Title: Pilot-scale microalgae harvesting with ceramic microfiltration modules: Evaluating the effect of operational parameters and membrane configuration on filtration performance and membrane fouling

Running title: TMP, CFV and membrane configuration and pore size effects on permeate flux and membrane fouling

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Abstract

BACKGROUND: Membrane fouling is one of the major drawbacks in microalgae harvesting which prevents the widespread application of membrane filtration. This study investigates the effects of operating parameters on the filtration performance and the fouling phenomenon of a pilot-scale microfiltration (MF) system equipped with different ceramic membranes for *Chlorella vulgaris* culture harvesting.

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RESULTS: A higher permeate flux was obtained with increasing the transmembrane pressure, resulting in a lower irreversible and reversible filtration resistances. On the other hand, at higher crossflow velocity with above 4 m s^{-1} , a high amount extracellular organic matter (EOM) released by microalgal cells led to a higher irreversible resistance which, in turn, caused a severe membrane fouling as well as a lower filtration flux. Furthermore, the membrane configuration and pore size have also significantly affected the permeate flux. Compared to the tubular membranes regardless of pore sizes, the multichannel MF membrane (pore size: $0.2 \text{ }\mu\text{m}$) exhibited higher permeate flux resulted in lower membrane resistances due to the lower release of EOM by microalgae cells. Moreover, the tubular membrane (pore size: $0.8 \text{ }\mu\text{m}$) showed a higher flux reduction with higher pore size because of the increase in the irreversible resistance with a high amount of EOM release.

CONCLUSION: The results revealed that the amount of EOM significantly associated with filtration conditions plays an important role in membrane fouling control. Considering the membrane configurations, the multichannel ceramic MF membrane (pore size: $0.2 \text{ }\mu\text{m}$) demonstrated a higher permeate flux due to its lower fouling property.

Keywords: microalgae harvesting, operational parameters, pilot microfiltration, tubular ceramic membrane, membrane fouling;

NOMENCLATURE

J_{PVF}	Permeate volumetric flux ($\text{L m}^{-2} \text{h}^{-1}$)
Δm	Variation of the mass of permeate collected (kg)
Δt	Variation of the time (h)
ρ_{eau}	Water density (kg m^{-3})
A	filtration surface of membrane (m^2)
J_{APVF}	Average permeate volumetric flux ($\text{L m}^{-2} \text{h}^{-1}$)
VRF	Volumetric reduction factor
V_{si}	Initial feed volume (kg)
V_{f}	Final volume of the retentate (kg)
V_{p}	Total volume of permeate collected (kg)
J_{f}	Water permeability just after microalgae suspension filtration ($\text{m}^3 \text{m}^{-2} \text{s}^{-1}$)

J_r	Water permeability just after rinsing the membrane area with DW ($\text{m}^3 \text{m}^{-2} \text{s}^{-1}$)
J_c	Water permeability after cleaning procedure ($\text{m}^3 \text{m}^{-2} \text{s}^{-1}$)
R_T	Total hydraulic resistance (m^{-1})
R_{ir}	Irreversible resistance (m^{-1})
R_r	Reversible resistance (m^{-1})
η_p	Permeate dynamic viscosity (Pa s)

1. Introduction

In recent years microalgae-based food and nutraceutical supplements have gained a growing interest due to their numerous benefits on human health.¹⁻³ They strengthen the human immune system against age-related diseases (hypertension, diabetes, hematopoiesis, cancer, heart disease, cataract, atherosclerosis) and other many diseases.^{1,2,4} Increased demand in microalgae-based products led to the increasing large-scale cultivation of microalgal biomass.⁵ However, widespread production of microalgae-based products is limited by the lack of an efficient microalgae harvesting technique because of the small size of microalgae cells, low concentration of the cultures, the requirement of high amount water to be processed.⁵⁻⁷ To overcome this challenge, many microalgal harvesting methods have been developed including centrifugation, gravity sedimentation, flotation screening and flocculation.⁸⁻¹⁰ However, most of these conventional techniques are energy-consuming (centrifugation), time-consuming (gravity sedimentation), toxic to microalgal biomass (chemical coagulation/flocculation) as well as require qualified staff and harmful to the surrounding environment.^{8,9,11,12}

Recently, membrane filtration has emerged as a promising alternative technology for microalgae harvesting.^{6,8,11} In this technology, no chemical is required, no heat-sensitive valuable compounds are denatured, the culture medium is purified and reused, investment cost and energy demand are relatively low.¹³⁻¹⁵ In addition, membrane technology is also environmentally friendly and harmless to human beings.¹⁶ These advantages make membrane technology unique for the production of high-value products.

However, a major challenge for microalgae harvesting by membrane process is membrane fouling, which causes a significant flux reduction and energy consumption as well as deterioration in membrane selectivity.^{9,11,12} The microalgae cells and their extracellular organic matter (EOM) including polysaccharides, proteins, lipids, and humic-like substances are formed a cake layer on the membrane surface and block or narrow membrane pores, leading to membrane fouling.^{8,17,18} In addition, EOM released by microalgae cells causes a formation of a thin impermeable gel layer, leading to a significant blocking into the membrane pores.^{8,17} Therefore, fouling control is crucial for membrane harvesting systems to ensure long term process stability. Several innovative techniques have recently been developed to control membrane fouling in the membrane filtration system. These include changing the

hydrophilicity and pore size of the membrane in tilted panel filtration system,¹⁹ changing the wave height and distance of patterned membrane,²⁰ the use of a finned spacer to improve the impact of air bulbs on membrane fouling²¹ and the use of standalone forward osmosis.²² However, these technologies are still not economically feasible due to the high capital investments and operating costs, which limit the scale up of them.

Recent reports have proved that crossflow membrane filtration technique offers a less costly and successful harvesting operation by providing a high rate of dewatering of microalgae biomass thanks to maintaining high shear forces onto the membrane surface.²³ However, the studies on polymer based membranes in crossflow filtration resulted in severe membrane fouling troubles in microalgae harvesting.^{11,24} Compared to polymeric membranes, the use of ceramic membranes in terms of the membrane fouling in microalgae harvesting is more viable due to their advantages including the narrow and well-defined pore size distribution, high surface hydrophilicity and mechanical resistance, thermal and chemical stability and long membrane lifespan.²⁵ Rios et. Al.²⁶ compared two methods, namely, dynamic and crossflow filtration by using ceramic membranes in microalgae harvesting to minimize the membrane fouling. They reported that the dynamic method initially provides higher water flux, but leads to more energy consumption and substantial reduction in permeability over time compared to crossflow filtration.²⁶ In another study, Hua et. Al.²⁷ introduced a crossflow filtration system equipped with electrochemically reactive ceramic membranes for microalgae harvesting. Although their reports show that electrochemical reactions and electrostatic repulsion on a reactive membrane surface provide membrane fouling mitigation, the energy consumption of the proposed system remains a big barrier for its industrial applications.²⁷ In addition, Purnima et al.²⁸ developed a low-cost kaolin based tubular ceramic membrane for crossflow microfiltration of microalgae solution. They stated that the fabricated membranes provide a high microalgal biomass recovery but not a good antifouling performance. This result suggests that the performance of commercial ceramic membranes is better than lab-made ceramic membranes in terms of maintaining the steady-state condition which is important to ensure the sustainability of the harvesting process. Another approach, Jana et al.²⁹ proposed to use a bioflocculant in microalgal harvesting to reduce the biofouling rate of the crossflow ceramic

MF system. However, the use of flocculant increases the operating costs and the disposing of the waste sludge generated from the system is environmentally harmful.³⁰

Despite the wide research on microalgae harvesting with membrane filtration systems, little is known regarding the performance or applicability of a pilot-scale crossflow MF system equipped with ceramic membranes for large scale applications.^{8,27,29,31} Therefore, investigation of the ceramic membranes at the pilot-scale for microalgae biomass harvesting is necessary to highlight some crucial issues such as membrane fouling and to suggest the solutions as well as to provide a reasonable hypothesis from a technical aspect for industrial applications of membrane technology.

The purpose of this study is to evaluate the performance of a pilot-scale crossflow microfiltration system for *Chlorella vulgaris* harvesting by using ceramic membranes with different configurations and pore sizes under different transmembrane pressures (50, 75 and 100 kPa) and crossflow velocities (2, 4 and 6 m s⁻¹). Membrane fouling was investigated in terms of the EOM such as protein and exopolysaccharide produced depending on operational conditions applied. This study provides relevant preliminary information that could be used to the applications of the membrane filtration process on an industrial scale for the harvesting of microalgae biomass.

2. Materiel and methods

2.1. Algal culture

Chlorella vulgaris microalgae collected from Greensea Company (Mèze, France) was used in this study. The culture was grown under axenic conditions in an 84 L photobioreactor as described previously by Bamba et al.³² Briefly, the PBR was continuously illuminated with four external white light blocks equipped with two daylight fluorescent tubes (6500K, T8, F-36 W / 54-765, 2.6 cm wide, 1.2 m long) and placed on the four sides the PBR giving a global photon flux density (PFD) of 336 $\mu\text{mol photon m}^{-2} \text{s}^{-1}$ which was measured using a digital illumination meter (Lutron LX-105, ref. 1308SI, IHM, China). A modified culture medium by Greensea Company was used to cultivate the microalgal culture under continuous aeration with sterile air at 400 L h⁻¹ enriched with 2% (v/v) of CO₂. The medium was kept at 30±1°C using

an immersed aquarium-type temperature-controlled heater (18-34°C, TSRH 3614 type, 25-100, 100 W, Eheim, Germany).

2.2. Determination of Biomass Concentration

Biomass concentration was determined as dry biomass according to Bamba et al.³² Briefly, 50 ml aliquot of algae suspension was sampled from PBR and filtered over a 0.6 µm preheated fiberglass filter and then dried with an infrared heating balance (Kern-Sohn, MRS 120-3, Germany) at 105°C until constant weight. Dry biomass, expressed as g L⁻¹, was calculated by subtracting the final filter weight from the raw filter weight. Analyses were replicated three times for each assay to achieve reproducibility. The results are given as an average of the triplicate value for each experiment. The Kruskal-Wallis analysis of variance (ANOVA) was performed to analyze the significance levels of the obtained results. Filtration experiments have been conducted with 1.04 ± 0.15 g L⁻¹ biomass concentration to compare the performance of the used membranes.

2.3. Determination of EOM content

Soluble exopolysaccharide and protein content were determined in retentate, permeate, and rinse water using the method of Dubois et al. and the Lowry method, respectively.³³ For determination of polysaccharides concentration, absorbance (A) measured at 450 nm (UV-visible Jenway 6705 spectrophotometer, Barloworld, France) against distilled water, was converted to mg L⁻¹ using a standard curve ($[\text{Polysaccharides}] = 0.1069 \times A$; $R^2 = 0.999$) prepared with glucose solutions (Panreac, Barcelona, Spain). For analysis of proteins, absorbance was measured at 750 nm and then converted to mg L⁻¹ using a standard curve ($\text{Proteins} = 0.506 \times A^{1.232}$; $R^2 = 0.998$) prepared with bovine serum albumin (Sigma-Aldrich, Missouri, USA). The EOM content was expressed in mg for 1 L by doing the sum of exopolysaccharides and protein in each mentioned part. The analyses for each sample were repeated three times to ensure reproducibility.

2.4. Properties of membrane used

Two different microfiltration membranes made of α-alumina supplied by Pall Exekia (Bazet, France) were used in the experiments. The membrane T1 consists of four hollow tubes

mounted in parallel in a cartridge, while the membrane P19-60 is equipped with 19 multichannel. All other characteristics related to the membranes were given in Table 1.

2.5. Experimental set-up

Membrane filtration tests were performed in industrial pilot-scale microfiltration equipment in permeate drained mode commonly named as concentration mode. The equipment was a closed-loop system consisted of 60 L feed tank, a feed pump (SolidC 2,2 KW, 3 m³ h⁻¹, 300 kPa, 3 000 tr min⁻¹, Alfa laval, Saint-Priest, France), a recirculation pump (SolidC 2,2 KW, 10 m³ h⁻¹, 220 kPa, 3 000 tr min⁻¹, Alfa laval, Saint-Priest, France), three pressure gauges P1, P2 and P3 connected to feed, retentate and permeate sides of filtration module, respectively to measure the transmembrane pressure (TMP). A flow sensor was used to monitor the crossflow velocity. A heat exchanger and a thermometer placed on the recirculation loop to keep and control the algae suspension temperature constant (≤ 30 °C) (Fig. 1) during the filtration process.

2.6 Microfiltration system operation and filtration tests

Prior to each filtration run, the microfiltration system was operated with deionized water for 5 min. Then, a feed tank was then filled with 50 L of the microalgal suspension and then pumped to the membrane module by using a feed pump. The pressure P1 was slowly set at 100 kPa according to the manufacturer procedure by using a needle valve (V1). Next, the recirculation pump was then turn on to generate a tangential recirculation of algal suspension in the recirculation loop and the crossflow velocity (CFV) was adjusted to the desired velocity using the frequency converter which automatically controls it through the electrical box. The pressure P2 and P3 were set from 100 to 300 kPa by using a needle valve V2 and V3 respectively to keep the transmembrane pressure at the desired level. TMP was defined as $(P1+P2)/2 - P3$.³⁴ The permeate water was collected in a permeate tank placed on an electronic balance and weighted to monitor its flux during the membrane filtration. The permeate volumetric flux J_{PVF} (L m⁻² h⁻¹) was calculated from the recorded mass of the permeate collected according to Equation 1:

$$J_{PVF} = \frac{\Delta m}{\rho_{eau} \times \Delta t \times A} \quad (1)$$

Δm and Δt are the variation of the permeate collected (kg) and time (hour) respectively, ρ_{eau} (kg m⁻³) is water density at 30°C and A (m²) is the filtration surface of the membrane.

In addition, average permeate volumetric flux J_{APVF} (L m⁻² h⁻¹) was calculated by dividing the total volume of permeate collected by the duration of filtration, and the membrane filtration surface area.

The filtration was performed until the volumetric reduction factor (VRF) reached 10 while taking into account the dead volume of the CFM pilot which was 5L. VRF was calculated by Equation 2:

$$VRF = \frac{V_{si}}{V_f} = \frac{V_{si}}{V_{si} - V_p} \quad (2)$$

where V_{si} is the initial feed volume, V_f is the final volume of the retentate and V_p is the total volume of permeate collected.

After each set of experiments, the system was rinsed and the membrane is chemically cleaned according to the manufacturer procedure using sodium hydroxide (12 g L⁻¹) at 70°C for 50 min followed by nitric acid (5 g L⁻¹) at 50°C for 30 min, and transmembrane pressure of 50 kPa until the complete restoration of hydraulic permeability which is controlled by the measurement of the permeate flux with deionized water at 30°C and at TMP of 100 kPa.

To assess the effect of operating conditions and membrane types on membrane fouling performance, nine filtration experiments were carried out under the experimental conditions summarized in Table 2. Each experiment was performed in triplicate.

2.7. Measurement of resistance

Microalgal suspension during membrane harvesting causes various resistances consisted of irreversible resistance (R_{ir}) due to the adsorption of microalgae debris and extracellular organic matters (EOM) on the membrane which might be removed by the cleaning process, reversible resistance (R_r) due to microalgae cells deposition on the membrane, which is removed by just rinsing the membrane with pure water. In addition to these resistances, the membrane showed intrinsic resistance which denotes clean membrane resistance (R_m). Then, total hydraulic

resistance (R_T) referred to the sum of these three resistances. In order to estimate these resistances, permeate flux have been measured with pure water after 10 min at 30°C and TPM of 100 kPa and the resistances were calculated as follows according to Darcy's law:³⁵

$$J_f = \text{TMP} / (\eta_p \times R_T) \quad (3)$$

$$R_T = R_m + R_{ir} + R_r \quad (4)$$

$$R_m = \text{TMP} / (\eta_p \times J_c) \quad (5)$$

$$R_m + R_{ir} = \text{TMP} / (\eta_p \times J_r) \quad (6)$$

$$R_{ir} = \text{TMP} / (\eta_p \times J_r) - R_m \quad (7)$$

$$R_r = \text{TMP} / (\eta_p \times J_f) - R_{ir} - R_m \quad (8)$$

where J_f , J_r and J_c ($\text{m}^3 \text{m}^{-2} \text{s}^{-1}$) denote to water permeability just after microalgae suspension filtration, after rinsing the membrane area with distilled water to remove microalgae cells and after cleaning procedure, and respectively; TMP (Pa) and η_p (Pa s) denotes to permeate dynamic viscosity and R_i (m^{-1}) denotes to different membrane resistances as described above.

3. Results and discussion

3.1 Effect of TMP on permeate flux and filtration resistances

Fig. 2(a) shows the flux behaviors of the tubular membrane (T1) as a function of time at different TMPs. The results show that the initial permeate flux at the beginning of filtration

increased proportionally with increasing TMP. Similar results are reported during the harvesting of different microalgal suspensions.^{11,31,36} This could be due to the fact that the increase in TMP causes an increase of driving force and also higher compaction of the fouling layer, which according to the mass transfer law of Fick results in higher flux.^{31,37}

The permeate flux trend consisted of two phases, the drastically declining phase and the gradually declining phase, which is commonly observed in membrane filtration processes.²⁷ The drastically declining phase extended 1 h from the start of filtration, and then the gradual flux decline occurred until the end of the process. However, the overall flux decline remained proportional to the TMP through the operation. The flux dropped from 79.12 ± 12 to 58.4 ± 15 , from 90.5 ± 10 to 62.5 ± 16 and from 114.3 ± 11 to 69.8 ± 13 L m⁻² h⁻¹ which represented a flux loss of 26.10, 30.55 and 38.95 % for 50, 75 and 100 kPa respectively. Indeed, the flux decline was due to the deposition of microalgae cells and EOMs released by the microalgae cells which clogged the membrane pores during the filtration process.^{11,27}

The permeate flux decline may originate from the resistances of several factors which were calculated in section 2.6. In principle, a high TMP leads to a high permeate flux while a high resistance results in low permeate flux. Fig. 2(b) shows the local fouling resistances under different TMPs during microalgae harvesting with the tubular membrane (T1) with 0.2 μm pore size. As shown in Fig. 2(b), it can be observed that all the filtration resistances showed a decreasing trend with an increase of TMP, except irreversible resistance (R_{ir}) at 50 and 75 kPa where R_{ir} were slightly different in magnitude respectively $6.0, 10^{11} \pm 600$ and $5.6, 10^{11} \pm 400$ m⁻¹. Moreover, regardless of TMP, the reversible resistance (R_r) due to cells deposition was higher than irreversible resistance (R_{ir}). The results of the study reported by Wu et al.⁸, performed with *Chlorella vulgaris* using submerged flat-sheet ceramic membrane at a pilot-scale are consistent with those observed in this study. In contrast, Jana et al.²⁹ reported a significant contribution of irreversible resistance to membrane fouling in *Arthorspira platensis* harvesting process using clay-alumina ceramic tubular membrane. This difference would be mainly related to the size, shape and structure of the microalgae used in our study. Indeed, *A. platensis* is a prokaryotic cell, has a filamentous form that breaks easy, whereas *C. vulgaris* is rather eukaryotic of small size and spherical, thus breaks difficult. In addition, Ahmad et al.¹¹ reported that the high rate of R_c could be attributed to the thickness of the fouling layer on the

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membrane surface and the size of *Chlorella* cells is higher than the membrane pore size, leading to the accumulation of cells on the membrane surface. Moreover, the decrease in individual resistances resulted in a negative correlation between the total resistance (R_T) and TMP while the permeate flux increased. The decrease in the filtration resistance when TMP increases could be due to the compressive force that it exerts on the fouling layer, thus reducing the fouling thickness while forcing permeate transfer. In addition, the increase in permeate flux while TMP increase showed that fouling occurs in deeper location of the membrane and mainly at the beginning of the filtration process.¹¹ Other results reported in the literature have indicated an increase in total resistance when increasing TMP.^{10,29} This phenomenon is attributed to the high compression of microalgae cells and EOM accumulated on the membrane surface, resulting in a thicker and dense fouling layer.^{10,29} However, the reported TMPs (> 180 kPa) are higher than those reported in our study. Furthermore, this observation suggests that the permeate flux decline at the pilot-scale is mainly due to reversible resistances increase. This observation is confirmed by the non-change in magnitude of intrinsic resistance ($R_m = 6.710^{10} \pm 500 \text{ m}^{-1}$) suggesting that membrane fouling can be easily cleaned by simple water rinsing after the harvesting process, which is seen a valid output to scale-up of this system.

3.2 Effect of CFV on microfiltration permeate flux and membrane fouling

Fig. 3(a) shows permeate flux behaviors of the tubular membrane (T1) during *Chlorella vulgaris* suspension harvesting by the MF process under various CFVs (2, 4 and 6 m s^{-1}). It has been observed that all the permeate fluxes declined rapidly within the initial 1 h of the filtration process, and then stabilized. The steady-state fluxes were 41.4 ± 2 , 69.9 ± 3 and $63.0 \pm 5 \text{ L m}^{-2} \text{ h}^{-1}$ for 2, 4 and 6 m s^{-1} , respectively. The enhancement in flux when increasing CFV is due to high turbulence creating by high CFV, which sweep the deposited biomass particles away from the membrane surface, thereby decreasing the fouling and enhancing the permeate flux.^{29,36}

However, when CFV was above 4 m s^{-1} permeate flux decreased which represented a flux loss of about 8%. The observed result demonstrates that microalgae concentration in the feed solution may play a limiting role on the permeate flux due to the exposure of the membrane surface to more microalgae content under increased CFV conditions.

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In order to elucidate the flux decline mechanisms depending on CFVs during microalgae harvesting, filtration resistances were given in Fig. 3(b). As shown in Fig. 3(b), all the resistances decreased significantly when CFV increased from 2 to 4 m s⁻¹, and then the resistances slightly decreased, except at 6 m s⁻¹ where irreversible resistance (R_{ir}) drastically increased from $9.0, 10^{10} \pm 400$ to $1.4, 10^{12} \pm 600$ m⁻¹, resulting in higher total resistance ($1.8, 10^{12} \pm 1000$ m⁻¹) compared to 4 m s⁻¹ ($1.5, 10^{12} \pm 500$ m⁻¹). This suggests that higher CFV until a certain level (4 m s⁻¹) is suitable for microalgae harvesting. Nevertheless, no change in intrinsic resistance was observed during microalgae harvesting. These results are consistent with those reported by Ahmad et al.¹¹ who found a decrease in resistances when increasing CFV from 0.13 to 4.0 m s⁻¹. Previous researches reported that a decrease in filtration resistances could be due to the decrease in mass and thickness of the foulant layer on the membrane surface, resulting from the strong shear force at higher CFV.^{9,11} In our study, the permeate flux decline could result in an increase in irreversible resistances which were generally related to the extracellular organic matters including proteins, polysaccharides and lipids released by microalgal cells during the crossflow filtration.^{9,11,38}

Table 3 shows EOM (protein + exopolysaccharides) content in the retentate, permeate and rinse water sides after filtration processes. It can be observed that the amount of EOM in the retentate significantly increases with the increase in CFV. However, no significant difference was observed in the permeate side. This suggests that higher CFV leads to higher microalgae cells broken. As a result, higher cell debris and EOM released in the suspension caused severe membrane pores blockage.^{12,38} Higher CFV of 6 m s⁻¹ caused higher shear stress on microalgae cells during their passage through the valves, pumps and the module, leading to higher EOM release. The EOM content in the rinse water at 6 m s⁻¹ CFV is lower than those of 4 m s⁻¹, this suggests that a great part of EOM released at 6 m s⁻¹ CFV remains adhered on the membrane surface, leading to the higher irreversible resistances at 6 m s⁻¹. Also, the highest amount of EOM in rinse water at 2 m s⁻¹ CFV would justify its higher reversible resistances. These results, which support the findings given in Fig. 3, can explain why crossflow velocity would be limited to 4.0 m s⁻¹ for microalgae filtration in the literature.³⁹

3.3 Effect of membrane configuration and pore size on permeate flux and membrane fouling

Fig. 4(a) shows the effect of pore size and membrane configuration on average permeate flux when using the P19-60 multichannel membrane (pore size: 0.2 μm) and T1 tubular membranes (pore sizes: 0.2 μm and 0.8 μm). When comparing the two configuration membrane with the same pore size, the average flux results obtained for each membrane revealed that the P19-60 membrane showed better performance compared to the T1 membranes. The average flux was 501.4 ± 25.0 and $103.6 \pm 13.2 \text{ L m}^{-2} \text{ h}^{-1}$ for the P19-60 and T1-0.2 μm membranes, respectively, which represents an average flux decline of 79.3%. Previous studies carried out with ceramic tubular membranes showed a greater permeate flux trend.^{29,40} With flashback tubular ceramic membrane (pore size: 1.2 μm), Jana et al.²⁹ observed a steady-state flux of $230 \text{ L m}^{-2} \text{ h}^{-1}$ during UF of *Arthrospira sp.* suspension with constant feed concentration of 0.99 g L^{-1} at 201 kPa and CFV of 6 L min^{-1} . This value is higher than that of the T1-0.2 μm membrane and about 2-fold lower than the value reported to the P19-60 membrane in spite of the higher size of the microalgae. However, Nędzarek et al.⁴⁰ reported the greatest steady-state permeate flux ($161280 \text{ L m}^{-2} \text{ h}^{-1}$) in the literature after 3 h filtration of *Monoraphidium contortum* at 100 kPa and CFV of 4 m s^{-1} using P2336 ceramic membrane with 1.4 μm pore size. The authors showed this high obtained flux could result from the elongated-shaped morphological structure of *M. contortum* making it easier to pass from a higher cut-off membrane (pore size: 1.4 μm) compared to those of oval-shaped such as *Chlorella* algae.⁴⁰ It can be concluded that algae morphology must be taking into consideration when choosing the membrane characteristic. Compared to recently published studies on membrane filtration (Table 4), the P19-60 membrane showed satisfactory performance in terms of permeate flux.

Fig. 4(a) shows the permeate flux values of the T1 membranes having different pore sizes. It can be seen that the smaller pores size (0.2 μm) showed a higher average flux than 0.8 μm one (flux difference: 30.7%). Similar results were observed by Elcik et al.⁹, Qu et al.⁴² and Hwang et al.⁴³, indicated that higher membrane pores size exhibited a faster decrease in permeate flux because of its vulnerability to blockage and constriction. However, the opposite result that is the larger pore size leads to the increase in permeate flux using a flat membrane immersed horizontally in the suspension is reported.⁴⁴ According to Lau et al.¹⁹, the impact of pore size on permeation flux is strongly membrane configuration dependent. They have shown

that the switching configuration is desirable for tilted panel membranes with small pores while the without switching mode would be good for those with large pores.

It can be seen in Fig. 4(b) that P19-60 showed the smallest total resistance (R_T) compared to the T1 membranes. The total resistances were $5.2, 10^{11} \pm 1050$, $1.8, 10^{12} \pm 800$ and $2.1, 10^{12} \pm 500 \text{ m}^{-1}$ for P19-60, T1-0.2 μm and T1-0.8 μm membranes, respectively. The same trend of total resistances with ceramic UF membranes with various pore sizes were reported with *Arthrospira platensis* ($5.0 \times 10^{12} \text{ m}^{-1}$) at CFV 3.0 m s^{-1} and TMP 100 kPa⁴⁵, and with *Scenedesmus diphormis* (4.0×10^{12} and $9.0 \times 10^{11} \text{ m}^{-1}$) when applying DC currents.²⁷ They showed a higher contribution of R_r than that of irreversible one to R_T . In contrast, Ahmad et al.¹¹ and Nedzarek et al.⁴⁰ reported a much lower R_T (4.8×10^9 and $2.2 \times 10^9 \text{ m}^{-1}$) at CFV 4.0 m s^{-1} with *Chlorella sp* (TMP 150 kPa) and *Monoraphidium contortum* (TMP 100 kPa) using polymeric membrane (pore size: 1.2 μm) and ceramic membrane (pore size: 1.4 μm), respectively. Hua et al.²⁷ indicated flux resistance depends on filtered liquid properties, temperature and composition, and operating parameters such as velocity and transmembrane pressures which influence the cake layer structure and compression state.

In addition, higher flux is correlated with lower filtration resistance due to its lower fouling tendency. This difference observed between the P19-60 and the T1-0.2 μm membrane fluxes could be due to the difference in the module configuration of these membranes. Indeed, the P19-60 has a small inner channel diameter (6 mm) compared to those of T1 which are larger about two times (15 mm) leading to Reynolds number of 22459.6 and 56149.1, respectively. Based on these Reynolds numbers, at the same CFV, internal hydrodynamic conditions change considerably due to the parameters such as hydraulic diameter and filtration surface. Reynolds number in the P19-60 channel was about two times slower than the T1-0.2 μm membrane taking into consideration the total number of channels of each membrane. Thus, microalgal foulants were greatly swept on the membrane surface leading to higher permeate flux. This is supported by the data reported in Table 5 where the P19-60 showed a higher amount of EOM in the retentate despite its small irreversible resistant and higher permeate flux. Moreover, the irreversible resistance of the P19-60 membrane is, however, 2 times lower than the reversible resistance. Contradicting result reported by Zhang et al.⁴⁶ showed 5 times higher reversible

resistance than irreversible resistance during microfiltration of *Microcystis aeruginosa* solution with a 7-channel tubular ceramic membrane of 0.1 μm of pores at dead-end mode.

Considering the T1 membranes, the T1-0.2 μm membrane showed higher reversible resistance than the T1-0.8 μm membrane, while for irreversible resistance, an opposite observation was found. On the other hand, Elcik et al.⁹ reported that reversible fouling is not related to membrane pore size for microalgae harvesting. This suggests that the filtration resistance would be related to membrane material and filtration system scale. Moreover, the total resistance of the T1-0.2 μm membrane is lower than the T1-0.8 μm membrane. This indicated that the lower flux obtained with the T1-0.8 μm membranes is due to their resistances (R_T , R_{ir} and R_r). In addition, lower permeate flux obtained with the T1-0.8 μm membrane could be due to its higher irreversible resistance, resulting from the clogging of membrane pore by EOM.

As shown in Table 5, filtration experiments with the P19-60 membrane resulted in a lower amount of EOM in the retentate and rinse water sides than those obtained with the T1 membranes whereas similar concentrations were obtained in the permeate side for both membrane types. This may be due to the short filtration time. Compared to the T1-0.2 μm membrane, while despite the same amount of EOM in the retentate, a significantly low amount of EOM in rinse water was obtained with the T1-0.8 μm membrane. This result revealed that under TMP of 75 kPa and CFV of 3 m s^{-1} more EOM deposited on the membrane surface, leading to its higher irreversible resistance. In addition, higher pore size leads to the higher accumulation of microalgae cells debris and released EOM into the membrane pores, resulting in a severe reduction of the permeate flux. The experimental results showed that the increasing the membrane pore size from 0.2 to 0.8 μm causes the membrane pores more vulnerable to blocking (shrinking pores), resulting in higher resistance to permeate transfer and eventually leading to lower permeate flux.

Conclusion

The influence of operational parameters and membrane characteristics which are very crucial to membrane fouling control during microalgae harvesting was investigated by using a pilot-scale MF system in this study. Permeate fluxes and filtration resistances known as a good

indicator to evaluate filtration performance were successfully monitored through the study. The following key findings were obtained from this study.

1. Cake layer formation on the membrane surface caused reversible resistance whereas blocking of the pores due to the EOM led to irreversible resistance.
2. The filtration resistances except for irreversible resistance were lower when applied higher TMP due to the increased compressive force on the membrane surface resulting in a higher permeate flux.
3. High shear force observed on the membrane surface above 4 m s^{-1} CFV caused a higher irreversible resistance, leading to a higher permeate flux reduction. In addition, this effect at higher CFV led to a higher amount of EOM release by microalgae cells in the retentate during filtration.
4. Compared to T1 tubular membranes, the P19-60 multichannel membrane (pore size: $0.2 \mu\text{m}$) exhibited better performance at the moderate operating conditions (TMP: 75 kPa and CFV: 3 m s^{-1}) with regards to permeate productivity due to its low fouling tendency.
5. For the tubular membranes (T1), the loose membrane, which has larger pores ($0.8 \mu\text{m}$), was exposed to a higher amount of EOM deposition into the pores, resulting in a high rate of irreversible resistance.

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Author contributions

Bio Sigui Bruno Bamba and Yves Lozano conceived and defined the problem; Bio Sigui Bruno Bamba developed the methodology and case studies; Bio Sigui Bruno Bamba implemented the model and run test cases; All authors analyzed the results; Bio Sigui Bruno Bamba wrote the

paper; Allassane Ouattara and Paul Lozano reviewed and contributed in discussions and conclusions; Harun Elcik did writing, review and editing the paper.

Conflicts of interest

The authors declare no conflict of interest.

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Table caption**Table 1.** Intrinsic characteristics of α -alumina ceramic membranes used in this study

Membrane configuration	Length (mm)	Hydraulic diameter (mm)	Thickness (mm)	Tube number per cartridge	Channel number per tube	Total Filtration surface (m ²)
T1	745	15	1	4	1	0.14
P19-60	800	6	-	1	19	0.304

Table 2: Experimental operating conditions for each pilot-scale MF process

Filtration trial	Membrane	Pore size (μm)	TMP (kPa)	CFV (m s^{-1})
F1	T1	0.20	50	5.00
F2	T1	0.20	75	5.00
F3	T1	0.20	100	5.00
F4	T1	0.20	100	2.00
F5	T1	0.20	100	4.00
F6	T1	0.20	100	6.00
F7	T1	0.20	75	3.00
F8	T1	0.80	75	3.00
F9	P19-60	0.20	75	3.00

Table 3. Flux characteristics and EOM amount per liter in the retentate, permeate and rinse water as influenced by crossflow velocity (VRF = 10, feed concentration = $1.04 \pm 0.15 \text{ g L}^{-1}$, TMP = 100 kPa, T1 membrane, pore size = $0.2 \text{ }\mu\text{m}$), average of three replicate $\pm$ standard deviations, different superscript letter on the lines mean significant difference ($p < 0.05$)

Permeate flux ($\text{L m}^{-2} \text{ h}^{-1}$), filtration duration (min) and EOM (mg)	Crossflow velocity (m s^{-1})		
	2	4	6
Steady-state permeate flux	41.4 ± 2^a	69.9 ± 3^b	64.3 ± 1^c
Protein + EPS in retentate	145.8 ± 0.3^a	190.6 ± 0.7^b	269.4 ± 0.6^c
Protein + EPS in permeate	10.1 ± 0.7^a	10.8 ± 0.6^a	9.7 ± 1.0^a
Protein + EPS in rinse water	41.4 ± 1.4^a	21.05 ± 1.2^b	39.1 ± 0.8^a

Table 4. Ceramic hydrophilic membrane configuration performance comparison with membrane filtration recent reports

Membrane configuration	Microalgae concentration and species in Feed (feed volume)	Filtration conditions	Membrane material with pore size	Permeate flux (L h ⁻¹ m ⁻²)	References
19-channel tubular 4 parallel tubular	1.04 g L ⁻¹ of <i>Chlorella vulgaris</i> (60 L)	CFMF concentration mode at 3 m s ⁻¹ and 75 kPa	Alumina with 0.2 µm	500 103.6	This study
Tubular	0.99 g L ⁻¹ of <i>Arthrospira sp.</i> (50 L)	MF flashback (5 min) recirculation mode at 600 L h ⁻¹ and 201 kPa for 1.17 h	kaolinite clay and alumina with 1 µm	200 - 250	29
4 submerged aerated Tilted panel	0.6 g L ⁻¹ of <i>Euglena sp.</i> (5 L)	MF concentration mode at 100 kPa for 1.67 h	PVDF + 3% PEG with 0.42 µm	72	19
4-flat-sheet in series	0.68 g L ⁻¹ of <i>Chlorella pyrenoidosa</i> (n.o L)	CFMF recirculation mode at 3.86 m s ⁻¹ and 130 kPa for 4.5h	Fluoro polymer with 0.2 µm	80	10
Electrochemical reactive monolithic Tubular	0.05 g L ⁻¹ of <i>Scenedesmus diphormis</i> (n.o L)	MF at 68.94 kPa for 40 min with DC currents of +2.5 or -2.5 mA·cm ⁻² ,	TiO ₂ with	198	27
Hollow fiber	0.6 g L ⁻¹ of <i>Chlorella pyrenoidosa</i> (55 L)	CF uniform shearing vibration in recirculation mode at 9 kPa for 48 h	PVDF with 0.05 µm	22	41
2 sided aerated sheet	1.1 g L ⁻¹ of <i>Chlorella vulgaris</i> (0.6 L)	One pass standalone Forward osmosis at 0.02 m s ⁻¹ for 1h	12.3 % of nylon 6,6 with 0.18 µm	17-20	22
Aerated finned projected spacer	1.1 g L ⁻¹ of <i>Chlorella vulgaris</i> (n.o L)	Submerged MF at 10 kPa	PVDF and 0.16 µm	718 - 870	21
23-channel tubular	g L ⁻¹ of <i>Monoraphidium contortum</i> (200 L)	CFMF recirculation mode at 4 m s ⁻¹ and 100 kPa for 3 h	Al ₂ O ₃ /TiO ₂ /ZrO ₂ with 1.4 µm	161280	40
Patterned membrane	0.78 g L ⁻¹ of <i>Desmodesmus sp.</i> (L)	CFMF at 0.015 m s ⁻¹ and 100 kPa for 4 h	Polysulfone + 7% PEG with	492	20
6-hollow fiber in series	1 g L ⁻¹ of <i>Scenedesmus acuminatus</i> (n.o L)	Backwashing CFUF at 0.9 m s ⁻¹ and 51 kPa for 2h	PVC with 50 kDa	94.4	6

MF = microfiltration, CFMF = Crossflow microfiltration, PVDF = Polyvinile fluoride, PEG = Polyethylene glycol

Table 5. Flux characteristics and EOM amount per liter in the retentate, permeate and rinse water as influenced by membrane configuration and pore size (VRF = 10, feed concentration = $1.04 \pm 0.15 \text{ g L}^{-1}$, TMP = 75 kPa, CFV = 3 m s^{-1}), average of three replicate $\pm$ standard deviations, different superscript letter on the lines mean significant difference ($p < 0.05$)

Permeate flux ($\text{L m}^{-2} \text{ h}^{-1}$), filtration duration (min) and EOM (mg)	Membrane configuration and pore size		
	P19-60	T1	
	0.2 μm	0.2 μm	0.8 μm
Protein + EPS in retentate	65.03 ± 0.6^a	168.6 ± 1.1^b	167.14 ± 0.8^b
Protein + EPS in permeate	11.75 ± 1.2^a	10.6 ± 0.6^a	14.49 ± 0.6^b
Protein + EPS in rinse water	7.41 ± 2.3^a	29.04 ± 1.1^b	15.38 ± 1.1^c

Figure list

Figure 1. Schematic of the crossflow membrane filtration unit

Figure 2. Influence of transmembrane pressure on microfiltration permeate flux (A) and filtration resistance (B) (VRF = 10, feed concentration = $1.04 \pm 0.15 \text{ g L}^{-1}$, CFV = 5 m s^{-1} , T1 Membrane, pore size = $0.2 \text{ }\mu\text{m}$, error bars).

Figure 3. Effect of crossflow velocity on permeate flux (A) and membrane resistance (B) (VRF = 10, feed concentration = $1.04 \pm 0.15 \text{ g L}^{-1}$, TMP = 100 kPa, T1 membrane, pore size = $0.2 \text{ }\mu\text{m}$, error bars).

Figure 4. Influence of membrane configuration and pore size on permeate flux (A) and membrane resistance during microalgae harvesting (B) (VRF = 10, feed concentration = $1.04 \pm 0.15 \text{ g L}^{-1}$, TMP = 75 kPa, CFV = 3 m s^{-1} , error bars).

Figure 1

Figure 2

Figure 3

Figure 4