Water circuit closure with membrane technology in the pulp and paper industry

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Abstract In this study, membrane filtration as an internal purification method, “the kidney”, in the pulp and paper industry is discussed. Membrane filtration is economically competitive and a very versatile process. It can be used to remove the enriched organic and/or inorganic loads either partially or totally from, for example, the mechanical pulping and paper making water circuits and it can be applied to various points within the process. With the so-called shear enhanced membrane modules very high fluxes, in ultrafiltration about 400 L/(m²h) and in nanofiltration fluxes almost 200 L/(m²h), have been obtained. Depending on the membrane, suspended solids (microfiltration), polysaccharides, extractives and high molar mass lignous substances (ultrafiltration) and multivalent salts (nanofiltration) can be removed. Ultrafiltration permeate can well be used in paper machine showers to replace fresh water. The quality of the nanofiltration permeate is significantly higher than that of ultrafiltration.

The membrane processes can be enhanced by various pre-treatment techniques to produce higher permeate fluxes and to eliminate membrane fouling. Such pre-treatment methods are, e.g., chemical treatment, ozonation and biological treatment. The most cost-effective processes were chemical flocculation, pH adjustment and thermophilic aerobic biological treatment.

Keywords Costs; hybrid process; nanofiltration; pre-treatment; ultrafiltration

Introduction

The fresh water consumption in the pulp and paper industry varies between 10 m³/ton up to 40 m³/ton depending on e.g. the paper grade produced and the technical age of the mill. Because of legislation (e.g. IPPC-directive), lack of water resources and customers’ demand the pulp and paper industry all over the world must find competitive ways to reduce their specific water consumption.

Process water recycling is required in an integrated pulp and paper mill when the specific water consumption is reduced below 10 m³/ton paper produced with the help of, for example, water segregation. At this stage different kinds of disturbing substances begin to enrich in the water circuits causing runnability and quality problems. Membrane filtration (micro- (MF), ultra- (UF) and nanofiltration (NF)) offers a competitive way to purify process waters before recycling (Jönsson, 1990; Gavelin, 1991, 1992; Lien and Simonis, 1995; Merry et al., 1995; Alho et al., 1998; Teppler et al., 1999). Membrane processes are versatile and economically feasible methods for internal purification in the pulp and paper mills compared to, for example, flotation or evaporation. Their energy consumption is low and they need relatively small floor space. Thus they are easy to fit also in an existing mill water circuit. The major drags for their wider use in the pulp and paper industry have been low fluxes and membrane fouling.

Membranes allow the water to be cleaned to a desired degree of purity. Suspended solids are retained by microfiltration, polysaccharides, extractives and high molar mass lignous substances by ultrafiltration and multivalent salts by nanofiltration. Fresh water used in paper machine showers can well be replaced with UF permeate (Teppler et al., 1999) and e.g. the paper machine slime problems are simultaneously overcome (Sutela, 2001).
Moreover, the capacity of the shear enhanced membrane processes is sufficient to fulfil the water supply demands of modern paper mills. The Cross Rotational (CR)-filter from Metso PaperChem Oy, offers high ultrafiltration fluxes (over 400 L/(m²h)) with very little fouling. Nanofiltration fluxes close to 200 L/(m²h) have been obtained with the Vibrating Shear Enhanced Processing (VSEP, New Logic Int. Inc.). The economy and the performance of membrane processes can be further improved by optimising the membrane process operating parameters (Mänttäri et al., 1997; Huuhilo et al., 2002; Nuortila-Jokinen et al., 2003a) or by pre-treating (Boman et al., 1991; Hall et al., 1995; Nuortila-Jokinen et al., 1998; Huuhilo et al., 2002) the process water.

In this study membrane filtration as an internal purification method used in various process points in an integrated mechanical pulp and paper mill is discussed. In addition, ways to enhance the membrane flux with pre-treatment are shown and the cost-effectiveness of these hybrid processes will be discussed.

Materials and methods

The waters

In this study circulation water from the grinding room of a mechanical pulp plant (later groundwood mill circulation water, GMW), the paper machine clarified white water (clear filtrate), paper mill total effluent and discharge water from an activated sludge plant were studied. All of these waters contain colloidal and dissolved substances originating from the wood itself (e.g. cellulose, hemicelluloses, lignin, lipophilic extractives and salts) and paper making chemicals, such as retention aids, antifoaming agents etc., in varying proportions. The paper machine clear filtrates originated either from an acidic process (pH 4–5) or neutral process (pH 7–8).

The membranes and modules

In the ultrafiltration experiments CR1000/10 (pilot), CR550/3 (pilot) and CR250/1 (laboratory) modules manufactured by Metso PaperChem Oy were used. The first number refers to the diameter of the membrane and the second to the number of cells in the module. One cell consists of two membrane sheets. The VSEP-filter (New Logic Int. Inc.) is the laboratory model (VSEP L) using a single sheet of membrane in one cell (area 465 cm²). In some experiments the laboratory scale cross-flow flat sheet module, membrane area 52.9 cm², was used.

In most of the ultrafiltration experiments regenerated cellulose membranes C30, C30G and C30F (MWCO 30 kDa) from Nadir Filtration GmbH were used. In addition, the PES 50H (poly(ethersulfone), 50 kDa), the PA 50H (poly(aramide), 50 kDa) ultrafiltration membranes (both from Nadir Filtration GmbH) were used in some experiments. Desal-5 (DS-5, Osmonics Ltd) membrane made from aromatic polyamide and polysulfone was used in the nanofiltration experiments. The NaCl-rejection of the DS-5 membrane is 50% according to the manufacturer. In some experiments a spiral wound element DL2540FXB (Desal-5 DL, Osmonics Ltd), effective membrane area 1 m², and the Dow NF270 polypiperazine flat sheet membrane were used.

The pre-treatments

Biological treatment. The experiments were made at the mill premises with a pilot unit. The pilot unit for the suspended carrier biofilm process (SCBP) was an automated Floobed-unit. In this process biomass grows on carrier elements moved by aeration. In the unit there are two reactors in series and the total volume of the reactors is 2 m³. The process was run under thermophilic conditions (T 45–55°C). Technical urea and phosphorous acid were added as nutrients so that the COD:N:P ratio was 100:5:1. A 1,000 µm bow screen was
placed before the pilot unit to remove the fibres. Before the pilot unit was also a heat exchanger, which adjusted the water temperature to 60°C. Aeration decreased the temperature in the reactors to 55°C. The thermophilic SCBP was operated applying increasing loading rates from 3–4 and 6–8 kg SCOD/(m³d) to 13.5–15 and 27–30 kg SCOD/(m³d) in reactors R1 and R2, respectively. The hydraulic retention time (HRT) decreased in the reactors from 8–9 to 2–3 h and from 4–4.5 to 1–1.5 h, respectively. The temperature in the reactors varied between 50 to 59°C.

The ultrafiltration unit was a CR550/3 pilot filter and the membrane was the regenerated cellulose membrane C30F (Nadir Filtration GmbH). The pressure in the filtrations was 0.8–1 bar and the rotor speed was 470 rpm. A 200 µm bow screen was set up between the biological process and the ultrafiltration unit to remove most of the biological sludge and fibers.

Ozonation. Ground wood mill circulation water was treated with continuous flow ozonation with different ozone doses delivered to the water. Adjusting the water feed rate and the ozone concentration in the gas inlet changed the ozone dose. The gas flow rate was kept constant at 5 dm³/min in all experiments. The calculated ozone dose in the reactor was 68, 120 or 147 mg O₃/dm³. Filtration tests were made with a CR200/1 filter equipped with the C30F membrane.

Flocculation. Several commercial flocculation agents from Kemira Chemicals were tested for their ability to remove turbidity. The chosen agents for further testing were FRC+PEO (resin + polyethylene oxide), K594 (cationic polyelectrolyte) and Altonit SF (bentonite, clay) + Fennopol A321 (anionic polyelectrolyte). The filtration tests were run with a laboratory scale cross-flow nanofiltration equipment. The membrane was Desal 5 DK and the filtration temperature was 50°C. The water was first treated with the flocculation agent and then the flocs were removed with a 150 µm sieve. Then the water was nanofiltered at 11 bar.

Results and discussion
Where to apply membrane filtration?
Membrane filtration can be applied in the pulp and paper manufacturing process to treat any separation task where the amount of, for example, dissolved substances – both organic and inorganic – micro-organisms or colour should be reduced. The degree of separation can be tailored by choosing an appropriate membrane process from microfiltration to reverse osmosis. However, the most suitable processes are ultra- and nanofiltration. Microfiltration in the pulp and paper applications does not usually produce any higher fluxes than ultrafiltration (often even lower), because the membrane foulants are about the same size as the pores in the microfiltration membrane thus resulting in severe fouling (pore plugging) and low fluxes. In reverse osmosis the applied external pressure is usually very high (≥ 100 bar) due to the high osmotic pressure caused by the high concentration of multivalent salts in the waters.

Membrane filtration is at its best when used to treat relatively small water flows with small variation in content. This is why they are a good choice for internal purification of process waters as shown in Figure 1. As the fresh water consumption is declining, the need for process water and effluent treatment increases and the treatment is shifted from the big external treatment plants closer to the points where the effluents are formed.

Membrane capacity
The membrane capacity (the flux in litres produced per square metre per hour) depends strongly on:
• the chemical characteristics of the water to be treated
• the membrane material and its surface properties and
• the filter, especially the shear forces produced in the module.

The fact that the paper making process usually operates at temperatures 40–60°C, limits the number of commercial membranes available. In the mechanical pulping the temperatures can well be above 80°C. So far there are not many polymeric membranes that can tolerate these high temperatures non-stop. The exceptions are ceramic membranes but the high cost of these membranes is a hindrance for their wider use even though their expected lifetime is much longer than that of the polymeric membranes. Moreover, generally the flux of the ceramic membranes is lower than those shown later in this study. The most widely used membrane materials in the pulp and paper applications are regenerated cellulose, poly(aramide), poly(ethersulfone) and poly(sulfone).

The membrane surface properties, such as charge and hydrophilicity/hydrophobicity, have been found to be the determining properties. However, no unambiguous conclusion can be given. The performance of the membrane is very case dependent and must be tested case by case. In general, the more hydrophilic the membrane, the higher the flux. In addition, the more hydrophobic the membrane, the more the hydrophobic substances, such as resin and fatty acids, are adsorbed in the membrane material thus causing irreversible fouling of the membrane. Moreover, if electrostatic repulsion is obtained between the membrane and the feed water, the less membrane fouling is observed.

The choice of the membrane module is also crucial. It has been shown (Nuortila-Jokinen, 1997) that high shear forces on the membrane surface increase the flux. Thus the CR- and the VSEP-filters have been found to perform best in the pulp and paper applications. Their additional benefit is their compact appearance compared to the traditional tubular or the spiral wound (SW) modules; they occupy relatively small floor space. Moreover, the spiral wound modules require a very heavy pre-treatment of the feed. In case there are any solids in the feed the SW modules are readily clogged and are very hard to clean. This is due to their very small flow channels. Besides, the spiral wound structure resists the flow efficiently and very low fluxes are obtained. In tubular membranes the cross flow velocity should exceed several metres per second until the turbulence is high enough to inhibit fouling.

The importance of the membrane material on the flux is clearly shown in Figure 2a. The most hydrophilic, regenerated cellulose C30G ultrafiltration membrane gave the highest flux, about 450 L/(m²h). The more hydrophobic membranes, PA50H and PES50H, gave a
very steady but very low (25 L/(m²h)) flux with the same water and the same filter. The explanation is that the hydrophobic constituents (extractives, mainly resin and fatty acids) of the GMW were more adsorbed into the hydrophobic membranes than in the hydrophilic membrane.

The influence of the paper making additives is shown when the flux of the groundwood mill circuit water is compared with that of the paper machine clear filtrates (CR-filter, the C30G membrane). Even though the paper making chemicals appear in the clear filtrate at very low concentrations, the flux is yet significantly lower, about 200 L/(m²h). It is also seen in Figure 2b that at acidic pH the flux is generally lower than at neutral conditions. This can be explained by the established electrostatic repulsion between the membrane and the feed water at neutral conditions.

The membranes C30G and C30F are modifications of the C30 membrane in which, for example, the membrane backing material (support layer on which the actual membrane is cast) has been changed. In the C30G, as well as in the C30 membrane, the backing material is poly(propylene) while in the C30F membrane the backing material is poly(ethylene terephthalate).

The superiority of the filtering device cannot be unequivocally concluded from this data because in Figure 2a and b only the maximum fluxes obtained in different experiments are shown. Thus the flux behaviour as a function of time has not been taken into account. The fact is, however, that in ultrafiltration the membranes in the CR-filter do not foul as quickly as in the VSEP-filter. For example, at M-Real Kirkniemi Paper Mills in Finland, the CR-filters treating PM3 clear filtrate are washed once a week (part of routine, not a necessity). In our test runs with the VSEP-filter in mill conditions the washing interval can be, in worst cases, only a few hours. This can be explained by the so called critical flux (Howell, 1995; Field et al., 1995). In the CR-filter the operation pressure (about 0.8 bar) is well below the critical flux of the membrane and thus hardly any fouling occurs. In the VSEP filter the lowest allowed operation pressure is 2.5 bar. This exceeds the critical flux and severe fouling is observed in the ultrafiltration. (Nuortila-Jokinen et al., 2003a).

In nanofiltration of the paper machine clear filtrates, the average capacity is around 80 L/(m²h) for the acidic clear filtrate and about 90 L/(m²h) for the neutral clear filtrate (Figure 3a). In this case the VSEP-filter gave a significantly higher flux than any other module. One reason for this is that the Desal 5 DK membrane was used in the other experiments instead of the Desal 5 DL. The DK-type membrane has, in general, a lower flux than the DL-type membrane.

The high flux obtained with the Desal 5 DL membrane in the VSEP-filter in the nanofiltration of paper mill clear filtrates (Figure 3a) is a result of using optimal operating parameters. The VSEP-filter is well suited for nanofiltration. Even though the operating pressures are relatively high (> 14 bar), the process can still be operated below the critical and...
limiting fluxes (Nuortila-Jokinen, 2002). This also allows the process to be run without fouling.

The Desal 5 and the WFN 4505 are both poly(aramide) membranes with poly(sulfone) support layer with similar sodium chloride rejection capabilities. The main difference appears in the module configuration; the latter being a tubular module. The flux obtained with the WFN 4505 membrane is very competitive, but the drawback is that the cross-flow velocity in these experiments was very high, 8.5 m/s (Nuortila-Jokinen, 1997). Thus the pumping energy needed to maintain this high cross-flow velocity will increase the operation costs significantly and due to the tubular configuration the plant will also occupy a very large floor space compared to the more compact modules, such as the VSEP.

In Figure 3b the effect of the module configuration is shown. The membrane and the spacer used in the laboratory scale flat sheet module were the same as in the tested spiral wound (SW) module (Mänttäri et al., 1999). The flux of the SW module was less than 30% of that of the flat sheet module. This result indicates that the spiral wound module has a very high resistance to flow and that there may also be “dead spots”, i.e. places that are not reached by the flow.

Membrane retention

The Table 1 gives roughly the magnitudes of the retentions obtained with different membrane processes. The ultra- and nanofiltration are highlighted because they have shown to be the most useful processes in the pulp and paper applications. The ultrafiltration removes about 30% of the organic load and the permeate is pure enough to replace mechanically treated fresh water, for example, as the paper machine shower water. Contrary to mechanically purified fresh water, the UF permeate is practically free from micro-organisms, which significantly reduces slime formation in the paper machine (Sutela, 2001).

Nanofiltration removes most of the organic load and also the multivalent ions, such as calcium, iron, aluminium, silicon, magnesium and sulphate. These ions are detrimental if enriched in the water circuit system because they can, for example, form deposits or accelerate or cause corrosion. Nanofiltration permeate can be used to replace fresh water even in the high pressure showers of the paper machine. Compared to chemically treated fresh water commonly used in paper mills, the benefit of the NF permeate is that its salt content is significantly lower.

In Figure 4 an example of the retentions obtained both in ultra- and nanofiltration of groundwood mill circulation water is shown. The retention of the organic material (TC) is more than three times higher in nanofiltration than in ultrafiltration. In addition, the retentions of the calcium, chloride and sulphate ions in nanofiltration were 96, 51 and 99% respectively.

Figure 3  Nanofiltration of a) the paper machine clear filtrates and b) paper mill total effluent using Desal 5 DL, Desal 5 DK, NF270 and WFN 4505 membranes. Tested modules/filters: CR-filter, VSEP-filter, Stork-Friesland tubular compact membrane module (tubular), spiral wound module (SW) and laboratory flat sheet module (flat sheet)
Enhancement of membrane filtration process with pre-treatment

By applying different pre-treatments to the feed, membrane filtration can be remarkably enhanced. The aim of the pre-treatment is to affect the membrane foulants either by disabling or by eliminating them. The result should conclude in increased and more stable flux.

The pre-treatment methods belonging to the former group are, for example, different chemical treatments. Naturally, a looser membrane process can be used as a pre-treatment for a tighter one, for example UF for NF. By using biological processes some foulants, such as low molar mass organics, can be eliminated. Ozone can be used to either degrade or to eliminate foulants depending on the ozone dose.

In Figure 5a, the results from the experiments where retention aids were used for pre-treatment of the groundwood mill circulation water, are shown. The chemicals formed flocs with the foulants. The flocs were removed prior to nanofiltration. As a result of the pre-treatment procedure the nanofiltration flux was significantly improved; in the best case the flux was almost doubled. The use of chemical pre-treatment is not, however, problem

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<th>Solids</th>
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Table 1 The retentions of different membrane processes, ++++ = very good, (+) = almost nil. HMM = high molar mass, LMM = low molar mass

Figure 4 Comparison of the retentions of some measured components obtained in the ultra- and nanofiltration of the groundwood mill circulation water. UF: C30F membrane; NF: Desal 5 DK membrane

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Figure 5 The effect of a) chemical pre-treatment on the nanofiltration flux and b) ozone pre-treatment on the ultrafiltration flux. Groundwood mill circulation water; UF: UF30F membrane; NF: Desal 5 DK membrane.

Relative flux: the NF flux of the groundwood mill circulation water = 1

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The quality of the feed water at mill site usually varies much according to the process circumstances. Thus extreme care should be taken when dosing the chemical. The optimum dose is the one where the cationic charge of the chemical is precisely equal to the anionic charge of the treated water. It has been shown that overdosing as well as underdosing the chemical may result in fouling of the membrane by the chemical (Nuortila-Jokinen et al., 1998).

However, the specific removal of some certain component does not always result in improvement of permeate flux. By ozonation hydrophobic substances, such as extractives, can be completely destroyed (Figure 5b). Yet, the flux decreases, which is most probably due to the ozonation degradation products. As also seen in Figure 5b, there is an optimum ozone dose where the flux is still somewhat increased. This could be explained by the disinfecting effect of the ozone treatment. Based on these results, it seems that biofouling causes more severe membrane fouling than hydrophobic substances.

Biological digestion can be successfully used for pre-treatment of membrane filtration. In general, biological digestion removes low molar mass organic compounds from the feed water – the COD-removal of e.g. thermophilic aerobic process alone, is about 60% (Huuhilo et al., 2002; Nuortila-Jokinen et al., 2003b).

The most simple way to develop a closed water circuit in a paper mill is to take the benefit of the external waste water treatment plants. For example, in Finland all paper mills have an activated sludge plant to treat their effluents. The discharged water from the activated sludge plant can be further membrane filtrated to produce water for reuse back at the mill. The ultra- or nanofiltration permeate is well suited for this purpose, because they do not contain biological activity. Moreover, the nanofiltration flux of the discharge water is almost tripled compared to the neutral clear filtrate (150 and 60 L/(m²·h) respectively, the NF270 membrane) (Määttäri and Nyström, 2004).

Biological processes can also be brought inside the mill. By applying thermophilic biological processes (temperature above 50°C), cooling and heating of the process water prior to or after biological treatment can be avoided. Figure 6 clearly shows how the nanofiltration flux is improved when different pre-treatment methods have been used.

The pH of the groundwood mill circulation water is about 5 and it has to be adjusted to neutral range before biological treatment. It is noteworthy that the pH adjustment alone increased the nanofiltration flux from 28 to 55 L/(m²·h). This can be explained by the electrostatic repulsion obtained between the feed water components and the membrane itself. When the pH adjusted water was further treated with thermophilic aerobic biological treatment, the NF flux increased to 76 L/(m²·h) and if an ultrafiltration step was added between the biology and the NF, the NF flux increased to 87 L/(m²·h). Thus the flux was increased.

![Figure 6](https://iwaponline.com/wst/article-pdf/50/3/217/421613/217.pdf)
68% when compared to NF without any pre-treatment. In addition, the fouling in the nanofiltration step decreased significantly.

Based on the retention values shown in Figure 6b, there is no indisputable evidence that removal of a specific component from the feed water would affect the flux behaviour. This is probably due to the complex interactions between the different substances.

The costs of membrane filtration
Membrane filtration is a very competitive method for internal water purification. The total cost for an ultrafiltration plant (CR-filters) treating 2,000 m³/d of groundwood mill circulation water is 0.42 €/m³ (10 years payback time). Respectively, for a nanofiltration plant the total costs would be 0.50 €/m³. Naturally the cost alters case to case with the flux (corresponding to the membrane area needed), price of the module and the membrane, washing frequency etc.

Figure 7 shows the cost-effectiveness of some of the hybrid processes discussed earlier. For example, the pH adjustment more than doubled the flux with minimal cost. Moreover, the ultrafiltration and biological treatment as pre-treatment steps are equally expensive, but the biological pre-treatment gave significantly higher flux. Involving the ultrafiltration step to the latter (Bio + UF + NF) increased the costs by 31% but gave only 13% higher flux.

Even though the chemical pre-treatments (pH adjustment or flocculation) show a good cost-effectiveness, it must be pointed out that both of them may cause problems to the paper making process. In acidic paper making process the pH cannot be changed back and forth without causing a danger of pH shocks. Moreover, the inorganic load, especially the concentration of sodium, chloride and sulphate ions, of the treated water is significantly increased. As discussed earlier, the chemical pre-treatment carries the risk of under or overdosing if sufficient online monitoring and control of, for example, process zetapotential is not arranged.

Conclusion
As the fresh water consumption decreases in the pulp and paper industry and the detrimental substances are enriched in the water circuit, membrane filtration offers a competitive alternative for internal water purification. The development of the shear enhanced modules along with the deeper understanding of the interactions between membranes and the feed waters have made high flux – low fouling membrane processes a reality.
The membrane processes can be further improved by applying different pre-treatment procedures. Chemical flocculation and thermophilic aerobic biological treatment increased the nanofiltration flux 2- to 3-fold and showed to be the most cost-effective.

Eventually membrane filtration, due to its easy applicability, versatility and relatively low cost, will be an essential part of any closed water circuit in the pulp and paper industry.

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