Conventional flotation deinking technology is widely used for deinking secondary fiber furnish which has been printed by the letterpress or lithographic (offset) processes. This technology is not effective for removing water-based flexographic inks due to the hydrophilic nature of their pigment particles, which form fine dispersions upon repulping. Washing is effective at removing flexographic ink dispersions from pulped secondary fiber, however, the resulting wash effluent contains significant quantities of pigment. The wash effluent must be clarified of pigment before it can be recycled within the mill without reducing pulp brightness. The difficulty in clarifying flexographic pigment dispersions from wash filtrate represents a significant barrier to closing the water loop of a deinking operation.

Ultrafiltration was investigated as a means to remove dispersed water-based pigments from wash effluent. Ultrafiltration of high concentration ink dispersions resulted in stable, high production rates of pigment free permeate. The use of high concentration feeds prevented irreversible membrane fouling, and actually demonstrated membrane regenerative properties. Inclusion of cellulose fines in the feed had mixed results on permeation rates.

ABSTRACT

Conventional flotation deinking technology is widely used for deinking secondary fiber furnish which has been printed by the letterpress or lithographic (offset) processes. This technology is not effective in removing flexographic ink residue, which form fine dispersions of pigment particles upon repulping. The same properties of flexographic ink dispersions which render them difficult to remove by flotation deinking, specifically colloidal dimensions and hydrophilic surfaces, lend them to efficient removal by washing technique. Washing is effective in the removal of flexographic ink residues from secondary fiber suspensions. However, the ink residues must be removed from the wash filtrate before this stream can be recycled to the process or discharged.

It has been shown that froth flotation under acidic, or at least non-alkaline conditions improves the efficiency of this operation in removing water-based flexographic inks. A two stage process consisting of non-alkaline flotation for removal of flexo inks, followed by alkaline flotation for oil-based ink removal has been proposed. There is also interest in developing flocculation aids (polymers) which can be added to the grey stock in order to render the flexographic pigment particles hydrophobic. This would result in an increased tendency for the flexo inks to be removed by conventional flotation deinking. Membrane separation technology is a potentially attractive method for the removal of flexographic ink residues from wash filtrate without further addition of chemicals.

A proposed flow diagram for the application of ultrafiltration techniques in a deinking process is shown in figure 1. Deinking a mixed old newsprint (ONP) furnish would rely on both froth flotation and washing. The wash filtrate would be sent to a multiple stage ultrafiltration operation to remove the flexographic pigments prior to being recycled to the process. The concentrated ink residue, consisting primarily of carbon black pigment, could be utilized in energy recovery. The number of stages, membrane surface area of each stage, and location in the deinking process to apply ultrafiltration must be optimized in order to maximize production rate and minimize capital (membrane) and pumping costs.

This study focuses on the feasibility of ultrafiltration techniques to remove dispersed water-based pigments from aqueous dispersions. An ultrafiltration apparatus was assembled and used to characterize the clarification of flexographic ink dispersions prepared from two commercially available water-based flexographic inks. The efficiency of the ultrafiltration separation process is characterized by parameters such as permeate flux, fouling rate, and cleaning requirements.
EXPERIMENTAL

The ultrafiltration apparatus is equipped with adequate instrumentation and controls to manipulate important operational parameters such as pressure drop across the membrane surface (transmembrane pressure, $\Delta P_{\text{m}}$), temperature, and flowrate through the membrane module. Two identical polysulfone hollow fiber ultrafiltration membranes were used in this study. Each membrane module is constructed in a shell and tube configuration, with a total of 1 ft$^2$ (0.0283 m$^2$) of surface area arranged into 68 hollow fibers of 0.043 inch (0.109 cm) ID. The molecular weight cut off (MWCO) of an ultrafiltration membrane is used as a measure of pore size, or retention ability, and is the approximate molecular weight of the smallest compound which will be retained by the ultrafiltration membrane. The MWCO of the membranes used in this study was 500,000. Figure 2 shows a schematic diagram of the ultrafiltration apparatus.

Feed solutions for the ultrafiltration experiments were prepared using tap water, flexographic ink, surfactant, and NaOH to adjust the pH to 9.7-10.7. A commercially available anionic and nonionic surfactant mixture was used, and the amount necessary to form a 0.01% solution was added (only 4 mL per 40 L of solution). Two formulations of flexographic ink were used in these studies. One was obtained from a regional newspaper publisher (ink #1), and was supplied as press ready ink. Another was obtained directly from an ink manufacturer (ink #2). Dispersions used in the ultrafiltration experiments had ink concentrations ranging from 0.04% to 10%. The concentration of ink was determined by visible light spectroscopy. Absorption at 457 nm by a diluted feed sample was compared to a calibration curve of light transmittance vs. ink concentration.

The feed solution was maintained at a constant ink concentration during most of the experiments by recycling the pigment free permeate back into the feed tank. However, during some of the experiments the permeate was removed from the ultrafiltration loop as it was produced. This resulted in the feed solution (ink dispersion) becoming more concentrated as the ultrafiltration operation proceeded (the total volume of feed also decreased during these experiments). The feed was kept well mixed by the action of the recirculation pump. In most experiments the temperature was maintained constant by passing the feed through a constant temperature water bath prior to the ultrafiltration module. An electric heater was incorporated into the ultrafiltration loop and used to heat the feed when performing experiments at elevated temperature. During these experiments the water bath was used for fine control of temperature.

Flux was measured by the timed collection of permeate in a tared beaker which rested on a balance. The specific gravity of the permeate was determined to be 1.0. After the flux measurement, the collected permeate was returned to the feed tank.

In some of the experiments cellulose fines were added to the feed. Cellulose fines were generated by beating unprinted newsprint stock, and collecting the fraction which passed through a 65 mesh screen and was retained by a 200 mesh screen. Fines were added in amounts necessary to make 0.025% and 0.05% (by weight) consistency dispersions.

The membrane module was cleaned between experimental runs. Immediately after an experiment in which flexographic ink dispersions were ultrafiltered the membrane module and entire flow path were rinsed with tap water to remove as much ink from the equipment as possible. A solution of tap water and a commercial detergent, with pH adjusted to 10 by addition of NaOH was then pumped through the membrane module and associated piping for several minutes. The dirty cleaning liquid was drained from the system, and a fresh cleaning solution was pumped through the system for at least 30 minutes. The third and final cleaning was performed using a fresh solution for as long as four hours. At this time the flux was measured using a fresh cleaning solution as the feedstream, at 25 psi $\Delta P_{\text{m}}$, 20°C, and 1 gpm. The resulting value is the water flux, which was used to evaluate the effectiveness of the cleaning procedure on restoring membrane performance.

RESULTS

Flux Performance When Processing Flexographic Ink Dispersions

Plots of permeate flux vs. $\Delta P_{\text{m}}$ for five dilute dispersions of ink #1 (less than 0.37%) are shown in figure 3. Ultrafiltration achieved complete retention of the flexographic pigment, producing clear permeate. All dispersions demonstrated linear flux vs. $\Delta P_{\text{m}}$ relationships, indicating that the system behaved as would be expected from the Hagen-Poiseuille law for streamline flow through channels, which is believed to best describe the pressure controlled flow of fluid through microporous membranes”. Flux vs. time data for batch ultrafiltration of a 0.05% dispersion of ink #1 (25 psi) are shown in figure 4. Flux stabilized at 48 1/m$^2$ hr during the six hour experiment, while the ink concentration increased from 0.05% to 0.12%. The results indicate that flux was independent of solids concentration. Flux vs. $\Delta P_{\text{m}}$ data of low concentration dispersions of ink #2 also demonstrated pressure controlled permeation rates, but at 25 psi became less sensitive to further increases in $\Delta P_{\text{m}}$ as shown in figure 5. The solids content of the feedstream appeared to have a greater influence on flux, which decreased with increasing solids content of the feedstream.
There were two distinct operating regions observed when processing the high ink content dispersions (those at 0.9% ink and above), as shown in figure 6. Flux was proportional to ΔP\text{m} at low pressure, and pressure independent at high pressure. The shift occurred at lower ΔP\text{m} as ink concentration increased, indicating that flux was strongly dependent on solids content. Higher flux values were attained while clarifying high concentration flexographic ink dispersions than when clarifying the low concentration dispersions. This is an interesting finding and will be discussed further in later sections.

During ultrafiltration of colloidal dispersions, particles are brought to the membrane surface by convective transport as fluid passes through the membrane. This effect is known as concentration polarization. At high flux levels the non-permeable material (ink pigment in this case) will consolidate into a "gel layer", which imparts additional resistance to permeate flux through the membrane\textsuperscript{13,14,15}. Shear forces associated with the flow across the membrane surface control the thickness of this layer, and at steady state convective transport and shear force dispersion balance each other\textsuperscript{16}. When this condition is achieved, increasing ΔP\text{m} will not result in increased flux. The flux will rise initially, but this will only serve to increase the amount of material transported to the membrane surface and thus the film thickness. The flux will then be reduced because of the increased resistance to permeate flow, restoring the equilibrium between pigment transport by convective flow and by shear dispersion. This operating region is known as the mass transfer controlled region. At these conditions the flux is predicted to decrease exponentially with increasing feed concentration\textsuperscript{13,14,15}.

Analysis of hysteresis patterns obtained during a cycle of increasing followed by decreasing pressure yields information on the mechanism of gel formation and pressure independent ultrafiltration. Figure 7 shows flux vs. ΔP\text{m} data for a complete pressure cycle (3.6% ink). The transition to pressure independence was gradual, due to dynamic effects, in which ΔP\text{m} was increased too fast for the gel layer to completely form as the conditions required for the onset of pressure independent operation were reached. The gel layer continued to consolidate as pressure increased, causing flux to decrease until the equilibrium flux was reached. Further increases in pressure did not affect flux. As the pressure was lowered flux decreased proportionally at first, characteristic of pressure controlled ultrafiltration. The low slope of the flux vs. ΔP\text{m} data at this point is indicative of high resistance to permeate flow due to the additive resistances of the membrane and the gel layer of retained pigment\textsuperscript{16}. As pressure was further reduced flux remained constant as the gel layer was being removed by shear forces on the membrane surface. As pigment was removed resistance to flux decreased, however ΔP\text{m} was lower so flux was unchanged. Flux increased dramatically and again became pressure controlled when the gel layer was completely removed. The hysteresis between increasing and decreasing ΔP\text{m} was observed because the gel layer is difficult to remove by shear forces once it has consolidated\textsuperscript{17}.

The flux vs. feed composition curve shown in figure 8 demonstrates the logarithmic relationship between flux and ink content which was observed for dispersions containing 0.9% ink and above (the high concentration ink dispersions). Porter observed this same trend when ultrafiltering colloidal suspensions (electro deposition primer and styrene butadiene latex), including the tendency for the flux vs. ln(C\text{f}) line to be concave up, which he attributed to variations in shear forces due to viscosity increasing as the feed was concentrated\textsuperscript{18}. The flux values for the low concentration dispersions (those containing less than 0.9% ink) do not follow the same relationship, and represent a discontinuity in the trend of exponential decrease in flux with increasing feed concentration. This data show that flux is strongly affected by feed concentration when processing high concentration dispersions, which indicates that the gel layer is the limiting resistance to permeate flux. Relative independence of flux on feed concentration was observed when processing low concentration dispersions, indicative of operating under conditions in which the gel layer is not the limiting resistance to permeate flux\textsuperscript{19}.

The data in figure 8 were extrapolated to zero flux, predicting that the gel concentration was approximately 30% solids. Figure 9 shows plots of flux vs. ln(C\text{f}) at 12 psi and at 18 psi, which predict the gel concentration of 67% and 42%, respectively. The data at low pressures predict a higher gel concentration than do the data at high pressures, indicating that flux is less strongly affected by solid content at low pressures. However, it is at high ΔP\text{m} when pressure independent gel controlled ultrafiltration is observed. Therefore the C\text{f} values predicted by high ΔP\text{m} data are probably more realistic. Porter's data for ultrafiltration of colloidal suspensions predicted C\text{f} between 60% and 70%, but the operating conditions during his experiments were not stated\textsuperscript{19}. Desaulniers and Hausslein attained a maximum solids content of 31% when ultrafiltering an aqueous dispersion of activated carbon particles (median size 11 µm) in a batch cell using an ultrafiltration membrane with MWCO of 250,000\textsuperscript{20}. They also determined that the carbon filter cake on the membrane surface controlled the filtration rate, and not the membrane itself.

The effect of flowrate on permeate flux during cross flow ultrafiltration is due to the shear forces exerted by flowing fluid at the wall of the membrane, which is instrumental in preventing or controlling the size of a layer of retained pigment on the membrane surface\textsuperscript{13,15,18,19}. Any pigment layer which forms on the membrane surface will act as a barrier to permeate flow and thus affect flux negatively. At high
flowrates the shear forces will be high, and thus the formation of a gel layer will be retarded. The effect of feed flowrate on ultrafiltration performance was investigated by processing a 4.6% flexographic ink dispersion at three flowrates (0.5 gpm, 1.0 gpm, and 1.5 gpm). The experiment was performed in a randomized manner to preclude any time effects (such as membrane fouling) from entering into the analysis. The desired conditions (flowrate and ΔP_in) were tabulated and numbered, and a random number generator was employed to randomize the order in which the data were obtained. The data in figure 10 demonstrate that better ultrafiltration performance (higher flux) is achieved when operating at high feed flowrates, and that the onset of pressure independent flux occurs earlier when operating at low flowrates. However, since the data were collected in a random order, complete steady state operation (including formation of stable pigment layers on the membrane surface) may not have been achieved. Therefore the effect of flowrate may be greater than that indicated by these results.

Effectiveness of Cleaning

The ultrafiltration membrane was cleaned after each ink dispersion was processed to ensure that a similar baseline performance was achieved with the membrane prior to processing the next dispersion, and also to evaluate the severity of any performance degradation which may have occurred. Membrane performance was characterized by the water flux, which is the flux when processing a 0.01% surfactant solution in water with the pH adjusted to 10, at 25 psi, 20°C, and 1 gpm feed flowrate. The following is a sequential summary of ultrafiltration experiments and the effectiveness of the cleaning procedure performed after each experiment. Figure 11 shows the post cleaning water flux after processing each of the ink dispersions.

When new the ultrafiltration membrane demonstrated a water flux of 400 l/m² hr. After processing the first ink dispersion (0.05%, ink #1), the water flux after cleaning had decreased to 90 l/m² hr. This represents significant membrane fouling which was not reversible upon membrane cleaning. This trend continued (figure 11, bars 2-6), and after processing the other low concentration dispersions of ink #1 (0.09%, 0.18%, 0.195%, and 0.37%), the final water flux was 104 l/m² hr, measured after the membrane had been allowed to soak in a cleaning solution for one month.

Higher concentration ink dispersions prepared from ink #2 were then processed. The water flux after processing a 0.9% ink dispersion and cleaning the membrane was 430 l/m² hr. This is a higher water flux than that measured with the new membrane, indicating that processing this ink dispersion actually had beneficial effects on membrane performance. Subsequent processing of other dispersions prepared from ink #2 (1.8%, 3.6%, 6.6%, and 9.8%) also resulted in high water flux values after cleaning, as shown in figure 11 (bars 7-11). The final water flux after processing the highest concentration ink dispersion (9.8%) was 415 l/m² hr.

Low concentration dispersions prepared from ink #2 were then processed. The water flux after processing a 0.05% dispersion and cleaning the membrane was 111 l/m² hr, indicating that irreversible membrane fouling had occurred. Two more low concentration dispersions were processed (0.1% and 0.4%), and as the ink content of the feedstream increased the post cleaning water flux followed, indicating that the membrane was becoming easier to clean (figure 11, bars 12-14). The final water flux after processing the 0.4% dispersion was 213 l/m² hr.

The next dispersion processed, which contained between 0.2% and 1% ink, was processed through the ultrafiltration membrane for approximately 30 minutes. The membrane was then cleaned and allowed to soak in a cleaning solution for approximately two months. The resulting water flux was 672 l/m² hr, which is the highest yet measured (figure 11, bar 15). At this point a series of experiments was performed using a 0.2% ink dispersion in which the feed temperature was varied. The results of the temperature study will not be presented here, but the results of the membrane cleaning procedures are quite interesting. The experiments were performed at 20°C, 35°C, 50°C and 65°C. Figure 11 (bars 16-19) shows the water flux values after each of these experiments, which demonstrate that high temperature processing was responsible for extensive membrane fouling.

Apparently there was some property of the high ink content dispersions which promoted membrane cleaning and restored the water flux, whereas processing low ink concentration dispersions tended to foul the membrane. In order to investigate this a 3.5% ink dispersion was processed at 25 psi, and 1 gpm (figure 12). During the first four hours the temperature increased from 26°C to 38°C, and was returned to 26°C over the next hour. Flux increased from 10 l/m² hr to 56 l/m² hr during the first four hours of the experiment (at this point the temperature was 38°C). When the temperature was returned to 26°C the flux decreased to 45 l/m² hr (demonstrating the effect of temperature on flux).

Afterwards the membrane was cleaned, resulting in a water flux of 350 l/m² hr. The water flux had increased from 57 l/m² hr to 350 l/m² hr, an increase of 514% (see figure 11, bar 20).

This is a very important finding. A similar experiment using a 3.9% ink dispersion and an identical ultrafiltration membrane (UF2) which had been fouled by processing low concentration ink dispersions at 65°C (the water flux of the membrane was 48 l/m² hr) was performed at 20°C (figure 13). Flux increased from 12 l/m² hr to 45 l/m² hr after 160 minutes. This is almost identical to the performance observed during the previous experiment using ultrafiltration membrane UF1. Afterwards
the membrane was cleaned, resulting in a water flux of 315 l/m² hr, an increase of 556%.

These results demonstrate that ultrafiltration of high concentration ink dispersions has beneficial effects on membrane cleaning. One would expect a suspension rich in submicron carbon black particles to foul the membrane in a thorough fashion! Although the pH and amount of surfactant added to the feed was maintained constant, the ink contains materials in addition to pigment which could affect ultrafiltration performance. These materials include binder resins, drying agents, and others, which have surface active properties. Higher ink content feedstreams prepared from commercial newsprint ink will contain not only higher concentrations of pigment particles, but also more surface active compounds, which may impart cleaning properties to high ink content dispersions.

**Stable Flux During High Concentration Ink Processing**

The flux vs. ΔP experiments consisted of collecting flux data during at least two complete pressure cycles. Typically during the first pressure cycle a great degree of hysteresis was observed as the membrane system stabilized. When processing low concentration ink dispersions, lower flux values were observed during the second pressure cycle than during the first pressure cycle, indicating that flux decreases with time. This effect was most noticeable for the 0.05% ink dispersion (the lowest concentration processed).

The flux vs. ΔP data from processing high concentration ink dispersions show different trends. Higher flux was attained during the second pressure cycle than during the first pressure cycle for each dispersion which contained 0.9% ink or more. This effect was less noticeable for the 6.6% and 9.8% ink dispersions, but was quite pronounced for the 0.9% and 1.8% dispersions. The 0.9% dispersion was processed through three pressure cycles, with maximum fluxes observed of 72 l/m² hr, 110 l/m² hr, and 130 l/m² hr during the first, second, and third pressure cycles, respectively. This indicates that flux increased with time.

A plot of flux vs. time for ultrafiltration of a 0.055% dispersion of ink #1 at constant pressure (25 psi) and temperature (22°C), with the permeate recycled back to the feed tank so that the ink concentration remained constant. The initial flux was 91 l/m² hr and decreased to 41 l/m² hr over a five hour period. When processing low concentration ink dispersions the flux tended to decrease with time due to membrane fouling. Over the next 60 minutes the flux changed very little. During ultrafiltration of a 3.5% ink dispersion, the flux decreased from 71 l/m² hr to 58 l/m² hr in the first 30 minutes, and then changed very little during the next 30 minutes with a final flux of 56 l/m³ hr after one hour of operation (figure 15). A 3.9% ink dispersion was processed using ultrafiltration membrane UF2. The flux decreased from 78 l/m² hr to 58 l/m² hr after only 20 minutes of operation (see figure 16). Continued processing resulted in the flux decreasing only slightly to 56 l/m² hr after 70 minutes of operation. These data demonstrate that stable flux can be achieved when processing high concentration ink dispersions. Not only is flux more stable with time, but higher flux levels were attained when processing the high ink content dispersions than when processing the low ink content dispersions. Mer one hour of ultrafiltration of a 0.055% ink dispersion, the flux had decreased to 65 l/m² hr and was still decreasing, as shown in figure 14. The final flux was 41 l/m² hr. Ultrafiltration of a 3.5% dispersion with the same membrane under identical conditions for one hour resulted in a stable flux value of 56 l/m² hr, over 36% higher than that achieved when processing the 0.05% dispersion.

The stable high flux values observed when processing high concentration ink dispersions could be due to a filtering action of the gel layer of pigment formed during pressure independent ultrafiltration. The pigment particles in flexographic inks are very small, with 90% of the particles less than 5 µm in size, with a considerable fraction below 0.2 µm. The smallest fraction of these particles could be small enough to enter, or at least partially block, some of the pores in the ultrafiltration membrane, reducing flux irreversibly. However the gel layer could act as a filter aid, preventing even the smallest pigment particles from reaching the membrane surface. Closely packed cakes of micron or sub-micron particles have high hydraulic permeabilities. If the gel layer is acting as a filter aid it would enable high flux levels, while promoting long term stability of flux by preventing membrane fouling. Membrane regeneration would also be facilitated if fouling can be prevented.

Effective clarification of flexographic ink dispersions entails concentrating the pigment to as high a level as possible as permeate (water and other dissolved species) is removed, while maintaining high flux rates. In order to evaluate the capabilities of ultrafiltration to achieve this goal, an experiment was performed in which permeate was removed from the system as it was produced, causing the ink concentration of the feed to increase as ultrafiltration progressed (figure 17). The initial ink concentration was 4.4%, and increased to 22.2% after 755 minutes of operation. After an initial rapid drop, flux decreased gradually as the ink concentration increased. At an ink concentration of 22.2% the flux had decreased to 18 l/m² hr.

A plot of flux vs. bulk feed concentration constructed from this data (figure 18) indicates that the gel concentration under these flow conditions was 50%. As C$_s$ approaches C$_e$, where
pressure independent operation predominates, the predicted value of \( C_d \) decreases. The ink dispersions clarified by ultrafiltration are complex colloidal suspensions, in which the primary particles are carbon black pigment, the majority of which are submicron in size. Flexographic ink dispersions also contain binder resins, defoaming agents, drying agents, and other materials, which complicate the interpretation of experimental results. In the above described experiment, as the pigment content of the feed increased, concentrations of the other materials increased also, but may not have increased proportional to the pigment concentration. Dissolved species may be removed with the aqueous permeate. In a continuous process where dilute ink is continuously added, while a permeate stream (clean water for re-use) and a solids rich stream (containing concentrated pigment) are continuously removed, the composition of the liquid phase would be determined by the degree of retention of all materials in the liquid. Nonetheless, composition of the liquid phase undoubtedly affects ultrafiltration performance. The important parameters have yet to be fully investigated.

Addition of Fines

In order to evaluate the potential effects of cellulose fines on ultrafiltration of flexographic ink dispersions, a series of experiments was performed in which fines were added to the ink dispersion. Constant pressure ultrafiltration (25 psi at 1 gpm) for one hour was followed by a pressure cycle in which the flux vs. \( \Delta P_{in} \) relationship was investigated. Cellulose fines were then added and the experiment repeated. The membrane was cleaned between each processing step to evaluate the effect of processing with and without fines on cleaning effectiveness.

A 3.5% ink dispersion was processed by ultrafiltration membrane UF1. The data are presented in figures 19 and 20. These results indicate that the presence of low amounts of cellulose fines decreased the ultrafiltration performance (flux levels), however the effectiveness of membrane cleaning did not appear to be significantly affected. The after cleaning water flux after processing these feeds were 290 l/m² hr (no fines), 302 l/m² hr (0.025% fines), and 306 l/m² hr (0.05% fines).

A 3.9% ink dispersion was processed by ultrafiltration membrane UF2 with results shown in figures 21 and 22. These results indicate that the presence of low amounts of cellulose fines did not significantly affect the ultrafiltration performance (flux levels), in contrast to the results when ultrafiltering using UF1. The presence of 0.025% fines had virtually no effect on ultrafiltration performance when using membrane UF2, and the presence of 0.05% fines had only a minimal effect. The effectiveness of membrane cleaning was not affected by fines when processing with membrane UF2, in agreement with the results obtained from membrane UF1.

The after cleaning water flux after processing these dispersions were 276 l/m² hr (no fines), 262 l/m² hr (0.025% fines), and 285 l/m² hr (0.05% fines).

CONCLUSIONS

During ultrafiltration of flexographic ink dispersions the permeate was free of ink pigments, demonstrating the capability of ultrafiltration to completely remove flexographic residues from wash effluent.

Flux was directly proportional to \( \Delta P_{in} \) during ultrafiltration of low concentrations of flexographic ink (less than 0.4%), and high ink concentrations (greater than 0.9%) at low operating pressure. This is indicative of operating under conditions in which the membrane, and not a gel layer of pigment, presents the limiting resistance to permeate flux.

Flux is relatively independent of ink concentration at low concentrations. At feed concentrations at 0.9% and above flux decreased exponentially with increasing ink concentration.

Ultrafiltration of low concentration flexographic ink dispersions resulted in irreversible membrane fouling. Performance could not be restored by cleaning after processing these dispersions. The cleaning procedures appeared to be quite effective after processing high concentration flexographic ink dispersions. In fact the processing of high concentration dispersions had beneficial effects on cleaning, and restored high flux levels to ultrafiltration membranes which had previously been fouled by processing low concentration dispersions.

Flux was observed to decrease with time when processing low concentration flexographic ink dispersions, indicative of membrane fouling. However, when ultrafiltering high concentration dispersions the flux was stable with time. In some cases higher flux was obtained by increasing the ink content of the feedstream. This indicates that the gel layer prevents small pigment particles from fouling the ultrafiltration membrane.

The addition of small amounts of cellulose fines to flexographic ink dispersions did not drastically affect the ultrafiltration performance when clarifying these dispersions. Cellulose fines had no effect on membrane fouling, as demonstrated by the effectiveness of the cleaning procedure after processing ink dispersions containing fines.

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Fig. 1. Proposed Flow Diagram

Fig. 2. Ultrafiltration Apparatus

Fig. 3. Flux vs. $\Delta P_{m}$ for Ink #1

Fig 4. Flux and Percent Ink Content vs. Time
Fig. 5. Flux vs. $\Delta P_m$ for Ink #2, Low Concentration

Fig. 6. Flux vs. $\Delta P_m$ for Ink #2, High Concentration

Fig. 7. Flux vs. $\Delta P_m$ for 3.6% Ink #2

Fig. 8. Flux vs. % Ink Solids at 24 psi.
Fig. 9. Flux vs. % Ink Solids at 12 psi and 18 psi.

Fig. 10. Flux vs. $\Delta P_m$ at Three Flow Rates. 4.6% Ink Dispersion

Fig. 11. Water Flux After Processing Flexographic Ink Dispersions

(* After Extended Soaking)
Fig. 12. Flux and Temperature vs. Time while Processing a 3.5% Dispersion at 25 psi

Fig. 13. Flux vs. Time while Processing a 3.9% Dispersion at 25 psi and 20°C

Fig. 14. Flux vs. Time while Processing a 0.055% Dispersion at 25 psi and 22°C

Fig. 15. Flux vs. Time while Processing a 3.5% Dispersion at 25 psi and 20°C
Fig. 16. Flux vs. Time whole Processing a 3.9 Dispersion at 25 psi and 20°C

Fig. 17. Flux and % Ink Solids vs. Time at 25 psi and 20°C

Fig. 18. Flux vs. % Solids in Feed

Fig. 19. Flux vs. Time, Ultrafiltration of 3.5% dispersion With and Without Cellulose Fines (25 psi 20°C, UF1)
Fig. 20. Flux vs. $\Delta P_m$ Ultrafiltration of 3.5% Dispersion With and Without Cellulose Fines (25 psi 20°C, UF1)

Fig. 21. Flux vs. Time, Ultrafiltration of 3.9% Dispersion With and Without Cellulose Fines (25 psi 20°C, UF2)

Fig. 22. Flux vs. $\Delta P_m$ Ultrafiltration of 3.9% Dispersion With and Without Cellulose Fines (25 psi 20°C, UF2)