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Industrial membrane processes in the treatment of process waters and liquors

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ABSTRACT

A review on pulp and paper industrial membrane processes using a variety of modules and processes is presented. Membranes are mostly used today to purify process waters and to recover coating colours. Ultrafiltration using tubular membrane modules or cross-rotational (CR) filtration has been widely applied for the purification of process waters. The reuse of UF membrane permeate has decreased the fresh water consumption to lower than 6 m^3/t of paper in some paper machines. Some industrial membrane processes also recover valuable products from different streams (e.g lignosulphonates). Membranes are also combined with biological degradation processes in some paper mills. Nanofiltration has been used to purify the effluents discharged from the activated sludge process. At least two reverse osmosis plants purify river water to be used as raw water in the mill. Furthermore, advantages of different membrane modules and the current ways to treat membrane concentrate are discussed.

Key words | concentrate, membrane filtration, membrane modules, pulp and paper industry

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INTRODUCTION

Although the water consumption in the pulp and paper manufacturing has decreased since the 1970's the pulp and paper manufacturing is still a very water and energy intensive process. Integrated pulp and paper mills may discharge water at a rate of even cubic meters per second.

Pulp and paper mill process waters and effluents are challenging to treat with membranes because they comprise heterogeneous combinations of different wood based compounds (polysaccharides, lignin and extractives including sterols, resin and fatty acids, triglycerides, lignans, flavonoids etc.), and their degradation products. In addition, the waters might contain the chemicals used in the processing of the raw materials (e.g. inorganic cooking chemicals) or additives used in the production of the end products (antifoaming agents, biocides, polymeric retention agents, optical brightening agents, fillers i.e starch and latex etc.). Some compounds are also known to be strongly hydrophobic in character causing fouling of membranes (Puro *et al.* 2002; Lacorte *et al.* 2003).

Futhermore, process waters and effluents contain suspended solids, e.g. fibres or debris, and have high contents of colloidal material. When the same paper machines are used to manufacture different paper grades, which all need tailored process conditions and special raw materials, the concentration and composition of the process streams to be filtered vary considerably (Nuortila-Jokinen *et al.* 2003). In spite of these demanding conditions several industrial scale membrane filtration processes have been installed in the pulp and paper mills and some of them are briefly reviewed in this paper.

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BRIEF HISTORY OF MEMBRANE INSTALLATIONS

Membranes have been evaluated for use in the pulp and paper industry for about 40 years. The initial experiments involved white water (circulation water) from a paper machine, which was purified for reuse, and bleach plant effluents, which were treated for the removal of colour. The first reported commercial application of membrane processes was as early as 1972 when Green Bay Packaging (Wisconsin, USA) installed a reverse osmosis (RO) system for the treatment of paper mill circulation water. Plate and frame type RO plants were installed at the Toten Sulfite Mill in Norway (1976) and at the Reed Lignosol Plant in Canada to concentrate spent sulphite liquor. During the 1980s three large-scale ultrafiltration (UF) plants started to treat caustic extraction stage effluents in Japan and Sweden to remove colour and reduce chemical oxygen demand (COD) and biological oxygen demand (BOD). The Taio Paper Company in Japan began to treat E-stage bleach plant effluent with tubular UF in 1981 and Sanyo Kokusaku pulp mill chose plate and frame type UF. Over 80% of the COD was removed. A UF plant with tubular membranes has been in use since 1981 in Norway (Borregaard Industries, Sarpsborg calcium bisulphite pulp mill) for the fractionation of a lignosulphonate stream (spent sulphite liquor) into two streams, which are further processed into by-products (vanillin and lignosulphonates). In 1988 the first high shear rate cross-rotational (CR) filter was installed in Sweden (MoDo, Husum). It was used to remove absorbed AOX (adsorbable organic halogens) and COD from the bleach plant effluent. This technology has now been installed in over 40 mills to reuse coating colour and process waters or to purify effluents (Jain & Miner 1998; Wagner 2000; Sutela 2008).

During the 1990's two new types of membrane processes, nanofiltration (NF) and the membrane bioreactor (MBR), were introduced in the pulp and paper industry. Today all kinds of modules (spiral wound, modified plate and frame, tubular and hollow fibers) are in use in the pulp and paper industry (Lien & Simonis 1995). This review tries to show how membranes can be used at very different sites and for various purposes. The mentioned processes are only examples of what has been done. The examples are ordered according to modules used.

MEMBRANE MODULES IN INDUSTRIAL INSTALLATIONS

Spiral wound modules

Spiral wound and hollow fibre modules have higher packing densities compared to e.g. tubular modules. Unfortunately, due to their small flow channels, these modules tend to plug easily and thus demand feed streams free of particulate contaminants (e.g. fibres and suspended solids). To reduce the plugging tendency of spiral elements, more open flow channels and special spacer constructions have been developed. Another drawback in spiral wound modules is a difficulty to maintain high flow rates because the flow rate also increases the pressure loss over the module and this will, when exceeding the upper allowed limit of the modules (typically 0.5–1.5 bar), devastate the module structure. Thus, with these types of modules the boundary layer, i.e the concentration polarisation layer, limits the flux and as a result the flux is often significantly lower than when high shear rate modules are utilised.

Spiral wound modules are "standardised" so that different modules from various manufacturers can be used in the same pressure vessels although minor changes might be needed. A wide variety of membrane materials are available for the spiral configuration and the modules have been the least expensive per unit area of membrane. NF and RO membranes are typically used in spiral wound form and in order to operate successfully a sophisticated pretreatment is usually needed (Table 1). In the pulp and paper industry these modules can be used for fairly clean non-fouling waters.

Spiral wound modules are common in seawater desalination processes. They have also been used to purify raw water in the pulp and paper industry at least in two mills (Table 1). Bahia Sul Celulose in Mucuri (Brazil) has used an RO water purification system (capacity 360 m^3/h, recovery 75%) to remove both dissolved solids and micro-organisms since 1991. In this application the membranes have originally suffered from high levels of biofouling that generate a high pressure drop on the system and demand frequent cleaning. This happened despite various pretreatments prior to RO (Table 1). The development of a new more fouling resistant membrane and the

Table 1 | Examples of spiral wound installations and their pretreatments (Kremser *et al.* 2006; Mänttäri & Nyström 2009)

Place	Mill	Application	Pretreatment
Mucuri, Brazil	Bahia Sul Celulose	Raw water treatment, RO	Chlorination, flocculation, sedimentation, sand filtration and filtration through activated carbon
Stendal, Germany	Mercel Int. pulp mill	Raw water treatment, RO	Coagulation, sedimentation, filtration and cartridge filters
Lohja, Finland	Kirkniemi paper mill	Process water, NF	Disc filter, cross-rotational ultrafiltration
Pacific Rim, Korea	Paper mill	Effluent, NF	Settling at several stages with chemicals, a sand filter, a back-washable screen filter and a bag filter
Eltmann, Germany	Papierfabrik Palm	Effluent, NF	Biological treatment (activated sludge plant) and a sand filtration
Chirk, UK*	Kronospan Ltd, MDF mill	Effluent, RO	Flocculation, filter press, sand filter and cartridge filters
Sarpsborg, Norway	Borregaard Industries	Spent liquor, RO	Ultrafiltration
New Mexico, USA	McKinley Paper board mill	Effluent, RO	Biological treatment, hollow fibre micro filtration

*More than 10 medium density fibreboard (MDF) mills in Europe use RO in their effluent treatment.

optimisation of the operation (maximum flux 22 L/(m^2 h)) and the cleaning conditions significantly reduced the fouling and the cleaning interval was doubled to 12 days. The salt retention is 98% corresponding to a permeate conductivity less than 6 μS/cm (Soalheiro 2000).

In the middle of the 1990s two spiral wound NF plants (USA and Korea) were installed for the treatment of effluent from a paper mill. Both plants had a very efficient pretreatment prior to the NF spiral wound modules (Desal-5 DK membrane) in order to prevent plugging of the filtration elements (Table 1). The use of a wide feed side spacer makes it possible to use a high cross-flow velocity in the spiral wound modules without causing a too high pressure drop over the module. In the Pacific Rim (Korea) plant nanofiltration removed over 95% of the total hardness and over 90% of the COD, and almost 80% of the conductivity but practically not at all SiO_2 and chloride ions from the biologically treated effluents (at 95% permeate recovery). Neither plant is in operation today (Lien & Simonis 1995; Eriksson 2005).

In Germany, Eltmann newsprint mill of Papierfabrik Palm has reduced COD, AOX and colour in their effluents using spiral wound nanofiltration since 1999. The mill uses activated sludge treatment and sand filtration prior to NF. The NF plant capacity is 190 m^3 permeate per hour. The flux of the Nadir NF-PES-10 membrane has been around 10–30 L/(m^2 h). The reported COD removal is 89%, AOX 61% and colour 93%. The reuse of NF permeate decreased the need to heat fresh water to the mill process temperature. The concentrate (36 m^3/h) is recirculated back to the activated sludge plant after lime precipitation (Schirm *et al.* 2001).

The reviewed mill scale examples prove NF and RO spiral wound modules to be useful in the treatment of raw water and pulp and paper mill effluents but extensive pretreatments are needed. Despite efficient pretreatments, the flux has commonly been only 5–40 L/(m^2 h) in spiral wound modules with NF or RO membranes.

Hollow fibres

Hollow fibres have the highest packing density of the existing modules but the fluxes are low. They are mostly used in submerged membrane bioreactors in the "outside-in" mode (MF and UF membranes) in the pulp and paper industry at close to subcritical fluxes to avoid fouling. In addition, their cleaning is improved by backflushing techniques. Aeration is used to minimise particle adhesion and concentration polarisation. Hollow fibres are commonly used in the membrane bioreactors but also as a pretreatment stage for RO.

Papeterie du Rhin's paper roll mill in France uses a membrane bioreactor with Zeeweed® UF membranes in their effluent treatment. The effluent (900 m^3/d) from the mill is first pre-screened with drum screens, then sent to an equalisation basin, from which it is pumped directly into the bioreactor. Brockmann & Praderie (2005) report an average flux of 15 L/(m^2 h) at a pressure of 0.15 bar. The MBR process decreased the COD from 4,000 mg/L to less than 200 mg/L and the BOD from 1,700 mg/L to less than 5 mg/L. The permeate is partly recycled as process water (Joore *et al.* 2001; Brockmann & Praderie 2005).

The McKinley Paper Company in New Mexico (USA) produces test liner from old corrugated containers (OCC). The lack of water made the mill minimise their raw water consumption to only 1.5 m^3/t board product. A waste stream of 3.3 m^3/t board produced is cleaned and finally concentrated using MF and RO membranes. First, flotation is used to separate fibres, fines and stickers from the water and then an activated sludge treatment is carried out prior to membrane filtration. The biological treatment reduced the COD and the total suspended solids content over 80%. The efficient pretreatment enables the use of hollow fibre microfiltration (USF Memcor Ltd.) to remove suspended solids from the effluent to less than 1 mg/L and to reduce the permeate silt density index (SDI) to less than three. Therefore, the MF permeate is suitable for reverse osmosis treatment (Pohjalainen 1999).

MBR (Memcor modules) has also been combined with downstream reverse osmosis in a board mill in Germany (Albert KohlerPappen board mill in Gengenbach). The system allows to reuse purified and desalted wastewater and reduces the volume of wastewater by some 90%. The COD concentrations decreased from 2,200 mg/L to less than 150 mg/L, and the average biological oxygen demand (BOD) levels were reduced from 1,000 mg/L to 2–3 mg/L (Junk *et al.* 2008).

Tubular membrane modules

Tubular modules are not easily plugged and can be used at high cross-flow velocity to minimise the concentration of retained substances on the membrane surface. Cross-flow velocity strongly influences the power consumption because under turbulent conditions the power will vary with the velocity raised to a power of 2.75 (Merry 1999). Unfortunately, flux is usually a function of the flow velocity and optimisation needs to be done between power consumption (flow velocity → flux) and the installed surface area of the membrane. Tubular modules are relatively easy to clean (even physical cleaning using foam balls is possible) and they can withstand some fibres in the feed water without becoming plugged, which has been the most typical reason to use them in the pulp and paper industry. In addition to the polymeric tubular membranes also ceramic tubular membranes are available on the market. Any industrial scale ceramic membrane filtration application has not yet been reported in literature but in future ceramics probably find their places in the applications where the temperature is high and/or the pH is very low or high. Table 2 shows some examples of tubular membrane filtration plants in the pulp and paper industry.

Since 2002 Artic Paper Munkedals (Sweden) has treated their white water using Norit tubular UF membranes. Sedimentation was used prior to UF, which removes over 95% of the remaining suspended solids. The ultrafiltered water is used in showers in the wire section. The flux in

Table 2 | Some examples of tubular membrane filtration installations (Merry 1999; Hepp *et al.* 2005; Mänttäri & Nytsröm 2009)

Place	Mill	Application	Type and size	Supplier	Year
Cowpens, SC, USA	LINPAC, paper mill	Mill effluent, 50 m^3/h	Tubular UF	Koch	2003
Munkedals, Sweden	Artic Paper	White water, 10 m^3/h	Tubular UF, 270 m^2	Norit	2002
Ugchelen, Netherlands	Van Houtum & Palm	Bleaching effluent, 10 m^3/h	MBR, tubular, 83 m^2	NORIT	2000
Munkedals, Sweden	Artic Paper	Mill effluent, 40 m^3/h	Tubular UF, 462 m^2	Norit	1999
Nymölla, Sweden	StoraEnso pulp mill	Bleach effluent, 300 m^3/h	Tubular UF, 4,600 m^2	PCI	1995
Norway, Sarpsborg	Borregaard Industries	Ca-sulphite spent liquor	Tubular UF, 1,120 m^2	PCI	1981

UF is about 40 L/(m^2 h) at 45°C and a pressure of 1–4 bar. The mill also purifies 50% of their effluents using UF (flux 50–80 L/(m^2 h)) (Hepp *et al.* 2005).

Probably the largest tubular membrane filtration plant having a total membrane area of 4,638 m^2 started in 1995 in StoraEnso Nymölla pulp mill in Sweden. The mill produces pulp from both hardwood and softwood using the magnesium bisulphite process. The COD of the effluent is about 10 g/L. The aim was to obtain a 50% reduction in COD and to remove the COD so concentrated that it could be incinerated. The whole plant processes > 300 m^3/h of bleach effluent producing a concentrate stream of 6 m^3/h (COD about 180 g/L). The permeate is discharged to an activated sludge plant. The fluxes are about 90 and 150 L/(m^2 h) at 7 and 8 bar pressure for softwood and hardwood effluents respectively. The high UF operation temperature (up to 82°C) eliminates the need for rapid cooling before the membrane plant and prevents precipitation of extractives on the membranes. Furthermore, keeping the pH above 11 reduced fouling (Merry 1999; Nordin & Jönsson 2008).

High shear rate modules

Because high fluxes and the ability to process streams containing suspended solids and fibres are often wanted in the pulp and paper industry, high shear rate modules have been developed. The currently existing high shear rate modules, excluding tubular modules, are modified plate and frame constructions. Both a cross-rotational (CR) module from Metso Paper and a vibration shear enhanced processing module (VSEP) from New Logic Inc. have been industrially used in the pulp and paper industry applications as Table 3 shows) (Jönsson *et al.* 1996; Anonymous 2005; Sutela 2008). Installation costs of high shear rate modules are typically higher than those of conventional modules.

In the cross-rotational (CR) filter the shear rate is generated by a rotating blade near the membrane surface, which forces the feed fluid to move with a high flow velocity across the membrane. The peripheral velocity of the blade can reach 13 m/s. The CR-filter can withstand and even might benefit when the feed water contains a small amount of short fibres. As a pretreatment only longer fibres and bigger particles are removed using a coarse screen prior to the CR ultrafiltration.

The industrial scale CR-filters were designed for UF membranes. Some piloting tests have also been made with a modified module and NF membranes (Mänttäri & Nytsröm, 2004). The CR-nanofiltration has shown to be an efficient way to polish effluents after biological treatment. Permeate fluxes from 90 to 150 L/(m^2 h) at a volume reduction of 12 and a pressure of 8 bar were achieved when discharge waters from activated sludge plants from different mills were filtered (Mänttäri *et al.* 2006).

Probably the best reported membrane installation in the pulp and paper industry is located in Lohja (Finland). The Sappi (former M-Real) Kirkniemi paper mill has totally 19 CR-units in the treatment of white water from paper machines and in the recovery of coating colour. The membrane area installed for white water treatment is 1,428 m^2 and for coating colour treatment 336 m^2. The first filter was installed in 1994. The high shear rate on the

Table 3 | Examples of membrane filtration plants using high shear rate membrane modules

Place	Company and mill	Application	Membrane unit	Year
Germany	Confidental customer	White water for OCC mill	CR-UF, 560 m^2	
Japan	Hokuetsu, Fine paper Niigata	White water	CR-UF, 560 m^2	
Turkey	Modern Karton, OCC	White water	CR-UF, 560 m^2	
Valkeakoski, Finland	UPM-Kymmene Tervasaari	White water, 120 m^3/h	CR-UF, 560 m^2	2005
Fuenlabrada, Spain	Holmen Paper Madrid	White water, 33 m^3/h	CR-UF, 140 m^2	2004
Cowpens, SC, USA	LINPAC, recycle paper mill	UF concentrate, 5 m^3/h	VSEP NF, 242 m^2	2004
Lohja, Finland	M-Real Kirkniemi mill	White water, 100 m^3/h	CR-UF, 420 m^2	2001
Örnsköldsvik, Sweden	Domsjö Fabriker	White liquor, 45 m^3/h	CR-UF, 252 m^2	2000
Lohja, Finland	M-Real Kirkniemi mill	White water, 220 m^3/h	CR-UF, 924 m^2	1996

membrane surface in UF is the key to extremely high fluxes. The average flux at a pressure of 0.8 bar was 315 L/(m^2 h) for the period 2001–2008. The UF membrane removes suspended solids, turbidity and bacteria completely. The reduction of anionic trash and COD are 50% and 10%, respectively. The permeate from the ultrafiltration system is used at the wire section showers and for chemical dilutions. In 2008 the total operating costs for CR-ultrafiltration was about 0.13 €/m^3 (used energy price 0.05 €/kWh).

Part of the UF permeates are nanofiltered using spiral wound modules and the permeate from the NF (flux 30–40 L/(m^2 h)) is led to a warm fresh water tank for reuse in nozzles and other critical parts of the process. Today the whole paper mill is operated at a fresh water consumption level of 9 m^3/t of paper and one fine paper machine consumes only 6 m^3 fresh water per tonne of paper (Sutela 2008).

Another type of high shear rate module, known as the VSEP filter, was introduced in 1991. The VSEP is a vertical plate and frame type construction, where membrane leaves are stacked on top of each other. In this system the membrane is moved and not the fluid. The VSEP employs torsional vibration of the membrane stack to generate shear forces at the membrane surface, an order of magnitude higher than those achievable in typical cross-flow modules. This reduces concentration polarisation and colloidal fouling significantly and makes filtration with minimally pretreated streams possible.

Linpac recycle paper mill installed VSEP-modules with NF membrane to concentrate the retentate from a two stage UF. This uncommon way to combine membrane processes makes it possible to remove impurities at really high concentration from the process. The VSEP-NF increased the total suspended solids content from 7% (UF concentrate) to 24% (Anonymous 2005).

Probably, the most successful application for membranes in the pulp and paper industry has been the recovery of coating effluents. Although the volumes of the coating waste waters are negligible compared to the overall mill waters, they cause significant colour in discharge water because they are not easily treated using conventional treatments. Furthermore, they represent a significant economical loss. Zero effluents from the coating plants, recycling of the concentrated coating colour and reuse of permeate are the main advantages when coating colour is treated by membranes, mostly by UF. The CR-technology dominates the installations in the coating colour treatment but tubular modules are also used. The payback time is typically 1–2 years when treating valuable coating colour (Alho *et al.* 1998). A good example about an extremely economical UF application is the CR-filter plant at ASSI Frövifors Bruk board mill in Frövifors (Sweden). The waste latex coating containing TiO_2 pigment is concentrated and recovered in the process. The UF operates at a constant concentrate solids concentration of 20% continuously for ten days between cleanings. The 50 kg/mol cut-off polysulphone membrane produces a flux of 200 L/(m^2 h) (Jönsson *et al.* 1996).

Treatment of membrane concentrates

The treatment of membrane concentrate depends on the application. A concentrate containing valuable compounds can be re-circulated back to the production process and a concentrate, which is a waste stream, needs to be further treated in an environmentally acceptable way before discharging it to the environment. Figure 1 shows some alternatives for the treatment of membrane concentrate in the pulp and paper industry.

The most economical way to dispose of the concentrate is to reuse it in the manufacturing process. The recovery of coating colour is one example of that kind of process. The payback times have been less than one year especially

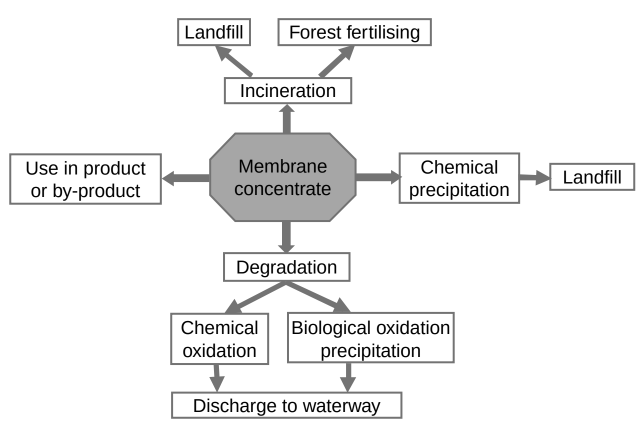

Figure 1 | Possibilities and used methods to treat membrane concentrates in the pulp and paper industry.

when valuable coating pigments such as titanium dioxide have been used to give brightness for the paper. Another process where the membrane concentrate is disposed of with the final product is the manufacturing of medium density fiberboard (MDF). The RO concentrate (COD around 200,000 mg/L) is simply disposed of by spraying it on the fibers before pressing in the manufacturing process. In that way there is no effluent to treat and the sprayed concentrate actually improves the board quality and saves a little glue since the sugars polymerise during heating and form a glue-like material (Alho *et al.* 1998; Wagner 2000).

If the produced concentrate is a waste stream, it can be incinerated. However, a low dry solids content in the membrane concentrate often restricts its incineration. Therefore, evaporation has been used prior to incineration to lower the water content of the concentrate (e.g. Rottneros Board mill in Sweden). Furthermore, a high concentration of inorganic compounds (e.g. silica) may sometimes set limits to the incineration of the concentrate e.g. in the chemical recovery boiler in the pulp mill.

A crystallisation process can remove salts from the membrane concentrate. For instance, the McKinley Paper Company's mill in New Mexico (USA) uses crystallisation for their RO concentrate (Pohjalainen 1999).

The Linpac paper mill in USA could concentrate UF retentate by NF prior to leading it to external disposal (Anonymous 2005). In Germany NF is used to purify effluents from an external biological treatment process to reuse in the mill. The NF concentrate is precipitated by lime and finally lead back to the biological process. The lime precipitation removes about 40% of the organic compounds (measured by COD and AOX) and over 70% of the coloured compounds. The iron removal was over 99% but sulphate ion removal only 30% (Schirm *et al.* 2001).

In many places where the concentrate cannot be reused it has to be degraded by e.g. biological processes prior to discharging it to the waterway. Chemical oxidants (e.g ozone) or enzymes can be used prior to the biological process to improve the biodegradability of the concentrate. Ozone has been used at least in two mills for that purpose in the combination: biological treatment–ozone degradation–biological treatment (Bierbaum & Öller 2005) but not yet connected to membrane concentrate treatment according to the authors' knowledge.

CONCLUSIONS

A wide range of membrane modules are used to treat different process streams and effluents in the pulp and paper industry. The reviewed examples show that membranes can solve many challenges in the purification of effluents, the reuse of process water and in the production of by-products. If the pulp mills are more and more converted to multi-product biorefineries membranes will be seen in many new applications in the future.

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