

ABSTRACT

Ultrafiltration is a potentially viable method of removing finely dispersed flexographic pigments from the deinking water loop. This work examines the effects of surface-active materials on ultrafiltration efficiency. A logarithmic relationship between permeate flux and pigment concentration was demonstrated at ink concentrations above 0.4%, permeation rates becoming independent of ink content at lower concentrations. The pressure independent ultrafiltration permeation rates observed at more highly concentrated ink dispersions were shown to be limited by mass-transfer effects, as predicted by the concentration-polarization model. Permeate flux at low ink concentration was limited by membrane fouling, which occurs because dilute ink dispersions have a low concentration of surface-active materials. Increasing the concentration of surfactant in dilute ink dispersions decreased the degree of membrane fouling. However, excessive surfactant decreased permeation rates because of micelle formation. Clarification of concentrated ink dispersions (>0.4% ink solids) resulted in relatively stable permeation rates and less fouling. The inherently higher concentrations of surface-active materials in concentrated ink dispersions appear to retard fouling.

Application:

Ultrafiltration shows promise as a means of clarifying ONP wash filtrates containing finely dispersed water-based flexographic inks. Control of surfactant concentration is key to prevention of membrane fouling.

FLEXOGRAPHIC PIGMENT RESIDUES are particularly well suited to removal by wash deinking (1). However, wash filtrate after deinking a stock consist-

Ultrafiltrative deinking of flexographic ONP: The role of surfactants

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ing of 100% flexographic ONP is estimated to contain between 0.05% and 0.1% finely dispersed pigment. This filtrate cannot be recycled to the process without reducing the brightness of the product pulp. To facilitate water reuse, the wash filtrate must be clarified of the flexographic pigments. Dissolved air flotation (DAF) is normally used to clarify wash filtrate. However, the hydrophilic flexographic pigments resist removal by DAF, even with the addition of cationic collectors and anionic flocculants (2).

Membrane separation technology is a potentially viable method for clarifying flexographic pigment dispersions from wash filtrate. Earlier work has shown that ultrafiltration and microfiltration are both capable of completely removing flexographic pigment from the aqueous dispersions formed by wash deinking of newsprint (3). Moreover, those results indicated that ultrafiltration can maintain higher clarification rates at most ink concentrations of interest, making this a better choice than microfiltration for clarifying flexographic ink dispersions (4). The present work is aimed at attaining a better understanding of the mechanism of membrane fouling and the role of surface-active materials in preventing it.

EXPERIMENTAL**Membranes and materials**

Two formulations of flexographic ink were used. One ink (Ink 1) was obtained from a regional newspaper

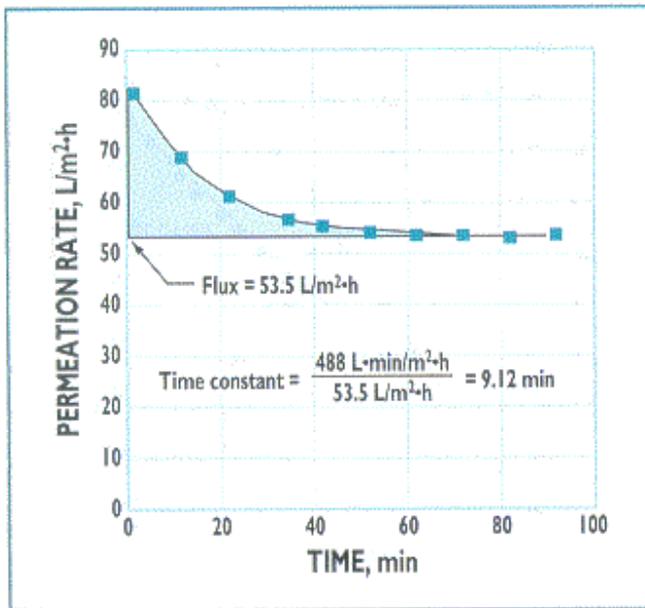
publisher and was supplied as press-ready ink. About one liter of this ink was made available, and it was used in preliminary tests only. Another ink (Ink 2) was obtained directly from an ink manufacturer. The majority of experiments were performed using this second ink.

The experimental apparatus is described in detail elsewhere (5). The polysulfone hollow-fiber ultrafiltration membranes used in this study were assembled in a shell-and-tube configuration of 68 hollow fibers with 0.043-in. (0.109 cm) inside diameter for a total of 1 ft² surface area, with a molecular-weight cutoff (MWCO, a measure of membrane pore size) of 500,000.

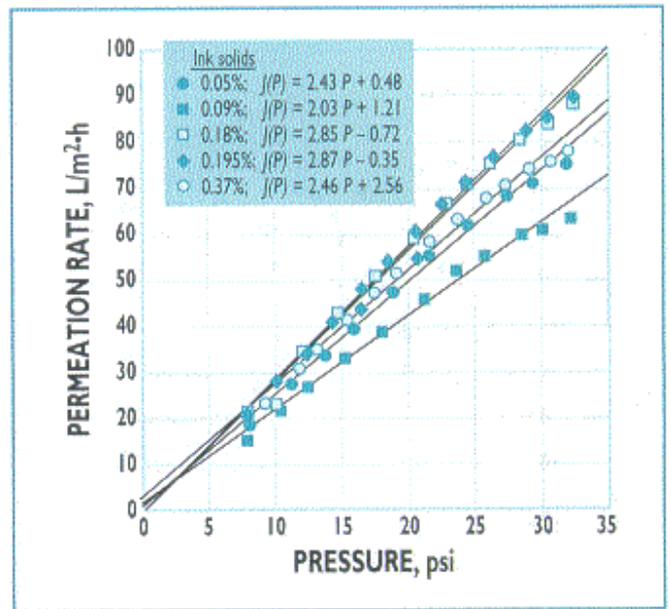
Unless otherwise specified, surfactant was added in the amount required to form a 0.01% (by volume) solution. The surfactant used was a commercially available concentrated cleaning solution (Micro 90, International Products Corp., Burlington, NJ). This is an anionic and nonionic surfactant mixture with major ingredients of water, ethylenediamine tetraacetic acid, benzenesulfonic acid dimethyl ammonium salt, triethanolammonium dodecylbenzenesulfonate, and polyoxyethylene undecylether (number of oxyethylene units not specified).

Flux vs. pressure

Permeation rates are reported as flux, which is measured as units of volume per unit of membrane surface area per unit time (L/m²·h). The relationship between permeate flux and applied pressure was investigated in a series of experiments in which the



1. Determination of the flux-decay time constant from flux-vs.-time data



2. Ultrafiltration permeation rates vs. pressure during clarification of dilute dispersions (<0.4% ink solids) of Ink I at 22°C and 1 gal/min crossflow rate

feed crossflow rate and temperature were held constant. The system was allowed to stabilize by operating at a constant pressure (25 psi) for one hour before flux-vs.-pressure data were collected. During some experiments, permeation rate data were collected through two or three cycles of increasing followed by decreasing pressure. In these cases, the stabilization period at constant pressure was omitted. The ink dispersion was maintained at a constant pigment concentration by recycling the permeate back to the feed tank.

Flux vs. time

An ultrafiltration experiment was performed in which permeate was removed from the filtration loop as it was produced and replaced with an equal volume of tap water that had been treated with NaOH to raise the pH to 10. This was done to maintain a constant pigment concentration while allowing the concentration of the dissolved components of the ink to decrease. Flux was measured as a function of time.

Immediately following each experiment, the membrane modules were cleaned and then tested to determine the effectiveness of cleaning.

Modeled characterization of ultrafiltration

A series of multivariable experiments was devised and implemented to investigate potentially interactive effects of temperature, ink content, and the concentration of supplemental surfactant on ultrafiltration performance. The effects were evaluated in terms of permeation rates and the ability to achieve steady-state clarification rates. The target ranges of the continuous variables were:

- Ink concentration, 0.1-10%
- Operating temperature, 30 -50°C
- Added surfactant, 0 - 1% (by volume)

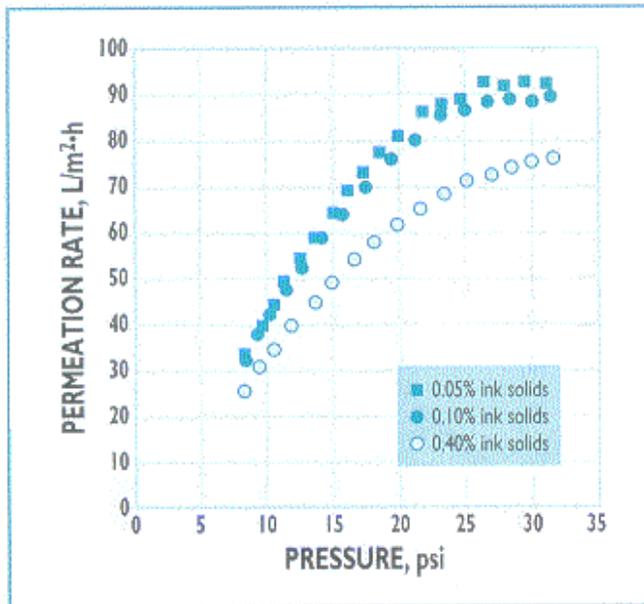
Each experiment was conducted at constant ink concentration, temperature, surfactant concentration, feed crossflow rate, and pressure. The detailed procedure has been described elsewhere (5). Permeate flux was measured at 10-min intervals for the duration of the experiment, which ranged from 90 to 140 min, depending on the stability of the flux measurements. The results of the experiment were reduced to a single data point, that being the average of the flux measurements taken within the time interval 80-100 min

| Ink conc., % solids | RESISTANCE, N·h/L | |
|---------------------|--------------------|------------------------------|
| | Fouling (R_f), | Boundary layer (R_{b1}), |
| 0.050 | 1734 | 677 |
| 0.090 | 1888 | 1082 |
| 0.181 | 1621 | 372 |
| 0.195 | 1814 | 162 |
| 0.370 | 1194 | 1183 |

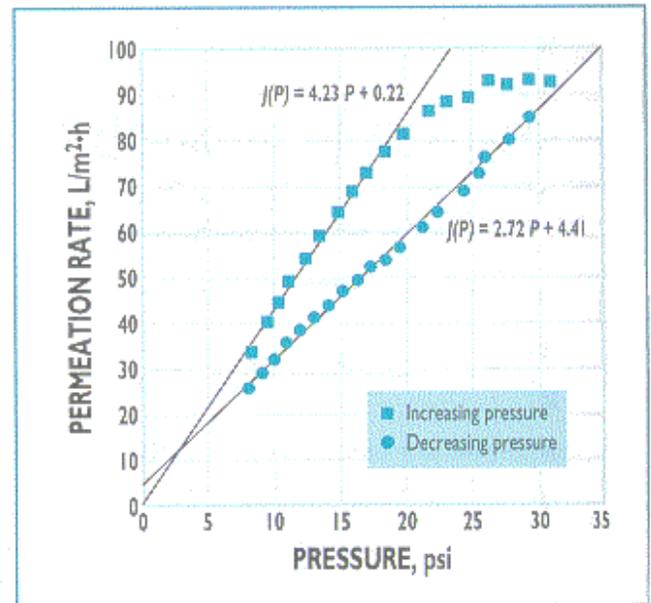
1. Fouling and boundary-layer resistances obtained after clarifying dilute dispersions of Ink I

(Fig. 1). At least two flux measurements (typically three or four) were used in obtaining this average value of permeate flux.

An estimate of permeate flux stability was determined for each experiment from the flux-vs.-time data. This estimate was obtained by integrating the permeation rate-vs.-time data for the period from 1 to 90 min of operation and subtracting the steady-state permeation rate (that measured after 90 min of operation) multiplied by the 89 min of operation. The resulting value is the area under the curve of the flux-vs.-time data minus the steady-state permeation rate, as illustrated in Fig. 1. This



3. Ultrafiltration permeation rates vs. pressure during clarification of dilute dispersions (10.4% ink solids) of Ink 2 at 22°C and 1 gal/min crossflow rate



4. Ultrafiltration permeation rates for a complete pressure cycle during clarification of Ink 2 dispersion containing 0.05% ink solids at 22°C and 1 gal/min crossflow rate

value was divided by the permeation rate after 90 min of operation to normalize the stability assessment for differences in pseudosteady-state flux inherent in clarifying dispersions of different composition and temperature. In this manner, the flux-decay time constant was determined, with units of minutes.

RESULTS

Laboratory ultrafiltration results

Distilled deionized water was processed through a new ultrafiltration membrane to obtain baseline permeation rate information. Permeation rates (flux) were measured as a function of applied pressure at a series of constant temperatures between 20°C and 50°C. Flux was directly proportional to pressure, as predicted by the pore model of ultrafiltration (6) based on the Hagen-Poiseuille equation, which also predicts an inversely proportional relationship between flux and permeate viscosity:

$$J = \frac{\epsilon r^2 P}{8 \eta L}$$

where

J = flux through the membrane

ϵ = surface porosity of the membrane

r = channel radius

P = transmembrane pressure

η = fluid viscosity

L = channel length

The intrinsic membrane resistance R_m is the resistance to permeation through the semipermeable membrane itself (7). When processing pure water, R_m is the only resistance to permeation that must be overcome by the applied pressure. Therefore R_m can be calculated using the relation:

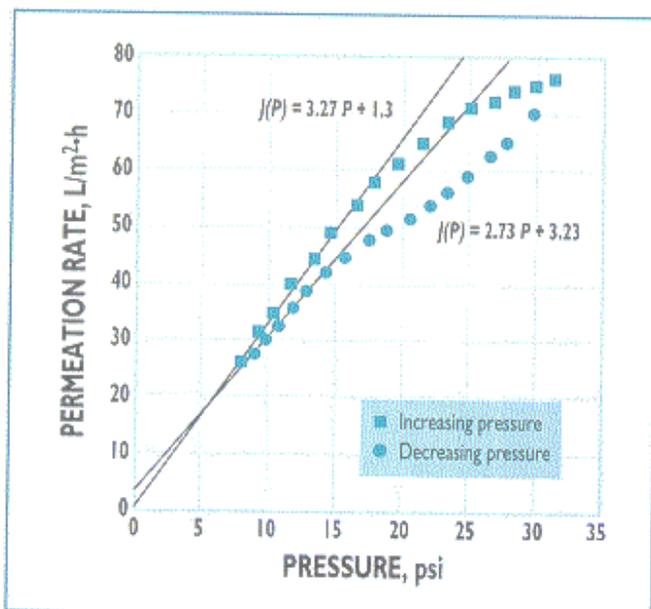
$$J(P) = \frac{P}{R_m}$$

The intrinsic membrane resistance R_m at 20°C was determined to be 426 N·h/L.

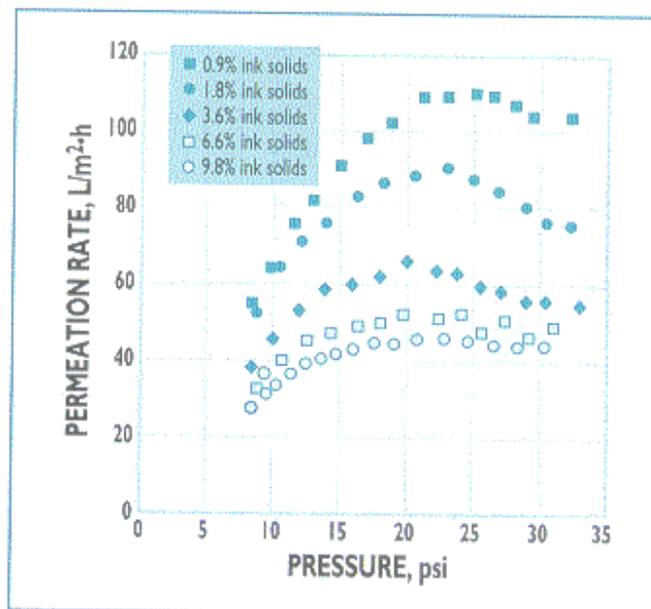
During ultrafiltration, colloidal solids are brought to the membrane surface by convective transport as solvent permeates through the membrane, resulting in a higher local concentration of retained solids at the membrane surface as compared with the bulk suspension. This phenomenon, known as concentration polarization (8), is believed to be responsible for the decrease in permeation rate observed when processing fluids containing rejected material as

compared with that of pure water. This is the basis of the film-theory, or gel-polarization, model of ultrafiltration.

Flexographic ink was dispersed in water to simulate the effluent from a wash operation used to deink stock containing flexographic printed ONP. Figure 2 shows permeation rate data as a function of pressure at 22°C and 1 gal/min crossflow rate (average linear velocity of 1 m/s) when clarifying dilute dispersions of flexographic Ink 1 (ink contents <0.4%). Approximately linear relations between permeation rate and applied pressure were observed. Permeate samples were analyzed by visible-light spectrophotometry and determined to be pigment free. The introduction of ink drastically reduced attainable permeation rates, with measured values ranging from 50 to 70 L/m²·h at 25 psi. It appears that the concentration of ink had little influence on permeation rate, as no trend was observed between ink content and permeate flux. The highest permeation rates were observed when clarifying the 0.195% ink dispersion, and the lowest when clarifying the 0.09% dispersion.



5. Ultrafiltration permeation rates for a complete pressure cycle during clarification of Ink 2 dispersion containing 0.4% ink solids at 22°C and 1 gal/min crossflow rate



6. Ultrafiltration permeation rates vs. pressure during clarification of concentrated dispersions (20.9% ink solids) of Ink 2 at 22°C and 1 gal/min crossflow rate

The slopes of the flux-vs.-pressure lines in Fig. 2 range from 2.03 (for 0.09% ink) to 2.87 (for 0.195% ink). These slope values can be interpreted as the overall resistance to pressure-controlled permeation, which would include terms associated with the intrinsic membrane resistance R_m and resistances caused by the concentration boundary layer R_{b1} and membrane fouling R_f (7). The fact that the overall resistance to permeation did not follow a trend with increasing ink content indicates that the contribution of the boundary layer to overall permeation resistance was minimal. Therefore, the predominant resistance must have been the result of membrane fouling. This was confirmed by measuring water flux after cleaning the membrane at the conclusion of each clarification experiment. Prior to the introduction of ink, the water flux was measured at 404.1 L/m²·h. After the first clarification run (with 0.05% ink), the water flux had decreased to 89.8 L/m²·h (measured at 25 psi and 25°C). From these data, the permeation resistance resulting from membrane fouling R_f was calculated based on the relation:

| Ink conc., % solids | RESISTANCE, N·h/L | |
|---------------------|-------------------|-----------------------------|
| | Fouling (R_f) | Boundary layer (R_{b1}) |
| 0.05 | 962 | 243 |
| 0.10 | 644 | 602 |
| 0.41 | 423 | 1259 |

II. Fouling and boundary-layer resistances obtained after clarifying dilute dispersions of Ink 2

$$J(P) = \frac{P}{R_m + R_{b1} + R_f}$$

Because $R_{b1} = 0$ for water, this reduces to

$$J(P) = \frac{P}{R_m + R_f}$$

The fouling resistance R_f was determined to be 1734 N·h/L.

The boundary-layer resistance can now be calculated. Since the overall resistance to permeation now includes all three contributions, the following relation can be used:

$$J(P) = \frac{P}{R_m + R_{b1} + R_f}$$

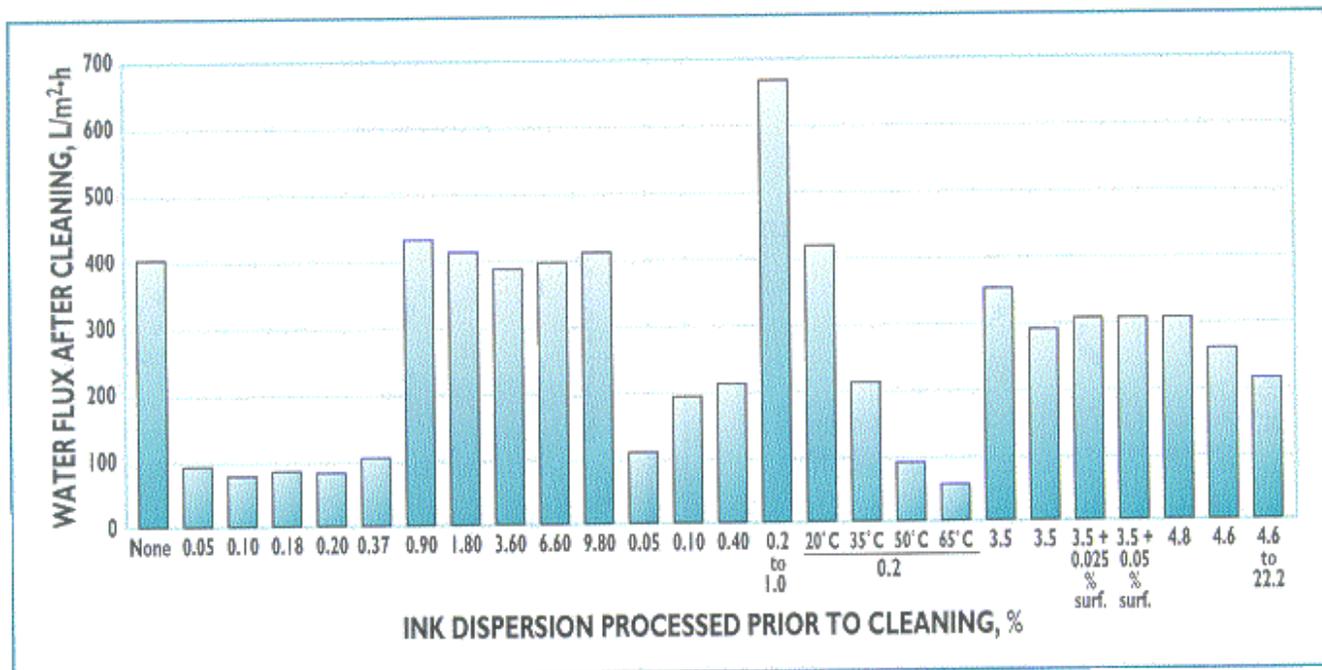
The boundary-layer resistance of the 0.05% bulk ink dispersion at the experimental conditions was deter-

| Ink conc., % solids | RESISTANCE, N·h/L | | |
|---------------------|-------------------|-----------------------------|---------------------|
| | Fouling (R_f) | Boundary layer (R_{b1}) | Gel layer (R_g) |
| 0.9 | -5.33 (0) | 681 | 1089 |
| 1.8 | -9.05 (0) | 721 | 1904 |
| 3.6 | -14.22 (0) | 1096 | 2582 |
| 6.6 | -14.90 (0) | 1448 | 2490 |
| 9.8 | -10.65 (0) | 1638 | 2594 |

III. Fouling, boundary-layer, and gel-layer resistances after clarifying concentrated dispersions of Ink 2

mined to be 677 N·h/L. Similar determinations of permeation resistance were made for the other dilute ink dispersions. The results in **Table I** indicate that membrane fouling was the major resistance to permeate flux in all cases. However, when the ink concentration approached 0.4%, the data suggest that the fouling resistance began to decrease and the boundary-layer resistance became more significant in determining permeate flux.

Clarification of dilute dispersions of Ink 2 showed similar flux-vs.-pressure relationships, as seen in **Fig. 3**. However deviation from linearity was observed as the applied pres-



7. Water flux through the membrane after clarifying various flexographic ink dispersions (* denotes after extended soaking)

sure was increased. The deviation from linearity could be the result of increases in (a) the fouling resistance with time of operation or (b) the concentration-boundary-layer resistance as the applied pressure was increased.

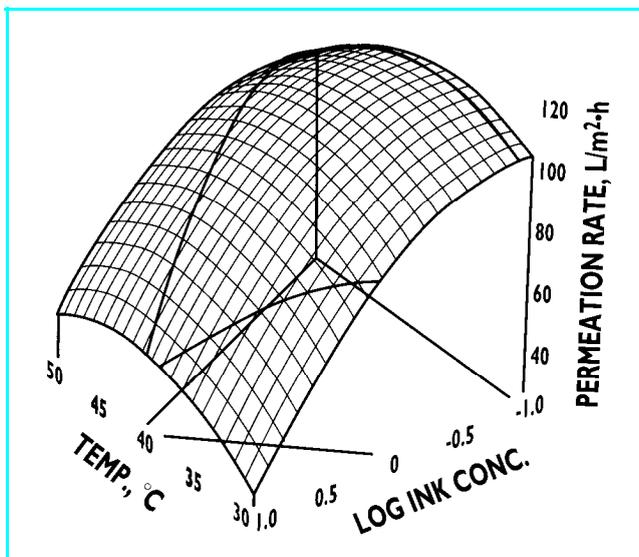
The film theory of ultrafiltration predicts an increase in the resistance of the concentration boundary layer at higher levels of applied pressure in response to increases in the boundary-layer thickness and in the concentration gradient that occur at higher permeation rates (9). However this effect should be reversed upon subsequent pressure reduction. The hysteresis patterns observed during clarification of the 0.05% ink dispersion under both increasing and decreasing pressure are shown in **Fig. 4**. Examination of these patterns could lead one to discount the possibility of increasing concentration-boundary-layer resistance as an explanation for the deviation from linearity during the increasing-pressure cycle. The linear relation between permeate flux and pressure while decreasing the applied pressure indicates a constant permeation resistance, independent of pressure. A more plausible explanation

for the departure from linearity with increasing pressure could be made based on the occurrence of membrane fouling resulting from clarification of this dispersion, with the fouling resistance increasing as the experiment proceeded. Indeed, the fouling resistance should be independent of pressure but dependent on time of operation. As with the dilute dispersions of Ink 1, clarifying dilute Ink 2 dispersions did result in decreased water-flux values. The water flux decreased from 415 L/m²-h to 111 L/m²-h after clarifying the 0.05% dispersion, indicating fouling of the membrane. However, the situation appears to be more complicated. **Figure 5** shows the hysteresis patterns for the 0.41% ink dispersion. In this case, examination of the increasing- and decreasing-pressure cycles leads to a different conclusion. The level of hysteresis decreased as pressure was lowered below 15 psi, indicating that permeation resistance decreased. This lends credibility to the theory that departure from linearity during the increasing-pressure cycle was due to increases in the boundary-layer resistance to permeation. In actuality, both fouling and increased bound-

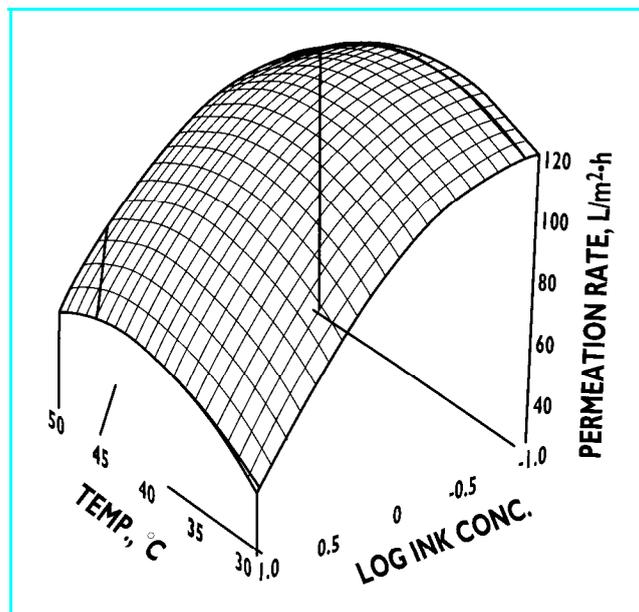
ary-layer resistance probably occur when clarifying these dilute ink dispersions. Moreover, it appears that the fouling resistance is predominant when clarifying the more dilute dispersions, with boundary-layer resistance becoming more significant at higher ink concentrations.

The amount of permeation resistance attributable to fouling and to the boundary layer was calculated for Ink 2 dispersions at different concentrations. The results, listed in **Table II**, verify the supposition that membrane fouling occurred to a greater extent when clarifying very dilute ink dispersions, with concentration-polarization boundary-layer resistance becoming more influential as the bulk ink concentration increased.

There were two distinct operating regions observed when clarifying dispersions containing 0.9% ink or more. Permeation rates increased proportionally with the applied pressure at low pressures and became pressure independent as the applied pressure was increased above a certain critical level, as seen in **Fig. 6**. There are two theories commonly used to explain this pressure-independent ultrafiltration phenomenon.



10. Permeation rate vs. ink content and temperature with no supplemental surfactant



11. Permeation rate vs. ink content and temperature with 0.5% supplemental surfactant

higher water-flux values regardless of the condition of the membrane prior to the experiment.

Membrane fouling resistances resulting from clarification of flexographic ink dispersions at 20–22°C were calculated. **Figure 8** is a plot of the fouling resistances vs. ink concentration. From these results, it can be seen that clarifying dispersions containing approximately 0.5% ink or more resulted in very low values for fouling resistance, whereas fouling resistance increased exponentially after clarifying lower-concentration ink dispersions. The binder resin concentration of a 0.5% ink dispersion is about 0.09% by weight, or 1.8×10^{-5} mole/L based on an average molecular weight of 50,000 for the binder resin (12). The present results indicate that binder resin concentrations lower than 1.8×10^{-5} mole/L are insufficient to prevent interaction between the carbon-black pigment particles and the nonpolar polysulfone membrane, which presumably leads to membrane fouling.

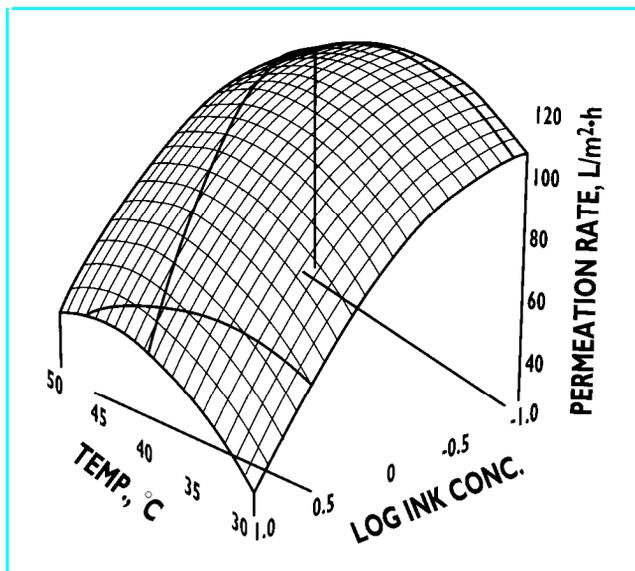
Figure 9 shows a plot of fouling resistance vs. temperature, calculated from the results of elevated-temperature experiments. Because the polyacrylic binder resin is ionized at alkaline conditions, its solubility—and thus its tendency to become desorbed from the pigment particles—increases at elevated temperature (13). The binder resin concentration of a 0.2% ink dispersion is approximately 7.2×10^{-6} mole/L. The data in Fig. 9 show that very little fouling resulted from clarifying the 0.2% ink dispersion at 20°C. However, the fouling resistance increased exponentially as the temperature was raised. The interaction between temperature and ink concentration in determining the extent of membrane fouling most likely reflects the temperature-dependent solubility of the binder resin. Therefore, when clarifying dilute ink dispersions

(with low binder resin concentration), increasing the temperature leads to particle-membrane interactions that foul the membrane.

Modeled characterization of ultrafiltration

Results from this experimental sequence were used to develop polynomial relations capable of predicting permeate flux and stability as a function of the design variables (temperature, ink concentration, and concentration of surface-active materials). Ink concentration was transformed to a log scale because previous results have shown the relationship between flux and ink concentration to be logarithmic (14). It should be noted that this model is empirical in nature. The data were fit to a polynomial model based on ease of modeling rather than on theoretical considerations. Therefore, the predictive results of the model should be viewed as qualitative descriptions of the influence of the design variables on ultrafiltration performance.

As expected, the concentration of ink in the dispersion being clarified had a controlling influence on permeate flux. An approximate logarithmic relation was observed between ink content and flux when clarifying dispersions containing between about 0.3% and 10% ink, as seen in **Fig. 10**. At lower ink concentrations, permeate flux was predicted to be relatively independent of ink content. Raising the temperature (from 30°C to 50°C) increased permeate flux when clarifying dispersions containing 1% to 10% ink and decreased permeate flux when clarifying dispersions containing less than about 0.8% ink.

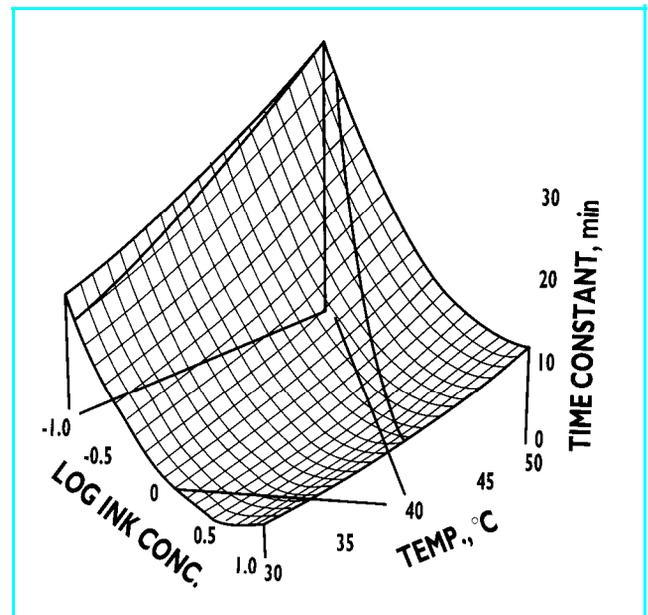


12. Permeation rate vs. ink content and temperature with 1% supplemental surfactant

The temperature dependence of permeate flux was similar at all concentrations of supplemental surfactant between zero and 1% by volume (Figs. 10-12). However at any given temperature, the maximum permeate flux was predicted when the feed contains 0.5% supplemental surfactant. Increasing the surfactant concentration to 1% decreased predicted permeation rates to levels only slightly higher than those predicted for dispersions containing no supplemental surfactant. The model predicts that, at any ink concentration, the maximum permeate flux was attained at approximately 40°C and 0.55% surfactant.

The least-stable permeation rates were predicted when clarifying dilute inks, especially when operating at elevated temperatures with no addition of supplemental surfactant, as seen in Fig. 13. At 30°C, the model predicted a minimum in the flux-decay time constant at an ink concentration of 2%. The ink concentration at which the most stable permeation rates were achieved increased logarithmically to 4.5% ink as the temperature was raised to 50°C. In general, the most stable operation was observed at high ink concentration and low temperature. The flux-decay time constant increased almost linearly with temperature, and the effect of temperature was greater when clarifying dispersions with high ink concentrations. Because the flux-decay time constant is a measure of (a) the time required to achieve pseudosteady-state permeate flux and (b) the rate of change in flux during this time period, these results indicate that clarifying dilute ink dispersions at elevated temperature leads to unstable permeation rates.

Increasing the surfactant concentration to 0.5% enhanced permeation rate stability by decreasing the flux-decay time constant at all ink concentrations and tem-

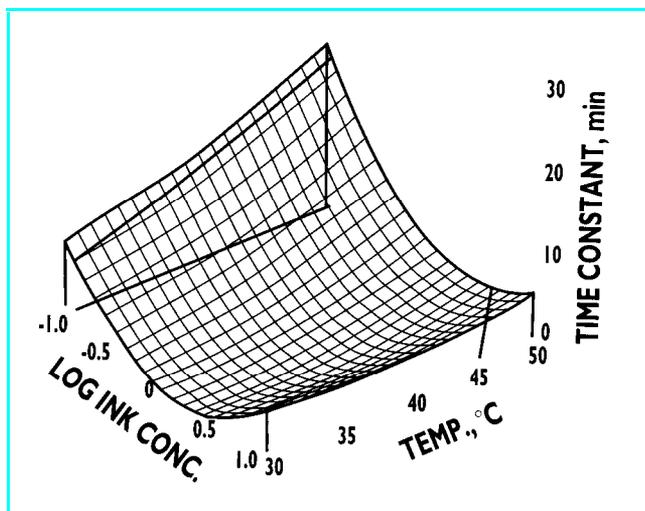


13. Flux-decay time constant vs. ink content and temperature with no supplemental surfactant

peratures, as seen in Fig. 14. The effect of surfactant was most pronounced when clarifying dilute ink dispersions at 50°C. Increasing the concentration of supplemental surfactant to 1% served to further decrease the flux-decay time constant when clarifying dilute inks at high temperature and to increase slightly the time constant when clarifying high-concentration ink dispersions at high temperature, as seen in Fig. 15.

These results show that permeate flux was less stable with time (i.e., it displayed more decay) when clarifying dilute ink dispersions than when clarifying concentrated dispersions. Figure 16 shows the predicted effect of surfactant addition and temperature on the flux-decay time constant when clarifying a 0.1% ink dispersion. This graph shows the interaction between temperature and surfactant content and its effect on permeation rate stability. The model demonstrates that stability can be enhanced by operating at low temperature or high surfactant concentration. The decrease in permeation rate with time when clarifying dilute ink dispersions (as reflected in high time-constant values) can be explained by the relatively low concentration of binder resin in the 0.1% ink dispersion, combined with increased solubility of the resin at high temperature, leading to a low level of adsorption on the pigment particles. Without the electrostatic and steric stabilization of the pigment dispersion by adsorbed binder resin, there was a higher tendency for particle-membrane interactions, which leads to membrane fouling and thus to low permeate flux stability.

Increasing the temperature increased the permeate flux at all ink concentrations except for the most dilute. The high values of the flux-decay time constant pre-

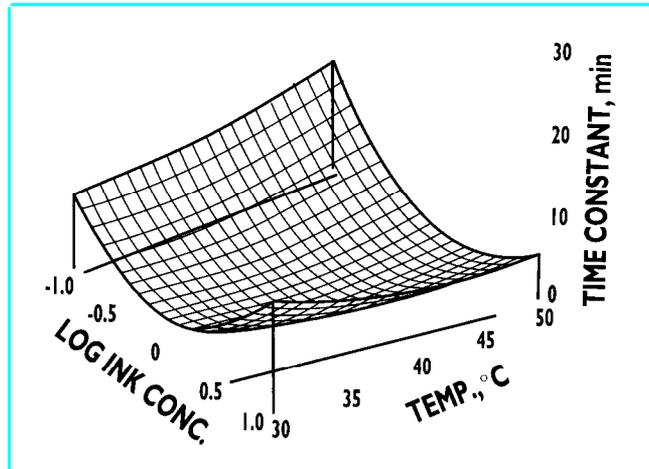


14. Flux-decay time constant vs. ink content and temperature with 0.5% supplemental surfactant

dicted when operating at these conditions indicate that a higher fraction of the pores became plugged.

Increasing the surfactant concentration in the feed promoted permeation rate stability when clarifying dilute ink dispersions. This was most likely due to surfactant-induced stabilization of the small pigment particles, thus decreasing the degree of interaction between particles and the membrane pores. Surfactant addition also increased permeation rates at low temperatures, probably because of the same stabilizing effect. Although surfactant addition appears to enhance stability at all temperatures, it can lead to depressed permeation rates at elevated temperatures, especially when clarifying concentrated ink dispersions. The decreased permeation rates cannot be explained by the presence of large concentrations of dissolved surfactant, whose molecular size would be much too small to result in retention. However, if the surfactant concentration is increased above a critical concentration, aggregates or micelles may be formed, which would result in the surfactant being rejected by the membrane and potentially contributing to the permeation resistance of the concentration boundary layer and gel layer, leading to low permeation rates.

The concentration at which surfactant molecules aggregate into micelles is called the critical micelle concentration (c.m.c.) (15). The critical micelle concentration is specific to each surfactant, and in general the c.m.c. of nonionic surfactants is approximately two orders of magnitude lower than that of anionic surfactants (16). The c.m.c. of anionic surfactants typically increases with temperatures above 25°C, whereas the c.m.c. of nonionic surfactants decreases with increasing temperature (13, 17). The presence of nonionic surfactants in surfactant mixtures leads to incorporation of anionic surfactant into mixed micelles at concentrations much lower than the c.m.c. of the pure anionic surfactant (17).



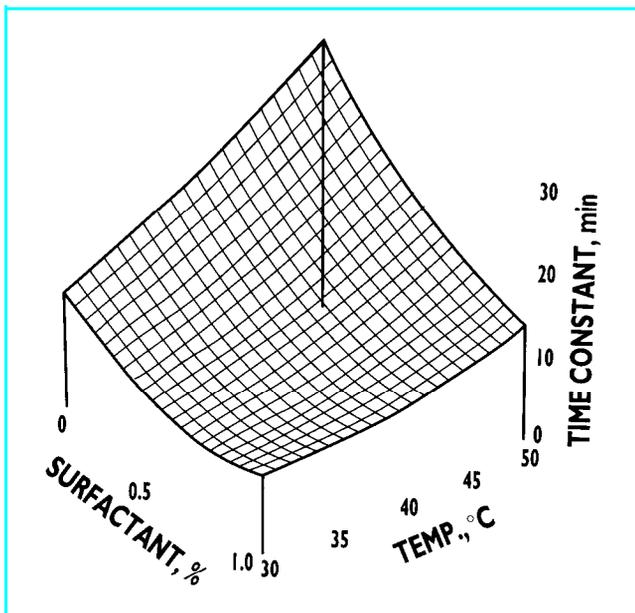
15. Flux-decay time constant vs. ink content and temperature with 1% supplemental surfactant

The effect of surfactant micelle formation on permeate flux is demonstrated in **Fig. 17**, which shows the reduction in distilled-water flux when surfactant is added in concentrations higher than its c.m.c. Addition of 0.1% surfactant had virtually no effect on permeate flux because the surfactant was dissolved and readily passed through the membrane. However, addition of 1% surfactant caused the flux to drop to about one-sixth of the value obtained with pure water, resulting in the onset of pressure-independent ultrafiltration. The surfactant micelles were brought to the membrane surface by convective transport, but were too large to pass through the membrane. The consequent concentration-polarization effects reduced the permeate flux.

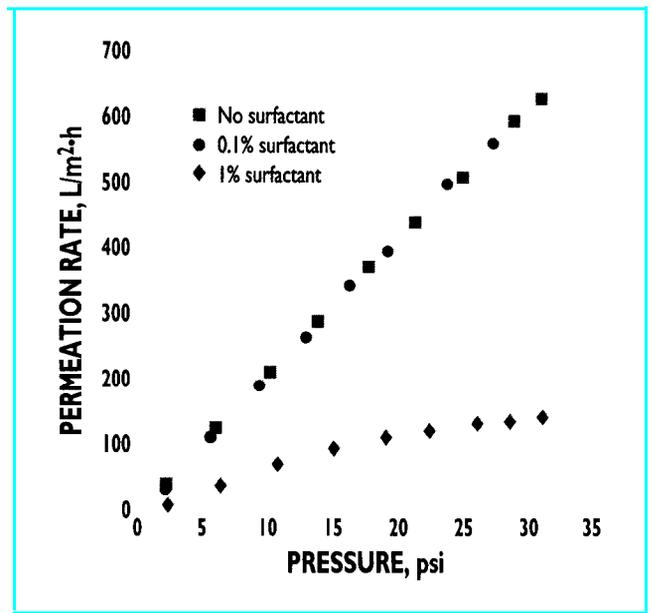
Interpretation of the given results is complicated by the variety of surface-active materials present in the flexographic ink dispersions that served as feed materials. However, the observed effect of temperature-surfactant-concentration interaction on permeate flux could reflect the temperature dependence of the c.m.c. for the anionic and nonionic surfactant mixture added. At 50°C, increasing the surfactant concentration could result in micelle formation and reduction of permeation rates. At 30°C, the added surfactant could have a more beneficial effect on dispersion stabilization, leading to higher permeation rates. The observation that surfactant addition is more detrimental to elevated-temperature clarification of concentrated ink dispersions than dilute inks also indicates that micelle formation could be the cause of this effect, since concentrated ink dispersions have an inherently higher concentration of surface-active materials.

CONCLUSIONS

When clarifying dilute flexographic ink dispersions (ink content <0.4%), linear relationships were observed for ultrafiltration permeate flux vs. pressure, as predicted by the pore model. Permeate flux was relatively



16. Flux-decay time constant vs. supplemental surfactant concentration and temperature for ultrafiltration of 0.1 % ink dispersion



17. Effect of surfactant concentration on ultrafiltration flux for distilled water

independent of ink content when clarifying dilute dispersions. The major resistance to permeation was membrane fouling, the result of inadequate surfactant concentrations. When clarifying more concentrated dispersions (ink content >0.4%), permeate flux was limited by the combined resistances of the concentration-polarization boundary layer and the gel layer. Permeation rate became independent of pressure between 15 and 20 psi when clarifying these dispersions. It was shown that the phenomenon of pressure-independent permeate flux was due to mass-transfer limitations, as described by the concentration-polarization model, rather than to osmotic-pressure limitations. No appreciable membrane fouling occurred when clarifying concentrated flexographic ink dispersions, since these contained higher concentrations of surface-active materials.

The experimental results were used to develop empirical polynomial relations that provide qualitative predictions of permeate flux and stability as functions of temperature, ink content, and supplemental surfactant concentration. Permeate flux demonstrated a logarithmic dependence on ink content at concentrations above approximately 0.5% ink, with a lesser dependence when clarifying more dilute dispersions. It was demonstrated that the effects of temperature, ink content, and surfactant concentration on permeation rate and flux stability are interdependent. The effect of temperature is twofold. Elevated temperature can increase permeation rates because of reduced fluid viscosity. However, when clarifying dilute inks—where the concentration of surface-active materials is low—elevated temperature can lead to extensive membrane fouling and thus low permeation rates and unstable permeate flux. Conversely, operation

at elevated temperature can reduce permeation rates in dispersions containing high levels of supplemental surfactant, presumably by promoting formation of surfactant micelles that contribute to the concentration-polarization limitations.

Addition of supplemental surfactant to dilute ink dispersions increased the permeation rate and enhanced permeate flux stability. Adsorption of surfactant onto the individual pigment particles stabilizes the electrostatic and steric properties of the dispersion, thus reducing the occurrence of the particle-particle and particle-membrane interactions that lead to membrane fouling. Adding surfactant to highly concentrated ink dispersions did not significantly affect flux stability, but it decreased permeate flux, especially at elevated temperature, because of the onset of surfactant micelle formation.

The results of this study demonstrate that ultrafiltration can be used to clarify ONP wash filtrates that contain water-based flexographic inks. Stable clarification of flexographic pigment dispersions can be achieved by controlling the surfactant concentration of the feed. However, there are a number of practical questions to be addressed prior to industrial scale up. For example, the membrane area required to treat a given effluent is dependent upon the number of stages and the desired percent recovery of the effluent. The percent recovery of clean water is limited only by the maximum solids content of the feed stream that can be handled by the membrane process. (Approximately 25% solids by weight was achieved at Auburn University using hollow-fiber ultrafiltration membranes.) At present, the most appropriate disposal route for rejects would be combination with the mill effluent prior to primary treatment. TJ

RECYCLING

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