PERFORMANCE OF A PILOT-SCALE MEMBRANE PROCESS FOR THE CONCENTRATION OF EFFLUENT FROM ALKALINE PEROXIDE MECHANICAL PULPING PLANTS

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A pilot-scale membrane process for the concentration of effluent from an alkaline peroxide mechanical pulping (APMP) plant was investigated. Specifically, the cross-flow velocity and volume reduction were optimized again for a higher flux and a lower system energy consumption. A mathematical model was established to obtain the optimal parameters. Estimates were obtained of the expected savings in energy and water. The obtained optimal concentration conditions were: molecular weight cut-off at 10,000 Dalton, trans-membrane pressure at 3 bar, feed temperature at 50 ^oC, cross-flow velocity at 2 m/s, and volume reduction at 0.9. The average permeate flux under these conditions was 43.21 l/m^2.h. The total solids content was increased from 25.47 g/L in the feed to 128.36 g/L in the concentrate. The permeate had low total solids content of 11.03 g/L, Chemical Oxygen Demand of 9180 mg/l, and Biochemical Oxygen Demand of 5870 mg/L. Such qualities would allow the permeate to be reused in the APMP process after a light biochemical treatment. With this new membrane concentration process, about 1402 kWh energy can be saved and 22 m^3 effluent discharge can be reduced for each ton of pulp produced.

Keywords: Pilot-scale membrane process; Concentrating; Alkaline peroxide mechanical pulping; Effluent; Mathematical model; Energy and water saving; Discharge reduction

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INTRODUCTION

The pulp and paper industry is one of the most water-dependent industries (Nurdan and Emre 2010). Increased knowledge about the environmental effects of industrial activities has led to a need to develop better techniques and more efficient waste management systems in order to reduce their environmental impact (Stoica et al. 2009). The pulp and paper industry has made major process investments in environmental protection, especially through changes in internal processes, during the last few decades (Gönder et al. 2011). Even though the current wastewater discharge limits have been fulfilled, it is important to investigate whether further improvements could be made in the future (Leiviska et al. 2009).

The alkaline peroxide mechanical pulping (APMP) process has been widely adopted all over the world, especially in Asia (Liu et al. 2011), due to its advantages of high yield, high brightness, high strength, and low pollution. The effluent of APMP plants comprises those coming from the processes of chip washing, hot-water impregna-tion, squeezing extrusion, chemical impregnation, and mechanical refining. It is a mixed effluent. To achieve a closed wastewater loop, several APMP plants in the world have attempted to concentrate the total effluent by using a multi-effect evaporation system. The concentrated effluent is then blended with black liquor to feed alkali recovery boilers. But this process is always associated with very high energy consumption (Zhang et al. 2010).

Membrane filtration processes have received even more interest in recent years because of the stringent standards for water supply and effluent discharge (Zhang et al. 2009). For a membrane filtration process to be successfully used in the pulp and paper industry as a kidney, it must be tailored according to its special requirements (Wallberg et al. 2003). The low-cost and easily-fabricated polyethersulfone (PES) membrane, which could produce a reasonably large amount of purified process water and does not foul easily compared to other commercial membranes (Ahmad et al. 2010), is believed to be applicable in the treatment of effluent from APMP plants.

During the past ten years, in the fields of using membrane filtration processes to treat the effluents coming from pulp and paper mills, the researchers in Lappeenranta University of Technology (Finland) and Lund University (Sweden) have done many studies. Their studies mainly have focused on the treatments of kraft black liquor (Wallberg et al. 2003; Holmqvist et al. 2005; Wallberg et al. 2005), bleaching effluent (Fälth et al. 2001), and white water of paper machines (Huuhilo et al. 2001). The membrane materials adopted for these applications have been either polymeric or ceramic (Wallberg et al. 2003; Holmqvist et al. 2005; Wallberg et al. 2005). The filtration performance of using polyvinilidene fluoride (PVDF) membrane cooperating with polyethyleneimine (PEI) and polyvinylalcohol (PVA), which were used as water-soluble polymeric macroligands, to remove trace metals and COD from pulp and paper industrial wastewater were discussed by the researchers in the University of Maringa (Brazil) (Vieira et al. 2001). As a feasible synthetic polymer, PES has been widely used as a membrane material for various applications such as biomedicine, food, hemodialysis, plasma separator, and water purification. But its use in pulp and paper industry applications has been rarely reported. Related studies of PES membranes applied in the pulp and paper industry have mainly emphasized the fouling and cleaning of the membrane but not the treatment of the effluent coming from an APMP plant (Väisänen et al. 2002; Maartens et al. 2002).

In this work, an energy saving membrane separation process to concentrate the effluent from APMP plants has been studied in detail. The concentrated retentate from the membrane filtration then enters a multi-effect evaporation system for further concentration. The permeate is evaluated relative to its reuse in the APMP process. The ultimate goal is to reduce the energy and water consumption as well as the effluent discharge in APMP mills. According to our previous study (Zhang et al. 2010), it is believed that ultrafiltration with a PES flat-sheet membrane is suitable for the concentration process of APMP plant's effluent. So we scaled up the concentrating experiment to a pilot scale. Specifically, the cross-flow velocity (CFV) and volume reduction (VR) were optimized again for a higher flux and a lower system energy consumption. A mathematical model was established to obtain the optimal parameters. An estimation of energy and water saving effect were also developed.

EXPERIMENTAL

Materials

Apparatus

The scaled up concentrating experiments were carried out using a flat-sheet cross-flow filtration apparatus, which is one part of the self-designed multifunctional mem-brane separation equipment. The available membrane area of the flat-sheet cross-flow module used was 0.063 m^2. The mass of permeate was measured using a scale. The cross-flow velocity was measured by a rotameter and it was controlled by changing the pump velocity or by adjusting the manual valves. The same valves and the pump velocity were used to control the trans-membrane pressure (TMP). Feed temperature (FT) was adjusted by a heat-exchanger. Figures 1 and 2 respectively show the photographic appearance and a schematic diagram of the multifunctional membrane separation equipment. The shaded region represents the flat-sheet cross-flow filtration apparatus used.

Fig. 1. Scene photograph of the self-designed multifunctional membrane separation equipment

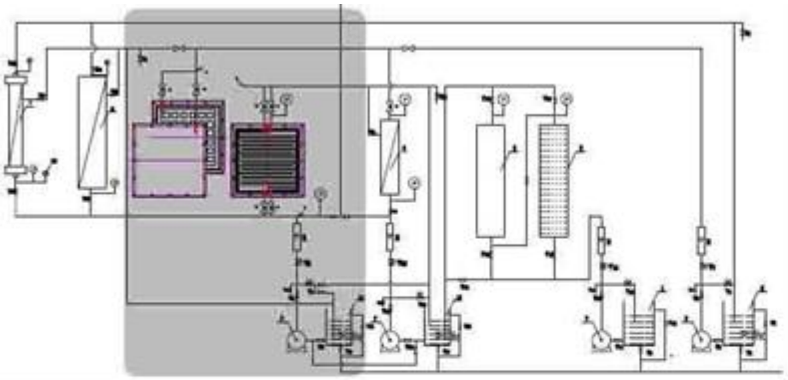
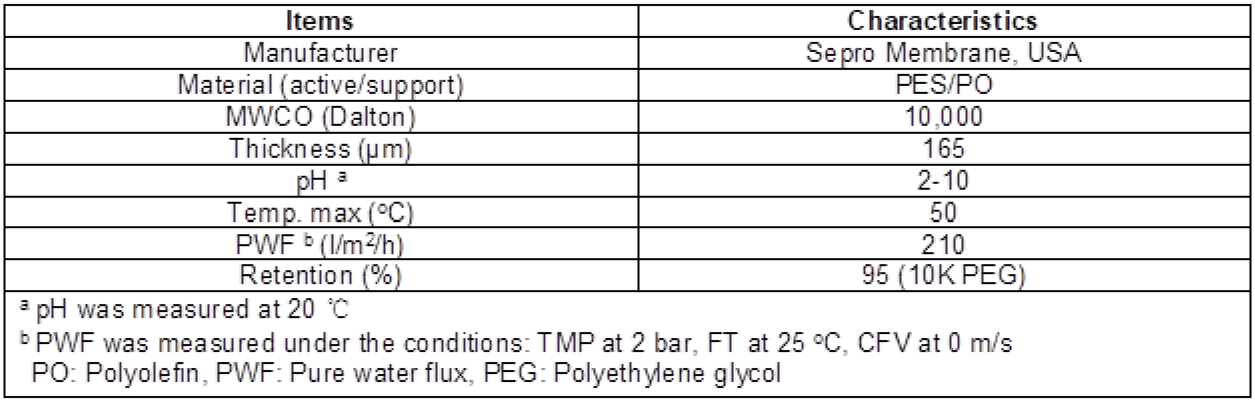


Fig. 2. Schematic diagram of the self-designed multifunctional membrane separation equipment

Membrane

According to our previous experimental results (Zhang et al. 2010), a PES 10 flat-sheet membrane, produced by Sepro Membrane, USA, with the molecular weight cut-off (MWCO) of 10,000 Dalton was chosen for this work. Table 1 shows its characteristics.

Table 1. Characteristics of the Used PES 10 Flat-sheet Membrane



Items	Characteristics
Manufacturer	Sepro Membrane, USA
Material (active/support)	PES/PO
MWCO (Dalton)	10,000
Thickness (μm)	165
pH [a]	2-10
Temp. max (°C)	50
PWF [b] ($l/m^2/h$)	210
Retention (%)	95 (10K PEG)

[a] pH was measured at 20 ℃

[b] PWF was measured under the conditions: TMP at 2 bar, FT at 25 °C, CFV at 0 m/s

PO: Polyolefin, PWF: Pure water flux, PEG: Polyethylene glycol

Effluent

The APMP plant's effluent came from Sun Paper Co., China, which uses poplar as the raw material for its APMP producing. It was a comprehensive wastewater that included a mixture of wastewaters from the chip washing process, hot-water impregna-tion process, squeezing extrusion process, chemical impregnation process, refining process, and pulp bleaching process. Generally speaking, the main components in the effluent were typically carbohydrates, extractives, lignin, low-molecular weight organic acids, proteins, and inorganic ions (Zhang et al. 2010). The effluent, pre-treated with a 150 μm bend screen, was used as the feed for the membrane concentration experiments. The main parameters of the feed are presented in Table 2.

Table 2. Characteristics of the Feed Used in the Membrane Concentration Experiments

Parameters	Average values
Total solids content (g/l)	25.47±0.32
Total organic matter (g/l)	14.59±0.04
Ash content (g/l)	11.09±0.08
Heating value (kJ/g dry solids)	12.68±0.63
COD (mg/l)	18300±880
BOD$_5$ (mg/l)	7300±310
pH	7.50±0.12
Conductivity (mS/cm)	15.89±0.10
Colour (PtCo)	1900±45

Methods

Before each filtration, a fresh membrane was first stabilized with distilled water under the conditions of 2 bar, 25 °C, and 0 m/s until its pure water flux remained stable. During the membrane concentration process, the concentrate was retained in the system and the permeate was collected from the outside. The endpoint of every concentration experiment was determined by the volume reduction (VR), which is the volume ratio between the permeate and the initial feed.

Optimal CFV and VR

The cross-flow velocity (CFV) and volume reduction (VR) are two important factors in this membrane concentration process, as they relate to concentration effect and production cost. A series of experiments for optimal cross-flow velocity and volume reduction were performed based on the optimized filtration conditions (Zhang et al. 2010). A mathematical model was developed using MATLAB 7.8 to calculate the optimal VR.

Analysis

Samples of the feed, concentrate, and permeate were all measured to obtain the values of total solid, total organic matter, ash content, heating value, COD, BOD_5, pH, conductivity, and colour. Total solid and ash content were analysed according to the SFS 3008 standard method. Total organic matter was calculated as the difference between total solids and ash content. Heating value analysis was carried out using a GR-3500 oxygen bomb calorimeter according to the ISO 1928 - 1995 standard method. COD was measured according to the SFS 5504 standard method. BOD_5 was analysed according to the ISO 5815 - 1, 2: 2003 standard method. The pH was determined according to the UDC 663. 6: 543. 06 standard method. Conductivity was analysed using the ISO 7888-1985 standard method. Colour was analysed according to the ISO 7887 - 1985 standard method.

Calculations

The volume reduction was calculated using Eq. (1), where V_p is the permeate volume (l) and V_f the initial volume of feed (l).

$$VRF = \frac{V_p}{V_f} \tag{1}$$

The average permeate flux at a specific VR ($l/m^2.h$) was calculated by Eq. (2), where V is the permeate volume (l), S the filtration area (m^2), and t the filtration time (h).

$$J = \frac{V}{S \times t} \tag{2}$$

The production cost of this membrane concentration process can be calculated by Eq. (3),

$$Pc = Cc + Oc - Ov \tag{3}$$

where Pc is the production cost, Cc the capital cost, Oc the operating cost; Ov the value of organics in concentrate. Then Cc, Oc, and Ov are respectively given by,

$$Cc = A \times (Cost_{\text{memb}} \times A_{\text{memb}} + Cost_{\text{equip}}) \quad (4)$$

$$Oc = B \times (Cost_{\text{electricity}} + Cost_{\text{replacement of memb}} + Cost_{\text{maintenance}} + Cost_{\text{cleaning}} + Cost_{\text{labor}}) \quad (5)$$

$$Ov = C \times (m_{\text{organics}} \times H_{\text{fuel}}) \quad (6)$$

where A, B, and C are the factors accounting for market prices. Because cross-flow velocity is the dominant factor to consumed power of pump, and volume reduction is the key factor to running time of pump, the relationship among CFV, VR and $Cost_{\text{electricity}}$ can be expressed with Eq. (7) as,

$$Cost_{\text{electricity}} = D \times CFV \times VR \quad (7)$$

where D is the price factor. Otherwise, in Eq. (6), $m_{organics}$ is the amount of organics in the concentrate and H_{fuel} the net heating value of the fuel, i.e., the heating value of organics in the concentrate minus the heating value needed for the evaporation of water in the concentrate during combustion. Thus H_{fuel} is given by,

$$H_{\text{fuel}} = H_{\text{organics}} \times X_{\text{organics}} - 20.5 \times [1 - TS] \quad (8)$$

where H_{organics} is the heating value of organics in the concentrate, X_{organics} is the weight percentage of organics, and TS the content of total solids in the concentrate. The contribution of other substances besides organics to the heating value of concentrate was assumed to be negligible. The heating values of lignin and carbohydrates are 25.5 MJ/kg and 12.5 MJ/kg, respectively (Grace et al. 1989). The value of 20.5 MJ/kg was used in the calculations based on the proportion of lignin and carbohydrates in hardwood. The influence of the VR on TS can be calculated as,

$$TS = TS_0 \times [1/(1 - VR)]^{R_{\text{ts}}} \quad (9)$$

where TS_0 is the initial total solids content in the feed, and R_{ts} the observed retention of total solids. The constant R_{ts} can be determined by fitting Eq. (9) to the data shown in Table 4, using nonlinear regression.

RESULTS AND DISCUSSION

Optimal CFV and VR

According to Eq. (5), the operating cost (Oc) consists of mainly power consumption, replacement of membrane, maintenance, cleaning, and labor costs. In a specific process, all the operating costs are constant other than power consumption, which is affected by cross-flow velocity and volume reduction. According to Eq. (9), the volume reduction is expected to have an indirect influence on the value of organics in the concentrate. So these two factors should need to be optimized again in this scale-up study to achieve the lowest production cost. Based on the previous results obtained from our laboratory-scale study (Zhang et al. 2010), it was first assumed that the optimal volume reduction was 0.93. The cross-flow velocities were set at 0.5 m/s, 1 m/s, 1.5 m/s, 2 m/s, 2.5 m/s, and 3 m/s, respectively, which are within the possible cross-flow velocity range for the used apparatus. Table 3 shows the influence of cross-flow velocity on membrane concentration effect. With the increasing of CFV, the permeate flux increased correspondingly from 11.62 l/m^2.h to 50.20 l/m^2.h. The power consumption decreased before CFV reached 2 m/s, but after that it increased quickly up to 8.12 kWh. So 2 m/s was chosen to be the optimal CFV for this membrane concentration process.

Table 3. Influence of CFV on Membrane Concentration Effect *

Parameters	Average values	
CFV (m/s)	Permeate flux (l/m^2.h)	Power consumption (kWh)
0.5	11.62	10.80
1.0	22.65	7.82
1.5	32.94	5.06
2.0	41.23	3.51
2.5	47.55	5.05
3.0	50.20	8.12

* Experimental conditions: MWCO at 10,000 Dalton, TMP at 3 bar, FT at 50℃, VR at 0.93
Feed volume: 15 L

A complete membrane concentration process was carried out from VR at 0.14 to VR at 0.95. The VR increased going with the increasing of filtration time. The total solids content in concentrate and the power consumption were also tested in this process. A trend line expressing the relationship between volume reduction and production cost was then fitted with MATLAB 7.8. This is presented in Fig. 3 and Table 4.

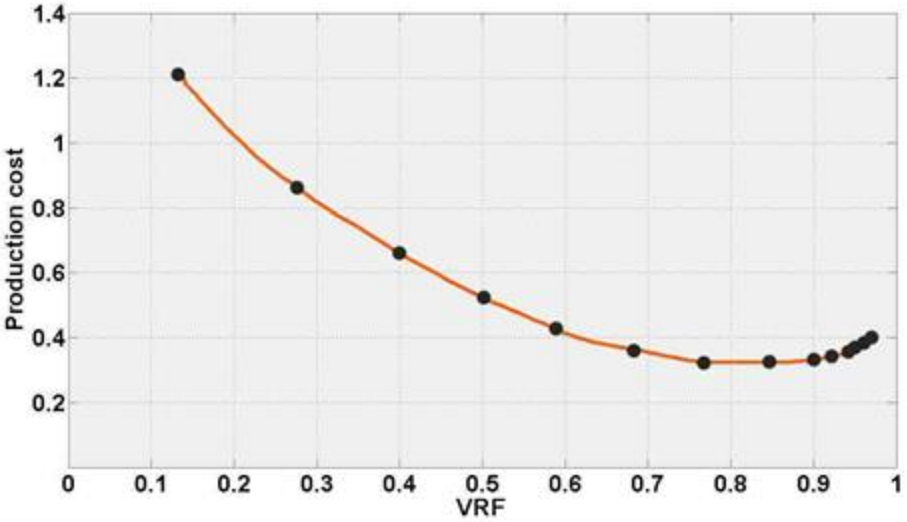
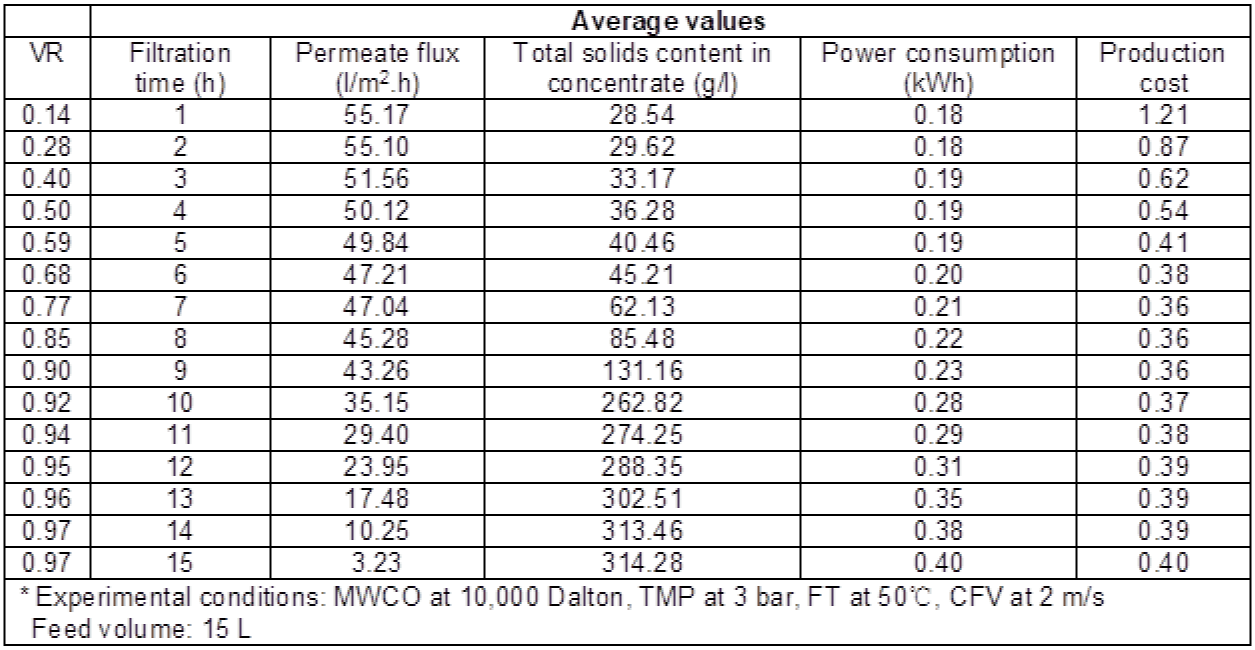


Fig. 3. Relationship between VR and production cost of membrane concentration process

Table 4. Influence of VR on Membrane Concentration Effect *



	Average values				
VR	Filtration time (h)	Permeate flux ($l/m^2.h$)	Total solids content in concentrate (g/l)	Power consumption (kWh)	Production cost
0.14	1	55.17	28.54	0.18	1.21
0.28	2	55.10	29.62	0.18	0.87
0.40	3	51.56	33.17	0.19	0.62
0.50	4	50.12	36.28	0.19	0.54
0.59	5	49.84	40.46	0.19	0.41
0.68	6	47.21	45.21	0.20	0.38
0.77	7	47.04	62.13	0.21	0.36
0.85	8	45.28	85.48	0.22	0.36
0.90	9	43.26	131.16	0.23	0.36
0.92	10	35.15	262.82	0.28	0.37
0.94	11	29.40	274.25	0.29	0.38
0.95	12	23.95	288.35	0.31	0.39
0.96	13	17.48	302.51	0.35	0.39
0.97	14	10.25	313.46	0.38	0.39
0.97	15	3.23	314.28	0.40	0.40

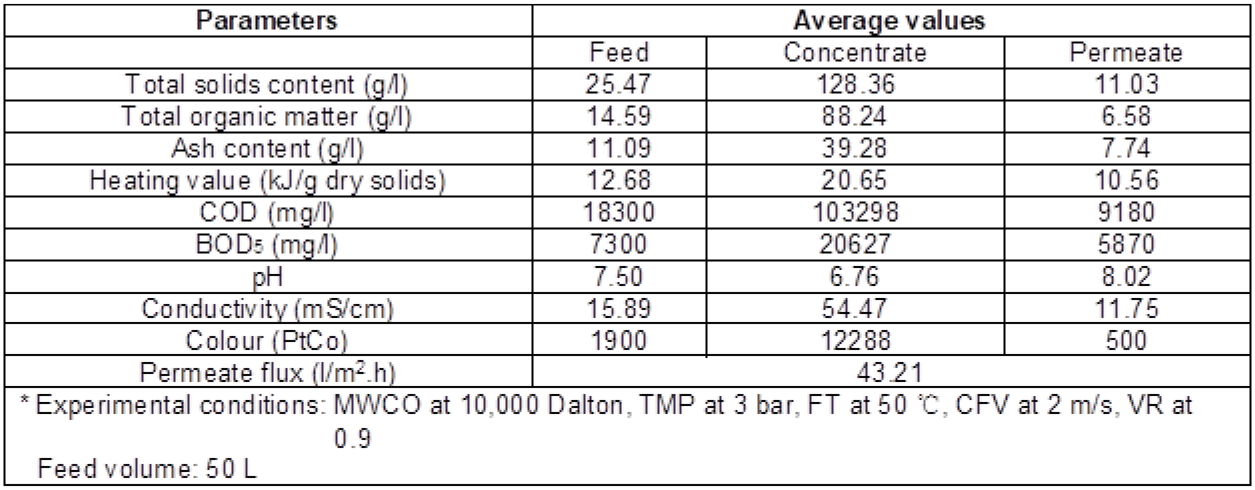
* Experimental conditions: MWCO at 10,000 Dalton, TMP at 3 bar, FT at 50℃, CFV at 2 m/s
Feed volume: 15 L

Before reaching 0.9, the production cost decreased gradually with increasing VR. But after that point, the production cost started to increase slowly. Meanwhile, the permeate flux decreased more quickly. So 0.9 was judged to be the optimal VR for this membrane concentration process.

Verification Experiment

A verification experiment was repeated immediately after obtaining the optimal filtration conditions. Table 5 shows the final concentration effects for APMP effluent feed, concentrate (retentate), and permeate. The average permeate flux under these conditions was 43.21 $l/m^2.h$.

Table 5. Characteristics of the Feed, Concentrate, and Permeate after the Verification Experiment *



Parameters	Average values		
	Feed	Concentrate	Permeate
Total solids content (g/l)	25.47	128.36	11.03
Total organic matter (g/l)	14.59	88.24	6.58
Ash content (g/l)	11.09	39.28	7.74
Heating value (kJ/g dry solids)	12.68	20.65	10.56
COD (mg/l)	18300	103298	9180
BOD_5 (mg/l)	7300	20627	5870
pH	7.50	6.76	8.02
Conductivity (mS/cm)	15.89	54.47	11.75
Colour (PtCo)	1900	12288	500
Permeate flux (l/m^2.h)	43.21		
* Experimental conditions: MWCO at 10,000 Dalton, TMP at 3 bar, FT at 50 ℃, CFV at 2 m/s, VR at 0.9 Feed volume: 50 L			

The content of total solid was raised from 25.47 g/L in the feed to 128.36 g/L in the retentate. The heating value of the retentate, relative to the feed, was increased from 12.68 kJ/g dry solids to 20.65 kJ/g dry solids, indicating that the utilization value of APMP plant's effluent was enhanced. The permeate had a low total solids content of 11.03 g/L, COD of at 9180 mg/L, and BOD_5 of 5870 mg/L, and was likely to be directly reused in APMP pulping process for wet feed preparation, coarse pulp washing, or lime mud washing after lightly biochemical treatment, according to the results of the later permeate reuse experiments (Zhang 2009).

Economic Benefits Calculation

The benefits in energy and water savings, as well as discharge reduction for every ton of APMP pulp produced using this new membrane concentration process under its optimal conditions, can be calculated based on the data in Table 5. Assume that there is 25 m^3 effluent generated for each ton of pulp produced. When the content of total solid is increased from 25.47 g/L in the feed to 128.36 g/L in the concentrate:

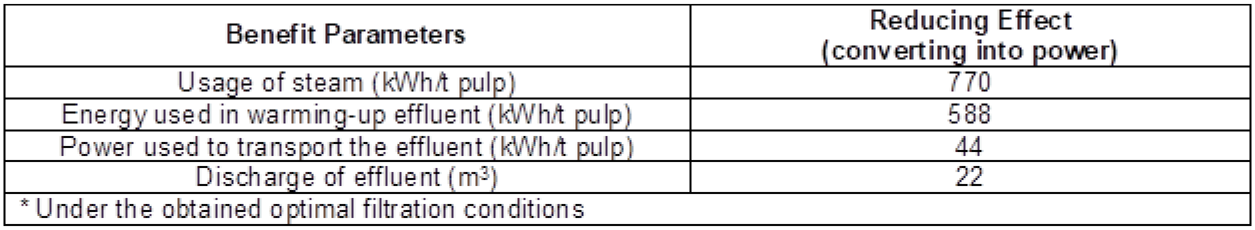
1) As a rule-of-thumb the energy requirement during evaporation is 35 kWh/m^3 vaporized water in five-effect evaporation station; thus the reduced usage of steam is 35×22=770 kWh/t pulp; where 22 m^3 is the permeate coming from the 25 m^3 total effluent;

2) Assuming that the specific heat of the effluent with the content of total solid between 25.0 g/L and 128.0 g/L is 3.85 kJ/kg.^{o}C; the heat energy used to preheat the permeate from 50 ^{o}C to 75 ^{o}C, converting into power, is about [3.85×22×1000×(75-50)]/3600=588 kWh/t pulp; so the reduced energy used in preheating effluent is 588 kWh/t pulp;

3) The energy consumption used to transport effluent from APMP plant to five-effect evaporation station is 2 kWh/t effluent; thus the reduced power used to transport the effluent is 22×2=44 kWh/t pulp;

4) Because of the reuse of all permeate, the reduced discharge of effluent is about 22 m^3; all of the above findings are summarized in Table 6.

Table 6. Benefits in Energy and Water Saving, and Discharge Reduction Obtained from this New Membrane Concentration Process *



Benefit Parameters	Reducing Effect (converting into power)
Usage of steam (kWh/t pulp)	770
Energy used in warming-up effluent (kWh/t pulp)	588
Power used to transport the effluent (kWh/t pulp)	44
Discharge of effluent (m^3)	22
* Under the obtained optimal filtration conditions	

With this new membrane concentration process, 88% of the water in the effluent can be removed. In an APMP plant that has an annual pulp production capacity of one million tons, for instance, 1.40 billion kWh power could be saved. The capital investment for multi-effect evaporation system could also be decreased by 88% correspondingly. The discharge of effluent could be reduced by 22 million m^3 per year. More details about the economic calculation were given by Zhang (2009).

CONCLUSIONS

1. A pilot-scale membrane separation process, using PES flat-sheet membranes, to concentrate an alkaline peroxide mechanical pulping (APMP) plant's effluent was investigated. The obtained optimal filtration conditions were: molecular weight cut-off at 10,000 Dalton, trans-membrane pressure at 3 bar, feed temperature at 50 ^oC, cross-flow velocity at 2 m/s, and volume reduction at 0.9. The average permeate flux under these conditions was 43.21 $L/m^2.h$. The total solids content was increased from 25.47 g/L in the feed to 128.36 g/L in the concentrate. The permeate had low total solids content of 11.03 g/L, Chemical Oxygen Demand of 9180 mg/L, and Biological Oxygen Demand of 5870 mg/L. Such qualities would allow permeate to be reused in an APMP process after light biochemical treatment.
2. Using this membrane concentration process under its optimal conditions, APMP mills could totally save 1402 kWh power and reduce 22 m^3 effluent discharge for every ton of pulp produced, and the capital investment in multi-effect evaporation system could also be decreased by 88%. These data suggested that the technology to concentrate APMP plant's effluent using ultra-filtration process with PES flat-sheet membrane has a great potential for industrial-scale application. Of course, large-scale pilot plant trials are needed for further validation.

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