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Unravelling the impact of oxic-settling-anaerobic cycle implementation and solid retention time on sludge generation, membrane operation, and contaminant removal in membrane bioreactors

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ABSTRACT

Sewage sludge production represents a critical issue nowadays. The oxic-settling-anaerobic (OSA) system is a promising strategy for reducing sludge production during biological wastewater treatment. However, the distinct effect of OSA process and solid retention time (SRT) on sludge production has not been clarified yet. Therefore, during this study, the impacts of OSA system implementation at different SRT on sludge generation, membrane operation, and contaminant removal were elucidated in two lab-scale membrane bioreactors working at 20 d (MBR_{20}) and 60 d (MBR_{60}). After OSA implementation, the $MBR\text{-}OSA_{20}$ and $MBR\text{-}OSA_{60}$ were operated at a hydraulic retention time in the sludge holding tank (HRT_{SHT}) of 6–12 h and of 6–24 h, respectively. The estimated observed sludge yield (Y_{obs}) showed a reduction of 38–48 % for the $MBR\text{-}OSA_{20}$, but no beneficial effect was observed for $MBR\text{-}OSA_{60}$ at 6 h of HRT_{SHT}. At the HRT_{SHT} of 24 h, Y_{obs} decreased by 17 % due to hydrolysis and acidogenic biomass fermentation. OSA implementation and long SRT had no negative effects on organic carbon and ammonium removal (96(±3) % and 100 %, respectively) and improved total nitrogen (TN) reduction by 19–41 %. Membrane fouling and sludge filterability were not significantly affected, whereas sludge settleability worsened after the implementation of the OSA cycle. This activity provided additional insights about the distinct impact of the OSA process and SRT on sludge production and characteristics, confirming the OSA process as a valid solution for reducing sludge production under a moderate SRT, but revealing a negligible effect on Y_{obs} under a prolonged SRT and a short HRT_{SHT}.

Abbreviations: CAS, conventional activated sludge; COD, chemical oxygen demand; CST, capillary suction time; DO, dissolved oxygen; EPS, extracellular polymeric substances; HRT, hydraulic retention time; IFAS, integrated fixed film activated sludge; IR, interchange ratio; MBBR, moving bed biofilm reactor; MLSS, mixed liquor suspended solids; MLVSS, mixed liquor volatile suspended solids; N_{org}, organic nitrogen; ORP, oxidation–reduction potential; OSA, oxic settling anaerobic; P, phosphorous; PAO, polyphosphate-accumulating organisms; PN, protein; PS, polysaccharides; SBR, sequencing batch reactor; SF, supernatant filterability; SHT, sludge holding tank; SMP, soluble microbial products; SRF, specific resistance to filtration; SRT, sludge retention time; SVI, sludge volume index; T, temperature; TIN, total inorganic carbon; TMP, transmembrane pressure; TN, total nitrogen; TP, total phosphorous; TSS, total suspended solids; VFA, volatile fatty acid; VSS, volatile suspended solids; WWTP, wastewater treatment plant.

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1. Introduction

Sewage sludge represents the major waste product of a WWTP, and its management and disposal are critical for WWTP operators. The problem of the huge volumes of sludge produced annually is mainly related both to the stringent discharge limits imposed by the current regulations and to population growth [1]. Moreover, in most contexts, sludge treatment and final disposal are critical issues that require attention and targeted interventions due to stringent environmental regulations and a lack of space, highlighting the urgency of minimizing sludge production at the source, as emphasized by the European Waste Framework Directive (2008/98 CE) [2].

Nomenclature

Y_{obs}	observed cell yield coefficient (mg VSS/mg COD)
Q_{WS}	wasted sludge flow (L/d)
Q_{INF}	influent flow (L/d)
X_{WS}	wasted sludge concentration (mg TSS/L)
X_{ws}	wasted sludge concentration (mg VSS/L)
X_{MBR}	biomass concentration inside the MBR (mg TSS/L)
X_{SHT}	biomass concentration inside the SHT (mg TSS/L)
V_{MBR}	working volume of the MBR (L)
V_{SHT}	working volume of the SHT (L)
COD_{IN}, COD_{EFF}	influent and effluent COD concentration (mg/L)
R_p	Pearson's correlation coefficient

Biological wastewater treatment in WWTP is the main contributor to sludge production. Among biological processes, CAS is still the most widely used in WWTP and, thereby, the primary driver for biological sludge production. For this reason, several innovative CAS upgrading strategies have been introduced and implemented in the last decades [3]. These strategies include modifications to the layout of the biological phase aimed at modifying the bacterial metabolism to pursue lower sludge yields with limited investment and operating costs, e.g., the OSA process, and the upgrading of pre-existing CAS with new-generation biological systems (e.g., MBR, MBBR/IFAS, and granular reactors), as well as a combination of these two strategies [4].

Among the innovative bioreactors upgrading a CAS, the MBR technology accounts for several applications at real scale nowadays [5]. Indeed, by combining the biological process with membrane filtration, the MBR enables the achievement of better treatment efficiencies and works with higher concentrations of microorganisms and longer SRTs compared to CAS at the same operating volume, with beneficial effects both in terms of sludge production and footprint [3,6]. By means of the OSA cycle, the RAS is retained inside an anoxic/anaerobic SHT and, therefore, it undergoes an alternation of aerobic and anoxic/anaerobic conditions triggering the mechanisms responsible for sludge reduction, such as maintenance of metabolism, uncoupling metabolism, cell lysis, EPS destruction, and development of slow-growing bacteria [6–8]. In the last years, the OSA process has been largely tested for upgrading CAS systems and, very recently, some applications to alternative and more innovative bioreactors systems have been reported in literature [6,9–11]. In particular, Fazelipour et al. [9] and Morello et al. [10] combined the OSA process with lab-scale IFAS systems under different operating conditions, estimating a Y_{obs} of 0.14–0.31 mg TSS/mg COD and a reduction of sludge production of 32–65 % compared to the reference configurations. Fida et al. [6] and Cheng et al. [12] operated a combined MBR-OSA process focusing on the sludge IR at lab- and pilot-scale, respectively, reducing the sludge production by 6–58 % compared to the reference MBR, while de Oliveira et al. [11] estimated a decrease in sludge production of 74 % in a pilot-scale MBR-OSA system operated at a short HRT_{SHT} of 6 h.

A common finding of previous studies coupling OSA to biological systems is that applying short exposure (HRT_{SHT} < 12 h) to anoxic/anaerobic conditions (ORP_{SHT} < -100 mV) and high SRT (>20 d) are beneficial in terms of sludge reduction, sludge characteristics, and footprint limitation [7,10,13,14]. Among the operating parameters, the SRT is crucial for controlling sludge production, and it is common knowledge that the SRT increase promotes a reduction in the sludge yields [15]. In previous literature studies, the OSA process was implemented at very different SRT values ranging between 10 d and "indefinite" values (SRT > 500 d). Furthermore, a significant increase in the SRT typically occurred after the implementation of the OSA cycle. Depending on the cases, the SRT was not controlled [8–10,16] or controlled to values higher than in the reference system [17,18]. Thus, a knowledge gap exists, as it was not possible to discriminate between the impact of the OSA process and that of the SRT increase on sludge reduction as well as on sludge quality. This gap limits the full comprehension of the OSA potential towards sludge minimization, hence restraining the correct application of the OSA process in real plants. Therefore, to overcome this research gap, the target of this study was to properly elucidate the impact of OSA implementation and SRT on sludge production in a MBR system. To explore the impact of the SRT, two lab-scale MBRs were run in parallel and operated at a SRT of 20 d (MBR_{20}) and 60 d (MBR_{60}). In the second phase, the OSA configuration was implemented at a constant SRT in order to clearly evaluate the distinct impact of the OSA process on sludge production. The SHT of the $MBR\text{-}OSA_{20}$ was operated at HRT_{SHT} of 12 and 6 h, whereas the SHT of the $MBR\text{-}OSA_{60}$ was operated at HRT_{SHT} of 6 and 24 h. Besides, to better understand the influence of both SRT and OSA implementation on the mechanisms governing sludge production and membrane operation, sludge characteristics in terms of EPS content, settleability, and filterability were monitored. Indeed, the relationship between OSA process operation and sludge characteristics is still controversial in the literature, and this study also aimed to elucidate this aspect. Additionally, carbon and nutrients concentrations were monitored to evaluate the impact of the applied operational conditions on their removal, providing a comprehensive insight into the performance of the innovative configuration tested.

2. Materials and methods

2.1. Experimental design

The experimental campaign was divided into three periods, both for the MBR_{20} (I-III) and for the MBR_{60} (I'-III'). The MBR set-up was as described by Yilmaz et al. [19]. During the first period (I and I'), two lab-scale cylindrical-shaped MBRs with submerged hollow-fiber UF (0.04 μm pore diameter and 0.23 m^2 of surface area) membranes and an operating volume of 7 L were started up (Fig. S1). The MBR_{20} and the MBR_{60} were run for 28 days and 72 days, respectively. The reactors were inoculated with 70 % (v/v) recycle sludge (~8 g VSS/L) collected from a municipal WWTP located at Istanbul, Turkey. The synthetic wastewater fed to the MBRs was prepared as described by Morello et al. [10] (Table S1). The influent tank was kept at a temperature below 15 °C and at pH of 7.66 ± 0.26. The membranes were operated intermittently (5 min suction, 1 min rest) with a flux of 13–15 LMH, resulting in an instant flux of about 15–18 LMH. The MBR_{60} (period I') was operated at an average and instantaneous fluxes of 5 LMH with a continuous filtration to reduce the operational issues with intermittent membrane operation. During the study, the HRT_{MBR} was maintained at 6 h, the DO concentration at 2–5 mg/L, and temperature at 21(±2)°C. The SRT was controlled at the fixed values of 20 and 60 d trough the daily withdrawal of a calculated amount of excess sludge. The MBRs operated during period I and period I' represented the reference systems to be compared with the MBR-OSA configurations.

From the second period on, the MBR-OSA systems were implemented

Table 1
Operating conditions of the MBRs and SHTs at SRTs of 20 d (I-III) and 60 d (I'-III').

MBR (aerobic)							
Periods	**SRT (d)**	**HRT (h)**	**DO (mg/L)**	**pH**	**ORP (mV)**	**MLVSS (g VSS/L)**	**Q_R/Q_{INF}**
I	20	6	3.3 ± 0.3	7.89 ± 0.22	99 ± 24	7.0 ± 0.5	
II	20	6	3.1 ± 0.7	7.76 ± 0.15	98 ± 36	4.0 ± 0.5	0.14
III	20	6	3.4 ± 0.6	7.71 ± 0.32	85 ± 22	3.0 ± 0.5	0.28
I'	60	6	4.3 ± 1.6	7.99 ± 0.27	49 ± 21	7.3 ± 0.5	
II'	60	6	3.8 ± 1.8	7.76 ± 0.23	39 ± 14	5.9 ± 0.7	0.28
III'	60	6	4.9 ± 0.7	7.88 ± 0.37	44 ± 16	3.0 ± 0.4	0.28

SHT (anoxic)					
Periods	**HRT_{SHT} (h)**	**DO (mg/L)**	**pH**	**ORP_{SHT} (mV)**	**MLVSS (g VSS/L)**
II	12	<0.2	7.76 ± 0.16	−156 ± 89	4.9 ± 0.7
III	6(*)	0.3 ± 0.3	7.81 ± 0.15	−153 ± 47	4.3 ± 0.8
II'	6	<0.1	7.81 ± 0.32	−198 ± 82	6.3 ± 0.7
III'	24(**)	<0.1	7.83 ± 0.46	−439 ± 113	3.6 ± 0.6

(*) V_{SHT} unchanged and the RAS flow rate doubled.
(**) RAS flow rate kept constant and V_{SHT} increased from 2 to 8 L.

by recirculating the sludge through an anoxic SHT with a V_{SHT} of 2 L. Excess sludge was withdrawn from the SHT and not from the MBR. Each experimental period was characterized by the same SRT of the reference MBR combined with a different HRT_{SHT} as described in Table 1.

During the entire experimental campaign, the TMP, DO concentration, temperature, and ORP were monitored both in the MBRs and SHTs by direct measurements in the bioreactors. TSS and VSS determinations of both mixed liquor and sludge collected from the SHTs were carried out twice a week. Influent, permeate, and supernatant from both the MBRs and SHTs were sampled and analysed three times a week to monitor the concentrations of COD, TN, TP, NH_4^+, anions (NO_2^-, NO_3^-, and PO_4^{3-}), and alkalinity. In order to evaluate membrane filtration performances and sludge characteristics, the SF, SRF, nCST, SVI, and EPS/SMP were regularly monitored at the end of each experimental period (when steady state conditions were established, after a period of at least one SRT). Membrane cleaning was applied when TMP values exceeded 0.2 bar through physical and chemical methods as described by Sahinkaya et al. [20]. Chemical cleaning was applied only when physical cleaning was not sufficient as pressure jump frequency increased significantly over time.

2.2. Estimation of sludge production and SRT

Sludge production was estimated through the calculation of Y_{obs}, which was calculated as the ratio between the biomass generated (equal to the excess sludge) and the organic substrate consumed (expressed as COD) according to Yilmaz et al. [19]:

$$Y_{obs} = \frac{Q_{ws} \cdot X_{ws}}{(COD_{IN} - COD_{EFF}) \cdot Q_{INF}} \quad (1)$$

The SRT was estimated as follows:

$$SRT = \frac{V_{MBR} \cdot X_{MBR} + V_{SHT} \cdot X_{SHT}}{Q_{WS} \cdot X_{WS}} \quad (2)$$

2.3. Analytical methods

The concentrations of COD, TSS, VSS, and alkalinity as well as the SVI were measured according to Standard Methods [21]. NO_3^-, NO_2^-, and PO_4^{3-} concentrations were analyzed by ion chromatography (Dionex ICS-5000+, Thermo Fisher Scientific, USA). TN, N-NH_4^+, and TP concentrations were measured using HACH kits according to the manufacturer's instructions. N_{org} was estimated as the difference between TN and TIN concentrations. TIN was calculated as the sum of N-NH_4^+, N-NO_2^- and N-NO_3^- concentrations. P_{org} was estimated as the difference between TP and P-PO_4^{3-} concentrations. Before COD, N-NH_4^+, TN, TP, and anions determination, the samples collected were firstly centrifuged (4100 rpm for 10 min) and the supernatant filtered through 0.45 μm filter paper. pH was measured using the HACH HQ40d multimeter 190 (Hach, Italy). Temperature, DO, and ORP were measured with an inoLab Multi 9620 IDS analyzer (WTW, Germany). SRF, SF, and CST analyses were performed on the MLSS from the MBR as described by Yilmaz et al. [19]. EPS and SMP were extracted through the method reported by Le-Clech et al. [22], and the PN (EPS-PN and SMP-PN) and PS (EPS-PS and SMP-PS) fractions were evaluated spectrophotometrically by the Folin method [23] and by the phenol–sulphuric acid method [24], respectively. EPS and SMP extraction and determination were performed both on the MLSS from the MBR and the sludge from the SHT. Turbidity was measured spectrophotometrically at 600 nm on sludge supernatant collected after the settling tests used for the SVI measurements. During period III', VFA analysis was carried out once a week on the SHT supernatant via gas chromatography (GC-2014, Shimadzu).

2.4. Statistical data analysis

The statistical differences of Y_{obs} among different periods were evaluated by one-way factor analysis of variance (ANOVA) using the Data Analysis Tool of Excel 2013 (Microsoft Corporation, USA). Correlation among variables was studied by running Pearson's Test. The significant difference was set at 95 % ($p < 0.05$).

3. Results and discussion

3.1. Excess sludge production in the MBR and MBR-OSA systems

During period I, when the MBR_{20} was operated as the reference system, the MLVSS concentration was about 7 g VSS/L and decreased to 4 and 3 g VSS/L during periods II and III, respectively, when the OSA process was implemented (Table 1). On the other hand, the MBR_{60} (period I') was characterized by a MLVSS concentration of about 7.3 g VSS/L. During period II', the MLVSS concentration was about 5.9 g VSS/L, while it halved to 3 g VSS/L during period III' due to the increase of the HRT_{SHT} from 6 to 24 h which promoted VSS degradation, as discussed below. Regarding the SHTs, the MLVSS concentration was similar to that of the MBRs and followed the same trend (Table 1). The VSS/TSS ratio inside the MBRs and SHTs was in the range of 0.84–0.89 during the entire study.

In terms of sludge production, Y_{obs} was 0.236(±0.025) and 0.075 (±0.009) mg VSS/mg COD for the MBR_{20} and the MBR_{60}, respectively (Fig. 1). These results are compliant with Y_{obs} values ranging between 0.104 and 0.154 mg VSS/mg COD reported by Wang et al. [25] who reviewed the literature studies involving MBR systems used for treating

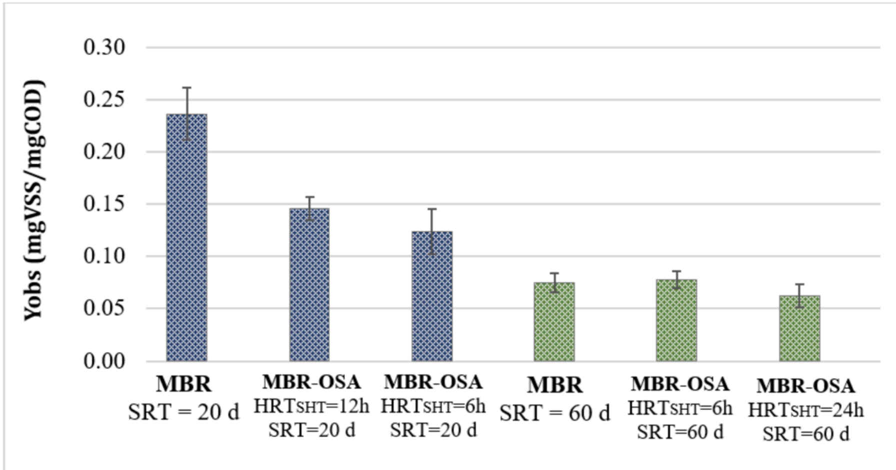

Fig. 1. Evolution of Y_{obs} during the study.

municipal wastewater. The Y_{obs} estimated during this study for the MBR_{20} confirms that the MBR alone operated under a moderate SRT could be a valid solution for implementing or completely replace a CAS for reducing biological sludge production, whose typical Y_{obs} is reported to be around 0.43 mg VSS/mg COD [15]. Indeed, the higher SRT of 20 d, compared to the typical range of a CAS (5–10 d), ensured a higher biomass concentration inside the reactor (~7 g/L), which triggered the mechanism of *maintenance and endogenous metabolism*, which reduces Y_{obs} due to the limited energy available to the microorganisms for growth and synthesis of new cells [3]. The operation of the MBR_{60} promoted a lower sludge production, as Y_{obs} was 68 % lower compared to the MBR_{20} ($p < 0.05$), confirming that manipulating the SRT has a significant effect on sludge production. The prolonged SRT of 60 d further limited the energy available for anabolic reactions, with a consequent reduction of Y_{obs}. Moreover, the growth of slow-growing bacteria with low sludge yields could be stimulated [3]. A similar result was reported by Wang et al. [26], who observed that the increase of SRT from 15 to 25 d reduced sludge production by 32 % in a lab-scale SBR fed with synthetic wastewater.

During period II, the $MBR\text{-}OSA_{20}$ was implemented and operated at HRT_{SHT} of 12 h, resulting in a Y_{obs} of 0.146(±0.011) mg VSS/mg COD. Therefore, Y_{obs} was 38 % lower than that measured for the MBR_{20}, showing that the implementation of the OSA process alone without changing the SRT reduced biological sludge production significantly ($p < 0.05$). During period III, the HRT_{SHT} was reduced to 6 h and Y_{obs} was 0.124(±0.022) mg VSS/mg COD, being about 48 % and 15 % lower than the Y_{obs} estimated during period I and period II, respectively. This result shows that the reduction of the HRT_{SHT} from 12 to 6 h contributed to further reducing sludge production ($p < 0.05$), confirming the outcomes of previous literature studies (Table 2). During periods II and III, the ORP_{SHT} was respectively −145(±110) and −153(±47) mV, showing the establishment of similar anoxic conditions, but the lower HRT_{SHT} of 6 h was more beneficial in reducing sludge production compared to the HRT_{SHT} of 12 h. This result was probably due to the more frequent alternation of oxic and anoxic conditions promoted at the HRT_{SHT} of 6 h, as the shorter HRT_{SHT} was obtained by increasing the RAS flow rate to the SHT without changing V_{SHT}. As a consequence, the high frequency of alternation between oxic and anoxic conditions induced a more severe

Table 2
OSA process in combination with innovative bioreactors: Comparison between operating conditions, sludge production and characteristics.

System scale and type	Feeding	Operating conditions	Sludge production	Sludge characteristics	Reference
Lab-scale MBR-OSA	Synthetic WW	SRT = 20 d HRT_{SHT} = 6–12 h ORP_{SHT} = −153 – −156 mV	0.124–0.146 g VSS/g COD	nCST = 18–32 s·L/g TSS SVI = 225–312 mL/g TSS	Our study
		SRT = 60 d HRT_{SHT} = 6–24 h ORP_{SHT} = −198–439 mV	0.062–0.078 g VSS/g COD	nCST = 19 s·L/g TSS SVI = 116–291 mL/g TSS	
Lab-scale MBR-OSA	Synthetic WW	SRT = n.a. HRT_{SHT} = 10.4 h ORP_{SHT}=- 250 mV	0.18 g TSS/g COD	SVI = 100 mL/g TSS	[13]
Lab-scale MBR-OSA	Synthetic WW	SRT = 10 d sludge IR = 0.11–0.22 (HRT_{SHT} = 15–17.2 d)	0.105–0.218 g MLVSS/g tCOD	SVI < 100 mL/g TSS	[6]
Pilot-scale MBR-ASSR	Real municipal WW	long SRT (*) (**) sludge IR = 0.2–1 HRT_{SHT} = 5–25 h	0.068–0.1272 g SS/g COD	n.a.	[12]
Pilot-scale MBR-ASSR	Synthetic WW	SRT > 500 d (**) HRT_{SHT} = 6 h ORP_{SHT} = −110.5 mV	0.08 g VSS/g COD	n.a.	[11]
lab-scale IFAS-OSA	Synthetic WW	SRT = 39–126 d (**) HRT_{SHT} = 6–12 h ORP_{SHT} = −91 – −229 mV	0.25–0.09 g VSS/g COD	CST = 74–155 s	[10]
pilot scale IFAS-OSA	Real municipal WW	SRT = 19–27 d (**) HRT_{SHT} = 2–4 h ORP_{SHT} = −148 mV	0.31–23 g MLSS/g COD	SVI = 49–63 mL/g TSS	[27]

n.a. = not available.
(*) specific value not reported.
(**) not controlled.

stress condition for aerobic bacteria promoting cell lysis and the occurrence of the uncoupling of metabolism, negatively impacting catabolism and, consequently, reducing bacteria aptitude to produce new biomass.

In terms of cost, upgrading an existing MBR with an OSA configuration would consists in the addition of an anoxic basin in the sludge recycle line, resulting in additional expenses for the realization and operation of both the basin and the recycle line, being not present in the typical MBR configuration. However, these additional costs would be offset by a significant reduction in sludge production (Y_{obs}), reaching up to ~ 50 % at an SRT of 20 days. This means that the costs for sludge handling and disposal can be halved compared to the reference MBR. Nowadays, the costs sustained by WWTP operators for sludge disposal can range between 100 and 400 €/tTS. Depending on the size of the WWTP and the amount of sludge produced annually, the benefits in terms of cost reduction of implementing the MBR-OSA process can be estimated for each case.

Regarding the MBR-OSA$_{60}$, Y_{obs} was 0.078(±0.008) mg VSS/mg COD (Fig. 1) during period II', similar to the Y_{obs} estimated for the MBR$_{60}$ ($p > 0.05$). This result indicates that OSA implementation had no beneficial effects on sludge production at the higher SRT of 60 d. Therefore, the first outcome of this work is that the OSA process could have a significant effect on sludge production when the MBR is operated under a moderate SRT (e.g., 20 d), but the effect of the OSA process could be anticipated to diminish or disappear as the SRT increases. In light of these results, it is possible to speculate that when the OSA process is implemented when microorganisms are already subjected to a prolonged SRT, as during some previous literature studies (Table 2), the sludge production is mainly influenced by the mechanisms promoted by the high SRT (e.g., endogenous metabolism), whereas the mechanisms triggered by the OSA process (e.g., uncoupling metabolism) may produce a limited effect.

In period II', Y_{obs} of the MBR-OSA$_{60}$ was 37 % lower ($p < 0.05$) than that of the MBR-OSA$_{20}$ with the same HRT$_{SHT}$ of 6 h (period III), which can be attributed to the higher SRT and lower ORP$_{SHT}$ of the MBR-OSA$_{60}$ (−198 ± 82 mV). In particular, the ORP$_{SHT}$ was 23 % lower than that measured for the MBR-OSA$_{20}$ during period III (Table 1), which could favor a more stressful environment for the microorganisms.

During period III', the HRT$_{SHT}$ was increased to 24 h by increasing the V$_{SHT}$ to 8 L to further reduce the ORP$_{SHT}$ and promote sludge hydrolysis. As expected, the ORP$_{SHT}$ dropped to −439(±113) mV testifying the establishment of a stricter anaerobic condition. Y_{obs} in this period was 0.062(±0.011) mg VSS/mg COD, resulting in a sludge reduction of about 17 % compared to the reference MBR$_{60}$ ($p < 0.05$). The increase of HRT$_{SHT}$ from 6 h (period II') to 24 h (period III') at the same SRT of 60 d contributed for about 17 % to the sludge reduction ($p < 0.05$) by promoting the destruction of the VSS in the anoxic tank, whose concentration decreased by 49 % compared to period II' (Table 1). Furthermore, the anaerobic exposure inside the SHT stimulated a first acidification with the consequent production of VFAs, whose composition was almost entirely represented by acetic acid with concentrations between 13 and 52 mg COD/L, the rest being propionic acid in trace amounts.

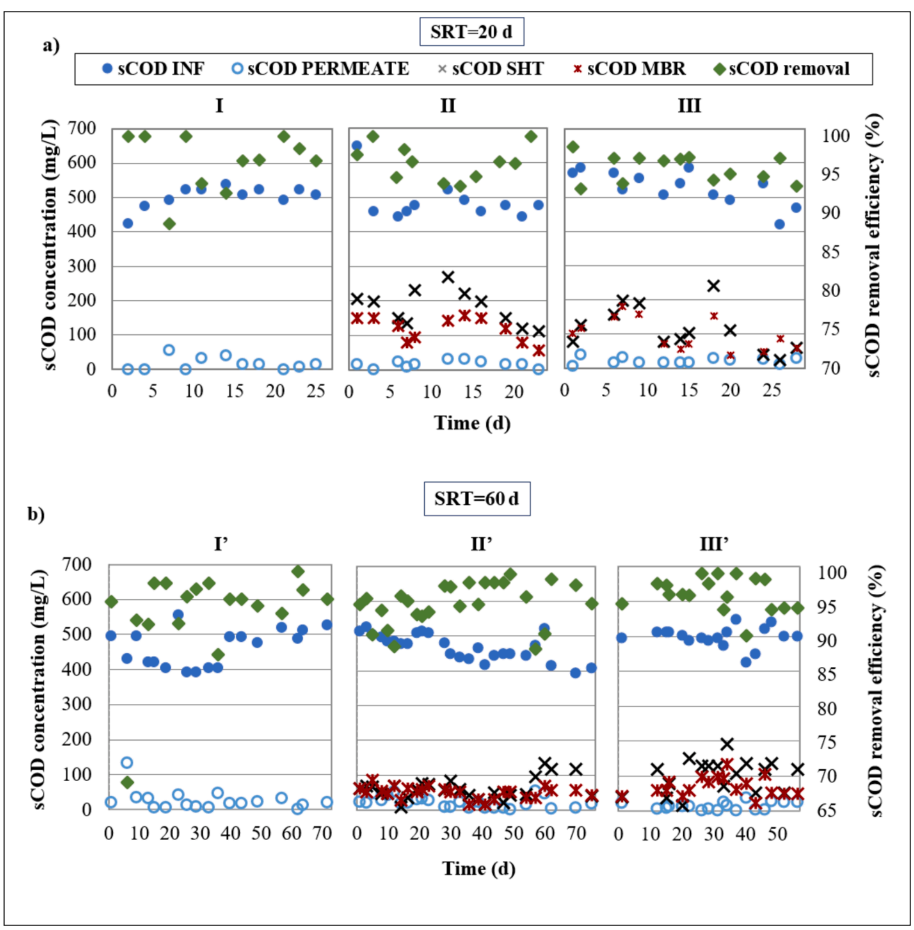

Fig. 2. Temporal profiles of sCOD concentration in the influent (sCOD INF), MBR supernatant (sCOD MBR), permeate (sCOD PERMEATE), and in the anoxic SHT supernatant (SCOD SHT) and COD removal efficiency for the systems operated at SRT of 20 d (a) and 60 d (b).

3.2. Impact on the evolution and speciation of organic carbon and nutrients

3.2.1. Organic carbon

The concentration trend of sCOD during the study is depicted in Fig. 2. sCOD removal efficiency was 95(±6)% during the MBR reference periods (I-I') (Fig. 2a) and 96(±2)% during the MBR-OSA periods (II, III, II' and III') (Fig. 2b), with effluent concentrations ranging between 10 and 55 mg/L. Therefore, both the OSA process implementation and SRT increase did not affect sCOD removal efficiency, as observed in previous studies [10,19]. Therefore, changes in the tested configuration and operating conditions did not influence the metabolic activity of heterotrophic bacteria, which were primarily responsible for the removal of organic carbon in the aerobic tank.

After implementing the OSA process, sCOD release in the SHT was observed. During MBR-OSA_{20} operation, sCOD concentration followed an increasing trend when increasing the HRT_{SHT}, as it was 96(±72) mg sCOD/L in period III (HRT_{SHT} = 6h) and 178(±57) mg sCOD/L in period II (HRT_{SHT} = 12 h (Fig. 2a). This event testifies the occurrence of cell lysis and hydrolization of part of bacterial cells as reported in previous studies [12,28,29]. In the MBR-OSA_{60}, when the HRT_{SHT} was increased from 6 h (period II') to 24 h (period III'), the sCOD level inside the SHT increased to 115(±43) mg sCOD/L due to the prolonged anaerobic exposure promoting a higher VSS destruction and sCOD release. Part of the sCOD released in the system could also be used as endogenous carbon source for nitrogen removal, as discussed in the next section.

3.2.2. Nitrogen

The concentration trend of the monitored inorganic N species is shown in Fig. 3. N-NH_4^+ concentration was always lower than 1 mg/L, resulting in complete NH_4^+ removal both during the reference MBR periods I-I' and the following MBR-OSA periods. These results show that OSA implementation did not affect nitrification efficiency throughout the study.

OSA implementation triggered a partial denitrification under the anoxic conditions inside the SHT which produced a decrease of NO_x ($NO_3^- + NO_2^-$) concentration of 43(±18)%, 67(±27)%, 90(±11)% and 95 (±7)% in period II, III, II' and III', respectively, resulting in improvement of TIN removal in the SHT (Table S2). The increasing denitrification efficiency observed during periods II, III and II' can be attributed to the enhanced carbon release due to cell lysis following the implementation of the OSA process, which was further promoted by increasing the sludge recirculation ratio for the MBR-OSA_{20} system (period III) and by the higher SRT set for the MBR-OSA_{60} system (period II') [29]. During period III', the extension of the HRT_{SHT} to 24 h at a constant sludge recirculation ratio caused the establishment of strict anaerobic conditions in the SHT (ORP = −439±(113) mV), which further promoted the hydrolysis of particulate organics originated from the lysate material [30] and also allowed a longer time for denitrification on slowly biodegradable COD. Furthermore, cell lysis occurring inside the SHT favored the hydrolysis of N_{org} (Table S2), which boosted its removal once recirculated in the MBR-OSA_{20} by 16–42 % compared to the reference MBR_{20}.

Partial denitrification and N_{org} hydrolysis inside the SHT contributed to the improvement of TN removal in the MBR-OSA system. TN removal was 16(±5)% during period I, but increased to 19(±3)% and 41(±10)% respectively in periods II and III. Therefore, TN removal at SRT of 20 d slightly improved during period II after OSA implementation at the

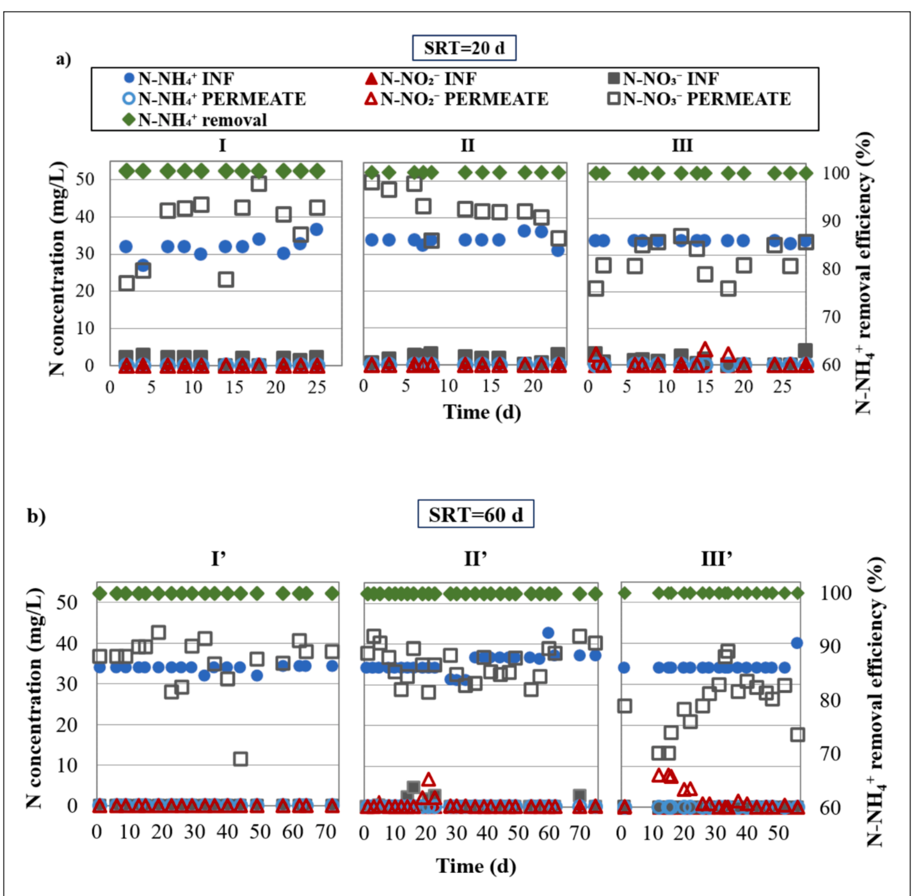

Fig. 3. Temporal profiles of N-NH_4^+, N-NO_3^-, N-NO_2^- concentrations in the MBR influent and permeate and N-NH_4^+ removal efficiency for the systems operated at SRT of 20 d (a) and 60 d (b).

HRT_{SHT} of 12 h, and improved considerably when the HRT_{SHT} was reduced to 6 h in period III due to enhanced denitrification (Fig. 3a). No significant differences in terms of TN removal were observed at the different SRT and similar HRTs (periods III and II'), nor at different HRTs when the SRT was set at 60 d (periods II' and III'). These results suggest that increasing the frequency of alternation between oxic and anoxic conditions by reducing the HRT_{SHT} seems beneficial in terms of TN removal up to a certain SRT, while this effect might diminish at higher SRT values.

3.2.3. Phosphorous

TP removal efficiency for the MBR_{20} and the MBR_{60} were respectively 62(±6)% and 65(±13)% and can be mainly attributed to P uptake due to microorganisms growth and periodical sludge withdrawal from the system. After the implementation of the OSA process, higher TP concentration were observed in the effluent and TP removal decreased to 24(±2)% and 45(±1)% in periods II and III ($MBR\text{-}OSA_{20}$) and to 38 (±4)% and 41(±8)% during periods II' and III' ($MBR\text{-}OSA_{60}$), respectively. This can be attributed to the reduced bacterial growth and diminished uptake of P following the decline of Y_{obs}. At SRT of 20 d, the lowest TP removal was achieved at HRT_{SHT} of 12 h, which indicates that prolonged exposure to anoxic conditions was detrimental for TP removal, which can be attributed to the release of P_{org} in the SHT as a consequence of cell lysis (Table S3). Indeed, P_{org} concentration was estimated to be 0 during period I (MBR only) but increased to 3(±1) and 1(±1) mg/L during periods II and III (MBR-OSA), respectively. In contrast, phosphates did not contribute to the increase of TP levels after OSA implementation, although significant P-PO_4^{3-} release was observed in the SHT (Fig. 4a) as a consequence of cell lysis and anaerobic PO_4^{3-} release from P-accumulating biomass.

In the $MBR\text{-}OSA_{60}$, TP removal during period II' was 16 % lower than the value observed in period III, which suggests that the higher SRT impacted negatively on TP removal. This result was due to the higher P-PO_4^{3-} release into the SHT observed at the SRT of 60 d, resulting much higher concentrations compared to those observed at SRT of 20 d (Table S3). Indeed, while P_{org} levels in the permeate and SHT effluent were lower at 60 d than at 20 d, the concentrations of P-PO_4^{3-} increased significantly at the higher SRT (Table S3). The observed increase of PO_4^{3-} levels indicates that prolonging the overall SRT and the HRT_{SHT}, and thus sludge exposure to the low ORP conditions in the SHT (Table 1), promotes the mechanisms of PO_4^{3-} release, including cell lysis and intracellular PO_4^{3-} release, which negatively affects TP removal. This should be considered in view of implementing a dedicated treatment for TP abatement.

3.3. Influence of the OSA process on membrane operation and sludge characteristics

3.3.1. EPS and SMP trend and composition

The trend and composition of EPS and SMP were evaluated at the end of each experimental period from the biomass collected both from the MBR and the SHT (Table 3) with the aim to elucidate their possible direct and indirect impacts on sludge production and characteristics as well as on membrane operation. In the reference MBR systems, the concentration of EPS_{TOT} was 15(±0) and 2(±0) mg EPS/g VSS for the MBR_{20} and MBR_{60}, respectively. These values are lower than the concentration of 50–60 mg EPS/g VSS reported for a lab-scale MBR run by Alizadeh Fard et al. [31] and reveal an almost negligible EPS_{TOT}

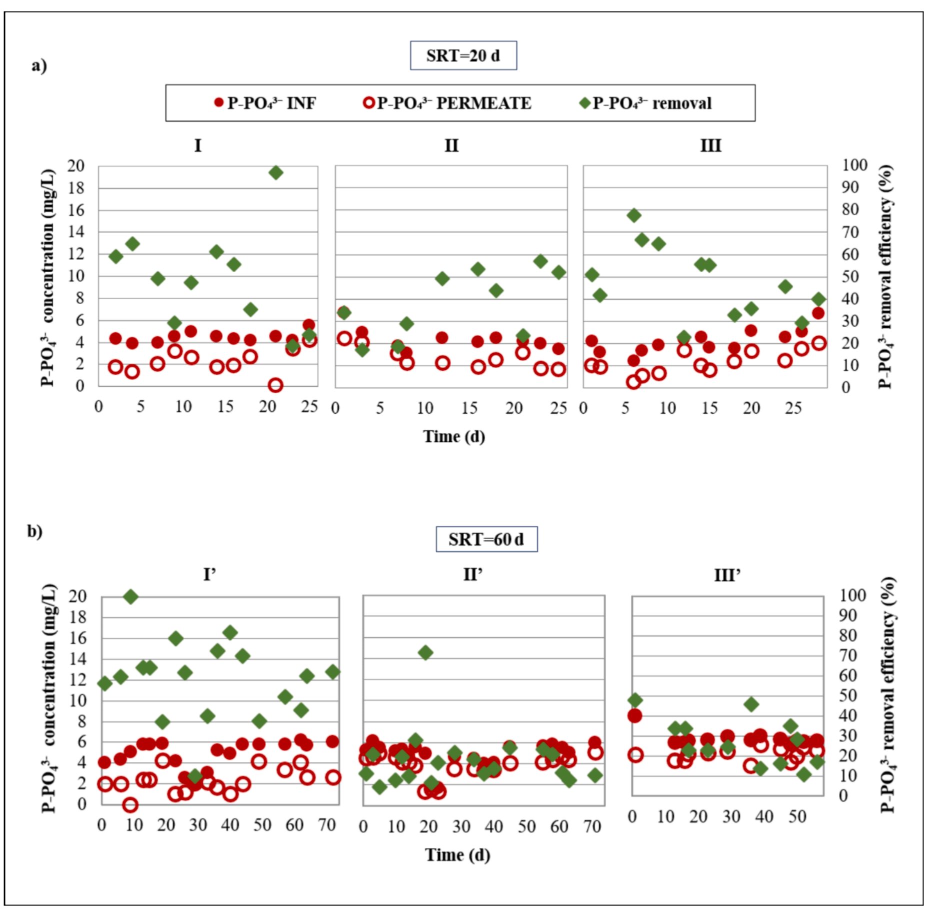

Fig. 4. Temporal profile of P-PO_4^{3-} concentration in the influent (P-PO_4^{3-} INF), MBR supernatant (P-PO_4^{3-} MBR SUPERNATANT), permeate (P-PO_4^{3-} PERMEATE), and in the anoxic SHT supernatant (P-PO_4^{3-} SHT SUPERNATANT), and P-PO_4^{3-} removal efficiency for the systems operated at SRT of 20 d (a) and 60 d (b).

Table 3
EPS and SMP concentrations inside the MBR and SHT measured at the end of each operational period.

MBR (AEROBIC)						
Periods	EPS_{PN} (mg/g VSS)	EPS_{PS} (mg/g VSS)	SMP_{PN} (mg/g VSS)	SMP_{PS} (mg/g VSS)	EPS_{TOT} (mg/g VSS)	SMP_{TOT} (mg/g VSS)
I	3 ± 0	12 ± 0	0	2 ± 0	15 ± 0	2 ± 0
II	7 ± 0	27 ± 6	3 ± 0	9 ± 1	34 ± 6	12 ± 1
III	4 ± 2	13 ± 8	0	13 ± 7	17 ± 6	13 ± 7
I'	1 ± 0	1 ± 0	0	9 ± 1	2 ± 0	9 ± 1
II'	1 ± 1	3 ± 2	0	0	4 ± 2	0
III'	3 ± 1	19 ± 2	0	5 ± 2	21 ± 2	5 ± 2
SHT (ANOXIC)						
Periods	EPS_{PN} (mg/g VSS)	EPS_{PS} (mg/g VSS)	SMP_{PN} (mg/g VSS)	SMP_{PS} (mg/g VSS)	EPS_{TOT} (mg/g VSS)	SMP_{TOT} (mg/g VSS)
II	20 ± 0.3	71 ± 9	5 ± 1	44 ± 2	90 ± 9	49 ± 2
III	4 ± 0.2	66 ± 7	2 ± 2	5 ± 0	71 ± 6	6 ± 1
II'	7 ± 1	21 ± 0	0	1 ± 0	21 ± 10	1 ± 0
III'	3 ± 1	36 ± 3	1 ± 1	12 ± 4	38 ± 3	13 ± 3

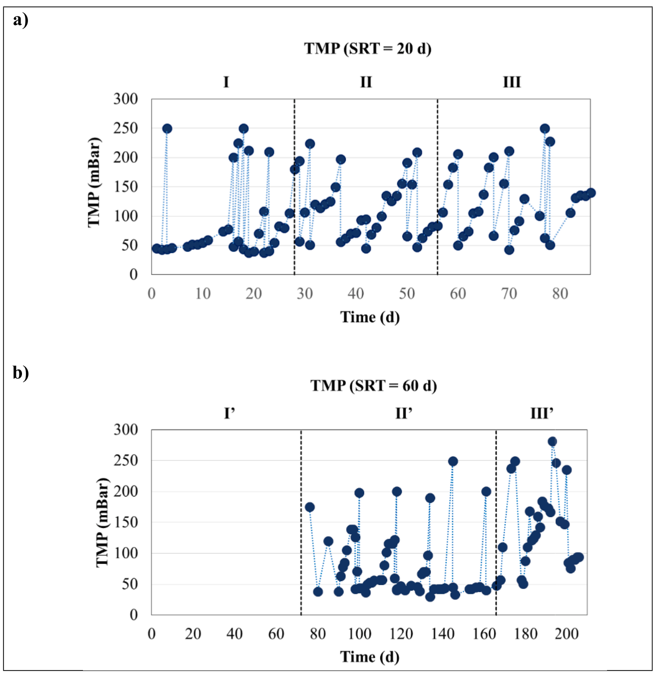

Fig. 5. TMP profile along the study at the SRT of 20 d (a) and 60 d (b).

concentration when the system was operated at the SRT of 60 d.

After OSA implementation (MBR-OSA_{20}), EPS_{TOT} concentration in the MBR increased to 34(±6) mg EPS/g VSS at the end of period II and to 17(±6) mg EPS/g VSS at the end of period III, the latter being similar to the concentration detected inside the MBR_{20} at the end of period I (Table 3). For the MBR-OSA_{60} system, EPS_{TOT} concentration in the MBR did not increase significantly in period II', being 4(±2) mg EPS/g VSS, while it increased to 21(±2) mg EPS/g VSS at the end of period III'. In the SHTs, EPS_{TOT} concentration was 90(±9) mg EPS/g VSS (period II) and 71(±6) mg EPS/g VSS (period III) at SRT of 20 d, while it was 21 (±10) mg EPS/g VSS (period II') and 38(±3) mg EPS/g VSS (period III') at SRT of 60 d (Table 3). In general, EPS_{TOT} concentration at SRT of 60 d were considerably lower than at 20 d, except when the HRT_{SHT} was increased to 24 h (period III'), which indicates that higher SRT stimulated EPS degradation. The reduction of EPS content is reported as a consequence of sludge flocs deterioration which can be preparatory for cell lysis [6,16,32], thereby contributing to the sludge reduction observed during the MBR-OSA periods. Furthermore, it can be noted that EPS_{TOT} concentrations inside the SHT were always higher (+45–81 %) than in the MBR for both systems (Table 3), indicating that EPS were produced under anoxic conditions and degraded under aerobic conditions. EPS_{TOT} concentration in the SHTs showed an increasing trend (+21–45 %) when increasing the HRT_{SHT}, indicating that longer exposure to anoxic conditions induced the proliferation of lysate material.

Regarding EPS composition, EPS_{PS} was the dominant fraction (50–86 % of EPS_{TOT}), therefore its variation impacted EPS_{TOT} trend significantly. However, both EPS_{PS} and EPS_{PN} varied consistently during the study, indicating that the operational conditions had similar impacts on both fractions (Table 3).

The concentration of SMP_{TOT} inside the SHT during the study was quite low (<13 mg SMP/mg VSS) and similar to the one detected inside the MBR (<13 mg SMP/mg VSS), except at the end of period II, as it was 49(±2) mg SMP/mg VSS. The low levels of SMP_{TOT} detected inside the SHT can be attributed to their immediate consumption as endogenous carbon source for denitrification (during periods II, III, II' and III') and PAOs activity (especially during period III') as previously discussed in Sections 3.2.2 and 3.2.3.

3.3.2. *Membrane fouling and sludge filterability*

The evolution of TMP during the study is shown in Fig. 5. TMP trend was similar for the reference MBR_{20} and the MBR-OSA systems at the different operating conditions, as previously observed by Fida et al. [6], testifying that the OSA process did not negatively affect membrane operation. As regards the MBR_{60}, TMP was close to zero as it was operated with a low flux. The frequency of chemical cleaning for the membrane in the MBR_{20} system was comparable during periods I and II (0.13 and 0.10 d^{-1}), but it increased in period III (0.25 d^{-1}). As regards

Table 4
SVI, SRF, SF and nCST, measured on the sludge from the MBR at the end of each experimental period.

Periods	SVI (mL/gTSS)	SRF (m/kg)	SF (mL/min)	nCST (s*L/g TSS)	Turbidity (abs)
I	116 ± 1	3.28E + 13	0.46	13 ± 2	0.195 ± 0.010
II	225 ± 33	3.27E + 14	0.68	18 ± 1	n.a.
III	312 ± 2	1.60E + 14	1.27	32 ± 4	0.273 ± 0.068
I'	109 ± 10	1.12E + 13	n.a.	5 ± 0	0.065 ± 0
II'	116 ± 6	7.77E + 13	1.62	19 ± 3	0.120 ± 0
III'	291 ± 7	5.81E + 13	1.24	19 ± 4	0.881 ± 0.195

n.a. = not available.

the MBR_{60}, no chemical washing was applied during period I', while during periods II' and III' chemical cleaning was applied with a frequency comparable to that of periods I-II (0.08 and 0.10 d^{-1} in periods II' and III', respectively). Taking into account also physical cleanings, the cleaning frequency of the membrane was 0.21–0.29 d^{-1} during periods I-III (SRT = 20 d) and 0.03–0.013 d^{-1} during periods I'-III' (SRT = 60 d), indicating that membrane fouling was mitigated at the higher SRT tested. This can be linked to the concentration of EPS_{TOT} and SMP in the two systems, being lower at SRT of 20 d than at 60 d (Table 3). Indeed, membrane fouling has been often associated to the accumulation of small-sized particles such as SMP and colloids on the membrane surface [19].

To understand the causes of fouling evolution during the study, as well as to investigate the effects of the OSA process on the sludge dewaterability, the SF, SRF and nCST were monitored at the end of each experimental period (Table 4). The SF in the reference MBR_{20} system (period I) was 0.46 mL/min and increased during the MBR-OSA_{20} periods to 0.68 to 1.27 mL/min in periods II and III. SF values of 1.24–1.62 mL/min were observed in the MBR-OSA_{60} system, indicating that OSA implementation had no detrimental effects on SF. The observed SF trend is consistent with the low SMP amounts detected in the MBRs during the entire study (Table 3) and may also suggests the absence of other colloidal species responsible for blocking the membrane pores. As a consequence, the TMP trend and membrane operation were not affected after OSA implementation (Fig. 5), as also indicated by the similar values of the cleaning frequency estimated with and without the OSA cycle (periods I-III).

Besides providing information about sludge filterability, the SRF and nCST offer an indication about the fouling propensity of the membrane [33]. The SRF was 3.28×10^{13} and 1.12×10^{13} m/kg for the MBR_{20} and the MBR_{60}, respectively, showing that the higher SRT had a beneficial effect on sludge filterability, as also observed by Pontoni et al. [34] and Yilmaz et al. [19]. After the implementation of the OSA process, the SRF increased (Table 4) without showing a clear relationship with the operating conditions tested ($p > 0.05$). As the SRF is reported to give an information about the cake quality and porosity [19,33,35], it could be speculated that the OSA process implementation produced a worsening of the cake permeability. However, this did not affect membrane operation and cleaning frequency. Overall, the SRF values estimated for both the MBR and MBR-OSA systems indicate limited filterability on industrial scale, being higher than 5×10^{12} m/kg (EN 14701-2:2006). However, this finding is common to several previous studies on lab-scale MBRs estimating SRF values in the range of 10^{12}-10^{16} [36,37], while lower values have been reported from full-scale evaluations [34,38].

The nCST was 13(±2) and 5(±0) s·L/g TSS for the MBR_{20} and the MBR_{60}, respectively. After implementing the OSA configuration, the nCST remained quite constant, as it was 18(±1) s·L/g TSS at the end of period II, but increased to 32(±4) s·L/g TSS at the end of period III. Similarly, the implementation of the OSA process to the MBR_{60} increased the nCST to the similar values of 19(±3) and 19(±4) s·L/g TSS at the end of periods II' and III', respectively. Overall, these results indicate that OSA implementation diminishes sludge filterability. Previous reports linked sludge filterability to EPS trend and composition to some extent [10]. In one of those studies (Table 2), the increase of nCST was associated with an increase of the EPS_{TOT} content of the sludge and to the presence of a higher fraction of EPS-PN over EPS-PS. Similarly, in this study both the nCST and the EPS_{TOT} increased after OSA implementation (Tables 3-4), which further strengthens the link between EPS concentration and sludge filterability.

3.3.3. Sludge settleability

Sludge settleability was evaluated by means of the SVI after proper settling tests, and the related results are reported in Table 4. During period I and I', the SVI was 116(±1) and 109(±10) mL/g TSS, showing that the sludge from the two reference MBRs was characterized by good settling properties (SVI ≤ 150 mL/g). After OSA implementation, SVI worsened at all the operating conditions tested. In particular, SVI increased to 225(±33) mL/g TSS after implementation of the OSA configuration to the MBR_{20} (period II), and it increased to 312(±2) mL/g TSS in period III following the reduction of the HRT_{SHT} from 12 to 6 h. As regards the MBR_{60}, SVI slightly increased to 116(±6) mL/g TSS after the implementation of the OSA process in period II' and it drastically worsened in period III', as it increased to 291(±7) mL/g TSS following the increase of HRT_{SHT} to 24 h. SVI values at the SRT of 60 d were always lower than at 20 d, indicating better sludge settleability at higher SRT. The relationship between sludge settling properties and OSA process reported in literature is controversial, as the behaviour of the SVI after OSA implementation appears to be very case-specific and not univocally affected by system scale, influent wastewater, or applied operating conditions [3]. Similar to our results, Corsino et al. [7] report a worsening of sludge settleability for a CAS-OSA system operated with a SRT of 33 d and a HRT_{SHT} of 6 h, as the SVI increased from 180 mL/g TSS of the reference CAS system to 230–770 mL/g TSS of the CAS-OSA system. In another study, Wang et al. [26] did not observe a beneficial effect of OSA implementation and SRT increase for a SBR system. Indeed, the SVI passed from 143 and 162 mL/g TSS of the reference SBR operated at SRTs of 15 d and 25 d, respectively, to a value of 157 mL/g TSS for the SBR-OSA. On the contrary, Saby et al. [13] and Romero Pareja et al. [14], who operated a MBR-OSA and a CAS-OSA (SRT = 21.7–46.1 d), respectively, with a HRT_{SHT} between 7 and 11 h, reported a SVI reduction between 31 % and 60 % after OSA implementation compared to the reference systems. They associated this result to the release of intracellular polymers inside the SHT that could have acted as "floc bridging agents". During our study, we also observed a release of EPS in the SHT, but their floc bridging potential beneficial for the sludge settleability probably failed as the EPS were degraded in the MBR under aerobic conditions. In this context, the turbidity evaluation performed in this study could provide useful information to help unravelling the impact of OSA implementation on sludge settleability (Table 4). Indeed, turbidity measured for the sludge supernatant collected after the settling test for SVI evaluation increased significantly after OSA implementation, independently from the SRT. In particular, turbidity exhibited the highest value in period III' (0.881 ± 0.195 abs) at the highest HRT_{SHT} tested of 24 h. Increase of turbidity was positively correlated with the concentration of EPS_{TOT} in the MBR ($R_p = 0.75$), but could also be the consequence of the increase in the concentration of dispersed microorganisms due to floc disruption at higher retention times in the MBR and SHT.

4. Conclusions

The clear distinction between the effect of OSA process implementation and SRT on sludge production and characteristics from a MBR was the novel target of this study. In particular, it was observed that the implementation of the OSA process to the MBR at the moderate SRT of 20 d contributed to reducing the sludge production by 38 % and 48 % at the HRT_{SHT} of 6 and 12 h, respectively. On the contrary, when the MBR was operated at the higher SRT of 60 d, no beneficial effect of OSA implementation was observed at the HRT_{SHT} of 6 h and a 17 % reduction of Y_{obs} was observed after the increase of the HRT_{SHT} from 6 to 24 h due to the establishment of strict anaerobic conditions inside the SHT ($ORP_{SHT} = -439$ mV), which improved hydrolysis and acidification of the biomass. This is a key finding of this study, as it reveals that when the biological process is operated at a prolonged SRT, the effect of the OSA process implementation on sludge production could be trivial. Consequently, the relationship between OSA process implementation and SRT increase when sludge minimization is pursued has been clearly elucidated. This new insight will represent an additional assessment factor for the OSA process future studies, in order to mindfully select the operating conditions which suit most with the application context (e.g., applied SRT) and avoid the overestimation of the effects of the OSA process on sludge production.

As regards the MBR operation, permeate quality was not negatively affected in terms of carbon and TN levels, as COD removal was around 96 % during the entire study and TN removal improved by 19–41 % during the MBR-OSA periods. On the contrary, TP removal worsened due to both OSA implementation and SRT increase, which stimulated the release of phosphate and organic-P. Sludge settleability and filterability exhibited a generalized worsening after the MBR-OSA implementation, while a positive correlation with the SRT was highlighted. However, despite the worsening of sludge characteristics, membrane fouling was not significantly affected by the OSA implementation, and it was mitigated by SRT increase. Based on these promising results, upscaling the system in future investigations could be useful for conducting an appreciable economic analysis in order to estimate the economic feasibility of this strategy on a real scale.

CRediT authorship contribution statement

Raffaele Morello: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Francesco Di Capua:** Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Conceptualization. **Cigdem Kalkan Aktan:** Methodology, Investigation, Formal analysis. **Tulay Yilmaz:** Methodology, Investigation, Formal analysis. **Giovanni Esposito:** Writing – review & editing, Supervision, Resources. **Francesco Pirozzi:** Supervision, Resources. **Umberto Fratino:** Supervision, Resources. **Danilo Spasiano:** Writing – review & editing, Supervision, Resources. **Erkan Sahinkaya:** Writing – review & editing, Supervision, Resources, Investigation, 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

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cej.2024.153800.

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