The relation of ultrafiltration membrane fouling caused by algae to algal growth phase

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ABSTRACT

In water treatment industry, ultrafiltration (UF) membrane technology is receiving more and more attention in treatment of algae laden eutrophic source waters. However, fouling of the membrane due to accumulation of algal organic matter on the membrane surface or in membrane pores is the main obstacle to the smooth operation of membrane filtration plants. This drawback is mainly due to the presence of the algae-rich and the algae-derived substances which are well known as algae organic matter (AOM). Membrane fouling behavior can varry as a result of different amount of AOM being produced by algae at different phases (lag phase, exponential phase, stationary phase, and death phase). A series of comprehensive membrane filtration experiments were performed in order to compare the two algae cell conditions, “intact” (mostly present in exponential phase) and “lysed” (mostly present in death phase), in terms of membrane fouling potential and membrane cleaning. Three types of marine algae were used as model water for filtration process: *Chlorella Sorokiniana (CS*), *Arthrospira Platensis* (AP), and *Thalassiosira Rotula* (TR). The results showed that the differences in size and shape of algae in “intact” condition resulting in the different filterability by the membrane. Additionally, moderate and severe fouling was observed in *TR* and *CS* in “lysed” condition compared to “intact” condition. In contrast, in case of *AP,* ”lysed” condition was slightly better in term of filterability. A chemical cleaning procedure close to usual chemical enhanced backwash was applied with which about 85 to 90% of the initial permeability could be recovered for all types of algae in both “intact” and “lysed” condition.

Keywords

Ultrafiltration (UF), Capillary Membranes, Algae Fouling, Algal Organic Matter, Membrane Cleaning, Algae Lysis

1. Introduction

Algae are globally abundant in surface waters like reservoirs, lakes, and seas. These algae-rich waters can cause challenges in water treatment. Depending on the algae characteristic and concentration, these algae-rich waters sometimes have undesirable effects on water treatment procedures especially during algal blooms (Henderson et al., 2008). Membrane filtration technology has been applied extensively as one of the procedures for the algae-rich water treatment. Microfiltration (MF) and ultrafiltration (UF) have been proven as effective technologies to retain all algae cells (Chow et al., 1997; Liu et al., 2017). However, cake formation and severe membrane fouling occur during the operation. This drawback is mainly due to the accumulation of the algae-rich and the algae-derived substances which have been well known as algae organic matter (AOM) on membrane surface (Babel & Takizawa, 2010; Loreen O. Villacorte et al., 2015). AOM can be categorized into extracellular organic matter (EOM) and intracellular organic matter (IOM). EOM is produced during algal metabolism and excretion and IOM is produced when algae cells die and subsequently lyse (Fang et al., 2010; Li et al., 2012; Paralkar & Edzwald, 1996; Thurman, 1985). AOM is able to fill void spaces in cake layers and aggravate the flux decline during filtration (Babel et al., 2002; Ladner et al., 2010).

Algal growth conditions including lag phase, exponential phase, stationary phase, and death phase are present simultaneously in surface water. As a consequence membrane fouling propensity could varry due to different amount of AOM being produced by algae at different phases. Regarding to this problem, in the study of Merle et al. (2016), UF filtration has been performed using algal cultures at death and exponential phases to evaluate the impact of algae phases toward membrane fouling behavior. They revealed that the algae at death phase have resulted in more severe flux reduction and irreversible fouling during filtration compared to the exponential phase.

Several studies have been conducted for algae-rich water treatment using UF membranes (Huang et al., 2015; Liu et al., 2017; Merle et al., 2016); nevertheless, until now, a detailed investigation of the correlation between the characteristics (class of algae, morphology, inhabitation, and growth phase) of the algae and fouling behavior and also solutions for fouling reduction and membrane cleaning is lacking. Moreover, so far, there are limited studies in simulating several filtration cycles in between two chemical assisted cleaning steps as found in real membrane filtration applications. Therefore, this research work is focused on a sequence of several filtration cycles with a comprehensive comparison regarding to membrane fouling potential and hydraulic backwash-ability with three different types of algae. These algaes can be categorized into two cell conditions, which denoted as “intact” (mostly present in exponential phase) and “lysed” (mostly present in death phase). Furhter on, the permeability reversibility using different chemical cleaning steps was addressed.

2. Materials and methods

2.1. Feed water

Three species of marine algae in both “intact” and “lysed” conditions were used as model water for filtration experiments: *Chlorella Sorokiniana* (Chlorophytes), a spherical form, and *Arthrospira Platensis* (Cyanobacteria), a filamentous helical shape, both as fresh water algae, and *Thalassiosira Rotula* (Diatoms), a cylinderical shape as sea water algae. *Chlorella sorokiniana* was cultivated in BG-11 media (Stainer et al., 1971) and exposed to a 16 h: 8 h light and dark photoperiod. Aeration was provided to increase oxygen concentration and buffering process. *Arthrospira Plantesis* and *Thalassiosira Rotula* was provided by Bluebiotech GmbH and was cultured in BG-11 medium and F-medium (Guillard & Ryther, 1962), respectively.

The disruption of algae cells was conducted using an ultra sound device (UIP 250, Hielscher, Germany) with 24 kHz and 250 Watt for 2 hours. During the process, the temperature was controlled between 15 - 20°C; to prevent the possible protein degradation caused by high temperature.

The feed suspension for experiments with “intact” and “lysed” cells was prepared by dilution of stock solution of fresh and disrupted algae in reverse osmosis (RO) water (Merlin Pentair Water, USA) with conductivity below 50 µS/cm and dissolved organic carbon concentration below 0.2 mg/L.

2.2. Analytical method

Chlorophyll-a concentration (Algae lab analyzer, bbe Moeldaenke, Germany) was measured as a parameter for presence or absence of “intact” cells. In order to inspect and differentiate the physical morphology of the algae in both “intact” and “lysed” condition, particle size distribution analysis (multisizer 4 particle size analyzer, Beckman Coulter Counter, USA) with the aperture size detection between 2 to 60 µm was performed. Particle number and particle volume was measured and used as a control parameter.

Organic carbon concentration in forms of total organic carbon (TOC)) in feed and permeate was measured using TOC-L analyzer, Shimadzu, Japan. Additionally ultraviolet absorbance at wavelength 254 nm (UV_{254}) in either “intact” or “lysed” algae condition was measured using Lambda 20 UV, Perkin Elmer, USA. For all filtration experiments, a minimum of 6 samples was collected after 30 minutes of filtration time and TOC and UV_{254} was measured.

2.3. Ultrafiltration experiments

The membranes used for the experiments were Polyethersulfone (PES) multibore® hollow fiber capillaries (inge GmbH, BASF, Germany). The membrane module consisted of ten fibers (length 30 ± 0.5 cm) with an active surface area of 0.051 m². UF process was conducted at room temperature (19 - 22°C) by commercially

available lab-scale membrane filtration equipment (Poseidon Convergence Inspector, Netherlands) in a vertically arranged inside-out dead end mode fed from the bottom side of the module.

Filtration experiments were conducted with a flux J of 100 $L \cdot m^{-2} \cdot h^{-1}$ for 45 minutes per each cycle, followed by a mechanical backwash using RO water with a flux of 230 $L \cdot m^{-2} \cdot h^{-1}$ for 55 seconds, with 30 seconds to the bottom side of the module and 25 seconds to the top side of the module. The filtration cycles ended when either the permeability reached 100 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$ or 20 filtration cycles were performed.

Reference permeability (P_0) and permeability at the end of the last filtration cycle (P_e) are determined by filtrating RO water for 15 minutes in the beginning of the first filtration cycle and end of last filtration cycle respectively (Calculation cf. chapter "Evaluation of the membrane performance").

Subsequently chemical cleaning sequences were performed. Soaking procedure, soaking time and concentration were chosen in such a way to simulate a maintenance cleaning more related to an intensive chemical enhanced backwash (iCEB) than an intensive cleaning in place (CIP). This kind of iCEB could be used in praxis on a daily base without too much loss of recovery or availability of the membrane unit as in case of CIP. Two kind of iCEB were conducted. For both sodium hypochlorite (NaOCl) for oxidation and sodium hydroxide (NaOH) to adjust pH were used: iCEB (50 ppm free chlorine at pH 12) was performed two times followed by one time $iCEB^+$ (200 ppm free chlorine at pH 12.5) both at room temperature. In both cases iCEB solution was pumped into the module with a flux of 120 $L \cdot m^{-2} \cdot h^{-1}$ for 10 minutes, continued by soaking for 15 minutes and rinsing for 20 minutes (10 minutes to bottom side, 10 minutes to top side) with a flux of 230 $L \cdot m^{2} \cdot h^{-1}$.

The filtration experiments for *CS*, *AP*, and *TR* were repeated for 5, 8, and 3 times, respectively, in order to investigate the reproducibility of the filtration behavior.

2.4. Evaluation of the membrane performance

The performance of the membranes were evaluated by assessment of the permeability, total permeability loss, and permeability recovery using the a concept established by Bogati et al. (2015) during filtration process. The permeability and total permeability loss can be defined as:

$$Permeability\ (P) = \frac{Flux\ (J)}{Transmembrane\ pressure\ (TMP)} \quad (1)$$

$$Total\ permeability\ loss = P_0 - P_e \quad (2)$$

Water flux J and transmembrane pressure (TMP) were recorded every 5 seconds.

Reference permeability (P_0), permeability after backwash in each cycle (P_a), permeability before backwash in each cycle (P_b), permeability after regular and

intensive CEB (P_{iCEB} & P_{iCEB^+}) and permeability at the end of last filtration cycle (P_e) were calculated. The average of 120 data points (10 minutes) was collected to determine the P_0, P_{iCEB}, P_{iCEB^+}, and P_e. For P_a and P_b, the average of 10 data points (50 second) was used. Permeability recovery in each cycle was defined as:

$$Permeability\ Recovery = \frac{(P_a - P_b)}{(P_o - P_b)} \quad (3)$$

The efficiency of chemical cleaning process in both regular and intensive cleaning in place (iCEB and $iCEB^+$) was defined as:

$$Efficiency\ of\ iCEB = \frac{P_{iCEB}}{P_0} \quad (4)$$

The membrane rejection of TOC and UV_{254} can be calculated as:

$$R = 1 - \frac{C_P}{C_F} \quad (5)$$

in which R is the rejection, C_P is the permeate concentration (either TOC or UV_{254}) and C_F is the feed concentration (either TOC or UV_{254}).

3. Results and discussion

3.1 Control parameters of the feed solution

As control parameters in preparation of the feed solution of the algae cells in the "intact" condition, the total particle volume and chlorophyll-a concentration of feed water solution for each type of the algae were measured as follows:

(i) *Chlorella Sorokiniana* $14.00 \cdot 10^6 \pm 1.44 \cdot 10^6$ µm³/mL with chlorophyll-a concentration of 197 – 225 µg/L

(ii) *Arthrospira Platensis* $6.11 \cdot 10^6 \pm 1.59 \cdot 10^6$ µm³/mL with chlorophyll -a concentration 25 – 50 µg/L

(iii) *Thalassiosira Rotula* $9.64 \cdot 10^6 \pm 2.80 \cdot 10^6$ µm³/mL with chlorophyll -a concentration 28 – 42 µg/L

For preparation of the feed solution of the algae cells in the "lysed" condition, the same solution for the feed in "intact" condition was used after disruption with ultra sound device.

3.2 Characterization of "intact" and "lysed" algae

3.2.1 Particle number and volume distribution

By physical appearance, it was reliable to differentiate the "intact" and "lysed" algae based on their particle number and volume. This is attributed to the structures of algae having impact on the size distribution. During the "intact" condition, *AP, CS*, and *TR* (Figure 2 a-c) can be categorized as a single-cell with particle size between 3 - 6 µm, 10 – 20 µm, and 8 – 15 µm, respectively. These particle sizes are found to

be corresponding to the previous report where *CS*, *AP,* and *TR* possesses the cells with diameter size between 2 – 6 µm (Azaman et al., 2017), 2.5 – 16 µm (Sili et al., 2012) and 12 – 40 µm (Maier et al., 2012; Olenina et al., 2006), respectively. Although algae are single-cell in “intact” condition, however, coulter counter results showed that there was another dominant particle size present in the sample. The occurrence of this dominant particle size could be attributed to aggregation between single cells which led to the formation of additional peak in both particle volume and number analysis (cf. Figure 1).

In constrast, “lysed” condition of algae can be indicated by the change of cell number and volume distribution. Based on both algae number and volume distribution presented in Figure 1, it can be observed that a peak, so-called “first peak” which has particle diameter ranged from 2 to 7 µm has quenched and shifted to the smaller particle size region. Further, second peak with particle diameter ranged from 7 to 25 µm has disappeared completely due to the breakage of the cells and release of AOM. Moreover, concentration of chlorophyll-a was found to decrease and reach 0 µg/L after 3-5 days as a result of cell lysis (Passow, 2002).

Mechanical stress by ultrasound was found effective to disrupt the algae cells (Figure 2 d-f). Aforementioned, chlorophyll-a concentration, total particle volume and number of algae were decreased in “lysed” condition. These finding were supported by a previous work explaining that ultrasonic treatment could degrade algae cells and thus led to chlorophyll-a concentration reduction (Dehghani, 2016).

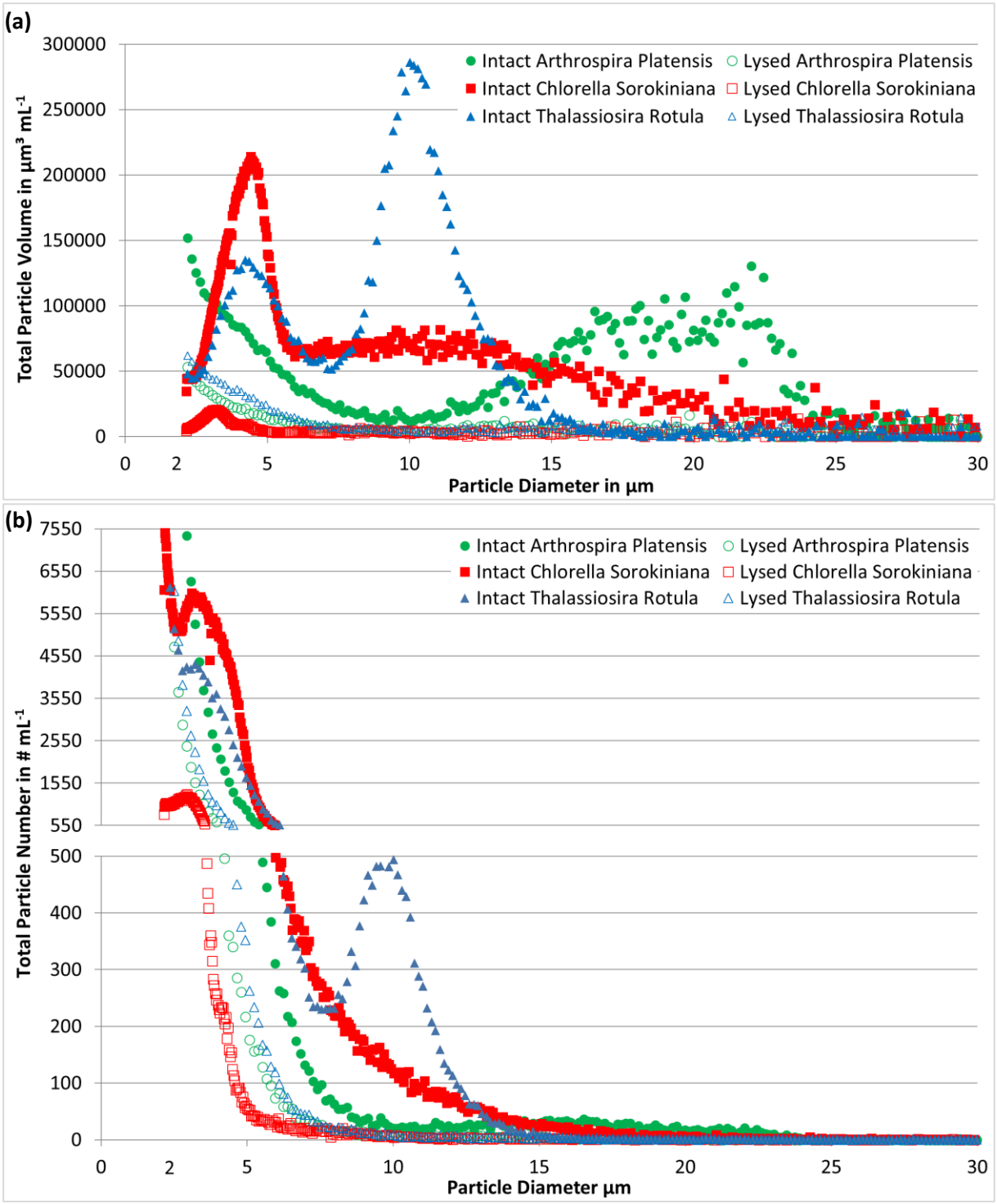

Figure 1. (a) Particle volume distribution and (b) particle number distribution both between 2 µm to 30 µm

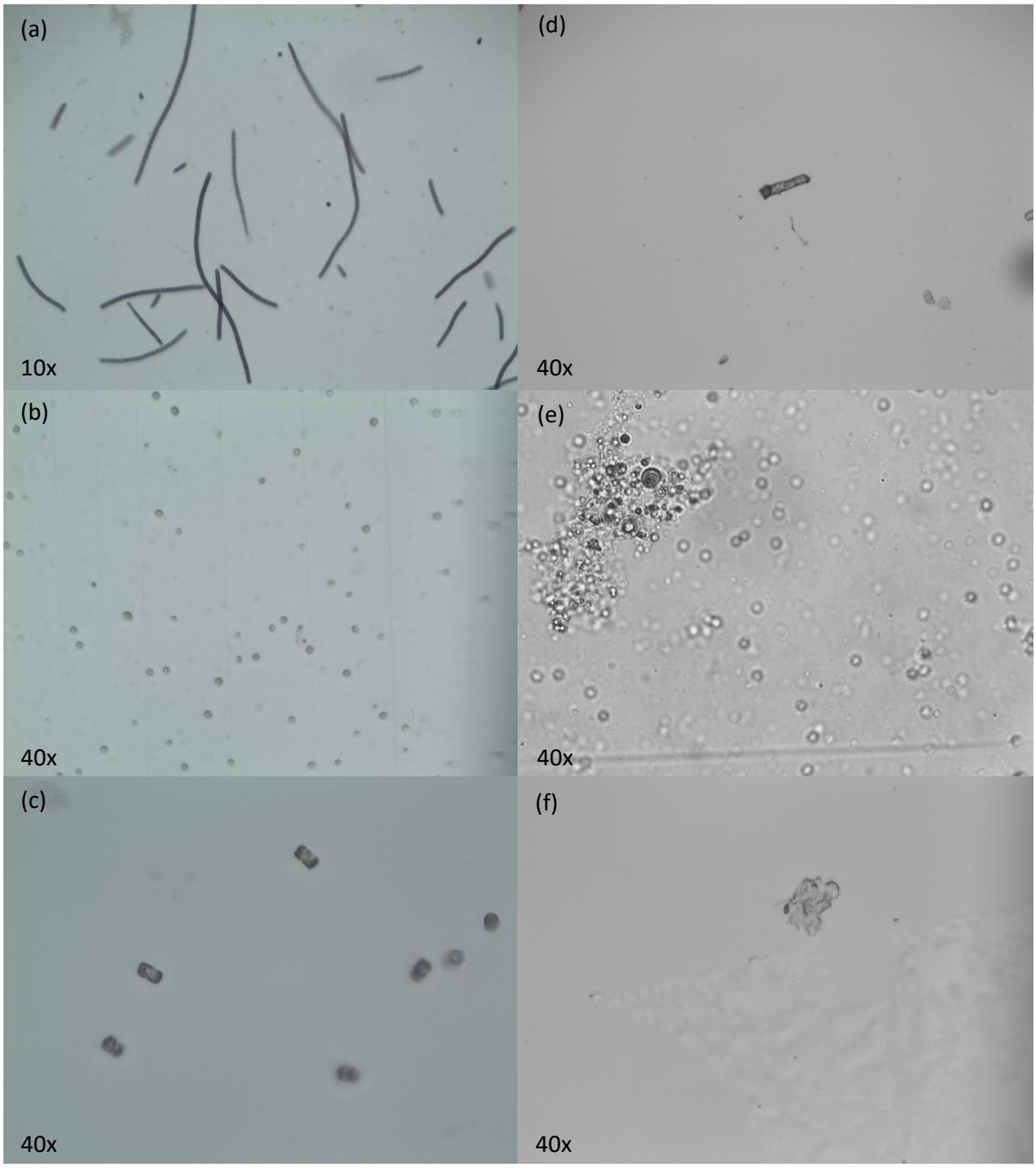

Figure 2. Microscopic image of “intact” and “lysed” *Arthrospira Platensis* (a & d), *Chlorella Sorokiana* (b & e), and *Thalassiosira Rotula* (c & f).

3.2.2 Total organic carbon and UV_{254} analysis

Basically, TOC and UV_{254} absorbance are employed to measure the concentration of organic matter present in water. Moreover, these analyses can be used to distinguish the type of organic matters, where TOC indicates all of organic content while UV_{254} represents UV-active substances with conjugated double bonds. Among these substances, protein-like substances are ascertainable by UV_{254} measurement but polysaccharides are not as they are not UV-active. Damaging algae by mechanical stress cause the release of the internal organelles and organic matter to the water

(Ladner et al., 2010). Resulting AOM contains a high proportion of protein-like and polysaccharide-like compounds (Hoyer et al., 1985). Regarding to this, the absorbance of organic indicator (UV_{254}) was expected to have different value for algae in “lysed” compared to “intact” condition.

As can be seen from Figure 3a and Figure 4a, the three types of “lysed” algae showed same TOC concentration but higher UV absorbance compared to “intact” indicating a strong increase in protein-like substances due to the release of algae substance into the feed water. The comparative strong deviations of the TOC values are caused by some trouble with the feeding system of the TOC measuring device especially for algae and their constituent parts. Moreover, higher TOC concentration and UV absorbing compounds were also found in the permeate side in “lysed” compared to “intact” (Figure 3b and Figure 4b) condition indicating that some of the UV active lyse products (probably protein-like compounds) can penetrate the membrane. Due to the above mentioned problems, TOC results of AP are not evaluable and thus not presented here.

3.3 Membrane performance

3.3.1 Membrane rejection

In all of the experiments, a higher maximum rejection of the UV_{254} absorbing compounds could be detected in the “lysed” condition compared to “intact” condition (see Figure 4c in which the ration of permeate to feed is presented). The maximum rejections of *CS, AP*, and *TR* in the “intact” condition were 34 %, 31 %, and 21 % respectively. This values were lower compared to the “lysed” condition (49 %, 39 %, and 37 %).This was in contradiction with findings of Ladner et al. (2010) in which the UV absorbance rejection in ”intact” condition was higher compared to “lysed” condition. Opposite to this, TOC rejection of *CS* and *TR* in “intact” condition was higher than in “lysed” condition (see Figure 3c). The median rejection for “intact” cells for *CS* and *TR* were 83% and 58%, respectively, and the median rejection for “lysed” *CS* and *TR* algae were 49% and 45%, respectively. This lower rejection values in “lysed” condition can be explained by the penetration of produced AOM after cell disruption through membrane and measurement of higher TOC values in the permeate (L. O. Villacorte et al., 2009). With respect to this result the conducted algal cell lyse produced for the considered types of algae more UV active compounds which can be rejected than these which can penetrate the membrane.

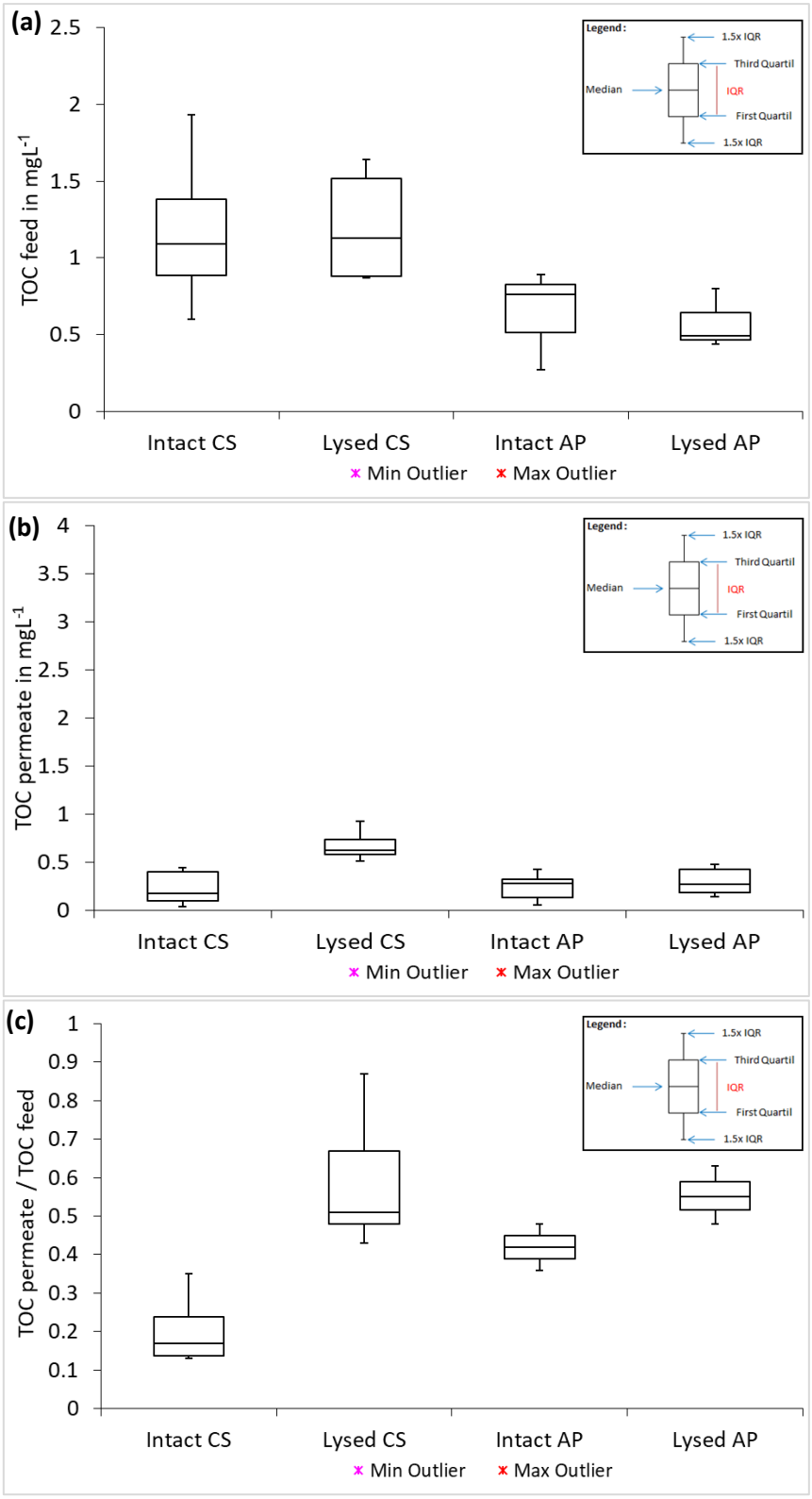

Figure 3. (a) TOC concentration in Feed, (b) permeate, and (c) ratio between TOC in permeate and feed

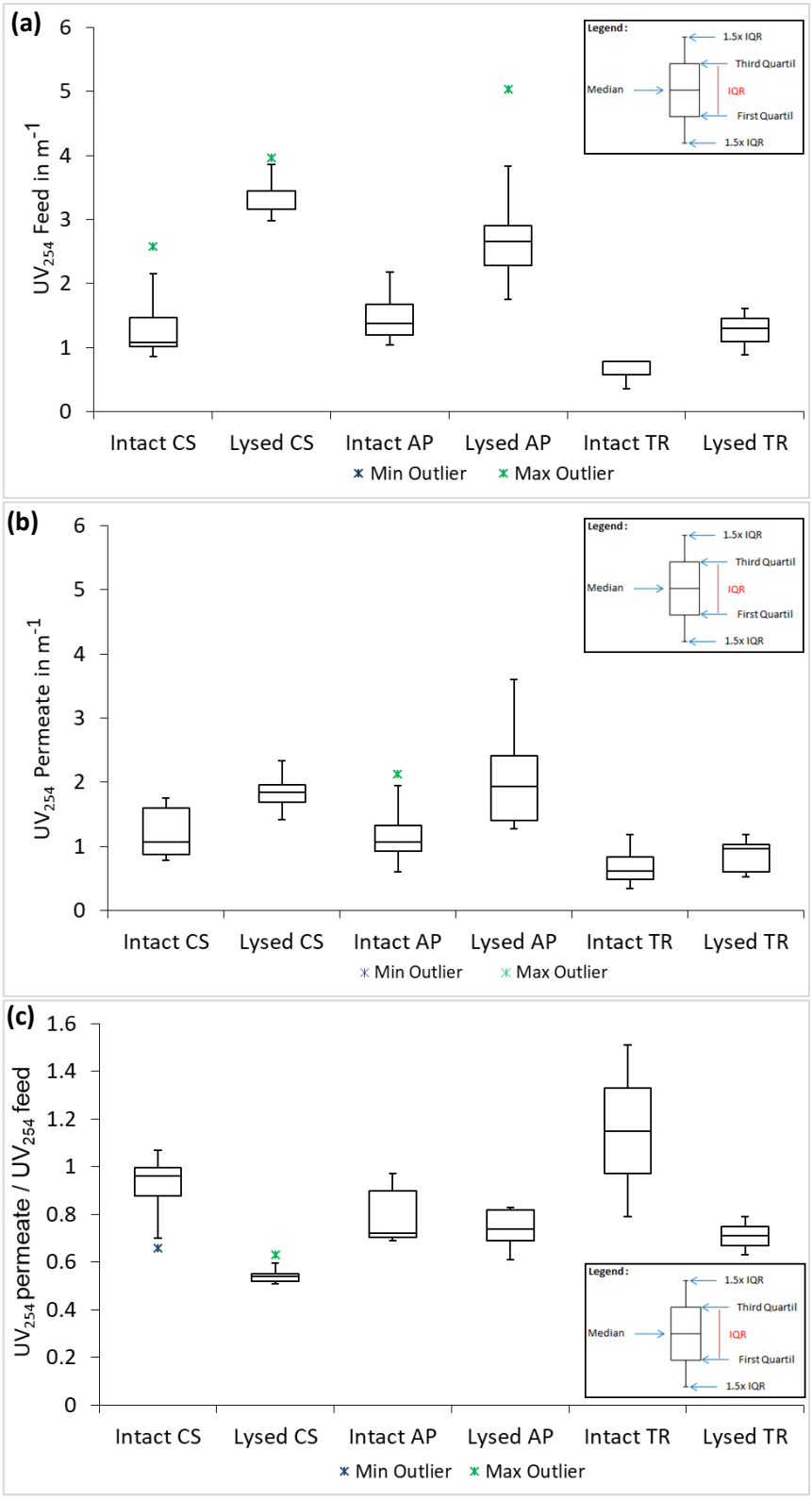

Figure 4. (a) UV_{254} absorbance feed, (b) permeate, and (c) Ratio between UV_{254} absorbance in permeate and feed

3.3.2 Membrane fouling

In order to assess the membrane fouling affinity filtration cycle tests with the 3 algae types in “intact” and “lysed” condition were conducted and permeability recovery

during each filtration experiment was calculated. As mentioned in the chapter “Material and methods”, filtration cycle tests had been finished when either the permeability reached 100 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$ or 20 filtration cycles were performed. Although total particle volume of all feed water suspensions with different algae types were almost the same, different fouling behavior was observed during the filtration of “intact” and “lysed” condition in different algae types.

As it can be seen in Figure 5a and Figure 5c, for the case of “intact” *TR*, a strong permeability reduction was found in the first 5 filtration cycles followed by a smooth reduction of the permeability during next 15 filtration cycles until it reached the limit of 20 filtration cycles with total permeability reduction of around 50 %. Moreover, both “intact” *CS* and *AP* showed a strong permeability reduction during the whole filtration cycles till the end of the filtration experiments (17 cycles for *CS* and 13 cycles for *AP*) with total permeability loss of around 90 % and 80 % for algae *CS* and *AP* respectively. This difference in the fouling behavior might be due to different particle size distribution and shape of different algae types (Figure 1 and Figure 2) causing differences in membranes pore plugging.

Figure 5b and Figure 5d shows the filtration experiment results for all three algae types in the “lysed” condition. A much more sever fouling was detected for “lysed” *CS* compared to its “intact” condition in which the experiment ended after only 5 filtration cycles. Similar to its “intact” condition, *TR* reached the end of the experiment after 20 filtration cycles, however the reduction of the permeability was around 60 % showing an only slightly stronger fouling compared to its “intact” condition. Unlike *CS* and *TR*, “lysed” *AP* showed better fouling behavior compared to its “intact” condition where 18 filtration cycles could be performed till the end of filtration time with a permeability loss of roughly 65 %.

In case of *CS* and *TR*, the more severe fouling can be explained by the strong release of AOM after damaging the algae cells. High proportion of protein-like and probably polysaccharide-like compounds in a sticky liquid form can partly penetrate into the membrane pores or stick on the membrane surface forming a gel layer and exacerbate the performance of the membrane. This was in agreement with the study of Merle et al. (2016) & Ladner et al. (2010) in which also stronger fouling was observed in the membranes during filtration of algae in the damaged condition or death phase compared to undamaged algae cells or cells in the exponential growth phase.

The better performance of *AP* in the “lysed” condition might be due to different characteristics of the released AOM from the AOM produced by *CS* and *TR*. The microscopic images and particle size analysis of *AP* (Figure 1 and Figure 2) in the “lysed” condition show much smaller filaments compared to “intact” condition. Additionally, the microscopic images of *CS* and TR (cf. Figure 2 (e) & (f)) show a slime form of substance. However, the slime substance could not be seen in microscopic images of “lysed” *AP*.

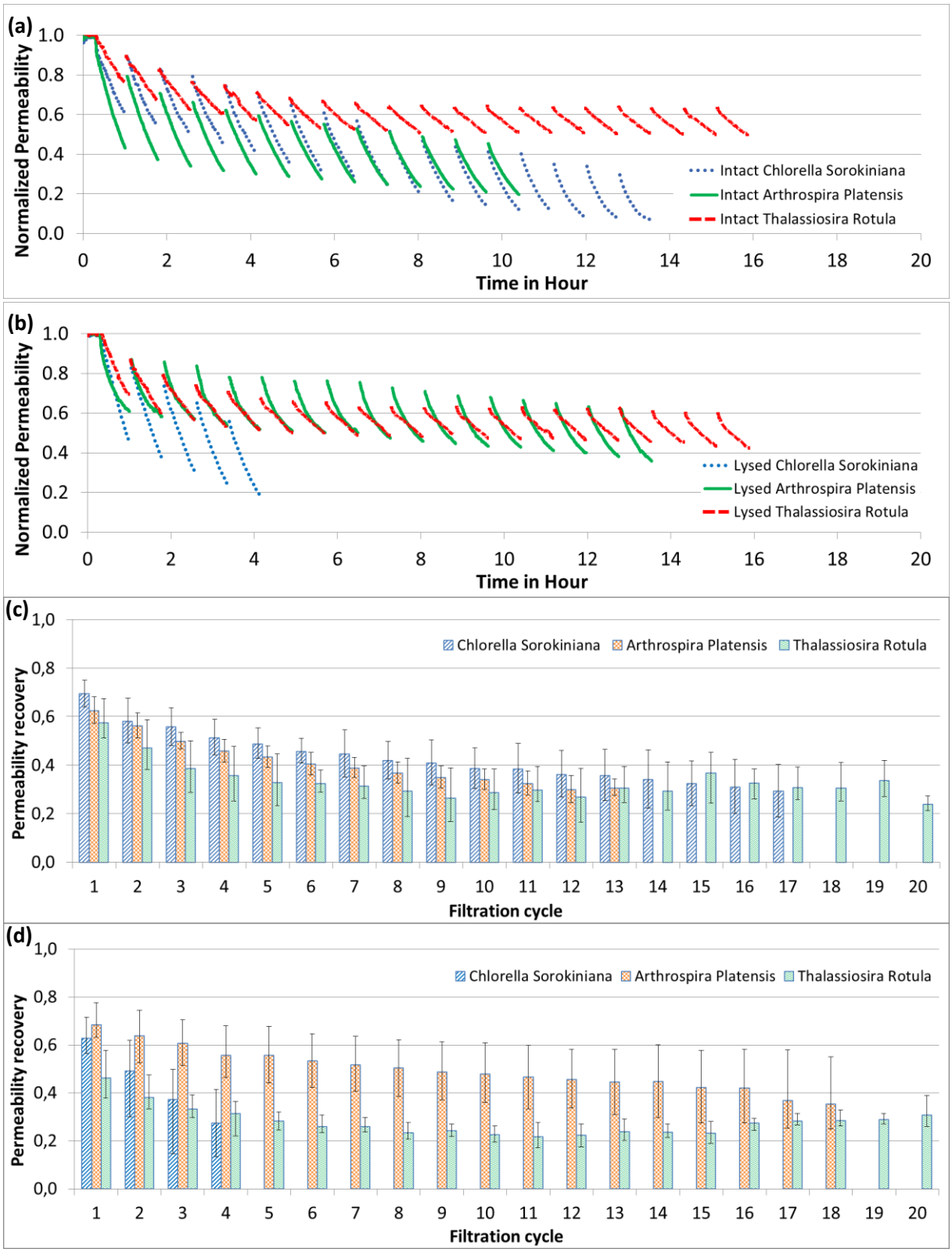

Figure 5. Permeability reduction and recovery in “intact” (a & c) and “lysed” (b & d) condition

3.3.3 Membrane cleaning

The chemical cleaning was found to be effective in cleaning and recovering the membrane permeability for “intact” and even better in “lysed” conditions. Figure 6 shows that already the first iCEB could recover 70 to 80 % of the initial permeability for ”intact” condition and even 80 to 90 % in “lysed” condition a little bit depending on the type of algae. The final $iCEB^{+}$ sequence further improved the recovery in both “intact” and “lysed” condition especially in the experiments with TR where more than 95 % recovery could be achieved. It can be concluded that the membrane cleaning procedure applied in this work is very effective, even if the cleaning step is more related to CEB than to CIP.

Similar cleaning process was employed by Merle et al. (2016) for outside-in PVDF UF hollow fibers. NaOCl with a concentration of 350 mg/L, not pH adjusted, feeding flux 120 $L \cdot m^{-2} \cdot h^{-1}$ for 3 minutes, soaking time 5 min had the recovery between 46 to 67 % and 16 to 50 % in restoring the permeability for alga-rich water in the “intact” and “lysed” condition respectively. This indicated that either optimization of chemical cleaning procedure or type of algae or membrane can play an important role in recovery of membrane permeability.

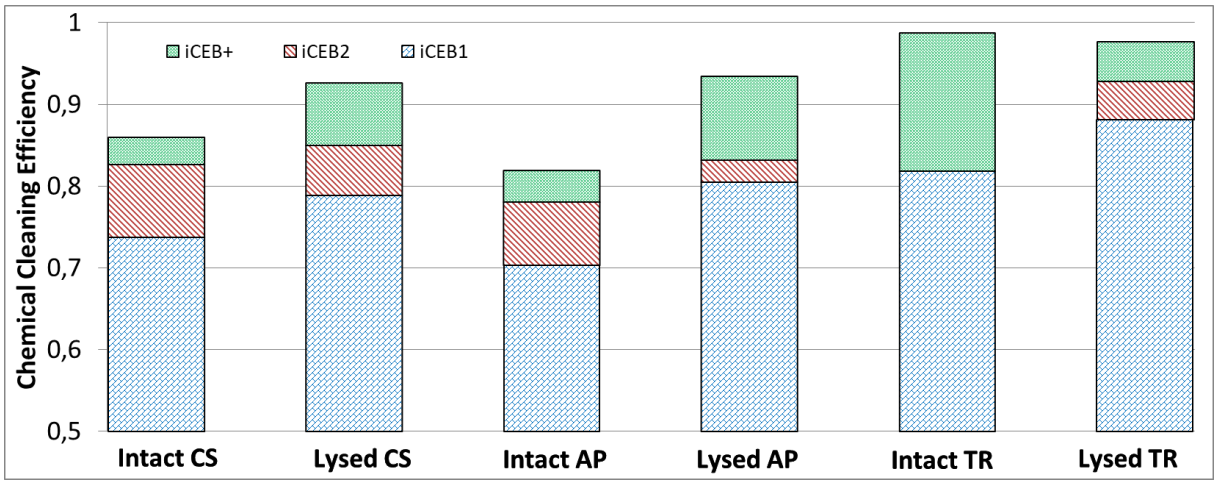

Figure 6. Membrane cleaning efficiency

4. Conclusion

This study aims to assess the impact of three types of algae (*Chlorella Sorokiniana*, *Arthrospira platensis*, and *Thalassiosira Rotula*) in “intact” and “lysed” condition on UF membrane. The main findings are:

1. The different size and shape of algae in “intact” condition resulted in different membrane fouling behavior

2. A better performance of the membrane during filtration of *Arthrospira Platensis* in “lysed” condition, compared to the “intact” condition is found

3. Moderate and severe membranes fouling during filtration of *Thalassiosira Rotula* and *Chlorella Sorokiniana* in the “lysed” condition compared to “intact” condition are found.

4. A chemical cleaning procedure close to usual chemical enhanced backwash is applied with which about 85 to 90% of the initial permeability could be recovered for all types of algae in both “intact” and “lysed” condition. In this context the achieved permeability recovery can be seen as success especially because an abnormal high concentration of AOM was filtrated in the lab experiments.

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