

## Research Article

# Fouling of Ultrafiltration Membranes during Isolation of Hemicelluloses in the Forest Industry

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The process streams in the forest industry contain a large amount of hemicelluloses that today ends up in the wastewater. This is an unfortunate loss of a renewable raw material. The hemicelluloses can be isolated from the process stream by using membrane filtration in a process that produces purified water as a by-product, thereby facilitating increased recirculation. However, process streams from the forest industry contain both aromatic compounds and inorganic ions that are known to cause fouling of the membranes. Thus, the most suitable membrane and pretreatment from a cost-efficient point of view must be applied to avoid fouling and life-time shortening of the membranes during operation. In the present investigation, fouling during ultrafiltration of a process stream from the production of Masonite was studied. The fouling of a hydrophilic membrane made of regenerated cellulose was less severe than the fouling of hydrophobic membranes made of polyether sulphone and a composite fluoropolymer. Pretreatment of the wastewater with activated carbon resulted in higher flux and less fouling of both hydrophilic and hydrophobic membranes. Lowering the pH of the wastewater led to less severe fouling of the regenerated cellulose membrane, but lower flux of the composite fluoropolymer membrane.

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## 1. Introduction

There is a universal aim in the forest industry to reduce the consumption of fresh water by recycling process streams as far as possible. This makes it necessary to introduce artificial kidneys in order to prevent the accumulation of matter in the internal circulation systems. Membrane processes have been used as kidneys in the forest industry since the late 1960s, mainly for the treatment of white water [1], and bleach plant effluent [2, 3] in pulp and paper mills. Recently, an additional benefit when treating process streams in the forest industry has attracted attention. Process streams in the forest industry are often rich in hemicelluloses that can be used as hydrogels [4], barrier films [5, 6] and paper additives [7]. The main interest in this area has been focused on the isolation of hemicelluloses in mechanical and thermomechanical pulp mills [8–11]. When studying the potential of isolation of hemicelluloses in process streams from the Masonite process, severe fouling was observed. This does not come as a surprise as aromatic compounds, such as lignin and extractives, act as natural glue in Masonite board and have been identified

as foulants during membrane filtration of pulp and paper effluent [12–14].

The aim of this work was to study different measures to reduce fouling when treating wastewater from the Masonite process. Fouling of six ultrafiltration (UF) membranes was investigated. The membranes were made of different materials including regenerated cellulose (hydrophilic), polyether sulphone (hydrophobic), and a composite fluoropolymer (hydrophobic material modified to be hydrophilic). The influence of pH on fouling and the removal of foulants by adsorption on active carbon before ultrafiltration was studied.

## 2. Materials and Methods

**2.1. Masonite Wastewater.** Experiments were performed with wastewater from Masonite AB (Rundvik, Sweden). Masonite is a type of hardboard formed by pressing a board of long cellulose fibres at elevated temperature. The main raw material used at Masonite AB is softwood chips and cutter dust. The chips are defibrated by steam explosion and

ground in a refiner together with the cutter dust, producing a wet fibre-rich pulp. The pH of the pulp is adjusted by formic acid. A hydrophobic dispersion of paraffin wax, glue (phenolic resin), retention aids, and catalysts (aluminium and ferric sulphate) is added. The pulp is dewatered and cut into lengths. These are then pressed and heated to form Masonite boards. All process streams are collected and treated by flotation at Masonite AB. The sludge is recycled to the fibre system, and 80% of the liquid is reused in the process. The remaining 20% of the liquid is discharged. The wastewater studied in this work was withdrawn after the flotation basin and had a temperature of 60°C. It contained 1–2 g/L of hemicelluloses, various inorganic compounds in low concentration (1–6 mg/L of Al, Fe, Si, Mn, 10 mg/L Mg and 50 mg/L Ca) and the UV light absorption at 280 nm, which is a measure of the content of lignin, was around 10 absorption units. Wastewater was withdrawn at various occasions, which means that the properties of the wastewater vary slightly between the four experiments performed.

**2.2. Concentration of Hemicelluloses.** The turbidity of the wastewater was rather high, 700 Nephelometric Turbidity Units (NTUs). The wastewater was therefore prefiltered in order to remove insoluble material which could otherwise block the feed channels in the ultrafiltration module. Prefiltration was performed in a Vibratory Shear-Enhanced Processing (VSEP) unit (series L/P, New Logic, Emeryville, Calif, USA) using a microfiltration membrane with a pore diameter of 10 µm. The total membrane area was 1.57 m<sup>2</sup>. Microfiltration was carried out at room temperature and with a vibration frequency of 50 Hz, which corresponds to an amplitude of 19 mm. The transmembrane pressure must be above 200 kPa at all times to avoid damage of the membranes caused by the vibrations. To ensure this, the microfiltration was performed at 250 kPa in this study. A higher pressure was not applied since it increases the transport of particles to the membrane surface and thus the probability of the formation of a filter cake. The turbidity after microfiltration was 18 NTU. The total solids (TS) content was decreased from 0.6% to 0.5%.

Ultrafiltration was carried out in a DDS 20 plate-and-frame module (Alfa Laval A/S, Nakskov, Denmark), equipped with eight double-sided membrane discs with a total membrane area of 0.28 m<sup>2</sup>. The ETNA10 membrane (Alfa Laval A/S) is a composite fluoropolymer membrane with a nominal molecular mass cut-off of 10 000 Da.

The membranes were cleaned before the experiment, at a volume reduction of 0.7 and after the experiment. The volume reduction (VR) is defined as the ratio between the volume of permeate withdrawn and the initial feed volume. The membranes were cleaned with an alkaline cleaning agent, 0.5 wt% Ultrasil 10 (Henkel Chemicals Ltd, Düsseldorf, UK) at 50°C for about 45 minutes. The system was thoroughly rinsed with deionized (DI) water after cleaning. The pure water flux (PWF) was measured at 50°C and 1.0 MPa.

During concentration of the wastewater, the transmembrane pressure was 1.0 MPa, the temperature was

50°C, and the circulation velocity was 0.5 m/s. The same operating conditions were previously successfully used to isolate hemicelluloses from pulp mill process water [8]. The retentate was recycled to the feed tank, and the permeate was continuously withdrawn. The permeate flow rate was measured gravimetrically with a balance (PL 6001-S, Mettler Toledo Inc., Columbus, Ohio, USA). Pressure, temperature, and permeate flow rate were recorded by a PC equipped with LabView 6.0 software (National Instruments Co, Austin, Tex, USA). Data were recorded every 5 seconds.

**2.3. Membrane Screening.** The wastewater was prefiltered in the VSEP unit as described above. Ultrafiltration was performed in an equipment with four cross-flow modules connected in parallel. Each module was equipped with a circular flat membrane with an area of 0.002 m<sup>2</sup>. Different membranes were used in each module. Fouling of six membranes was studied. The characteristics of the membranes are given in Table 1. Both retentate and permeate were recirculated to the feed tank during ultrafiltration. New membranes were used in all experiments. The transmembrane pressure was 1.0 MPa. The membranes were cleaned before the experiment. Pressure, temperature, and permeate flow rate were recorded every 60 seconds. The temperature was 50°C and the permeate flow of the UP005, UC005, and PLCC membranes was measured gravimetrically with a balance (PL 6001-S, Mettler Toledo Inc.), and the permeate flow of the ETNA01, ETNA10, and NP030 membranes was measured with a PhaseSep flow meter (Phase Separations Ltd, Queensferry, UK).

Octanoic acid was used to study the hydrophobicity of the ETNA10, UP005, and UC005 membranes. Octanoic acid is a saturated fatty acid with similar properties as some of the extractives in wood. A solution with 0.5 g/L of octanoic acid was used as feed at a temperature of 20°C and a pressure of 0.2 MPa. After stabilizing the flux for 20 minutes, the flux was measured and compared to the pure water flux at the same conditions. The hydrophobicity of the membranes was reflected by the magnitude of the flux decrease.

**2.4. Adsorption by Activated Carbon.** The wastewater was prefiltered in the VSEP unit as described above. Ultrafiltration was performed in the same way and with the same equipment as used in the membrane screening but with the same type of membrane in all four modules. The pressure was 0.7 MPa in order to decrease the flux and thus reduce the transport of foulants to the membrane surface. The membranes were cleaned before and after the experiment. Adsorption with activated carbon was performed in a stirred tank for 30 minutes at room temperature. Activated carbon with a size of 3–5 mm (KEBO AB, Stockholm, Sweden) was added at a ratio of 1 : 24 (g/g wastewater). The same ratio has been used for the removal of phenolic compounds from rice straw hemicellulosic hydrolysate [15]. The activated carbon was removed by filtration through a filter paper (Quality 3, Munktell Filter AB, Falun, Sweden) before ultrafiltration. The retentate was recycled to the feed tank, and the permeate was continuously withdrawn during ultrafiltration.

TABLE 1: Properties of the ultrafiltration membranes used in the investigation. The pure water flux (PWF) was measured at 1.0 MPa and 50°C.

Manufacturer	Membrane	Material	Max. operating temperature (°C)	Cut-off (Da)	PWF (L/m <sup>2</sup> h)
Alfa Laval	ETNA01	Composite fluoropolymer	60	1000	290
Alfa Laval	ETNA10	Composite fluoropolymer	60	10 000	890
Microdyn-Nadir	NP030	Polyether sulphone	95	1000	110
Microdyn-Nadir	UP005	Polyether sulphone	95	5000	590
Microdyn-Nadir	UC005	Regenerated cellulose	55	5000	180
Millipore	PLCC	Regenerated cellulose	50	5000	200

**2.5. Adjustment of pH.** The wastewater was prefiltered in the VSEP unit as described above. Ultrafiltration was performed in a cross-flow module with a circular flat membrane with an area of 0.002 m<sup>2</sup>. The permeate flow was measured with a PhaseSep flow meter (Phase Separations Ltd). The retentate and the permeate were recycled to the feed tank

The transmembrane pressure corresponding to a pure water flux of 100 l/m<sup>2</sup>h at 50°C was measured at the pH to be investigated before each experiment. The pH of the pure water (deionized water) and the wastewater was adjusted with sodium hydroxide (50 wt%, Merck, Darmstadt, Germany) and hydrochloric acid (32 wt%, Merck) in an external tank at room temperature before ultrafiltration. The transmembrane pressure corresponding to a pure water flux of 100 l/m<sup>2</sup>h at the investigated pH and a temperature of 50°C was then used during ultrafiltration of the pH adjusted waste water. Thus, all membranes were subjected to similar conditions during the experiments. New membranes were used in each experiment. They were cleaned before the experiment.

**2.6. Analysis.** The content of total solids was determined according to the standardized method of the National Renewable Energy Laboratory (NREL, U.S. Department of Energy, USA) [16], and the turbidity was measured with a turbidimeter (HACH 2100P, Hach Lange, Düsseldorf, Germany).

The concentration of hemicelluloses was analyzed by acid hydrolysis according to the standardized method of the NREL [17]. Monomeric sugars were analyzed before and after acid hydrolysis, and the hemicellulose content was calculated from the difference in monosaccharide concentration before and after hydrolysis. Anhydro corrections of 0.9 and 0.88 were used for hexoses and pentoses, respectively. High-performance anion-exchange chromatography coupled with pulsed amperometric detection (HPAEC-PAD) using an ED40 electrochemical detector (Dionex, Sunnyvale, Calif, USA), was used to analyze the monomeric sugars. The chromatograph was equipped with a gradient pump (GP40, Dionex), an autosampler (AS50, Dionex) with a CarboPac PA1 guard, and a PA10 analytical column (Dionex). Millipore water with 2 mM NaOH was used as eluent at a flow rate of 1 mL/min, and the injection volume was 10 µL. D-mannose, D-glucose, D-galactose, D-xylose, and L-arabinose (Fluka Chemie AG, Buchs, Switzerland) were used as standards.

The content of lignin residues was measured with Shimadzu UV-160 spectrophotometer (Kyoto, Japan) at a wavelength of 280 nm. Before measurement, the samples were diluted with deionized water. The concentration of inorganic cations was determined by inductively coupled plasma atomic emission spectroscopy (ICP AES) with a Perkin Elmer (Waltham, Mass, USA) Optima 3000DV ICP AES instrument.

### 3. Results

**3.1. Concentration of Hemicelluloses.** The concentration of hemicelluloses in the prefiltered wastewater was 1.4 g/L. Mannose, galactose, and glucose residues constituted about 80% of the hemicellulose content. The ratio between these sugar residues in the wastewater equals that in the hemicellulose galactoglucomannan [18]. The remaining 20% of the hemicellulose content consisted of arabinose and xylose residues.

The wastewater was concentrated using the ETNA10 membrane. This membrane had been found to be successful when treating a process stream from a thermomechanical pulp mill in a previous study [8]. However, the flux when concentrating the Masonite wastewater declined rapidly, as shown in Figure 1. The membrane was therefore cleaned at a VR of 0.7. The flux was partially recovered after cleaning, as can be seen in the figure.

Concentration was interrupted at VR 0.99 (corresponding to a volume reduction factor VRF of 100). The content of total solids was 0.5% initially, 1.1% at VR 0.7, and 12.4% in the final retentate. The increase in the concentration of hemicelluloses in the retentate is shown in Figure 2. The concentration increases steeply at VR 0.95, as can be seen in the figure. The concentration of hemicelluloses at VR 0.7 was 4.5 g/L, and the final concentration was 65 g/L.

The pure water flux was 260 L/m<sup>2</sup>h before the experiment, 235 L/m<sup>2</sup>h after cleaning at VR 0.7, and 25 L/m<sup>2</sup>h after cleaning at the end of the experiment. It was not possible to restore the pure water flux by repeated cleaning with the alkaline cleaning agent Ultrasil 10.

**3.2. Membrane Screening.** A screening study was carried out to find a membrane less sensitive to fouling than the ETNA10 membrane. Hydrophilic membranes are less susceptible to fouling than membranes with a more hydrophobic character in most applications [12, 13, 19, 20], but the chemical

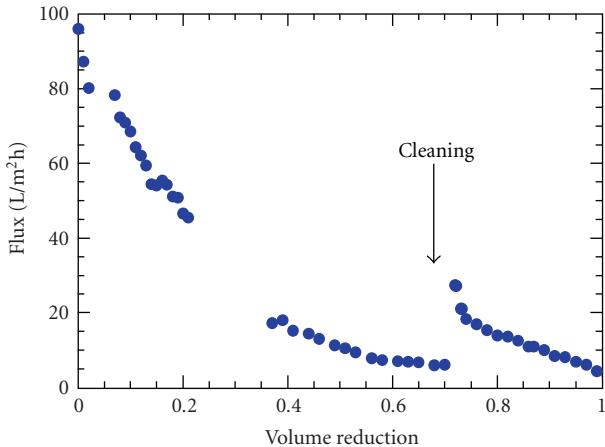


FIGURE 1: Flux during ultrafiltration of prefiltered Masonite wastewater. There was no sampling of flux overnight between VR 0.21 and 0.36.

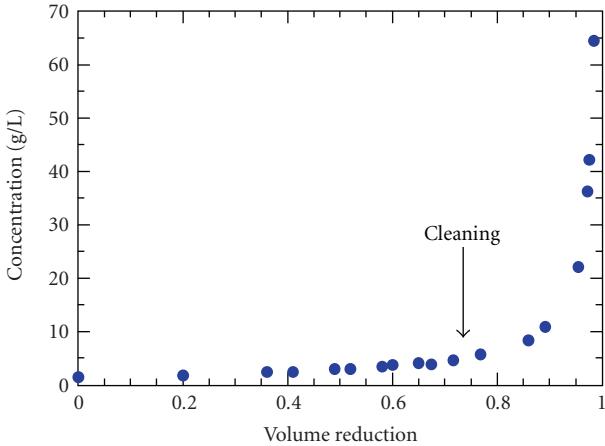
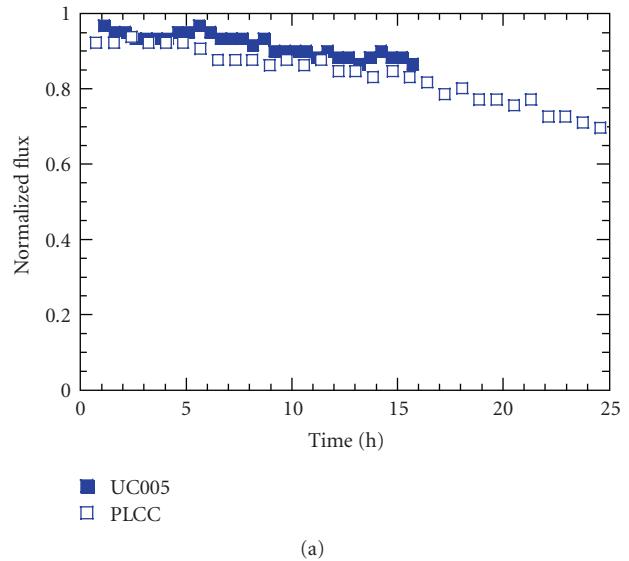


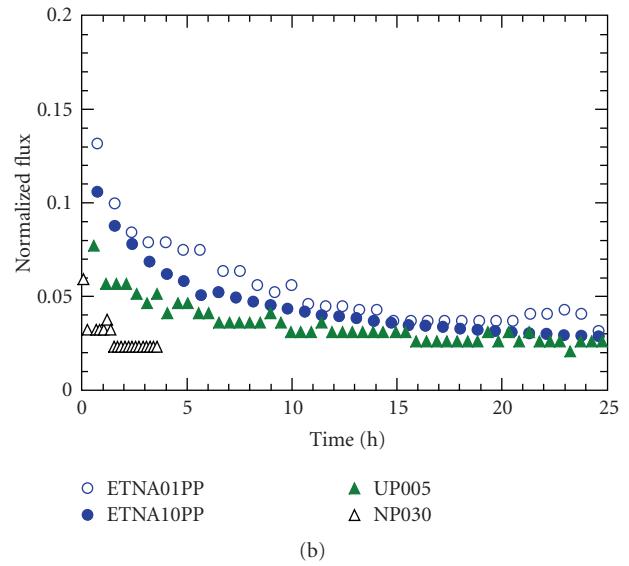
FIGURE 2: Concentration of hemicelluloses in the retentate during the experiment illustrated in Figure 1.

stability of hydrophobic membranes is generally superior [20]. In this investigation, hydrophilic membranes made of regenerated cellulose (UC005 and PLCC), hydrophobic membranes of polyether sulphone (NP030 and UP005), and membranes made of a hydrophobic material modified to be hydrophilic (ETNA 01 and ETNA10) were included. The influence of the pore size was studied using membranes with cut-offs between 1000 and 10 000 Da.

The TS content of the prefiltered wastewater used in the experiments was 0.4%, and the concentration of hemicelluloses was 1.0 g/L. A significant difference in flux decline between the hydrophilic and the hydrophobic membranes was observed. The flux decline of the hydrophilic membranes was minor (see Figure 3(a)), whereas the flux of all other membranes was less than 5% of the original pure water flux after 24 hours (see Figure 3(b)). The normalized flux used in the figure is the ratio between the wastewater flux and the pure water flux before the experiment. The cut-off had no significant influence on the flux decline.



(a)

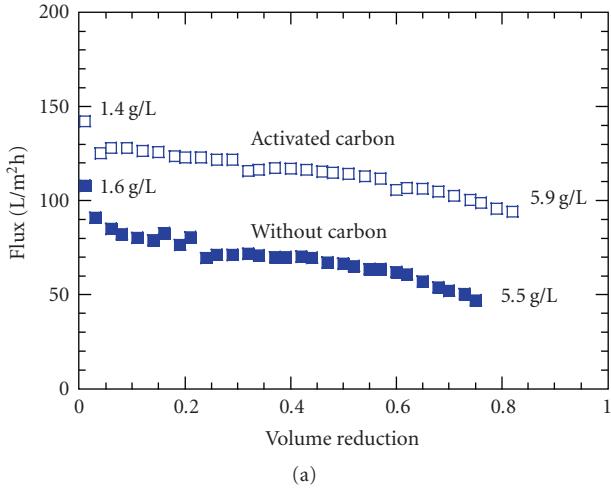


(b)

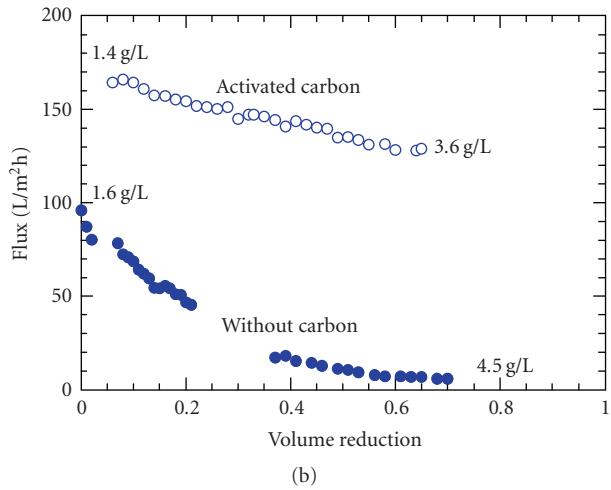
FIGURE 3: Flux decline during ultrafiltration of wastewater. Flux of (a) hydrophilic and (b) hydrophobic and modified membranes. The sampling of flux from the UC005 membrane was interrupted after 15 hours because of instrument failure. Note the difference in the scale of the y-axes.

The hydrophobicity of the ETNA10, UP005, and UC005 membranes were analyzed by using a solution containing 0.5 g/L octanoic acid and measuring the magnitude of the flux decrease caused by the fatty acid. No flux decrease was observed for the hydrophilic UC005 membrane. A minor flux decline was noticed for the ETNA10 membrane (flux being 90% of the pure water flux). The hydrophobic UP005 membrane was severely fouled. The flux was less than 70% of the pure water flux. These results agree with the results from the screening study shown in Figure 3, which implies that the flux decline of the hydrophobic membranes was due to fouling by organic molecules.

**3.3. Adsorption by Activated Carbon.** Adsorption by activated carbon was employed prior to ultrafiltration in order to



(a)

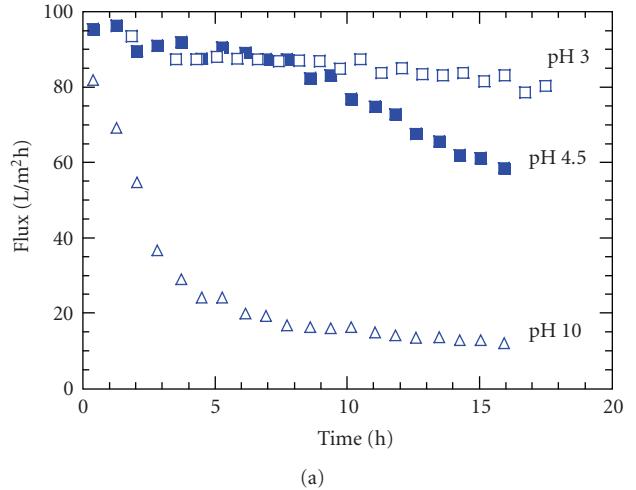


(b)

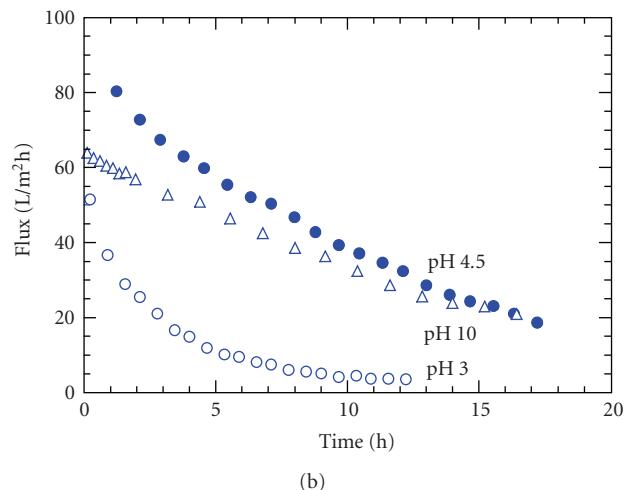
FIGURE 4: Flux during ultrafiltration of wastewater with and without pretreatment with activated carbon. Flux of (a) the UC005 membrane, and (b) the ETNA10 membrane. The curve “without carbon” in (b) originates from the “concentration of hemicelluloses” study. The hemicellulose concentration in the beginning and in the end of the experiments is shown in the figure.

reduce fouling of the ETNA10 and the UC005 membrane. The TS content decreased from 0.5% to 0.4%, and the concentration of hemicelluloses decreased from 1.6 g/L to 1.4 g/L after adsorption with activated carbon. Pretreatment with activated carbon increased the flux of both the UC005 membrane (see Figure 4(a)) and the ETNA10 membrane (see Figure 4(b)). Both membranes had an average hemicellulose retention of 85%, which shows that both can be used to concentrate the hemicelluloses.

The positive effect of the pretreatment with activated carbon was most marked for the ETNA10 membrane. Fouling was not only reduced, the flux of the ETNA10 membrane was even higher than the flux of the UC005 membrane. When using activated carbon, the pure water flux was fully recovered after the experiment with the ETNA10 membrane and to 90%, for the UC005 membrane.



(a)



(b)

FIGURE 5: Influence of pH on flux during ultrafiltration of wastewater with, (a) the UC005 membrane, and (b) the ETNA10 membrane.

**3.4. Influence of pH.** To change the properties of the foulants, the initial pH of the wastewater (4.5) was adjusted to pH 3 and pH 10 by adding hydrochloric acid and sodium hydroxide. The TS content of the prefiltered wastewater used in the experiments was 0.3%, and the concentration of hemicelluloses was 1.0 g/L. The influence of pH on the flux of the hydrophilic UC005 membrane was significant, as can be seen in Figure 5(a). The flux of the UC005 membrane was stable for a longer time at pH 3 than at pH 4.5. At pH 10, the flux decreased rapidly. On the other hand, the flux of the ETNA10 membrane at pH 3 was considerably lower; whereas the flux at pH 4.5 and 10 was quite similar, as shown in Figure 5(b).

The pressure needed to reach a pure water flux of 100 L/m<sup>2</sup>h varied with pH for the ETNA10 membrane, but not for the UC005 membrane. A pressure of about 0.28 MPa was needed for the UC005 membrane at both pH 3 and pH 10. The pressure needed to be increased from 0.02 MPa at pH 10 to 0.07 MPa at pH 3 for the ETNA10 membrane.

These results suggest that the surface charge of the UC005 membrane is independent of pH in the investigated pH interval and that the charge increases with increased pH for the ETNA membrane. That might explain the rapid decline of the flux at pH 3 in Figure 5(b) for the ETNA membrane since a decreased surface charge will increase the hydrophobicity and thus the fouling.

## 4. Discussion

Fouling of the UC005 membrane was much less than fouling of the ETNA10 membrane, as shown in Figure 3. In addition, the flux of the UC005 membrane could be increased by lowering the pH from 4.5 to pH 3 (see Figure 5(a)), and by pretreatment of the wastewater with activated carbon (see Figure 4(a)). The increase in flux at pH 3 might be due to the increased solubility of inorganic compounds at this pH [20]. Activated carbon is known to adsorb both organic molecules, such as phenolic compounds [15], and various inorganic ions, such as aluminium [21]. Hence, the removal of inorganic ions from the wastewater could explain both results.

The influence of pretreatment on the flux of the ETNA10 membrane was somewhat different. The flux increased in a similar way after pretreatment with activated carbon (see Figure 4(b)). However, the flux decreased more rapidly at pH 3 than at pH 4.5 and 10 (see Figure 5(b)). To investigate if the ETNA membrane was fouled by organic molecules, new samples of ETNA10 and UC005 membranes were cleaned with 0.5 wt% Ultrasil 10 and placed in a beaker with waste water for 20 hours. The pressure needed to reach a pure water flux of 100 L/m<sup>2</sup>h after rinsing with deionized water was measured before and after the experiment. The pressure had to be increased by 18% for the ETNA membrane to reach the same flux after the experiment, while no significant difference could be seen for the UC005 membrane. This suggests that adsorption by organic foulants is significant for the ETNA membrane but not for UC005 membrane.

The fouling was successfully inhibited by using activated carbon, but it would be very costly to use this method in larger scale due to the high price of activated carbon (2000 €/metric ton). With the investigated method, it corresponds to an additional cost of 80 €/m<sup>3</sup> waste water containing approximately 1 kg of hemicelluloses. This can be compared with the total cost of a similar isolation process developed for isolating hemicelluloses from pulp mill process streams which was 0.7 €/kg of hemicelluloses [8]. The dosage of activated carbon must be decreased significantly if used in this application.

To decrease fouling by lowering pH is a less costly method than using activated carbon. The buffer capacity of the waste water is very low; hence, only a small dosage of acid (34% HCl) is needed to lower the pH from 4.5 to pH 3 (0.5 g/kg waste water). The price of hydrochloric acid is around 100–150 €/metric ton which corresponds to a treatment cost of less than 0.1 €/m<sup>3</sup> waste water, which is several times less expensive than using activated carbon. However, this method was only successful for the UC005 membrane.

## 5. Conclusions

The results of this study could be of great importance in the treatment of process streams from forest industries by ultrafiltration. Several important results that support this belief have been achieved in this work, for example,

- (i) it is possible to reach 99% volume reduction during ultrafiltration,
- (ii) the hemicelluloses can be concentrated to at least 60 g/L,
- (iii) fouling can be avoided by using a suitable combination of pretreatment methods and ultrafiltration membrane.

In this application, both the ETNA10 and the UC005 membranes could be used. The ETNA10 can withstand harsher operating conditions, which is beneficial since the temperature of the wastewater is 60°C and must be cooled to 55°C if the more foulant-resistant UC005 membrane is to be used in a future application. However, if the ETNA10 membrane is to be used, a cost-efficient fouling-reduction pretreatment step will be necessary.

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