Coagulation Pretreatment for Ultrafiltration of Deinking Effluents Containing Flexographic Inks

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ABSTRACT

This study was carried out to determine the potential of coagulation pretreatment with organic or inorganic coagulants to improve ultrafiltration performance during processing of wash deinking effluents containing flexographic inks. Wash filtrate effluents generated from mixtures of old flexographic and offset newspapers and old magazines were pretreated with a cationic coagulant and alum before ultrafiltration. Membrane performance was assessed from permeate flux, fouling rate, and ease of membrane regeneration. Coagulation pretreatment of wash filtrate effluents with a commercial cationic coagulant or alum significantly improved ultrafiltration and reduced fouling up to a coagulant concentration of 10 mg/L. Addition of an excessive amount of a cationic coagulant had a very detrimental effect on membrane performance. Alum had a better effect on permeation rate with less fouling tendency compared to the cationic coagulant studied. The methods of analysis used to determine dissolved and colloidal substances removals were not sensitive enough to establish a correlation between substance removal and ultrafiltration performance.

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KEYWORDS

Coagulant, Deinking, Effluent, Flexography, Magazines, Newspapers, Ultrafiltration, Washing.

INTRODUCTION

The problem associated with deinking of flexo-printed newspapers remains unsolved and still presents a unique challenge to the deinking operations. It is well established that conventional deinking systems such as flotation and washing processes have limited capacity for the treatment of recovered paper furnishes containing flexo-printed newspapers. Under alkaline repulping conditions, flexographic inks cannot be efficiently removed by flotation because fibers interfere with flotation by preventing the growth of ink aggregates (1) or tend to redeposit on fibers during washing or thickening stages (2-4). Although washing technology has been shown to effectively remove flexographic inks from pulp suspensions (3, 5), large quantities of filtrate are produced which cannot be recycled to the deinking process without detrimental effects on deinking operations and deinked pulp quality. Dissolved air flotation (DAF) units most commonly used for the clarification of gray water in deinking mills are also adversely affected by the presence of flexographic inks, requiring significantly higher levels of coagulant to achieve a proper clarified water quality (6-8) Consequently, lower ink removal in the DAF process increases the build-up of fine ink particles in the water loops and causes a drastic reduction in deinked pulp brightness.

The application of membrane separation technology for clarification of flexographic ink laden water has been investigated by Upton et al. (9, 10). Recently, Chabot et al. (11) have studied the potential of ultrafiltration to clarify process water from a flexo-printed newspaper deinking facility. Results indicated that ultrafiltration is capable of completely removing ink pigments from an effluent stream, producing clear permeate. However, permeation rate and fouling tendency were shown to be dependent on effluent composition and operating conditions. Flux decline caused by concentration polarization and irreversible fouling of the membrane affects ultrafiltration performance and is one of the major practical limitations of ultrafiltration operation (12). Various strategies for controlling fouling problems such as modification of the membrane, pretreatment of the feed, or adjustment of the operating conditions have been discussed by Fane and Fell (13).

Coagulation pretreatment has been suggested as a means to improve flux rates and reduce fouling for ultrafiltration of various process waters (14-16). Results have shown that partial removal or deactivation of dissolved and colloidal substances (DCS) improved permeation rates during ultrafiltration. Since typical deinking wastewaters contain a wide variety of suspended and dissolved organic and inorganic materials such as latex, fatty acids, tackifiers, resins, and hot melts which both influence deinking processes and deinked pulp quality (17), it is likely that those substances would also interfere with the performance of the membrane during treatment of deinking effluents. Coagulation pretreatment can thus be thought of as a process with the potential of converting dissolved organic matter into a filterable particulate phase larger than the pore size of the membrane.

The objective of this work was to determine if coagulation pretreatment of wash filtrate effluent can improve flux rate and reduce fouling. Furnishes containing flexographic and offset newspapers (ONP) and old magazines (OMG) were deinked under alkaline conditions. Coagulation pretreatments of wash effluents using organic and inorganic coagulants were carried out prior to ultrafiltration. Bench top ultrafiltration apparatuses were used to clarify wash filtrates. Effectiveness of these systems were evaluated by monitoring flux rates, fouling, and permeate clarity. The reductions of some measurable parameters of the effluents were also determined.

EXPERIMENTAL

Deinking

Recovered paper furnishes consisting of 70% ONP and 30% OMG were used throughout the study. The flexo-printed newspaper content was adjusted between 10% and 70%.

Repulping was carried out in a laboratory pulper (Adirondak Machine Corp., Glens Falls, NY) operated for 15 min at a pulp consistency of 3% and a temperature of 45 °C. Deinking chemicals consisting of 1% sodium hydroxide, 1% sodium silicate (42 °Bé), and 0.1% nonionic surfactant were used. All chemical dosages were based on an oven-dry paper basis. After repulping, the pulp slurry was diluted with tap water to 0.8% consistency. Flotations were then carried out in a Denver D12 flotation cell (Svedala, Colorado Springs, CO) for 5 mm at 1200 rpm and 45 °C. Foam was manually scraped off from the surface of the cell. The accepted pulp from the flotation cell was kept for thickening. The pulp suspension (accepts) was diluted with tap water to 0.45% consistency prior to thickening. Thickening was carried out on a 150 mesh screen in order to increase the pulp consistency from 0.45% to about 12%. The filtrate was collected for coagulation pretreatment and ultrafiltration treatments.

Coagulation Pretreatment

The wash filtrate effluent was first prefiltered using a bag filter (Cole-Parmer, Vernon Hills, IL) with a nominal pore size of 100 µm to remove fibers and fines. Then, coagulation tests were conducted to identify the most effective coagulant based on the reductions of turbidity, conductivity, lignin content, cationic demand, and total solids. A commercial low molecular weight cationic coagulant with a charge density of 5.08 mequ./g and alum were selected for experimentations. Tests were performed according to the standard jar test procedure described previously (11).

Ultrafiltration Procedure

Experiments were carried out with two bench-top laboratory membrane filtration devices. Preliminary trials were first performed with a polysulfone hollow fiber membrane (Koch Membrane System, Wilmington, MA) with a nominal molecular weight cut-off of 500,000 Da, and an effective surface area of 0.0929 m^2. The membrane apparatus has been described in earlier paper (11). Experiments were performed at 45 °C. Transmembrane pressure (ΔP_{TM}) and cross-flow rate were maintained constant at 172 kPa and 3.78 L/min, respectively. Further experiments were also carried out with a polyethersulfone membrane cassette (Ultrasette, Pall Filtron Corp., Northborough, MA) with a nominal molecular weight cut-off of 100,000 Da. The cassette was an open channel flat membrane with a surface area of 0.07 m^2. The process water was delivered to the UF unit by a peristaltic pump with variable speed drive (Masterflex L/S, Barnant Co., Barrington, IL). Experiments were conducted at 45 °C, and at a transmembrane pressure (ΔP_{TM}) and cross-flow rata of 172 kPa and 1.8 L/min, respectively. In both experimental set-ups, retentate and permeate were recirculated to the feed container to keep the feed concentration constant. Flux was measured gravimetrically: by collecting permeate in a tared beaker on a digital balance at 5 or 10 min intervals.

In preliminary experiments with the hollow fiber cartridge, the pure water flux before filtration was determined. In the. second series of experimentations, the pure water flux before filtration and after filtration was measured in order to estimate fouling effect. Cleaning of the membrane was carried out after each experiment to restore membrane performance. The pure water flux after cleaning was also measured.

Methods of Analysis

For each experiment, feed samples (before and after coagulation pretreatment), and a permeate sample were collected. Samples were filtered on a Whatman 934-AH glass-fiber filter paper (Whatman Inc., Clifton, NJ) to remove suspended solids. Filtrates were collected and then analyzed for pH, conductivity (YSI 35, Yellow Springs, OH), turbidity (Hach 2100N, Loveland, CO), total solids (gravimetry), lignin residuals (UV absorption at 280 nm and 400 nm), and cationic demand (back titration with an excess of 0.001N p-DADMAC (cationic polymer) and 0.001N polyvinyl sulfate (anionic polymer) solutions (18, 19). Analysis of samples were carried out the same day as the experiment was performed in order to limit variation of the content with time or contamination.

The flux reduction during ultrafiltration was calculated according to:

$$FR = \left(1 - \frac{J_F / J_{0b}}{J_I / J_{0b}}\right) \times 100 \qquad (1)$$

where

FR = Flux reduction, %
J_F = Wastewater flux at the end of the ultrafiltration, L/m^2-h
J_I = Wastewater flux at the beginning of the ultrafiltration, L/m^2-h
J_{ob} = Pure water flux before ultrafiltration, L/m^2-h
J_F / J_{ob} = Relative waste water flux at the end of the ultrafiltration (70 min.)
J_I / J_{ob} = Relative waste water flux at the beginning of the ultrafiltration (2 min.)

The fouling effect was determined by calculating the pure water flux (PWF) reduction before and after the ultrafiltration as follows:

$$FR_{PWF} = \left(1 - \frac{J_{0a}}{J_{0b}}\right) \times 100 \qquad (2)$$

where

FR_{PWF} = Flux reduction of pure water flux, %
J_{oa} = Pure water flux after ultrafiltration, L/m^2-h
J_{ob} = Pure water flux before ultrafiltration, L/m^2-h

The effectiveness of the cleaning procedure on restoring membrane performance was determined by calculating the reduction of the pure water flux before and after cleaning the membrane as follows:

$$E_c = \left(\frac{J_{0c}}{J_{0b}}\right) \times 100 \qquad (3)$$

where

E_c = Efficiency of the cleaning procedure, %
J_{oc} = Pure water flux after cleaning, L/m^2-h
J_{ob} = Pure water flux before ultrafiltration, L/m^2-h

The reductions of measured substances were calculated by comparing the content in the permeate with the content in the feed as follows:

$$R = \left(1 - \frac{C_P}{C_F}\right) \times 100 \qquad (4)$$

where

R = Reduction, %
C_P = Concentration in the permeate
C_F = Concentration in the feed

RESULTS AND DISCUSSION

Coagulation Pretreatment Before Ultrafiltration

Preliminary trials with a hollow fiber ultrafiltration membrane were first carried out. Figure 1 shows flux decline with time for feed effluents containing various flexographic ink contents. Effluents were not subjected to coagulation pretreatment before ultrafiltration.

In all cases, flux decreased rapidly within 10 min of operation. After this rapid initial decay, the rate of flux decline progressively lessened until a near equilibrium filtration rate was achieved. Results showed that increasing the proportion of flexo-printed newspapers in the furnish had a significant effect on membrane performance. Flux reduction, which is a measure of how fast the permeate flux decreases during filtration, was reduced at higher flexo ink content suggesting that some components of the offset newspapers had a very detrimental effect on permeation rate. Similar behavior was reported for furnishes containing newspapers only (11). Although the flux behavior observed is typical of membrane filtration processes, it is likely that concentration variations of the dissolved and colloidal substances in each of the feed effluents affected the ultrafiltration membrane performance. Typical deinking effluents contain a variety of organic dissolved and colloidal substances derived mainly from paper chemicals added in the previous cycle. These include latex, hot melt adhesives, tackifiers, resins, fatty acids, as well as inorganic components such as calcium and aluminium compounds, tine minerals, and silicates (17, 20-21). However, offset newspapers which are printed with oil-based inks contain oil binders which could possibly be adsorbed on the polysulfone membrane reducing their performance severely. It is well known that hydrophobic solutes such as low molecular weight oil components or fatty acids are readily adsorbed on hydrophobic polysulfone membranes decreasing considerably permeation rates (14, 22-24). These studies have shown that adsorption of hydrophobic compounds was mainly responsible for membrane fouling. Reversible fouling was caused by the deposition of contaminants on the membrane surface (concentration polarization) while irreversible fouling was caused by deposition of macromolecules within the membrane structure pores (22). Therefore, both phenomenon are likely to be responsible for the extensive reduction of membrane performance observed in Fig. 1.

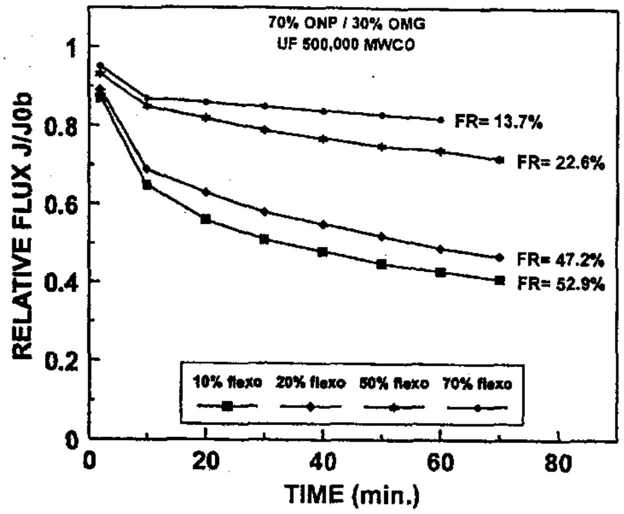

Figure 1: **Permeate flux decline for feed effluents containing various proportions of flexo-printed newspapers. No *coagulation* pretreatment.**

In order to improve flux rates of the ultrafiltration membrane during treatment of wash deinking effluents, coagulation pretreatments with organic and/or inorganic coagulants were carried out before ultrafiltration. Figure 2 presents preliminary trials with a deinking effluent generated from a recovered paper furnish consisting of 20% flexographic newspapers, 50% offset newspapers, and 30% magazines. A catiomc coagulant (20 mg/L) or alum (pH adjusted to 4.5) were added to the feed prior to ultrafiltration.

Results showed that addition of the cationic coagulant or alum improved significantly permeation rates, compared to feed without any pretreatment. Flux decreased at a lower rate for pretreated effluents, indicating that coagulation pretreatment reduced the membrane fouling potential during

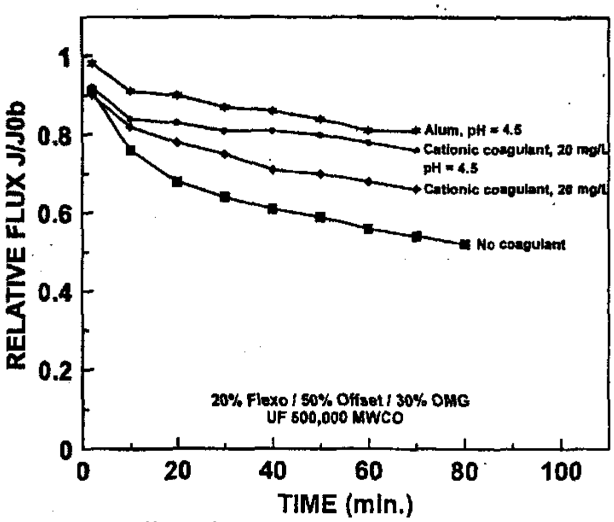

Figure 2: **Effect of coagulation pretreatment on permeation rate for printed wastepaper d&king effluents.**

Table 1. Flux Reduction Values for Untreated and Pretreated Effluents of Printed Recovered Paper Furnishes.

Experiment	J_I / J_{ob}	J_F / J_{ob} [a]	FR (%)
No coagulant	0.91	0.54	40.7
Cationic Coagulant (20 mg/L)	0.90	0.66	26.7
Alum (pH = 4.5	0.98	0.81	17.3
Cationic Coagulant (20 mg/L, pH = 4.5	0.92	0.78	15.2

[a] Final flux measured after 70 min operation.

treatment of these streams. Table 1 presents the flux reduction (FR) values for untreated and pretreated effluents under various coagulation conditions for printed wastepaper furnishes. Results clearly indicate that coagulation pretreatments decreased significantly flux reduction compared to untreated effluent resulting in higher permeation rates at steady state and possibly less fouling potential as shown in Fig. 2.

Alum produced better flux improvement compared to the cationic coagulant used. However, this behavior may be attributed to a pH effect as shown by the higher final relative flux values or comparable flux reduction (FR) achieved with the cationic coagulant at acid pH. Improvement in flux rates may be attributed to the removal and/or neutralization of dissolved and colloidal substances in the feed effluent before ultrafiltration. Since the majority of those substances carry a negative electrostatic charge, destabilization and subsequent agglomeration of these materials into larger aggregates by cationic compounds reduced fouling of the membrane during ultrafiltration treatment.

Cleaning efficiency (E_c) can be used to qualitatively evaluate the fouling propensity of the ultrafiltration membrane. Figure 3 presents the pure water flux restoration or the cleaning efficiency for each pretreatment condition studied. Results showed that membrane permeability decreased during various treatments while cleaning efficiency remained relatively high, indicating, that irreversible fouling occurred during filtration. Coagulation pretreatment with a cationic coagulant may affect fouling of the UF membrane as shown by the lower cleaning efficiency achieved after filtration of pretreated feed. Fouling is mainly caused by adsorption of substances onto the walls of the membrane pores and pore plugging, or by the deposition of solute molecules at the surface of the membrane where a cake is formed (12). Adsorption is determined by the membrane material and solute interactions, while the degree of pore plugging depends on the relative size of solute and pore (25). Moreover, solutes are more readily adsorbed onto hydrophobic membrane surfaces such as the polysulfone membrane used in this series of experiments (12). Since the UF membrane used had a molecular weight cut-off (MWCO) of 500,000 Da, it means that any dissolved or colloidal material with a molecular weight lower than 500,000 could permeate through the pores with the solvent and subsequently interact with the membrane surface, reducing the effective pore diameter. Electrostatic interaction between solute molecules and membrane surface could also partly explain the lower flux restoration observed. It is possible that the amount of cationic coagulant used (20 mg/L) for pretreatment was too high Therefore, free cationic polymer molecules which are bearing positive charges were possibly adsorbed on the negative sites of the polysulfone membrane resulting in partial fouling of the membrane. However, it is clear that partial removal of dissolved and colloidal substances with both organic and inorganic coagulants improved ultrafiltration performance.

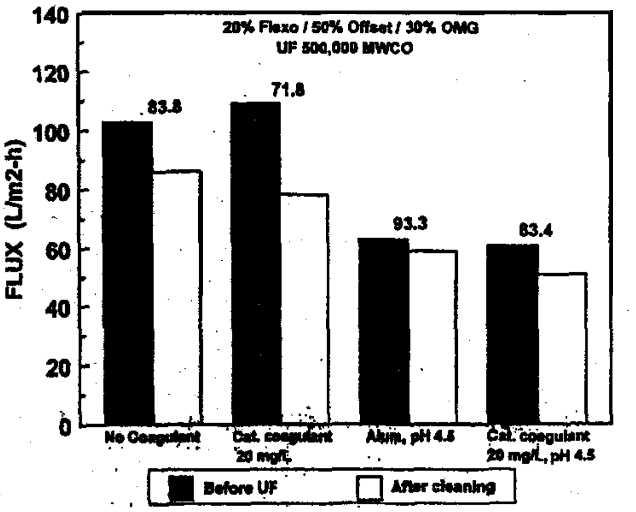

Figure 3: **Effect of coagulation pretreatment on membrane fouling potential for printed wastepaper deinking effluents. Values reported on the top of the bars are the cleaning efficiency, (E_c) of the membrane after processing feed effluents.**

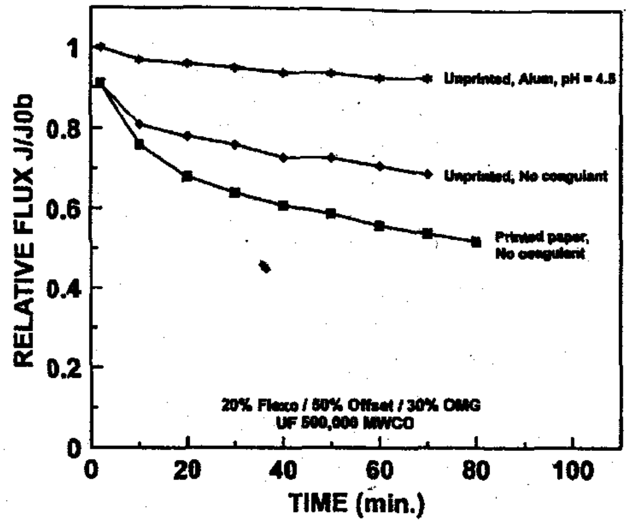

Figure 4: **Effect of coagulation pretreatment on permeation rate for unprinted wastepaper deinking effluents.**

Dissolved organic materials found in most deinking effluents may originate from recovered papers (fibers and printing inks) and from the deinking process itself. To determine their relative contributions on ultrafiltration performance, a furnish (70% ONP/30% OMG) consisting of unprinted trims from both printed papers was processed following the same experimental procedure used previously for printed papers. Results are presented in Fig. 4. Table 2 presents the flux reduction (*FR*) values for untreated and pretreated effluents under various coagulation conditions for unprinted wastepaper furnishes.

Results indicated that the flux decline occurring during treatment of effluent from unprinted recovered papers may be partly attributed to dissolved and colloidal substances present in unprinted papers. Although effluent from unprinted recovered paper furnishes resulted in a higher flux at the end of the filtration and a lower flux reduction compared to effluent from printed recovered paper furnishes, it is clear that dissolved end colloidal substances present in the former effluent have a significant contribution to the total effect observed for the printed recovered paper effluent. Therefore, flux declines shown in Fig. 1 were the result of a combination of both printing and papermaking materials contributions. Substances such as lignin, hemicelluloses, and wood extractives are generally associated with virgin pulps (26), while fine clay particles and starch as well as fatty acid and bleaching chemicals derived from the recycled raw material and the deinking operations (27). Deinked pulps may also contain colloidal synthetic stickies.

Coagulation pretreatment with alum at pH 4.5 improved significantly permeation rats as shown in Fig. 4 and Table 2. Flux decay was very low indicating that no fouling of the membrane occurred during treatment of this stream. It. is clear that alum pretreatment was very efficient at controlling detrimental substances and thus improved significantly membrane performance. Flux stability was also achieved more rapidly compared to the same untreated effluent.

Table 2. Flux Reduction Values for Untreated and Pretreated Effluents from Printed and Unprinted Recovered Paper Furnishes

Experiment	J_I / J_{ob}	J_F / J_{ob} [a]	*FR* (%)
Printed papers No coagulant	0.91	0.54	40.7
Unprinted papers No coagulant	0.91	0.69	24.2
Unprinted papers Alum (pH = 4.5)	1.00	0.93	7.0

[a] Final flux measured after 70 minutes operation

Effect of Coagulant Concentration Used for Pretreatment on Ultrafiltration Performance

In the previous series of experimentations, preliminary trials with a hollow fiber ultrafiltration membrane have shown that coagulation pretreatment of the wash deinking effluent. with organic or inorganic coagulants improved permeation rates during ultrafiltration. To further investigate this, a second series of experiments were carried out. Emphasis has been put on the removal of various components and their resulting effects on permeation rates and fouling tendency of the membrane. A cationic polymer and alum were used as coagulants. Results are presented in Table 3.

Results indicated that pretreatment, of the effluent with the cationic coagulant or alum reduced significantly the level of dissolved and colloidal substances of the resulting stream. Lignin content determined from UV absorbance was minimum for cationic coagulant concentration of 15 mg/L while the cationic demand (CD) steadily decreased with increasing amounts of cationic polymer.

Table 3. Influence of Coagulant Concentratione on Turbidity, Conductility, UV, and Cationic Remand of Recycled Pulp Filtrate.					
Coagulant	Concentration (mg/L)	Turbidity (NTU)	Conductivity (μS/cm)	UV 280 nm	CD (μequ./L)
None	0	293	213	0.691	55
Cationic coagulant	5	300	221	0.415	35
Cationic coagulant	10	320	224	0.305	32
Cationic coagulant	15	309	228	0.288	30
Cationic coagulant	20	302	231	0.336	17
Alum a		241	361	0.277	7

[a] Alum was added to reach a pH of 4.5

However, inorganic substances determined from conductivity measurementa increased slowly with cationic coagulant concentration. Although the addition of the cationic coagulant destabilized suspensions at all level studied, turbidity increased slightly probably due to light scattering of larger aggregates. Results also showed that alum was more effective than cationic coagulant for the cotrol of dissolved and colloidal substances as indicated by lower UV and CD values.

lb study the effect of pretreatment conditions on ultrafiltration performance, various concentrations of the cationic coagulant were tried. Alum was also studied for comparison. Ultrafiltration experiments were carried out with a flat membrane configuration. Permeation rates are presented in Fig. 5. Results indicated that addition of a cationic polymer or inorganic alum improved permeation rates as in previous series of experimentations with the hollow fiber membrane. However, the amount of cationic coagulant added during pretreatment had a significant effect on ultrafiltration performance. Permeation rates improved for coagulant, concentration up to 10 mg/L. Further addition decreased membrane performance.

Figure 6 clearly shows that the lowest flux decrease was achieved with a coagulant concentration of 10 mg/L. Pretreatment with lower or higher coagulant concentrations affected significantly the membrane performance. It is likely that the addition of au excessive amount of cationic coagulant decreased the ultrafiltration performance. Since these molecules bear positive charges, they were probably readily adsorbed on the negatively charged sites of the polyethersulfone membrane, resulting in a drastic reduction of the permeate flux.

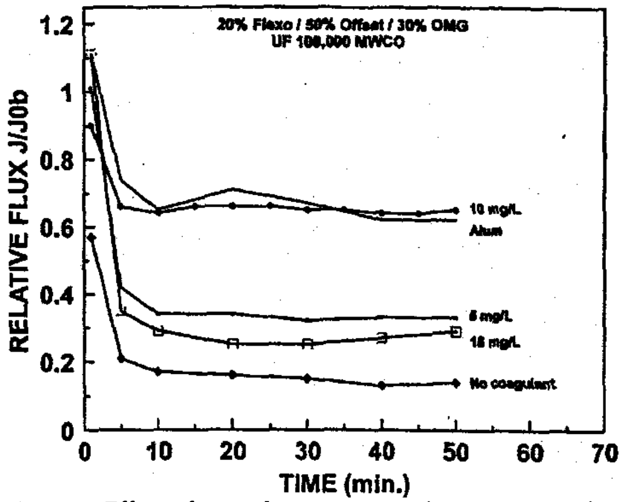

Figure 5: **Effect of coagulant concentration on permeation rate.**

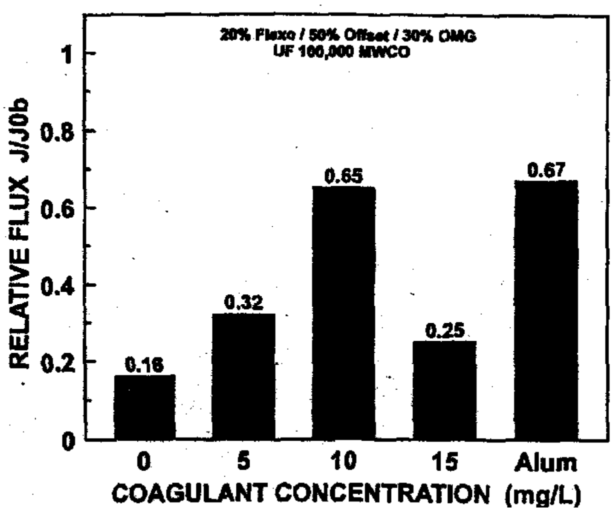

Figure 6: **Effect of coagulant concentration on relative flux at steady-state (flux measured after 30 minutes operation).**

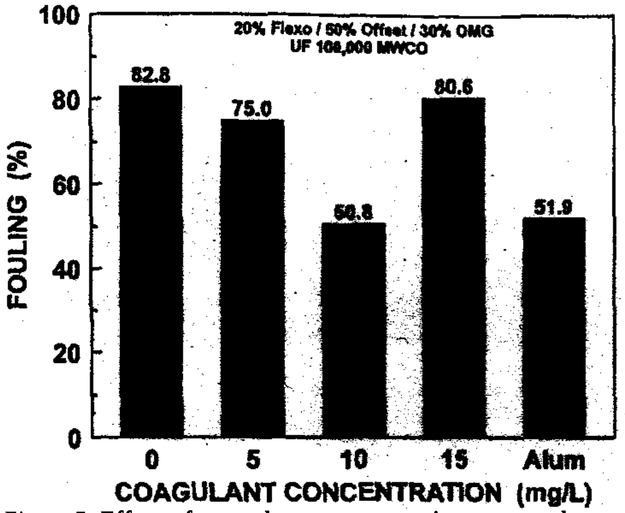

Figure 7: **Effect of coagulant concentration on membrane fouling during ultrafiltration of deinking effluents.**

Although the permeation rates decreased rapidly within 5 min of operation for all concentration tested, it seems that the coagulant concentration had a significant effect on membrane performance. Alum, which has previously been shown to better control dissolved end colloidal substances (see Table 3), produced a similar flux reduction pattern to the cationic coagulant at optimum concentration (10 mg/L) as shown in Fig. 5. Relative fluxes at steady-state (Fig. 6) were also similar indicating that both compounds effectively removed dissolved and colloidal substances and thus improved permeation rates.

Figure 7 presents the fouling effect resulting from various pretreatments studied. It is clear that addition of the cationic coagulant reduced fouling up to a concentration of 10 mg/L Further addition had a very detrimental effect on membrane fouling. Results are in perfect agreement with previous observations for permeation rates. Therefore, an optimum coagulant concentration reduced fouling of the membrane improving permeation rate. The lower fouling tendency can be attributed to the lower quantity of small particles resulting from coagulation which could block and/or tightly adsorb onto the membrane pore surface (14-16). During ultrafiltration, larger aggregates can form deposits on the membrane surface with lower specific resistance which facilitate the permeation of the solvent through the cake layer. It is also known that shear-induced lift which promotes back-diffusion of deposit materials increases with increasing particle size resulting in thinner cake layer (28). Lower fouling can also be attributed to the minimization of solute-membrane interactions (29) resulting from the pretreatment with organic or inorganic coagulants.

The effect of coagulant concentration used for pretreatments on the reductions of various substances measured in the ultrafiltration feeds and permeates is presented in Fig. 8. Results showed that feed pretreatments had no significant effect on inorganics removal (conductivity), indicating that ions cannot be retained by ultrafiltration. However, dissolved and colloidal substances such as lignin compounds (UV_{280} and UV_{400}) as well as anionic trash measured as cationic demand were more or less equally removed depending on the cationic coagulant concentration used. Although dissolved and colloidal substances slightly increased with cationic coagulant concentration, it was not pos-

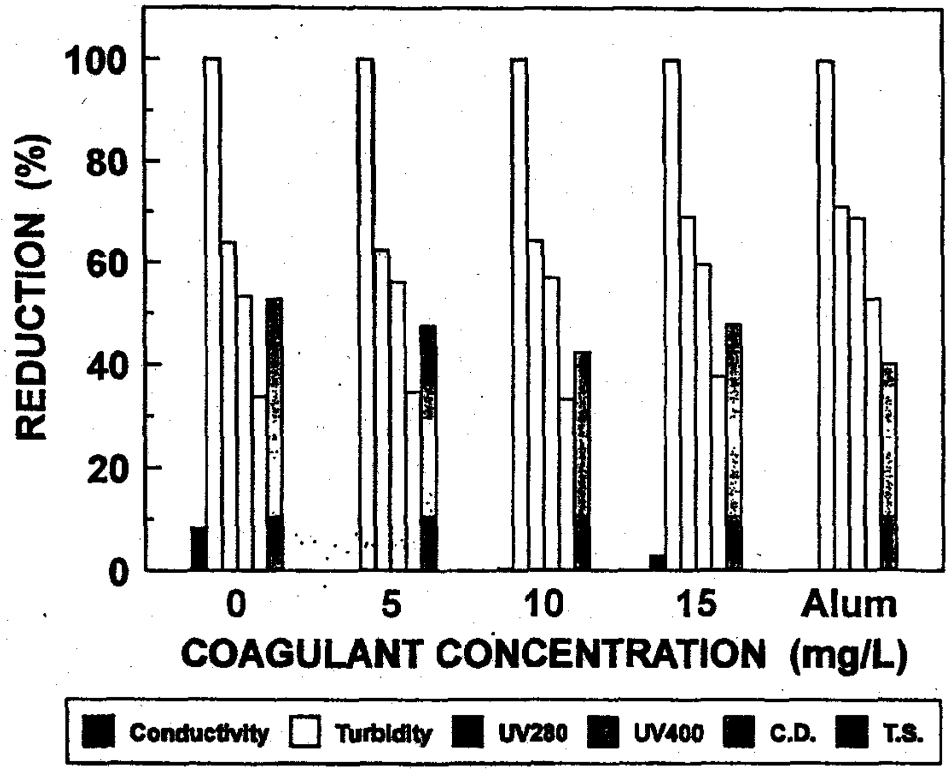

Figure 8: **Effect of coagulant concentration on reductions of detrimental substances during ultrafiltration of deinking effluents.**

sible to correlate DCS removal with flux improvement previously observed in Fig 5 or Fig. 6. Therefore, it is likely that part of the interfering substances were not detected by the methods of analysis used to determine concentrations of dissolved and colloidal substances in both feed and permeate samples.

CONCLUSIONS

This study focused on the development of an experimental procedure aimed at the improvement of ultrafiltration performances during treatment of wash deinking effluents containing flexographic inks. The results can be summarized as follows:

Coagulation pretreatment of wash deinking effluents with a commercial cationic coagulant or inorganic alum significantly improved permeation rates compared to untreated effluents. The rate of flux decay with time was lower for pretreated effluents and was mainly attributed to a lower fouling effect due to the partial removal and/or neutralization of dissolved and colloidal substances before ultrafiltration. Flux decline occurring during treatment of wash deinking effluents was mainly attributed to the presence of dissolved and colloidal substances from both printing and permaking materials. Alum pretreatment produced better ultrafiltration performance with lower flux decline and fouling effect compared to cationic coagulant.

Pretreatment of a wash deinking effluent with increasing amounts of a cationic coagulant significantly decreased the level of dissolved and colloidal substances in permeate. Ultrafiltration of those pretreated effluents showed improved permeation rates and lower fouling tendency up to a coagulant concentration of 10 mg/L. However, an excessive amount of the cationic coagulant had a very detrimental effect of membrane performance attributed to adsorption of free cationic molecules on membrane surfaces. Finally, the methods used to determine removal of detrimental substances were not adequate to establish a clear correlation between ultrafiltration improvement and substance removals.

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