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Internal Purification of Thermomechanical Pulp Clear Filtrate with a Combined Biological and Membrane Filtration Method: A Preliminary Study

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ABSTRACT

In this study a combined anaerobic biological–nanofiltration method was studied as a means of internal purification of a thermomechanical pulp (TMP) plant effluent. A TMP plant clear filtrate was first subjected to a thermophilic anaerobic treatment at 55 or 70°C in an upflow anaerobic sludge blanket reactor and then nanofiltered in a flat-sheet laboratory-scale module. The anaerobic treatment removed 55% of the chemical oxygen demand at 70°C and 65% at 55°C. Sugars were removed both at 55 and 70°C while the low molar mass ligneous material was removed only at 55°C. By nanofiltration the remaining low molar mass ligneous material was removed by about 98–99% and the high molar mass ligneous material by 96–99%. Sugar was removed by 88–98% and chemical oxygen demand by 78–81%. It was also shown that most of the pulp-brightness-decreasing substances had been removed. The permeate flux depended on the sample but was at its best [about 38 L/(m^2·h) at 8 bar] for the first hour of filtration for the sample anaerobically treated at 55°C. The samples did not cause permanent fouling of the membrane. In this study it was shown that the combined anaerobic biological–nanofiltration method is a competitive internal purification method for TMP plant clear filtrate resulting in a very clean water, which could be reused in the water circula-

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tion system of the plant or in the paper machine white-water system. However, further studies for optimization of the process are needed.

Key Words. Thermophilic; Anaerobic; UASB; Nanofiltration; Thermomechanical pulping; Internal purification; Closed water circulation system

INTRODUCTION

Today the totally closed water circulation system in the pulp and paper industry has become an object of intensive research due to stringent environmental legislation and economic reasons. In a totally closed water circulation system the consumption of fresh water and the losses in fiber and fines are minimized. On the other hand, the temperature in the closed system is elevated and, together with the enrichment of, e.g., dissolved and colloidal substances and charged material, this leads to increased amounts of deposits and corrosive effects as well as to a decrease in the optical characteristics of pulp. Therefore an internal purification of process waters becomes a necessity if undisturbed runnability in an integrated pulp and paper mill and a high quality of products are to be maintained.

It has previously been shown that, e.g., ultrafiltration of kraft mill bleach effluents improves mill closure and facilitates anaerobic or aerobic external effluent treatment (1). A new purification process for multicomponent wastewaters has recently been developed by Rautenbach and Mellis (2). This process consists of an activated sludge bioreactor, a nanofiltration stage, and either a chemical oxidation or an adsorption stage.

The use of a biological process as an internal treatment unit alone or combined with other operation units has been studied on the laboratory- and the pilot-scale (3–5) and has also been applied in a full-scale plant (6). Recent studies have demonstrated that a thermophilic anaerobic process can be used to treat hot and concentrated pulp and paper industry process waters and wastewaters at around 55°C (7–9) and even at 70°C (9).

Anaerobic biological treatment removes only low molar mass compounds, which, e.g., in thermomechanical pulping (TMP) plant circulation waters are mainly polysaccharides. Anaerobic treatment removes only slightly or not at all the ligneous material or low molar mass ionic compounds present in pulp and paper industry process waters.

Membrane filtration methods, such as ultra- and nanofiltration as well as reverse osmosis, can be used as efficient internal purification methods in the pulp and paper industry (10–12). Ultra- and nanofiltration of pulp and paper effluents, such as bleach effluents (13–15), effluents from debarking (16), mechanical pulp manufacture (15), and also paper machine circulation waters have been studied (15, 17–21). In these studies it was shown that most of the ligneous material and low molar mass ionic compounds can be removed from a paper machine clear filtrate by nanofiltration.

In this study the possibilities of applying a combined anaerobic biological–nanofiltration method as an internal purification process for TMP plant clear filtrate were studied.

MATERIALS AND METHODS

The TMP Clear Filtrate

The TMP clear filtrate used in this study originates from an integrated Finnish TMP plant–paper machine where spruce is used in TMP manufacture. The TMP clear filtrate is one of the two water streams resulting after the accept from the centrifugal cleaners have been thickened with a save-all disc in the TMP plant. At the moment the TMP clear filtrate is an effluent, but after sufficient purification it could be reused in the TMP plant and in the paper machine white-water system.

For anaerobic studies the clear filtrate was first filtered through a paper filter (Whatman Qualitative No. 4 filter paper) to avoid clogging in the small diameter feed tubing. Micro and trace nutrients were added to the clear filtrate as described previously (9). Sodium bicarbonate (1 g $NaHCO_3$/g COD) was added to provide buffer capacity, and HCl was used to adjust the pH of the feed solution to 6.9–7.1. A new feed solution was prepared 1 to 3 times a week. The feed solution was flushed with nitrogen and then stored at 4°C under nitrogen.

In the nanofiltration studies three different TMP clear filtrate samples were used: untreated clear filtrate (no nutrient additions and no pH adjustment, TMP1), clear filtrate treated with anaerobic bacteria at 55°C (TMP2) and at 70°C (TMP3). The TMP2 and TMP3 samples were exposed to oxygen prior to nanofiltration. The pure water used throughout the nanofiltration experiments was ion exchanged and filtered through a Millipore reverse osmosis (RO) unit giving a conductivity < 1 μS/cm.

Anaerobic Reactor Experiments

Two glass upflow anaerobic sludge blanket (UASB) reactors (referred to as R1 and R2) with a total liquid volume of 400 mL were used in this study. R1 was run at 55°C and R2 at 70°C. The experimental set-up was as described by Jahren and Rintala (5).

The reactors were inoculated with granular sludge [17 g volatile solids (VS) per reactor] from a full-scale mesophilic UASB reactor treating starch industry wastewaters in Jokioinen, Finland. The sludge was stored anaerobically for 3 months before the experiments.

Nanofiltration (NF) Experiments

The nanofiltration experiments were carried out in a laboratory crossflow flat-sheet module made of poly(carbonate) with an effective membrane area of 53 cm^2. NTR-7250 membranes from Nitto Denko were used. They are hydrophilic thin film composite membranes consisting of a poly(vinyl alcohol)/poly(piperazine amide) skin layer on a polysulfone support. The isoelectric point (IEP) of the NTR-7250 membrane is about pH 4 (22), thus at a pH higher than 4 the membrane has a small negative charge. The pure water flux (PWF) measured at 8 bar and 25°C was 30.8 $L/(m^2 \cdot h)$.

Before the experiments the membrane was stabilized and the PWF at 25°C was measured as a function of pressure from 4 to 12 bar. The nanofiltration experiments were performed under constant conditions: pressure 8 bar, flow rate 2 m/s, and temperature 40°C. The same membrane was used throughout the experiment, but it was washed with pure water for 30 minutes between the runs. The permeate flux was measured during the runs. The membrane cleaning procedure after the experiments was: 1) pure water at maximum flow rate (4 m/s) for 1 hour, 2) pure water for 1.5 hours, 3) pure water 6 bar and 2.8 m/s flow rate. Then the PWF at 25°C was measured as a function of pressure as above.

Methods of Analysis

The original water samples (feeds) as well as the resulting retentates (enriched fraction) and permeates (cleaner fraction) were analyzed for their ionic content (conductivity and pH), lignin residuals (UV/VIS absorption at 280 and 400 nm), sugar content (anthrone-sulfuric acid color method), and chemical oxygen demand, COD (Hach reactor, SFS 5504). The brightness decreasing/increasing effect was tested by filtering the sample through a cake made of eucalyptus pulp and pure RO water (100 mL 3 times through, 40°C). The brightness, Y-value, and yellowness were measured by an Elrepho 2000 spectrophotometer. The total amount of iron-containing compounds was analyzed by a FerroVer method (λ = 512 nm) for the untreated TMP water (TMP1) only, because the strong color in the other samples disturbed the analysis. Volatile solids (VS) were analyzed according to standard methods (APHA, 1992).

The reductions R of different measured substances have been calculated by comparing the concentrations of the substance in the permeate and in the feed. The concentrations of substances in feeds and permeates were presented relative to their concentrations in the TMP1 feed (= 100), called here the relative content.

RESULTS AND DISCUSSION

Anaerobic Reactor Experiments

The anaerobic reactors were operated for 106 days (5). The COD removal started at 55°C and at 70°C after 17 and 23 days of operation, respectively. The load was gradually increased up to 5.7–5.8 kg COD/(m^3·d) during the first 70 days of operation. The COD reduction improved gradually to reach a stable level after 50 days (independent of the load). At the end of the runs (days 102–106) the mean COD removals were 64.7 and 54.8% at 55 and 70°C, respectively (Table 1).

More detailed analysis (Table 2) showed that the sugar content of the clear filtrate decreased by 85 to 95% during the thermophilic treatment. The decrease of low molar mass ligneous material (UV_{280}) was significant only at 55°C. The treatments lowered the brightness and the Y-value and increased the yellowness of the clear filtrate. The clear filtrate looked like water with a very light yellow color. The clear filtrate treated at 55°C had a medium/dark brown color while the clear filtrate treated at 70°C had a dark reddish-brown color and a strong unpleasant urea-like odor. The effluents from the anaerobic reactors looked immediately after sampling almost similar to the untreated clear filtrate. The color in the anaerobically treated clear filtrates was apparently due to exposure of the samples to oxygen (9, 23, 24), and it has been suggested by Sierra-Alvarez et al. (23) that it is due to autoxidation of phenolic compounds.

The present results are in good agreement with previous studies (9) with clear filtrate from the same mill. In the present study high loading rates

TABLE 1
Performance of 55 (R1) and 70°C (R2) UASB Reactors Treating TMP Clear Filtrate

	R1	R2
Load, kg COD/m^3·d	5.7 ± 0.4	5.8 ± 0.1
HRT, h	9.4 ± 1.0	9.0 ± 0.0
COD removal, %	64.7 ± 1.2	54.8 ± 2.3

TABLE 2
Characteristics of TMP Clear Filtrate Samples, Untreated and Treated with Anaerobic Bacteria. TMP1 = untreated, TMP2 = treated at 55°C, TMP3 = treated at 70°C. Brightness, Y-Value, and Yellowness were Determined Using Eucalyptus Pulp. nm = Not Measured

	TMP1	TMP2	TMP3
Sample mass, g	2695	2620	2369
pH, —	4.6	7.5	7.6
Conductivity, mS/cm	0.61	4.22	4.64
COD, mg/L	2200	770	970
Sugar content, ppm	748	105	48
UV_{280}, —	7.81	4.44	7.02
UV_{400}, —	0.49	1.06	1.28
Total Fe content, ppm	26	nm	nm
Brightness, —	83.6	41.7	51.6
Y-value, —	88.6	53.2	60.2
Yellowness, —	7.3	30.4	20.2

were not attempted while earlier (9) in a 55°C UASB reactor loading rates even up to about 80 kg COD/(m^3·d) corresponding to 55 minutes hydraulic retention time (HRT) were achieved. Thus, these results together with the previous results clearly indicate that anaerobic treatment of TMP clear filtrate at temperatures of 55 and 70°C is feasible.

However, the suitability of an anaerobic process as a part of an internal purification system requires further studies. For example, the need for nutrient additions and pH adjustment should be more closely investigated as they also affect the performance of the following membrane unit.

Nanofiltration Experiments

The flux of the TMP2 sample decreased by 50% during an experiment of 160 minutes (Fig. 1). However, until 60 minutes the flux of the TMP2 was better than those of the TMP1 and the TMP3 but started rapidly to fall afterward. This phenomenon could be explained by the formation of bacterial aggregates which may form a preventive secondary membrane due to concentration polarization. The flux of the TMP1 sample decreased by 22% in 180 minutes and 35% in 442 minutes. The TMP3 flux remained almost constant; only a 2% decrease in 180 minutes was observed. Thus the higher pH improved the performance of nanofiltration. However, the samples did not cause permanent fouling of the membrane because the pure water flux was more or less recovered after the experiments.

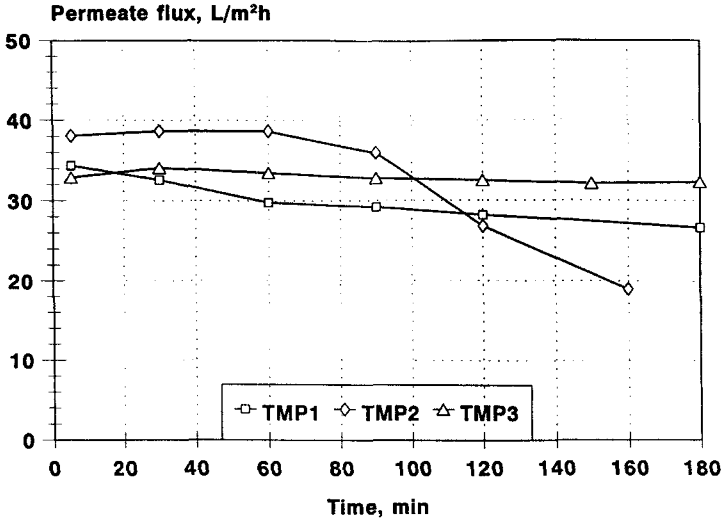
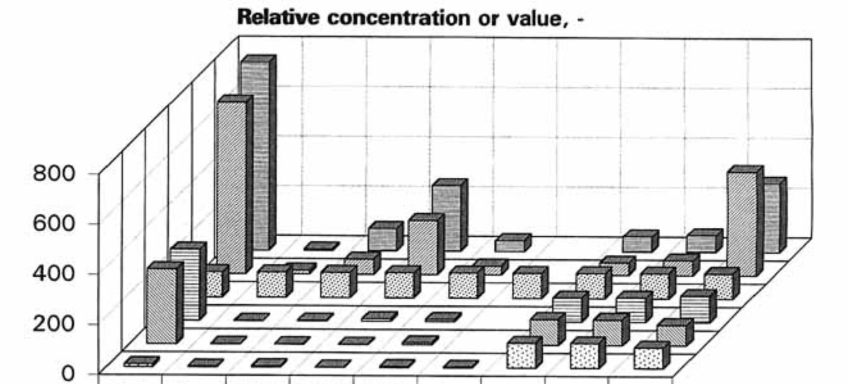


FIG. 1 The permeate fluxes in nanofiltration experiments, pressure 8 bar, flow rate 2 m/s, and temperature 40°C, membrane NTR-7250. TMP1 = untreated, TMP2 = treated at 55°C, TMP3 = treated at 70°C.

The anaerobic treatment increased the amount of high molar mass ligneous material (UV_{400}) and decreased the sugar content (Fig. 2). The low molar mass ligneous material (UV_{280}) was decreased significantly only after the anaerobic treatment at 55°C. The increase in the UV_{400} of the TMP2 and TMP3 feeds was probably due to the existence of autoxidated phenolic compounds (9, 23) and the higher pH of these samples.

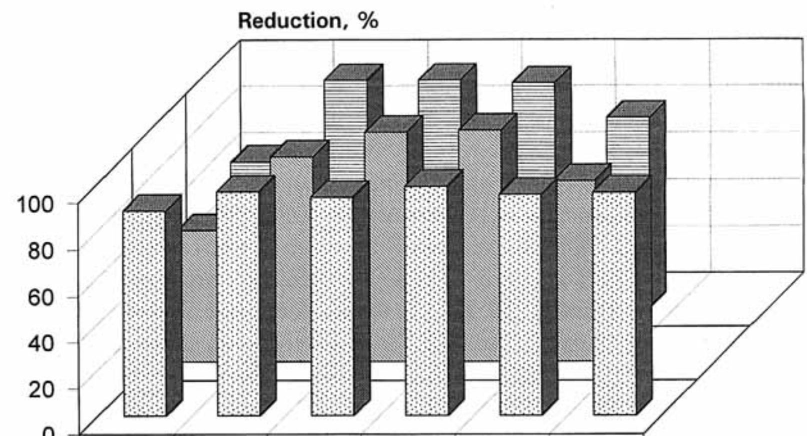
The higher temperature in the anaerobic stage resulted in better reductions of sugar and COD, as seen in Fig. 3, when the reductions obtained for the TMP2 (55°C) and the TMP3 (70°C) were compared. Comparing the TMP3 with the untreated TMP1, it was seen that almost equal reductions were obtained except for the conductivity and COD. The COD reduction was more than 10% lower in the NF of the TMP3 than in the NF of the untreated TMP1. Considering the fact that the COD contents in the anaerobically treated samples were much lower than in the untreated sample, it seems likely that some very low molar mass compounds were formed at the anaerobic stage by biodegradation.

The reductions of low and high molar mass lignous material were high (>96%) for both TMP2 and TMP3 (Fig. 3). Thus the temperature in the anaerobic stage did not have any significant effect on it. However, the



	Cond.	Sugar	UV(280)	UV(400)	COD	TotFe	Bright.	Y-val.	Yell.
TMP1 P	11.4	3.4	5.7	1	4.6	3.9	100.9	99.9	83.6
TMP2 P	297.2	1.7	0.8	1.4	7.7		102.3	101.2	81.6
TMP3 P	282.6	0.2	2.2	9.6	8.2		95.8	96	104.1
TMP1 F	100	100	100	100	100	100	100	100	100
TMP2 F	687.3	14.1	59.9	216.2	35		49.9	60	415.3
TMP3 F	755.7	6.5	89.9	262.7	44.1		61.7	67.9	275.3

FIG. 2 The relative concentrations of different substances and relative values of brightness, Y-value, and yellowness in feeds (F) and permeates (P) in NF experiments of TMP1, TMP2, and TMP3. The concentrations and values of the TMP1 feed were set equal to 100. The NF parameters were: pressure 8 bar, flow rate 2 m/s, and temperature 40°C, membrane NTR-7250. TMP1 = untreated, TMP2 = treated at 55°C, TMP3 = treated at 70°C.



	Cond.	Sugar	UV(280)	UV(400)	COD	TotFe
TMP1	88.6	96.6	94.3	98.9	95.4	96.2
TMP2	56.8	88.1	98.6	99.3	77.9	
TMP3	62.6	97.5	97.5	96.3	81.4	

FIG. 3 The reductions of different measured parameters in the nanofiltration experiments, pressure 8 bar, flow rate 2 m/s, and temperature 40°C, membrane NTR-7250. TMP1 = untreated, TMP2 = treated at 55°C, TMP3 = treated at 70°C.

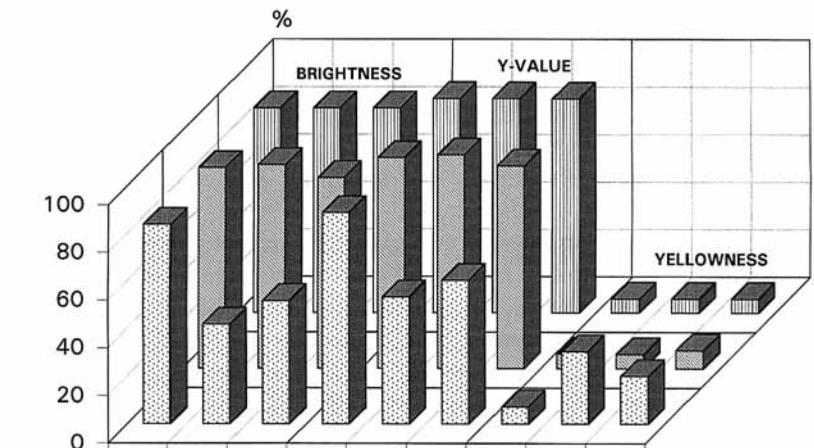
anaerobic treatment enhanced the removal of UV_{280} at both temperatures and UV_{400} at 55°C, which was probably due to the different pH causing some structural changes in these compounds.

The reduction of conductivity in nanofiltration of the treated samples was rather low (57–63%) compared to the untreated TMP1 (87%). This was, however, to be expected, because in aqueous solution the sodium bicarbonate and HCl, which were added to the feed prior to the anaerobic stage, dissociate into monovalent ions, which are not well retained by the nanofiltration membrane. The membrane manufacturer reports a NaCl rejection of 50% and a $MgSO_4$ rejection of 98% for NTR-7250 membranes. Nevertheless, the ion content should be further studied in order to clarify the nature of the charged material left in the waters and to judge its harmfulness to a closed water circulation system.

The brightness decreasing/increasing tendency of the permeates, retentates, and feeds was judged by their brightness, Y-value, and yellowness. As a reference, values given by pure RO water were used. In the brightness test the wavelength of light used was 457 nm and in the Y-value test 557 nm, which describes more accurately what a human eye can see. In general, the higher the values, the better. In the yellowness test the yellow part of spectral light is emphasized. Thus the smaller the yellowness, the better.

The anaerobic treatment at 55°C had a slight brightness increasing (+1.2 brightness units) and the treatment at 70°C a brightness decreasing tendency (−4.2 brightness units) compared to the untreated sample as seen very clearly in Fig. 4. The TMP2 permeate had about 2 units higher brightness than the TMP1 feed (the original clear filtrate) and was almost equal to the brightness given by pure RO water. Also the Y-value and yellowness of the TMP2 permeate were almost equal to those of RO water. Thus most of the brightness-decreasing substances in the TMP2 were removed by NF treatment. From the brightness point of view, as good results were not obtained by only nanofiltering the clear filtrate. This could indicate that the degradation products formed during the anaerobic treatment at 55°C were better retained in nanofiltration due to electrostatic repulsion, which would also explain the TMP2 permeate flux behavior during the first 60 minutes of filtration seen in Fig. 1.

However, a straight correlation between the concentrations of different compounds in the samples and brightness cannot be drawn (Fig. 2). The anaerobic treatment at 55°C (TMP2 feed) increased the yellowness and decreased the brightness and the Y-value more than the treatment at 70°C (TMP3 feed) compared to the TMP1 feed level. Moreover, the UV_{400} was over two times higher in the TMP2 and TMP3 feeds than in the TMP1 feed. This could indicate that the anaerobic treatment at lower temperature increases the content of chromophoric groups in the high molar mass



	TMP1	TMP2	TMP3	TMP1	TMP2	TMP3	TMP1	TMP2	TMP3
Feed	83.6	41.7	51.6	88.6	53.2	60.2	7.3	30.4	20.2
Permeate	84.3	85.5	80.1	88.6	89.7	85.1	6.1	5.9	7.6
RO-water	86.1	86.1	86.1	90.2	90.2	90.2	5.9	5.9	5.9

FIG. 4 The brightness, Y-value, and yellowness of TMP1, TMP2, and TMP3 feeds and permeates obtained in the NF experiments. The reference values were obtained by RO-water. Determinations were made with eucalyptus pulp. The NF parameters were: pressure 8 bar, flow rate 2 m/s, and temperature 40°C, membrane NTR-7250. TMP1 = untreated, TMP2 = treated at 55°C, TMP3 = treated at 70°C.

ligneous material through structural changes. On the other hand, the TMP2 and TMP3 feeds also had a higher pH, which also is known to give a brightness decreasing effect. It also seemed that the lower level of sugar and low molar mass ligneous material (UV_{280}) content in the TMP2 feed compared to the TMP3 feed improved the brightness and other optical characteristics obtained after nanofiltration.

Obviously the color of the treated clear filtrate greatly affects its reuse in the mill. However, the color change after the anaerobic stage and prior to nanofiltration should not affect the permeate quality, because color-forming substances are well retained by NF, but it affects the retentate reuse in the mill. So far it has been suggested that the retentate should be either evaporated or dried (25). Thus different kinds of experimental setups should be tried in order to avoid color formation.

CONCLUSION

The anaerobic treatment and its temperature affected the performance of the nanofiltration stage. Even though almost as good results were obtained by only nanofiltering the TMP clear filtrate, it was seen that the changes in the composition of the clear filtrate caused by the anaerobic bacterial treatment enhanced the nanofiltration result.

In general, all the nanofiltration permeates were very clean and should be suitable for recycling purposes. However, further studies on experimental setups together with, e.g., membrane optimization and more detailed analysis are still needed.

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