Protein recovery by selective separation using ceramic membranes

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Abstract

Processing of shrimp shells for the production of chitin makes commercial use of shell waste with economic benefits. Although chitin possesses the valuable properties of a biopolymer, with many useful applications, significant environmental pollution during its production hampers growth opportunities for industrial processing. In this study, a filtration process at different molecular weight cut-offs is assessed for protein recovery from the discharged alkaline processing stream of an industrial chitin manufacturing unit. Three tubular ceramic membranes (0.1 μm, 450 D and <300 D) have been investigated under a constant temperature of 70 °C, at chosen trans-membrane pressures of 1.3–5 bar, high cross flow velocities of 3.3–3.5 m/s, and at a volumetric concentration factor of 5. Results of concentration runs indicate a significant increase of recovered proteins, between 7 and 16%, can be achieved in the concentrate stream by reducing the chosen membrane cut-offs. A second product the permeate stream – solid-free hydroxide sodium solution – can be re-utilized in the chitin production line. Retention of the organic load led to a 56% decrease of chemical oxygen demand and total bound nitrogen in the permeate stream.

Key words: ceramic membrane, chitin, filtration, protein recovery

INTRODUCTION

The world's fish and fishery production is growing steadily with a crucial contribution from aquaculture, which continues to expand at an annual rate of 8.8 percent (FAO 2012). As one of the major seafood exporters worldwide, 55% of Vietnam's total fish production originates from aquaculture activities, of which the key species, Pangasius and shrimp, reached a total amount of 1.2 million and 488,000 tons, respectively, in 2012 (Vietfish International 2014). The growth of aquaculture, and especially of the shrimp industry, provides a stable raw material source for the exploitation of chitin and its derivatives. Approximately 40–50%-wt. of raw shrimp is solid waste (Ngoan *et al.* 2000, Lertsutthiwong *et al.* 2002). Processing of shrimp residues helps countries producing shellfish to dispose of bio-waste in an ecological and economical manner.

For chitin recovery from shrimp waste on an industrial scale, sodium hydroxide and hydrochloric acid are widely employed as reagents (Lertsutthiwong *et al.* 2002) to remove the associated proteins (thermal alkaline extraction) and minerals (acidic extraction) in the crustacean shells. Consequently, organic and nitrogenous compounds originate mostly from the alkaline partial stream that accumulates in the wastewater. Water is utilized intensively during production, for washing and purification, and wastewater with a high organic load is also an unavoidable issue for such processing companies.

Because the potential recovery of useful proteins or sodium hydroxide solution from industrial processes has attracted attention recently, studies on alkaline wastewater from chitin processing have

been performed. Benhabiles *et al.* (2013) reported on the possibility of recovering proteins with ceramic ultrafiltration membranes (0.5 µm) and reviewed the influences of chitin processing conditions on the properties of the alkaline hydrolyzate. Filtrations were run on lab-scale, at 20 °C and pH 7, with synthetic deproteinized shell wastewater. Zhao & Xia (2009) combined a stainless steel ultrafiltration (cut-off 20 kD) and a polysulfone nanofiltration (NF) (cut-off 150 D) for caustic solution recovery. Feed solution was sampled from a local shrimp waste-processing factory. Trials were carried out at a technical scale for the ultrafiltration at 70–80 °C, trans-membrane pressures (TMP) of 3 bar, and for the NF at 50 °C, TMP of 31 bar. Both investigations applied a very large range of tangential velocities 4–6 m/s. Chemical oxygen demand (COD) and protein concentrations were relatively low: less than 7 g/L and 1 g/L, respectively.

With respect to protein recovery, membrane technology has been extensively investigated as a process-integrated unit for the separation and enrichment of production-related streams or liquid substrates from food processing, e.g. in cheese-making (Butylina *et al.*, 2006; Galanakis *et al.* 2014), amaranth starch production (Middlewood & Carson 2012), soy bean flour production (Noordman *et al.* 2003), and in seafood processing industries (Afonso & Borquez 2002, Bourseau *et al.* 2009). Researchers focused not only on membrane performances and recovery capacity but also on operational conditions for preventing fouling, a common obstacle in nutrient-rich liquid filtration. Ceramic membranes have been proposed as a solution and are being increasingly applied because of their thermal and chemical stability with regard to influent properties.

This study reports on a comparison of three tubular ceramic membranes for protein recovery from alkaline extraction lysate, under consideration of local chitin processing conditions. Operating parameters, permeate flow rate, and recovery efficiency in pilot-scale testing conditions were evaluated to propose suitable cut-offs related to increasing the concentration of recovered proteins.

MATERIALS AND METHODS

Applied ceramic membranes and membrane testing plant

The current experiments were performed with multi-channel tubular ceramic membranes: a microfiltration (MF) membrane 0.1 micron (Atech GmbH, Germany) and two NF membranes 450 D, <300 D (Inopor GmbH, Germany) with a filtration surface area of 1.68 m² and 1.75 m², respectively. The cut-offs of membranes were chosen on the basis of previous lab-scale experiments (Nguyen *et al.* 2014). Each membrane tube has a length of 1.2 m and an active layer of alumina (MF) or of zirconia/titania (NF). Table 1 shows the membrane cut-off (manufacturers' information) and measured values of membrane permeabilities under testing condition with softened water: temperature 35 °C, transmembrane pressure 1.5 bar, and cross flow velocity 3 m/s.

Table 1 | Properties of tested membranes

Membrane	Cut-Off	Permeability [L/(m² h bar)]
MF	0.1 µm	300–400
NF 450	450 Dalton	40–60
NF <300	<300 Dalton	20–30

Trials were carried out in a pilot plant (EnviroChemie GmbH, Germany) installed at the chitin production section of a factory in Vietnam (Figure 1). Immediately after each protein removal step, the alkaline processing stream was collected in a feed reservoir with a volume of 2.5 m³. For filtration

experiments, this stream is initially pre-screened into a storage chamber, then pre-filtered with a pore size of 100 μm, and, finally, pumped into a buffer tank (300 L) equipped with an immersed heater. The sieve and the bag filter protect the membranes against blockage caused by coarse particles larger than the pore size of the applied devices.

The membrane testing unit is characterized by a feed-and-bleed system supported by a pressure pump and a recirculation pump located in front of the membrane modules. This configuration is identical for both MF and NF. The membrane module splits the alkaline stream (feed stream) into a permeate stream and a concentrate stream.

Experimental performance

Experimental performances presented in this paper for three ceramic membranes were carried out with the alkaline stream, in a one-step filtration, at a constant temperature of 70 °C. Prior to the experiments, membranes were rinsed with softened water. The pure water permeability was measured at a temperature of 30–35 °C, at a TMP of 1.5 bar (MF) or at different TMPs 1.5–9 bar (NF) and at a cross flow velocity of 3 m/s. After each experiment, chemical cleaning was carried out to recover the original filtration capacity. The pure water permeability was also re-checked before starting a new trial.

The MF and NF membrane can be backwashed at the pressure of 4.5 bar from the permeate side by pneumatic valves with compressed air and the permeate stream left in the manifold pipe (Figure 1). An integrated backwash was applied impulsively at intervals of 15 and 5 min and with a duration of 4 and 2 s during concentrating runs for MF and NF, respectively.

For the determination of optimal operating conditions, the critical flux phenomenon or critical conditions of the process were investigated. Filtrations were run without backwash in a closed loop. Both concentrate and permeate streams were returned to the buffer tank, to keep the feed concentration constant. Table 2 shows the process with various operating adjustments of pressures and cross flow velocities applied for the tested MF and NF membranes. The process was carried out step-wise. The upward flux value was recorded every 5 s during 10 to 15 min in each pressure step, after the operating conditions had been adjusted.
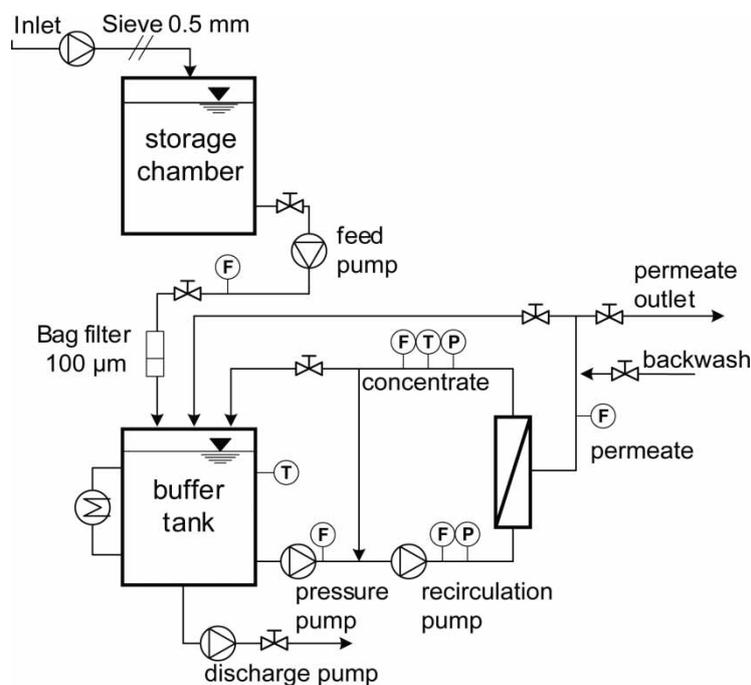

Figure 1 | Layout of investigated filtration process in technical scale (F for flow meter, T for temperature and P for pressure gauge).

Table 2 | Stepwise pressure/velocity adjustments for determination of optimal hydrodynamics

for the MF membrane

TMP [bar]		1.5	2	2.5	3	
v [m/s]	2.5	start	→	→	end	
	3	start	→	→	end	
	3.5	start	→	→	end	
	3.8	start	→	→	end	

for the NF membranes

TMP [bar]		1.5	3	5	7	9
v [m/s]	2.5	start	→	→	→	end
	3	start	→	→	→	end
	3.5	start	→	→	→	end

For the targeted recovery of proteins, filtrations were set in a so-called concentrating mode. This means that the concentrate stream is routed back to the buffer tank, thus generating a concentration increase, while the permeate stream is collected in a storage tank outside the plant. The volumetric permeate recovery (VPR) rate (calculated according to Equation (3)) increases with the running time. In these experiments, the transmembrane pressure was set constantly to 1.5 bar (MF), 3 bar (NF 450), and 5 bar (NF <300). A tangential velocity of 3.5 m/s was applied. To investigate the system stability or membrane performance in a long-term filtration at a constant VPR, the liquid in the buffer tank can be regularly discharged, using the discharge pump during the filtration mode.

Process assessment

In order to differentiate the membrane performance from the system capabilities, two types of retention rates for a component i are calculated: membrane retention and system retention.

$$R_{i,M} = 1 - \frac{c_{Pi,M}}{c_{Bi,M}} \tag{1}$$

$$R_{i,B} = 1 - \frac{c_{Pi}}{c_{Fi,B}} \tag{2}$$

Membrane retention $R_{i,M}$ (Equation (1)) refers to the *nearly real retention rate* at a given point of time during the filtration process and is calculated using the measured permeate concentration of i $c_{Pi,M}$ and the bleed concentration $c_{Bi,M}$ in the recirculation loop of the filtration unit at this moment. *System retention $R_{i,B}$* (Equation (2)) is calculated using the unified permeate concentration c_{Pi} after the end of the filtration and the concentration of the influent at the beginning of the filtration $c_{Fi,B}$.

The VPR rate, a measure of the volumetric reduction of the feed solution, is defined as the ratio between the virtual filtered volume $V_{P,virtual}$ and the total feed volume $V_{F,total}$ (Equation (3)). The virtual filtered volume is the difference between the total permeate volume (measured by the permeate flow meter) and the permeate volume (caused by intermittent backwash) that is still left in the manifold pipeline.

$$VPR = \frac{V_{P,\text{virtual}}}{V_{F,\text{total}}} \tag{3}$$

Sample analysis

COD was measured with a COD online analyzer (LAR Process Analysers AG, Germany), using the combustion method. The measured COD values ($COD_{Analyzer}$) represent the oxygen consumption to combust the organic compounds, including nitrogenous compounds, within the samples. The virtual COD is therefore revised by Equation (4).

$$COD = COD_{Analyzer} - 2*TN_b \tag{4}$$

To assess the nitrogen content, both total bound nitrogen (TN_b) and total Kjeldahl nitrogen (TKN) were determined. TN_b was analyzed according to the Koroleff method (spectrometric method with 2.6-Dimethylphenole after digestion with sodium persulfate) and TKN according to the Kjeldahl method (digestion with H_2SO_4, distillation and titration via Foss 2300 Kjeltec). Because the results for TN_b and TKN are almost identical, values of TN_b are chosen to calculate crude protein (CP) concentration by multiplying with a factor of 6.25 (Ngoan *et al.* 2000).

For process controlling, samples were also additionally checked with Hach Lange Cuvette Tests (Dr. Lange LCK 514, LCK 314 for COD and LCK 238, LCK 338 for TN_b).

Ammonia (NH_4-N) was determined via photo spectroscopy with the indophenol blue method (DIN 38406-5). Sodium hydroxide concentration was measured by titration with HCl 0.5 N. The total concentration of phosphorus (TP) was measured photometrical using cuvette tests LCK 350 (2–20 mg P/L) of Hach Company. Total suspended solids (TSS) and residue on ignition (ROI) were determined according to the standard German method DIN 38409-2 (1987). For TSS analysis, the sample was filtered with a glass fiber filter (Whatman GF/C, undefined pore size) and dried at 105 °C until its mass became constant. The ignition loss of the filtered residues was determined by heating the sample at 550 °C for 60 min in a furnace.

RESULTS AND DISCUSSION

Properties of the alkaline processing stream

The alkaline stream is generated from the chitin production by thermally extracting shrimp residues (>75 °C) with soda hydroxide. This stream is normally treated together with other process flows in the factory's decentralized wastewater treatment plant (Figure 2). Samples were taken directly after the base extraction at the production site, according to an every-three-month monitoring program.

The alkaline stream is characterized by the key parameters given in Figure 3. CP, chemical oxygen demand (COD_h in homogenized sample, COD_f in filtered sample), and suspended solids were much higher than those reported by Zhao & Xia (2009), especially the CP concentration: 4.7% compared to 0.5–1.2% (Zhao & Xia, 2009). A total COD of 113 g/L was found in homogenized samples, in comparison to 5.8 g/L (Zhao & Xia, 2009) and 1.6 g/L (Benhabiles *et al.*, 2013). In contrast, sodium hydroxide after extraction had a concentration of 1%, which possibly indicates a high rate of protein removal. Sodium hydroxide seemed to be completely used up. Around 30% of the COD is apparently associated with solid matter.

Similar values for TN_b and TKN indicate negligibly low concentrations of nitrite, nitrate, and ammonia, and also indicated that a significantly large amount of organic bound nitrogen, e.g. proteins, was present in the sample.

Although basic process parameters, e.g. alkali concentration, temperature, solid-solvent ratio, and extraction time, were reported to have a critical effect on the protein removal step (Benhabiles *et al.*, 2013), not all factors could be taken into account in the large-scale production of chitin. The
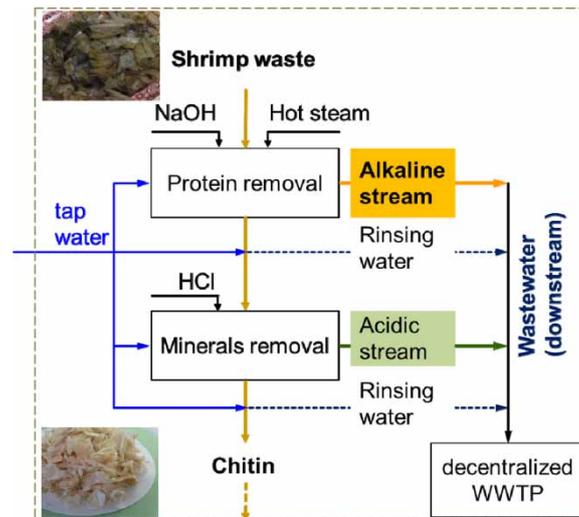
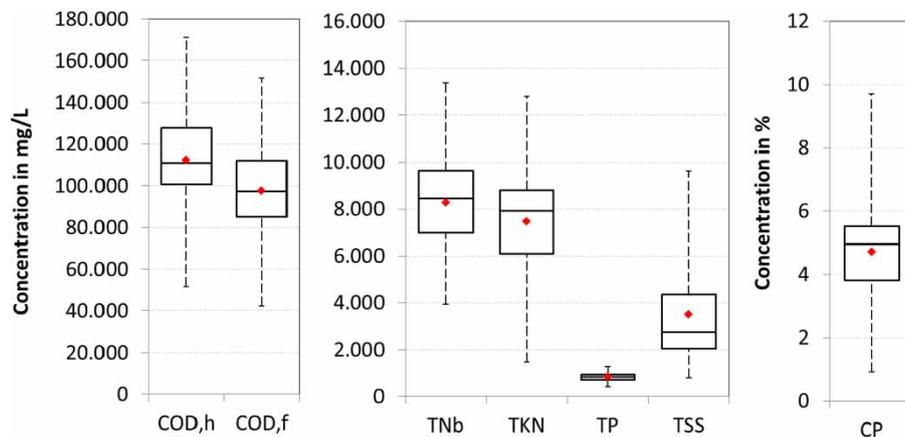**Figure 2** | Basic method for chitin recovery from shrimp waste at the factory site.

Figure 3 | Characteristics of the alkaline processing stream as feed solution in the membrane testing plant (blot graph of $n_{sample} = 88$ shown as values for the lower quantile 25%, the upper quantile 75%; min, max, median and mean).

quality of the shrimp waste, the proportion of heads and shells in one charge, the recirculation rate of alkaline hydrolyzate in the first extraction step, etc., profoundly influence the production process and the quality of the alkaline discharged stream. Therefore, analyses varied considerably from batch to batch. The min – max range with a large variation in the investigated parameters corresponds to the real and varying conditions at the production site.

In relation to the downstream, the segments of the pie chart illustrate the percentage ratio of the alkaline stream in terms of volumetric flow and loads of COD and TN_b (Figure 4). Separating the alkaline partial flow eliminated approx. 39% of the total organic load and 33% of the nitrogen load in the entire wastewater.

Effect of hydrodynamic parameters on membrane performances

A sustainable flux is desirable in the filtration process to reduce the running costs. Field *et al.* (1995) presented a critical flux concept for MF. This concept stated that, *on start-up, there exists a flux below which a decline of flux with time does not occur; above it fouling is observed. This flux is the critical flux and its value depends on the hydrodynamics and probably other variables* (Field *et al.* 1995, p. 267). Apparently, adapting the operating conditions of the system, namely, transmembrane pressure
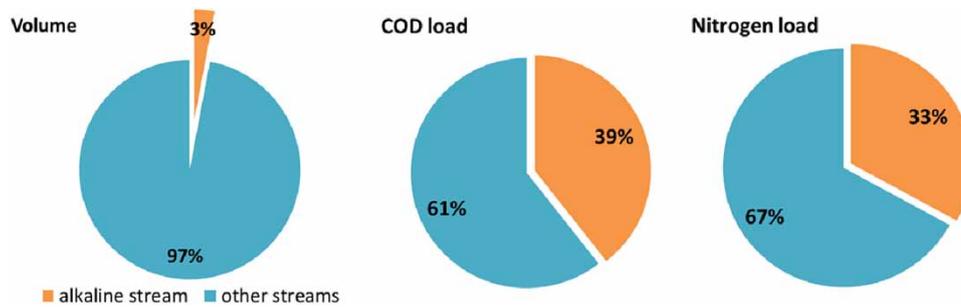
Figure 4 | Percentage share of the alkaline stream in terms of volumetric flow and loads (COD and nitrogen) in the entire wastewater.

and cross flow velocity, is one of several methods to minimize fouling and improve the flux (van den Berg *et al.*, 1989). With regard to the issue of critical flux, extensive studies have been performed on MF and UF (Chen *et al.* 1997; Madaeni *et al.* 1999; Rayess *et al.* 2011) (Chen *et al.*, 1997; Madaeni *et al.*, 1999; Wu *et al.*, 1999; Rayess *et al.*, 2011; Sim *et al.*, 2014), as well as on NF (Mänttäri & Nyström (2000), Ellouze *et al.* 2012), especially in terms of measuring methods for critical flux (Espinasse *et al.* 2002).

Accumulation of suspended particles or soluble molecules, or both, adjacent to the membrane surface limits the liquid flow through the membrane. Protein-rich feed solutions are considered to be a particular challenge for separation. Due to their viscosity, gels may form on the filter's surface. High pressure or low tangential velocity could accelerate cake layer formation on the membrane surface. High tangential velocity enhances the mass transport through the membrane but is associated with increasing energy consumption. Therefore, optimal hydrodynamics have to be assessed.

The critical flux concept was used to investigate the effect of hydrodynamics on membrane performances. The objective of this study was to identify the optimal pressure and optimal cross flow velocity for membranes MF 0.1 μm, NF 450 and NF <300 in order to filtrate the alkaline process stream steadily. The determination of critical flux was carried out by following the pressure-step measuring method of Mänttäri & Nyström (2000) and Ellouze *et al.* (2012).

The variation of permeate flux per pressure-step was measured at different cross flow velocities (Figure 5). The MF started with a flux of 80–90 L/(m^2·h) (TMP = 1.5 bar, T = 70 °C), a value significantly lower than that of the corresponding pure water flux: 825 L/(m^2·h). An almost linear increase of permeate flux was found as a function of trans-membrane pressure at a cross flow velocity of v = 3 and 3.5 m/s (see Figure 5(a)-1). This flux increase does not correspond to rising cross flow velocities of v = 2.5 to 3.0 m/s and v = 3.5 to 3.8 m/s at TMP = 1.5 and 2.0 bar (see Figure 5(a)-2). A cross flow velocity higher than 3.5 m/s was not beneficial for flux increase at all tested transmembrane pressures although a high velocity enhances the mass transfer through the membrane because of the increasing value of the Reynolds and Sherwood numbers (van den Berg *et al.* 1989). The labile operation field in the upper limit of the cross flow pump might be the reason for this velocity – flux performance which conflicts the theory. The highest MF flux of 130 L/(m^2·h) was found at v = 3.5 m/s and TMP = 3 bar.

According to Bacchin *et al.* (2006), the flux performance below the pure water line indicates a very early fouling at the initial stage of the filtration process. At the starting point of this test, the flux-line already deviated from the pure water line and presented itself non-linearly (see Figure 5(b) and 5(c)). This performance is defined as the 'weak form' of critical flux (Field *et al.* 1995) and suits the complexity of treated feed solution properly.

The diagrams 5b and 5c show a similar flux-TMP relationship between the two tested NF membranes. Both exhibited a great flux deviation, especially NF 450, compared to the pure water flux value at the beginning (TMP = 1.5 bar). The permeation of the caustic solution varied from 50 to 158 L/(m^2·h) (NF 450) and from 7 to 89 L/(m^2·h) (NF <300), as a function of TMP. It slopped
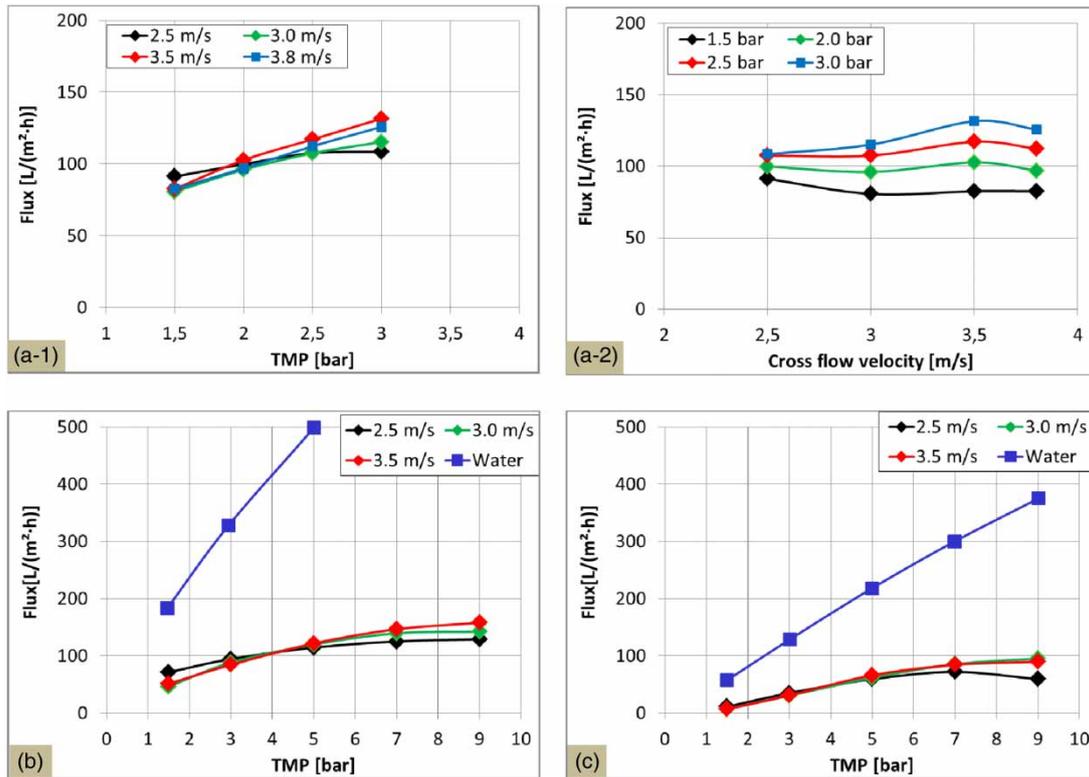
Figure 5 | Variation of permeate flux of MF 0.1 (a), NF 450 (b), and NF <300 (c) as a function of TMP and diverse cross flow velocities, at T = 70 °C.

considerably at a trans-membrane pressure of 5 bar (NF 450) and 7 bar (NF <300), independent of the applied velocities.

In addition, Figure 6 demonstrates that an instable flux region appeared at high transmembrane pressure, e.g. at TMP = 7 and 9 bar for NF <300. A highly turbulent force (v = 3.5 m/s) increased the permeate flow but could not keep the flow constant, in particular for NF <300, from an initial drop at the critical point (TMP = 7 bar). Flux performances among velocities, e.g. v = 2.5 and 3.5 m/s below the mentioned critical TMPs, were also extensively present in this figure. Stable flux
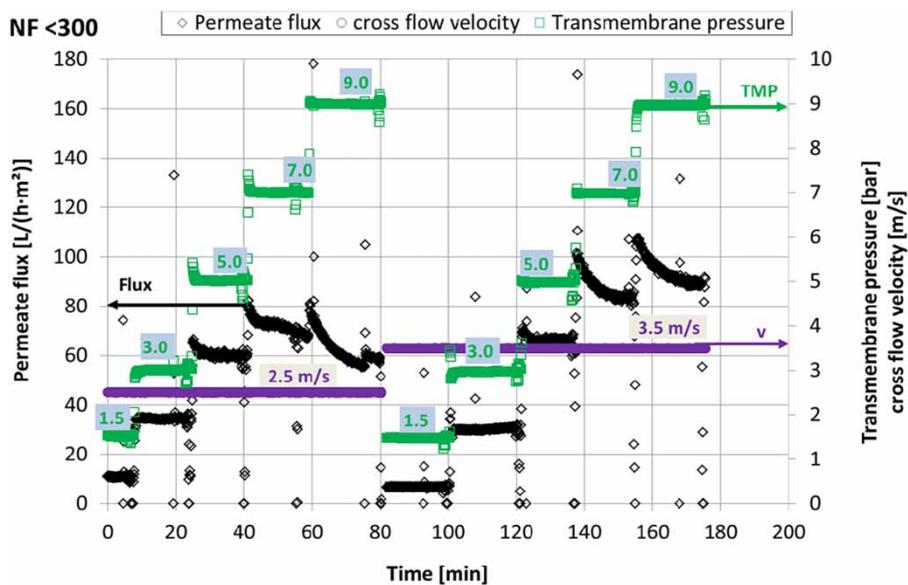

Figure 6 | Instable flux region at a high transmembrane pressure of NF <300.

regions were observed at TMP = 5 bar at both applied velocities. At the same pressure condition (TMP = 5 bar), the permeate flux at v = 3.5 m/s was slightly higher.

Regarding membrane retention, e.g. for total nitrogenous compounds (in short nitrogen), substantial differences between MF and NF are evident in Figure 7(a). It is obvious that denser membranes can retain more nitrogenous compounds than loose membranes. This retention of MF tended to be independent of applied velocities and transmembrane pressures, whereas there was a definite pressure-dependent retention of tested NF membranes. The feed concentration was kept constant during the test by recirculating the permeate and concentrate streams in the buffer tank. For NF <300, the nitrogen retention increased with increasing transmembrane pressure from 66 to 70%, which was, at v = 3.5 m/s, a little higher than those at v = 2.5 m/s (Figure 7(b)). The NF 450 membrane has the same retention performance. It can be concluded that high turbulence and high pressure have a positive effect on the retention of the NF membranes as more water as well as caustic solution is forced to penetrate the membrane.
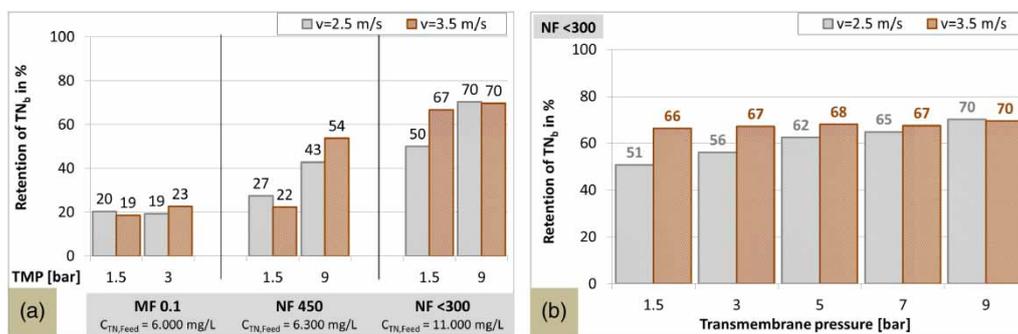

Figure 7 | Nitrogen retention as a function of transmembrane pressures and applied membrane cut-offs.

In terms of hydrodynamic effects, especially on membrane permeation and retention, TMP = 1.5 bar (for MF), TMP = 3 bar (for NF 450), TMP = 5 bar and v = 3.5 m/s (for both) were sustainable conditions and are recommended for further trials.

MEMBRANE PERFORMANCE AND PROTEIN RETENTION

Under sub-critical conditions, the chosen transmembrane pressures of TMP = 1.5 bar (MF), 3 bar (NF 450) and 5 bar (NF <300) and a tangential velocity of v = 3.5 m/s were kept constant during the investigation. Figure 8(a) shows the filtration performances of the tested membranes with regard to permeability. An accumulation of CP in concentrate samples was observed and is presented in Figure 8(b)–8(d) for MF, NF 450, and NF <300, respectively. The concentrating process ended at a VPR of 80%, equivalent to a concentration reduction rate of 5.

Both NF membranes appeared to have a similar trend, with nearly identical initial permeabilities of approx. $J_P = 20$ L/(m²·h·bar), whereas the MF started at a higher value of $J_P = 48$ L/(m²·h·bar). The MF permeate flux sloped continuously with filtration time. The cross flow velocity of 3.5 m/s may have succeeded in reducing cake layer formation but failed to limit the interactions of solid matter and colloids in open membrane channels. The presence of particles, macromolecules, and dissolved substances that cover the membrane surface might induce pore blockage and reduce the liquid flow through the membrane surface. A nearly identical flow performance observed for stainless steel UF 20 kD was reported by Zhao & Xia (2009). Within 15 hours, a VPR of 80% was reached, at which the UF permeability decreased by more than 50% of its initial value.
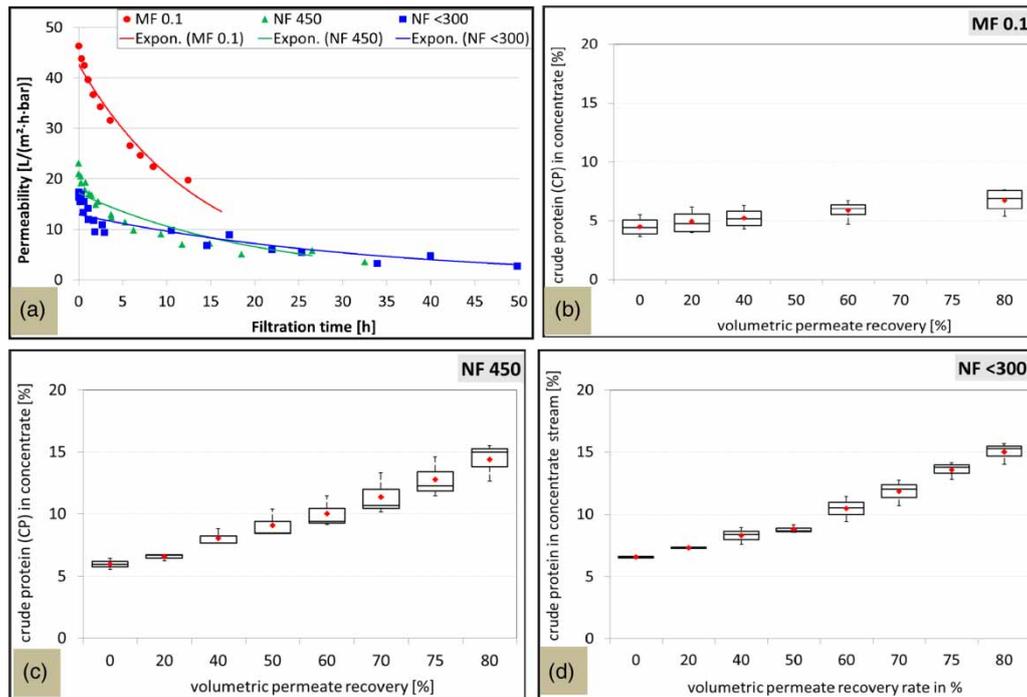
Figure 8 | Membrane performances (a) and CP concentration in concentrate samples taken from 20 trials of MF (b), 4 trials of NF 450 (c), and 4 trials of NF <300 (d) in correlation with VPR.

The flow performance of both NF membranes can be described in two stages. The permeability declined sharply in the first 5 hours of operation, and steadily sloped up to a permeability of $J_P = 4$ L/(m²·h·bar) at the end of the filtration time (Figure 8(a)) or at the VPR rate of VPR = 80% (Figure 9, in the case of NF <300). The permeate flow was found to be steady when the VPR was kept constant, e.g. at VPR = 75% for 10 hours. The NF <300 membrane also demonstrated a stable permeability at VPR = 80% in the long-term test of nearly 40 filtration hours. At the same volumetric permeate reduction ratio, with much lower COD and TN_b, Zhao & Xia (2009) reported a permeability of $J_P = 1$ L/(m²·h·bar) at a temperature of 50 °C and an operational pressure of TMP = 30 bar.
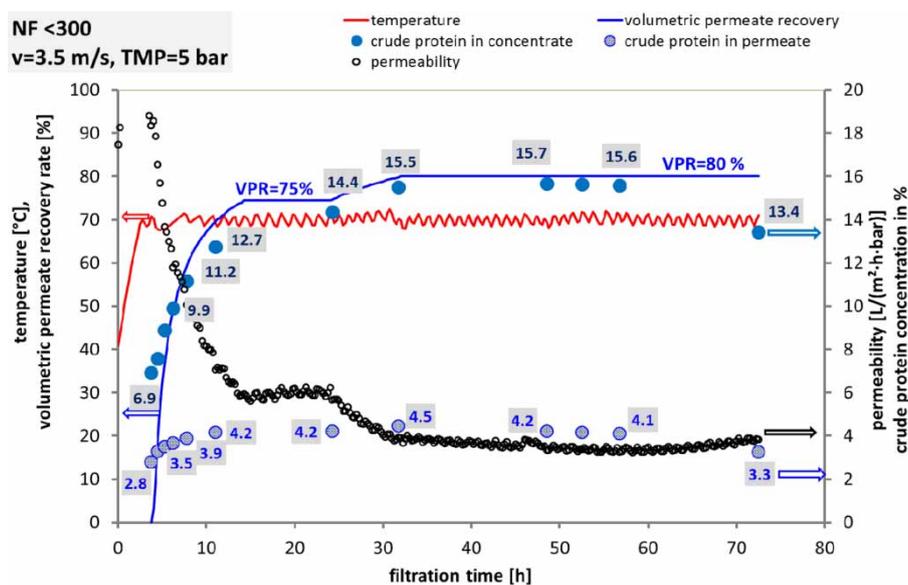

Figure 9 | Performance of NF <300 in a long-term test.

TN$_b$ was analysed in the concentrate stream to assess the protein retention efficiency. With gradual reduction of the concentrate volume, CP accumulated in concentrate stream from an initial value of approx. 4% to 6.7, 13.7, and 15.8%-wt. by single-step filtration with MF 0.1, NF 450, and NF <300, respectively (Figures 8(b)–8(d) and 9). Whereas MF retains only particulate proteinaceous matter, the increase of CP concentration from ca. 60 to 156 g/L was obtained using only NF membranes. Approximately 80% of the feed volume was recovered as a clear, yellowish permeate with 1%-wt. NaOH. The hot caustic solution is suitable for reuse in the production line. Close to 4% of dissolved nitrogen compounds, e.g. peptides, amino acids, and molecules smaller than 300 D still remain in the permeate stream (Figure 9).

Along with protein recovery, COD removal increased with decreasing membrane cut-offs (Figure 10). A maximum membrane retention of nearly 70% COD was obtained by using a cut-off lower than 450 D. According to system retention, approx. 53% of COD in the feed stream was observed, taking the effect of the concentration polarization into account.
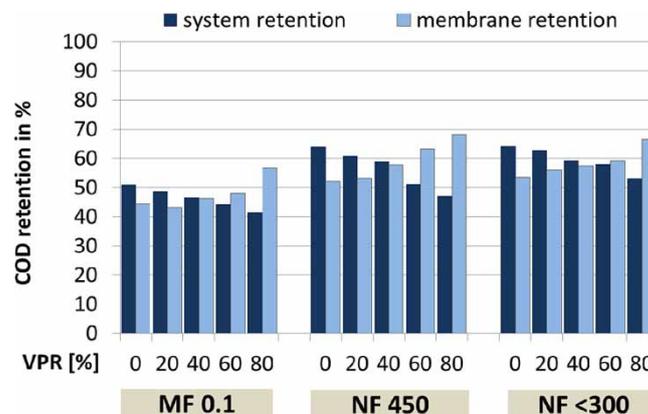

Figure 10 | COD retention as a function of VPR with different membrane cut-offs.

CONCLUSIONS

This paper reports on the potential recovery of proteins and caustic solution from the partial alkaline stream of a chitin manufacturer in a tropical region. Three ceramic multi-channel membranes with different molecular weight cut-offs were investigated on a pilot-scale for optimal operating parameters, filtration performance, and protein recovery efficiency. The process stream was identified to be rich in nitrogen, organic substances, and suspended solids. For a sustainable flux, a trans-membrane pressure lower than 5 or 7 bar was proposed as the cut-off for 450 D and <300 D, respectively. A high tangential flow velocity of 3.5 m/s should be applied. Approximately 6.7–13.7–15.6%-wt. of CP can be accumulated in the concentrate with the corresponding MF 0.1, NF 450 and NF <300 membranes. Protein-rich concentrate is an alternative protein source for animal feeding. Furthermore, reuse of sodium hydroxide solution in the chitin production line helps to reduce chemical and heating costs and limits the discharge load to the connected wastewater treatment plant. This concept addresses a sustainable solution for the chitin industry and contributes to maintaining the eco-friendliness of chitin-based products. Characteristics and reuse possibilities of the concentrate stream – a crucial issue – will be the focus of a subsequent publication.

ACKNOWLEDGEMENTS

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