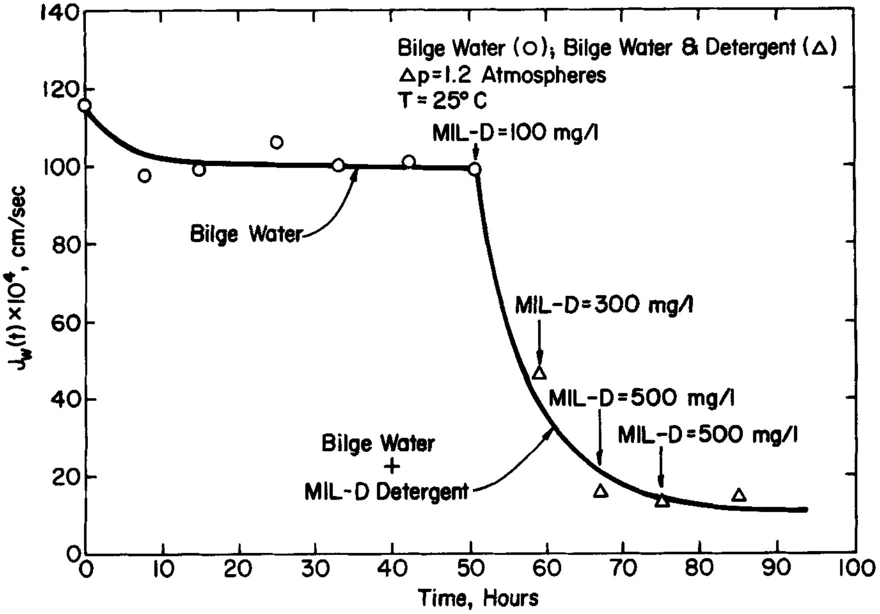

FIG. 14. Time dependence of water flux for bilge water systems.

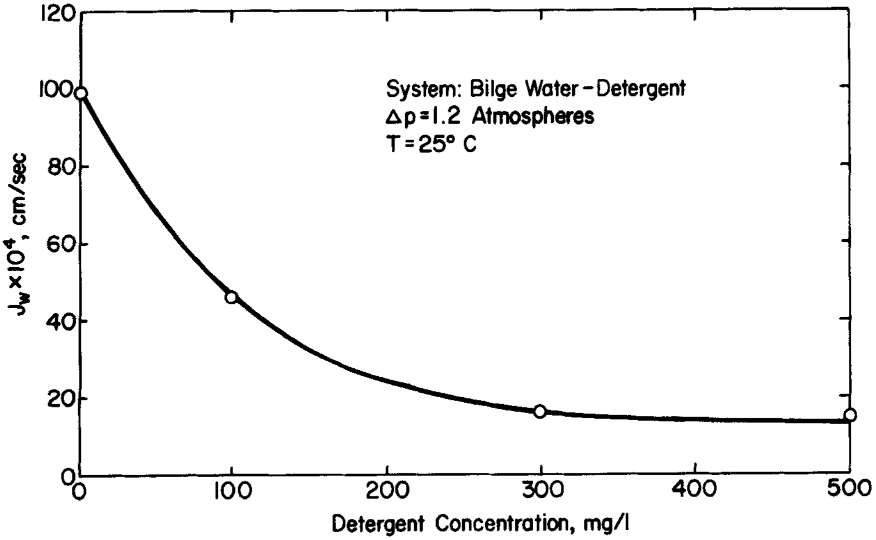

FIG. 15. Steady-state water flux for bilge water in presence of detergent at various concentrations.

interesting to observe that the water flux became constant above a critical concentration. This behavior was similar to that observed in Fig. 4. The higher critical detergent concentration (compared to the detergent–distilled water system) was due to partial oil–detergent emulsification. The ultrafiltrate oil concentration was always less than 8.5 mg/l.

Membrane Water Flux Recovery (Cleaning)

Figure 16 shows the water flux recovery obtained after flushing with tap water at the end of each run. With the detergent system, flux recovery was 100% even with membranes of high initial water flux. With oil systems (with and without detergents), the high surface fouling that occurred with membranes of $J_w > 50 \times 10^{-4}$ cm/sec could not be removed by simple water flushing. For example, with the oil–detergent–distilled water system, the steady-state water flux (Fig. 12) for membranes of initial flux 100 ×

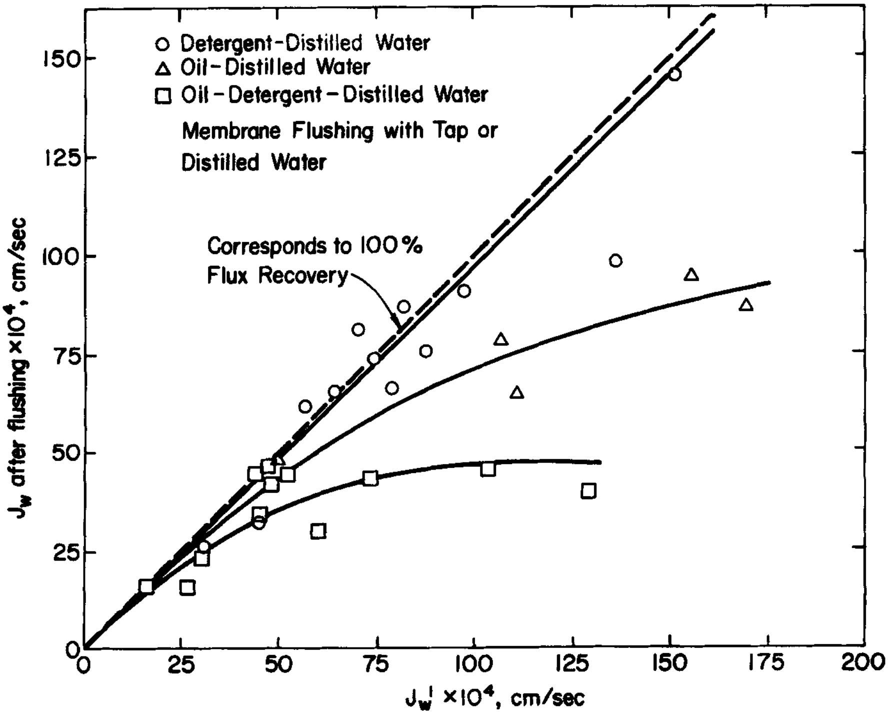

FIG. 16. Water flux recovery with tap water flushing.

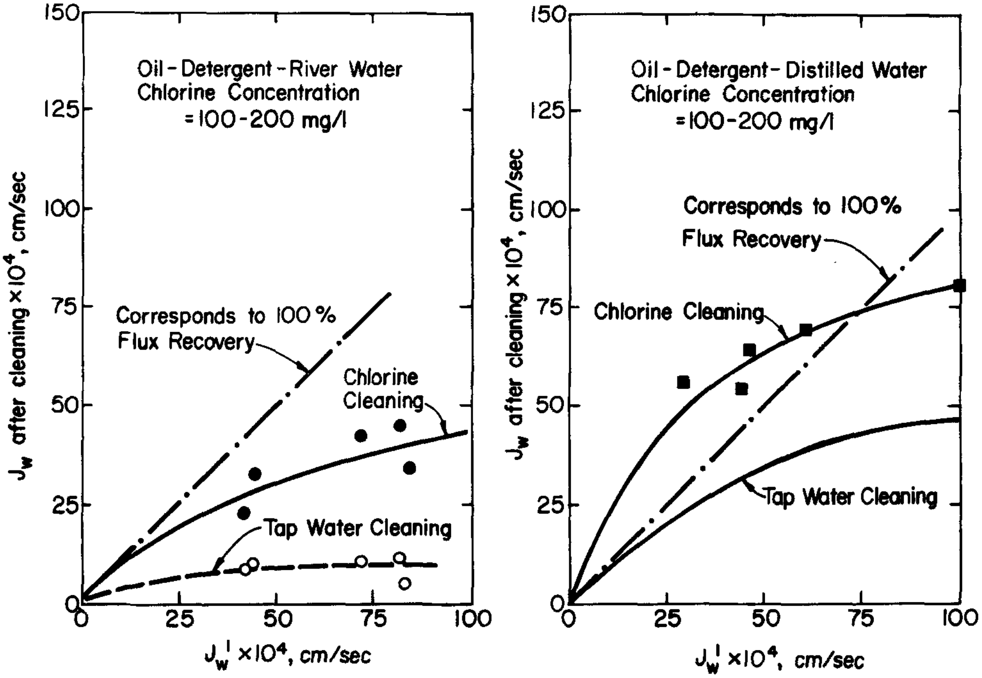

FIG. 17. Water flux recovery with chlorine cleaning.

10^{-4} cm/sec was 32×10^{-4} cm/sec, and tap water flushing recovered the flux to 48×10^{-4} cm/sec (Fig. 16). Chlorine cleaning (Fig. 17, right) provided complete flux recovery for the oil–detergent–distilled water system. Because the oil–detergent–river water system produced extensive surface fouling problems (Fig. 11), particularly with membranes of high initial water flux, the flux recovery even with chlorine cleaning was incomplete (Fig. 17, left). With membranes of high J_w' (100×10^{-4} cm/sec), chlorine cleaning increased the water flux from 8×10^{-4} to 44×10^{-4} cm/sec. It was also observed that flux recovery (by chlorine cleaning) to values greater than 50×10^{-4} cm/sec could not be achieved for membranes exposed to the oil–detergent–river water system.

CONCLUSIONS

Ultrafiltration studies with oil–detergent–water systems indicate that a nonionic surfactant causes substantial water flux drops due to adsorption and/or micelle formation of detergent molecules in the membrane pores. The water flux approaches a constant value above a critical detergent concentration. For all systems the steady-state water flux is a function of the initial water flux. Membrane surface fouling due to stable oil–detergent emulsions and fine suspended solids can be minimized by membrane operation at low pressure or for membranes of initial water flux less than 50×10^{-4} cm/sec. The oil–detergent–river water system causes maximum surface fouling at an operating temperature of 25°C. Flux drop can be minimized by operating at higher temperature (40°C) and/or with membrane depressurization at intervals of 8 to 10 hr for at least 1 hr. Depending on the system, partial to complete flux recovery can be obtained with chlorine cleaning. Membrane cleaning with chlorine concentrations greater than 200 mg/l, however, is found to have an adverse effect on the membrane intrinsic properties.

At 25°C the steady-state water flux values (in cm/sec $\times 10^4$) for a membrane with an initial water flux of 100×10^{-4} cm/sec and with distilled water systems (oil 500 mg/l, detergent 100 mg/l), are 27 with detergent only, 52 with oil only, and 32 with oil plus detergent. With river water systems the steady-state water flux values are 15 with oil only and 8 for oil plus detergent with membranes of initial flux $>50 \times 10^{-4}$ cm/sec. The water flux with the oil–detergent–river water system can be increased to 21×10^{-4} cm/sec by operating at 40°C. The water flux behavior is explained in terms of membrane resistance increase due to detergent interaction and to surface fouling in the presence of emulsions and suspended solids. Excellent oil rejections are obtained in all cases: even with oil–detergent systems, an ultrafiltrate oil concentration of less than 10 mg/l can be achieved.

Membrane area requirements are dependent on the type of oil–water mixture to be processed; that is, whether the mixture contains distilled, river, or seawater. Processing oil–river water mixtures requires approximately threefold more membrane area than oil–distilled water or oil–seawater mixtures.

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Received by editor November 6, 1978