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Membrane Fouling during Constant Flux Crossflow Microfiltration of Dilute Suspensions of Active Dry Yeast

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ABSTRACT

Fouling of microporous and ultrafiltration membranes during crossflow microfiltration of rehydrated active dry yeast (ADY) was investigated using measurements of the transmembrane pressure as a function of time at constant flux. By centrifuging the suspensions and comparing the increase in transmembrane pressure produced by both the original suspensions and the supernatant alone, it was determined that this increase was mainly caused by soluble components in the supernatant. This finding is consistent with previous observations that considerable quantities of intracellular matter leak from cells of ADY when they are rehydrated. The increase in transmembrane pressure caused by the supernatant alone was found to be independent of tangential flow rate, suggesting that the underlying mechanism was one of internal membrane fouling. Fouling was found to be enhanced by increasing the transmembrane flux and reducing the membrane pore size. Membrane fouling by the supernatant was modeled as a process involving simultaneous deposition and removal of foulant, onto and from, the walls of membrane pores. In contrast to the standard blocking model, but in agreement with experimental observations, the new model does not predict that the transmembrane pressure will increase indefinitely, but will reach a constant value which will depend on the flux and the pore diameter.

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INTRODUCTION

Under conditions of constant transmembrane pressure and crossflow velocity, the filtrate flux in continuous crossflow microfiltration declines to a steady-state value which can be as much as two orders of magnitude lower than the initial or "pure-water" value (1). The severity of this decline is a major impediment to the practical application of crossflow microfiltration. Basic filtration theory suggests that two phenomena are responsible for this decline in flux. Using the equation (2)

$$J = \frac{\Delta P}{\mu(R_M + \alpha M)} \quad (1)$$

where J is the flux (i.e., the filtrate flow rate per unit membrane area), ΔP is the transmembrane pressure, μ is the filtrate viscosity, R_M is the membrane resistance, α is the specific cake resistance, and M is the cake mass per unit membrane area, it is clear that the decline in flux is due to a combination of cake formation (increasing M) and membrane fouling (increasing R_M). The latter phenomenon would typically be a result of the blocking of membrane pores by the deposition or adsorption of suspension components (both particulate and soluble) onto the membrane surface or the walls of the membrane pores.

There is considerable evidence for cake formation in crossflow microfiltration (3-8). As a consequence, numerous models, both empirical and theoretical, have been developed which attempt to explain the mechanism of cake formation and its influence on the transmembrane flux (9-12). In contrast, while there is now a significant body of data which suggests that membrane fouling is a common phenomenon, especially during microfiltration of microbial suspensions where medium components can interact with the membrane (5, 13-17), there are, as yet, very few models which give even a semiempirical description of the effect of membrane fouling on the flux (1). This deficiency in current research is attributable to three main factors. First, experimentation involving the adsorption or deposition of particles or molecules onto synthetic membranes is difficult since formidable problems arise with regard to fully characterizing the membranes and in obtaining reproducible data. Second, even if reliable data are obtained, interpretation is difficult since theories of the interaction of particles and macromolecules with surfaces are not at an advanced stage of development. Finally, in situations in which both cake formation and membrane fouling are occurring, there is, at present, no simple methodology for determining the separate contributions of both of these phenomena to the decline in filter performance.

The experiments reported in this paper are an attempt to develop a methodology for the study of membrane fouling in crossflow microfiltration of microbial suspensions. Rehydrated active dry yeast (ADY) is a convenient microbial "particle" for microfiltration experiments. It has been used previously by a number of workers (18-20) and is employed in this study. ADY is obtained when *Saccharomyces cerevisiae* is washed and pressed to a solids content of 30-32%, and then dried under controlled conditions to 92% solids. A factor considered in only one previous filtration study (20) is that, on resuspension, as much as 30% of the intracellular mass of ADY is lost due to leakage through the cell wall (21). This effect is more pronounced when rehydration is carried out at low temperatures, where the cell wall is slow to regain its normal fully-hydrated structure. It was found that at 4.5°C, approximately 12% of the protein, 20% of the phosphorus, 40% of the carbohydrate, and 50-60% of the inorganics leaked from the cells into distilled water. These losses were halved at 43°C. Typical protein losses ranged from 54 mg/g dry cells at 4.5°C to 26 mg/g at 43°C. This behavior was found to be typical of a range of commercial ADY samples. Much of the material which leaks from the cells during rehydration, particularly the proteins, can be expected to interact in some way with synthetic membranes during microfiltration. Therefore, the study of the filtration performance of suspensions of rehydrated ADY should provide a convenient and useful starting point for assessing the effects of membrane fouling during microfiltration of biological suspensions.

MATERIALS AND METHODS

Suspensions

ADY (Distillers Company Ltd., Scotland) was resuspended in deionized water at 20°C. This yeast is supplied in pellet form and was, therefore, thoroughly mixed for 1 hour prior to filtration. Suspensions produced in this way are referred to as "unwashed" yeast throughout this paper. Unwashed yeast suspensions were also prepared by suspending the yeast in isotonic solutions, i.e., deionized water into which was dissolved 9 g/L NaCl and 1 g/L glucose.

"Washed" yeast suspensions were produced by centrifuging the unwashed yeast in a Sorval centrifuge (Dupont, UK) at 5000 rpm for 10 minutes. The supernatant was decanted and the yeast pellet resuspended in deionized water. Microscopic examination at 400 \times magnification showed that the supernatant was free of particulate matter.

Microfiltration

Microfiltration was performed at 20°C in a Pellicon (Millipore, UK) flat sheet module. Suspensions were pumped using a Bio-2000 (Bio-Flo Ltd., Scotland) peristaltic pump which has a maximum operating pressure of 200 kPa. Experiments were carried out in total recycle mode, i.e., both the retentate and filtrate were returned to the feed reservoir. The reservoir volume (including module hold-up) was 1 L in all runs. The experiments were performed under conditions of *constant transmembrane flux*, in contrast to the more conventional method of employing constant pressure operation. The constant flux mode is a convenient method for controlling the flow conditions in the membrane pores, thus allowing data to be more easily interpreted in terms of membrane properties. It should be noted that constant flux operation has also been used in ultrafiltration where it was desired to keep the protein concentration at the membrane surface at a constant value (22).

Instrumentation

The feed flow rate was determined from the pump calibration. As the membrane performance deteriorated during a run, the flux was kept constant by continually adjusting the retentate valve in order to keep the reading on the filtrate rotameter at a constant value. The average transmembrane pressure was determined from the feed, permeate, and retentate pressures which were measured using Bourdon gauges on the Pellicon module. All pressures reported in this paper represent the average of three runs.

Membranes

Three membranes were used in this study. A list of the relevant membrane properties is provided in Table 1. Membranes were cleaned with solutions of sodium hypochlorite or Neutracon detergent (Decon Laboratories Ltd., UK). Experimental runs were begun only if the "pure-water" resistance of the membrane was within 10% of the value given in Table 1. This value was determined by measuring the flux of pure water at a known pressure and using the expression $J = \Delta P / \mu R_M$, where the symbols are defined as before.

RESULTS AND DISCUSSION

All experiments reported below involve dilute suspensions. Accurate data were difficult to obtain at high concentrations due to the rapid rise

TABLE I
Membrane Properties

Material	Pore diameter/cutoff	$R_M \times 10^{-11} (\text{m}^{-1})$
PVDF	0.22 μm	4.0
PVDF	0.45 μm	3.2
Polysulfone	$3 \times 10^5 \text{ Da}$	5.6

in transmembrane pressure. However, behavior qualitatively similar to that reported in this paper has been found at concentrations up to 20 g/L (dry weight) of unwashed yeast.

Figure 1 is a plot of transmembrane pressure versus time for unwashed yeast, washed yeast, and the supernatant. It is apparent that microfiltration of the unwashed yeast leads to a much greater rise in pressure than the washed yeast. An obvious explanation for this phenomenon is that intracellular matter which has leaked from the cells during rehydration

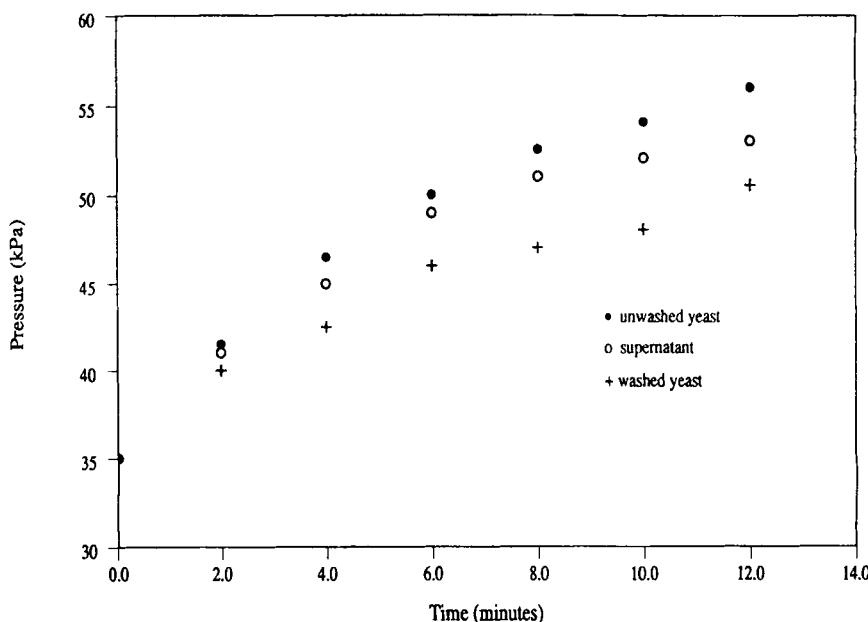


FIG. 1 Transmembrane pressure versus time for unwashed yeast, washed yeast, and supernatant. Yeast concentration: 2 g/L (dry weight). Flux: $10.2 \times 10^{-5} \text{ m/s}$. Membrane: 0.45 μm .

has fouled the membrane. This leaked matter is present in the supernatant which is seen to cause a significant rise in transmembrane pressure when filtered on its own. An interesting feature of the data is that the separate effects of the washed cells and the supernatant are nonadditive, i.e., the increase in transmembrane pressure due to the unwashed yeast is less than the sum of the separate effects of the washed yeast and the supernatant. An explanation for this behavior is that cells which deposit on the membrane surface prevent soluble foulant from gaining access to its pores in a similar manner to a filter aid precoat. Comparable behavior has been observed during the separation of enzymes from cell debris where a number of authors have found that the debris significantly reduces the transmission of enzyme through the membrane, leading to reduced product recovery (23, 24).

Leakage of intracellular matter during rehydration should be suppressed by suspending the cells in an isotonic solution rather than in the deionized water used to obtain the data in Fig. 1. However, no reproducible difference was found between data for "isotonic" supernatant and data for "nonisotonic" supernatant.

As mentioned in the Introduction, lowering the rehydration temperature tends to increase the leakage of intracellular matter from the yeast cells [21]. Experiments were performed in which the cells were rehydrated at different temperatures ranging from 7 to 40°C, and then microfiltered at 20°C as before. No reproducible reduction in membrane fouling was obtained by resuspending the cells at higher temperatures. The lack of any significant salt or temperature effect demonstrates that membrane fouling by leaked intracellular matter is an inherent problem with suspensions of reconstituted ADY. It appears to be significant even when the cells are rehydrated under conditions that minimize leakage.

In order to investigate the mechanism of membrane fouling by the supernatant, a series of experiments were performed in which the effects of tangential flow rate, transmembrane flux, and membrane properties on the transmembrane pressure were investigated. It was found that the pressure rise obtained with the supernatant alone is independent of tangential flow rate, suggesting that fouling is associated with deposition of material in the pore interior and not the blocking of pores by deposition at the surface of the membrane. A similar conclusion was reached by Bowen and Quan (25) when they investigated the fouling of 0.45 μm membranes by proteins.

Figure 2 gives the results of experiments in which the *flux* was varied at fixed tangential flow rate during microfiltration of supernatant. The data indicate that the rate at which $\Delta P/\Delta P_0$ increases with time is enhanced at higher fluxes, *J*, where ΔP_0 is the initial transmembrane pressure and ΔP

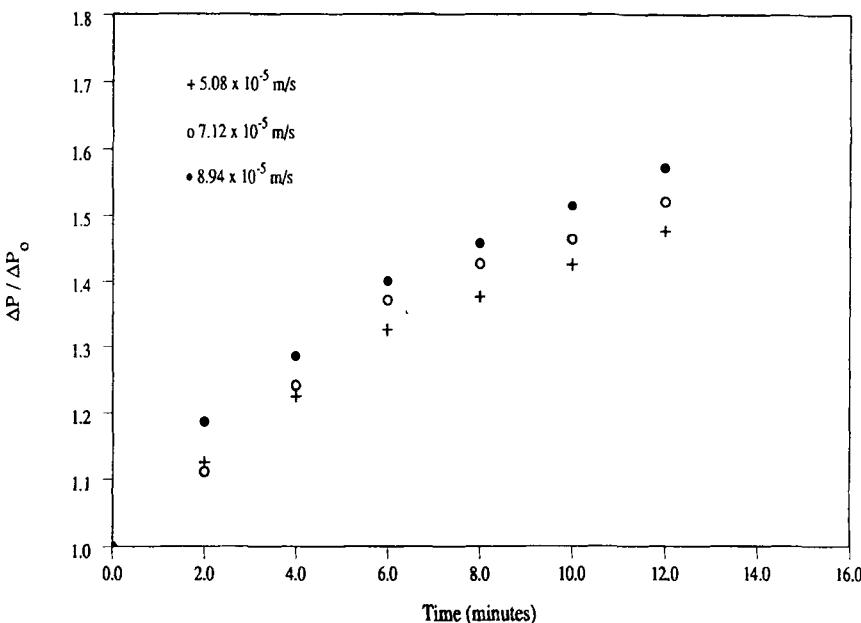


FIG. 2 Effect of flux on transmembrane pressure during microfiltration of supernatant.
Yeast concentration: 2 g/L. Membrane: 0.45 μ m.

is the pressure at time t . Similar behavior was observed with all three membranes.

Comparison with the Standard Blocking Model

The apparent lack of a dependence of the fouling process on the tangential flow rate suggests that the basic mechanism is one of internal pore plugging. A possible explanation for enhanced fouling at higher fluxes is that more fluid passes through the membrane and therefore has more opportunity to block the membrane pores. This is the explanation of the standard blocking model (26) which predicts (Appendix A)

$$\left[\frac{\Delta P_0}{\Delta P} \right]^{1/2} = 1 - \frac{bJ}{\epsilon_0 L} t \quad (2)$$

where b is a constant, J is the flux, L is the pore length, and ϵ_0 is the initial or "unfouled" porosity of the membrane. Equation (2) is qualitatively correct in predicting the increase in transmembrane pressure to be flux-dependent. However, when the data of Fig. 2 are replotted, as is

done in Figure 3, for direct comparison with Eq. (2), it is clear that the standard blocking model is incorrect in predicting that the quantity $(\Delta P_0 / \Delta P)^{1/2}$ should decrease *linearly* with time.

A further limitation of the standard model is that it does not include an explicit pore-size dependence for $\Delta P_0 / \Delta P$. The pressure rise obtained during microfiltration of the supernatant with each of the three membranes used in this study is shown in Fig. 4. While recognizing the fact that the (asymmetric) ultrafiltration membrane is different in material and construction from the (symmetric) microporous membranes, one can make the general observation that the smaller the pore size, the greater the pressure rise. In the next section, a simple model is proposed which offers a rationalization for this behavior.

A Deposition Model for Membrane Fouling

A possible strategy for modeling the fouling process is to assume that it is the net result of simultaneous deposition and removal of foulant, onto and from, the walls of membrane pores. This approach is frequently used to model the fouling of heat transfer surfaces by both biological and non-

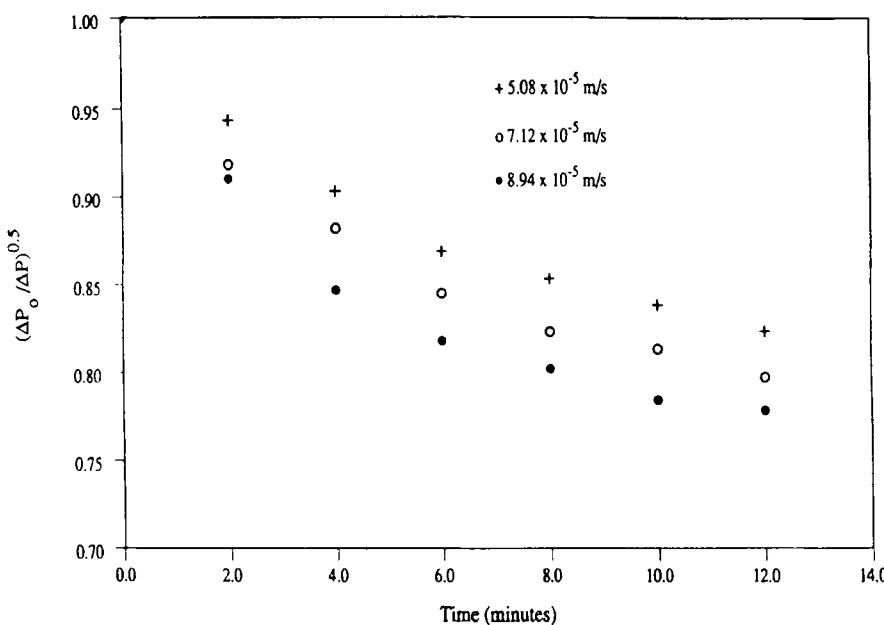


FIG. 3 Standard blocking model plot of data in Fig. 2.

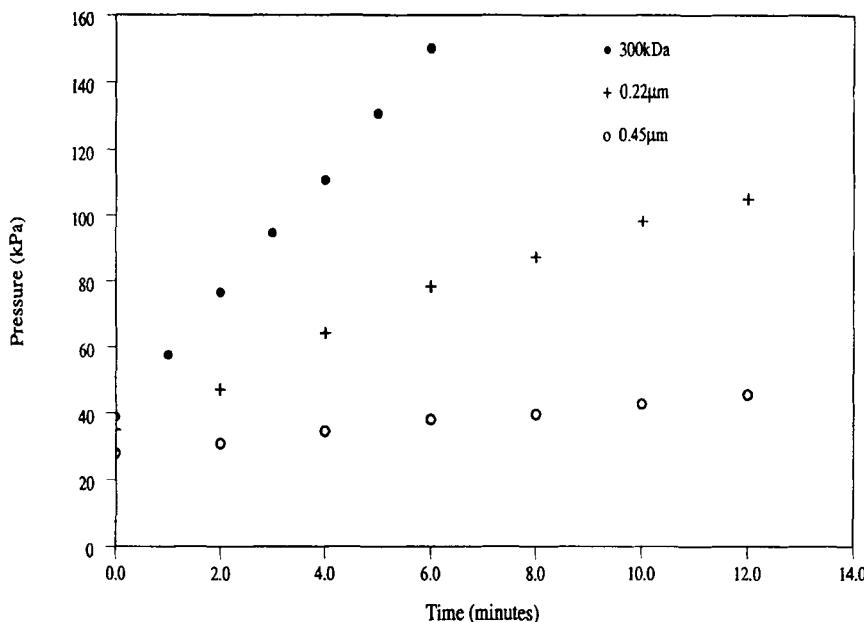


FIG. 4 Effect of membrane type on the transmembrane pressure during microfiltration of supernatant. Yeast concentration: 2 g/L. Flux: 7.12×10^{-5} m/s.

biological materials (27). It has also been used to model cake formation on the *surface* of microfiltration membranes (28). The basic equation for the fouling process can be written in the form

$$dh/dt = v_D - kh \quad (3)$$

where h is the fouling layer thickness at time t , v_D is the rate of increase of h due to material deposition, and k is a constant. The first-order dependence of the foulant removal rate on the foulant layer thickness implies that foulant removal is essentially a random process.

For a constant deposition rate, v_D , Eq. (3) predicts that the deposit layer thickness increases until a dynamic steady-state between particle deposition and removal is reached. This is in contrast to Eq. (2) which allows the deposit layer thickness to increase until the pore is completely filled by fouling material.

Assuming laminar flow through the membrane, Eq. (3) leads to the following expression for the transmembrane pressure as a function of time (Appendix B):

$$\left[\frac{\Delta P_0}{\Delta P} \right]^{1/4} = 1 - \frac{v_D}{kR_0} [1 - e^{-kt}] \quad (4)$$

where R_0 is the unfouled pore radius. Equation (4) is in qualitative agreement with the observed dependence of the pressure rise on membrane pore size. However, a drawback of this model is that, in contrast to the standard blocking model, it does not explicitly predict the pressure rise to be flux-dependent. This deficiency may be corrected by assuming either or both v_D and k to be functions of J .

In order to test the model in a more quantitative way, the data of Fig. 4 were fitted to Eq. (4) by least-squares regression. The results of this fitting procedure are given in Fig. 5. The agreement between model and experiment is satisfactory. In addition, Table 2 presents the best-fit values of v_D and k evaluated at different fluxes for each of the two PVDF membranes. The data for the ultrafiltration membrane were not considered, as it has a different structure and as polysulfone membranes typically have a greater tendency to be fouled by proteins (29). Thus, direct comparison with the PVDF membranes is not very meaningful. It appears from these data that both v_D and k are functions of pore shear rate, since both

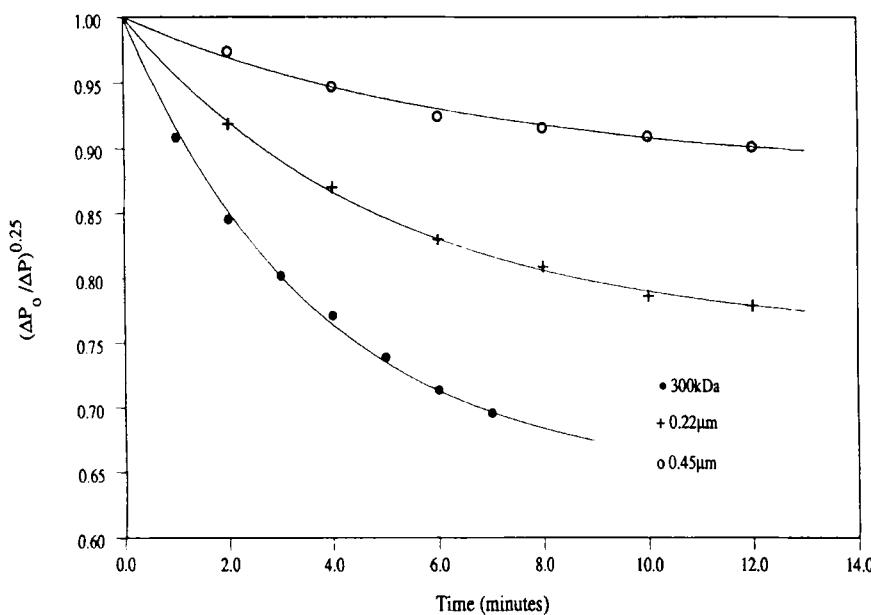


FIG. 5 Fit of Eq. (4) to data in Fig. 4.

TABLE 2
Fouling Model Parameters as a Function of Membrane Pore
Diameter and Transmembrane Flux

$J \times 10^5$ (m/s)	$v_D \times 10^{11}$ (m/s)		$k \times 10^3$ (s ⁻¹)	
	0.45 μm	0.22 μm	0.45 μm	0.22 μm
5.08	6.07	7.57	2.39	2.90
7.12	6.59	8.93	2.45	3.34
8.94	8.17	10.91	3.14	4.00

of these parameters increase with increasing flux and decreasing pore size. Strictly speaking, therefore, their shear dependence should be incorporated at an earlier stage of the model, i.e., a more complete model would include the dependence of v_D and k on the deposit layer thickness, h . It is questionable whether such an increase in complexity is warranted, given the semiempirical nature of the model.

Regression of the data in Table 2 gives

$$v_D = 7.53 \times 10^{-12} \left(\frac{J}{d_0} \right)^{0.44} \quad (5)$$

$$k = 3.58 \times 10^{-4} \left(\frac{J}{d_0} \right)^{0.39} \quad (6)$$

where d_0 is the unfouled pore diameter and all units are as given in Table 2. From these equations it appears that the experimentally observed dependence of $\Delta P/\Delta P_0$ on the flux (for a given membrane) is due to the fact that the foulant deposition rate has a slightly greater dependence on shear rate than the foulant removal constant.

The shear-dependence of v_D is in agreement with the data of Bowen and Quan (25) who found that protein deposition onto the pores of a 0.45- μm membrane was enhanced by increasing the flux. They attributed this observation to a process termed "shear-induced" deposition, in which the shear forces in the pore altered the protein structure in such a way as to overcome the repulsive forces between a depositing molecule and previously-deposited or adsorbed molecules. The driving force for migration of a molecule from the bulk solution to the pore walls was not explained. The shear dependence of k suggests that higher shears seem to promote the sweeping of deposited material from the pore wall.

When both cells and foulant are present, the fouling process becomes more complex and application of the model becomes more difficult. As

seen in Fig. 1, a deposit of cells on the surface of the membrane seems to reduce the access of foulant to the pores of the membranes, i.e., the foulant concentration in the pores is reduced by the presence of cells on the surface of the membrane. Thus, the deposition rate, v_D , may also be a function of *cell* concentration.

Another issue which neither the standard blocking model nor the new model presented here addresses is that of irreversibility. It has been found throughout this study that the membrane cannot be returned to its initial state by simply flushing with pure water. Cleaning agents must be used. This is not in agreement with Eq. (3) which suggests that if $v_D \rightarrow 0$, as it would during flushing with water, the deposit layer thickness should reduce to zero. It is likely that incorporating the phenomenon of irreversibility would require more information as to the precise physical phenomena involved in the fouling process. While Eq. (3) could be considered a transport model, irreversibility probably involves adhesion or adsorption processes. Additional research, probably with more well-defined systems, is needed before these issues can be addressed.

CONCLUSIONS

The work presented in this paper has a number of implications. A methodology has been established in which the contribution of noncellular suspension components to the decline in filter performance can be assessed. While suspensions of ADY were used here, the same methodology is equally applicable to fermented broths of yeast or bacteria (13).

The work has also shown that suspensions of rehydrated ADY are not ideal systems for studying cake formation unless it is recognized that the material which leaks from the cells can interact with the membrane and increase its resistance. However, ADY suspensions should prove to be convenient systems for investigating simultaneous cake formation and membrane fouling.

It is apparent that the basic mechanism of membrane fouling is a subject worthy of further study. The data presented suggest that internal plugging of membrane pores will be affected by the membrane material, the pore size of the membrane, and the transmembrane flux. In addition, deposition of particulate matter on the membrane surface appears to reduce access of foulant material to the pore interior.

The modeling work presented here has demonstrated that simplistic approaches typified by the standard blocking model are not particularly useful in explaining fouling data. Greater emphasis must be placed on microscopic analyses from which gross behavior may be predicted. The

deposition model developed in this paper is a very simple example of such an analysis.

APPENDIX A. THE STANDARD BLOCKING MODEL APPLIED TO CONSTANT FLUX MICROFILTRATION

The standard blocking model makes the assumption that in a small time interval, dt , the change in pore volume is proportional to the volume of filtrate which has passed through the pore, i.e.,

$$-2\pi RNLdR = bdV \quad (A1)$$

where R is the effective pore radius, N is the number of pores, L is the pore length, dR is the change in pore radius in time dt , b is a constant, and dV is the volume of filtrate passed in time dt .

A filtrate balance can be written

$$dV = JAdt \quad (A2)$$

where J is the flux and A is the membrane area. Substituting into Eq. (A1) and integrating for constant J yields

$$\pi NL(R_0^2 - R^2) = bAJt \quad (A3)$$

where R_0 is the initial or unfouled pore radius.

Assuming that the flow in the pores is laminar, then

$$J = \frac{\epsilon_0 R_0^2 \Delta P_0}{8\mu L} \quad (A4)$$

and

$$J = \frac{\epsilon R^2 \Delta P}{8\mu L} \quad (A5)$$

where ϵ_0 is the initial or unfouled membrane porosity, ϵ is the membrane porosity at time t , ΔP_0 is the initial pressure drop across the membrane, and ΔP is the pressure drop at time t .

The porosities ϵ_0 and ϵ are related to the radii R_0 and R by the expressions

$$\epsilon_0 = \frac{N\pi R_0^2}{A} \quad (A6)$$

and

$$\epsilon = \frac{N\pi R^2}{A} \quad (A7)$$

Combining Eqs. (A4) to (A7) and substituting into Eq. (A3) gives

$$\left(\frac{\Delta P_0}{\Delta P}\right)^{1/2} = 1 - \frac{bJ}{\epsilon_0 L} t \quad (A8)$$

APPENDIX B. A DEPOSITION MODEL FOR MEMBRANE FOULING

In this model it is assumed that the net accumulation of matter on the pore walls can be expressed as the difference between deposition and removal, i.e.,

$$dh/dt = v_D - kh \quad (B1)$$

where h is the fouling layer thickness at time t , v_D is the rate of increase of the deposit layer thickness due to the deposition of fouling matter, and k is the foulant removal constant. Assuming v_D and k to be independent of h , Eq. (B1) can be integrated to give

$$h = \frac{v_D}{k} (1 - e^{-kt}) \quad (B2)$$

Now, h is related to R_0 and R through the expression

$$h = R_0 - R \quad (B3)$$

and therefore

$$\frac{R}{R_0} = 1 - \frac{v_D}{kR_0} (1 - e^{-kt}) \quad (B4)$$

Combining this expression with Eqs. (A4) to (A7) gives

$$\left(\frac{\Delta P_0}{\Delta P}\right)^{1/4} = 1 - \frac{v_D}{kR_0} (1 - e^{-kt}) \quad (B5)$$

NOMENCLATURE

A	membrane area
b	constant in standard blocking model
d_0	unfouled pore diameter
h	foulant layer thickness
J	transmembrane flux
k	foulant removal constant
L	pore length

M	cake mass per unit membrane area
N	number of pores
R_M	membrane resistance
R_0	unfouled pore radius
R	pore radius
t	time
v_D	foulant deposition rate
V	filtrate volume
α	specific cake resistance
ϵ	membrane porosity
ϵ_0	unfouled membrane porosity
μ	filtrate viscosity
ΔP	transmembrane pressure
ΔP_0	initial transmembrane pressure

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