



Article

Optimal Mesh Pore Size Combined with Periodic Air Mass Load (AML) for Effective Operation of a Self-Forming Dynamic Membrane BioReactor (SFD MBR) for Sustainable Treatment of Municipal Wastewater

Senouci Boulerial ^{1,2,†}, Carlo Salerno ^{1,*,†}, Fabiano Castrogiovanni ¹, Marina Tumolo ¹, Giovanni Berardi ¹, Abdelkader Debab ², Boumediene Haddou ³, Abdellah Benhamou ³ and Alfieri Pollice ¹

- ¹ CNR IRSA (National Research Council of Italy, Water Research Institute), V.le F. De Blasio 5, 70132 Bari, Italy; senouci.boulerial@univ-usto.dz (S.B.); fabiano.castrogiovanni@ba.irsa.cnr.it (F.C.); marina.tumolo@ba.irsa.cnr.it (M.T.); giovanni.berardi@ba.irsa.cnr.it (G.B.); alfieri.pollice@cnr.it (A.P.)
- Laboratory of Process Engineering and Environment, University of Science and Technology of Oran, BP 1505, Elmnouar, Oran 31000, Algeria; abdelkader.debab@univ-usto.dz
- Laboratory of Physical Chemistry of Materials, Catalysis and Environment, University of Science and Technology of Oran, BP 1505, Elmnouar, Oran 31000, Algeria; boumeddiene.haddou@univ-usto.dz (B.H.); abdellah.benhamou@univ-usto.dz (A.B.)
- * Correspondence: carlo.salerno@cnr.it
- [†] These authors contributed equally to this work.

Abstract: A self-forming dynamic membrane bioreactor (SFD MBR) is a cost-effective alternative to conventional MBR, in which the synthetic membrane is replaced by a "cake layer," an accumulation of the biological suspension over a surface of inert, low-cost support originated by filtration itself. Under optimized conditions, the cake layer is easy to remove and quick to form again, resulting a "dynamic membrane." The permeate of the SFD MBR has chemo-physical characteristics comparable to those of conventional ultrafiltration-based MBR. In this paper, two nylon meshes with pore sizes of 20 and 50 µm, respectively, were tested in a bench-scale SFD MBR in which an air mass load (AML) was periodically supplied tangentially to the filtration surface to maintain filtration effectiveness. The SFD MBR equipped with 20 µm nylon mesh coupled with 5 min of AML every 4 h showed the best performance, ensuring both a permeate with turbidity values always below 3 NTU and revealing no increases in transmembrane pressure (TMP) with manual maintenance needs. A benchmark test with the only difference of a suction break (relaxation) instead of AML was conducted under identical operating conditions for validation with an already known maintenance strategy. This latter test produced a permeate of very good quality, but it needed frequent TMP increases and consequent manual cleanings, showing that a periodic AML coupled with the use of a 20 μm mesh can be an optimal strategy for long-term operation of SFD MBR.

Keywords: biological membrane; SFD MBR; trans-membrane pressure; dynamic membrane; turbidity; air mass load

check for updates

Citation: Boulerial, S.; Salerno, C.; Castrogiovanni, F.; Tumolo, M.; Berardi, G.; Debab, A.; Haddou, B.; Benhamou, A.; Pollice, A. Optimal Mesh Pore Size Combined with Periodic Air Mass Load (AML) for Effective Operation of a Self-Forming Dynamic Membrane BioReactor (SFD MBR) for Sustainable Treatment of Municipal Wastewater. *Processes* 2024, 12, 323. https://doi.org/10.3390/ pr12020323

Academic Editors: Dimitris Zagklis and Georgios Bampos

Received: 10 January 2024 Revised: 26 January 2024 Accepted: 31 January 2024 Published: 2 February 2024



Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

1. Introduction

The membrane bioreactor (MBR) is an established technology for the treatment and reuse of domestic and industrial wastewater [1–3]. It is based on solid/liquid activated sludge separation through synthetic membranes made of different materials and operated by positive or negative (suction) force. The membrane filtration pore size range for MBR includes microand ultra-filtration [4]. In MBR, the role of the membrane is to separate the supernatant from the suspended solids, and this may be obtained by adopting mainly two possible configurations: (i) membranes submerged in the bioreactor (submerged MBR), (ii) membranes immersed in the secondary clarifier or in another separate vessel (sidestream MBR). MBR technology offers

Processes **2024**, 12, 323 2 of 11

several advantages with respect to conventional activated sludge (CAS) systems, including a significantly reduced footprint and improved degradation of pollutants. This is mostly due to the possibility of operating the system with higher concentrations of suspended solids in the mixed liquor (MLSS) and to the absence of a secondary clarifier. Submerged MBR allows for the adsorption, biodegradation, and membrane separation in the same biological tank [5]. Moreover, wastewater treatment plants (WWTPs) based on MBR technology usually produce permeates of excellent quality with very low levels of total suspended solids (TSS), turbidity, chemical oxygen demand (COD), biological oxygen demand (BOD), and pathogens [6]. In specific situations, MBR can be coupled with or integrated into other technologies to ameliorate wastewater treatment performance [7].

Nevertheless, a limitation of the application of MBR is the occurrence of membrane fouling and pore clogging, which deteriorate the system's performance, require maintenance efforts, and may shorten the membrane's service life. Both phenomena are detectable by monitoring the resistance to filtration imposed by the materials that tend to accumulate over the membrane surface or into the membrane structure, called transmembrane pressure (TMP) [8]: when the TMP (in absolute values) rapidly increases, the membrane is fouling/clogging, leading to a decrease in permeate flux. The mechanisms of fouling are: (i) adsorption of soluble microbial products (SMP), extracellular polymeric substances (EPS), colloids, and other particles into/on the membranes; (ii) deposition of sludge flocs on the membrane surface with consequent formation of a "cake" layer on the membrane surface; (iii) changes in membrane and/or mixed liquor composition during long-term operation (e.g., changes in bacterial community and biopolymer components in the cake layer, degradation of membrane composition) [9].

Periodic maintenance of MBR systems is often accomplished either by backwashing the membranes, i.e., temporarily reversing the permeate flow, or by cyclic relaxation from suction, which simply involves stopping permeate extraction for a defined time interval. These techniques do not influence the ordinary functionality of the bioreactor, as they are conventionally incorporated into most MBR designs as standard operational strategies for fouling control, and normally do not require chemical reagents, preventing any risk of membrane degradation/damage [10,11].

When the TMP thresholds determining significant and critical reductions in flux are passed despite periodic maintenance, the membrane needs to be removed from the biological tank to be manually cleaned [12–14]. For MBR in treating municipal wastewater, water jet rinsing is ordinarily enough to remove the pore clogging and excess sludge accumulation and to recover the initial set flux. If the flux is not recovered due to a deep fouling of membrane pores, a chemical treatment is needed [15]. On the contrary, when the TMP does not tend to increase and the quality of permeate decreases, the integrity of the membrane should be tested, with its possible (partial or complete) replacement [16]. This may imply a relevant burden in terms of investment cost.

In the last decade, self-forming dynamic membrane bioreactors (SFD MBRs) were developed as a cost-effective alternative to conventional ultrafiltration (UF)-based MBR, and their application in wastewater treatment has been studied [17,18]. The SFD MBR is a particular MBR in which inert materials (meshes, nets) with medium-large pore-size (in the range of 10– $500~\mu m$) are used as supports for the formation of cake layers, these becoming the real biological membrane [19]. Different studies have revealed that the main chemical and physical characteristics of the SFD MBR permeate can be similar to those of conventional MBR permeate, apart from the microbiological quality indicators, so a post-disinfection step is still required, especially in the case of effluent reuse. An advantage of SFD MBR with respect to classical MBR is that chemical or other deep cleaning procedures are rarely used because the medium-large pore-size support media are less exposed to critical clogging than the UF membranes used in MBR, and a physical cleaning is usually enough to remove the cake layer from the support surface. In conventional MBR, the gel layers that may develop over the long term can clog the membrane pores [17]. To solve this, the modules are submitted to chemical treatment for the oxidation and removal of the

Processes **2024**, 12, 323 3 of 11

sticky and colloidal substances that pass inside the small pores. In SFD MBR, controlling and limiting the clogging gel layer is easier due to the larger pore size, and often physical methods, such as water jet rinsing, surface air sparging, permeate backwashing, and flux relaxation, are efficient [20]. Afterwards, the filtration system can soon be restored so that the biological membrane can form again. The easy removal and then reforming of the biological membrane explain why it is also called "dynamic membrane" (DM) [21].

In a green economy context, SFD MBR is a lower-pollutant and energy-saving technology because no chemicals are used for cleaning and lower pressure is required for filtration (in the range of a few hundred mbar, also achievable by gravity) with respect to conventional UF-based MBR.

In a previous paper, Salerno and co-authors showed the effectiveness of SFD MBR for the treatment of municipal wastewater and with limited maintenance needs in tests with low sludge retention time (SRT) [20]. The purpose of the present paper is to evaluate the performance of a bench-scale SFD MBR in treating real municipal sewage with a medium-high SRT of 30 days, having support media with two different pore sizes, and with a maintenance strategy based on a periodic air mass load (AML, large bubbles causing turbulence at the filtration surface). In the first experiment, called test A, a 50 μ m nylon mesh was used as the support material for the development of DM with a periodic cleaning of the mesh using a high air mass flow rate in short time. The second test, named test B, was identical to the first but used a 20 μ m nylon mesh. Test B had better performance than test A, so it was finally compared to a benchmark test, called test C, under the same conditions and mesh as test B, but with a different and already known maintenance strategy based on periodic relaxation from permeate suction. Finally, the best performance, both in terms of permeate quality and support cleaning requirements, was shown by test B (20 μ m SFD MBR coupled with an AML of 5 min every 4 h).

2. Materials and Methods

All bench-scale SFD MBR plants, the features of which are summarized in Table 1, were operated at room temperature, continuously aerated, and under the same operating conditions, except for the pore size of the support mesh and the strategy of periodic maintenance.

| Parameter | Test A | Test B | Test C |
|------------------------------|----------------------------------|----------------------------------|----------------------------------|
| SRT | 30 days | 30 days | 30 days |
| Volume | $16.0~\mathrm{L}$ | 16.0 L | 16.0 L |
| Filtering area | 0.0072 m^2 | 0.0072 m^2 | 0.0072 m^2 |
| Target flux | $73 \mathrm{L m^{-2} h^{-1}}$ | $73 \mathrm{L m^{-2} h^{-1}}$ | $73 \mathrm{L m^{-2} h^{-1}}$ |
| Mesh pore size | 50 μm | 20 μm | 20 μm |
| Periodic maintenance * | AML | AML | relaxation |
| No suction time distribution | 3' break + $5'$ AML + $3'$ break | 3' break + $5'$ AML + $3'$ break | 11′ break |

Table 1. Main characteristics and operating conditions of the bench-scale SFD MBR plants.

In the bench-scale SFD MBR, two filtration modules were positioned vertically and face-to-face, at a distance of about 3 cm from one another, and every single module had a 6×6 cm filtration surface, for a total surface of 72 cm². Aeration was provided in the reactors by four external air pumps (M2K3, Schego, Frankfurt am Main, Germany), respectively connected to four fine-bubble diffusers placed on the reactor bottom. The pumped air also ensured the necessary mixing of sludge to achieve homogeneity of the suspended biomass. For every test, permeate suction was ensured by a peristaltic pump connected to the filtration modules with a set flow rate of 12.6 L d⁻¹. The TMP was measured by an analogic manometer placed between DM and the suction pump and recorded at least every hour between 9:00 A.M. and 5:00 P.M. from Monday to Friday. In test A, a support nylon mesh with a pore size of 50 μ m was used, while a 20 μ m nylon mesh was employed in tests B and C. When the TMP overcame the threshold of -200 mbar, the modules were temporarily removed from the bioreactor, washed by tap water jet rinsing,

^{*} every 4 h.

Processes **2024**, 12, 323 4 of 11

and finally reassembled to restart. As summarized in Table 1, all systems had a periodic 4-h cycle consisting of 229 min of suction and 11 min of no suction. In tests A and B, the no suction time was organized as follows: 3 min of simple suction break, 5 min of AML with an air flow rate of 42.0 $L_{\rm air}$ min⁻¹ supplied tangentially to the filtration surface (still without any permeate suction), and another 3 min of suction break, as described by Salerno and colleagues [20]. In Test C, the whole 11 min period was in simple no suction mode, called relaxation. The bioreactor's operating volume was maintained constant through a level control switch connected to the feed pump. The latter was turned on as the level control detected a decrease in the reactor's operating volume, and it was turned off when the volume had been restored. The general scheme for all plants is illustrated in Figure 1.

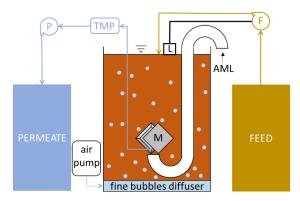


Figure 1. Plant scheme common to all tests. F is the feed pump; L is the level control that activates F; M is the couple of filtration modules; TMP is the manometer measuring transmembrane pressure; P is the permeate suction pump; AML is the periodic air mass load pipeline (not present in test C).

Real pre-settled municipal wastewater was collected twice per week from the municipal wastewater treatment plant of Giovinazzo (41°10′39.6″ N 16°41′04.5″ E, Area Metropolitana di Bari, Italy), managed by Acquedotto Pugliese S.p.A. (Bari, Italy). The wastewater was characterized, diluted to the target value of 460 mg COD $\rm L^{-1}$, and finally given as feed to the SFD MBR. Table 2 shows the average characteristics of the feed.

| Table 2. Main conventional parameters of the wastewater feeding the SFD MB | R. |
|---|----|
| | |

| Parameter | Unit | Average \pm St.Dev. |
|--------------------------------|-------------------------------|--|
| TSS | ${\sf mg}~{\sf L}^{-1}$ | 248.8 ± 103.6 |
| VSS | $mg L^{-1}$ | 243.2 ± 95.9 |
| COD | $ m mg~L^{-1}$ | 460.0 ± 22.6 |
| soluble COD | $mg L^{-1}$ | 112.3 ± 49.0 |
| TN | $mg L^{-1}$ | 65.5 ± 17.3 |
| N-NH ₄ ⁺ | $ m mg~L^{-1}$ | 42.0 ± 11.1 |
| N-NO ₂ - | $mg L^{-1}$ | 0.1 ± 0.0 |
| N-NO ₃ | $mg L^{-1}$ | 0.2 ± 0.2 |
| pН | - | 7.4 ± 0.2 |
| Electr. conductivity | $\mathrm{mS}\mathrm{cm}^{-1}$ | 1.3 ± 0.5 |
| Tot. coliforms | $MPN \ 100 \ mL^{-1}$ | 2.5×10^7 (median); 2.0×10^6 (min); 7.9×10^7 (max) |
| E. coli | $\rm MPN~100~mL^{-1}$ | 7.9×10^6 (median); 3.0×10^5 (min); 2.9×10^7 (max) |

Both the feeding wastewater and the produced permeate were characterized twice per week in terms of total and volatile suspended solids (TSS and VSS, respectively), chemical oxygen demand (COD), total nitrogen (TN), ammonium, nitrite, and nitrate, according to standard methods [22]. Electrical conductivity and pH were measured with an InnoLab[®] Multi 9420 IDS (WTW, Weilheim, Germany), while permeate turbidity was determined by a 2100P turbidimeter (HACH, Loveland, CO, USA). The activated sludge was characterized on the same days as the feed and permeate. The mixed liquor suspended solids (MLSS) and the sludge volume index at 30 min (SVI₃₀) of the SFD MBR activated sludge were measured according to standard methods [22]. Conventionally, the SVI₃₀ is an evaluation

Processes **2024**, 12, 323 5 of 11

test of sludge settling capacity [23]. A phase-contrast microscope BX50 (Olympus, Tokyo, Japan) was used to evaluate the morphological characteristics of the activated sludge.

3. Results

3.1. Activated Sludge Characteristics

The activated sludge features during the three tests are displayed in Table 3.

Table 3. Activated sludge characteristics during the different tests.

| Parameter | Unit | Test A | Test B | Test C |
|-------------------|------------------------------|------------------|-----------------|-----------------|
| MLSS | $ m gL^{-1}$ | 3.4 ± 1.2 | 4.4 ± 1.3 | 2.9 ± 1.6 |
| MLVSS | $ m g~L^{-1}$ | 3.0 ± 1.0 | 3.8 ± 1.1 | 2.6 ± 1.4 |
| SVI ₃₀ | $\mathrm{mL}\mathrm{g}^{-1}$ | 64.3 ± 14.1 | 92.1 ± 8.6 | 43.9 ± 9.9 |
| Temperature | °C | 20.0 ± 0.6 | 20.2 ± 0.2 | 22.5 ± 0.8 |
| DO | ${ m mg~L^{-1}}$ | 6.3 ± 1.1 | 4.1 ± 1.2 | 6.2 ± 1.8 |
| ORP | $m\widetilde{ m V}$ | 305.5 ± 39.1 | 294.6 ± 6.2 | 314.8 ± 9.7 |
| pН | - | 6.8 ± 0.5 | 7.1 ± 0.5 | 7.0 ± 0.8 |

Generally, all of the SFD MBR tests had average concentrations of mixed liquor suspended solids (MLSS) between 3 and 4.5 g L $^{-1}$, with about 90% volatile suspended solids (MLVSS). The SVI $_{30}$ values of the tests never approached the threshold of 150 mL g $^{-1}$, after which sludge bulking generally occurs [24]. The pumped oxygen ensured aerobic conditions in all experiments, achieving dissolved oxygen (DO) values always well above 3 mg L $^{-1}$, and the redox potential (ORP) and pH average values were always around 300 mV and 7.0, respectively. Images of fresh activated sludge taken by a phase-contrast microscope at $100 \times$ magnification and related to four different moments for each test are shown in Figure 2.

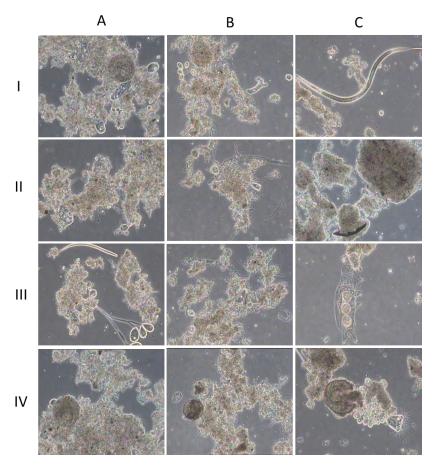


Figure 2. Activated sludge samples under phase-contrast microscopy at $100 \times$ magnification. The three tests are reported in columns (A–C), each one represented by four pictures in each column (I–IV).

Processes **2024**, 12, 323 6 of 11

3.2. Performance of the SFD MBR Tests

Figure 3 shows the trends in permeate turbidity, flux, and wash events for every test.

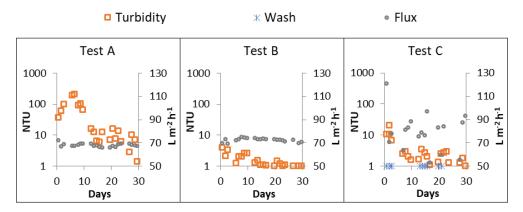


Figure 3. Trends in permeate turbidity, flux, and wash events of Tests A, B and C, respectively. Turbidity values are expressed in terms of nephelometric turbidity unit (NTU), while flux values are expressed in terms of liters per square meter per hour (L m^{-2} h^{-1}).

Tests A and B maintained the set flux over time, while test C was revealed to be more problematic and the flux was more heavily affected by the filtration efficiency. Indeed, the higher frequency of TMP increase observed in test C caused a decrease in permeate flux and was only temporarily solved with module washing. On the contrary, in tests A and B, the TMP did not tend to increase over time, and consequently no module washing was required. Moreover, test C showed some fluctuations in the permeate turbidity, but still with an average value of around 5 NTU. Test B always produced an effluent with turbidity values even below 3 NTU. Generally, when the mesh with 20 μ m pore size was adopted (tests B and C), the permeate turbidity was consistently under 10 NTU. On the contrary, in test A (equipped with 50 μ m mesh), the permeate turbidity was always higher than 50 NTU during the first 10 days (with a peak of 211 NTU), which decreased to lower values around 10 NTU in the following 20 days, reaching values below 5 NTU toward the end of the test. In Table 4, the main quality parameters of the three permeates are compared. The total coliforms and *Escherichia coli* contents in all permeates showed median, minimum, and maximum values between 4 and 5 Log and between 3 and 5 Log, respectively.

| Table 4. Comparison of the p | produced permea | tes from every | SFD MBR. |
|-------------------------------------|-----------------|----------------|----------|
|-------------------------------------|-----------------|----------------|----------|

| Parameter | Unit | Test A | Test B | Test C |
|--------------------------------|-----------------------|---------------------------------|---------------------------------|---------------------------------|
| TSS | ${\rm mg~L^{-1}}$ | 366.7 ± 78.5 | 4.7 ± 1.9 | 6.4 ± 6.2 |
| COD | $ m mg~L^{-1}$ | 103.0 ± 86.7 | 30.4 ± 5.0 | 32.8 ± 6.2 |
| TN | ${ m mg}~{ m L}^{-1}$ | 98.7 ± 52.1 | 55.1 ± 5.3 | 41.3 ± 8.8 |
| $N-NH_4^+$ | $ m mg~L^{-1}$ | 1.0 ± 2.3 | 0.1 ± 0.1 | 0.3 ± 0.3 |
| N-NO ₂ ⁻ | $ m mg~L^{-1}$ | 0.0 ± 0.0 | 0.0 ± 0.0 | 1.6 ± 0.9 |
| N-NO ₃ ⁻ | $ m mg~L^{-1}$ | 24.7 ± 5.1 | 35.7 ± 6.7 | 27.1 ± 7.3 |
| Electr. conductivity | ${ m mS~cm^{-1}}$ | 1.0 ± 0.1 | 0.8 ± 0.0 | 1.1 ± 0.0 |
| рН | - | 7.1 ± 0.8 | 7.4 ± 0.3 | 7.3 ± 0.3 |
| Tot. coliforms | $ m MPN~100~mL^{-1}$ | $1.6 	imes 10^5$ (median) | $4.4 	imes 10^5$ (median) | 1.6×10^4 (median) |
| | | $1.3 	imes 10^5$ (min) | $5.0 \times 10^4 (min)$ | $1.0 \times 10^4 (\text{min})$ |
| | | $1.9 \times 10^5 (\text{max})$ | $4.6 \times 10^5 (\text{max})$ | $2.2 \times 10^4 (\text{max})$ |
| E. coli | $ m MPN~100~mL^{-1}$ | 6.0×10^4 (median) | 1.0×10^5 (median) | 8.2×10^3 (median) |
| | | $5.8 \times 10^4 (\text{min})$ | 2.0×10^4 (min) | $6.3 \times 10^3 \text{ (min)}$ |
| | | $6.3 \times 10^4 \text{ (max)}$ | $2.2 \times 10^5 \text{ (max)}$ | $1.0 \times 10^4 \text{ (max)}$ |

Processes **2024**, 12, 323 7 of 11

4. Discussion

4.1. Activated Sludge Characteristics

The characteristics of the activated sludge were evaluated applying the microscopy methods indicated by Jenkins and colleagues [25]. Generally, the phase-contrast microscopy revealed very similar morphological features among the activated sludges sampled during the three tests. Particularly, all three bioreactors had an average floc size in the range of $150-500~\mu m$. Moreover, in all cases, the flocs appeared to be irregular but compact, with the presence of eukaryotic organisms typical of activated sludge (e.g., both swimming and stalked ciliates, nematodes, tardigrades, or rotifers). The bacterial filaments/floc ratio was also monitored, always resulting in the range of 2-5, as normally expected. The whole of these observations indicated that all sludges had a general state of good health.

The relatively lower DO concentration of test B (still well above the normal threshold recommended for aerobic activated sludge bioreactors) with respect to the other two tests may have depended on the higher average MLSS concentration observed, considering that all three plants had the same air flow rates. The SVI₃₀ values revealed a higher settling capacity of the sludge of test C, followed by those of tests A and B. Moreover, the SVI₃₀ average value of test B was twice that of test C, while the average value of test A was more or less halfway between tests B and C, highlighting some differences in the physical properties of the three sludges. Figure 3 showed that test C faced several stops for mesh cleaning with removal of the DM from the mesh, its recovery, and re-entry of the sludge cake material in the suspended activated sludge. This may have affected the sludge settling ability. Similarly, also in Figure 3, test A showed the loss of part of the suspended biomass in the permeate during the first weeks, possibly influencing the sludge settling. The SVI₃₀ can also give indications about the possible bulking phenomenon due to the presence of filamentous bacteria, about the sludge density, and about the presence of sticky substances in the supernatant of the mixed liquor [26–28]. Nevertheless, the bulking threshold was not exceeded in any test, and the physiological features of all three sludges appeared similar, as already described above. Therefore, further functional investigations of the effects of activated sludge imbalances or disturbances on the settling ability, such as those shown in the described tests, are suggested for future research.

4.2. Permeate Quality in the Different SFD MBR Tests

The 20 µm SFD MBR tests produced permeates with very low turbidity values (Figure 3). Besides turbidity, the lower quality of the permeate of test A (50 μm) was also confirmed by other parameters. Lower solid retention was clearly revealed by the permeate TSS value, which was on average one order of magnitude higher with respect to tests B and C, but also by the permeate COD and TN average values, which were three and two times higher than the other two tests, respectively. This suggested that under the applied operating conditions, the 50 µm mesh had lower efficiency in supporting the cake layer than the 20 μm mesh, as shown in the other two tests. The aerobic conditions ensured very good nitrification for all SFD MBR tests, considering the average values of ammonium, nitrite, and nitrate in the permeates. The average ammonium value in the permeate of test A was affected by a high punctual value of 6.6 mgN L^{-1} on day 7, when a peak in solids in the permeate was also detected. In the rest of test A, the ammonium measured in the permeate was always less than 1 mgN L⁻¹. In terms of nitrite content, the permeate of test C had an average value of nitrite equal to 1.6 mgN L⁻¹, while under the applied aerobic conditions, it was expected to be almost null (Table 3). This could be explained by the low MLVSS concentration, which represents an estimate of the active bacterial content [29], and the relatively high concentration of ammonium in the feeding wastewater (Table 2). These results suggested that ammonium oxidation may have tended toward its maximum rate, which was higher than the maximum rate of nitrite oxidation, leading to a slight residual nitrite accumulation [30].

The total coliforms and *E. coli* assays in all SFD MBR permeates showed the relative independence of these microbiological indicators from physical determinations such as turbidity and TSS of permeates, confirming the need for further disinfection steps of the

Processes **2024**, 12, 323 8 of 11

SFD MBR permeates in case of reuse. In this sense, the use of already tested on demand UV disinfection systems could be recommended [3]. As a possible alternative, direct exposure to solar light could represent an easy and green solution [31].

4.3. Effects of the Mesh Pore Size and the AML on SFD MBR Performance

The pore size of the support material was demonstrated to play a relevant role during the initial formation phase of dynamic membranes and after cleaning of the support itself [32]. In the same paper, the authors asserted that the mesh pore size had negligible effects on the cleaning requirements and a small influence on the effluent quality under their tested conditions (overall a permanent slight air scouring), i.e., a stop for TMP increase at least once every 5 days, in their best case. In the present paper, the choice of 20 or 50 μm was demonstrated to be relevant when coupled with AML. As matter of fact, differently from "50 μ m mesh + AML", the combination "20 μ m mesh + AML" was revealed to be optimal for better permeate quality and quicker formation of a new DM after the periodic manual cleaning of the support. Cai and colleagues demonstrated that a larger pore size of the support material can cause a significant loss of biomass in the early phase of cake formation [33]. In the same paper, before the formation of the self-forming dynamic membrane (SFDM), the turbidity obtained using a 50 µm mesh could be higher than 250 NTU, similar to the turbidity peak of test A (50 µm) described herein. Adopting two other meshes with 25 and 10 µm pore sizes, the same authors obtained turbidity values lower than 40 and 10 NTU, respectively. Nevertheless, once the SFDM was formed and stable operations were achieved, no correlation between the pore size of the support material and the quality of the permeate was observed. Saleem and coworkers [34] reported that using a 50 μm pore size support net, although it contributed to improving the SFDM effluent quality in terms of turbidity values with respect to the 200 µm net, accelerated the mesh clogging, resulting in a faster TMP increase, and therefore in more frequent cleanings. Another study from Sreeda and coworkers [35] reported that the pore size of the support medium did not affect the formation of the SFDM, and that the bacterial composition of an SFDM grown on a support with a pore size of 2 mm was similar to the one observed on other much smaller pore size nets. In a previous work by Chuang and colleagues [36], the use of a 14 µm support material led to a supernatant with more than 95% of particles between 0.2 and 6.4 μ m in size, and large particles (>10 μ m) accounted for less than 1%. Nevertheless, some particles accumulated inside the pores and caused clogging. Moreover, in a work by most of the authors of the present paper [37], a continuous slight air scouring was used to control the excess DM growth. Those conditions were observed to be effective at relatively high MLSS values. In the tests described in the current manuscript, the combination of "20 µm mesh + AML" showed no TMP increases (on the contrary of Vergine and colleagues, wherein even the best experiment required at least one manual cleaning per week), suggesting good control of the excess DM growth over time and very low effluent turbidity.

There are different possible approaches to mesh cleaning in order to resume the filtration performance. As previously described, water jet rinsing proved to be effective when the sludge was sufficiently dense and in "good health" (i.e., not subject to stress conditions due to feed or operation). Nevertheless, physical mesh cleaning usually has temporary effects on the system's filtration performance, with losses of suspended solids through the mesh and a decrease in overall effluent quality during the transient phase of new DM formation [21]. When the activated sludge faces stress, it can produce bioproducts such as soluble microbial products (SMP), extracellular polymeric substances (EPS), or other classes with colloidal characteristics that could reduce the pore size, stick on the support surface, and make physical cleaning less effective. Under these conditions, water jet rinsing should be integrated with chemical cleaning. Weak acids, bases, and oxidants are typical cleaning reagents, while metal-chelating chemicals, surfactants, and formulated detergents may also be used [38]. Guan and colleagues [39] compared the cleaning effects of sodium dodecylsulfate (SDS), NaOH, and NaClO on three identical fouled modules. Their

Processes 2024, 12, 323 9 of 11

results showed that the main fouling of SFD MBR was a complex mixture of bacterial flocs and EPS, and that NaClO was the best performing reagent for SFD MBR chemical cleaning in terms of TMP, flux recovery, and total resistance reduction, successfully oxidizing both the EPS and the bacterial flocs. The authors also found that SDS and NaOH were effective in removing the EPS, but they were not effective in removing mixed fouling as well as β -polysaccharides.

The use of air sparging for mesh cleaning is well known in the literature [19]. Rezvani and colleagues [40] adopted an SFD MBR with a similar configuration as that proposed in this paper, except for the use of synthetic wastewater, a lower suction flux for long-term operation (30 L m⁻² h⁻¹), and overall continuous permeate suction until TMP reached the value of 26 kPa (equal to 260 mbar, to use the same unit of the present paper). At that moment, an air flow rate of $0.3 \text{ m}^3 \text{ h}^{-1}$ (5 L min⁻¹) was applied for 30 min to clean the mesh. This means that a total of 150 L of air was sparged for cleaning the mesh when the TMP had reached the threshold. In the present paper, an air flow rate of 42 L min⁻¹ adopted for 5 min (i.e., 210 L of air) was used for periodic preventive air cleaning. Nevertheless, the permeate quality of the best combination reported in this paper (20 µm mesh + AML) was better than the one obtained by Rezvani and co-workers in terms of filtration performance. Anyway, it must be taken into account that the same authors used synthetic wastewater, different from the real wastewater used in the present paper. As matter of fact, it is very likely that the activated sludge of Rezvani's test and those described here had different compositions, possibly influencing the quality of DM. Further investigations of the air cleaning flow rate and time for AML to optimize preventive fouling control of SFD MBR in treating real wastewater shall be conducted. Indeed, a preventive, quick, intense, and periodic air mass load for mesh cleaning coupled with the more appropriate pore size to achieve a very performant SFD MBR has not yet been optimized. The results of the present research have shown that the SFD MBR plants having mesh supports with 50 µm and 20 µm pore sizes operated under the same operating conditions, including a stable working flux of more than $70 \,\mathrm{L}\,\mathrm{m}^{-2}\,\mathrm{h}^{-1}$, had different permeate quality trends from the first days. This can be attributed to the speed of sludge cake formation and DM development. In particular, the 20 µm mesh demonstrated more efficiency in rapid DM build-up, with consequent production of permeates with turbidity always lower than 3 NTU. A second important finding was the demonstration that a periodic air mass load with a flow rate of 42.0 L_{air} min⁻¹ for 5 min every 4 h achieved and kept the stability of the system with no need of washing the mesh support on site. Test B was compared to the benchmark (test C) and confirmed the effectiveness of the combination of 20 µm with AML with respect to a conventional maintenance strategy based on simple periodic interruption of filtration (relaxation). Other studies are required to investigate the optimal air mass load flow rate and duration to achieve the best cleaning efficiency and most sustainable operation for meshes of different pore sizes in order to optimize the overall system performance.

5. Conclusions

Two different nylon meshes of 20 and 50 μm pore sizes were used as corresponding supports for the development of a biological DM in three parallel SFD MBR tests for the treatment of real municipal wastewater. In two tests, the nylon meshes had different pore sizes, but both were periodically cleaned through an AML (air mass load, i.e., large bubbles causing turbulence) with a flow rate of 42.0 L_{air} min⁻¹ for 5 min every 4 h. The third test was equipped with the 20 μ m pore size and operated with periodic interruption of filtration for DM relaxation. The SFD MBR with 20 μ m nylon mesh was revealed to be more efficient in the production of a high-quality permeate in comparison with the larger 50 μ m pores. The maintenance strategy based on an intense AML of 5 min every 4 h was effective in controlling the excessive build-up of the cake layer and maintaining a relatively stable DM. On the other hand, relaxation during the maintenance breaks was not very efficient in controlling the excessive DM growth under the experimental conditions tested. Optimization of the AML in terms of flow rate and time will require further

Processes 2024, 12, 323 10 of 11

investigation, also depending on the mesh pore size adopted, the sludge characteristics, and the operating conditions. Nevertheless, the present results confirm the sustainability and effectiveness of the approach proposed for long-term operation of SFD MBR for municipal wastewater treatment.

Author Contributions: Conceptualization, C.S.; methodology, C.S., F.C. and A.P.; software, M.T.; validation, A.D., B.H. and A.B.; formal analysis, G.B. and F.C.; investigation, S.B. and G.B; resources, A.P.; data curation G.B., F.C. and M.T.; writing—original draft preparation, S.B. and C.S.; writing—review and editing, S.B., C.S. and A.P.; visualization, A.D., B.H. and A.B.; supervision, A.P.; project administration, A.P.; funding acquisition, A.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research was partially supported by the EU-India project "Pavitra Ganga" funded by the EC with contract n. 821051, call H2020 SC5-12-2018.

Data Availability Statement: The data presented in this study are available upon request from the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

References

- 1. Friha, I.; Karray, F.; Feki, F.; Jlaiel, L.; Sayadi, S. Treatment of cosmetic industry wastewater by submerged membrane bioreactor with consideration of microbial community dynamics. *Int. Biodeterior. Biodegrad.* **2014**, *88*, 125–133. [CrossRef]
- 2. Hoinkis, J.; Gukelberger, E.; Atiye, T.; Galiano, F.; Figoli, A.; Gabriele, B.; Mancuso, R.; Mamo, J.; Clough, S.; Hoevenaars, K. Membrane Bioreactor (MBR) Treated Domestic Wastewater for Reuse in a Recirculating Aquaculture System (RAS). In Water-Energy-Nexus in the Ecological Transition: Natural-Based Solutions, Advanced Technologies and Best Practices for Environmental Sustainability; Naddeo, V., Choo, K.-H., Ksibi, M., Eds.; Advances in Science, Technology & Innovation; Springer International Publishing: Cham, Switzerland, 2022; pp. 153–155. [CrossRef]
- 3. Vergine, P.; Amalfitano, S.; Salerno, C.; Berardi, G.; Pollice, A. Reuse of ultrafiltered effluents for crop irrigation: On-site flow cytometry unveiled microbial removal patterns across a full-scale tertiary treatment. *Sci. Total Environ.* **2020**, 718, 137298. [CrossRef] [PubMed]
- 4. Asif, M.B.; Zhang, Z.; Vu, M.T.; Mohammed, J.A.H.; Pathak, N.; Nghiem, L.D.; Nguyen, L.N. Membrane Bioreactor for Wastewater Treatment: Current Status, Novel Configurations and Cost Analysis. In *Cost-Efficient Wastewater Treatment Technologies: Engineered Systems*; Nasr, M., Negm, A.M., Eds.; The Handbook of Environmental Chemistry; Springer International Publishing: Cham, Switzerland, 2023; pp. 147–167. [CrossRef]
- 5. Judd, S.J.; Le-Clech, P.; Taha, T.; Cui, Z.F. Theoretical and experimental representation of a submerged membrane bio-reactor system. *Membr. Technol.* **2001**, 2001, 4–9. [CrossRef]
- 6. Wu, Y.; Huang, X.; Wen, X.; Chen, F. Function of dynamic membrane in self-forming dynamic membrane coupled bioreactor. *Water Sci. Technol.* **2005**, *51*, 107–114. [CrossRef] [PubMed]
- 7. Borea, L.; Castrogiovanni, F.; Ferro, G.; Hasan, S.W.; Belgiorno, V.; Naddeo, V. Hydrogen Production in Electro Membrane Bioreactors. In *Frontiers in Water-Energy-Nexus—Nature-Based Solutions, Advanced Technologies and Best Practices for Environmental Sustainability*; Naddeo, V., Balakrishnan, M., Choo, K.-H., Eds.; Advances in Science, Technology & Innovation; Springer International Publishing: Cham, Switzerland, 2020; pp. 85–87.
- 8. Hasan, S.W.; Elektorowicz, M.; Oleszkiewicz, J.A. Correlations between trans-membrane pressure (TMP) and sludge properties in submerged membrane electro-bioreactor (SMEBR) and conventional membrane bioreactor (MBR). *Bioresour. Technol.* **2012**, 120, 199–205. [CrossRef] [PubMed]
- 9. Meng, F.; Chae, S.-R.; Drews, A.; Kraume, M.; Shin, H.-S.; Yang, F. Recent advances in membrane bioreactors (MBRs): Membrane fouling and membrane material. *Water Res.* **2009**, *43*, 1489–1512. [CrossRef]
- 10. Le-Clech, P.; Chen, V.; Fane, T.A.G. Fouling in membrane bioreactors used in wastewater treatment. *J. Membr. Sci.* **2006**, 284, 17–53. [CrossRef]
- 11. Judd, S.; Judd, C. (Eds.) The MBR Book, 2nd ed.; Butterworth-Heinemann: Oxford, UK, 2011. [CrossRef]
- 12. Field, R.W.; Pearce, G.K. Critical, sustainable and threshold fluxes for membrane filtration with water industry applications. *Adv. Colloid Interface Sci.* **2011**, *164*, 38–44. [CrossRef]
- 13. Stoller, M.; Bravi, M.; Chianese, A. Threshold flux measurements of a nanofiltration membrane module by critical flux data conversion. *Desalination* **2013**, 315, 142–148. [CrossRef]
- 14. Xie, W.; Li, J.; Sun, F.; Dong, W.; Dong, Z. Strategy study of critical flux/threshold flux on alleviating protein fouling of PVDF-TiO₂ modified membrane. *J. Environ. Chem. Eng.* **2021**, *9*, 106148. [CrossRef]
- 15. Wei, C.-H.; Huang, X.; Ben Aim, R.; Yamamoto, K.; Amy, G. Critical flux and chemical cleaning-in-place during the long-term operation of a pilot-scale submerged membrane bioreactor for municipal wastewater treatment. *Water Res.* **2011**, *45*, 863–871. [CrossRef]

Processes **2024**, 12, 323

16. Moattari, R.M.; Mohammadi, T.; Rajabzadeh, S.; Dabiryan, H.; Matsuyama, H. Reinforced hollow fiber membranes: A comprehensive review. *J. Taiwan Inst. Chem. Eng.* **2021**, 122, 284–310. [CrossRef]

- 17. Mohan, S.M.; Nagalakshmi, S. A review on aerobic self-forming dynamic membrane bioreactor: Formation, performance, fouling and cleaning. *J. Water Process Eng.* **2020**, *37*, 101541. [CrossRef]
- 18. Xiao, T.; Zhu, Z.; Li, L.; Shi, J.; Li, Z.; Zuo, X. Membrane fouling and cleaning strategies in microfiltration/ultrafiltration and dynamic membrane. Sep. Purif. Technol. 2023, 318, 123977. [CrossRef]
- 19. Ersahin, M.E.; Ozgun, H.; Dereli, R.K.; Ozturk, I.; Roest, K.; Van Lier, J.B. A review on dynamic membrane filtration: Materials, applications and future perspectives. *Bioresour. Technol.* **2012**, 122, 196–206. [CrossRef] [PubMed]
- Salerno, C.; Berardi, G.; Casale, B.; Pollice, A. Comparison of fine bubble scouring, backwash, and mass air load supply for dynamic membrane maintenance and steady operation in SFD MBR for wastewater treatment. J. Water Process Eng. 2023, 53, 103846. [CrossRef]
- 21. How, S.W.; Kang, C.; Min, S.; Carrera, P.; Siddiqui, M.A.; Chen, G.; Wu, D. 13 Self-Forming Dynamic Membrane BioReactors (SFDMBRs) for wastewater treatment. In *Current Developments in Biotechnology and Bioengineering: Membrane Technology for Sustainable Water and Energy Management*; Bui, X.-T., Guo, W., Chiemchaisri, C., Pandey, A., Eds.; Elsevier: Amsterdam, The Netherlands, 2023; pp. 293–311. [CrossRef]
- 22. American Public Health Association (APHA); American Water Works Association (AWWA); Water Environment Federation (WEF). Standard Methods for the Examination of Water and Wastewater, 24th ed.; Lipps, W., Braun-Howland, E., Baxter, T., Eds.; APHA Press: Washington, DC, USA, 2023; Available online: https://www.standardmethods.org/ (accessed on 16 November 2023).
- 23. Kim, Y.; Yeom, H.; Choi, S.; Bae, H.; Kim, C. Sludge settleability detection using automated SV30 measurement and comparisons of feature extraction methods. *Korean J. Chem. Eng.* **2010**, 27, 886–892. [CrossRef]
- 24. Han, H.; Wu, X.; Ge, L.; Qiao, J. A sludge volume index (SVI) model based on the multivariate local quadratic polynomial regression method. *Chin. J. Chem. Eng.* **2018**, *26*, 1071–1077. [CrossRef]
- 25. Jenkins, D.; Richard, M.G.; Daigger, G.T. Manual on the Causes and Control of Activated Sludge Bulking, Foaming, and Other Solids Separation Problems, 3rd ed.; CRC Press: Boca Raton, FL, USA, 2003. [CrossRef]
- 26. Chen, X.; Kong, F.; Fu, Y.; Si, C.; Fatehi, P. Improvements on activated sludge settling and flocculation using biomass-based fly ash as activator. *Sci. Rep.* **2019**, *9*, 14590. [CrossRef]
- 27. Maltos, R.A.; Holloway, R.W.; Cath, T.Y. Enhancement of activated sludge wastewater treatment with hydraulic selection. *Sep. Purif. Technol.* **2020**, 250, 117214. [CrossRef]
- 28. Nittami, T.; Batinovic, S. Recent advances in understanding the ecology of the filamentous bacteria responsible for activated sludge bulking. *Lett. Appl. Microbiol.* **2022**, *75*, 759–775. [CrossRef]
- 29. Gerardi, M.H. Appendix I: F/M, HRT, MCRT, MLVSS, Sludge Age, SVI. In Settleability Problems and Loss of Solids in the Activated Sludge Process; John Wiley & Sons, Ltd.: Hoboken, NJ, USA, 2002; pp. 153–156. [CrossRef]
- Zhao, W.; Bi, X.; Bai, M.; Wang, Y. Research advances of ammonia oxidation microorganisms in wastewater: Metabolic characteristics, microbial community, influencing factors and process applications. *Bioprocess Biosyst. Eng.* 2023, 46, 621–633. [CrossRef]
- 31. Vivar, M.; Fuentes, M.; Torres, J.; Rodrigo, M.J. Solar disinfection as a direct tertiary treatment of a wastewater plant using a photochemical-photovoltaic hybrid system. *J. Water Process Eng.* **2021**, 42, 102196. [CrossRef]
- 32. Vergine, P.; Salerno, C.; Casale, B.; Berardi, G.; Pollice, A. Role of Mesh Pore Size in Dynamic Membrane Bioreactors. *Int. J. Environ. Res. Public. Health* **2021**, *18*, 1472. [CrossRef]
- 33. Cai, D.; Huang, J.; Liu, G.; Li, M.; Yu, Y.; Meng, F. Effect of support material pore size on the filtration behavior of dynamic membrane bioreactor. *Bioresour. Technol.* **2018**, 255, 359–363. [CrossRef]
- 34. Saleem, M.; Masut, E.; Spagni, A.; Lavagnolo, M.C. Exploring dynamic membrane as an alternative for conventional membrane for the treatment of old landfill leachate. *J. Environ. Manag.* **2019**, 246, 658–667. [CrossRef]
- 35. Sreeda, P.; Sathya, A.B.; Sivasubramanian, V. Novel application of high-density polyethylene mesh as self-forming dynamic membrane integrated into a bioreactor for wastewater treatment. *Environ. Technol.* **2018**, *39*, 51–58. [CrossRef]
- 36. Chuang, S.-H.; Lin, P.-K.; Chang, W.-C. Dynamic fouling behaviors of submerged nonwoven bioreactor for filtration of activated sludge with different SRT. *Bioresour. Technol.* **2011**, 102, 7768–7776. [CrossRef] [PubMed]
- 37. Vergine, P.; Salerno, C.; Berardi, G.; Pollice, A. Self-Forming Dynamic Membrane BioReactors (SFD MBR) for municipal wastewater treatment: Relevance of solids retention time and biological process stability. *Sep. Purif. Technol.* **2021**, 255, 117735. [CrossRef]
- 38. Wang, Z.; Ma, J.; Tang, C.Y.; Kimura, K.; Wang, Q.; Han, X. Membrane cleaning in membrane bioreactors: A review. *Membr. Clean. Membr. Bioreact. Rev.* **2014**, 468, 276–307. [CrossRef]
- 39. Guan, D.; Dai, J.; Ahmar Siddiqui, M.; Chen, G. Comparison of different chemical cleaning reagents on fouling recovery in a Self-Forming dynamic membrane bioreactor (SFDMBR). *Sep. Purif. Technol.* **2018**, 206, 158–165. [CrossRef]
- 40. Rezvani, F.; Mehrnia, M.R.; Poostchi, A.A. Optimal operating strategies of SFDM formation for MBR application. *Sep. Purif. Technol.* **2014**, 124, 124–133. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.