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ABSTRACT

Oxygen transfer in wastewater treatment is significantly influenced by the mixed liquor suspended solids (MLSS). The effect is more pronounced at MLSS concentrations higher than 20 g L^{-1} when supplying air by conventional diffused aeration systems. The oxygen transfer performance of a supersaturated oxygenation technology (*i.e.*, the supersaturated dissolved oxygen (SDOX) system) was evaluated in clean water and in activated sludge with MLSS concentrations from 4 to 40 g L^{-1} as a promising technology for uncapping such limitation. The evaluation was carried out at the laboratory facilities of the faculty of food technology and biotechnology at the University of Zagreb. The sludge was collected from a full-scale conventional activated sludge (CAS) wastewater treatment plant (WWTP) operated at a solid retention time (SRT) of approximately 5 days. The evaluation was carried out using a laboratory-scale setup consisting of a bench-scale SDOX system (2.75 L) supplying pure oxygen to a 5 L biological reactor. The SDOX exhibited oxygen mass transfer rate coefficient (K_La) values (2.6 h^{-1}) in clean water lower than for fine bubble diffusers (11 h^{-1}). However, higher oxygen transfer rate (OTR) values and alpha factors (mass transfer ratio of process-water to clean-water) as a function of the MLSS concentration were observed. A standard oxygen transfer efficiency (SOTE) of approximately 100 % in clean water was reported. The SDOX technology can be presented as a promising alternative for supplying dissolved oxygen (DO) into mixed liquor solutions; particularly, at the high MLSS concentrations required by high-loaded membrane bioreactor (HL-MBR) systems and aerobic digesters.

1. Introduction

Supplying dissolved oxygen (DO) into conventional activated sludge (CAS) wastewater treatment plants (WWTP) may represent up to 75 % of the total operational costs (Capodici et al., 2019; Li et al., 2017; Mannina et al., 2020). The oxygen transfer in CAS systems is influenced by the mixed liquor suspended solids (MLSS) and other substances present in the liquid phase. Particularly, this negative effect has been clearly observed and reported when utilizing conventional diffused aeration systems (*e.g.* fine and coarse bubble diffusers) (Cornel et al., 2003; Durán et al., 2016; Germain et al., 2007; Henkel et al., 2011; Muller et al., 1995; Xu et al., 2017). In particular, at MLSS concentrations higher than 20 g L^{-1}, the alpha factor (mass transfer ratio of process-water to clean-water) is severely affected (Cornel et al., 2003; Durán et al., 2016; Germain et al., 2007; Henkel et al., 2011; Kim et al., 2019; Muller et al., 1995). Beyond that MLSS concentration it is either not technically feasible (due to hardware limitations), or extremely inefficient to supply DO (because of the higher associated costs).

Membrane bioreactor (MBR) systems are arguably the most suitable technology to operate at high MLSS concentrations. Advantages of MBRs include: (i) the production of a high quality treated wastewater able to comply with strict effluent discharge standards; (ii) lower generation of digested sludge (compared to CAS systems) at longer solid retention times (SRTs); and (iii) more robust systems able to handle severe shock loads (Bagheri et al., 2019; Fortunato et al., 2018; Khouni et al., 2020; Kim et al., 2019). Moreover, if, for instance, MBRs can operate at higher than usual MLSS concentra-

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tions (*e.g.* higher than 20 g L^{-1}) the system footprint and sludge generation can be further reduced lowering the capital and operational costs (Barreto et al., 2017; Livingston, 2010). This concept of an MBR operated at high MLSS concentrations (from approximately 15–40 g L^{-1}) was introduced by Kim et al. (2019) and presented as the high-loaded MBR (HL-MBR). A HL-MBR can also bring the opportunity to design more compact and containerized mobile wastewater treatment systems that can be suitable for on-site and/or decentralized municipal and/or industrial wastewater treatment applications. In addition, such containerized HL-MBR systems can be considered as an alternative for the provision of sanitation after the occurrence of natural disasters (Barreto et al., 2017; Zakaria et al., 2015). However, Kim et al. (2019) reported that, if conventional aeration systems are used, it is neither technically nor economically feasible to operate such systems at MLSS concentrations higher than 20 g L^{-1}. Notwithstanding the intrinsic benefits offered by both conventional MBR and HL-MBR, there is a need for more efficient oxygen supply systems able to work at MLSS concentrations higher than 20 g L^{-1} in an economic manner.

Different innovative, non-conventional aeration technologies have been recently proposed to enhance the oxygen transfer process. Aeration processes such as the deep-shaft (U-Tube contactor or vertical shaft), high purity oxygen (HPO) aeration systems, and pressurized oxygenation systems (supersaturated oxygen aeration systems) have been developed to enhance the oxygen transfer rate (OTR) and oxygen transfer efficiency (OTE) (Xu et al., 2016). Most of these systems rely on increasing the amount of DO in the liquid phase by increasing the partial pressure of oxygen in the gas phase (Barber et al., 2015). Such systems can achieve high OTE values of up to 90 % (Mueller et al., 2002). The disadvantages of these technologies historically include the high capital, operation, and maintenance costs. Besides, most of them have not been tested at MLSS concentrations higher than 20 g L^{-1} (Bernat et al., 2017; Singh, 2017).

Among the non-conventional aeration systems, the supersaturated oxygen aeration systems have been developed for working with HPO and at high-pressure conditions achieving higher OTRs (Berktay and Ellis, 1997; Jin et al., 2010). The two most relevant supersaturated oxygen aeration technologies include the Speece cone technology and the supersaturated dissolved oxygen (SDOX) system. The Speece cone system appears as a promising technology for supplying DO at high MLSS concentrations in biological wastewater treatment systems. However, the oxygen transfer performance of such technology needs to be further evaluated at a high MLSS concentration range. The SDOX system is a novel alternative to supply DO into biological wastewater treatment systems. It consists of a pressurized chamber operated at a high pressure (> 800 kPa) connected to an HPO source. A stream of the mixed liquor to be oxygenated is recirculated through the pressurized chamber where it gets in contact with the HPO at the high-pressure conditions in the chamber (Gerling et al., 2014). The influent stream enters at the top of the pressurized chamber, where a large gas-liquid interface is created between the wastewater and pure oxygen. The high-pressure conditions exerted in the pressurized chamber allows reaching DO concentrations of up to 350 mg L^{-1} (Jones, 2010), higher than other technologies. OTE values higher than 95 % have been reported for the SDOX system (https://www.blueingreen.com, accessed on February 2020). However, the SDOX technology has been mostly evaluated for lake and river restoration. Thus, no information has been reported in the literature regarding the performance of the SDOX system in activated sludge mixed liquors, despite the potential advantages and benefits that this technology can offer in terms of (i) reaching highly-supersaturated DO concentrations, (ii) higher OTE, (iii) easiness to install, and (iv) relatively lower maintenance and operational needs than other aeration systems. As such, there is a need to evaluate the performance of the SDOX at higher MLSS concentrations (> 20 g L^{-1}) in biological wastewater treatment systems.

The main objective of this research was to evaluate the oxygen transfer performance of an SDOX system at a high MLSS concentration range of 4–40 g L^{-1}. The oxygen mass transfer rate coefficient (K_La), the maximum standard oxygen transfer rate (SOTR), the standard oxygen transfer efficiency (SOTE), and the alpha factor were determined at the evaluated conditions. Moreover, the energy requirements of the SDOX system as a function of the MLSS concentration are presented and compared to the energy requirements of conventional diffused aeration systems. If the SDOX technology is successful at such MLSS concentration range, the limitations imposed by conventional diffused aeration for working at high MLSS concentrations may be uncapped, promoting the implementation of HL-MBR units and achieving the benefits that such systems can bring. The oxygen transfer performance of the SDOX technology was evaluated following the non steady-state batch test under endogenous respiration conditions (WEF and ASCE, 2001).

2. Materials and methods

2.1. Design of experiments

The oxygen transfer performance of the SDOX system was evaluated in clean water and in activated sludge mixed liquor at MLSS concentrations of approximately 4, 10, 20, 30, and 40 g L^{-1}. Fresh mixed liquor (activated sludge) was taken from the municipal WWTP of the city of Zagreb (Zagreb, Croatia). The initial and original concentration was approximately 4 g L^{-1}. Thus, the sludge was concentrated up to the desired target MLSS concentrations. At each of the evaluated experimental conditions, the oxygen transfer performance of a bench-scale SDOX system was assessed determining the K_La, the maximum SOTR, and the alpha factors. Moreover, the SOTE of the SDOX system was also determined in clean water. In all the evaluations, the oxygen intrusion from the atmosphere was also evaluated and incorporated into the oxygen transfer performance assessment. All the experiments were conducted at the laboratory facilities of the faculty of food technology and biotechnology at the University of Zagreb in Croatia.

2.2. Analytical methods

The MLSS and mixed liquor volatile suspended solids (MLVSS) concentrations were analysed according to Standard Methods (APHA, 2017). The temperature and DO were determined with a DO probe (WTW Oxi 3310, Germany). The pH was determined with a pH probe (SI Analytics GmbH, Germany). Both the DO and pH determinations were corrected by the actual temperature. The particle size distribution (PSD) was determined using a Malvern Mastersizer 2000 laser diffraction particle counter (Malvern Instruments Ltd, Malvern, UK).

2.3. Oxygen uptake rate

The oxygen uptake rate (OUR) determinations were carried out with a biological oxygen meter (BOM) based on the batch respirometric method (Drewnowski et al., 2019)). The BOM consisted of a glass container equipped with a DO probe (WTW Oxi 3310, Germany), and a stirring plate (IKA® RH B2, Germany). A Master flex peristaltic pump (Cole-Parmer, USA) recirculated the sludge from the aerobic reactor under evaluation through the BOM. When the BOM was filled with the activated sludge, the pump was stopped and the decrease of the DO as a function of time was monitored and recorded by the DO probe. After determining the OUR values, the sludge was returned back to the reactor. A DO range from 6.5 to

2.5 mg L^{-1} was used to calculate the OUR values. OUR values were determined in triplicate before and after conducting each specific experiment. The average value of the calculated OUR from each experiment was used for the determination of the reported K_La.

2.4. Experimental procedures

2.4.1. Collection and preparation of the sludge

Fresh activated sludge was collected from the WWTP of the city of Zagreb in Croatia. The WWTP was designed only for organic matter removal. The plant was operated as a CAS process at an SRT of approximately 5 days and at an average MLSS concentration of approximately 4 g L^{-1}. The general characteristics of the sludge used in this study were as follows: MLVSS/MLSS ratios of 0.78, average sludge floc size of 150 μm, and sludge volume index of 65 mL g^{-1}. The sludge was collected from one of the aerobic basins at the WWTP. The sludge was thickened by either gravity settling or membrane filtration to reach the desired MLSS concentrations. For reaching the lower range of MLSS concentrations (*i.e.*, 4 and 10 g L^{-1}) the sludge was concentrated mostly by gravity settling at the WWTP facility. The 4 g L^{-1} MLSS concentration was directly prepared by sampling sludge from the aerobic basin without any further concentration step. To prepare the 10 g L^{-1} MLSS concentration approximately 100 L of sludge were sampled and introduced into 20 L containers. The mixture was settled for approximately 30 min until reaching the desired MLSS concentration by periodically removing the supernatant. The target MLSS concentration was confirmed by determining the total suspended solid (TSS) concentration at each sample. To reach the higher range of evaluated MLSS concentrations (*i.e.*, 20, 30 and 40 g L^{-1}) the sludge was thickened by membrane filtration. A rectangular based (24 × 24 × 93 cm) 40 L bench-scale MBR provided with vertically submerged hollow fibre membranes (Zenon ZeeWee™-10, 0.4 μm pore size, 0.92 m^2 surface area) was used to concentrate the sludge. Sludge with a starting MLSS concentration of approximately 10 g L^{-1} achieved by gravity settling was introduced into the MBR and concentrated to the desired sludge concentration. Sludge volumes of approximately 60, 90, and 120 L were introduced into the MBR to achieve MLSS of approximately 20, 30, and 40 g L^{-1}, respectively. The target MLSS concentration was confirmed by determining the TSS concentrations. The sludge transport time from the WWTP to the laboratory, where the sludge concentration step was conducted, was less than an hour.

2.4.2. Experimental setup

The experimental setup is described in Fig. 1. The setup consisted of a biological reactor with a working volume of approximately 5 L. The biological reactor was equipped with a mixer that had a propeller length of approximately 0.25 m (Heidolph instruments GmbH, RZR 2102 control, Germany). The reactor was provided with a DO probe connected to a data logger (WTW Oxi 3310, Germany) and with a pH probe (SI Analytics GmbH, Germany). The BOM equipment was placed next to the biological reactor to determine the OUR. The DO was supplied to the biological reactor by means of a bench-scale SDOX system. The bench-scale SDOX system consisted of a pressurized chamber connected to an HPO source (Fig. 1). A sludge stream was recirculated from the biological reactor through the SDOX system; the sludge stream was supersaturated with pure oxygen at the high-pressure conditions in the pressurized chamber. The supersaturated sludge stream was then released from the pressurized chamber back into the biological reactor introducing in such way the DO into the reactor. The pressurized chamber had a total volume of approximately 2.75 L. Approximately 40 % of that volume (1.1 L) was occupied by the solution to be oxygenated, while the 60 % remaining (1.65 L) consisted of the headspace of the pressurized chamber. The pressure at the pressurized chamber was set at 500 kPa for the entire series of experiments carried out. The pressurized chamber was provided with two analogic pressure gauges (McDaniel Controls, USA). Moreover, both a pressure digital sensor (SICK AG, Germany) and a level digital sensor (Setra Systems, USA) were also located at the pressurized chamber. An electro-pneumatic valve (NVF3-MOH-5/2-K-1/4-EX, FESTO, Germany) was also placed at the effluent drainage of the pressurized chamber. The pressure sensors, level sensors, and the electro-pneumatic valve were used to monitor and control the level and pressure of the pressurized chamber by the aid of a program logic controller (PLC) system.

The sludge stream was introduced into the pressurized chamber of the SDOX system through a 6 mm orifice by a high-pressure peristaltic pump (EW-74203-24, Cole-Palmer, USA). A flow rate of approximately 0.825 L min^{-1} was set on the peristaltic pump for all the evaluated experimental conditions. The supersaturated sludge was released back to the biological reactor on a semi-continuous way by the action of the electro-pneumatic valve connected to the PLC system. The opening intervals of the valve were properly adjusted to maintain a constant flow of the sludge out of the pressurized chamber, so a constant level could be maintained in the pressurized chamber. The pure oxygen was supplied into the pressurized chamber from a pure oxygen cylinder (MESSER, Croatia) through a gas flow meter provided with mass totalizer capacities (Model # 32908-59, Cole-Palmer, USA). In practice, the SDOX system is operated in a continuous fashion, constantly pumping water through the saturation vessel and constantly discharging the oxygenated solution back into the process.

The oxygen transfer performance of the SDOX system was evaluated by measuring the DO transfer performance in the biological reactor operated with clean water and then with sludge at MLSS concentrations of 4, 10, 20, 30, and 40 g L^{-1}.

To determine the SOTE of the SDOX system in clean water some minor modifications were introduced to the experimental setup. A 100 L biological reactor was used to monitor the OTE on the system for a longer period. Moreover, the recirculation flowrate from the biological reactor to the SDOX system was set at 0.225 L min^{-1}. All the rest of the operational conditions remained unchanged.

2.4.3. Air intrusion experiments

The oxygen intrusion from the atmosphere into the biological reactor was evaluated and considered in the subsequent calculations. The K_La due to air intrusion in clean water was determined by the non steady-state batch test in clean water (WEF and ASCE, 2001). Nitrogen was sparged into the reactor until reaching a DO concentration below approximately 0.5 mg L^{-1}. Then, the mixer was started at an identical mixing intensity as to be used in the oxygen transfer experiments. The DO concentration was continuously monitored and recorded until reaching a DO concentration of approximately the DO atmospheric saturation value. The K_La value was then calculated by performing a non-linear regression using the Eq. 1 with the aid of the Microsoft Excel software add-in SOLVER getting the best fit between the measured and calculated DO.

$$intrusion\ accumulation\ rate = V\left(\frac{dC_{intrusion}}{dt}\right)$$
$$= K_L a_{intrusion}\left(C_s - C_{Reactor}\right) \times V \quad (1)$$

Where:

V = Biological reactor working volume

$V(dC_{intrusion}/dt)$ = Accumulation rate of DO in the biological reactor due to the atmospheric oxygen intrusion ($kgO_2\ d^{-1}$)

$K_L a_{intrusion}$ = Oxygen mass transfer rate coefficient due to intrusion (h^{-1})

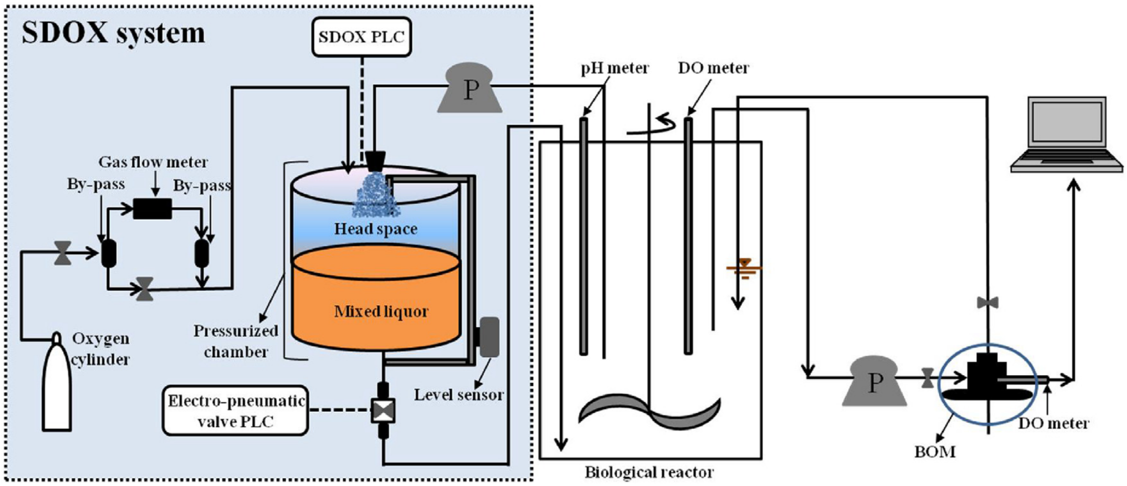

Fig. 1. Schematic diagram of the SDOX system with a bioreactor using pure oxygen used in this study.

C_s= Atmospheric DO saturation concentration in clean water ($mgO_2\ L^{-1}$)

$C_{Reactor}$= Measured DO concentration in the biological reactor ($mgO_2\ L^{-1}$)

2.4.4. Oxygen transfer performance experiments in clean water

The K_La values in clean water were determined by the non-steady state batch test in clean water (WEF and ASCE, 2001). The biological reactor was filled with tap water. The DO concentration was depleted by sparging nitrogen gas until measuring a DO concentration below 0.5 mg L^{-1}. Then, the SDOX unit was started to supply the DO into the biological reactor. The DO concentration was continuously monitored and recorded in the biological reactor until reaching a stable DO concentration. The K_La value was then calculated by performing a non-linear regression using the Eq. 2 with the aid of the Microsoft Excel software add-in SOLVER getting the best fit between the measured and calculated DO. The oxygen intrusion from the atmosphere was taken into account for adjusting the K_La values in clean water. The experiments were conducted in triplicate, and an average K_La value was reported.

$$V\frac{dC_{Reactor}}{dt} = \left[K_La\left(\beta C_{s\,HPO} - C_{Reactor}\right)\right] \times V + \left[K_La_{intrusion}\left(C_s - C_{Reactor}\right)\right] \times V \quad (2)$$

Where:

$V(dC_{Reactor}/dt)$ = Accumulation rate of DO in the biological reactor ($kgO_2\ d^{-1}$)

K_La = Oxygen mass transfer rate coefficient (h^{-1})

β= Correction factor for the influence of dissolved solids on the oxygen saturation concentration. β = 1 on the clean water experiments; β = 0.95 on the mixed liquor experiments

C_{sHPO} = Oxygen saturation concentration inside the SDOX ($mgO_2\ L^{-1}$)

The maximum SOTRs were calculated as described by Eq. 3. The SOTE values were determined by using Eq. 4 and reported when using the 100 L biological reactor.

$$\max_S OTR = K_La \times C_{s\,HPO} \times V \quad (3)$$

Where:

max_SOTR = Maximum standard oxygen transfer rate ($kgO_2\ d^{-1}$)

$$SOTE = \frac{SOTR}{HPO\ mass\ flow} \times 100 \quad (4)$$

Where:

SOTR = Standard oxygen transfer rate ($kgO_2\ d^{-1}$)

HPO mass flow = High-purity oxygen mass flow supplied to the system ($kgO_2\ d^{-1}$)

2.4.5. Oxygen transfer performance experiments in the mixed liquor

The sludge collected from the WWTP and concentrated up to the desired MLSS concentration was aerated overnight prior to the experiments. The values of K_La of the sludge at the evaluated concentrations were determined by the non steady-state batch test under endogenous respiration conditions (WEF and ASCE, 2001). The experiments were conducted from the most concentrated MLSS concentration (approximately 40 g L^{-1}) to the most diluted MLSS concentration (approximately 4 g L^{-1}). For all the evaluated experimental conditions, the same experimental procedure was carried out as follows. The reactor was filled with mixed liquor at the desired MLSS concentration. The DO concentration was depleted by sparging nitrogen gas until the DO concentration dropped below 0.5 mg L^{-1}. Then, the SDOX unit was started thus introducing DO into the biological reactor. The DO concentration was continuously monitored and recorded in the biological reactor until reaching a stable DO concentration. The OUR values were determined before and after each evaluation as described in Section 2.3 Oxygen uptake rate. Moreover, samples were taken at the end of each evaluation to determine the MLSS, MLVSS, and PSD. The K_La value was then calculated by performing a non-linear regression using the Eq. 5 with the aid of the Microsoft Excel software add-in SOLVER getting the best fit between the measured and calculated DO. Thus, the values were corrected considering the oxygen intrusion from the atmosphere. The experiments were performed in triplicate and the average K_La was reported for each experimental condition. All the determined K_La values were corrected to the standard environmental temperature (20 °C). The maximum SOTR were calculated as described by Eq. 3. The alpha factors were determined as described by Eq. 6.

$$V\frac{dC_{Reactor}}{dt} = \left[K_La\left(\beta C_{s\,HPO} - C_{Reactor}\right)\right] \times V + \left[K_La_{intrusion}\left(C_s - C_{Reactor}\right)\right] \times V - OUR \quad (5)$$

Where:

OUR= endogenous respiration rate (oxygen consumption rate by the biomass community in the mixed liquor at endogenous respiration) ($kgO_2\ d^{-1}$).

$$\alpha = \frac{K_La_{process\ water}}{K_La_{clean\ water}} \quad (6)$$

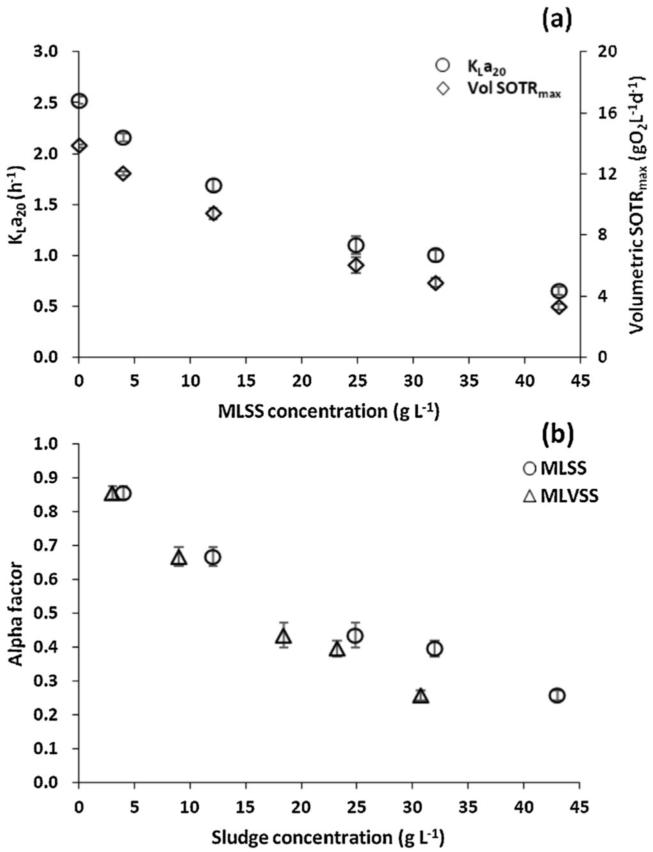

Fig. 2. (a) Oxygen mass transfer rate coefficient (K_La) and maximum volumetric standard oxygen transfer rates (SOTRs) as a function of the MLSS concentrations; (b) Alpha factors as a function of MLSS and MLVSS concentrations.

3. Results and discussion

3.1. Determination of the K_La and SOTR for the SDOX in clean water

The K_La and SOTR (Fig. 2a) were determined both in clean water and at each of the evaluated MLSS concentrations. The values reported at an MLSS concentration of zero g L^{-1} corresponds to the evaluations carried out in clean water. Both the K_La and SOTR decreased exponentially as a function of the MLSS concentrations.

A K_La of approximately 2.6 h^{-1} was obtained for the SDOX system when working in clean water. The K_La coefficients in clean water were also determined by the same authors (Kim et al., 2019) at the same experimental conditions as in this research, but using a fine bubble diffuser (SANITAIRE® Silver Series 2, Xylem, USA). Kim et al. (2019) reported K_La coefficients in clean water of 11, 23, 32, and 64 h^{-1} at the specific air flow rates (AFRs) of 5, 25, 50, and 200 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$, respectively. That is, higher K_La coefficients were reported when using fine bubble diffusers as compared to this research with the SDOX system. However, the evaluation was performed at specific AFRs ranging from 5 to 200 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$ higher than the specific AFRs commonly used in full-scale WWTPs (typically ranging from approximately 1–7 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$) (Cornel et al., 2003; Krause et al., 2003). When comparing the K_La values obtained in this research with the K_La obtained by Kim et al. (2019) at the lowest evaluated specific AFR of 5 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$ (typical AFRs applied in full-scale systems), still, higher K_La coefficients were obtained for the fine bubble diffusers (11 h^{-1}) compared to the SDOX system (2.6 h^{-1}); however, the differences are not that considerable. Henkel (2010) also reported the K_La coefficients in clean water when introducing DO by means of both fine and coarse bubble diffusers at specific AFRs ranging from 2 to 5 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$. When using fine bubble diffusers the K_La coefficients were similar to those observed by Kim et al. (2019). However, when using coarse bubble diffusers lower K_La coefficients were reported (4 h^{-1} at a specific AFR of 3 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$) closer to the K_La coefficients reported for the SDOX system (2.6 h^{-1}). Therefore, the K_La coefficients obtained for the SDOX system in clean water were lower than the commonly reported K_La coefficients for fine bubble diffusers in clean water and similar to the K_La coefficients reported for coarse bubble diffusers. The K_La coefficients in clean water have also been evaluated and reported for other supersaturated oxygen delivery technologies such as the Speece cone system (Barreto et al., 2018). Barreto et al. (2018) evaluated the oxygen transfer performance of a Speece cone system and the reported K_La coefficients of approximately 2.0 h^{-1} in clean water, which is similar to the K_La value obtained for the SDOX system (2.6 h^{-1}). Supersaturated oxygen aeration systems exhibit lower K_La coefficients compared to conventional diffuser system. Therefore, considering only an oxygen mass transfer coefficient perspective, conventional diffusers perform better than supersaturated oxygen aeration systems. However, when considering full-scale applications of the evaluated technology, other factors need to be considered such as the SOTR and the overall oxygen consumption efficiency explained as follows.

The maximum SOTR was also determined in clean water and reported as the volumetric maximum SOTR (Fig. 2a). A volumetric maximum SOTR of 14 g O_2 L^{-1} d^{-1} was observed in clean water at the evaluated operational conditions. Kim et al. (2019) reported volumetric maximum SOTR in clean water of 2.4, 5.0, 7.0, and 14.6 g O_2 L^{-1} d^{-1} at specific AFRs of 5, 25, 50, and 200 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$, respectively, at the same experimental conditions but using a fine bubble diffuser. Despite the SDOX system exhibited lower K_La coefficients than the fine bubble diffuser, higher volumetric maximum SOTRs were obtained, in particular, when comparing to the lowest (and more realistic) specific AFRs evaluated by Kim et al. (2019). At a specific AFR of 5 $m^3{}_{AIR}\,m^{-3}\,h^{-1}$, Kim et al. (2019) observed a volumetric SOTR of 2.4 g O_2 L^{-1} d^{-1} (K_La of 11 h^{-1}) compared to a volumetric SOTR of 14 g O_2 L^{-1} d^{-1} (K_La of 2.6 h^{-1}). The SDOX system was operated with pure oxygen under pressurized conditions, while the fine bubble diffuser reported by Kim et al. (2019) was operated with air at approximately atmospheric pressure. These differences may explain the higher volumetric maximum SOTR of the SDOX system. Without considering any energy consumption or OTE aspects (discussed later on in this manuscript), the advantages introduced by the SDOX system capable of working at high-pressure conditions and fed pure oxygen may overcome the lower K_La coefficients (when compared to the fine bubble diffusers). In this regard, the SDOX system can reach higher OTRs compared to conventional bubble diffusers, regardless the K_La values.

3.2. Determination of the alpha factor for the SDOX system

The alpha factors for the SDOX system were determined as a function of the MLSS and MLVSS concentrations (Fig. 2b). As expected, the alpha factors followed a similar trend as the K_La coefficients. Alpha factors of approximately 0.9 and 0.3 were reported at MLSS concentrations of approximately 5 and 45 g L^{-1}, respectively. The alpha factors exponentially decreased as a function of the MLSS concentration, while they linearly decreased as a function of the MLVSS concentration.

The K_La coefficients, and consequently the alpha factors, were negatively affected by the MLSS concentration. The impact is more noticeable at the highest evaluated MLSS concentrations. Particularly, the higher the concentration of the suspended solids in the

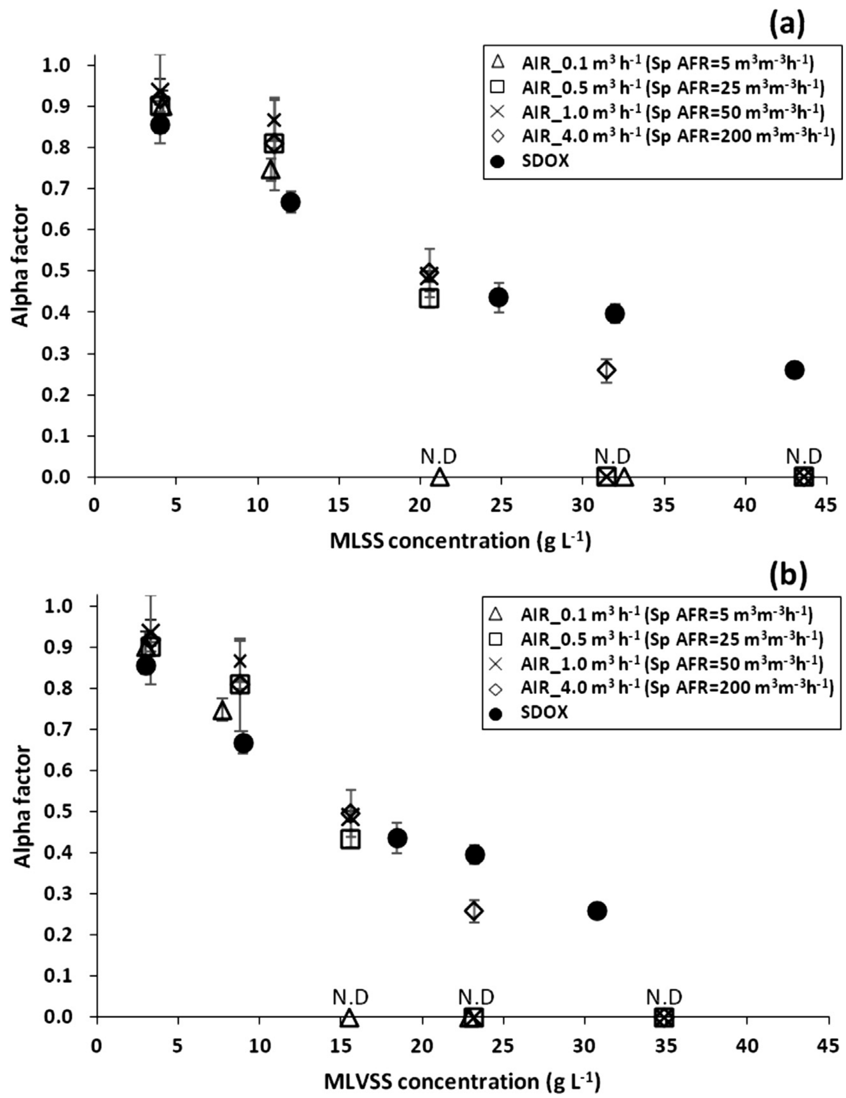

Fig. 3. Alpha factors determined in this study as a function of the MLSS concentration by the SDOX system compared to the alpha factors reported by Kim et al. (2019) as a function of the MLSS concentration obtained by fine bubble diffusers at AFR of 0.1, 0.5, 1.0, and 4.0 $m^3 h^{-1}$ (specific AFRs of 5, 25, 50, and 200 $m^3{}_{AIR}\ m^{-3}\ h^{-1}$).

mixed liquor, the higher the limitations imposed for the oxygen to diffuse from the gas phase (pure oxygen) into the liquid phase (mixed liquor). This observation is in line with previously reported studies evaluating the relationship between the oxygen transfer (expressed as alpha factor) and the MLSS concentration for conventional diffusers, both at the very same experimental conditions as in this research (Kim et al., 2019), as well as at other different experimental conditions (Cornel et al., 2003; Germain et al., 2007; Günder, 2000; Muller et al., 1995). Most of the authors concluded that the oxygen transfer is considerably inhibited by the presence of high MLSS concentrations, although an exact correlation could not be established due to differences in the operational conditions such as the AFRs, reactor configurations, sludge characteristics, types of influent wastewater and aeration systems, among others.

Generally, the higher the MLSS concentration, the more noticeable the impact of the suspended solids impeding the oxygen molecules in the gas phase to diffuse into the liquid phase. However, Henkel (2010) concluded, and as observed in our research, that the volatile fraction of the MLSS concentration (*i.e.*, the MLVSS) tends to limit the oxygen transfer process. Moreover, the author reported a direct correlation between the alpha factor and the MLVSS concentration. Firstly, the author reported a reduction in the available gas/liquid interfacial oxygen transfer area due to the accumulation of volatile solids on such transfer area. Henkel et al. (2009) examined the impacts of the volatile suspended solids of the sludge flocs on the gas-liquid interface. The sludge flocs are inclined to make contact with the gas surface area because of the inherent partially hydrophobic surface of the sludge flocs and the hydrophobicity of the gas-liquid interface. As the MLVSS concentrations increased, the gas-liquid interface area was covered with a higher amount of sludge flocs. This reduces the net interfacial area available for the oxygen transfer. Secondly, there is a direct dependence of the sludge floc volume determining the free water content of the solution. The MLVSS concentration relates to the microorganisms and extracellular polymeric substances (EPS) content of the sludge, which consists primarily of water (Ramirez-Mora et al., 2018). The more water bound in the sludge by the organic matter, the larger the volume that the floc occupies and the less free water available for the undisturbed oxygen transfer from the gas to the liquid phase. The MLVSS component of the sludge not only reduces the net interfacial area available for the oxygen transfer to occur but also increases the difficulty for the oxygen molecules to diffuse into the liquid phase. Therefore, the MLVSS concentration tends to exhibit a direct negative impact on the oxygen transfer rather than the MLSS concentration. Similar findings were obtained in our research. Fig. 2b shows an exponential decrease of the alpha fac-

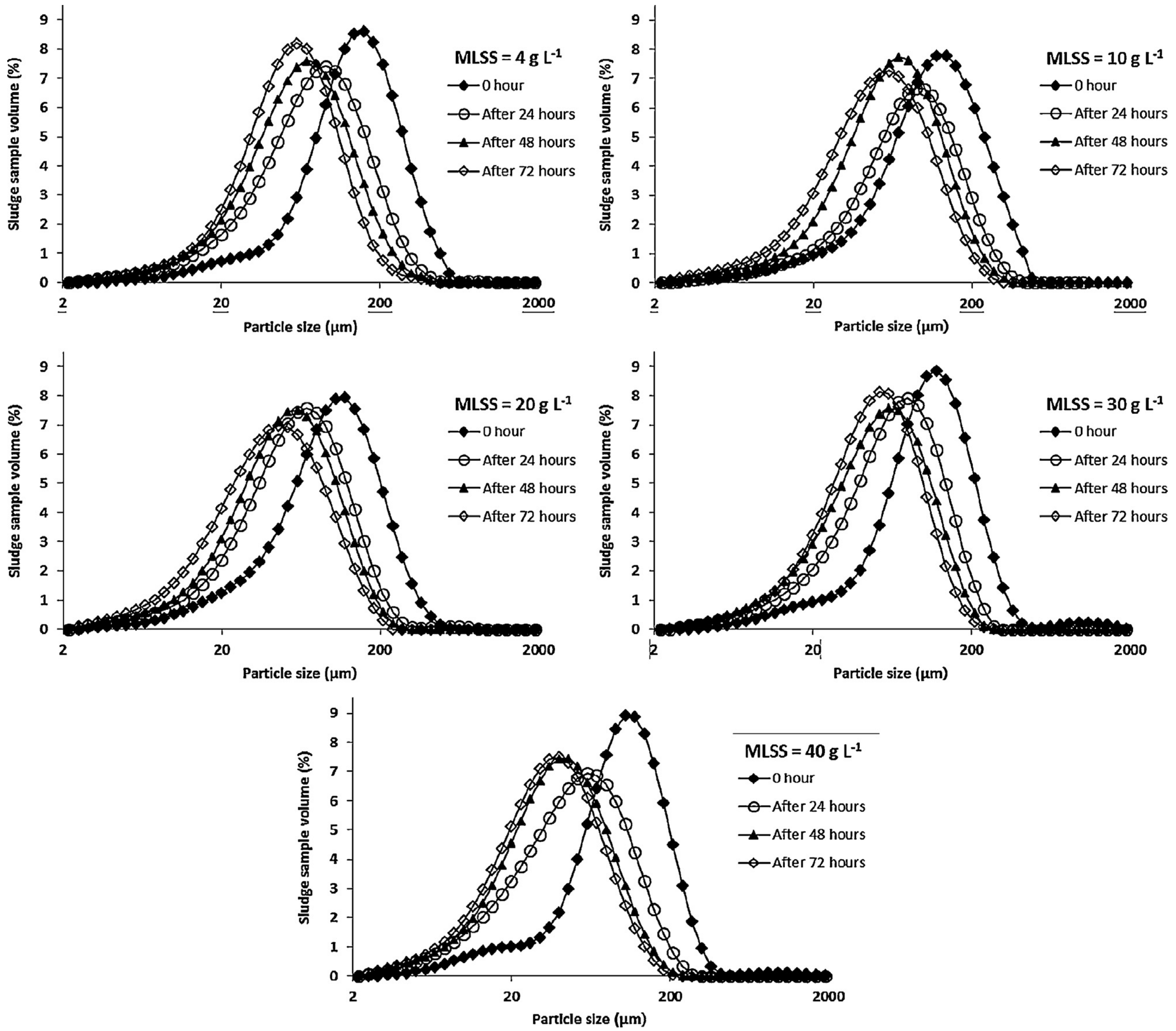

Fig. 4. Particle size distribution (PSD) of the sludge at each MLSS concentration as a function of the oxygenation time in the SDOX system.

tor as the MLSS concentration increases. However, Fig. 2b shows a direct negative relationship between the alpha factor and the MLVSS confirming the direct impact of the volatile solids fraction as reported by Henkel (2010) and Henkel et al. (2009).

3.3. Comparison between the alpha factor for the SDOX system and conventional bubble diffusers at the evaluated MLSS range

Fig. 3 shows a comparison between the alpha factors obtained in the current research when the DO is supplied with an SDOX system, and the alpha factors obtained in the research performed by Kim et al. (2019) working with the same experimental conditions but supplying oxygen by means of a fine bubble diffuser. The same sludge was used in both evaluations, so the comparison of the alpha factor as a function of the MLVSS concentration between the two studies is not influenced by the characteristic of the sludge. Almost the same alpha factors were reported at MLSS concentrations below 20 g L^{-1}. However, above that MLSS concentration higher alpha factor values were reported for the SDOX system compared to the fine bubble diffusers, even at the highest specific AFRs on the fine bubble diffusers.

The better performance observed for the SDOX system on the alpha factors could be inherent to the SDOX technology used in this study, which is conceptually and technologically different from diffused aeration systems. However, despite the potential technological advantages exhibited by the SDOX system, the K_La values reported in clean water for the SDOX system were not higher compared to those for fine bubble diffusers. Rather, lower K_La values were reported for the SDOX system compared to fine bubble diffusers. Therefore, the technological features of the SDOX system seemed not to enhance the oxygen mass transfer performance from the mass transfer rate coefficient perspective, but the SDOX seems to reduce the negative effects of the mixed liquor on the oxygen transfer (as reflected by the higher alpha factors observed on the SDOX system). Moreover, because the SDOX system can work with pure oxygen and under pressurized conditions, it can reach higher OTRs than fine bubble diffusers in spite of the lower K_La values.

The structure and morphology of the sludge can change both due to the high-pressure conditions set in the SDOX chamber, and due to the high shear effects at which the sludge is exposed (when pumping the sludge into and out of the pressurized compartment). These changes on the sludge properties could have influenced the oxygen transfer process. Fig. 4 shows the changes in the PSD of the sludge as a function of the exposure time in the SDOX system at different MLSS concentrations. As observed in Fig. 4, a considerable reduction of the average PSD was observed at all the evaluated MLSS concentrations. Specifically, the exposure of the sludge mixture to the conditions of the SDOX system created a shift in the PSD of the sludge decreasing substantially the average size of the flocs. As such, the average sludge floc size decreased approximately ten times from approximately 200 μm to 30 μm at all the evaluated conditions. The "smashing" effects of the SDOX system could not only reduce the size of the flocs, but eventually decrease its water content. Therefore, this effect could be the reason why the oxygen transfer increased by (i) reducing the specific area of the flocs that "obstruct" the gas/liquid oxygen transfer area; and (ii) increasing the free water available for an undisturbed oxygen transfer between the gas phase and liquid phase due to the reduction of the water content of the flocs. The effects of the reduction of the particle size are shown in Fig. 3 where alpha factor values were almost undetectable in the fine bubble diffuser system at MLSS concentrations higher than 20 g L^{-1}, while alpha factors ranging from approximately 0.4 to 0.2 were obtained in the SDOX system within the same MLSS concentrations range. Therefore, the SDOX system can deliver DO at higher OTRs, and at the same time reach alpha factor values higher than those reached with fine bubble diffusers at high MLSS concentrations.

Several authors have reported on the effects of the MLSS concentration on the alpha factor at MLSS concentrations of up to approximately 40 g L^{-1} on a wide range of experimental wastewater treatment setups (from laboratory-scale reactors to full-scale plants) provided with diffused aeration systems (Cornel et al., 2003; Germain et al., 2007; Günder, 2000; Muller et al., 1995). Remarkably, the alpha factors reported in this research (Fig. 2b) are higher than those reported in the literature. For instance, Muller et al. (1995) and Günder (2000) reported the highest alpha factor values as a function of the MLSS concentration. Muller et al. (1995) observed alpha factors of 0.50, 0.30, and 0.20, while Günder (2000) reported alpha factors of 0.27, 0.12, and 0.04 at MLSS concentrations of 16, 26, and 39 g L^{-1}, respectively. Muller et al. (1995) did not indicate what type of bubble diffusers were used in their study, while Günder (2000) operated pilot-scale MBRs with both fine and coarse bubble diffusers. In this research, as observed in Fig. 2b, alpha factors of approximately 0.6, 0.45, and 0.32 for the SDOX system were determined at the MLSS concentrations of 16, 26, and 39 g L^{-1}, respectively (*i.e.*, at the same MLSS concentrations reported by Muller et al. (1995) and Günder (2000)). Moreover, the studies of Muller et al. (1995) and Günder (2000) applied more favourable conditions than those used in this research to increase the oxygenation capacity: infinite SRTs (without waste of sludge) that promote the removal of surfactants (fatty acids and lipids commonly found in municipal wastewater) and lead to lower MLVSS fractions. Baquero-Rodríguez et al. (2018) reported on the negative impact of surfactants on the oxygen transfer and the benefits of longer SRTs on the aeration efficiency. On the opposite, this research was conducted with sludge obtained from a WWTP operated at a short SRT of approximately 5 days. That is, a less favourable set of conditions regarding the oxygen transfer; nevertheless, the alpha factors reported in this research are higher than those reported using bubble diffusers.

Fig. 5 compares the values of the alpha factors obtained in this research (with the SDOX system) *versus* the alpha factors reported by Germain et al. (2007) as a function of the MLSS and MLVSS concentrations. Germain et al. (2007) investigated the effect of the MLSS concentration on the oxygen transfer in fine bubble diffuser systems using sludge from full and pilot-scale municipal and industrial MBRs. Specifically, Germain et al. (2007) evaluated the oxygen transfer performance under standard conditions, and not at infinite SRTs (arguably unrealistic conditions) like those applied by Muller et al. (1995) and Günder (2000). Similar alpha factors were obtained in this research (with the SDOX system) and in that one of Germain et al. (2007) up to sludge concentrations slightly lower than 10 g L^{-1}. However, at higher sludge concentrations, higher alpha factors were obtained in this research compared to those reported by Germain et al. (2007). Therefore, the SDOX system can reach higher alpha factors than those obtained with conventional bubble diffusers under similar experimental conditions (particularly, at similar sludge characteristics).

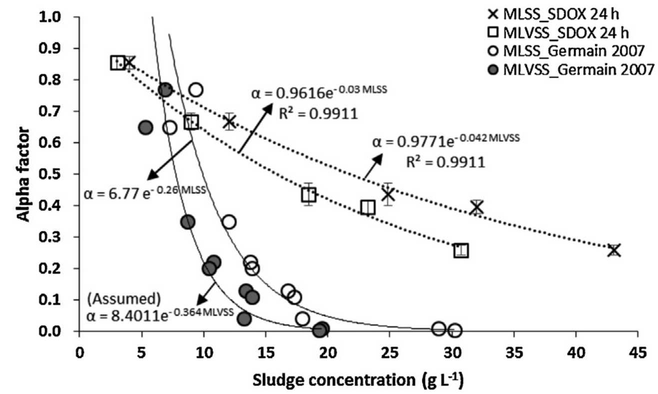

Fig. 5. Comparison of alpha factor between conventional aeration systems and SDOX at different sludge concentrations.

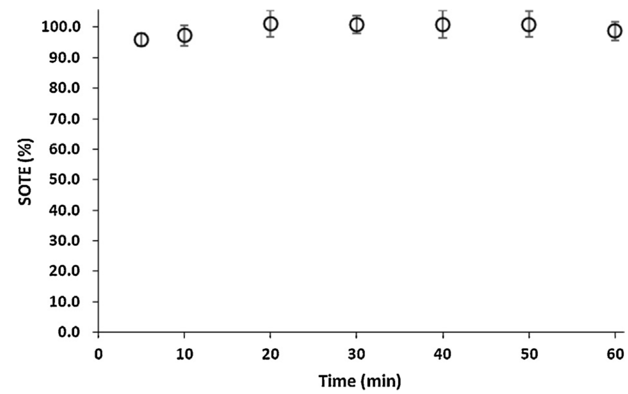

Fig. 6. Standard oxygen transfer efficiency (SOTE) for the SDOX system as a function of time in clean water.

3.4. Determination of the OTE for the SDOX system in clean water

The OTE of the SDOX system in clean water was evaluated by measuring with a mass flow controller by calculating the amount of oxygen delivered by the SDOX system, and by determining the amount of oxygen actually dissolved into the system. Fig. 6 shows the SOTE obtained for the SDOX system in clean water as a function of time. The results clearly indicate an OTE of almost 100 % for the evaluated period. That is, the SDOX system on top of achieving a higher SOTR, and exhibiting much higher alpha factors compared to fine bubble diffusers at high MLSS concentrations, also exhibited an almost 100 % OTE. This represents a major advantage for the SDOX technology compared to conventional diffused aeration system. The K_La exhibited by the SDOX system in clean water was not higher (actually even lower) than the K_La reported for fine bubble diffusers. However, the higher achievable OTRs, the higher reported alpha factors as a function of the MLSS concentration, and the higher observed SOTE (approximately 100 % in clean water) compared to conventional diffused aeration may position the SDOX technology as a promising alternative for DO supply into mixed liquors solutions.

3.5. Evaluation of the energy requirements of the SDOX system

The SDOX technology has several advantages in terms of oxygen transfer compared to diffused aeration systems; however, the technology requires to operate with pure oxygen and to recirculate (pump) the mixed liquor through a pressurized vessel, which introduces considerable operational (energy) costs to drive the SDOX system. Therefore, to better assess the applicability of the SDOX technology (or the niche is the technology may be applied) a theoretical evaluation on the energy requirements of the system was carried out at the entire range of MLSS concentrations evaluated in this research and compared to the energy needs for conventional diffused aeration systems.

The theoretical evaluation was carried out on an MBR wastewater treatment system proposed by Kim et al. (2019) exhibiting a biological oxygen demand of 2,530 kg O_2 d^{-1}. The MBR system can operate within a broad range of MLSS concentrations from approximately 4–40 g L^{-1} exhibiting different alpha factors at each of the MLSS concentration operational set points. Three different aeration systems were compared: fine bubble diffusers, coarse bubble diffusers, and the supersaturated oxygen system (SDOX technology). For each of the evaluated systems, three scenarios were evaluated at a low, middle, and high standard aeration efficiency (SAE) values (Henze et al., 2008; https://www.blueingreen.com, accessed on July 2017). The power requirements for each system were calculated at the evaluated range of MLSS concentrations from 4 to 45 g L^{-1}. The alpha factors selected for this evaluation for the fine and coarse bubble diffusers were taken from literature as follows: (i) for the fine bubble diffusers the alpha factors were taken from Germain et al. (2007), and Kim et al. (2019), and (ii) for the coarse bubble diffusers the alpha factors were taken from Günder (2000). Germain et al. (2007) evaluated the oxygen transfer performance of fine bubble diffusers on sludge obtained from full-scale and pilot plants (*i.e.*, alpha factors obtained at more realistic full-scale conditions). Kim et al. (2019) operated at the same experimental conditions like in this research, but introducing DO using a fine bubble diffuser. The alpha factors obtained by Kim et al. (2019) at a specific AFR of 5 m^3_{AIR} m^{-3} h^{-1} were considered in this evaluation. Günder (2000) operated pilot-scale MBRs with both fine and coarse bubble diffusers on sludge obtained from a plant operated at infinite SRTs (*i.e.*, these alpha factors represent one of the highest reported alpha factor in the literature). For the SDOX system, the alpha factors obtained in this research were selected for the current evaluation. Moreover, to estimate the energy needs for the SDOX system, it was assumed that pure oxygen is delivered using an on-site oxygen generator with an additional power consumption of 50 kW for the amount of oxygen delivered in this evaluation (https://www.pcigases.com, accessed on August 2019). Alternatively, pure oxygen can be supplied at an oxygen cost of approximately 100 USD ton ${O_2}^{-1}$ approximately two times more expensive than the option of using the on-site oxygen generator assuming a power cost of 0.1 USD kWh^{-1} (Fabiyi, 2008).

The theoretical results of the evaluation are described in Table 1. When working at standard CAS-relevant concentrations (4 g L^{-1}), fine bubble diffusers demand less power (28 kW at SAE of 4.2 kg kWh^{-1}) compared to coarse bubble diffusers (147 kW at SAE of 1.0 kg kWh^{-1}) and to the SDOX system (80 kW at SAE of 4.1 kg kWh^{-1}) at all the evaluated SAE conditions. Alpha factors stated above for bubble diffusers systems are higher than reported in studies from full-scale operating WWTPs. For example, Rosso et al. (2005), based on operating data from approximately 30 WWTPs, reported an average alpha factor of 0.3 for systems operating at low SRTs. Bubble diffuser systems are prone to fouling when operated in biological systems, and it is common practice to apply a fouling factor when estimating performance of bubble diffusion systems. Considering the alpha factors suggested by Rosso et al.

Table 1
Comparison of the energy requirement for each of the aeration systems as a function of MLSS concentration.

MLSS (g L^{-1})	Type of aeration systems															
	Fine bubble diffuser								Coarse bubble diffuser				SDOX			
			Power required (kW)							Power required (kW)				Power required (kW)		
	Alpha factor		Low SAE (3.6)		Mid SAE (4.2)		High SAE (4.8)		Alpha factor	Low SAE (0.6)	Mid SAE (1.0)	High SAE (1.5)	Alpha factor	Low SAE (2.7)	Mid SAE (4.1)	High SAE (5.5)
	Ger*	Kim	Ger*	Kim	Ger*	Kim	Ger*	Kim	Gunder				SDOX			
4	1.00	0.90	29	33	25	28	22	24	0.72	245	147	98	0.85	96	80	72
10	0.50	0.75	58	39	50	33	44	29	0.44	403	242	161	0.71	105	86	77
20	0.04	ND	784	-	672	-	588	-	0.19	924	554	370	0.53	124	99	86
30	ND	ND	-	-	-	-	-	-	0.08	2,119	1,271	848	0.39	150	116	99
45	ND	ND	-	-	-	-	-	-	0.02	7,359	4,416	2,944	0.25	207	153	127

Ger*: Germain et al. (2007).s.

(2005), applying such fouling factor for long-term operations, fine bubble diffusers may demand similar power (84 kW at SAE of 4.2 kg kWh^{-1}) to the SDOX system (80 kW at SAE of 4.1 kg kWh^{-1}) at CAS-relevant MLSS concentrations. At a sludge concentration of 10 g L^{-1} (MBR concentration range), fine bubble diffusers demand less energy (33 kW at SAE of 4.2 kg kWh^{-1}) than both coarse bubble diffusers (242 kW at SAE of 1.0 kg kWh^{-1}) and SDOX system (86 kW at SAE of 4.1 kg kWh^{-1}). Considering the same fouling factor as before for a long-term operation, fine bubble diffusers may demand more power (99 kW at SAE of 4.2 kg kWh^{-1}) compared to the SDOX system (80 kW at SAE of 4.1 kg kWh^{-1}). At the 20 g L^{-1} MLSS concentration, the SDOX system (99 kW at SAE of 4.1 kg kWh^{-1}) outperforms significantly both the fine (672 kW at SAE of 4.2 kg kWh^{-1}) and coarse bubble (554 kW at SAE of 1.0 kg kWh^{-1}) aeration systems. Moreover, the operation at this point would not be even practically feasible with fine bubble diffusers at standard specific AFRs. Therefore, based on this evaluation, the SDOX technology has key advantages with regard to operational power requirements compared to conventional bubble diffusers at an MLSS concentration range between 10 and 20 g L^{-1}, potentially applicable for biological systems operated at higher organic loading rates. This MLSS concentration range represents biological wastewater treatment system working at higher than usual MLSS concentrations, like for instance the HL-MBR concept introduced by Kim et al. (2019). Beyond the 20 g L^{-1} MLSS concentration (*i.e.*, at 30 and 40 g L^{-1}) the differences are even more noticeable. Above 20 g L^{-1} the first observation is that it is not even feasible to introduce DO by fine bubble diffusers since non-detectable alpha factors have been reported by Germain et al. (2007) and Kim et al. (2019). Therefore, in that range not only the SDOX technology would be more efficient with regard to energy consumption than fine bubble diffusers, but also would not be even possible to supply DO by means of fine bubble diffusers. This operational MLSS range fits well with the HL-MBR concept described by Kim et al. (2019) and eventually, it could be an appropriate technology to supply DO to aerobic digester systems.

When comparing the evaluated aeration technologies, it is important to highlight that the SDOX system is operated with pure oxygen, while conventional diffusers are operated with air. For establishing a fair comparison between technologies, ideally both systems should have been fed the same oxygen source. Kim et al. (2019) compared the performance of bubble diffusers operated with both pure oxygen and air, and reported very similar K_La and alpha factors regardless the source of oxygen at the same volumetric AFRs. Therefore, the SDOX system exhibits lower K_La and higher alpha factors compared to bubble diffusers regardless the oxygen source. When feeding pure oxygen both systems deliver similar SOTR values; however, conventional bubble diffusers exhibit such low SOTE values (approximately 8 % per meter of submergence) that for most of the applications are not economically feasible fed pure oxygen. On the other hand, the SDOX system reported much higher SOTE (of approximately 100 % in clean water) making it attractive to feed pure oxygen achieving such high SOTR values.

In Table 1 the SDOX technology was only theoretically compared to conventional diffused aeration systems since those are the most widely used systems to provide DO into engineered wastewater treatment system (Mueller et al., 2002). This comparison did not include other systems such as underground systems (the deep shaft reactor), HPO activated sludge systems, Praxair I-SO systems, UNOX system, OASES system, among others. Furthermore, the evaluation was carried out for a medium MBR system with a biological oxygen demand of 2,530 kg of dissolved O_2 per day. Results may differ when considering systems of different scales. Moreover, the alpha factors considered for coarse bubble diffusers reported by Günder (2000) were obtained on sludge from pilot plants operated at infinite SRTs. Therefore, the alpha factors for the coarse bubble diffusers were probably overestimated. Besides, on this comparison, only energy (power) requirements were discussed. Additional costs for maintenance activities were not considered. The SDOX technology may require less maintenance compared to diffused aeration system representing other potential advantages for that system that may prove the system competitive on the lowest range of evaluated MLSS concentration and expand even beyond the advantages in the high MLSS concentration range. Additionally, the evaluation only presents and considers the energy needs for introducing the DO without considering the effect of the SDOX technology on the biological activity of the system or on the downstream unit operations in a WWTP. For instance, the shear forces and high-pressure effects at which the sludge is exposed to the SDOX system may affect the biological performance of the sludge. Moreover, the observed reduction in the PSD may influence the post sedimentation of the sludge in the secondary clarifiers or the membrane filtration performance in MBR systems. Therefore, further research is needed to address these pending aspects and provide a full evaluation of the advantages and disadvantages of the SDOX system.

4. Conclusions

- The SDOX system exhibits a KLa of 2.6 h-1 in clean water similar to the ones observed for coarse bubble diffusers (4 h-1) and lower than for fine bubble diffusers (11 h-1) at standard specific AFRs. Moreover, similar KLa values were reported for the SDOX system in clean water compared to other supersaturated oxygen delivery systems such as the Speece cone system.
- Higher OTRs were reported for the SDOX system (14 g O2 L-1 d-1) compared to fine bubble diffusers (2.4 g O2 L-1 d-1 at AFR of 5 m3AIR m-3 h-1) in clean water at the experimental conditions evaluated in this research; operating the SDOX system with pure oxygen at pressurized conditions contributed significantly on obtaining such high OTRs.
- Considerably higher alpha factors were reported for the SDOX system compared to both fine and coarse bubble diffusers; particularly at MLSS concentrations higher than 20 g L-1.
- SOTEs of approximately 100 % were reported for the SDOX system in clean water; *i.e.*, much higher SOTEs compared to conventional diffused aeration.
- Above 20 g L-1, the SDOX system demands much less energy compared to conventional diffused aeration systems. The higher operational MLSS concentration range can be a niche for the application of the SDOX system in the HL-MBR concept and aerobic digesters.

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.

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