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Volatile fatty acids from sewage sludge by anaerobic membrane bioreactors: Lesson learned from two-year experiments with fouling analysis by the resistance in series model

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ABSTRACT

Volatile fatty acid (VFA) production from sewage sludge has become one of the main biotechnologies implemented in view of the circular economy application in wastewater treatment plant (WWTP) management. In this study, domestic sewage sludge collected from three WWTPs over two-year experiments was subjected to acidogenic fermentation. The fermented liquid was recovered through an ultrafiltration membrane. The membrane fouling was analysed in detail by applying the resistance in series model, revealing the major role of the extra polymeric substances in the reversible fouling, accounting for 91.2 % of the total resistance. Finally, the major contribution of the carbon footprint assessment was due to the indirect emissions (1.30 kg CO2eq/m3). The study has the novelty of providing an in-depth understanding of MBR membrane fouling used for solid/liquid separation in a plant aimed at VFA recovery from sewage sludge acidogenic fermentation. Also, the carbon footprint assessment provides insights regarding the environmental impact of VFA recovery through ultrafiltration membrane.

1. Introduction

Recently, sewage sludge has become more of a resource than a waste [1,2]. Indeed, thanks to the spread of the circular economy concept, wastewater treatment plants (WWTPs) are evolving into Water Resource Recovery Facilities (WRRF). In this view, sewage sludge represents a zero-cost resource from which several valuable products can be obtained thanks to innovative biotechnologies [3,4]. Among the different recovered resources (e.g., biofuel, biopolymers, cellulose, inorganic compounds, etc.), volatile fatty acids (VFA) are of great interest due to their economic value and their role as a biochemical building block [5]. VFA are important intermediate products of the anaerobic digestion (AD) process. More precisely, VFA are produced during AD's acidogenic step, during which sugars, fatty acids and glycerol are converted into simpler molecules and ammonia is released [6].

VFA can be used as substrate for many biochemical processes such as biopolymer production, bioenergy production and biological nutrient removal [7,8]. Despite the increasing interest in these high value-added chemicals, several bottlenecks still need to be addressed regarding the VFA production yield and extraction from sewage sludge coming from civil WWTPs [9,10]. With this regard, the solid/liquid rich in VFA separation represents a challenging issue in the production process. Indeed, the anaerobically digested sludge is a stable mixture rich in biopolymers, particles, stable heterogeneous colloidal matters and extracellular polymeric substance (EPS) that are difficult to be separated from the water [11–13]. Several separations methods such as distillation, gas stripping, electrodialysis and solvent extraction have been investigated over the years. Still, their applications are limited due to their low effectiveness and high operational cost [14–18].

Regarding the VFA recovery from acidogenic fermentation, membrane-based processes are usually preferred due to the high selectivity and lower energy demand compared to the previously cited methods (e.g., distillation, gas stripping and electrodialysis) [19–21]. Among them, pressure driven processes such as micro filtration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) are often preferred due to the lack of pretreatment required and the easier membrane management which positively impact the sustainability of the entire process [22–24]. The pressure-driven membrane process also allows direct in-line VFA recovery, usually performed by anaerobic membrane bioreactors (AnMBRs) and MBRs. Wainaina et al. [25]

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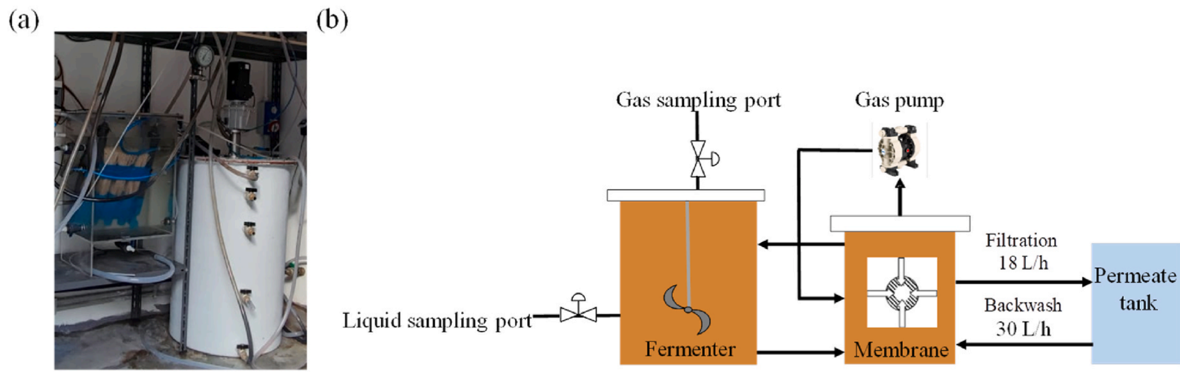

Fig. 1. Picture (a) and schematic representation (b) of the experimental set-up for the fermentation-filtration tests.

Table 1
Average values of the main parameters for UNIPA, Corleone and Marineo WWTP.

Parameters	Symbol	Unit	UNIPA Sludge (A)	Marineo Sludge (B)	Corleone Sludge (C)
Flow rate	Q	$m^3 \cdot h^{-1}$	0.48	90	154
Sludge retention time	SRT	day	27	20	33
Food to microorganism	F/M	kg BOD·kg $TSS^{-1} \cdot day^{-1}$	0.26	0.16	1.16

evaluated the VFA's continuous *in situ* recovery from food waste and sludge co-fermentation by applying an immersed MBR at high total suspended solids (TSS) concentrations. Based on the transmembrane pressure (TMP) and permeate flux, the operation was evaluated as stable for up to 42 days. Jomnonkhaow et al. [26] adopted a similar concept with different substrate fermentation reaching a stable long-term operation of 114 days. Membrane-based processes also allow the customisation of the direct in-line recovery process using chemicals that enhance the substrate hydrolysis and filterability. Longo et al. [27] evaluated the influence of wollastonite on the VFA production and recovery from sewage sludge fermentation. To apply the fermented liquid as a carbon source, a membrane based solid-liquid separation was performed by employing two tubular cross-flow UF membrane modules. The results demonstrated that the wollastonite can be considered an efficient additive to enhance both the VFA production and recovery since a 10 g/L wollastonite concentration maintained the pH above 7 thus increasing the VFA production and the filtration flux from 9.5 to 12.5 L/m^2 h. Despite the increasing spread of membrane based VFA recovery, several challenges are yet to be overcome. The substrate's several organic compounds and microbial biomass will act as fouling agents, lowering the permeate flux and reducing the membrane VFA selectivity. Despite the deep fouling analysis performed during the years regarding the AnMBRs, there is still a lack of appropriate analysis for MBR used to recover VFA from sewage sludge acidogenic fermentation. Indeed, to the authors' knowledge, this paper is the first comprehensive experimental study regarding the implementation of a hollow fiber membrane in VFA recovery from sewage sludge acidogenic fermentation providing insights on the fouling mechanism. Three different sewage sludge samples were used in this paper to identify the correlation between the membrane fouling and the sludge properties as well as the efficiency in VFA production, providing insights regarding the optimal sludge features to produce and recover VFA. A detailed fouling analysis was conducted for each fermentation test, considering the EPS content and applying the resistance in series (RIS) model. Finally, the carbon footprint was assessed for each fermentation test to compare the environmental impact of different sludges. Further studies are required for long-term analysis under full-scale applications.

2. Materials and methods

2.1. Pilot plant description

The experiments were run at the pilot plant built at the WRRF at Palermo University [28]. Fig. 1 shows the pilot plant layout comprising a fermenter, an ultra-filtration unit and a permeate storage tank. The fermenter was used as a Continuous Stirred Tank Reactor (CSTR) with a total volume of 225 L, equipped with liquid and gas sampling points at the lower side of the reactor and on the reactor's cover, respectively. Also, two probe ports are installed inside the reactor. The fermenter is connected to the ultra-filtration (UF) unit (total volume of 40 L). The unit has a hollow fibre membrane of polyvinylidene fluoride (PVDF) with 0.03-μm porosity and 1.4-m^2 surface area. A gas recycle pump (Gilian GilAir Plus, Recom *Industriale*) is also connected to the UF unit to reduce the membrane fouling while keeping the anaerobic environment. At the end of the fermentation process, the sludge was pumped (Qdos 60, Watson Marlow) to the UF unit where it was filtered with an initial flow rate of 13.2 L/h (9 min filtration at 18 L/h, 1 min backwash at 30 L/h) while the gas pump flow rate was set at 5 LPM.

2.2. Experimental design

Three different sewage sludges (i.e., A, B and C) produced from the pilot WWTP installed at the WRRF of Palermo University (UNIPA) and the civil WWTPs of Marineo and Corleone (Table 1), respectively, were used in this study. The UNIPA pilot plant considers the conventional activated sludge (CAS) process coupled with oxic settling and anoxic process (OSA) aimed at reducing the amount of sewage sludge production. On the other hand, the full-scale WWTPs of Marineo and Corleone apply the classical CAS process. The features of the sewage sludge used are reported in Table 2. For each sewage sludge, three fermentation-filtration tests were carried out. The acidogenic fermentation was run for 5 days and stopped after reaching the sCOD peak, while the filtration was carried out for 1 or 2 days and stopped once all the fermented mixture was filtrated. The fermentation tests were conducted in series by using the fermenter described above.

Table 2
Sewage sludge and fermentation test features.

Parameters	Sludge A			Sludge B			Sludge C		
	A1	A2	A3	B1	B2	B3	C1	C2	C3
F/M (kg BOD·kg SS^{-1}·day^{-1})	0.24	0.16	0.14	0.08	0.04	0.06	0.36	0.46	0.14
SRT (days)	9.62	4.80	4.80	15.26	17.52	17.86	18.38	16.16	58.17
TSS (g L^{-1})	4.56	6.45	4.70	7.96	8.05	5.33	5.51	6.03	6.67
VSS (g L^{-1})	3.71	4.76	3.29	4.78	4.7	2.65	4.13	4.31	3.8
pH	6.71	7.11	6.95	7.13	7.24	7.47	7.17	6.91	6.99
sCOD (mg L^{-1})	50.45	67.63	30.24	96.34	71.82	58.79	98.21	142.67	50.26
TCOD (mg L^{-1})	8124	7954	8257	7931	8702	7856	4201	6194	6078
T (°C)	16.17	16.10	15.87	19.47	18.73	17.93	23.33	20.63	19.07

Table 3
sCOD concentration and VFA distribution obtained from the three adopted sewage sludge.

Parameters	Sludge fermentation tests								
	A1	A2	A3	B1	B2	B3	C1	C2	C3
sCOD peak day (mg $L^{-1)}$	300.2	297.2	320.2	342.2	256.0	185.3	230.6	266.4	341. 8
VFA/sCOD peak day (%)	42	47	41	36	38	38	39	48	47
Acetic acid (%)	73.2	56.3	71.5	100	100	64.1	87.4	49.1	53.3
Propionic acid (%)	12.3	25.2	13.8	–	–	21.7	15.6	18.4	19.3
Butyric acid (%)	14.8	18.5	14.7	–	–	14.2	–	32.5	27.4

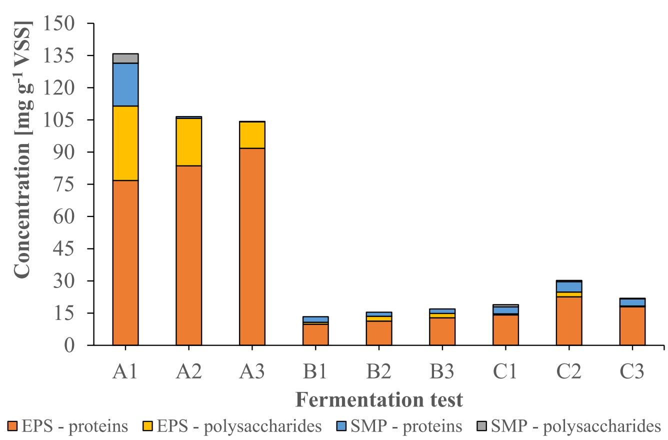

Fig. 2. EPS and SMP sludge content at the start of the filtration.

2.3. Analytical methods

The analysis of total and soluble chemical oxygen demand (TCOD and sCOD, respectively), total and volatile suspended solids (TSS, VSS), ammonium (NH_4^+- N) and phosphate (PO_4^{3-}-P) was carried out by applying the standard methods [29]. VFA were measured according to Mineo et al. [30]. Briefly, 0.45-μm filtered sludge samples were treated with 1 mL of dimethyl carbonate (DMC-OEI) and 0.1 mL of potassium bisulfate ($KHSO_4$) solution. Treated samples were centrifuged at 4000 rpm for 10 min, and the upper layer was analysed with an Agilent Technologies 7820A gas chromatograph (GC) equipped with a flame ionization detector (FID) and a DB FFAA column (30 m × 0.25 × mm × 0.25 μm). The protocol proposed by Montiel-Jarillo et al. [31] was adopted to analyse different VFA. VFA concentrations were converted into COD, expressed as mg COD/L, using the conversion factors proposed in the literature [32]. VFA production is calculated as the percentage ratio of VFA (expressed as mg COD/L) and sCOD. Finally, the COD solubilization was calculated according to Equation (1) [33]:

$$COD\ solubilization = \frac{sCOD_t - sCOD_0}{TCOD_0} \quad [1]$$

where $sCOD_t$ and $sCOD_0$ are the soluble COD concentrations at the final (t) and initial (0) fermentation time, respectively.

2.4. Membrane fouling

During the filtration, the transmembrane pressure (TMP) was monitored using a vacuum gauge during filtration and backwashing. The net permeate volume was measured to calculate the total membrane resistance (R_{T1}) defined as the ratio between the average TMP and the average permeate flux (J) multiplied by the permeate viscosity (μ) [34] (Equation (2)).

$$R_{T1} = \frac{TMP}{J \bullet \mu} \quad [2]$$

The resistance in series (RIS) model has been used to assess the type of membrane fouling by applying the protocol described by Di Bella et al. [35]. Based on the RIS model R_{T1} can also be expressed according to Equation (3).

$$R_{T1} = \frac{TMP}{J \bullet \mu} = R_m + R_{PB} + R_{C,rev} + R_{C,irr} \quad [3]$$

where R_m is the intrinsic membrane resistance, R_{PB} is the resistance due to pore blocking, $R_{C,rev}$ is the resistance due to reversible cake deposition and $R_{C,irr}$ is the irreversible cake resistance.

In view of applying the RIS model, after measuring the R_{T1}, the membrane was removed from the reactor and physically cleaned with water. After the cleaning, the membrane was operated in clean water. It was subjected to a filtration cycle to assess the resistance to filtration in clean water ($R_{T,CW}$) (using the same Equation (2)). $R_{T,CW}$ represents the sum between R_m and R_{PB} (Equation (4)).

$$R_{T,CW} = R_m + R_{PB} \quad [4]$$

Subsequently, the membrane was operated again in the reactor and was subjected to a filtration cycle using mixed liquor to assess the final total resistance (R_{T2}). Since R_{T2} is measured after a physical cleaning, it does not include $R_{C,irr.}$ Consequently, R_{T2} can also be expressed according to Equation (5).

$$R_{T2} = R_m + R_{PB} + R_{C,rev} \quad [5]$$

Therefore, after applying the RIS model, all the resistance fractions can be discriminated according to equations [6–8].

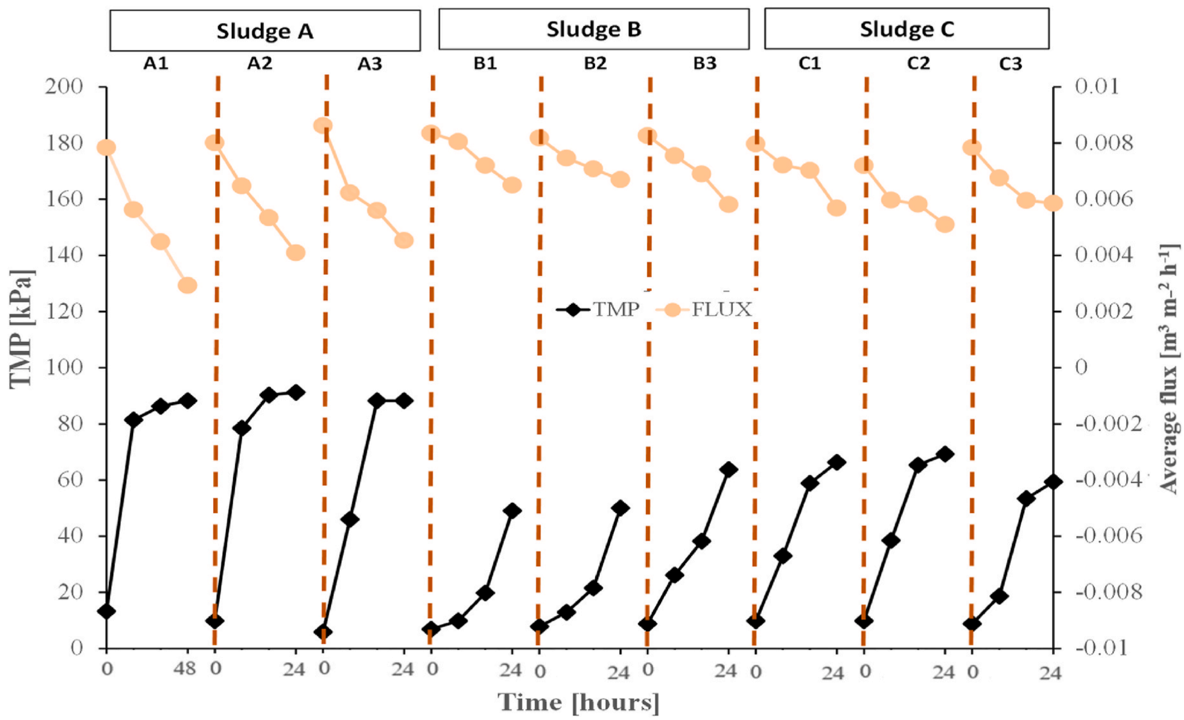

Fig. 3. Filtration monitoring for the tests.

$$R_{PB} = R_{T,CW} - R_m \quad [6]$$

$$R_{C,irr} = R_{T1} - R_{T2} \quad [7]$$

$$R_{C,rev} = R_{T2} - R_{T,CW} \quad [8]$$

2.5. Carbon footprint assessment

The carbon footprint (CF) has been quantified based on the procedure proposed by Boiocchi et al. [36]. According to Boiocchi et al. [36], CF can be assessed as the sum between direct, indirect and derivative emissions. Direct emissions (DE) are related to the greenhouse gases (GHGs) produced during biological processes. Indirect emissions (IE) are due to energy consumption and sewage sludge management. Derivative emissions (DerE) are due to the effluent contaminant loads.

Since we are dealing with anaerobic acidogenic fermentation here, the concentration of the greenhouse gases with the highest global warming potential (such as methane or nitrous oxide) can be considered negligible in off-gas. Thus, one can dismiss the DE contribution in the CF calculation. Analogously, the DerE have also been neglected since the fermented liquid is adopted as a feedstock for producing polyhydroxyalkanoates. Therefore, only the IE contribution has been considered in the CE calculation.

IE ($kgCO_{2eq}/d$) was calculated as the sum of equivalent CO_2 related to energy consumption ($CO_{2eq,En}$) (Equation (9)) and to the sludge management and disposal ($CO_{2eq,Sludge}$) (Equation (10)).

$$CO_{2eq,EN} = E_n \bullet FC_{En} \quad [9]$$

where E_n [kWh/d] represents the daily energy consumption and FC_{En} [$kgCO_{2eq}$/kWh] is the conversion factor of the energy (equal to 0.252 $kgCO_{2eq}$/kWh according to EEA, 2016). The E_n [kWh/d] has been evaluated by multiplying the operating time of each piece of equipment by its absorbed power.

$$CO_{2eq,Sludge} = M_{Sludge} \bullet FC_{Sludge} \quad [10]$$

where M_{sludge} [ton/day] is the mass of wasted sludge per day and FC_{Sludge} [$kgCO_{2eq}$/ton] is the emission factor connected with sludge management (714.74 $kgCO_{2eq}$/ton according to Zhao et al. [37]).

3. Results and discussion

3.1. Sewage sludge acidogenic fermentation

The sewage sludge acidogenic fermentation was carried out 9 times with three different sludges. Each fermentation test was conducted without performing sludge pre-treatment, chemical addition, pH, or temperature controlsince enhancing VFA production was not the goal of this study. This allowed to perform a comprehensive study on the fouling mechanism of AnMBR and correlate it with sludge properties.

Table 3 summarises the sCOD, VFA/sCOD ratio, and VFA composition measurements performed on the sCOD production peak day. Sludge A showed the best performance, both for sCOD (305.87 ± 12.54 mg L^{-1}) and VFA production (43.3 ± 3.2 %), while sludge B and C achieved 261.18 ± 78.53 mg L^{-1} and 37.3 ± 1.1 %, 279.58 ± 56.75 mg L^{-1} and 44.7 ± 4.9 %, respectively. Sludge A was the most performant despite having a low food to microorganism (F/M) ratio compared to sludge C (Table 2). This result might be explained by the low SRT and high VSS/TSS ratio of sludge A compared to the others [38]. Sludge's A performance is considerably lower than other fermentation tests presented in the literature, with no pre-treatment performed. Zhang et al. [39] evaluated the influence of urea's implementation during sewage sludge fermentation. The highest VFA concentration of 5500 mg COD/L was reached with only 0.2 g urea/g TSS. This result is reported to be due to the enhancement of sewage sludge decomposition, enhanced by the urea addition, which increased the bioavailable organic matter and the metabolic activities. Still, even without pre-treatment, control sludge achieved 1092 mg COD/L of VFA in 6 days, a concentration almost 10 folds higher than the one reported in this work. This result has to be correlated to the sewage sludge's properties, since Zhang et al. used concentrated sludge to perform the fermentation. Indeed, average TSS concentration accounted for around 22 g/L with a TCOD of around 21 g/L, values 5 and 3 folds higher than the one reported in this study, respectively. Despite appearing lower compared to other literature

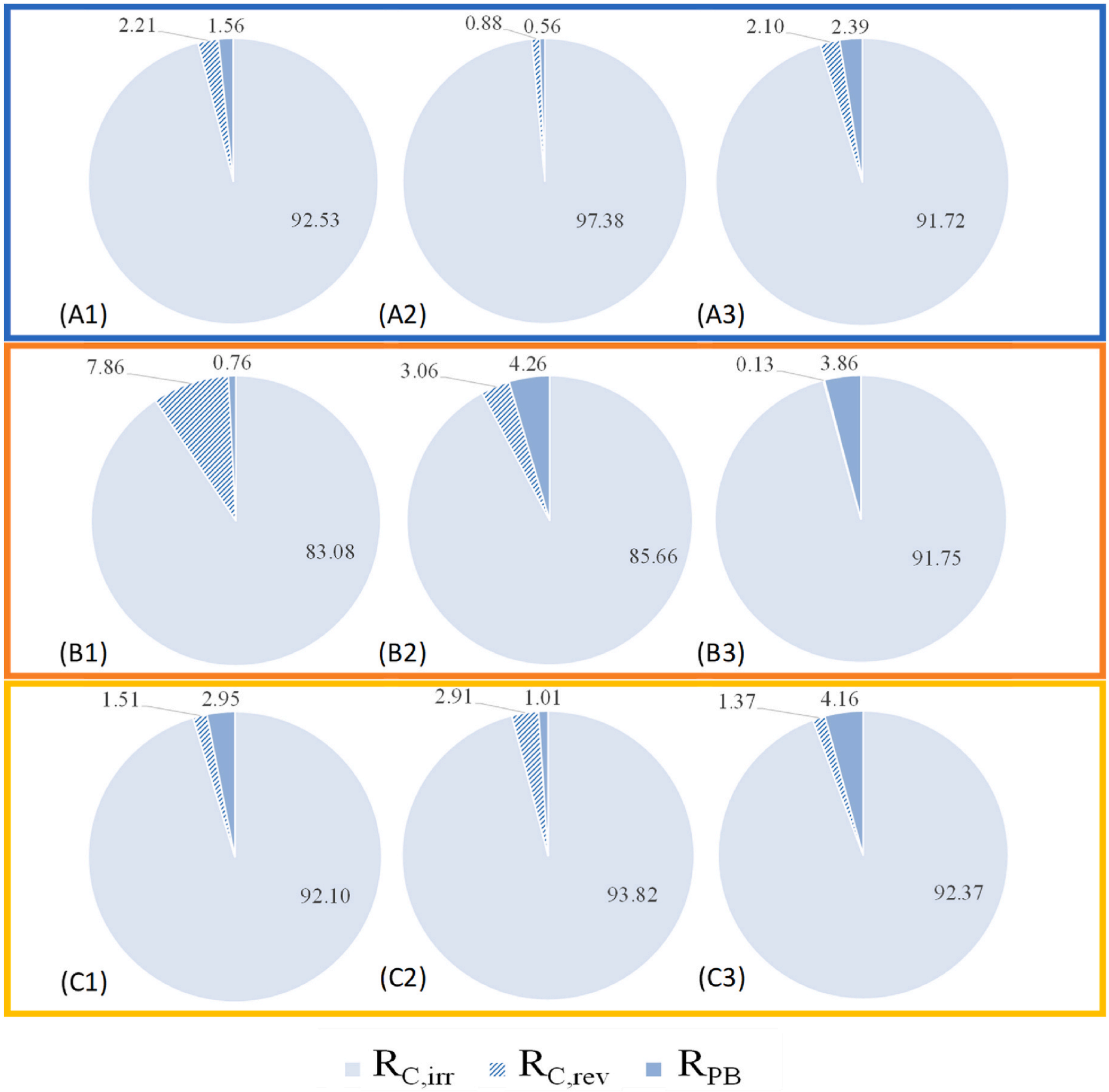

Fig. 4. Percentage of $R_{C,irr}$, $R_{C,rev}$ and R_{PB} for sewage sludge type A – test 1–3 (a1-a3), type B – test 1–3 (b1-b3) and type C – test 1–3 (c1-c3).

studies, the VFA concentration reported in this work is comparable to those reported in literature for waste activated sludge's fermentation without pre-treatment [30,40,41]. SRT played a crucial role also in the VFA composition since high SRT enhanced the butyric acid production during fermentation tests C [42] while it did not affect the fermentation of sludge B. This latter result was likely due to sludge B features (in terms of initial sCOD, TCOD and VSS/TSS ratio), which worsened the organics solubilization [43,44]. This result is confirmed by the average COD solubilization rate, which accounted for 3.3, 2.3 and 3.3 % for sludge A, B and C, respectively.

3.2. Membrane fouling monitoring

Once the sCOD production peak was obtained, a hollow fibre membrane separated the fermented sludge from the liquid part rich in VFA. The membrane was not operated continuously because of the VFA production kinetics during the acidogenic fermentation. VFA concentration reaches the peak corresponding to the soluble COD peak, at the end of the acidogenic step in the digestion process. In view of that, the membrane was operated to obtain the highest amount of VFA possible at the sCOD peak. The adopted hollow fibre membrane did not show any differences in the separation of the different VFA. Around 95 % of the produced VFA was always recovered in the filtration step, independently of the carbon chain length. Fig. 3 shows the TMP and fluxes monitored during the filtration for all the fermentation tests. In all the cases, the increase in TMP decreased the net flux due to the membrane fouling. The average TMP were 63.96, 26.24 and 40.93 kPa, while the fluxes accounted for 5.82E-03, 7.34E-03 and 6.53E-03 m^3/m^2 h, for tests A, B and C, respectively. Tests with sludge A suffered the most severe membrane fouling, with an average flux decrease of 53 %. While, during tests with sludge B and C a decrease of 24 and 27 % for the average flux was recorded, respectively. The rapid TMP increase in tests with sludge A was attributed to the adsorption of suspended solids to the membrane surface. As shown in Fig. 2, tests with sludge A had the highest EPS and SMP concentration measured, respectively 84.02, 23.06, 6.99 and 1.43 mg g^{-1} VSS for the average EPS proteins, polysaccharides, SMP protein and polysaccharides. The results show how the EPS and SMP concentration played a pivotal role in VFA production and sludge filtration. A higher amount of EPS might be the reason which sludge achieved the highest VFA and sCOD concentration [45,46] but significantly worsened the sludge filtration at the same time [47,48].

Fig. 4 shows the RIS model application to the filtration process. The

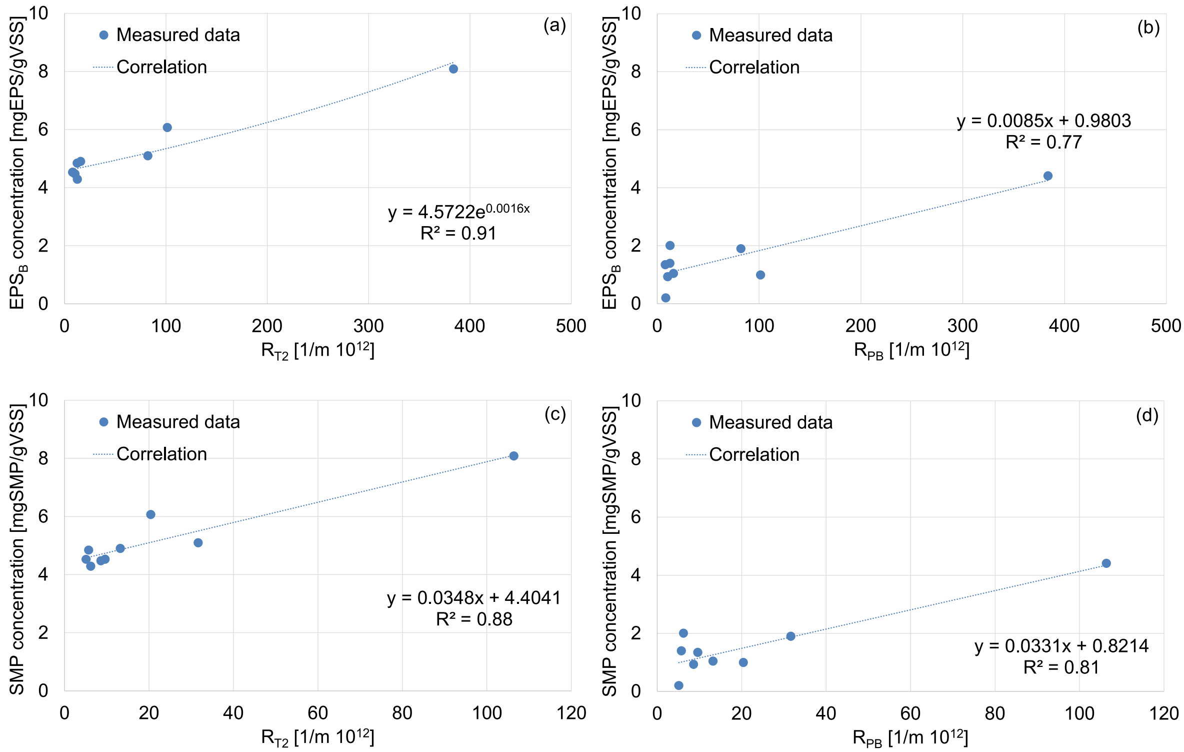

Fig. 5. Correlation between EPS_B and R_{T2} (a) and R_{PB} (b), correlation between SMP and R_{T2} (c) and R_{PB} (d).

main fouling mechanism was the irreversible cake deposition as described by the $R_{C,irr}$ values accounted for 95.34, 87.90 and 92.83 % for sludge A, B and C, respectively. Reversible cake deposition resistance, $R_{C,rev}$ was always lower than 3 % of the total resistance except B1. In contrast, resistance due to pore blocking, R_{PB}, accounted for 1.10, 3.22 and 2.71 %, showing an opposite trend compared to the irreversible cake deposition. These results show how the different sludges' properties affected the membrane fouling and confirm the differences with the AnMBR process. Zhang et al. [49] studied the long-term operation of AnMBR, focusing on the fouling mechanism. Compared to our study, the sludge Zhang et al. [49] had a lower protein to carbohydrate ratio (less than 1), which worsened the fouling mechanism [50]. Also, as reported in several AnMBR studies [51–53], the main particle size distribution is between 10 and 100 μm, which can be considered as SMP [54]. Smaller particles mainly influence the pore blocking fouling, which can cause severe damage to the membrane and require specific cleaning protocols [48,55]. Despite achieving less severe fouling, the fouling mechanism for AnMBR reported in this study was found to be comparable to anaerobic baffled biofilm-membrane bioreactor (AnBB-MBR) [56]. Buakaew et al. [56] adoperated a AnBB-MBR system with microareation for organic and nitrogen removal achieving a cake resistance always higher than 95 % of the total resistance which is comparable to the average $R_{C,irr}$ values reported in this study. This correlation is mainly devoted to the similar protein to carbohydrate ratio (higher than 1.5) reported in the studies, despite a more severe fouling was reported by Buakaew et al. because of the higher SMP concentrations. Microareation was able to mitigate the membrane fouling rate by 26 %, suggesting that the same set up could be used to mitigate the fouling also in AnMBR for VFA recovery from sewage sludge fermentation since a similar fouling mechanism was reported. This suggestion could be particularly useful when the sludge pre-treatment is performed since, as reported by Zhang et al. [39], the increase of bioavailable organic matter will lead to an increase of SMP, thus leading to a more severe fouling.

The bound EPS and SMP measured concentrations were correlated with R_{T2} (Fig. 5a and c) and R_{PB} (Fig. 5b and d) to assess which fouling mechanism is influenced by the presence of these substances. The bound EPS has a good correlation with R_{T2} ($R^2 = 0.91$) (Fig. 5a). However, the correlation between the bound EPS and R_{PB} gets worse ($R^2 = 0.77$) (Fig. 5b). This result is likely due to the relatively high dimensions of the bound EPS particles which are easily removed during backwash ($R_{C,rev}$) and physical cleaning ($R_{C,irr}$) [52,57,58]. On the other hand, SMPs provide a good correlation both with R_{T2} and R_{PB}.

By comparing the correlation results presented in Fig. 5 to other MBR studies, membrane fouling is more contained and can be easily recovered by physical cleaning [55]. The RIS model application proved that, with low R_{PB}, hollow fibre membranes are a valid solution for solid/liquid separation after the acidogenic sewage sludge fermentation.

3.3. *Carbon footprint*

The calculated CF accounted for 1.30 kg CO_{2eq}/m^3, obtained by considering the influent cubic meters needed to produce the surplus sludge used to feed the fermenter each week. DEs and DerEs were null since the layout adopted is a closed system where no gases are produced and emitted to the environment. Regarding the DerEs, the fermented sludge liquid obtained at the end of the fermentation process is used as a carbon source to enrich PHA producers' microorganisms [28]. Therefore, there is no discharge of contaminants into receiving water bodies. Compared to AnMBR systems, where the fouling mitigation and recovery are the main contributors [59,60], in MBR deputed to VFA recovery the major CF contribution are the IE. The equivalent CO_2 emission due to energy consumption accounted for 1.27 kg CO_{2eq}/m^3 while the equivalent CO_2 emission due to the wasted sludge treatment accounted for 0.03 kg CO_{2eq}/m^3.

4. Conclusions

Filtration tests were performed using a hollow fibre membrane to separate fermented sewage sludge for producing VFA. Membrane fouling was mainly attributed to the irreversible cake. Indeed, the $R_{C,irr}$ was the highest resistance fraction in all the tested cases (on average 91.16 % of the total resistance), conversely to the digested sludge treated by AnMBR. The carbon footprint assessment revealed that indirect emissions are the major contributors to MBR assisted VFA recovery (1.30 $kgCO_{2eq}/m^3$). Finally, EPS and SMP correlation with different resistances revealed that the SMP concentration mainly influenced the most severe fouling, R_{PB}. Future studies regarding the VFA production and recovery from sewage sludge should provide an in-depth analysis of the fouling mechanisms, also exploring the micromorphology of the different membranes adopted.

CRediT authorship contribution statement

Antonio Mineo: Writing – review & editing, Writing – original draft, Investigation, Formal analysis, Data curation. **Alida Cosenza:** Writing – review & editing, Writing – original draft, Formal analysis, Data curation, Conceptualization. **How Yong Ng:** Writing – review & editing, Visualization. **Giorgio Mannina:** Writing – review & editing, Visualization, Supervision, Methodology, Investigation, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

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