

Sidestream Superoxygenation and Activated Sludge Pre-Conditioning in a High- Solids Membrane Bioreactor (MBR) for Wastewater Treatment

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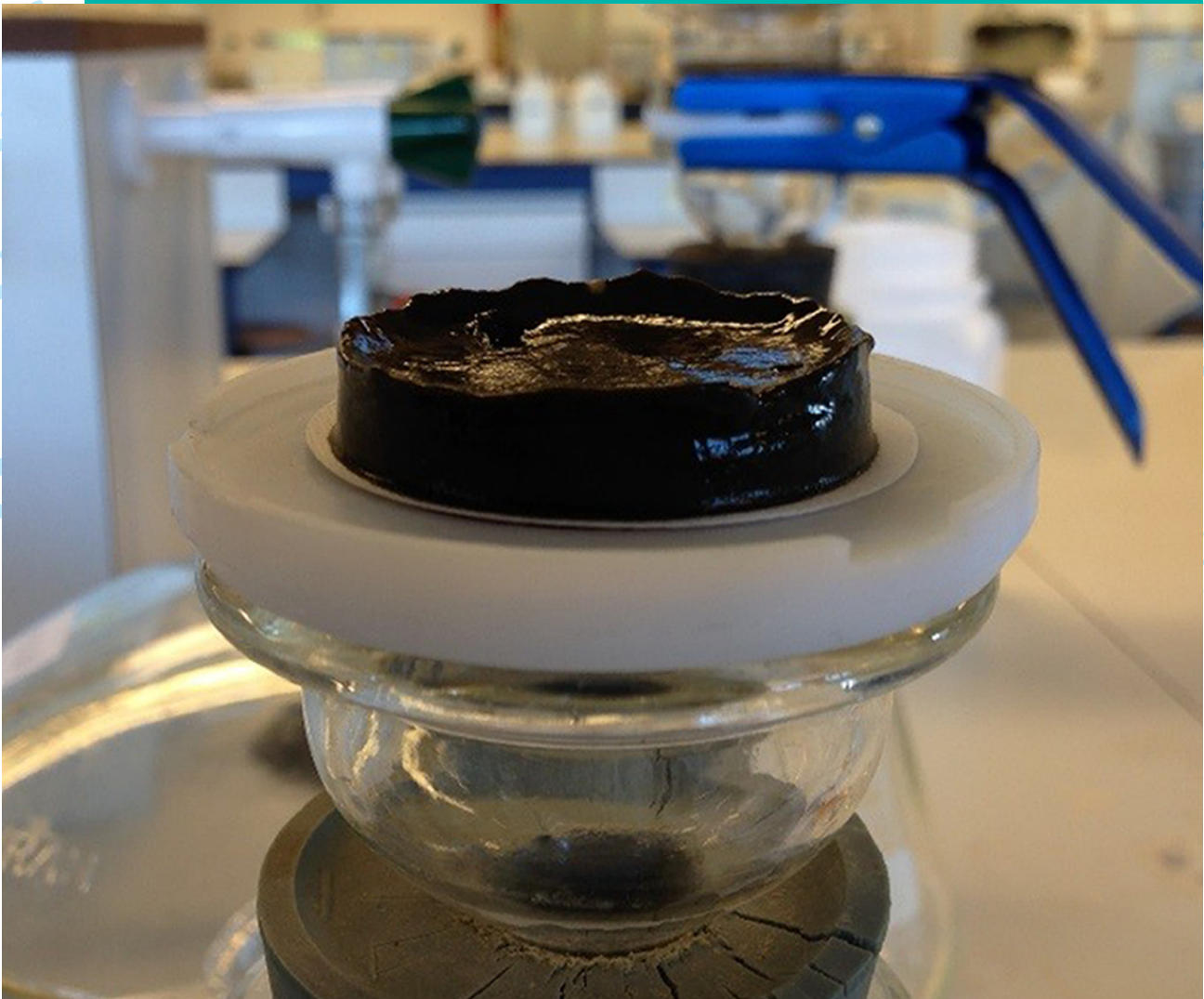
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Carlos Mauricio Barreto Carvajal

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Carlos Mauricio Barreto Carvajal

Sidestream Superoxygenation and Activated Sludge Pre-Conditioning in a High-Solids Membrane Bioreactor (MBR) for Wastewater Treatment

DISSERTATION

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at Delft University of Technology
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chair of the Board for Doctorates
and
in fulfilment of the requirement of the Rector of IHE Delft
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by

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(To...my Family)

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SUMMARY

The reduction of the volume and footprint requirements of membrane bioreactors (MBR)s is constrained by the maximum amount of biomass that can be accommodated in the aerobic basin. However, the biomass concentration is mainly limited by the extremely low oxygen transfer efficiency (OTE) experienced by conventional aeration bubble diffuser systems at mixed liquor total suspended solids (MLSS) concentrations higher than 20 g L^{-1} . Another potential limitation for the operation of MBRs at such high MLSS concentrations is the reduction on the membrane permeability due to excessive fouling.

High MLSS (high rate) MBRs require considerably smaller reaction volumes and area as compared to the the conventional activated sludge (CAS) process. Operation of high rate MBR can lower the footprint requirements for new wastewater treatment plants (WWTP)s in places where space is limited and improve the treatment capabilities of existing WWTPs that can be upgraded. In addition, movable/portable containerized MBRs for a diverse range of wastewater treatment applications ca be developed under the high MLSS MBR concept. Applications may include the provision of municipal/industrial wastewater treatment in remote areas without sewerage (decentralized sanitation), and the provision of sanitation services under challenging site-specific conditions such as after the occurrence of a human-made or a natural disaster.

Membrane bioreactors offer a wastewater treatment alternative with superior effluent quality, suitable for reuse, resource recovery or discharge. Besides being a robust and flexible system able to adapt to load and flow changes, the MBR is the most compact version of the activated sludge process due to the higher biomass concentrations that can be achieved thanks to the membrane filtration used for the solid-liquid separation. However, further MBR footprint reduction is currently constrained by two main factors:

- i) the extremely low oxygen transfer observed at mixed liquor suspended solids (MLSS) concentrations higher than 20 g L^{-1} .
- ii) the very high membrane fouling that occurs at such high solids concentrations.

Tackling these two limitations is the main objective of this research as a contribution for making the MBR more capable, compact, and portable.

The oxygen transfer limitations at high solids concentrations are a well-known fact that has been largely documented by the scientific community in the last decades. Conventional aeration methods like fine bubble diffusers and surface aerators are widely used around the globe and have proved to deliver enough dissolved oxygen for running the activated sludge process at low MLSS (<15 mg L⁻¹). However, the scientific community consensus is that with increasing solids concentrations the oxygen transfer coefficients decrease exponentially to nearly zero beyond 20 g L⁻¹ MLSS (Cornel *et al.*, 2003a; Krampe & Krauth, 2003; Germain *et al.*, 2007; Zhichao Wu *et al.*, 2007; J. Henkel *et al.*, 2009; Durán *et al.*, 2016). In order to cope with the hindered oxygen diffusion and oxygen mass transfer, this research explored the capabilities of an alternative aeration method via sidestream supersaturation using a Speece cone. This method consists of a pressurized vessel where oxygen gas is dissolved into a mixed liquor sidestream before the supersaturated mixture is returned to the biologic reactor. The Speece cone has been used in the past for hypolimnetic aeration of lakes mainly in the US for ecosystem remediation delivering supersaturated streams with dissolved oxygen concentrations of up to 30 mg L⁻¹ (Ashley *et al.*, 2008). To date, there are no scientific reports of this method being used for the aeration of membrane bioreactors.

Despite the membrane prices have lowered considerably in the last 10 years, the MBR technology is still perceived as expensive due to the membrane replacement cost and the high energy demand mainly for membrane scouring, a practice used for the mitigation of membrane fouling. Finding new methods to reduce or mitigate the fouling rates or extend the service time of membranes in bioreactors opens the possibility for MBRs to become more economically feasible at large scale for industrial and municipal applications.

A new alternative intended to contribute to the fouling mitigation research line is being proposed in this research. It consists of the introduction of hydrocyclones for solid liquid separation prior to the membrane filtration (mixed liquor pre-conditioning) in order to reduce the fouling potential and enhance the membrane performance in terms of permeate production (flux) and power demand by potentially reducing the need for membrane scouring aeration. In the proposed arrangement, the mixed liquor is pumped directly from the high MLSS MBR into the hydrocyclone where the bigger (heavier) particles are separated from the treated water or the mixed liquor supernatant (MLS) by means of centrifugal and gravitational forces due to the difference in specific weight of water and suspended solids. On the other hand, the even more concentrated activated sludge is taken out of the hydrocyclone through the underflow line and is sent back to the anoxic chamber as recirculating activated sludge (RAS), while the MLS overflows to a separate membrane tank for the permeate extraction step.

These combined new methods for overcoming both the aeration and fouling problems in high MLSS operational conditions comprise the Speece cone-MBR (Sc-MBR) concept

as the main output for this research work. The (Sc-MBR) might open the door for treating not only wastewater but also highly concentrated septic sludge in decentralized settlements. Having an effluent of superior quality, the (Sc-MBR) also offers the possibility of resource recovery via water reuse for secondary uses like urban agriculture and aquaculture on the water line. On the sludge line, it is possible to reuse the stabilized (digested) discarded sludge from the (Sc-MBR) for farming purposes.

While operating at steady state, the Speece cone provided effectively enough oxygen to sustain an MBR treating municipal wastewater while keeping DO concentrations of approximately 2 mg L^{-1} at MLSS concentrations of up to 28 g L^{-1} . Regarding sludge respiration, oxygen uptake rates (OUR) higher than $200 \text{ mg L}^{-1} \text{ h}^{-1}$ were recorded at 14 g L^{-1} MLSS and higher than $300 \text{ mg L}^{-1} \text{ h}^{-1}$ at 22 g L^{-1} MLSS. For a very short hydraulic retention time (HRT) of 3.7 hours, the (Sc-MBR) reached COD removal efficiencies of up to 99% once dissolved oxygen was not limited.

Results showed that at higher oxygen flowrates, higher transfer rates can be achieved; this however, at expense of the transfer efficiency. As expected, lower transfer efficiencies were observed in mixed liquor compared to clean water. Alpha factors, or the process-water to clean-water ratio varied between 0.6 and 1.0. However, values of approximately 0.9 and in some cases even higher can be obtained in all cases by fine tuning the oxygen flowrate delivered to the system, meaning the deviation between the mass transfer ratio in process water as compared to that in clean water was minimal.

When operating at a very low high purity oxygen (HPO) flow of 5% of the cones maximum capacity, the alpha factors obtained with the Sc-MBR setup showed values of 0.6 and 0.2 for MLSS concentrations 5 and 20 g L^{-1} respectively; these alpha values are very similar to those of conventional aeration, more specifically fine bubble aeration; in contrast, at higher HPO flow to the cone of 30%, the Sc-MBR system showed alpha factors of 0.9 and 0.7 for the same MLSS concentrations. In addition to the HPO flow, the system performance was strongly influenced by the inlet velocity and the suspended solids concentration. The Speece cone exhibited very high performance in terms of SOTE and alpha factors even at high MLSS, presenting an alternative for operation at high biomass concentrations without the mass transfer limitations of conventional aeration methods such as fine bubble, coarse bubble diffusers and mechanical aerators like paddle wheel and splash type aerators.

The Speece cone performance was affected mainly by the inlet velocity and the HPO flowrate. On the contrary, the pressure did not have a major effect on the SOTE nor the alpha factor. The apparent viscosity played a major role in the gas-liquid mass transfer efficiency.

On the membrane filtration side, long term membrane permeability reduced by two thirds (from 33 to 11 lmh bar^{-1}) when the MLSS concentration was increased from 18.7 to 27.8

g L^{-1} . Sludge filterability values expressed as the added resistance (ΔR_{20}) fell in the range of "poor filterability" for all the evaluated operational conditions.

This research contributes with the applied knowledge necessary to implement in the short term the Sc-MBR concept and develop portable systems for sanitation provision in emergency conditions. It also opens the door for a wider range of MBR applications on treating highly concentrated industrial wastewater streams taking advantage of the high MLSS concept in combination with the enhanced aeration and reduced fouling features that could be achieved if a sludge pre-conditioning stage is introduced between the bioreactor and the membrane tank or membrane module in the case of sidestream filtration. This research demonstrated that the Speece cone technology can be implemented as an effective aeration alternative for supplying large amounts of dissolved oxygen with precision at high oxygen transfer efficiencies and minimizing wastage in wastewater treatment applications.

SAMENVATTING

De reductie van het volume en de benodigde ruimte van membraanbioreactoren (MBR's) wordt beperkt door de maximale hoeveelheid biomassa die in het aerobe bekken kan worden geplaatst. De biomassaconcentratie wordt echter voornamelijk beperkt door de extreem lage zuurstofoverdrachts efficiëntie (OTE) die conventionele diffusie beluchtingsystemen ervaren bij MLSS-concentraties (mixed liquor total suspended solids) hoger dan 20 g L^{-1} . Een andere potentiële beperking voor de werking van MBR's bij zulke hoge MLSS-concentraties is de verminderde doorlaatbaarheid van het membraan door overmatige vervuiling.

MBR's met een hoge MLSS (hoge concentratie) vereisen aanzienlijk kleinere reactie volumes en -oppervlaktes in vergelijking met het conventionele proces met geactiveerd slib (CAS). Het gebruik van MBR's met hoge deeltjes concentratie kan de benodigde voetafdruk voor nieuwe afvalwaterzuiveringsinstallaties (RWZI's) verkleinen op plaatsen waar de ruimte beperkt is en de behandelingscapaciteit verbeteren van bestaande RWZI's die opgewaardeerd kunnen worden. Bovendien kunnen verplaatsbare/draagbare gecontaineriseerde MBR's voor een breed scala aan afvalwaterzuiveringstoepassingen worden ontwikkeld binnen het concept van MBR's met een hoge MLSS. Toepassingen zijn onder andere de behandeling van gemeentelijk/industriële afvalwater in afgelegen gebieden zonder riolering (gedecentraliseerde sanitatie) en de levering van sanitaire diensten onder uitdagende locatie specifieke omstandigheden, zoals na een door mensen veroorzaakte ramp of een natuurramp.

Membraan bioreactoren bieden een alternatief voor afvalwaterbehandeling met een superieure effluent kwaliteit, geschikt voor hergebruik, terugwinning van grondstoffen of lozing. Naast het feit dat het een robuust en flexibel systeem is dat zich kan aanpassen aan veranderingen in belasting en concentratie, is MBR de meest compacte versie van het actiefslibproces vanwege de hogere biomassa concentraties die kunnen worden bereikt dankzij de membraanfiltratie die wordt gebruikt voor de scheiding van vaste en vloeibare stoffen. Een verdere verkleining van de MBR-voetafdruk wordt momenteel echter beperkt door twee belangrijke factoren:

- i) de extreem lage zuurstofoverdracht die waargenomen wordt bij MLSS-concentraties (mixed liquor suspended solids) hoger dan 20 g L^{-1} .
- ii) de zeer hoge membraanvervuiling die optreedt bij zulke hoge concentraties vaste stoffen.

Het aanpakken van deze twee beperkingen is het hoofddoel van dit onderzoek om de MBR beter, compacter en draagbaarder te maken.

De beperkingen van de zuurstofoverdracht bij hoge concentraties vaste stoffen zijn een welbekend feit dat de afgelopen decennia door de wetenschappelijke gemeenschap uitgebreid is gedocumenteerd. Conventionele beluchtings methoden zoals diffusors met fijne bellen en oppervlakte beluchters worden wereldwijd veel gebruikt en hebben bewezen dat ze voldoende opgeloste zuurstof leveren om het actiefslibproces te laten werken bij lage MLSS ($<15 \text{ mg L}^{-1}$). De wetenschappelijke gemeenschap is het er echter over eens dat met toenemende vaste stofconcentraties de zuurstof overdrachts coëfficiënten exponentieel afnemen tot bijna nul boven 20 g L^{-1} MLSS (Cornel et al., 2003a; Krampe & Krauth, 2003; Germain et al., 2007; Zhichao Wu et al., 2007; J. Henkel et al., 2009; Durán et al., 2016).

Om de belemmerde zuurstofdiffusie en zuurstof massatransfer aan te pakken, onderzocht dit onderzoek de mogelijkheden van een alternatieve beluchtingsmethode via zijstroomoververzadiging met behulp van een Speece kegel.

Deze methode bestaat uit een drukvat waarin zuurstof gas wordt opgelost in een zijstroom van gemengde vloeistof voordat het oververzadigde mengsel wordt teruggevoerd naar de biologische reactor. De Speece kegel is in het verleden gebruikt voor hypolimnetische beluchting van meren, voornamelijk in de VS voor ecosysteemherstel waarbij oververzadigde stromen met opgeloste zuurstof concentraties tot 30 mg L^{-1} werden geleverd (Ashley et al., 2008). Tot op heden zijn er geen wetenschappelijke rapporten over het gebruik van deze methode voor de beluchting van membraanbioreactoren. Ondanks het feit dat de membraanprijzen de laatste 10 jaar aanzienlijk zijn gedaald, wordt de MBR-technologie nog steeds als duur beschouwd omwille van de vervangingskosten van het membraan en de hoge energievraag, voornamelijk voor membraan schuren, een techniek die gebruikt wordt om membraan vervuiling tegen te gaan. Het vinden van nieuwe methoden om membraan vervuiling tegen te gaan of te verminderen waardoor de levensduur van membranen in bioreactoren kan worden verlengt. Opent de mogelijkheid om MBR op grote schaal economisch haalbaar te maken voor industriële en gemeentelijke toepassingen.

In dit onderzoek wordt een nieuw alternatief voorgesteld om bij te dragen aan onderzoek naar de beperking van fouling. Het bestaat uit de introductie van hydrocyclonen voor vaste-vloeistofscheiding voorafgaand aan de membraanfiltratie (mixed liquor pre-conditioning) om de kans op fouling te verminderen en de membraan prestaties te verbeteren op het gebied van permeaat productie (flux) en stroom verbruik door noodzaak voor membraan schurende beluchting te verminderen. In de voorgestelde opstelling wordt de gemengde vloeistof rechtstreeks vanuit de MBR met hoge MLSS in de hydrocycloon gepompt waar de grotere (zwaardere) deeltjes worden gescheiden van het behandelde water of het supernatant van de gemengde vloeistof (MLS) door middel van centrifugale en gravitatie krachten als gevolg van het verschil in soortelijk gewicht van water en gesuspendeerde vaste stoffen. Anderzijds wordt het nog geconcentreerdere actieve slib uit de hydrocycloon gehaald via de onderstroomleiding en teruggestuurd naar de

zuurstofloze kamer als recirculerend actief slib (RAS), terwijl het MLS overloopt naar een aparte membraantank voor de permeaat extractie stap.

Deze gecombineerde nieuwe methodes voor het overwinnen van zowel de beluchtungs- als de aangroei problemen in operationele omstandigheden met een hoge MLSS vormen het Speece cone-MBR (Sc-MBR) concept als het belangrijkste resultaat van dit onderzoekswerk. De (Sc-MBR) zou de deur kunnen openen voor de behandeling van niet alleen afvalwater, maar ook van sterk geconcentreerd septisch slib in gedecentraliseerde gebieden. Met een effluent van superieure kwaliteit biedt de (Sc-MBR) ook de mogelijkheid om hulpbronnen terug te winnen via hergebruik van water voor secundair gebruik zoals stadslandbouw en aquacultuur. Op de sliblijn is het mogelijk om het gestabiliseerde (uitgegist) afvalslib van de (Sc-MBR) te hergebruiken voor landbouw doeleinden

Bij een stabiele werking leverde de Speece kegel effectief genoeg zuurstof op om een MBR die huishoudelijk afvalwater behandelt in stand te houden, terwijl de opgelost zuurstof-concentratie ongeveer 2 mg L^{-1} bleef bij MLSS-concentraties tot 28 g L^{-1} . Wat betreft de slibademhaling werden zuurstof opname snelheden (OUR) van meer dan $200 \text{ mg L}^{-1} \text{ h}^{-1}$ geregistreerd bij 14 g L^{-1} MLSS en van meer dan $300 \text{ mg L}^{-1} \text{ h}^{-1}$ bij 22 g L^{-1} MLSS. Bij een zeer korte hydraulische retentietijd (HRT) van 3,7 uur bereikte de (Sc-MBR) CZV- een verwijderings efficiëntie tot 99% wanneer de opgeloste zuurstof niet beperkt werd.

De resultaten toonden aan dat bij hogere zuurstof concentraties hogere transfer rates bereikt kunnen worden; dit gaat echter ten koste van de transfer efficiency. Zoals verwacht werden lagere transfer rendementen waargenomen in gemengde vloeistof in vergelijking met schoon water. Alfafactoren, of de verhouding tussen proceswater en schoon water, varieerden tussen 0,6 en 1,0. Waarden van ongeveer 0,9 en in sommige gevallen zelfs hoger kunnen echter in alle gevallen worden verkregen door de zuurstofstroom naar het systeem nauwkeurig af te stellen, wat betekent dat de afwijking tussen de massa overdrachts ratio in proceswater vergeleken met die in schoon water minimaal was.

Wanneer gewerkt werd met een zeer laag HPO-debiet van 5% van de maximale capaciteit van de kegel, vertoonden de alfa-factoren verkregen met de Sc-MBR opstelling waarden van 0,6 en 0,2 voor MLSS-concentraties van respectievelijk 5 en 20 g L^{-1} ; deze alfa waarden komen sterk overeen met die van conventionele beluchting, meer bepaald beluchting met fijne bellen; bij een hoger HPO-debiet naar de kegel van 30% vertoonde het Sc-MBR systeem daarentegen alfa-factoren van 0,9 en 0,7 voor dezelfde MLSS-concentraties. Naast het HPO-debiet werden de systeem prestaties sterk beïnvloed door de inlaatsnelheid en de concentratie zwevende deeltjes. De Speece kegel vertoonde zeer hoge prestaties in termen van SOTE en alfa-factoren, zelfs bij hoge MLSS, wat een alternatief is voor gebruik bij hoge biomassa concentraties zonder de beperkingen van de

massaoverdracht van conventionele beluchtingsmethoden zoals diffusors met fijne bellen, diffusors met grove bellen en mechanische beluchters zoals schoepenrad en spatelbeluchters.

De prestatie van de Speece conus werd voornamelijk beïnvloed door de inlaatsnelheid en de HPO-concentratie. Daarentegen had de druk geen grote invloed op de SOTE of de alfa-factor. De schijnbare viscositeit speelde een grote rol in de gas-vloeistof massaoverdracht efficiëntie.

Aan de kant van de membraan filtratie verminderde de membraan permeabiliteit op lange termijn met tweederde (van 33 naar 11 lnh bar⁻¹) wanneer de MLSS-concentratie werd verhoogd van 18,7 naar 27,8 g L⁻¹. Waarden voor de filtreerbaarheid van slib, uitgedrukt als de toegevoegde weerstand (ΔR_{20}), vielen in het bereik van "slechte filtreerbaarheid" voor alle geëvalueerde bedrijfsomstandigheden.

Dit onderzoek draagt bij met de toegepaste kennis die nodig is om op korte termijn het Sc-MBR concept te implementeren en draagbare systemen te ontwikkelen voor sanitaire voorzieningen in noodsituaties. Het opent ook de deur voor een breder scala aan MBR-toepassingen voor de behandeling van sterk geconcentreerde industriële afvalwaterstromen, waarbij gebruik wordt gemaakt van het hoge MLSS-concept in combinatie met de verbeterde beluchting en verminderde aangroei die kunnen worden bereikt als een slib voorbehandelingsfase wordt geïntroduceerd tussen de bioreactor en de membraan tank of membraan module in het geval van zijstroomfiltratie. Dit onderzoek toonde aan dat de Speece kegel technologie kan worden geïmplementeerd als een effectief beluchtings alternatief voor het nauwkeurig leveren van grote hoeveelheden opgeloste zuurstof met een hoge zuurstofoverdrachts efficiëntie en het minimaliseren van verspilling in afvalwater behandeling toepassingen.

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1

INTRODUCTION

1.1.MBR technology

The application of Membrane bioreactor (MBR) technology to domestic and industrial wastewater treatment offers effluent of high quality suitable for reuse; the reclaimed water can be used according to specific conditions in secondary uses such as irrigation, or with additional treatment can be fully reused in industrial process and even can be suitable for human consumption in places with water scarcity if required. In addition to superior effluent quality, the reduced requirement for the area or system's footprint is the most attractive feature of MBRs as compared to conventional activated sludge (CAS) process and this is due to the higher biomass concentration which allows for bioreactors volume reduction in an MBR system. Smaller bioreactors, in combination with membrane technology which can replace the secondary clarifiers, are the key features of this more compact wastewater treatment system.

Another interesting possibility for application of MBR technology is the sanitation provision in temporary human settlements such as emergency or refugee camps which can be improved with the application of MBR technology; the reclaimed water and stabilized sludge can be used for irrigation and farming respectively. Moreover, the ease to deploy and ability to operate at high biomass concentrations (thus low footprint) make membrane bioreactors an interesting option for sanitation in emergency conditions.

Membrane bioreactors unlike CAS systems, use a physical barrier, meaning an ultrafiltration (UF) membrane to separate the treated water or the permeate from the mixed liquor. The use of a membrane allows to obtain superior water quality in the effluent because it also retains the non-settleable material that would escape from a secondary clarifier in a CAS system. In this way, the solid-liquid separation no longer relies only on hydraulic principles and the settling properties of the sludge, but instead, will be determined among others, by the pore size of the filtration (membrane) element (*Geilvoet, 2010*).

In order to separate the treated water from the bulk biomass, and depending on the wastewater type, polymeric and ceramic Ultra Filtration (UF) membranes are commonly used in two different arrangements, namely sidestream and immersed filtration depending on whether the membrane is inside or outside the bioreactor as depicted in Figure 1 and Figure 2.

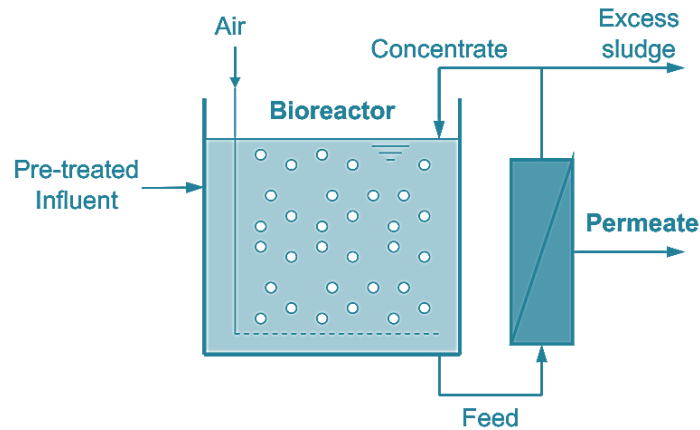


Figure 1 MBR sidestream configuration. (Geilvoet, 2010)

Beyond the obvious difference regarding the membrane's location inside or outside the biologic reactor, the main difference between these two arrangements is the way they deal with fouling prevention. Sidestream filtration relies on the liquid crossflow velocity to drag away particles that could sit on the membrane surface; for this, it is necessary to apply crossflow velocities of approximately 1 to 6 m s^{-1} in order to create enough shear forces along the membrane (Evenblij, 2006). The substantial energy input required for pumping can be as high as 10 times the required for the immersed filtration arrangement (Geilvoet, 2010). On the other hand, the immersed filtration configuration uses air scouring systems for fouling mitigation. This requires less energy but at the same time is less effective in terms of the achievable shear force that can be applied on the membrane surface. Therefore, the immersed membrane configuration is characterized by lower applied fluxes (20 to $40 \text{ L m}^{-2} \text{ h}^{-1}$) when compared to sidestream systems (50 to $100 \text{ L m}^{-2} \text{ h}^{-1}$) according to Geilvoet (2010).

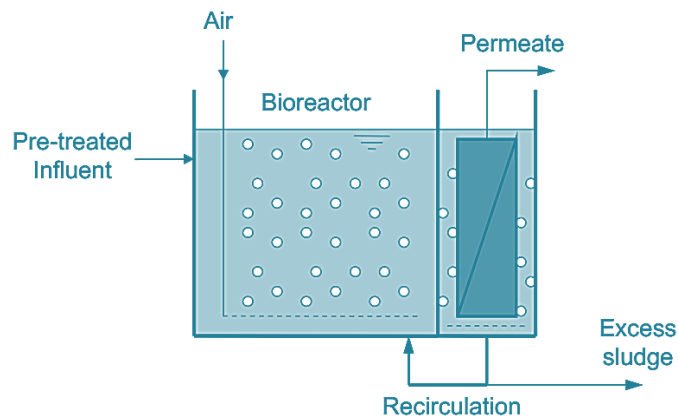


Figure 2 MBR immersed configuration. (Geilvoet, 2010)

Another difference between these two membrane configuration processes is the operational pressure or Transmembrane Pressure (TMP). According to Drews (2010),

Geilvoet (2010) Henze et al., (2008), Judd (2008), Judd (2010), Le-Clech et al., (2006) Zhang et al., (2006), the sidestream membranes require typically a higher pressure (2 to 6 bar) compared to the hollow fiber and flat sheet membranes commonly used in the immersed membrane configuration, which can operate in a lower pressure range (0.1 to 0.5 bar).

The most relevant operational parameters in the operation of MBR systems are described in this section, namely the mixed liquor suspended solids (MLSS) concentration, the trans-membrane pressure (TMP), membrane permeability, sludge filterability and membrane fouling.

1.2. Mixed liquor suspended solids (MLSS)

The MLSS concentration in the bioreactor determines not only its volume, but also the load treatment capacity, the allowable flux and the oxygen requirements according to Henze et al. (2008). Together with sludge retention time (SRT), it is a critical design parameter usually determined by the strength of the influent wastewater or in other words by the capacity of the available substrate to maintain such bacterial population.

MLSS and the specific sludge properties dependant mainly on wastewater origin, will determine the fouling potential which directly affects the permeability, durability and maintenance demands for each type of membrane.

1.3. Trans-membrane pressure (TMP)

The pressure drop across the membrane or in other words, the required pressure to produce certain permeate flux ($L\ m^{-2}\ h^{-1}$) is referred to as the TMP (bar). As fouling occurs while controlling the system for constant Flux the TMP will rise to compensate for the higher resistance to filtration in the upstream side of the membrane. The TMP is directly measured in the permeate line and needs to be corrected for the relative location of the pressure metering device. The rate at which the TMP increases in time at a fixed flux is used for calculating the membrane fouling rates, this topic is further discussed in section 1.6. membrane fouling.

$$\Delta P = -P_{measured} - \rho \cdot g (Z_{PI} - Z_W) \quad [1]$$

Where:

- ΔP : TMP, trans membrane pressure (bar)
- $P_{measured}$: Pressure reading in the permeate line (bar)
- Z_{PI} : Height of the pressure indicator (PI) (m)
- Z_W : Height of the water level (m)

1.4. Permeability

Determining permeability is the most applied method to monitor membrane operation and control cleaning cycles because it is relatively easy to measure on site. However, it is not representative of the fouling processes that cause the decrease in permeate production, it is just a snapshot of the current status in a system showing the fouling related symptoms. When measured continuously, permeability can be plotted in a time series to evaluate the rate at which permeate production reduces.

Permeability (P) is defined as the specific flow rate (per unit area) per unit pressure, or the Flux divided by the Trans Membrane Pressure (TMP) (Judd, 2010).

$$P = \frac{J}{TMP} = \frac{Q}{A \cdot TMP} \quad [2]$$

1.5. Sludge Filterability

Filterability is a sludge property that refers indirectly to the fouling potential of an activated sludge. It is related to the resistance the sludge opposes to be filtered under specific operational conditions depending on the method used to measure it. It has been the focus of attention for many researchers in the last years and there have been new methods developed mainly in *Van den Broeck et al. (2011)*, *(Rosenberg et al., 2002; Evenblij et al., 2005; Evenblij, 2006; Q.-Y. Yang et al., 2009; Geilvoet, 2010; Lousada-Ferreira et al., 2010; Thanh et al., 2010; Gil et al., 2011; Thiemig, 2012; Krzeminski, 2013; Lousada-Ferreira et al., 2015)*.

Filterability allows monitoring how the permeability is affected by the sludge characteristics and will help to identify new methods to prevent reversible fouling and also to develop more efficient filtration systems for sludge with low filterability (*M Lousada-Ferreira et al., 2010*).

No relation between permeability and filterability was found in the studies carried out by *Gil et al., 2011* in different membrane configurations (hollow fiber vs. tubular membranes) in a full scale municipal WWTP in The Netherlands. The filterability is highly influenced by the sludge temperature due to its strong relation with the fluid viscosity, however *Krzeminski (2013)* defined the viscosity's influence on filterability to be negligible when compared to other major influencers like membrane cleaning regimes and operational conditions.

The filterability is a complex indicator that represent the 'difficulty' on performing the permeate extraction from activated sludge or as *Van den Broeck et al. (2011)* proposed: "...it is dictated by the interactions between the biomass, wastewater and operational conditions...". It has been long researched in recent years (*Rosenberg et al., 2002; Evenblij et al., 2005; Evenblij, 2006; Q.-Y. Yang et al., 2009; Geilvoet, 2010; Lousada-Ferreira et al., 2010; Thanh et al., 2010; Gil et al., 2011; Thiemig, 2012; Krzeminski, 2013; Lousada-Ferreira et al., 2015*) becoming more relevant in the field of MBR

performance and operational assessment since it gives a more precise approximation of the actual process conditions.

The most popular methods to determine filterability are:

- Time to filter (TTF): measures the time it takes to produce 100 mL of permeate through a Whatman N°1 paper filter using a vacuum pressure of 51 KPa. (AWWA, 2014) Figure 3.

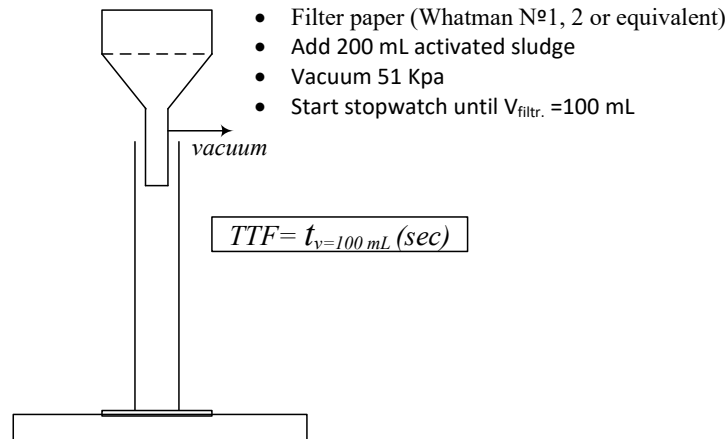


Figure 3 Time to filter (TTF) method to determine filterability. Adapted from (Geilvoet, 2010)

- The Sludge Filtration Index (SFI): relates the time to filter a given volume of permeate with the MLSS concentration, the sludge sample is filtered through a Schwarzband MN85/70 paper filter by gravity but simulating crossflow conditions using a rotating blade on top of the sample.

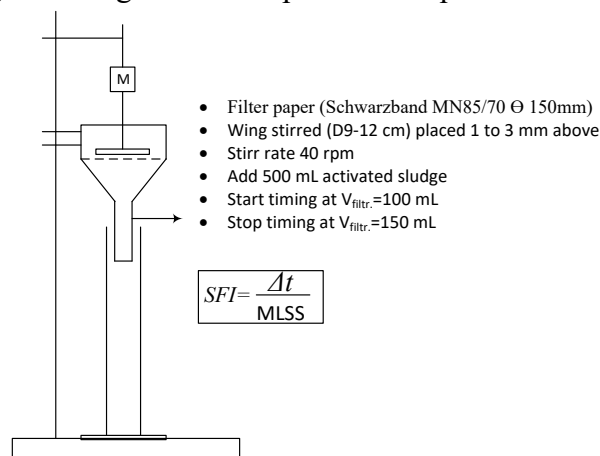


Figure 4 Sludge filtration Index (SFI) to determine filterability. Adapted from (Geilvoet, 2010)

- The Delft Filtration Characterization method (DFC_m): it is a state-of-the-art method to measure sludge filterability, and the one going to be used in this research. The Delft Filtration Characterization installation (DFC_i) consists of a complex set up with an UF single crossflow filtration element operated at very specific flux and TMP conditions.

The DFCi is a complex analytical set up consisting of a single UF cross flow membrane element where the activated sludge is continuously filtered in a closed circuit. The feed, permeate, and concentrate pressures are continuously measured to determine the TMP during the test. The DFCi reports the resistance increase ΔR_{20} [$\times 10^{12} \text{ m}^{-1}$], meaning the filterability, which is a property of the sludge. The filterability represents the added resistance to filtration of the sludge, as the sludge cake layer builds up on top of the membrane. The added resistance value (ΔR_{20}) is calculated after producing 20 L m^{-2} of permeate through the membrane at a specific flux of $80 \text{ L m}^{-2} \text{ h}^{-1}$ (lmh) and at a crossflow velocity of 1 m s^{-1} .

A schematic diagram and a picture of the installation is presented in Figure 4. The DFCi consists of the following ancillary equipment:

- ✓ Sludge feed pump (peristaltic)
- ✓ Sludge Tank
- ✓ Tater Tank
- ✓ Water pump (submersible)
- ✓ Membrane flush pump (peristaltic)
- ✓ Permeate pump
- ✓ Inflow damper
- ✓ Membrane holder
- ✓ Temperature probe
- ✓ pH probe
- ✓ DO probe
- ✓ Pressure transmitters
- ✓ Automated Electrovalves
- ✓ Mass balance (weighing scale)
- ✓ Control panel (signal collection)
- ✓ User interface (Laptop)

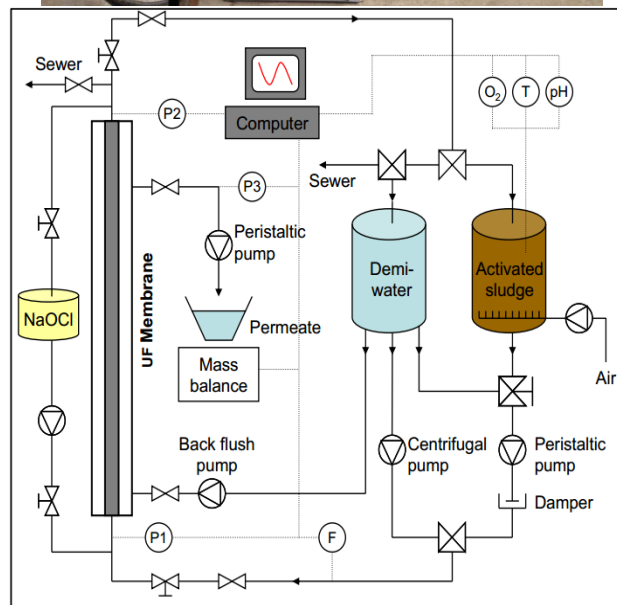
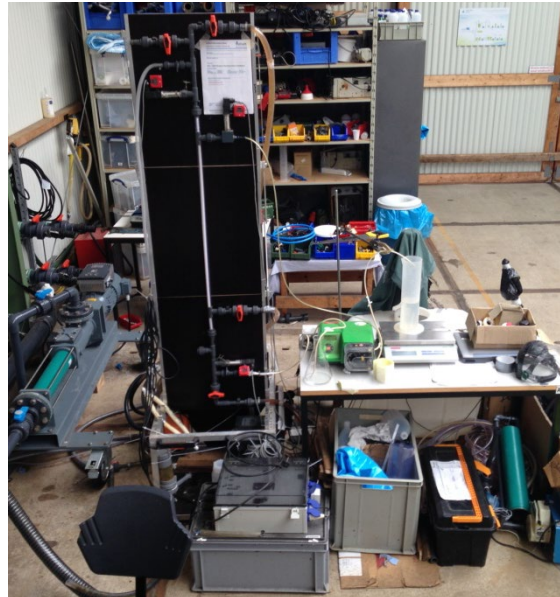


Figure 5 The Delft Filtration Characterization installation (DFCi) (Maria Lousada-Ferreira et al., 2014)

The filtration resistance can be calculated according to Darcy's law as follows:

$$J = \frac{TMP}{\eta_p R_t} \quad [3]$$

The total resistance (R_t) can be calculated by rearranging Equation 3. Having control over the operational conditions at which the filterability test takes place, allows to either measure directly or calculate the other terms in Equation 3, flux (J), transmembrane pressure (TMP) and the dynamic viscosity (η_p). Then, the resistance values obtained during the test can be plotted against the specific permeate production (V_s in Equation 4) and using a power law regression it is possible to obtain a mathematical expression as follows:

$$\Delta R = aV_s^b \quad [4]$$

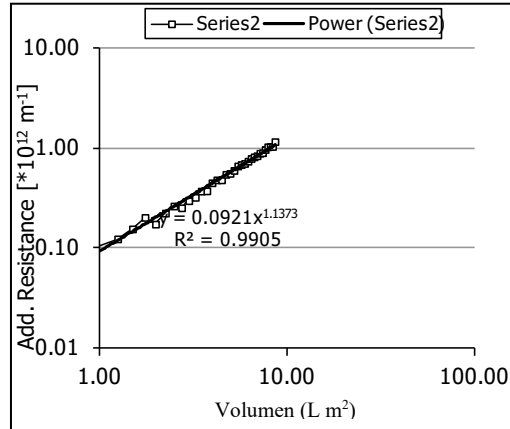


Figure 6 DFCi output file. The added resistance is plotted against the specific permeate production.

Where "a" and "b" are the known absolute coefficients describing each filtration test obtained from the previously described plot and regression. Then using a fixed permeate production value ($V_s = 20 \text{ L m}^{-2}$), the ΔR_{20} values can be calculated as follows:

$$\Delta R_{20} = a20^b \quad [5]$$

The cake layer filtration theory (Equation 6) describes the added resistance (ΔR) with an expression that matches the form of Equation 4 as follows:

$$\Delta R = (\alpha_R \cdot c_i)^{\frac{1}{1-s}} \cdot V_s^{\frac{1}{1-s}} \quad [6]$$

From these two equations it is possible to identify the experimental coefficients around V_s "a" and "b" as:

$$a = (\alpha_R \cdot c_i)^{\frac{1}{1-s}} \quad [7]$$

$$b = \frac{1}{1-s} \quad [8]$$

Where:

α_R : Specific cake resistance [m kg⁻¹]

c_i : Concentration of fouling particles [kg m⁻³]

s : Compressibility coefficient [1]

Following the expressions in Equation 7 it is possible to calculate the product ($\alpha_R \cdot c_i$) and the "s" coefficient to validate the measurements.

The previous equations (3, 4, 5, 6, 7 and 8) and descriptions have been adapted from Lousada-Ferreira (2011).

1.6. Membrane fouling

Membrane fouling refers to the accumulation of materials on top of the filtrating surface that affects in some way the permeate production, the deposited materials can be both from physicochemical or biological origin (Henze *et al.*, 2008). Even though MBR capex have been reduced as manufacturing methods are being constantly improved, it is still the opex related to the energy consumption necessary to overcome fouling which represents the major constraint for implementing large scale membrane bioreactors. Excessive fouling can lead to relevant decrease in permeate production, an increase in the backflush/cleaning frequencies and even to membrane permanent damage due to irreversible fouling (Drews, 2010).

There are many ways to classify fouling depending on the fouling mechanism, the type of deposited material and based on the definitive or temporary character of the permeate production loss, meaning reversible or irreversible fouling.

Reversible fouling is associated to materials that can be removed from the membrane surface applying physical methods like relaxation or back flushing; on the contrary, irreversible fouling can only be removed by chemical methods that are usually applied with a lower frequency (Henze *et al.*, 2008), this condition allows irreversible fouling to build up as shown in Figure 7.

When fouling cannot be removed physically or chemically it is called irrecoverable because the permeate production capacity of that filtration element cannot be recovered anymore, it is represented as the dashed line with the lowest slope in Figure 7 and can be understood as the membrane aging.

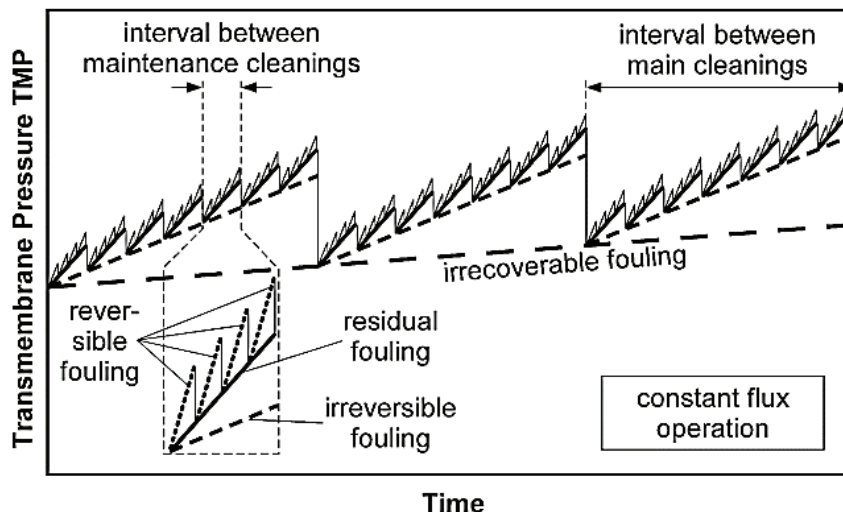


Figure 7 Schematic of fouling rates in long term operation of full scale MBRs. (Drews, 2010)

However, fouling is a very complex process depending on many factors that include operational and different process characteristics variations such as the type and concentration of wastewater, site conditions, type of membrane, maintenance and filtration regimes, applied flux, cleaning in place procedures and frequency among the most relevant parameters; this has been described extensively by other researchers in the past who have laid a solid ground for further research *Le-Clech et al. (2006)*; *Zhang et al. (2006)*; *Judd (2008)*; *Drews (2010)*; (*Judd, 2010*), for the sake of simplicity, the main parameters influencing membrane fouling can be grouped in four main categories for submerged membranes as shown in Figure 8 and Figure 9.

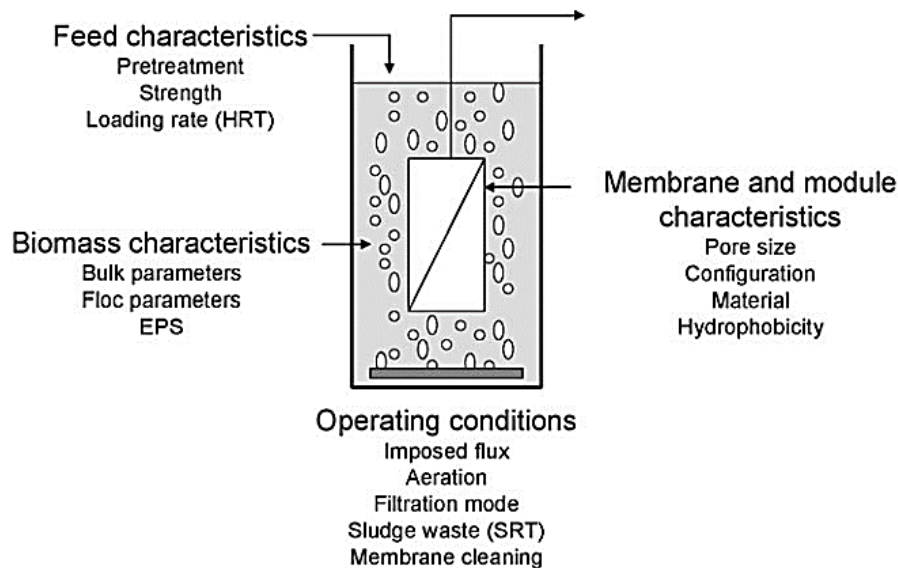


Figure 8 Factors affecting submerged membranes fouling. (Pierre Le-Clech et al., 2006)

Feed characteristics like wastewater strength, the loading rate and pre-treatment level will influence the biomass characteristics and determine amongst others the floc size and structure which directly affect the fouling potential. On the other hand, membrane characteristics together with the operation regimes will determine mainly the rates at which the fouling processes will occur and the permanent or temporal character of the fouling (reversible or irreversible). the relations between these parameters are further explained by *Judd (2010)* as presented in Figure 9.

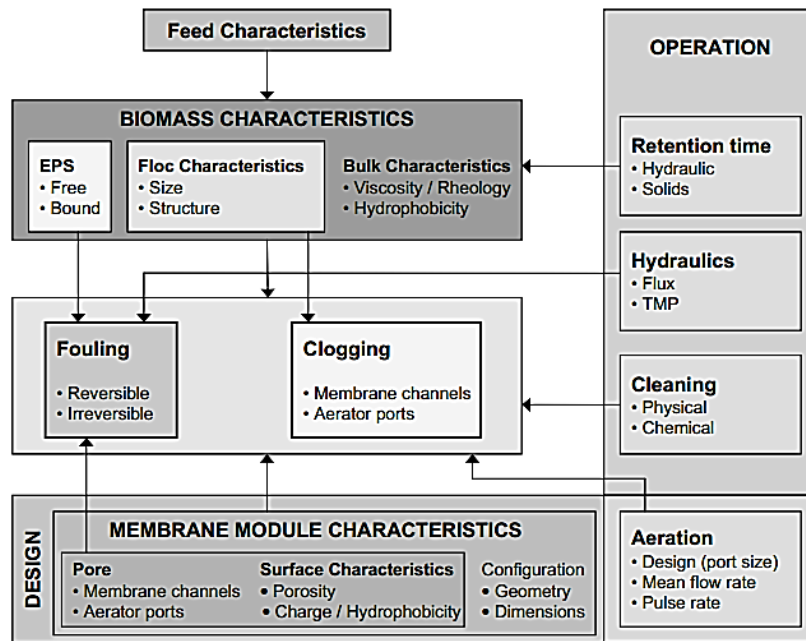


Figure 9 Inter relationships between MBR parameters and fouling. (Judd, 2010)

Another approach for graphically describing membrane fouling was presented by Zhang *et al.* (2006). In his description, Zhang *et al.* (2006) present two main charts, *i*) The fouling road map and (Figure 10) *ii*) the fouling mechanisms map (Figure 11). The fouling road map displays on the right-hand side, the design and operational parameters that have an influence over the elements on the left, the so-called fouling factors. The main rationale in the road map is that all the design parameters can be controlled. Since at some point decisions are taken in order to assign values to the listed variables, either during the design or the operational stages, the result of their interactions will determine the overall fouling nature for a particular membrane filtration facility.

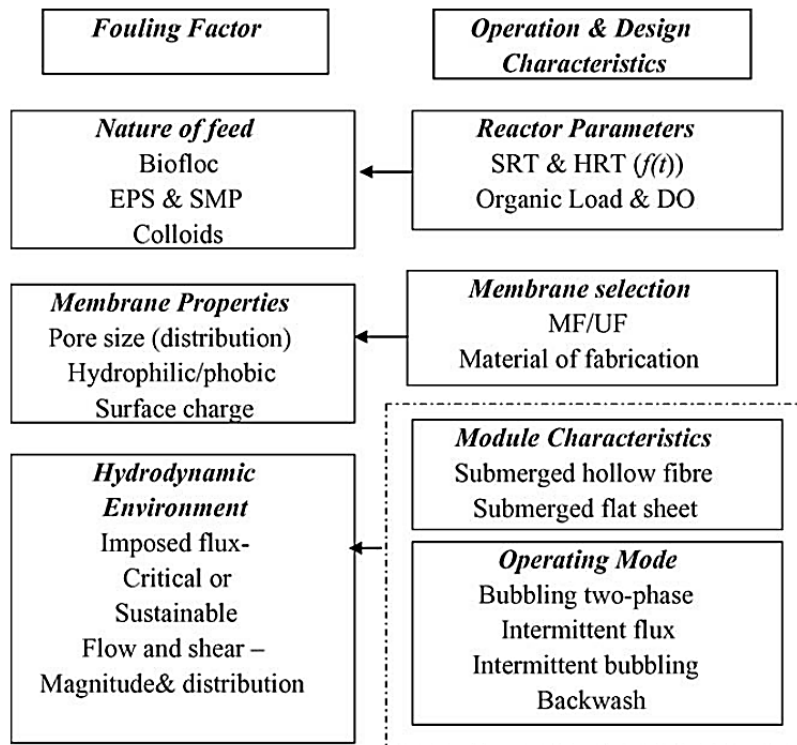


Figure 10 The fouling road map (Zhang et al., 2006)

On the other hand, the fouling mechanisms map describes how fouling occurs in time during membrane operation. Zhang et al. (2006) divides the fouling process into three main stages as follows:

- 1) Short term TMP increment due to membrane conditioning (stage 1).
- 2) Long term TMP increase which can present either a linear or exponential behaviour depending on the specific fouling characteristics (stage 2).
- 3) The TMP jump, which is characterized by a steep slope due to the pressure change rate ($dTMP/dt$) (stage 3).

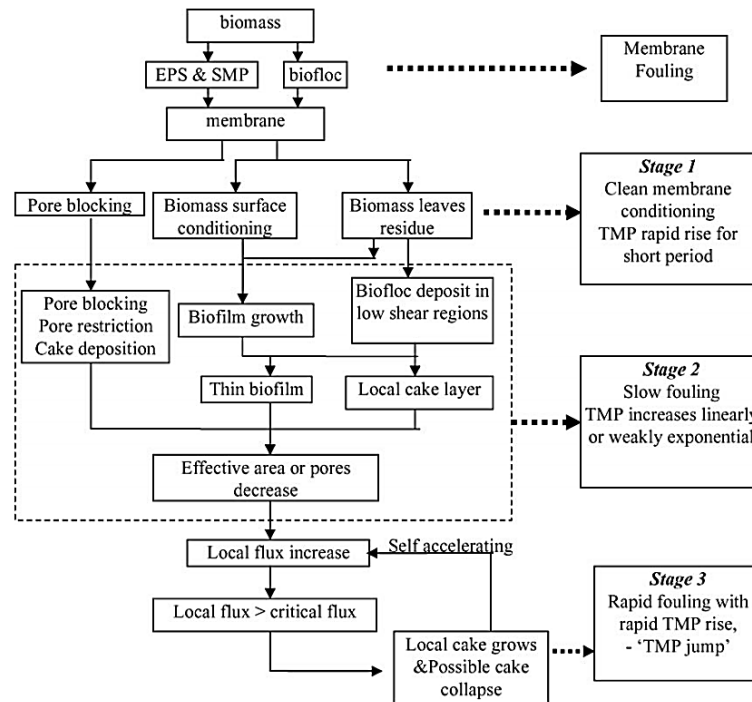


Figure 11 The fouling mechanisms map (Zhang et al., 2006)

1.7. Recent developments on Fouling control

Membrane fouling research has evaluated several factors in the last years including: the accumulation of substances and particles on the membranes (Delrue et al., 2011), the effects the SRT has on the production of extracellular polymeric substances (EPS) that affect the membrane performance (Van den Broeck et al., 2012), differences between the fouling potential on membranes exposed to suspended and attached growth conditions (Khan et al., 2011), or more recently about the membrane preconditioning by the application of a membrane coating for fouling reduction (Deowan et al., 2016). In other case, the influence of the operational conditions and membrane cleaning routines on membrane fouling processes was studied Delrue et al., (2011), Zsirai et al., (2012). A considerable number of studies have been conducted on membrane fouling by Huang et al., (2008), Ivanovic et al., (2008), Drews (2010), (Lee et al., 2013), Chen et al., (2015), Chen et al., (2016), Deng et al., (2016).

The current studies and advances in fouling research have focused on evaluating the effects of MLSS concentrations on membrane fouling, leading the way for the exploration of these phenomena in higher biomass concentrations range. These studies include the evaluation of the biomass characteristics (Chen et al., 2015), the fouling characteristics at such biomass concentrations (Lee et al., 2013), and fouling mitigation mechanisms using bio carriers (Huang et al., 2008; Ivanovic et al., 2008; Chen et al., 2016; Deng et al., 2016), also known as mechanical scouring. Despite the valuable scientific contribution of the previously mentioned research work, most of these studies were performed after diluting or concentrating the sludge samples to reach the desired MLSS

concentrations. This dilution or concentration process could probably affect some sludge characteristics namely the sludge matrix in terms of the extracellular polymeric substances (EPS) and sub-micron particles (SMP), when compared to the high MLSS sludge from a full scale system (*Barreto et al.*). In other words, by manipulating the activated sludge samples it is more likely that its structure and rheology can be modified, for instance, by de-flocculation (*Lousada-Ferreira et al., 2015*), or even worse, the release of polymeric materials that contribute to membrane fouling might be triggered (*Drews, 2010*). For these reasons, it is necessary to better understand the membrane fouling mechanisms at high biomass concentrations and evaluate the effects of such, using real activated sludge produced in MBRs operated at MLSS conditions that occur above 20 g L^{-1} ; However, sometimes this is difficult to achieve specially in pilot scale and short-term evaluations. Nevertheless, the results obtained from these types of experiments using concentration and dilution as a way to achieve MLSS concentrations of interest can give a good indication of what is occurring with the sludge rheology and its interaction with the membranes.

1.8.Fouling mitigation and control

Cleaning and recovery

Currently, the applied membrane cleaning techniques are basically divided into two groups, namely, the physical and the chemical methods. They are both applied to remove different types of fouling materials, the cleaning frequency depends on the fouling nature and the operational conditions more specifically on the applied flux. since flux is usually linked to a fixed permeate production value, TMP will increase as fouling builds up, the maximum allowable TMP (P_{max} , usually given by the membrane manufacturer) will determine when the cleaning procedure is required.

Physical methods are intended to remove reversible fouling, backflushing and membrane relaxation are carried out in a timescale of minutes (1 to 2 minutes backflushing and/or relaxation every 10 to 30 minutes operation), but it is highly dependent on the characteristics of the whole system, namely the wastewater characteristics, activated sludge rheology, the process conditions and most importantly on the operation regime and maintenance scheme.

Chemical cleaning of submerged membranes aims to remove irreversible fouling that cannot be removed by physical methods. It uses different chemical solutions (normally organic acids and sodium hypochlorite) to take away the fouling layer and bring the membrane closer to its original permeability, however it never comes back to the original value due to the irrecoverable fouling.

A combination of these two methods called Chemically-Enhanced Backwash (CEB) is being applied using a low concentration solution of cleaning product to perform backwashing (*Henze et al., 2008*).

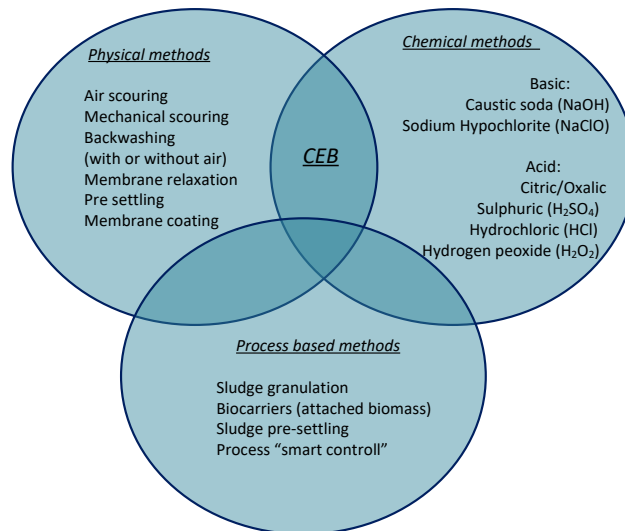


Figure 12 Simplified scheme of membrane cleaning methods. Adapted from (Henze et al., 2008)

Chemical cleaning usually takes place two to four times per year but that can be more frequent depending on the mixed liquor-membrane interactions. Cleaning in Place (CIP) is the most common practice to perform chemical cleaning by filling the membrane chamber with the cleaning agent, but it can also be performed in a separate tank.

Membrane fouling research has evaluated several factors including: the accumulation of substances and particles on the membranes (Delrue et al., 2011), the effects of the SRT (Van den Broeck et al., 2012), differences between suspended and attached microorganisms growth (Khan et al., 2011), the application of membrane coating for fouling reduction (Deowan et al., 2016), the influence of operational conditions, membrane cleaning routines (Delrue et al., 2011) other state-of-the-art process automation and control strategies (Ferrero et al., 2012). A considerable number of studies have been conducted on membrane fouling (Drews, 2010); however, limited research has been conducted on evaluating the effects of high MLSS concentrations ($>20 \text{ g L}^{-1}$) on membrane fouling or on a strategy for sludge pre-conditioning that can introduce a low-solids stage prior to the membrane filtration tank.

Mechanical scouring

A recent development by Chen et al. (2016) presents a novel method for reducing fouling rates in a relatively simple way. By adding biocarriers to the aerobic reactor, a double benefit is attained: *i*) a fraction of the mixed liquor suspended solids goes to the carrier in the form of attached biomass. *ii*) as the biocarriers move randomly due to the mixing effect provided by the aerobic basin aeration, they hit the membrane causing a mechanical abrasion effect on the sludge cake layer. The effect of this phenomenon has been found to be very positive in terms of extending the filtration time, which is the period before the system reaches the maximum operational TMP before it needs to be cleaned. The TMP series for a conventional lab scale MBR, an MBR with carriers (MBMBR) and an MBR with mechanical scouring (MBMBR_{sc}) are presented in Figure 13.

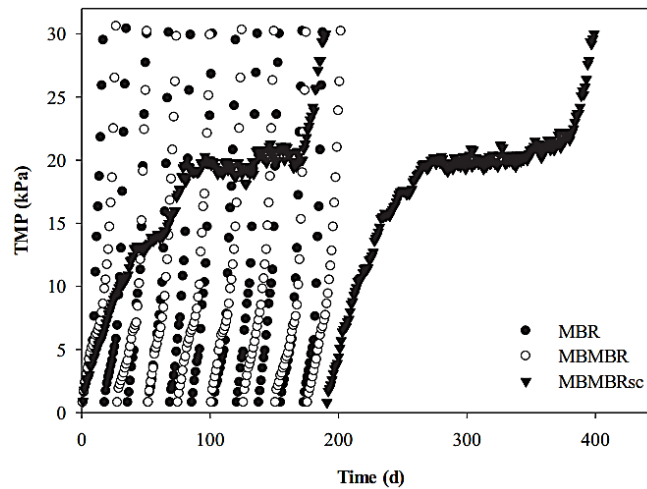


Figure 13 TMP profiles for MBRs with and without mechanical scouring (F. Chen et al., 2016)

According to Chen et al. (2016), the filtration time was extended up to 11 times as compared to the conventional MBR. That is, on day 17, the conventional MBR required membrane cleaning whereas the TMP for the MBMR_{sc} increased at a lower rate for the first 80 days, after which it stabilized on a relatively stable value for three months before increasing rapidly on day 190. This is a remarkable finding that opens the possibility for membrane operation with reduced fouling rates. However, since the mechanical effect relies on the energy that could be transmitted during the carrier collision, it is very likely for this effect to reduce with increased viscosity under high MLSS concentrations.

1.9. Fouling characterization

Membrane fouling can be characterized in two main different ways, firstly in terms of quantity or magnitude of the fouling phenomena e.g. by measuring the resistance increase or by determining to which extent it can be reversed; and secondly, in terms of quality or, in other words, by describing the factors and processes responsible for fouling and the way they interact and influence each other (Drews, 2010).

Flux step method

The flux step method (FS_m) is one of the most widely used for fouling quantification. The FS_m applies the concept of the critical flux, originally introduced by Field et al. (1995) and developed in more detail by Le Clech et al. (2003). The critical flux concept proposes that during membrane startup, there is a flux below which the flux reduction in time equals zero (Field et al., 1995). This concept, however, is not entirely applicable in practice because, like Drews (2010) stated, besides the fouling caused by the convective flow of particles towards the membrane when permeate is being produced, adsorption processes take place even at zero flux, generating membrane fouling to a certain extent. Nevertheless, the flux step method has the advantage of being a relatively simple test that allows to quantify the membrane fouling rates producing results that are comparable with several other studies.

The FS_m consists of operating a submerged membrane under constant flux conditions at different flux values applied in steps, one at a time while recording the changes in the TMP. The protocol variables that must be defined (unfortunately in an arbitrary manner) for the test are the step height, meaning the applied flux for each step, and the step duration or the time for which the TMP will be recorded on each flux step. A typical FS_m output plot is presented in Figure 14.

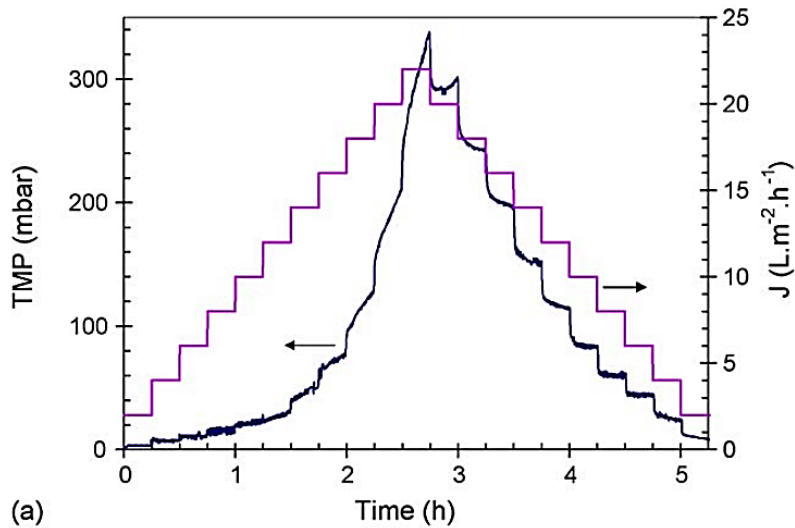


Figure 14. Flux step method output plot (Pierre Le-Clech et al., 2006).

Figure 14 shows a series of flux steps in magenta color starting at 2 lmh and increasing up to approximately 22 lmh and then all the way down again forming a symmetric shape with well defined square steps like a stair going up and down. In this series to the right-hand side of Figure 14, it is possible to identify and understand the meaning of both the flux step duration on the X axis (h), and flux height concepts on the Y axis which is expressed in $L\ m^{-2}\ h^{-1}$ (lmh). A more irregular data series in black color shows the recorded TMP increment for the series of experiments corresponding to each one of the flux steps, it can be read on the left-hand side vertical axis expressed in mbar. In the TMP series it is possible to observe that at low fluxes the TMP increase rate is almost negligible, and that there is a flux value from which the TMP becomes ever increasing. The flux value right before the drastic change or leap in the $dTMP/dt$ rate takes place is the so called critical flux (Le-Clech et al., 2006). With this input data it is possible to quantify the membrane fouling rates and the critical flux for a particular system. A schematic representation of the FS_m installation is presented in Figure 15.

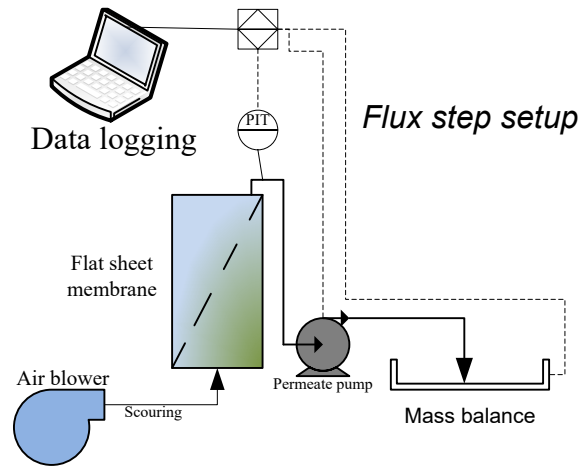


Figure 15 The flux step method experimental setup

Modified flux step method.

A modified flux step method for the determination of the critical flux has been presented by a research group in The Netherlands *van der Marel et al. (2009)*. This relatively new method presents some advantages when compared to the original flux-step method presented by *Le Clech et al. (2003)*. These advantages include, for instance, the ability to determine not only the critical flux (J_c) but also the critical flux for irreversibility (J_{ci}), which defines the boundary above which irreversible fouling will occur. Another advantage and the main difference to the original method is the introduction of membrane relaxation intervals in between the filtration steps as presented in Figure 16 and Figure 17. Membrane relaxation is performed by continuing the filtration but at a much lower reference flux (J_L in Figure 17); by allowing the membrane to relax, the effect of cumulative fouling (or fouling history) is removed from the picture, therefore it is possible to visualize on the method's output plot the flux step for which fouling cannot be removed by physical methods. Due to these advantages, the authors named this modified version the "improved flux-step method".

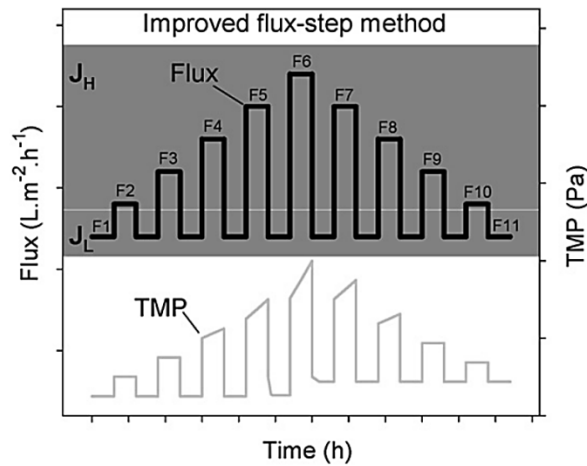


Figure 16 Typical output plot for the modified flux step method. (van der Marel et al., 2009)

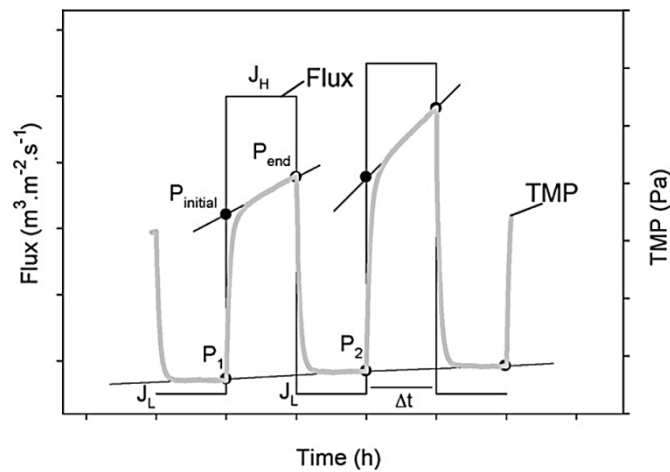


Figure 17 Detail of the output plot with method variables. (van der Marel et al., 2009)

The research conducted by *van der Marel et al. (2009)*, assessed the critical flux for two identical homemade membranes using the same activated sludge with two methods, the original and improved flux methods. In both output plots (Figure 18) the critical flux is clearly observed as an abrupt slope change in the TMP series which indicates the magnitude of the critic flux; however, for the original method, this slope is much steeper since the pressure increase is being affected by the fouling history from previous flux steps. On the other hand, the improved method presents a smooth distribution both for the ascending and descending steps with the added value of showing in a very precise way where the irreversible fouling begins. Figure 18 also shows the difference in the test duration as a downside for the improved method, meaning it takes longer to perform one test, this extended time is due to the introduced relaxation steps.

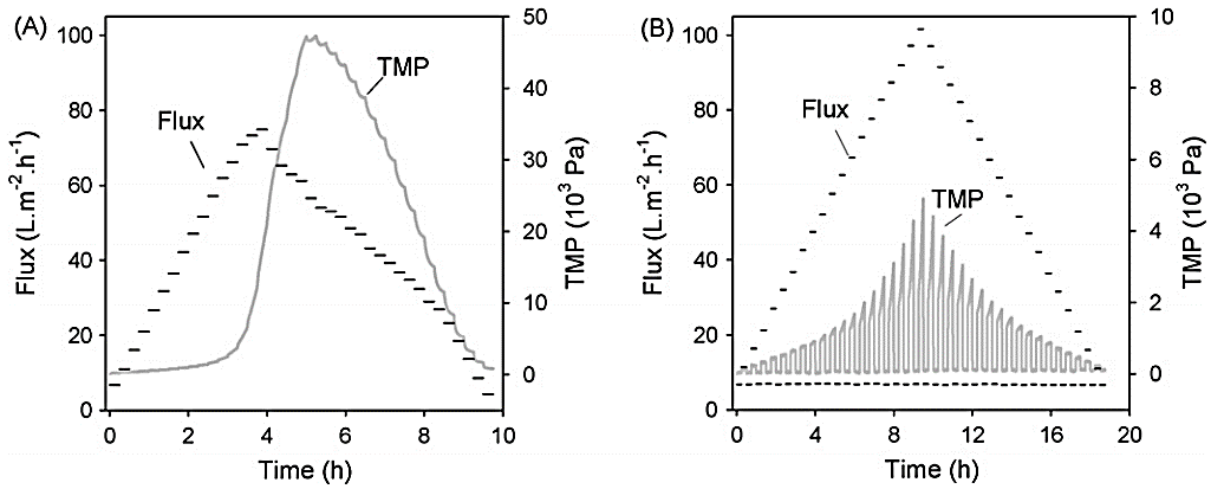


Figure 18 Comparison between the output plots for the original (A) and modified (B) flux step method. (van der Marel et al., 2009)

2. Aeration

Many studies have been conducted on the oxygen mass transfer in the wastewater field. Most of them evaluate among other operational parameters the alpha factor (α), which represents the ratio between the oxygen mass transfer coefficient to the liquid phase (k_{LA}) in wastewater divided by the same coefficient obtained for identical testing conditions in clean water, meaning the deviation of the mass transfer rate in process conditions as compared to the baseline as presented in Equation 16 in section 2.5. On the other hand, fewer studies have been conducted on the evaluation of the oxygen transfer and efficiency of aeration devices at high MLSS concentrations, particularly when talking about supersaturation or in the case of this research thesis, superoxygenation devices. *Krampe and Krauth (2003)* demonstrated the occurrence of a decrease on the oxygen transfer efficiencies at high biomass concentrations of up to 28 g L^{-1} , expressed as a reduction on the alpha factor ($\alpha < 0.1$ above 20 g L^{-1}) when using fine bubble aeration. In another study by *Karkare and Murthy (2012)* the relationship between dissolved oxygen and MLSS concentrations up to 40 g L^{-1} was explored on a laboratory scale batch reactor provided with a mechanical diffusion aeration device and presented a kinetic model to predict the COD removal on agrochemical wastewater. More recently, *Mitra et al. (2016)* evaluated the oxygen transfer efficiency of an alternative aeration technology comparing an eductor to conventional fine bubble diffusers, finding that the eductor was indeed more efficient in delivering oxygen to the system ($\alpha = 0.91$ and 0.75 for eductor and diffuser respectively), but also noticing a positive effect on the membrane fouling rates possibly attributed to higher bubble rising velocity due to higher bubble size. However, that study was still in the low MLSS range (up to 6.5 g L^{-1}). *Durán et al. (2016)* evaluated the effect of suspended solids on the oxygen transfer efficiency in activated sludge at a MLSS concentrations up to 10.2 g L^{-1} finding that the apparent viscosity plays a crucial role in the oxygen transfer at varying operational conditions such as increasing MLSS

concentration. Therefore, despite recent research, there is still a need and a clear interest for advancing alternative oxygen delivery systems that can supply dissolved oxygen to the bioreactor operating at high MLSS concentration.

2.1. Conventional aeration methods

Aeration is one key aspect in terms of operational expenses with 45 to 75% of the energy demand for aerobic treatment processes including CAS and MBR (Henze *et al.*, 2008). Low pressure aeration with fine bubble diffusers is the most popular method to deliver the system oxygen demand. Diffusers come in many different shapes and configurations but essentially, they are comprised of a structural part providing support to a perforated polymeric membrane that breaks down the air flow into small bubbles. Disc and tubular diffusers are used extensively worldwide, and recently flat panels are taking over a portion of the market due to the simplified requirements for installation and the fact that can be easily removed from the aeration basin for maintenance or replacement.



Figure 19 Disc and tubular diffusers for fine bubble aeration (Stanford Scientific International LLC).

Aeration efficiency depends on the temperature, the oxygen partial pressure at site conditions and on the effective contact area between the two phases (liquid and gas phase), therefore the relevance of bubble size, in that sense the smaller the bubbles the higher the contact area and the better the oxygen transfer. The final dissolved oxygen concentration (at equilibrium) in activated sludge depends also on *i*) the rate at which the oxygen mass transfer process from the gaseous to the liquid phase occurs; *ii*) the oxygen transfer rate into the biofloc where it is used, and *iii*) the rate of usage by the microorganisms or Oxygen Uptake rate (OUR) (García-Ochoa & Gómez, 2009). These concepts will be further addressed in the following sections.

Aeration is not only important to carry out the microbiological processes in the activated sludge process but also for keeping the membrane running for a longer time (fouling mitigation) in the case of the MBR technologies (see section 1.8. fouling mitigation and control).

2.2. Alternative aeration methods

2.2.1. Supersaturated oxygen aeration system SDOX

Alternative oxygenation systems are needed to cope with the high oxygen demands and low oxygen transfer efficiencies exhibited by MBRs operated at high MLSS concentrations in low water depth systems. Oxygen transfer efficiencies on conventional bubble diffusers are highly impacted at high MLSS concentrations and low submergence conditions. Concentrated oxygen delivery systems (such as the Speece cone), commonly used for hypolimnetic aeration mainly in lakes (*Ashley et al., 2008*), may present a feasible alternative for providing the additional required oxygen at such high MLSS and low submergence conditions. The oxygen transfer capabilities and oxygen transfer efficiencies of similar concentrated oxygenation systems using a side-stream saturation concept (super saturated dissolved oxygenation system - SDOX) has been evaluated by *Kim et al. (2015)*. The SDOX system uses a nozzle to generate an activated sludge mist to enhance the liquid-gas phase contact at a high pressure enhancing the oxygen transfer process. The results obtained in that study were very promising in terms of both oxygen transfer rates, and oxygen transfer efficiencies at high MLSS concentrations.

2.2.2. Speece cone

The Speece cone was developed in the United States in 1971 for hypolimnetic aeration of ponds and lakes for remediation purposes by Richard Speece.

The Speece cone (Figure 20) consists of a conic structure fed with water from the top at a high velocity (approximately 3 m s^{-1}). Pure oxygen is injected right at the inlet close to the smallest cone diameter. Once the oxygen is introduced, the gas buoyancy will lift the gas up, but the down-flow velocity of the water stream will keep the oxygen in the throat section of the cone where an interface is formed. The oxygen bubbles have both enough retention time and surface contact area under mid-high pressure (from 2 to 5 bar) to get the oxygen dissolved into the water. Steady dissolved oxygen concentrations of up to 30 mg L^{-1} at the discharge outlet were reported by (*Ashley et al., 2008*). The bubble dynamics and oxygen transfer were studied by (*Burris & Little, 1998*) and in a more advanced level, the mathematical expressions for modelling the oxygen transfer dynamics in a Speece cone have been presented by *McGinnis and Little (1998)*.

The supersaturated flow is discharged from the bottom of the cone to either a tank or a lake (or any other water source). The available research publications describing the performance of a Speece cone being used for membrane bioreactors at high MLSS concentrations is limited to a single study performed by *Barreto et al. (2017)*, *Barreto et al., (2018)*, which is part of this research thesis.



Figure 20 Speece cone at the Harnaspolder WWTP, The Netherlands (source: C. M Barreto).

The Speece cone has shown very impressive oxygen transfer efficiency results regarding both the oxygen transfer coefficient (k_{La}), and the standard oxygen transfer rate (SOTE). The process can be controlled using the cone's flow discharge velocity and the oxygen input flow rate as shown in Figure 21 by Ashley *et al.*, (2008).

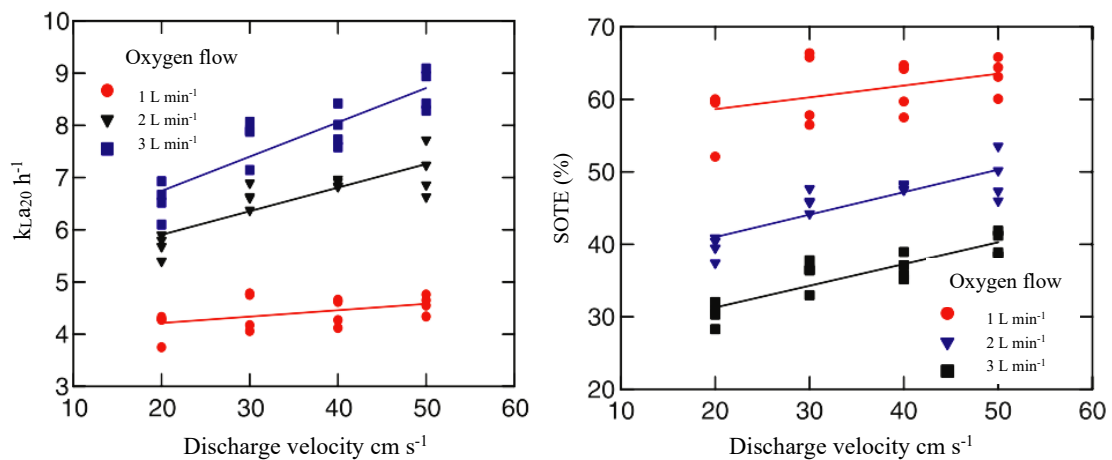


Figure 21 Speece cone k_{La} coefficient and SOTE at different discharge velocities and oxygen flowrates (KI Ashley *et al.*, 2008).

According to the research conducted by (Ashley *et al.*, 2008), the flow discharge velocities should be higher than 70 cm s^{-1} in order to achieve a higher transfer. In addition, the authors reported the importance of keeping the gas-liquid interphase stable at the cone's throat; otherwise, the efficiency will decrease drastically. The operation of the Speece cone on air instead of on pure oxygen is not economically feasible due to the

low energetic efficiency caused by the increase in the recirculation pumping needs required to provide similar results (*Ashley et al., 2008*).

2.3. Oxygen transfer

Aeration is a mass transfer process driven by a difference in concentration or gradient. This diffusion process for a liquid-gas interphase can be expressed with Fick's law:

$$N = -D_L A \frac{dc}{dy} \quad [1]$$

Where:

N	: mass transfer per unit time
A	: cross section area
D_L	: diffusion coefficient
dc/dy	: concentration gradient perpendicular to cross sectional area

When two phases (liquid and gas) are involved, the transfer rate in equilibrium can be expressed as:

$$N = k_L A (C_s - C_L) = k_g A (P_g - P) \quad [10]$$

Where:

C_s	: oxygen saturation concentration
C_L	: oxygen concentration in the liquid
k_L	: liquid film coefficient defined as D_L/Y_L
k_g	: gas film coefficient defined as D_g/Y_g

The mass transfer process for gases with low solubility (O_2 , CO_2) is liquid film controlled, that is, the transfer rate is determined by the liquid film resistance, for this case, the overall transfer rate can be expressed in terms of concentration as follows:

$$\frac{1}{V} N = \frac{dc}{dt} = k_L \frac{A}{V} (C_s - C_L) \quad [21]$$

The term $k_L AV^{-1}$ is most expressed as $k_L a$, representing the overall liquid film transfer coefficient for aeration processes, giving as a result:

$$\frac{dc}{dt} = k_L a (C_s - C_L) \quad [3]$$

During the activated sludge process, the oxygen is being consumed by the active biomass in the reactor for both endogenous respiration and substrate uptake (growth), including this factor in the equation we obtain:

$$\frac{dC}{dt} = k_L a (C_s - C_L) - r_t \quad [4]$$

Where:

$$r_t = r_s + r_e$$

- r_t : total oxygen uptake rate [$\text{mg L}^{-1} \text{min}^{-1}$]
 r_s : endogenous oxygen uptake rate [$\text{mg L}^{-1} \text{min}^{-1}$]
 r_e : exogenous oxygen uptake rate [$\text{mg L}^{-1} \text{min}^{-1}$]

When equilibrium is reached ($dC/dt \rightarrow 0$) and in absence of substrate for exogenous respiration ($r_e \rightarrow 0$), the equation can be rearranged to calculate $k_L a$.

$$k_L a = \frac{r_s}{(C_s - C_L)} \quad [14]$$

2.4. Factors affecting oxygen transfer

The oxygen transfer in bioreactors is a complex process that has been studied extensively in the past years (*Wagner, 1999; Mueller et al., 2002; Cornel et al., 2003a; Germain et al., 2007; Garcia-Ochoa & Gomez, 2009; Henkel, 2010; Henkel et al., 2011*). This mass transfer process is affected by many factors including physical properties of the activated sludge (viscosity, SRT, MLSS, particle size), process conditions (atmospheric pressure, altitude, temperature), influent characteristics, interface contact area (bubble size, biofloc free water content), oxygen concentration gradient among others. A clear simplification of the most relevant variable affecting the oxygen transfer rate (OTR) was presented by *García-Ochoa and Gómez (2009)* and is shown in Figure 22.

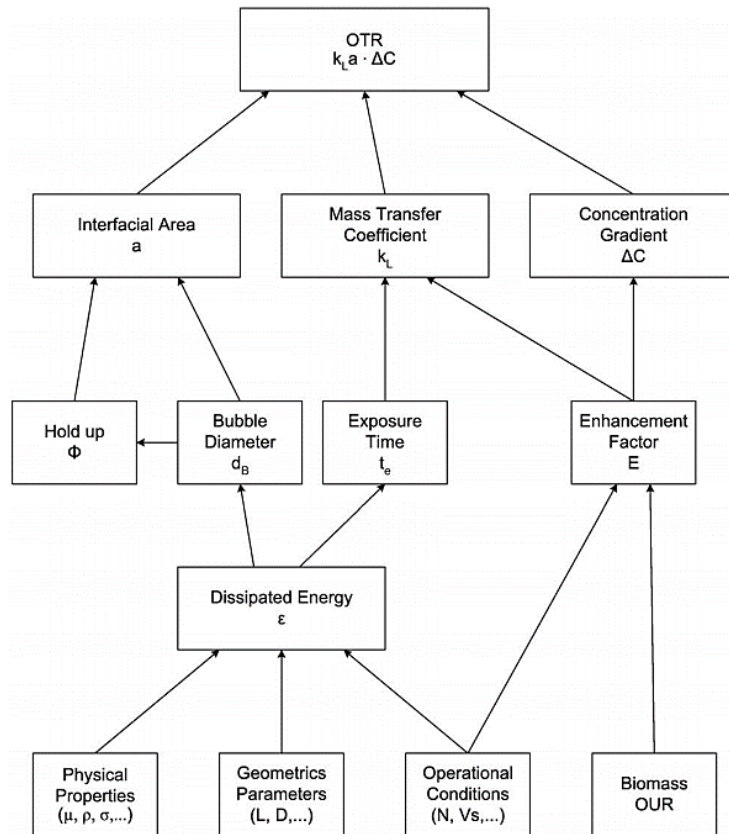


Figure 22 Interactions of the main parameters affecting the OTR in activated sludge. Modified from (García-Ochoa & Gómez, 2009).

The liquid film coefficient $k_L a$ can be corrected for temperature variations with the following equation:

$$k a_{(T)} = k_L a_{(20^\circ C)} \theta^{(T-20)} \quad [5]$$

2.5. Alpha factor

This factor represents the ratio between the transfer rates for process and clean water. Since alpha is an experimental coefficient product of field measurements under the actual process conditions, it accounts for the settings of a given system, considering the aeration method, environmental conditions, wastewater, and sludge characteristics.

$$\alpha = \frac{k_L a (\text{wastewater})}{k_L a (\text{tap water})} \quad [16]$$

2.6. Oxygen saturation concentration

The maximum amount of oxygen that can be dissolved in water depends on the site temperature, barometric pressure, submergence, and water salinity. The corresponding equations for these correction factors are:

2.7. Temperature

$$\tau = \frac{C_{st}^*}{C_{s20}^*} \quad [17]$$

Where:

τ : Temperature correction factor

C_{st}^* : Oxygen saturation concentration at site temperature

C_{s20}^* : Oxygen saturation concentration at 20°C equals to 9.09 mg/L

(*) indicates the saturation concentration at 1 atm and zero total dissolved solids (TDS)

2.8. Wastewater characteristics (salinity)

$$\beta = \frac{C_s^* \text{ wastewater}}{C_s^*} \quad [6]$$

Where:

β : Salinity correction factor

However, the beta factor can also be calculated from the following expression within a TDS concentration from 0 to 20,000 mg L⁻¹.

$$\beta = 1 - 5.7 \times 10^{-6} \times TDS \quad [19]$$

2.9. Barometric pressure

$$\Omega = \frac{C_{\infty Pb}^*}{C_{\infty Ps}^*} = \frac{P_b}{P_s} \quad [20]$$

Where:

$C_{\infty Pb}^*$: Oxygen concentration saturation at 20°C at site conditions

$C_{\infty Ps}^*$: Oxygen concentration saturation at 20°C at standard conditions

The barometric pressure at different altitudes can be calculated as follows:

$$P_b = P_{b0} \left(1 - \frac{alt (m)}{9100} \right) \quad [7]$$

2.10. Submergence

The oxygen transfer is also affected by the depth at which the diffuser is located, the correction factor to account for this can be calculated as:

$$\delta = \frac{C_{\infty 20}^*}{C_{s20}^*} = \frac{P_s + p_{de} + p_v}{P_s - P_v} \quad [22]$$

Where:

P_s : standard atmospheric pressure (1atm)

p_{de} : liquid static pressure at diffuser depth

p_v : vapor pressure at 1 atm and 20°C

The overall expression to calculate the oxygen saturation concentration under field conditions for an activated sludge suspension.

$$C_{\infty f}^* = \tau\beta\Omega C_{\infty 20}^* \quad [8]$$

$$C_{\infty 20}^* = \delta C_{s20}^* \quad [9]$$

The previous equations (From Equation 10 until Equation 24) and the corresponding definitions have been adapted from (Ros, 1993; Mueller et al., 2002).

2.11. Sludge retention time, MLSS and viscosity

The influence of the SRT and MLSS on the oxygen transfer, more specifically on the alpha factor, has been demonstrated in the past by many authors (*Groves et al., 1992; Wagner, 1999; Rosso et al., 2005; Gillot & Héduit, 2008; Henkel, 2010*), as shown in Figure 23, Figure 24, Figure 25, Figure 26, Figure 27 and Figure 28. This effect has been long established by either addressing the direct influence that SRT has over the mixed liquor viscosity or by establishing direct or indirect relations between MLSS-SRT-viscosity (*Krampe & Krauth, 2003; Trussell et al., 2007; Delrue et al., 2011; Durán et al., 2016*). A simple but clear schematic showing the relations between some factors having an effect over the oxygen transfer by *Germain and Stephenson (2005)* is presented in Figure 25. From this scheme, it is clear that at higher solids concentration and viscosity, a lower oxygen transfer will take place.

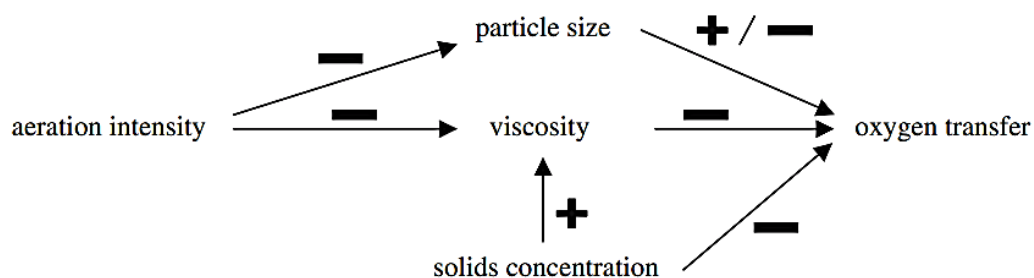


Figure 23 Summary of MBR biomass characteristics effects on oxygen transfer (*Germain & Stephenson, 2005*)

The SRT-MLSS effects over the oxygen transfer from other research groups have been summarized by *Henkel et al. (2011)* in simple but very concise short sentences as follows:

Modified from *Henkel et al. (2011)*

- “...with increasing mixed liquor suspended solids concentration, the α factor declines.”
- “...with increasing sludge retention time (SRT), the α factor enhances” (see Figure 26)
- “...daily or weekly changes in influent characteristics can lead to variations in α factors at the same treatment plant”.
- “...Other factors, at the microbiologic end, including DNA content or EPS production can also affect the α factor”.

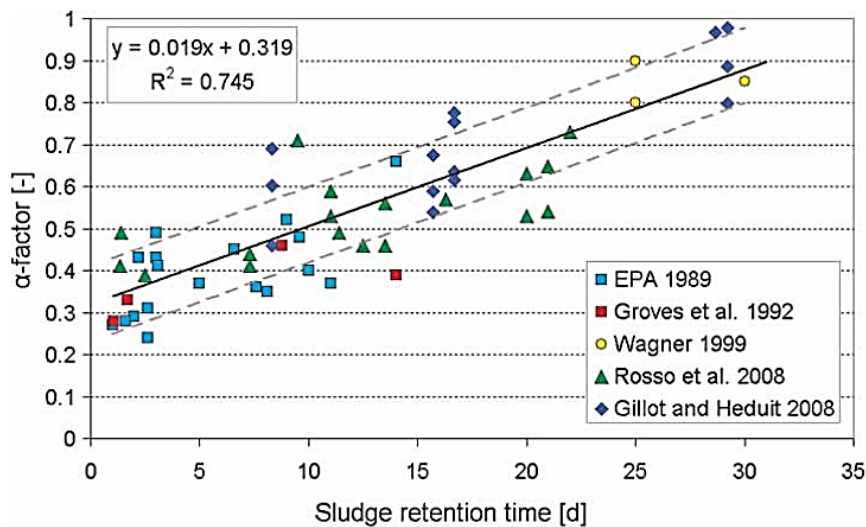


Figure 24 Alpha factor variations at different SRT (*J. Henkel et al., 2011*)

Henkel et al. (2011) presents the idea that the α factor is not a static but instead a dynamic operational parameter that can be manipulated in order to optimize the WWTP demands. In their research, the idea of the MLVSS (instead of MLSS) having a better correlation with α factor is presented; When compared to MLSS where the data points are very dispersed, a better linear fit can be observed by plotting the α factor against the MLVSS as shown in Figure 27 and Figure 28.

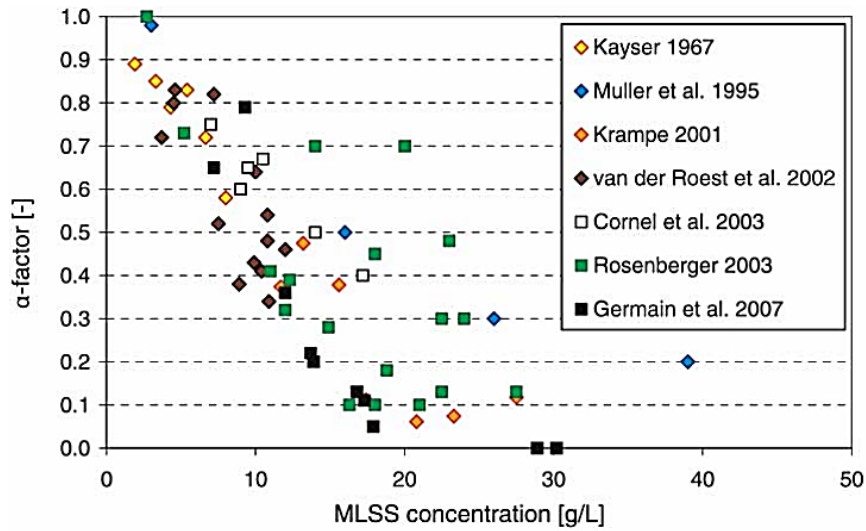


Figure 25 Alpha factor relation with MLSS (Henkel et al., 2011)

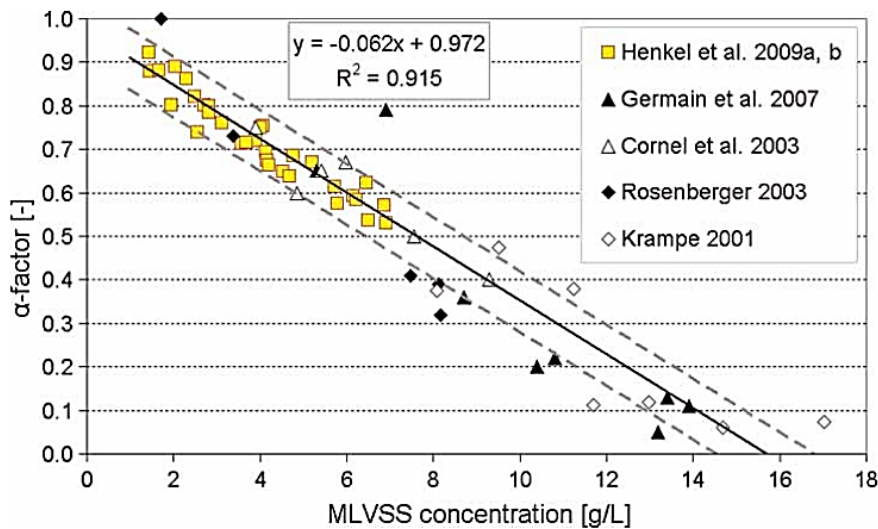


Figure 26 Alpha factor relation with MLVSS (Henkel et al., 2011)

The SRT-MLVSS- α relation is so clear that a series of expressions were proposed to calculate the resulting dynamic α as follows:

α factor as a function of the MLVSS:

$$\alpha \text{ factor} = -0.062 \cdot \text{MLVSS} + 0.972 \pm 0.070 \quad [10]$$

α factor as a function of the SRT:

$$\alpha \text{ factor} = 0.019 \cdot \text{SRT} + 0.533 \pm 0.093 \quad [26]$$

α factor as a combined function of both MLVSS and the SRT:

$$\alpha \text{ factor} = 0.51 - 0.062 \cdot MLVSS + 0.019 \cdot SRT \pm 0.114 \quad [27]$$

for MLVSS between and 12 g L^{-1} and SRT between 1 and 25 days

These expressions were derived from real data in pilot, laboratory and full-scale treatment systems.

In another publication, *Henkel et al. (2009)* presented his findings in a three-dimensional plot, see Figure 27. Besides the relation to SRT and MLVSS, an alpha factor dependency on the activated sludge floc free water content is proposed. The results presented by *Henkel et al. (2009)* show a relation between the volumetric fraction of free water in the biofloc, which is related to the available interface surface area for the mass transfer process to take place. See Figure 28. These recent findings open the possibility for other methods to be applied to overcome the aeration drawbacks present at high biomass concentrations, particularly low alpha factor, low OTE and low OTR, which are specifically the aim of this thesis research.

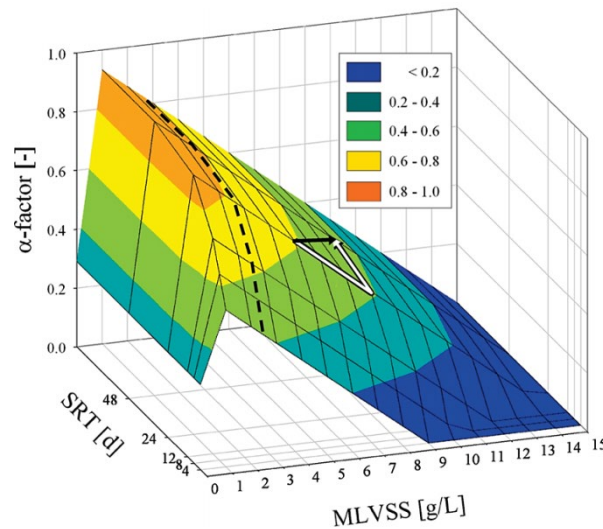


Figure 27 Alpha factor double dependency on SRT and MLVSS (*J. Henkel et al., 2009*)

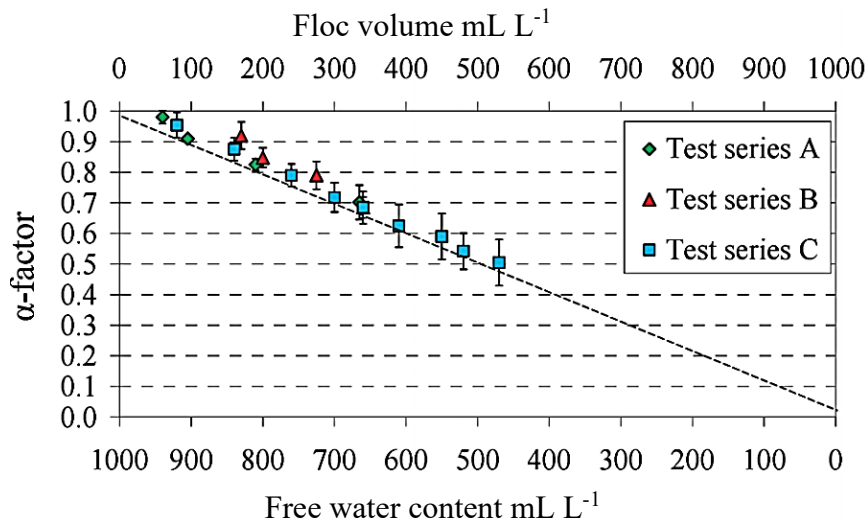


Figure 28 Alpha factor dependency on the activated sludge floc free water content (Henkel et al., 2009)

2.12. Activated sludge respiration

The biodegradation of organic matter present in wastewater via aerobic mineralization requires the presence of dissolved oxygen as the main driving parameter, meaning the oxygen is used by microorganisms to accomplish the whole purpose of water treatment via aeration processes which fuel the biochemical reactions necessary to do so. In order to measure the extent of the biological activity, a different parameter based on the rate at which oxygen is consumed (respirometry) was developed and the early developments were reported initially by many authors including (Montgomery, 1967; Heddle, 1980; Cailas & Gehr, 1989; Sollfrank & Gujer, 1990; Ros, 1993; Kappeler & Gujer, 1994; Spanjers et al., 1998).

The oxygen uptake rate (OUR) is a measure of the oxygen depletion in an oxygen saturated sludge sample under standard conditions (20°C, 1 atm). The OUR measuring procedure consists of registering the rate at which dissolved oxygen is depleted from the activated sludge sample. It is also referred to as the respirometry test of the activated sludge. The sludge respiration can be divided into two main phases, endogenous and exogenous. Endogenous respiration is the rate at which oxygen is consumed just for “maintenance” (without growth) once the soluble substrate has been depleted or consumed. The active or exogenous oxygen uptake rate considers both the maintenance and growth processes oxygen demand, i.e. in presence of soluble substrate (Ros, 1993).

Performing a respirometric test requires a series of conditions to make the results consistent and reliable, such conditions can be listed as:

- The air flow must be constant during the whole experiments set. This condition can be guaranteed by using the same aeration device under the same operational conditions.
- The volume and shape of the reactor must be known.
- The reaction tank-recipient has to be completely mixed.

- Temperature must be constant during the experiment, ideally 20 °C.

The measuring procedure must comply with the following stages:

1. Activated sludge saturation: The sludge sample is aerated allowing the biomass to take up the remaining substrate. Alternatively, the bulk sludge can be rinsed repeatedly with aerated tap water. Once the DO concentration is constant under constant aeration, an endogenous respiration rate condition is considered to be reached.
2. Determination of the sludge respiration rate: The oxygen uptake rate of an activated sludge suspension after the substrate has been depleted is considered constant, and can be expressed as:

$$r_s = k_L a (C_s - C_e) = \text{constant} \quad [28]$$

Where:

r_k	: sludge respiration rate (endogenous)	[mg L ⁻¹ min ⁻¹]
$k_L a$: liquid phase mass transfer coefficient	[min ⁻¹]
C_s	: DO saturation concentration	[mg L ⁻¹]
C_e	: DO saturation concentration on site	[mg L ⁻¹]

When C_e is constant (meaning endogenous respiration is achieved), the determination of the respiration rate can be performed as follows:

- i) Measure the change in DO concentration.
- ii) Calculate the endogenous respiration rate from the linear part of the DO concentration curve, this slope represents:

$$r_s = \left| \frac{dC}{dt} \right| \quad [11]$$

Where:

r_s	: OUR	[mg L ⁻¹ min ⁻¹]
-------	-------	---

for calculating the specific OUR (SOUR), the OUR is divided by the biomass content in terms of MLVSS (X_v)

$$SOUR = R_s = \frac{1.44 r_s}{X_v} \quad [\text{g gVSS}^{-1} \text{ day}^{-1}] \quad [12]$$

3. Liquid phase mass transfer coefficient ($k_L a$):

When the sludge suspension is at equilibrium without the addition of external substrate, the $k_L a$ can be calculated with the following expression:

$$k_L a (C_s - C_e) - r_s = 0 \quad [13]$$

$$k_L a (C_s - C_e) = r_s \quad [14]$$

$$k_L a = \frac{r_s}{(C_s - C_e)} \quad [15]$$

However, this expression is applicable for static or equilibrium conditions. In order to calculate the dynamic k_{La} the following procedure and expression can be used:

- i) Allow the activated sludge to take up the remaining oxygen until the DO concentration is below 0.5 mg/L.
- ii) Start the aeration device and record the increasing DO concentration.
- iii) From the linear part of the curve calculate the k_{La} using:

$$k_{La} = \frac{\ln \frac{(C_e - C_1)}{(C_e - C_2)}}{(t_1 - t_2)} \quad [16]$$

4. Measuring the sludge respiration:

Now, it is possible to measure the total AS respiration (endogenous+exogenous). Adding a known amount of COD on a saturated sludge sample will disturb the equilibrium causing the DO to drop. The change in DO can be expressed as:

$$\frac{dC}{dt} = k_{La} (C_s - C_e) - r_T \quad [17]$$

$$r_T = r_s + r_e \quad [36]$$

Where:

- r_T : total respiration rate (endogenous+exogenous) [mg L⁻¹ min⁻¹]
 r_s : sludge respiration rate (endogenous)
 r_e : sludge respiration rate (exogenous)

from the previous calculations, and knowing r_s , [$r_s = k_{La} (C_s - C_e) = \text{constant}$], the expression for the change in DO can be rearranged as:

$$\frac{dC}{dt} = k_{La} (C_s - C) - k_{La} (C_s - C_e) - r_e \quad [37]$$

or

$$r_e = k_{La} (C_e - C) - \frac{dC}{dt} \quad [38]$$

At the lowest point of the DO consumption curve in the respirogram, t_2 in Figure 29, all the added substrate has been used up and the change in DO concentration (dC/dt) will be zero; therefore, the exogenous respiration rate can be expressed as:

$$r_e = k_{La} (C_e - C_{min}) \quad [39]$$

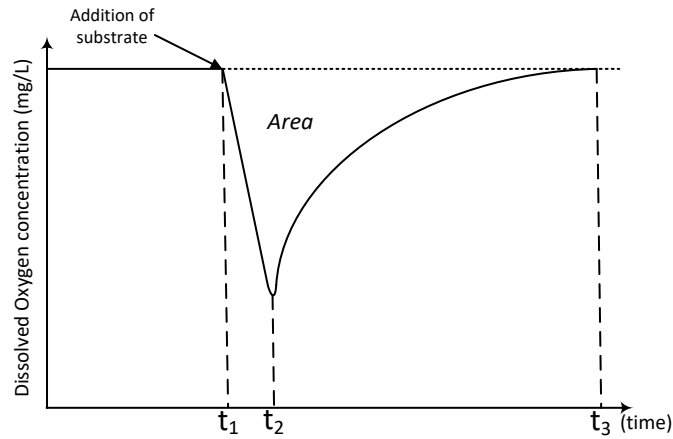


Figure 29 Schematic of a typical respirogram curve with substrate addition. Adapted from (Ros, 1993)

And, by integrating,

$$\int_0^{\infty} r_e dt = k_L a \int_0^{\infty} (C_e - C) dt \quad [18]$$

And since all of the substrate will be used from t_1 to t_3 , being t_3 the time when DO concentration equals DO at t_1 (see Figure 31), or in other words when DO concentration is in equilibrium again at the end of the respirometry curve, the previous equation becomes:

$$\int_{t_1}^{t_3} r_e dt = k_L a \int_{t_1}^{t_3} (C_e - C) dt \quad [19]$$

After calculating the *Area* highlighted in Figure 29, it is possible to apply the following equation:

$$\int_{t_1}^{t_3} r_e dt = k_L a \cdot A - BOD \quad [20]$$

Additionally, the following parameters can be derived from the previous experiments:

Time required to reach the minimum DO concentration:

$$\Delta t = t_2 - t_1 \quad [21]$$

Time to degrade the added substrate:

$$t_s = t_3 - t_1 \quad [22]$$

Maximum difference in DO concentration:

$$\Delta C_{max} = C_e - C_{min} \quad [23]$$

Exogenous OUR:

$$OUR_e = r_e = k_L a \cdot \Delta C_{max} \quad [\text{mg L}^{-1} \text{ min}^{-1}] \quad [46]$$

Exogenous SOUR:

$$SOUR_e = R_e = \frac{r_e}{X_v} \quad [\text{g gVSS}^{-1} \text{ d}^{-1}] \quad [47]$$

All the previous equations (from Equation 35 until Equation 47) and descriptions have been adapted from *Ros (1993)*.

3. Hydrocyclone technology

The hydrocyclone principle uses the difference in specific weight and centrifugal forces to separate a fluid composed by multiple phases. This devices can reach shear forces equivalent to 10,000 the gravitational acceleration (*McKetta, 1996*), making them robust and especially compact. Due to its remarkable compactness, it has been extensively used in the oil and gas industry offshore platforms for separation of drilling sludge-water-gas mixtures. A schematic representation of a hydrocyclone arrangement is shown in Figure 30.

Hydrocyclones have been used for decades in a very wide range of industrial applications, some of the main uses are:

- Liquid clarification
- Slurry thickening
- Solids washing
- Solids classification by particle size
- Solids sorting according to density or particle shape.
- Particle size measurement (offline or online)
- Degassing of liquids
- Separation of two immiscible liquids
- Oil water separation for oil and gas industry

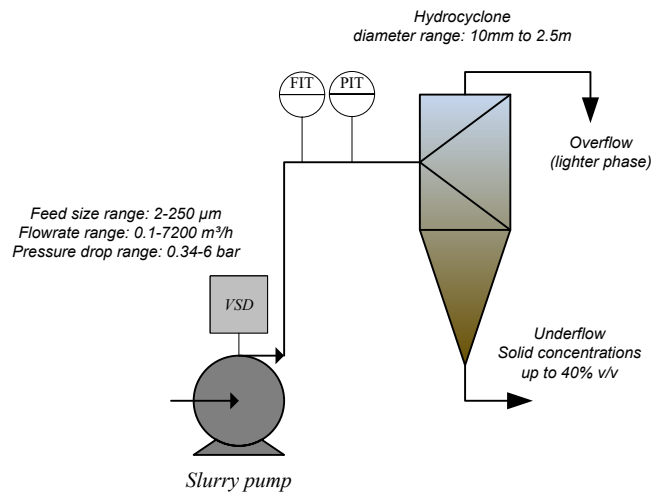


Figure 30 Schematic of the Hydrocyclone configuration

There are different theories and approaches describing the separation process in a hydrocyclone, the most relevant are:

1. Simple, fundamental theories disregarding the flow rate effect, the feed concentration and the feed size distribution.
2. The two-phase flow theory considering the effect of the feed concentration and the mean feed particle size.
3. The crowding theory considering the strong effect of the underflow orifice size
4. Empirical models based mostly on regressions analysis of measured data; however, these models are only applicable for the specific system under testing.
5. The chemical engineering approach based on dimensionless groups combining all of the previously mentioned theories.

The main advantages of hydrocyclones are:

- Have been extensively tested and used in real applications with high solid concentrations.
- Can be custom made for very specific desired particle cut size, removal efficiency, velocity and pressure drop.
- Extensive research has been done on hydrocyclones and there are several computational fluid dynamics tools for process simulation.
- It is simplified low-cost equipment with very low maintenance requirements and no moving parts.
- Due to the very high centrifugal forces that can be applied the hydrocyclone footprint is minimum compared to other solid liquid separation devices.

The collection efficiency increases with the particle size and the solids concentration (see Figure 29 and Figure 30). As depicted in these two figures, nearly all of the particles are removed when the particle size approaches to 10 μm and the solids concentration is higher

than 2 g L^{-1} (kg m^{-3}), this condition is particularly convenient for the high MLSS activated sludge separation.

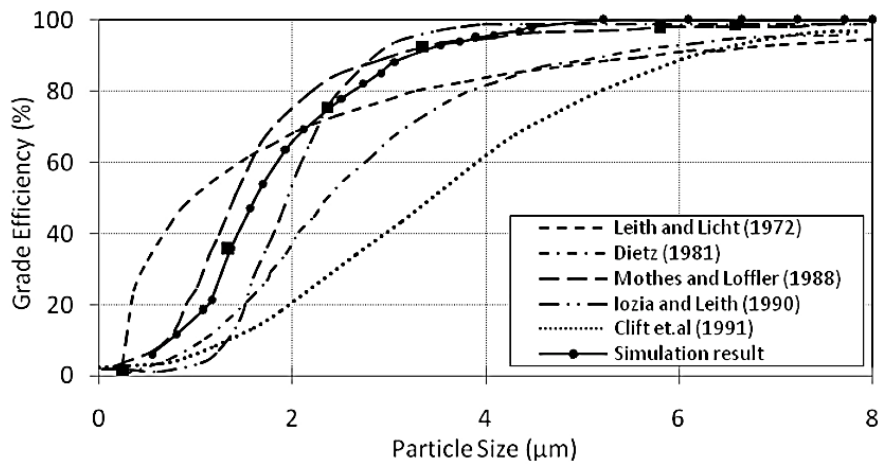


Figure 31 Collection efficiency comparison (Utikar, 2010)

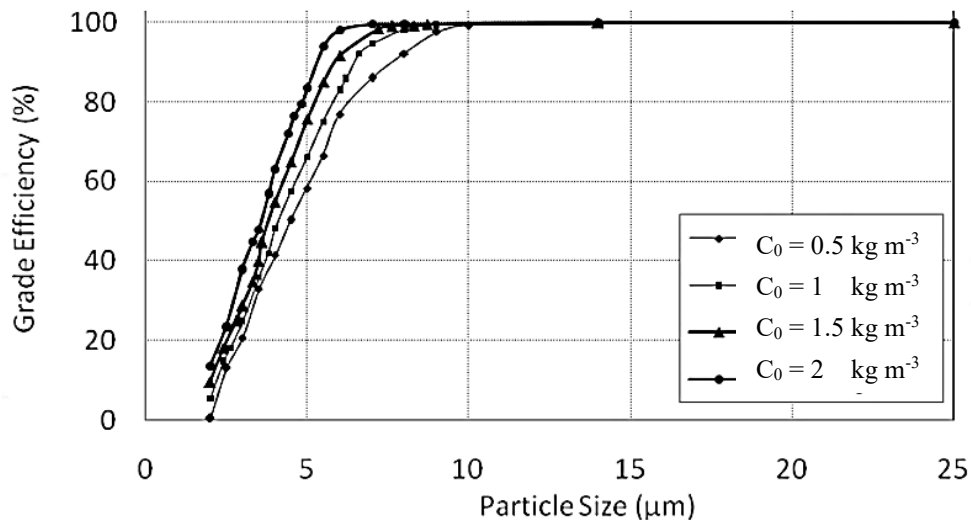


Figure 32 Separation efficiency at different inlet particle concentrations (Utikar, 2010)

During this research, centrifugation tests were performed to mimic the effect of a hydrocyclone in testing the potential of mixed liquor pre-conditioning prior to the membrane filtration process. The aim of this conditioning process is to separate the solid phase (activated sludge) from the liquid one (mixed liquor supernatant). The obtained results and a more detailed description of the testing is given in Chapter 5.

4. Research Scope

Despite MBRs being operated in MLSS concentrations higher than 20 g L^{-1} in some industrial applications for many years, the scientific literature available about this is limited to some studies aimed at comparing their performance in removing organic load while using conventional aeration methods. Besides this research thesis, there is only one other academic research that focused on the evaluation of an MBR operated at high MLSS conditions providing oxygen with a supersaturation method different than the Speece cone, the so called super saturated dissolved oxygenation system (SDOX) *Kim et al., (2020, 2021, 2022)*, therefore there is still a need for deeper understanding of such supersaturation methods and how they can contribute to the advance of the wastewater treatment field and membrane bioreactors. In addition, the membrane fouling studies that are available in literature, focus mostly on finding methods for the mitigation of fouling under more conventional operational conditions, meaning lower solids concentrations below 15 g L^{-1} and with conventional aeration methods. It is important to remark the need for scientific studies that evaluate the changes in sludge rheological properties namely in viscosity, filterability and particle size distribution when the aeration method is changed from fine bubble diffusers to sidestream supersaturation; Mainly due to the high shear stress the sludge is exposed to in the recirculation through the saturation device (the Speece cone) which can have a grinding effect thus shifting the PSD towards smaller sizes that can increase membrane fouling via pore blocking mechanism. On the other hand, the higher solids content that occur at high MLSS concentrations changes radically the sludge viscosity and therefore all membrane filtration dynamics. It is for these reasons that this research thesis can contribute to a better understanding of such phenomena by answering the following research questions:

Q1 :What are the variables that can lead to improving the oxygen transfer in a high MLSS membrane bioreactor when using a sidestream supersaturation system such as the Speece cone?

Q2 :To which extent can the Speece cone system improve the standard oxygen transfer efficiency (SOTE) and the alpha factor in a high MLSS MBR?

Q3 :Is there a net improvement in the high MLSS activated sludge respiration when more oxygen is available (not limited) by introducing a superoxygenation method such as the Speece cone?

Q4 : to which extent can the mixed liquor suspended solids be reduced in high MLSS MBR activated sludge when introducing a pre-conditioning stage by means of centrifugation?

Q5 :How can the membrane fouling be mitigated in a high MLSS MBR using centrifugation methods such as hydrocyclone?

This research aimed at evaluating the ultra-compact, high-MLSS-MBR-Speece cone configuration as an alternative for portable sanitation by examining its limitations regarding the negative impact derived from the operation under high solids concentrations regarding the decline in oxygen transfer with conventional aeration methods and the excessive fouling which translates in reduced permeate production.

The overall objective of this research is to examine the operational performance of an aerobic membrane bioreactor operating at biomass concentrations higher than usual in municipal wastewater treatment, meaning above 15 g L^{-1} . This research focused on evaluating the performance of the high MLSS MBR treatment capabilities in regards of the two main process variables which are currently hindering a further footprint reduction of such bioreactors. Such process variables are the oxygen transfer to the mixed liquor and the membrane performance under the high MLSS concept.

The specific research objectives are:

- i. To assess the MBR performance in terms of effluent quality and treatment efficiency under increasing biomass concentrations from 5 and up to 40 g L^{-1} .
- ii. To characterize the Speece cone operational performance and evaluate the applicability of this aeration method under the high MLSS-Speece cone MBR concept.
- iii. To monitor the biomass activity using respirometry tests (OUR) under high MLSS ($>15 \text{ g L}^{-1}$) and supersaturation conditions, meaning with dissolved oxygen concentrations higher than the saturation concentration ($>9 \text{ mg L}^{-1}$).
- iv. To determine the process limitations of main operational parameters (membrane flux, biomass concentration, OTE, SOTR, alpha factor) for the operation of the high MLSS- Speece cone MBR.
- v. To assess the sludge filterability, viscosity and particle size distribution at high biomass concentrations and supersaturated aeration.
- vi. To assess the membrane fouling rates, membrane resistance and permeability under the Speece cone aeration conditions and high biomass content.
- vii. To evaluate the impact and potential benefits (on fouling mitigation) of introducing an activated sludge pre-conditioning stage for centrifugal solid-liquid separation under high MLSS MBR operation.

This research addresses the following hypotheses:

1. (H1) The use of alternative aeration methods such as sidestream supersaturation (Speece cone) can enhance the oxygen transfer at high MLSS concentrations.
2. (H2) Higher oxygen transfer efficiency (OTE) and alfa factors can be achieved when using the Speece cone for side-stream superoxygenation as compared to conventional aeration methods.
3. (H3) The overall oxygen transfer performance of a speece cone can overcome the mass transfer limitations in MLSS concentrations above 15 g L^{-1} .

4. (H4) By integrating a hydrocyclone for solid-liquid separation (sludge pre-conditioning), it is possible to introduce a low MLSS stage for membrane filtration with reduced fouling rates and enhanced membrane permeability.
5. (H5) Sludge pre-conditioning using a hydrocyclone in a separate membrane tank offers an alternative for membrane fouling mitigation in high MLSS MBR operation.

5. Research approach

The Speece cone-MBR Sc-MBR pilot setup was tested at different flowrate, pressure and oxygen flowrate combinations in order to characterize its oxygen transfer capabilities under high MLSS conditions in different phases as follows:

- The Sc-MBR was operated in steady state treating municipal wastewater at MLSS concentrations of up to 28 g L^{-1} at sludge retention times (SRT)s ranging from 30 to 35 days. The MBR performance was evaluated by monitoring the influent and effluent water quality, the membrane permeability, the sludge filterability, the dissolved oxygen (DO) concentration, and the OUR. (CHAPTER_2)
- The performance of the pilot scale Sc-MBR was examined in clean water and activated sludge in order to determine the overall oxygen mass transfer rate coefficient (k_{La} , h^{-1}), the standard oxygen transfer rate (SOTR, $\text{kg O}_2 \text{ d}^{-1}$), and the standard oxygen transfer efficiency (SOTE, %). In addition, the alpha factor (α) was determined for activated sludge with suspended solids concentration of approximately 5 g L^{-1} . (CHAPTER_3)
- The Sc-MBR ability to dissolve oxygen into high MLSS activated sludge was evaluated in the range of 5 to 30 g L^{-1} . The liquid phase mass transfer coefficient k_{La} , Standard oxygen transfer efficiency (SOTE), Standard aeration efficiency (SAE), alpha factor, endogenous respiration, sludge viscosity and particle size distribution were determined. The process variables influencing the process included the High purity oxygen (HPO) flow to the cone, pressure and inlet velocity. (CHAPTER_4).

The principle for improving the membrane performance while operating at high MLSS relies on performing mixed liquor pre-conditioning before the membrane filtration process. This is, decoupling the high MLSS stage (the aerobic bioreactor) from the filtration process (the membrane tank) by introducing a pre-conditioning step between the two, in order to lower the fouling rates, and possibly reducing the need (and cost) for additional scouring air which would be required with mixed liquor without pre-conditioning and therefore higher solids and viscosity. This pre conditioning step can be completed by using a hydrocyclone that allows the separation of a heavier mixed liquor fraction (to be recirculated to the aerobic tank) from its supernatant (the pre-conditioned mixed liquor), which will reach the membrane tank in order to achieve a better membrane

performance with diminished fouling and increased permeate production that are comparable to low MLSS filtration conditions, while still keeping the high conversion rate in the aerobic bioreactor.

This research aimed to perform the preliminary experiments for solid-liquid separation at laboratory scale using a laboratory scale centrifuge as an analogue for a hydrocyclone in order to establish the operational range for rpm and separation characteristics that could be achieved as follows:

- Short term filtration tests were performed using three types of submerged membranes (flat sheet, tubular and ceramic) in the high MLSS range from 5 to 40 g L⁻¹. The membrane permeability, membrane resistance, fouling rates and fouling recovery were evaluated while characterizing the activated sludge particle size distribution and viscosity (CHAPTER_5).
- Additional laboratory scale experiments on mixed liquor pre-conditioning were performed using centrifugal methods to mimic the solid-liquid separation process in order to obtain a supernatant, measuring its particle size distribution and viscosity to determine the effect of introducing such separation step as a fouling mitigation alternative (CHAPTER_5).

Introduction

The experimental work of this research took place at the Harnaschpolder wastewater treatment plant in Delft, The Netherlands (Figure 33), more specifically at the research (pilot) hall (Figure 34) which is part of the Delft Blue Innovations (DBI) research initiative integrated by diverse institutions including research, governance and water service companies www.delftblueinnovations.nl. DBI is managed and Evides, Delfland and Delfluent Services. Other partners include TU Delft, UNESCO-IHE, KWR, Delft Blue Water and RINEW.



Figure 33 The Harnaschpolder wastewater treatment plant. Delft, The Netherlands.



Figure 34 Delft Blue Innovations (DBI) pilot hall at the Harnaschpolder WWTP.

2

ASSESSING THE PERFORMANCE OF AN MBR OPERATED AT HIGH BIOMASS CONCENTRATIONS

This Chapter is based on Barreto, C. M., García, H. A., Hooijmans, C. M., Herrera, A., & Brdjanovic, D. Assessing the performance of an MBR operated at high biomass concentrations. *International Biodeterioration & Biodegradation*, 119, 528-537.
doi:<https://doi.org/10.1016/j.ibiod.2016.10.006>

Abstract

Reducing the footprint requirements of membrane bioreactors (MBR)s can both decrease the surface area needs for new wastewater treatment plants (WWTP) and increase the treatment capacities of existing WWTPs at a given surface area. In addition, it may promote the development of movable/portable containerized MBRs for a diverse range of wastewater treatment applications. Applications may include the provision of municipal/industrial wastewater treatment in remote areas without sewerage, and the provision of sanitation services under challenging site-specific conditions such as after the occurrence of a human-made or a natural disaster. The reduction of the footprint requirements of MBRs is constrained by the maximum amount of biomass that can be accommodated in the aerobic basin. The biomass concentration is mainly limited by the extremely low oxygen transfer efficiency (OTE) experienced by conventional aeration bubble diffuser systems at mixed liquor total suspended solids (MLSS) concentrations higher than 20 g L^{-1} . Another potential limitation for the operation of MBRs at such high MLSS concentrations is the reduction on the membrane permeability due to excessive fouling. A pilot MBR with a treatment capacity of $1 \text{ m}^3 \text{ d}^{-1}$ was installed at the research hall facilities at the Harnaschpolder wastewater treatment plant in Delft, The Netherlands. The MBR was operated at MLSS concentrations of up to 28 g L^{-1} at sludge retention times (SRT)s ranging from 30 to 35 days. The MBR was provided with a Speece cone concentrated oxygen delivery system to overcome the oxygen transfer limitations of conventional bubble diffuser aeration systems at high MLSS concentrations. The MBR performance was evaluated by monitoring the influent and effluent water quality, the membrane permeability, the sludge filterability, the dissolved oxygen (DO) concentration, and the OUR. The Speece cone proved to be effective in delivering enough oxygen to maintain DO concentrations in the MBR of approximately 2 mg L^{-1} at MLSS concentrations of up to 22 g L^{-1} . OUR values above $200 \text{ }^{-1} \text{ h}^{-1}$ were observed at 14 g L^{-1} MLSS and higher than $300 \text{ mg L}^{-1} \text{ h}^{-1}$ at 22 g L^{-1} MLSS. The MBR exhibited COD removal efficiencies of up to 99% even at a hydraulic retention time (HRT) as low as 3.7 hours. A reduction in permeability from 33 to 11 lmh bar^{-1} was observed when the MLSS concentrations increased from 18.7 to 27.8 g L^{-1} . Sludge filterability values expressed as the added resistance (ΔR_{20}) fell in the range of "poor filterability" for all the evaluated operational conditions; however, a lower filtration resistance in the range of "moderate filterability" at approximately 23 g L^{-1} MLSS was noticed. The experimental results suggested that at the evaluated experimental conditions the existent limitations on poor oxygen transfer and low permeability when operating a MBR at high MLSS concentrations can be overcome; therefore, the footprint requirements of MBR systems may be further reduced.

1. Introduction

Considering all the existent alternatives for the provision of wastewater treatment, MBRs present some additional advantages including the production of a high quality effluent suitable for water reuse, (Stephenson, 2000; Melin et al., 2006; Arceivala, 2008; Henze et al., 2008; Judd, 2008, 2010; Hai & Yamamoto, 2011), the reliability of the technology, the potential production of small amounts of already stabilized sludge, and the operational flexibility to adjust to changes in the organic loads, among others. The reduction of the footprint requirements of membrane bioreactors MBRs can allow both the reduction of the surface area needs when constructing new wastewater treatment plants (WWTP)s, and the increase of the treatment capacities of existing WWTP at a given surface area. In addition, the achievement of an additional footprint reduction on MBRs may promote the development of movable/portable containerized MBRs for a diverse range of applications including the provision of municipal/industrial wastewater treatment in remote areas without sewerage and the provision of sanitation services under challenging site-specific conditions such as after the occurrence of a human-made or a natural disaster. However, the reduction of the footprint requirements of MBRs is constrained by the maximum amount of biomass that can be accommodated in the aerobic basin.

The maximum biomass concentration that can be achieved in a MBR is mainly limited by the extremely low oxygen transfer efficiency (OTE) experienced by conventional aeration systems such as fine and coarse bubble diffusers at MLSS concentrations higher than 20 g L^{-1} (Germain et al., 2007). Another limitation for the operation of MBRs at such high MLSS concentrations is the reduction on the membrane permeability observed due to excessive fouling. This drastic decrease in permeability is caused mainly by the accumulation of fouling substances and the increased mixed liquor viscosity (Trussell et al., 2007). The relation between the potential benefits of operating a high MLSS MBR and the negative impact on the system permeability has been addressed in the literature as the “*Capex-Opex dichotomy*” (Judd, 2008). Therefore, and in order to avoid these adverse conditions, conventional MBR systems are currently designed to operate at MLSS concentrations of approximately 10 g L^{-1} setting the footprint requirements of this technology.

Oxygen transfer in aerobic wastewater treatment processes has been extensively addressed in the past decades. Several studies demonstrated that both the suspended solids as well as the mixed liquor viscosity negatively affect the oxygen transfer process (Cornel et al., 2003a; Germain & Stephenson, 2005; Germain et al., 2007; Trussell et al., 2007; Zhichao Wu et al., 2007; Moreau et al., 2009a). Krampe and Krauth (2003) reported a decrease on the OTE as the biomass concentration increased. The evaluation was conducted on a biological system provided with a conventional fine bubble diffuser up to biomass concentrations of approximately 28 g L^{-1} . Alpha factors as low as 0.1 were reported at a 20 g L^{-1} MLSS concentration demonstrating an extremely low OTE at the evaluated conditions. A study conducted by Jochen Henkel et al. (2009) investigated the OTE of fine and coarse bubble diffusers at MLSS concentrations ranging from 4.7 to

19.5 g L⁻¹ under different air flow conditions and operating the biological systems at high SRTs. A decrease on the alpha factor was reported as the biomass concentrations (expressed as MLSS) increased. In addition, a more direct correlation was noticed between the decrease of the alpha factor and the increase of the mixed liquor volatile suspended solids (VSS). The authors concluded that the mixed liquor VSS concentration in the reactor is the main factor impacting on the oxygen transfer process. At mixed liquor VSS concentrations higher than 20 g L⁻¹ negligible alpha factors were reported; therefore, very little DO at a very low OTE could be supplied at the evaluated experimental conditions. The rheological and physiological properties of MBRs were investigated by Zhichao Wu et al. (2007); the authors demonstrated that the MLSS concentration has a direct impact on the mixed liquor apparent viscosity, which consequently affects the oxygen diffusion process. The effect of the high MLSS concentration on the apparent viscosity was also demonstrated by Trussell et al. (2007). The negative impact of the apparent viscosity on the oxygen transfer process was reported in a more recent publication by Durán et al. (2016) for fine bubble diffuser aeration. In a comparative study carried out by Krampe and Krauth (2003), different bubble diffuser aeration systems were evaluated at MLSS concentrations of up to approximately 20 g L⁻¹. In accordance with previously reported studies, the authors concluded that the alpha factor decreases exponentially with increasing MLSS concentrations. In addition, an increase on the viscosity was observed as the MLSS concentration increased. The authors proposed that the increased viscosity of the mixed liquor could promote the formation of large bubbles via coalescence resulting in a reduced available interfacial gas-liquid area negatively impacting the oxygen transfer process. Even though several studies were carried out evaluating the OTE on biological systems at different MLSS conditions, there is still a need and a clear interest for advancing on alternative oxygen delivery systems for efficiently supplying DO; particularly, when designing biological systems to operate at higher than usual MLSS concentrations.

Alternative aeration systems are needed to cope with the high oxygen demands and low OTEs commonly observed on MBRs operated at high MLSS concentrations. The oxygen transfer rates and OTEs of innovative concentrated oxygen delivery systems such as the super saturated dissolved oxygenation system – (SDOX) were recently evaluated by Kim et al. (2015). The SDOX system recirculates activated sludge through a chamber that is pressurized with pure oxygen. The activated sludge is introduced into the pressurized chamber through a nozzle generating a mist enhancing the gas-liquid interaction; consequently, the oxygen mass transfer between the pure oxygen gas phase and the mixed liquor solution is maximized. The authors reported similar alpha factors compared to conventional bubble diffuser systems; however, the SDOX system exhibited nearly 100% OTEs when working at MLSS concentrations of up to 40 g L⁻¹. That is, nearly all of the oxygen supplied to the pressurized chamber ended up as DO in the biological reactor. In addition, such aeration systems are not subject to clogging or scaling as it is the case for

membrane fine bubble diffusers. The clogging or scaling of the diffuser reduces the OTE even further causing an increased backpressure in the air distribution line (Garrido-Baserba et al., 2016). Another concentrated oxygen delivery technology, the Speece cone system, may present a feasible alternative for providing the required DO in biological systems working at high MLSS concentrations. The Speece cone system has been commonly used in the past for hypolimnetic aeration applications mainly for bioremediation of lakes and other water courses (KI Ashley et al., 2008). The Speece cone system recirculates the mixed liquor from the aerobic basin of the reactor through a pressurized inverted cone structure. Pure oxygen gas is directly supplied at the top of the cone and is dissolved into the mixed liquor, which is introduced into the top of the pressurized inverted cone without the use of any nozzle, as compared to the situation previously described for the SDOX system. For this reason, the Speece cone system minimizes the head losses of the system allowing to process large volumes of mixed liquor without large energy expenditures (McGinnis & Little, 1998). The improvement on the oxygen mass transfer observed at the Speece cone is based on both the high pure oxygen pressure conditions inside the cone, and on the specially designed cone geometry. That is, based on the geometry of the inverted cone and on the selected mixed liquor flow rate through it, a particular downward velocity can be set for the mixed liquor. The mixed liquor velocity at the top of the cone is higher than the pure oxygen bubbles buoyancy due to the small cross-sectional area. Therefore, the oxygen bubbles are forced down inside the cone to be in contact with the mixed liquor. As the oxygen bubbles and mixed liquor travel down, the cross-sectional area of the inverted cone increases and the mixed liquor's downward velocity decreases preventing the oxygen bubbles from escaping the cone at the bottom of the structure. Therefore, the contact time between the pressurized pure oxygen and the mixed liquor inside the cone is maximized enhancing the oxygen mass transfer into the mixed liquor. The Speece cone system may present a feasible alternative for providing DO in biological systems working at high MLSS concentrations.

The operational performance of MBRs is commonly assessed in terms of water quality, permeability, transmembrane pressure (TMP), membrane fouling rates, and more recently in terms of sludge filterability. Previous research on membrane fouling has focused on evaluating several factors influencing this phenomenon such as: the accumulation of substances and particles on the membranes (Delrue et al., 2011), the effects of the SRT on fouling (R. Van den Broeck et al., 2012), differences between suspended and attached microorganisms growth (Jamal Khan et al., 2011), the application of membrane coating for fouling reduction (Deowan et al., 2016), and the influence of operational conditions and membrane cleaning routines, (Delrue et al., 2011) among others. A considerable number of studies were conducted on membrane fouling (Drews, 2010); however, limited research was carried out evaluating the effects of high biomass concentrations above 20 g L⁻¹ MLSS on membrane fouling. Some of these studies included the evaluation of the biomass characteristics on membrane fouling (C.-H. Chen et al., 2015), the fouling characteristics at different MLSS and COD loadings (S. Lee & Kim, 2013), and the fouling mitigation mechanisms using bio carriers (F. Chen et al., 2016). However, most

of these studies were carried out by severely diluting or concentrating the sludge samples to reach the desired MLSS concentrations. This dilution or concentration processes could affect some properties of the sludge when comparing to the fresh and naturally occurring high MLSS sludge from a full-scale system. That is, the manipulation of the sludge could modify its structure for instance by de-flocculation (Maria Lousada-Ferreira et al., 2015), or could promote the release of polymeric materials (Drews, 2010). Hence, there is a need for better understanding the performance of the membrane filtration component on MBRs operating at high MLSS concentrations using fresh activated sludge produced *in situ*.

The objective of this study was to evaluate the technical feasibility of a MBR operated at high MLSS concentrations provided with a Speece cone concentrated oxygen delivery system. This study evaluated the overall performance of the MBR at different MLSS concentrations by monitoring the influent and effluent water quality, the membrane permeability, the sludge filterability, DO concentration, and the OUR. In addition, this study assessed to which extent the footprint of MBRs can be reduced by increasing the MLSS concentration in the aeration basin.

2. Materials and methods

This research evaluated the performance of a pilot MBR operated at MLSS concentrations ranging from approximately 7 to 28 g L⁻¹. Specific MLSS concentrations set points were reached in that MLSS range by modifying the operational conditions and loads to the MBR system. The system performance was evaluated at each of the targeted MLSS concentration set points.

2.1 Pilot MBR-Setup description

A pilot MBR with a standard treatment capacity of approximately one m³ day⁻¹ was operated at the Delft Blue Innovations (DBI www.delftblueinnovations.nl) research hall at the Harnaschpolder wastewater treatment plant in Delft, The Netherlands. The pilot MBR consisted of the following main components: an anoxic chamber (volume 0.25 m³), an aerobic chamber (volume 0.85 m³), a permeate collection tank (volume 0.2 m³), a low-pressure blower (SECOH, EL-S-250; USA), and a bidirectional pump for permeate production and membrane backwash (Liverani EP NEOS; Italy). The MBR was provided with a submerged ultrafiltration membrane module made of single tubular polyvinylidene fluoride membrane elements with an average pore size of 0.01 µm and a total filtration area of 20 m² (Memos; Germany). A coarse bubble distribution manifold was installed at the bottom of the membrane module for membrane scouring. A ceramic fine bubble diffusers was introduced for supplying much of the DO needs to the MBR. However, anticipating the low OTE commonly observed by fine bubble diffusers when operating at high MLSS concentrations (*Krampe & Krauth, 2003*), a concentrated oxygen delivery system – Speece cone was provided as an additional source of DO. A schematic of the experimental MBR system is presented in Figure 35.

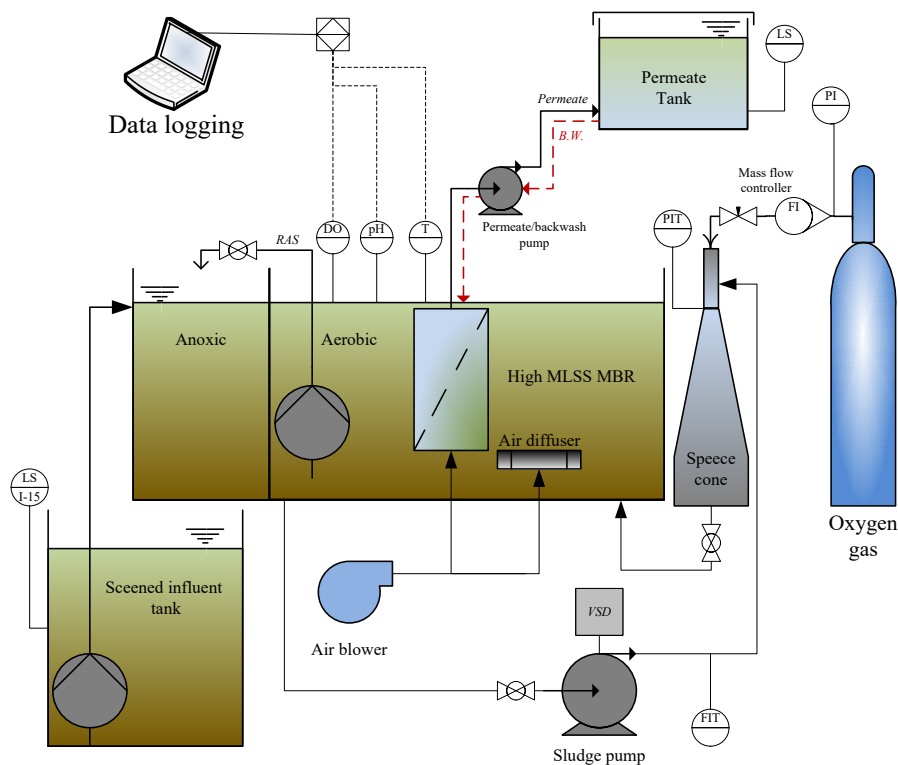


Figure 35 Schematic process flow diagram. High MLSS MBR-Speece cone pilot setup (VSD: Variable speed drive, PIT: Pressure indicator transmitter, FI: Flow indicator, LS: Level switch, B.W: Backwash, DO: Dissolved oxygen, RAS: Return activated sludge, T: Temperature)

The Speece cone system continuously recirculated the mixed liquor from the aeration basin of the MBR into a pressurized inverted cone by means of a progressive cavity pump (Netzsch, NEMO NM045BY02512B; Germany). At the cone, the mixed liquor got in contact with pressurized pure oxygen; therefore, the oxygen gas was dissolved into the mixed liquor for supersaturation. The oxygen flow to the cone was regulated by means of a gas mass flow controller (Alicat, MC-5SLPM-D; USA). The amount of DO transferred by the Speece cone system was governed by two main parameters: the pressure, and the recirculation flowrate through the cone.

During the entire execution of this research the MBR was fed screened (0.45 mm) non settled municipal wastewater. The membrane operating cycles were controlled by a PLC (Mitsubishi FX3G-24M, USA). One cycle corresponded to 10 minutes of permeate production and one minute backwash. A 10 minute long backwash was performed automatically every 50 cycles.

2.2 Operational conditions

The pilot MBR was seeded with returned activated sludge from the Harnaschpolder wastewater treatment plant, and it was operated for approximately six months. Different

operational conditions were established to achieve the targeted/desired MLSS concentration set points as indicated below. A unique numerical identification code (IDs 1 to 13) was assigned to each targeted MLSS concentration (MLSS set point) which corresponds to a particular combination of operational parameters such as SRT, influent flowrate, influent strength, and aerobic basin volume.

(i) First phase (numerical identification code IDs 1 to 5 corresponding to $MLSS_{target}$ concentrations of: 8, 9, 10, 15, and 18 g L^{-1}). The increase on the MLSS concentrations for this phase was achieved by setting the SRT at 30 days; no activated sludge was purged, while the influent flowrate and the reactor volume were kept constant at $3.5 \text{ m}^3 \text{ d}^{-1}$ and 0.85 m^3 , respectively. During this phase the influent COD concentration was not modified.

(ii) Second phase (IDs 6 to 11 corresponding to $MLSS_{target}$ concentrations of: 23, 24, and 36 g L^{-1}). Once the SRT was fixed at 30 days, the MLSS concentration was increased by modifying the applied COD load to the MBR. In addition, the MBR aerobic volume was reduced to 0.73 m^3 to achieve the desired MLSS concentrations.

(iii) Third phase (IDs 12 to 13 corresponding to $MLSS_{target}$ concentrations of: 27, and 29 g L^{-1}). The MLSS targeted concentrations in this phase were achieved by applying higher COD loads by using sugar cane molasses. The reactor volume was returned back to its original value (0.85 m^3), and the flowrate was halved to approximately $2 \text{ m}^3 \text{ d}^{-1}$. In addition, the SRT was increased to 35 days.

The MLSS concentration in the MBR can be increased by changing any of the following operational parameters: the SRT, the influent load (flow and COD concentration), and/or the reactor volume. The achievement of the desired MLSS set points requires changing at least one of the operational parameters of the MBR. After reaching the desired SRT of approximately 30 days, it was decided to keep the SRT as constant as possible for the entire MLSS range to have a similar sludge with a similar biomass active fraction for the entire evaluated MLSS range allowing to perform a better comparison of the performance of the MBR-Speece cone systems.

The working operational conditions for each operational set point or IDs are depicted in Figure 36.

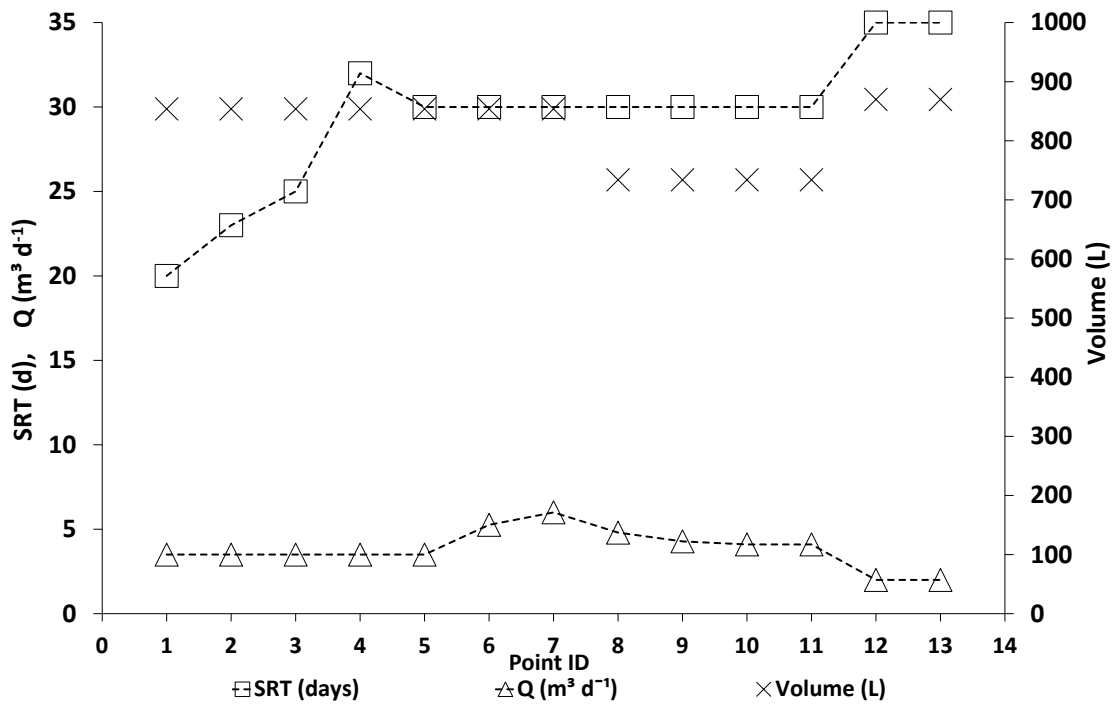


Figure 36 MBR operational conditions including solid retention time (SRT), flow rate (Q), and aerobic chamber volume.

2.3 System evaluation

The MBR was evaluated at the different MLSS concentration set points as previously indicated and shown in Figure 36 by monitoring the following parameters:

(i) Transmembrane pressure (TMP) and MBR permeability. The reported TMP values correspond to the mean value in a series of 50 cycles for each operational MLSS set point. From the reported TMP and applied flux, the operational permeability was calculated. The permeability was later normalized at 25°C and reported as a normalized operational permeability (OP_n). Neither fouling mechanisms nor fouling rates were assessed during this study; the TMP and OP_n were reported as indicators of the overall performance of the system.

(ii) Permeate water quality (in terms of COD removal)

(iii) Sludge activity by measuring the OUR

(iv) Speece cone theoretical DO delivery capabilities

(v) Sludge filterability

In addition, a comparison of the footprint requirements was carried out in terms of the required volume for a conventional activated sludge systems (CAS), a conventional MBRs, and the high MLSS MBR concept.

2.4 Analytical procedures

Water quality analyses were performed following the standard methods for the examination of water and wastewater (AWWA, 2014). Chemical oxygen demand (COD) and total suspended solids (TSS) were determined at each MLSS concentration set point. The reported values correspond to the average from duplicate determinations for TSS and volatile suspended solids (VSS) and triplicate determinations for COD. DO and pH were measured daily using electrode probes (CellOx325 and WTW SenTix21-3 respectively) mounted on portable data loggers (WTW3310, Germany).

2.4.1 Oxygen uptake rate

The total OUR was measured twice at each MLSS concentration set point following the EPA method 1683 (Specific Oxygen Uptake Rate in biosolids) with addition of substrate for maximum OUR measurement. The mixed liquor sample (600 mL) was saturated with pure oxygen gas to a concentration of approximately $10 \text{ mg O}_2 \text{ L}^{-1}$, then the oxygen flow was stopped, and 50 mL of influent wastewater were added. The decrease in the DO concentration (DO) was recorded automatically every 5 seconds. The decrease on the DO concentration indicated the velocity at which the DO was consumed (the total OUR) both for substrate oxidation and for the endogenous respiration. For data analysis and comparison, the OUR values were normalized at $20 \text{ }^\circ\text{C}$ and are reported as OUR_{20} ($\text{mg L}^{-1} \text{ h}^{-1}$).

2.4.2 Filterability

The sludge filterability was evaluated twice at each MLSS concentration set point using the Delft Filtration Characterization method (DFC_m) developed at the Delft University of Technology (TUD), The Netherlands (*H Evenblij et al., 2005; Geilvoet, 2010*). This method allows to calculate the sludge added resistance to the filtration process (ΔR). The method uses a single membrane element (X-flow F5385, The Netherlands) to filtrate a mixed liquor sample of approximately 30 L in a recirculation circuit, at a controlled flux ($80 \text{ L m}^{-2} \text{ h}^{-1}$) and crossflow velocity (1 m s^{-1}) (*H Evenblij et al., 2005; Geilvoet, 2010; M Lousada-Ferreira et al., 2010; Gil et al., 2011*). The reported values correspond to the ΔR_{20} which is the mean resistance increment after producing 20 L of permeate per membrane square meter. The sludge filterability depends on the temperature due to its influence on the fluid viscosity; however, the impact of viscosity on the filterability was considered negligible when compared to the influence of other factors such as the operational conditions and membrane maintenance and cleaning regimes (*Krzeminski, 2013*). The temperature at which the filterability tests were carried out ranged between 18.1 and $23.2 \text{ }^\circ\text{C}$.

3. Results and discussions

3.1 MLSS concentrations and MBR permeability

The target and actual mixed liquor total suspended solids (MLSS) concentrations and the mixed liquor VSS concentrations in the aerobic basin of the MBR were monitored at each MLSS set point (IDs 1 to 13). Figure 37 describes both the desired/target and measured/actual MLSS as well as the measured mixed liquor VSS values.

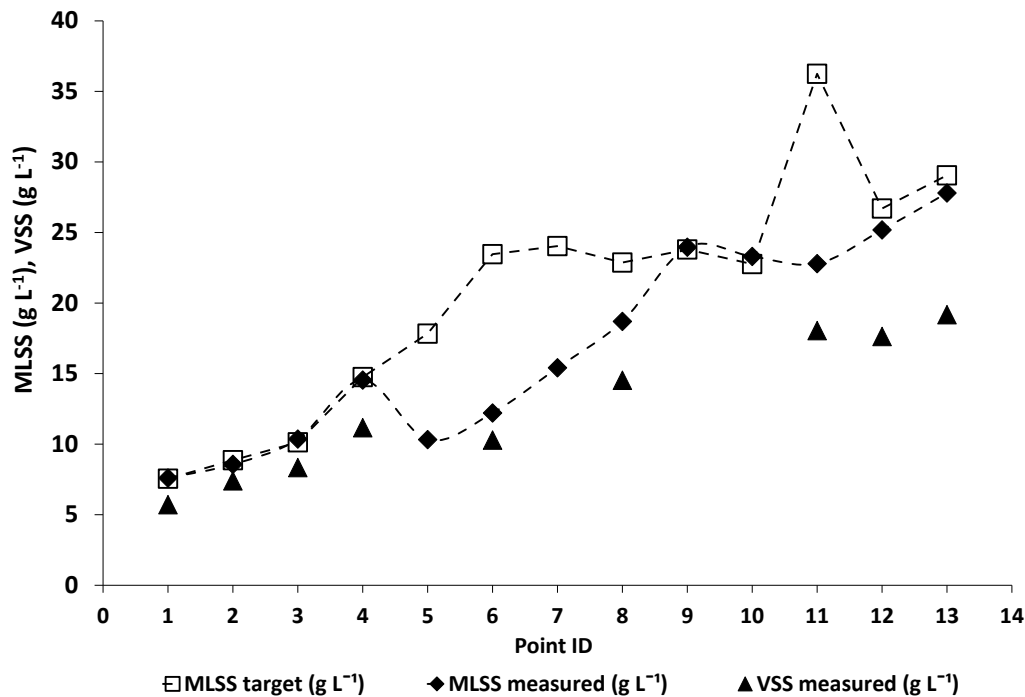


Figure 37 MLSS (target and measured) and VSS measured concentration at the different experimental set points.

For the first range of evaluated points (first phase - IDs 1 to 5 as described in the materials and method section), the influent flowrate was kept constant and the increase on the MLSS concentration was achieved mostly by increasing the SRT up to 30 days as can be observed in Figure 36. During this initial phase the MBR was still running at biomass concentrations below 15 g L⁻¹ MLSS. The targeted and measured MLSS concentration exhibited very similar values as observed in Figure 37 for IDs 1 to 4. The solids concentration difference between the target and measured MLSS for set point ID 5 was caused by operational problems which disturbed the continuous influent feeding and thus the final MLSS concentration.

For the second range of evaluated points (second phase - IDs 6 to 11) the influent flowrate was initially increased from 3.5 to 6 m³ d⁻¹ as observed in Figure 2 corresponding to a higher organic load applied to the reactor. However, the flow rate was needed to be reduced back for the set point IDs 8 to 11 to compensate for the observed increase on the TMP values typically reported at higher MLSS concentrations (*S. Lee & Kim, 2013*).

When higher flowrate conditions were not possible to maintain due to the reduced permeability observed in set points IDs 8 to 11, the reactor volume was reduced by 14% (0.12 m³) to achieve the desired MLSS concentrations as also indicated in Figure 36. This was done before adding the external substrate to increase the overall influent COD concentration. The solids concentration difference between the target and measured MLSS (IDs 6 to 8) was caused by operational problems which disturbed the continuous influent feeding and thus the final MLSS concentration. An unusually high concentration of influent TSS coming to the wastewater treatment plant was observed during the set point ID11. This high influent TSS value of 1,006 mg L⁻¹ led to a very high target MLSS of 36.3 g L⁻¹ as observed in Figure 37. Therefore, that explains the large differences between the targeted and measured MLSS corresponding to the set point ID 11. Despite the gaps observed between the targeted and measured MLSS at this second phase, it was confirmed that the MBR system was biologically active and performing well by evaluating the COD removal of the system. As reported more precisely below in section 3.2. of this chapter, COD values in the effluent as low as 12 mg L⁻¹ were observed for the evaluated range even at HRT conditions as low as 3.7 hours as reported for set point ID 8.

For the third and last operational range corresponding to IDs 12 and 13, sugar cane molasses was added as an external source of COD to increase the influent concentration. Since the MBR influent feed was steady during this final stage, the measured and targeted MLSS values matched very closely for these last two points as observed in Figure 37.

Figure 38 reports the transmembrane pressure (TMP), flow rate (Q), and normalized operational permeability (OP_n) at the evaluated range of measured MLSS concentrations. Figure 38 shows that the operational permeability of the MBR system overall decreased as the MLSS increased. In addition, an increase on the TMP was observed as the MLSS concentration increased at the evaluated flow rates. Both the increase on TMP values as well as the decrease on the OP_n indicate an overall negative impact on the performance of the filtration system under these operational conditions. Previous studies indicated that the increase on TMP and the decrease on permeability can be related both to the increase of the MLSS concentrations and viscosity (*Delrue et al., 2011*), and to the presence of extracellular polymeric substances (EPS) that contribute to foul the membranes (*Krzeminski, 2013*). The decrease on the filtration system performance can be ultimately translated as higher pressure (energy) demand to produce a progressively reduced permeate volume (*Trussell et al., 2007*).

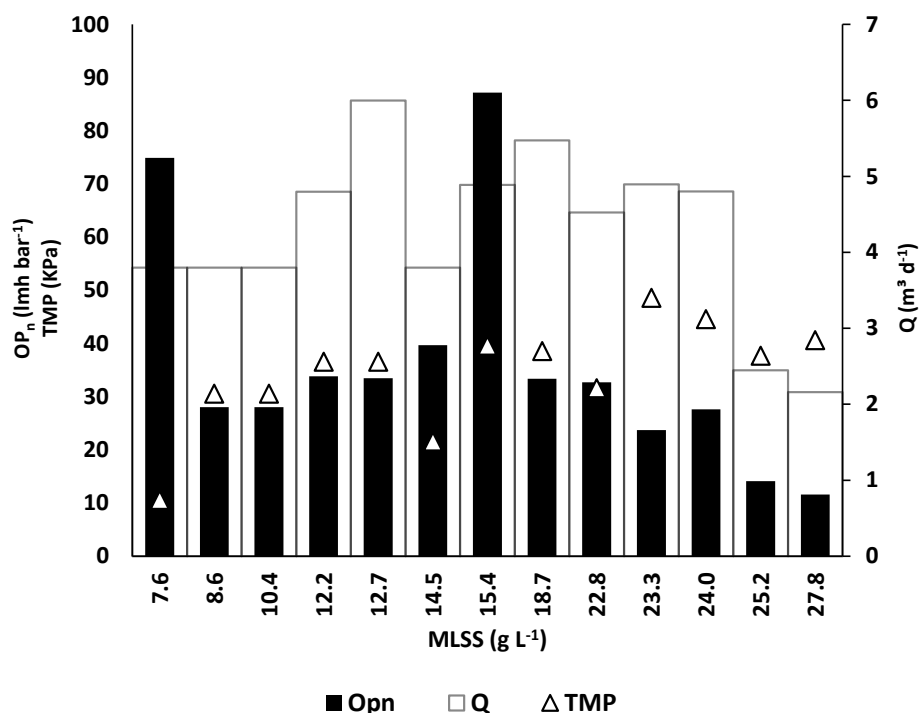


Figure 38 Normalized operational permeability (OP_n), flow (Q), and transmembrane pressure (TMP) and at the evaluated $MLSS$ concentrations in the MBR.

3.2 COD removal

The system was fed wastewater from a full-scale treatment plant; therefore, the influent COD concentration was not steady. The influent COD concentration to the pilot MBR system was ranging approximately from 600 to 1,000 mg COD L⁻¹ for IDs 1 to 10. An external source of COD was added for the experimental set points IDs 11 to 13 to strengthen the influent wastewater in order to provide enough substrate to sustain higher biomass concentrations. The performance of the MBR system regarding COD removal is presented in Figure 39. The COD effluent concentrations were mostly below 35 mg COD L⁻¹ with a maximum value of 61 and a minimum of 12 mg COD L⁻¹ (except for the last two set points IDs 12 and 13). Similarly, the COD removal showed efficiencies above 90% during most of the operational period with the exception of the last two set points IDs 12 and 13 where it decreased to 77% and 79%, respectively. The cause for this reduced COD removal values was attributed to an insufficient dose of DO to the MBR; a theoretical OTE provided by the Speece cone manufacturer based on clean water tests results was used for calculating the oxygen delivery capacity of the Speece cone without considering the effects of the $MLSS$ on the OTE (similar to the alpha factor effects on conventional bubble diffuser systems). As indicated by the removed COD values for set points IDs 12 and 13, enough DO was effectively delivered to remove nearly 80% of the applied substrate. However, not sufficient oxygen was supplied to satisfy all the oxygen needs of the system and leave a residual DO to be measured.

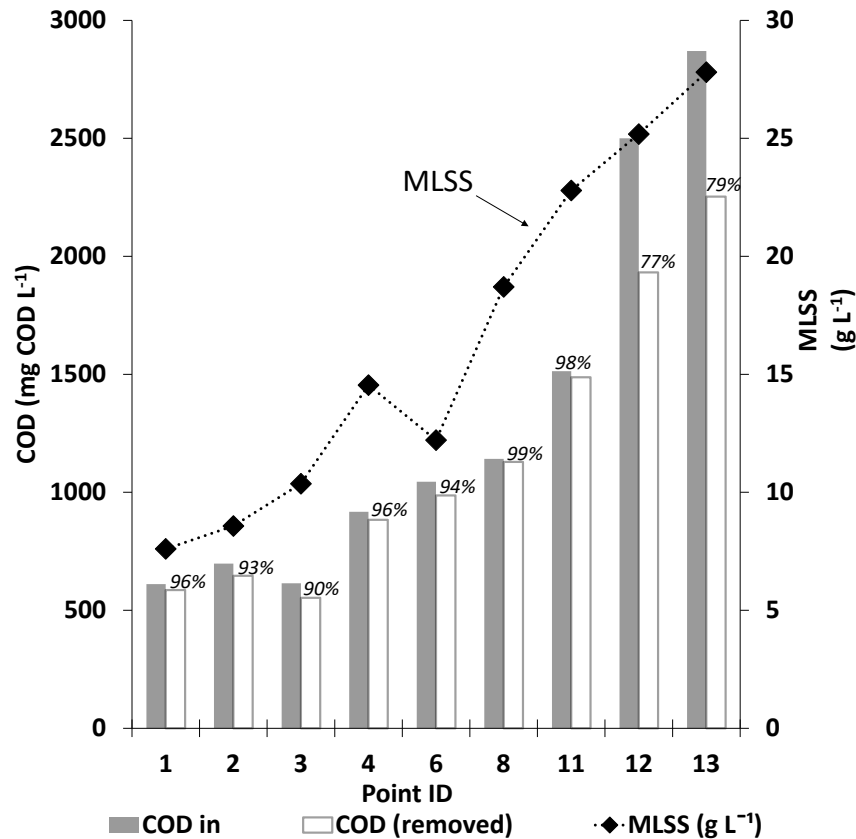


Figure 39 Applied and removed COD at the measured MLSS concentrations range.

3.3 DO and OUR

The OUR increased following a similar trend as observed with the increase of the MLSS concentration except for the last two MLSS set points (ID 12 and 13) as indicated in Figure 40. The trend observed for set points ID 1 to 11 was as expected with more active biomass consuming more DO resulting in a maximum reported OUR value of $332 \text{ mg L}^{-1} \text{ h}^{-1}$ corresponding to a measured MLSS concentration of 22.8 g L^{-1} . The reported trend for the OUR values was as expected assuming that enough DO was available in the aerobic basin of the MBR system. However, that was not the case for the set points ID 12 and 13 when the system was oxygen limited due to an insufficient dose of oxygen as explained in the previous section. The reported OUR for these last two set points, 82.8 and $105.6 \text{ mg L}^{-1} \text{ h}^{-1}$ for measured MLSS of 25.2 and 27.8 g L^{-1} , respectively, were comparable to the OUR values observed at the lower range of MLSS concentrations. Nevertheless, as observed in Figure 5, considerable COD removal still took place on the system considering that most of the applied substrate load ($2,870 \text{ mg COD L}^{-1}$) was removed ($2,253 \text{ mg COD L}^{-1}$).

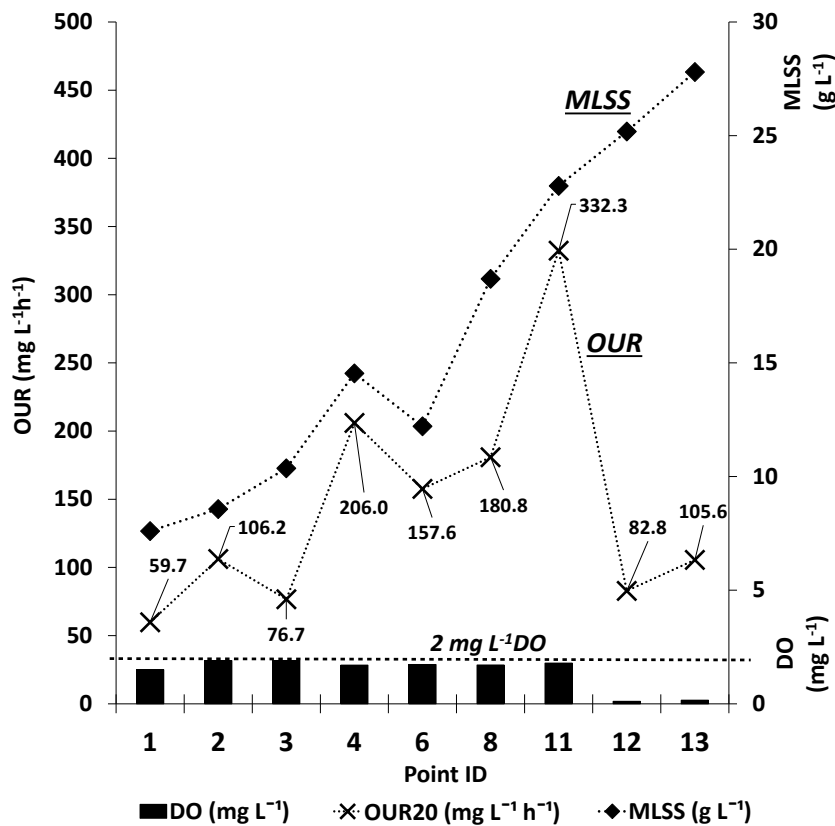


Figure 40 Dissolved oxygen concentrations (DO), oxygen uptake rates normalized at 20°C (OUR₂₀), and MLSS concentrations at the different MBR operational set points.

Figure 41 shows the theoretical OUR calculated using the oxygen flux (kg O₂ d⁻¹) required to carry out the oxidation of: (i) the removed COD - this would be the system boundary or the maximum potential oxygen consumption value; (ii) the ultimate BOD (UBOD) which represents the soluble and particulate substrate subject to biological oxidation; and (iii) the calculated BOD₅. These three calculated OUR series were compared to the actual (measured) OUR values for validation. From this comparison, the UBOD provided a better approximation to the measured OUR values with exception of the last two points where insufficient DO was provided as described on the previous section. The OUR values calculated using the UBOD represent all the substrate that could be subject to biological oxidation; thus, consuming oxygen. Therefore, under the particular experimental conditions evaluated in this research the OUR_{UBOD} could be

used to estimate the expected and the actual OUR.

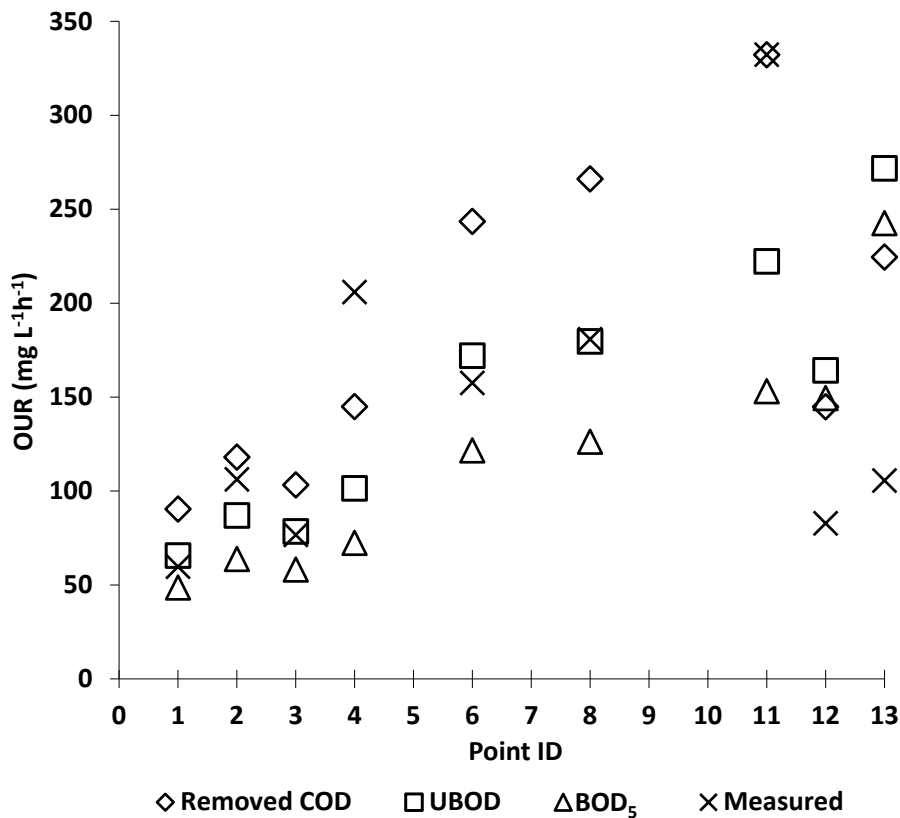


Figure 41 Calculated OUR values with respect to: (i) removed COD ◇; (ii) UBOD □; and (iii) BOD₅ △; compared to measured OUR values ×.

3.4 Speece cone aeration system

Figure 42 presents a comparison between the theoretical DO delivered by the Speece cone system in clean water, and the calculated oxygen requirements as oxygen flux (FO_c) considering both the total measured removed COD (FO_c 1) and the calculated theoretical UBOD (FO_c 2). Figure 8 indicates that the theoretical DO supplied by the Speece cone system was apparently more than enough to satisfy the theoretical UBOD oxygen demand of the pilot MBR system (FO_c 2). However, according to the observed DO concentrations previously reported in Figure 6 the MBR system was oxygen limited in the last two operational set point points (ID 12 and 13). This oxygen limitation also imposed a negative effect on the partial COD removal noticed for the same operational set points as previously indicated in Figure 39. The theoretical DO delivered by the Speece cone system was provided by the manufacturer based on oxygen transfer evaluation conducted in clean water without considering the impact of the MLSS concentrations on the oxygen transfer. Therefore, the theoretical DO delivery by the Speece cone system needs to be corrected considering the negative impact of the MLSS concentration on the oxygen transfer. However, the evaluation of the oxygen transfer efficiencies of the Speece cone system at different MLSS concentrations (that is, the impact of the different MLSS

concentrations on the OTE of the Speece con system) was not part of the scope of this research. Even though the oxygen transfer efficiency of the Speece con system was negatively affected by the MLSS, still the amount of DO supplied by the Speece cone to the MBR system was enough to remove 77 and 79% of the influent COD at the most challenging evaluated experimental conditions as indicated in Figure 39.

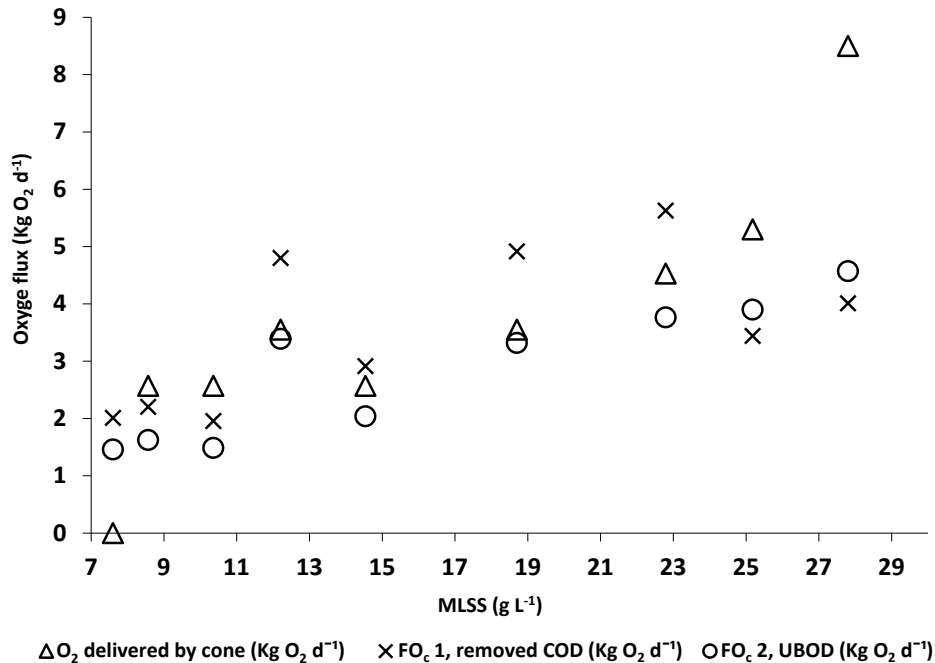


Figure 42 Theoretical oxygen delivered by the Speece cone in clean water, and theoretical oxygen requirements by the system. O₂ delivered by the cone = theoretical dissolved oxygen delivered by the cone in clean water; FO_{c1} removed COD = calculated oxygen required.

3.5 Filterability

As indicated in Figure 43, most of the measured filterability values fell in the poor filterability range ($\Delta R_{20} > 1 \times 10^{12} \text{ m}^{-1}$) with the exception of the set point ID 11 (MLSS concentration of 22.8 g L^{-1}) which showed a remarkable low resistance value falling in the range of moderate filterability ($\Delta R_{20} = 0.1 \times 10^{12} \text{ m}^{-1}$). This effect may be in accordance with the decrease in the resistance values at higher MLSS reported by (M Lousada-Ferreira et al., 2010; Gil et al., 2011). As observed in Figure 43, the filtration resistance did not increase proportionally to the MLSS concentration; on the other hand, the filtration resistance showed similar values at different MLSS concentrations; that is, the added resistance at very high MLSS concentrations ($>20 \text{ g L}^{-1}$) was not much different than the values observed at the mid-range MLSS concentrations ($\Delta R_{20} < 20 \text{ g L}^{-1}$). On previous studies conducted at the Delft University of Technology (TUD), it was suggested that there may be a breakpoint or as the author suggested an MLSS critical concentration for which the resistance to filtration (ΔR_{20}) is reduced (M Lousada-Ferreira et al., 2010; Gil, et al 2011). Similarly, (M Lousada-Ferreira et al., 2010) proposed that high

concentrations of mixed liquor could act as a sludge blanket retaining most of the fouling particles causing a reduction in the resistance to filtration; in other words, this might indicate the possibility of better filterability set points at some specific operational conditions as it was observed for the set point ID 11 during this research. That is, operating a MBR at such high MLSS concentrations may probably require the same operational efforts in terms of filtration resistance as when operating a conventional low biomass concentration MBR, but having the associated advantages such as the footprint reduction and the lower capital expenses.

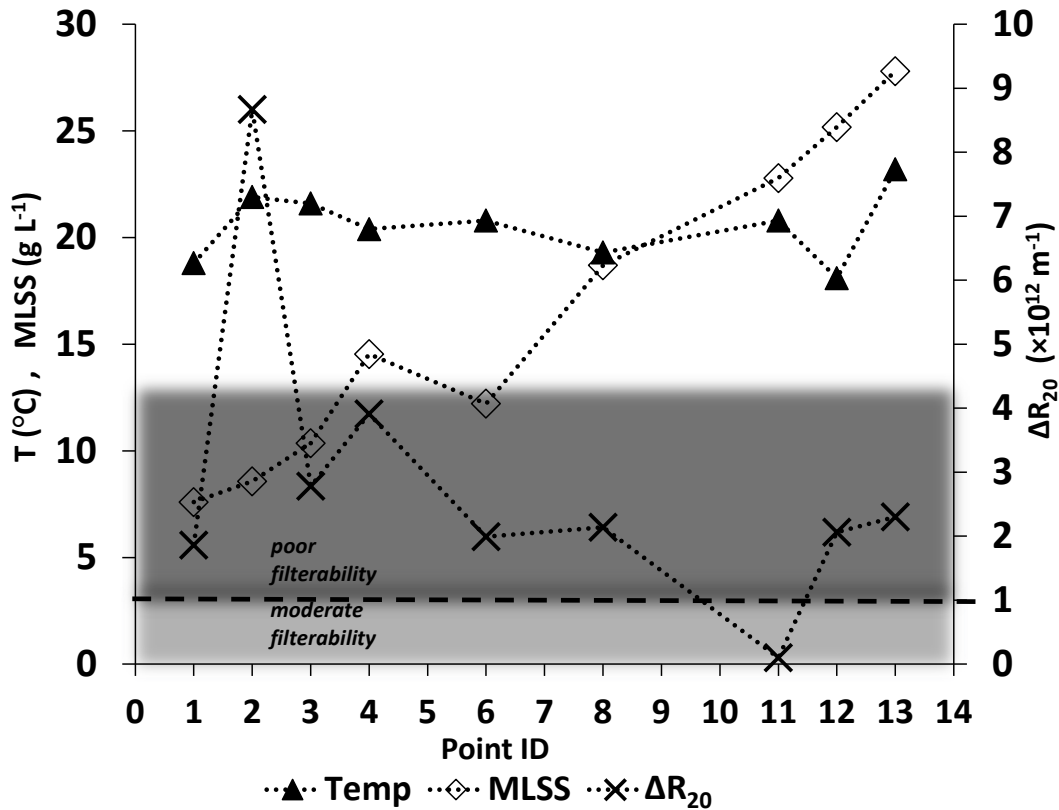


Figure 43 Sludge filterability (ΔR_{20}) at the evaluated MLSS concentration.

3.6 A comparison with conventional systems

One of the main advantages of operating an MBR at high MLSS concentrations is the reduction of the footprint requirements of the system. A comparison between a conventional activated sludge (CAS) system, a conventional MBR operated at low MLSS concentrations, and a high MLSS MBR system is shown in Figure 44. The same operational conditions were considered for all the proposed scenarios as follows: flow rate (Q) = 4 m³ d⁻¹; solid retention time (SRT) = 20 d; temperature 20 °C, and UBOD = 500 mg L⁻¹. The only parameter that was changed during this evaluation was the MLSS concentration which is the theoretical biomass corresponding to the applied organic load. Since this amount of biomass is the same in all cases, the changes on the MLSS

concentrations were obtained by just changing the required volume of the systems necessary to accommodate that mass for that particular MLSS concentration. Figure 44 shows the theoretical volume reduction that can be achieved by operating a high MLSS MBR compared to both a CAS system operated at 3 g L⁻¹ MLSS and to a conventional (low MLSS) MBR operated at 9 g L⁻¹ MLSS. The volume reductions and consequently the footprint requirement reductions can be as large as 90% and 70% compared to CAS and conventional MBRs, respectively.

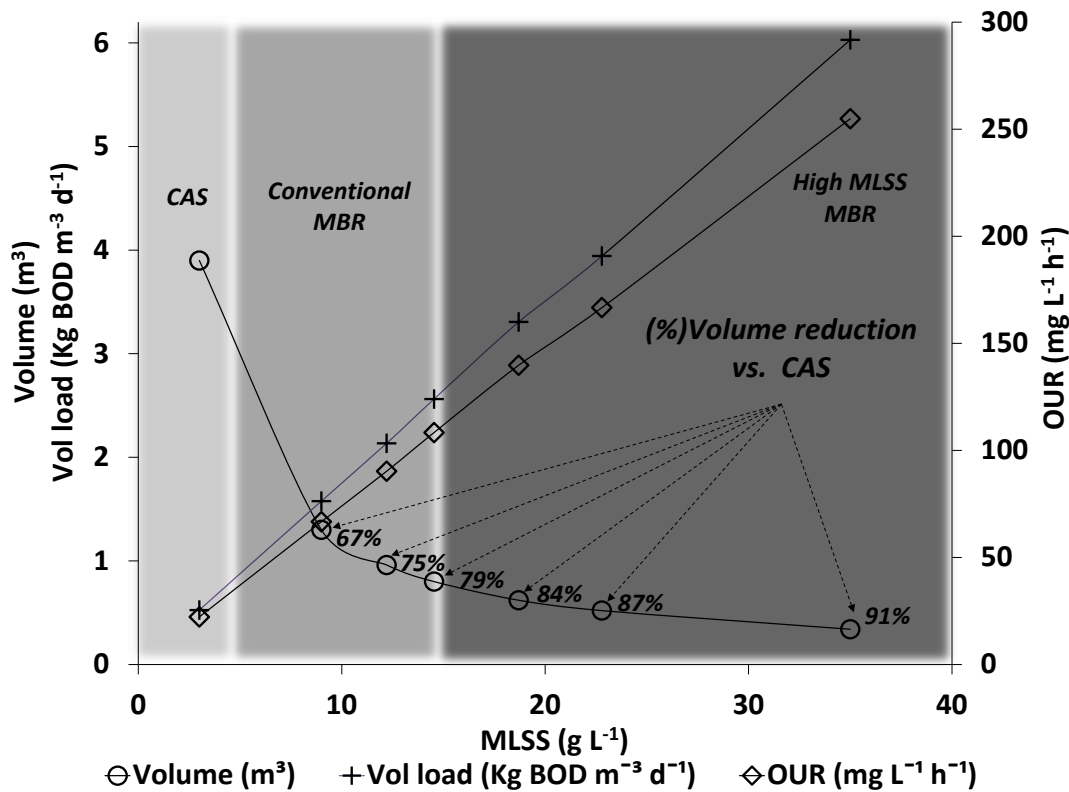


Figure 44 Comparison of the required biological system volumes (Volume), theoretical OURs (OUR), and volumetric organic loads (Vol load) for: (i) a conventional activated sludge system (CAS) – operated from 0 to 5 g L⁻¹ MLSS, (ii) a conventional MBR – operated from 5 to 15 g L⁻¹ MLSS, and (iii) a high MLSS MBR-Speece cone – operated from 15 to 40 g L⁻¹ MLSS. Assumed operational conditions: Flow rate (Q) = 4 m³ d⁻¹; SRT = 20 d; Temperature 20 °C, and UBOD = 500 mg L⁻¹.

Advantages of operating MBRs at high MLSS concentrations include the reduction on the footprint requirements lowering the associated capital costs. At the experimental conditions evaluated in this research, it was demonstrated that an MBR can be operated at high MLSS concentrations; a good quality effluent was obtained in terms of COD concentrations, high OURs were observed, and the filterability of the sludge was not much affected at the evaluated high MLSS concentrations compared to conventional systems.

Another scenario that may be feasible for implementing the high MLSS MBR concept may include the upgrade of existing CAS systems or conventional MBR wastewater treatment facilities to cope with ever increasing treatment demands. This situation may be particularly attractive in developing countries where funding availability could be limited. In the case of treatment plant upgrading, most of the required infrastructure would be already in place (i.e. pre-treatment, biological reactors, pumping systems, and control-instrumentation systems). The installed treatment capacity may be increased by providing additional membrane area to compensate for the increased flow rate but keeping the same reactor volumes. The additional treatment capacity is provided by the higher biomass concentration and increased membrane area. This, combined with the introduction of an alternative aeration system such as the concentrated oxygenation system - Speece cone or similar could make the necessary capital investment significantly less compared to the cost of building a whole new additional treatment train to cope with an increased influent load or treatment demand.

Moreover, the reduction of the footprint requirements may promote the development of innovative systems such as movable/portable containerized MBRs for a diverse range of applications including the provision of municipal/industrial wastewater treatment in areas without sewerage and the provision of easily deployable sanitation services under challenging site-specific conditions such as after the occurrence of a human-made or a natural disaster.

4. Conclusions

The results obtained under the experimental conditions of this research suggest that the existent limitations for reducing the footprint requirements of MBR systems can be overcome. The oxygen transfer limitations can be solved by incorporating an alternative aeration system. The measured OUR values matched closely the calculated values corresponding to the UBOD required oxygen flux at the evaluated MLSS concentrations. Filterability values falling in the range of poor filterability (ΔR_{20} above $1 \times 10^{12} \text{ m}^{-1}$) were observed for most of the evaluated MLSS concentrations. However, the decrease on filterability was not much worse for the upper MLSS concentration range (MLSS > 25 g L⁻¹) compared to the middle MLSS concentration range. Consequently, MBRs can be designed to operate at higher than usual MLSS concentrations. Further research should be carried out evaluating both the MBR performance on the very high MLSS range above 30 g L⁻¹, as well as the OTE of the Speece cone system at the evaluated range of MLSS concentrations. That is, further research is needed for pushing even further the limits for MBR applications operating at high MLSS concentrations.

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3

SIDESTREAM SUPEROXYGENATION FOR WASTEWATER TREATMENT: OXYGEN TRANSFER IN CLEAN WATER AND MIXED LIQUOR

This Chapter is based on Barreto, C. M., Ochoa I., García, H. A., Hooijmans, C. M., Livingstone, D., Herrera, A., & Brdjanovic, D. Sidestream superoxygenation for wastewater treatment: oxygen transfer in clean water and mixed liquor. *Journal of Environmental Management*, 219,125-137.

doi:<https://doi.org/10.1016/j.jenvman.2018.04.035>.

Abstract

The performance of a pilot scale superoxygenation system was evaluated in clean water and mixed liquor. A mass balance was applied over the pilot-scale system to determine the overall oxygen mass transfer rate coefficient (k_{LA} , h^{-1}), the standard oxygen transfer rate (SOTR, $kg\ O_2\ d^{-1}$), and the standard oxygen transfer efficiency (SOTE, %). Additionally, the alpha factor (α) was determined at a mixed liquor suspend solids (MLSS) concentration of approximately $5\ g\ L^{-1}$. SOTEs of nearly 100% were obtained in clean water and mixed liquor. The results showed that at higher oxygen flowrates, higher transfer rates could be achieved; this however, at the expense of the transfer efficiency. As expected, lower transfer efficiencies were observed in mixed liquor compared to clean water. Alpha factors varied between 0.6 and 1.0. However, values of approximately 1.0 can be obtained in all cases by fine tuning the oxygen flowrate delivered to the system.

1. Introduction

Aeration is a key process in aerobic biological wastewater treatment. Making enough oxygen available for bacterial growth allows the mineralization and removal of most of the pollutants from wastewater. Diffused aeration has been widely used for decades as the preferred alternative for introducing dissolved oxygen (DO) into water, despite its low oxygen transfer efficiency. The SOTE for fine bubble diffusers in clean water ranges from 2 to 7% per meter depth of reactor (*Mueller et al., 2002*), and even lower SOTEs have been reported in activated sludge process water; that is, most of the air that is compressed and pumped into the aerobic reactor in a wastewater treatment plant (WWTP) is released back to the atmosphere. In order to overcome the low SOTE in bubble aeration, sufficient retention time for the bubbles to interact with the mixed liquor needs to be provided by applying relatively low air flow rates, and by having deeper bioreactors. That is, to sustain the highest possible SOTE in bubble aeration, the maximum air flowrate allowed through each individual diffuser is compromised. This makes the surface area available in the aerobic reactor a key design parameter, since it determines the amount of diffusers that can be accommodated in the reactor; consequently, the amount of air that can be supplied. Assuming that the volume of the aerobic basin is a fixed outcome in the design process, the deeper the aerobic basin, the smaller the actual surface area available for placing diffusers. The reactor depth and available surface area, together with the biological oxygen demand and atmospheric site conditions, ultimately determine the motor size and power consumption of the air blower-compressor. Moreover, lower SOTEs are expected in process water compared to clean water due to: i) the effect of the suspended solids in the mass transfer process which translates into alpha factors of approximately 0.6 and 0.5 for conventional activated sludge (CAS) systems and for membrane bioreactors (MBR), respectively (*Krause et al., 2003; Germain et al., 2007; Trussell et al., 2007; Henkel et al., 2011*); and ii) the biofouling of the diffusers which progressively reduces the OTEs and demands additional maintenance (*Garrido-Baserba et al., 2016*). The aforementioned disadvantages worsen at high biomass concentrations due to: i) the increased resistance the suspended solids oppose to the mass transfer process (lower alpha values); ii) the diminished gas-liquid contact area due to enlarged bubble size when biofouling of the diffusers causes unwanted coalescence; and iii) uneven air distribution accompanied by progressively reduced air flowrates due to diffuser pore-blocking.

The low SOTEs provided by conventional fine bubble diffusers introduces limitations on the design of wastewater treatment systems. For instance, when designing portable wastewater treatment systems (low depth), high airflow rates are required to compensate for the low SOTEs introduced by the low available water depth provided at this type of wastewater treatment systems. That is, the high airflow rates requirements introduce high energy demands limiting the treatment capacity of these portable wastewater treatment systems. Another example of an application constraint includes the limitations imposed by bubble diffusers on expanding the design possibilities for MBR systems. The

maximum designed MLSS concentration in MBRs (therefore, the MBRs treatment capacity and footprint) is limited to approximately 15 to 18 g L⁻¹ due to the low SOTE exhibited by fine bubble diffusers at higher MLSS concentrations. Alpha factors as low as 0.2 have been reported at MLSS concentrations in the 20 g L⁻¹ range; and negligible alpha factors have been reported at approximately 40 g L⁻¹ (Muller *et al.*, 1995; Germain *et al.*, 2007; Judd, 2008; Racault *et al.*, 2010; Henkel *et al.*, 2011; Durán *et al.*, 2016). Therefore, innovative aeration technologies, and their integration in wastewater treatment processes, are needed to overcome the current limitations imposed by conventional bubble diffusers aeration systems for designing more efficient and compact wastewater treatment systems. Sidestream superoxygenation systems may offer an alternative to cope with the extremely low SOTEs imposed by bubble diffusers; particularly, when working at high MLSS concentrations. Pure oxygen based systems have been recently studied with promising results (Livingston, 2010; Barber *et al.*, 2015; Barreto *et al.*, 2017).

Superoxygenation systems, such as the Speece cone, have been used mainly for the ecological restoration of aquatic ecosystems. The Speece cone reported high oxygen transfer efficiencies and operational flexibility under a wide range of applications without most of the limitations observed in conventional aeration methods (Speece, 1975; Ashley, 1985; Ashley *et al.*, 2008; Ashley *et al.*, 2014). The oxygen transfer process occurs in a pressurized vessel built in a side-stream configuration. An oxygen supersaturated stream can be delivered to a receiving basin without depending on the current limitations imposed by bubble diffusers such as the maximum number of submerged bubble diffusers that can be placed on a given surface area. The use of this aeration method may allow both the design of shallow wastewater treatment reactors for portable sanitation applications, as well as the operation of high oxygen demand wastewater treatment systems such as MBRs operated at high MLSS concentrations.

Previous studies reporting on the factors influencing the oxygen transfer on Speece cone systems in clean water have been conducted for hypolimnetic aeration of aquatic ecosystems applications (Ashley, 1985; McGinnis *et al.*, 1998; Ashley *et al.*, 2008; Ashley *et al.*, 2014). However, the integration of the Speece cone system in the wastewater treatment field has not been explored. For instance, the effects of the MLSS concentration on the oxygen transfer capacities of the Speece cone technology have not been researched. Despite the enormous potential advantages of the superoxygenation technologies over conventional aeration methods, the performance of these systems have not been evaluated in the context of wastewater treatment applications. Once proven successful in wastewater treatment applications, more compact, portable, and energy efficient wastewater treatment systems can become a standard practice in the wastewater treatment field. For instance, MBR systems operated at high MLSS concentrations in combination with other strategies such as energy optimization (Gabarrón *et al.*, 2014) can maximize the impact and application of decentralized wastewater treatment systems for water reclamation (Atanasova *et al.*, 2017). Potential advantages of a high MLSS MBR system

include: reduced generation of waste activated sludge (WAS), reduced footprint requirements, better portability, and enhanced operational flexibility, among others as reported in a previous study on a high MLSS MBR conducted by *Barreto et al.*, (2017).

The objective of this study was to evaluate the oxygen transfer performance of a Speece cone system in the context of wastewater treatment applications. Particularly, the effects of the operational conditions on the oxygen transfer performance were assessed both in clean and process water at relevant CAS MLSS concentrations. The evaluated operational conditions included: i) the pressure inside the cone, ii) the inlet velocity to the cone, iii) the recirculation flowrate through the cone, and iv) the pure oxygen flow rate into the cone.

2. Material and methods

2.1. Experimental setup description

The evaluations were performed feeding high purity oxygen (HPO) to the cone at gas flow rates ranging between 5 and 40% of the cone's theoretical maximum capacity. The overall oxygen mass transfer rate coefficient (k_{LA} ; h^{-1}), the standard oxygen transfer rate (SOTR; $kg\ O_2\ d^{-1}$), and the standard oxygen transfer efficiency (SOTE; %), were determined by performing a mass balance over the system. These parameters were determined under pressures inside the cone of 10 and 40 psig, recirculation flowrates of 3 and 6 $m^3\ h^{-1}$, and liquid inlet velocities of 1.2 and 3.4 $m\ s^{-1}$. In addition, when working with mixed liquor, the alpha factor (α) was determined at an MLSS concentration of approximately 5 $g\ L^{-1}$.

The experiments were performed at the Delft Blue Innovations research facility (www.delftblueinnovations.nl) at the Harnaschpolder wastewater treatment plant in Delft, The Netherlands. The oxygen transfer performance of the Speece cone was evaluated using the same pilot scale membrane bioreactor (MBR) described by *Barreto et al.* (2017). This work is part of a research project investigating the oxygen transfer characteristics of a Speece cone in activated sludge for its application in the wastewater treatment field.

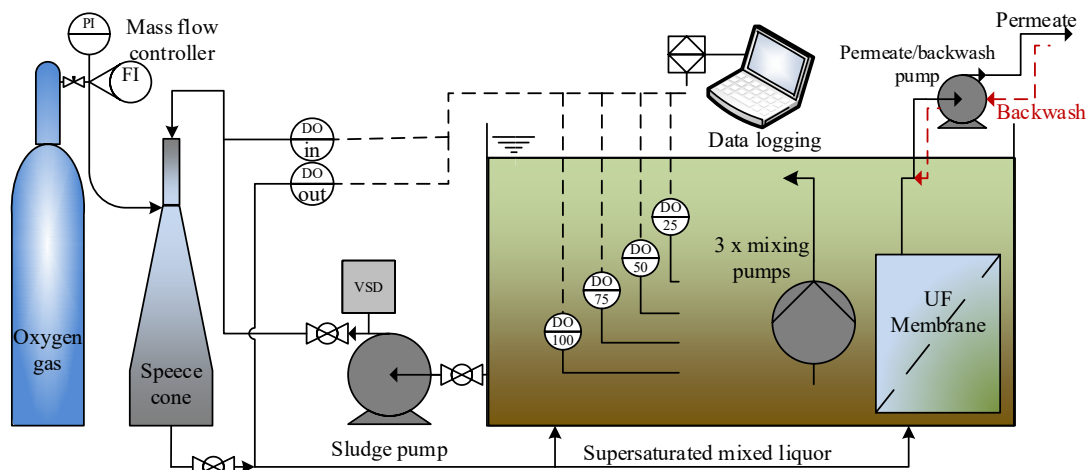


Figure 45 Speece cone-MBR. Process flow diagram.

2.1.1. Speece cone-MBR system

A pilot scale Speece cone (Speece, 1975) with a theoretical oxygen delivery capacity of approximately $5 \text{ kg O}_2 \text{ d}^{-1}$ was coupled to a pilot scale MBR. The equipment arrangement is presented below (Figure 45). The pilot Speece cone (ECO Oxygen Technologies LLC, USA) had the following dimensions: 115 cm height with base and top diameters of 30 and 10 cm respectively; the intake and discharge connections were coupled to ANSI flanges 4 inches in diameter. A progressive cavity pump (Netsch, NM045BY02512B, Germany) was provided with a variable frequency drive to feed the cone at different flowrates. The desired inlet velocity was set by placing PVC inserts with different diameters at the cone's inlet. The internal pressure could be monitored and adjusted by means of a manometer and a discharge valve at the cone's outlet. The cone discharged an oxygen supersaturated stream in two opposite corners of the MBR's aerobic chamber; the location of the discharge streams was selected to enhance the mixing conditions in the aerobic reactor. The aerobic chamber in the MBR had a maximum volume capacity of one cubic meter ($1 \text{ m} \times 1 \text{ m} \times 1 \text{ m}$). Additionally, and in order to provide complete mixing conditions, three submersible pumps (LEO, LKS-256P, Belgium) were installed inside the MBR. Pure oxygen from a certified industrial gases supplier was dosed and monitored by means of a temperature compensated and gas specific mass flow controller (Alicat Scientific, MC-5SLPM-D, USA).

2.2. Analytical procedures

Dissolved oxygen was measured using portable electrode probes (CelloX325, Germany) mounted on portable data loggers (WTW3310, Germany) with a measuring range of $0 - 50 \text{ mg L}^{-1} \pm 0.5\%$. A total of six DO probes were placed in the following locations (Figure 45): one at the cone inlet, one at the cone outlet, and four submerged in the MBR at depths of 25, 50, 75 and 100 cm to account for concentrations differences along the vertical axis

of the aerobic tank. The probes were calibrated according to the user's manual at least once a week when working with clean water, and daily when working with mixed liquor. The total and volatile suspended solids were measured according to the standard methods for the examination of water and wastewater (AWWA, 2014). The oxygen uptake rate in the activated sludge was determined following the U.S. Environmental Protection Agency (EPA) method 1683 Specific oxygen uptake rate in biosolids (EPA, 2001).

2.3. Experimental Procedure

The oxygen transfer in clean water and mixed liquor was evaluated following the reaeration method as defined by the Standards of the American Society of Civil Engineers (ASCE, 1997). The influence of the atmospheric oxygen intrusion in clean water and mixed liquor was determined and accounted for. Changes in the DO concentration due to intrusion were measured for a period of six hours under the same experimental conditions as in the oxygen transfer experiments without the addition of any oxygen gas. For the clean water experiments, tap water with the following characteristics was used: Turbidity: < 0.1 NTU; pH: 7-8; Electric conductivity (EC): 585 $\mu\text{S cm}^{-1}$. The ASCE method requires to remove the dissolved oxygen to at least a concentration of 0.5 mg L^{-1} by dosing sodium metabisulphite as an oxygen scavenger. After the dissolved oxygen was removed, the inlet velocity was set by placing a PVC insert of 17 or 25 mm diameter, and by adjusting the pump flowrate to the desired value, either 3 or 6 $\text{m}^3 \text{h}^{-1}$. The pressure inside the cone was adjusted by means of a valve located in the discharge line. Then, the oxygen gas flowrate was set in the mass flow controller and the data loggers started to record the DO concentrations without oxygen addition for approximately two minutes to confirm the oxygen scavenger had been fully consumed. After each experiment, the water was discarded and replaced with fresh tap water to avoid interferences by any chemical residuals. For the mixed liquor evaluations the procedure was exactly the same, but without the addition of the oxygen scavenger. In this case, the oxygen was used up by the active biomass for endogenous respiration. In order to assure that the endogenous respiration was reached, the activated sludge was aerated without substrate addition for a period of at least 30 hours before the experiment. The activated sludge was loaded into the MBR aerobic reactor from one of the Harnaspolder wastewater treatment plant secondary settlers; the MLSS was adjusted to approximately 5 g L^{-1} using the MBR's membrane module to extract the excess water until the desired suspended solids value was reached.

2.4. Experimental design

A series of preliminary experiments (nearly 50) were performed to identify the major operational variables influencing the oxygen mass transfer process in the Speece cone. During these experiments, one of the following three variables was changed at a time to observe its effects on the oxygen transfer process: i) pressure inside the cone (2, 10, 15,

25 and 40 psig); ii) recirculation water flow rate (3 and 6 m³ h⁻¹); and iii) HPO gas flowrate (ranging between 5 and 107% of maximum flow rate). For these experiments, the inlet velocity (0.6 and 1.20 m s⁻¹) changed as a consequence of the selected recirculation water flowrate, since the same inlet diameter at the top of the Speece cone was used for all the experiments. The results from this preliminary phase (not reported in this article) showed the importance of the HPO gas flowrate on the oxygen transfer rate compared to the other variables. The performance of these preliminary experiments allowed to identify a significant amount of oxygen bubbles escaping from the cone's bottom beyond certain HPO flow. The fact that this gas volume could not be accounted for, allowed to define an upper limit in the HPO flow of 40% of the maximum theoretical oxygenation capacity of this particular cone at the specific experimental conditions for the next round of experiments.

The experimental design for the clean water experiments is presented in Table 1. The individual effect of each operational parameter (pressure, inlet velocity, and recirculation flowrate) was evaluated by keeping two of the three operational parameters constant as follows: i) evaluated pressures of 10 and 40 psig for a constant recirculation flowrate of 3 m³ h⁻¹ and an inlet velocity of 3.4 m s⁻¹; ii) evaluated inlet velocities of 1.2 and 3.4 m s⁻¹ for a constant pressure of 10 psig and a recirculation flowrate of 3 m³ h⁻¹; and iii) evaluated recirculation flowrates of 3 and 6 m³ h⁻¹ for a constant pressure of 10 psig and an inlet velocity of 3.4 m s⁻¹. The operational parameters were evaluated at different HPO flow rates of approximately 5%, 10%, 20%, 30% and 40% of the theoretical maximum oxygen dissolution capacity in the cone at the experimental conditions. A similar approach was followed to evaluate the effects of mixed liquor on the Speece cone as described in Table 2; however, only the effect of the operational pressure inside the cone was evaluated at 10 and 40 psig at a MLSS of approximately 5 g L⁻¹ for the same HPO gas flowrate range previously described.

2.5. Data analysis and oxygen transfer parameter estimation

The oxygen concentration profile for all the experiments was fit to a mass balance applied over the MBR (Equation 48) in order to calculate the net oxygen transfer rate into the MBR reactor (Net OTR_(MBR)) by means of a numerical integration method for each DO probe separately. The reported values in this study correspond to the average result of the profiles per probe at different depths. The homogeneity and replicability of the experiments were determined by performing triplicate experiments in the preliminary phase evaluation; the difference between experiments under the same operational conditions was established with the standard deviation of the calculated results, which is plotted as error bars for each set of experiments on the respective result and discussion section.

$$V \left(\frac{dC_{MBR}}{dt} \right) = \text{Net OTR}_{(MBR)} + \text{Intrusion} - \text{OUR}_{\text{en}} \quad (48)$$

2.5.1. *Determination of the intrusion oxygen mass transfer rate coefficient $k_{LA_intrusion}$*

The atmospheric oxygen intrusion into the MBR was calculated by measuring the oxygen mass transfer rate under the same experimental conditions as during the experiments described in Tables 1 and 2, without the addition of oxygen gas (that is, net OTR is zero). The corresponding oxygen mass transfer rate coefficient $k_{LA_intrusion}$ for the atmospheric intrusion was calculated by fitting the DO data obtained in the intrusion experiments to Equation (49) using the MS Excel solver tool. The theoretical C_s value was determined using the DOTABLES software of the United States Geological Survey. The endogenous respiration term (OUR_{en}) was only considered when performing the experiments with mixed liquor; for the clean water experiments this term equals zero.

$$V \left(\frac{dC_{\text{intrusion}}}{dt} \right) = k_{L}a_{\text{Intrusion}}(C_s - C_{MBR}) \times V - \text{OUR}_{\text{en}} \quad (49)$$

The calculated $k_{LA_intrusion}$ values were incorporated into the overall mass balance in Equation (48) to account for any atmospheric oxygen dissolved into the system.

2.5.2. *Determination of the net oxygen transfer rate*

Once the values for the intrusion term ($k_{LA_intrusion}$) were determined, as described in previous section 2.5.1 of this chapter, the DO data from the experiments performed both in clean water and in mixed liquor was fit to the Equation (50), and the net oxygen transfer rate was calculated at each experimental condition. From the DO profiles only the measured concentrations in the range of 10 to 75% of the atmospheric C_s were taken, according to the methods reported by (Boyd, 1986; Casey, 1997; Mueller et al., 2002; Ashley et al., 2014). The endogenous respiration term (OUR_{en}) was only considered when performing the experiment with mixed liquor; for the clean water experiments this term was equal to zero.

$$V \frac{dC_{MBR}}{dt} = \text{Net OTR}_{(MBR \text{ tank})} + [K_L a_{\text{Intrusion}}(C_s - C_{MBR})] \times V - \text{OUR}_{\text{en}} \quad (50)$$

The calculated net oxygen transfer rate term represents the observed oxygen transfer rate (OTR kg d^{-1}); that is, the oxygen mass transferred to the system.

2.5.3. *Determination of the overall oxygen mass transfer rate coefficient $k_{LA_transference}$*

The overall mass transfer rate coefficient k_{LA} is often used for the characterization of different types of aeration systems in which gas into liquid mass transfer processes are involved with a well-mixed liquid phase. The net oxygen transfer rate term included in Equation (50) can also be expressed as the product between an overall oxygen mass transfer rate coefficient ($k_{LA_transference}$) and the driving force for the oxygen transfer. This driving force is given by the difference between the oxygen saturation concentration inside the cone (C_{sHPO}) and the DO concentration in the aerobic MBR reactor (C_{MBR}). The resulting expression is described in Equation (51). The DO profiles from the experiments performed both in clean water and in mixed liquor were fit to Equation (51) using MS Excel solver. The overall oxygen mass transfer rate coefficient ($k_{LA_transference}$) was calculated for each experimental condition. As described in section 2.5.2 of this chapter, also for these DO profiles the measured concentrations in the range of 10 to 75% of the atmospheric C_s were used. The endogenous respiration term (OUR_{en}) was equal to zero in the case of clean water experiments. The oxygen saturation concentration (C_{sHPO}) inside the cone was calculated taking into account the temperature, pressure, and oxygen gas purity inside the cone as described in Equation (52).

$$V \frac{dC_{MBR}}{dt} = [k_{LA_Transference}(\beta C_{sHPO} - C_{MBR})] \times V + [k_{LA_intrusion}(C_s - C_{MBR})] \times V - OUR_{en} \quad (51)$$

$$C_{sHPO} = P_A \times C_s \times O_2F \quad (52)$$

The calculated $k_{LA_transference}$ values previously obtained from the Equation (51) were standardized to 20°C using the expression described in Equation (53).

$$k_{La_{20}} = k_{La_T} \theta^{(20-T)} \quad (53)$$

2.5.4. *Determination of the observed standard oxygen transfer rate SOTR and max_SOTR*

The observed OTRs in $kg\ O_2\ d^{-1}$ were calculated as the net OTR by applying the mass balance previously described in section 2.5.2. The net OTR was obtained by subtracting the measured intrusion from the accumulation term on the left-hand side of Equation (50). The observed OTR values were then standardized to 20°C by applying the expression described in Equation (54).

$$SOTR = OTR \times \theta^{(20-T)} \quad (54)$$

The maximum SOTR, or the system oxygen transfer capacity when the conditions are optimal, was calculated by using the expression described in Equation (55). That is, the maximum SOTR can be obtained when the maximum difference is observed between the oxygen saturation concentration and the oxygen concentration in the MBR tank; that is, when the DO concentration in the MBR reactor (C_{MBR}) is equal to zero.

$$\max_SOTR = k_L a_{\text{Transference20}} \times C_{s\text{HPO}} \times V \quad (55)$$

Both the observed SOTR and the calculated max_SOTR are presented in this study to compare the actual with the maximum achievable optimum performance.

2.5.5. Standard oxygen transfer efficiency SOTE

The SOTE (%) was determined by considering both the SOTR together with the high-purity oxygen mass flow supplied to the system at each evaluated experimental condition as described by the expression in Equation (56). The SOTE indicates what percentage of the pure oxygen gas that was introduced into the Speece cone system in fact ended up as dissolved oxygen in the liquid phase, meaning in the MBR aerobic reactor.

$$SOTE = \frac{SOTR}{\text{HPO mass flow}} \times 100 \quad (56)$$

2.5.6. Alpha factor

The effects of the MLSS on the oxygen transfer was evaluated in the experiments carried out with mixed liquor at a CAS relevant MLSS concentration of approximately 5 g L⁻¹. Alpha is a factor widely used in the wastewater treatment field to account for the hindered mass transfer of oxygen that occurs in the presence of MLSS. This factor plays an important role in the design phase of a system since it allows to establish the increase in the oxygen mass flow to the aerobic reactor necessary to compensate for the additional resistance the MLSS oppose to the oxygen dissolution. Alpha is a ratio that represents how close the k_La in process water (mixed liquor) is to the k_La in clean water. The alpha factor can be determined as described by the following expression (Equation (57)) by dividing k_{LA}_process by the k_{LA}_clean water.

$$\alpha = \frac{k_{L}a_{\text{process water}}}{k_{L}a_{\text{clean water}}} \quad (57)$$

Sidestream superoxygenation for wastewater treatment: Oxygen transfer in clean water and mixed liquor

Table 1 Experimental design for the evaluation of the operational parameters effects on the Speece cone oxygen transfer performance in clean water

Evaluated parameter	Recirculation flowrate (m ³ h ⁻¹)	Pressure (psig)	Inlet velocity (m s ⁻¹)	% of max HPO flow	Theoretical max oxygen output (kg d ⁻¹)	Oxygen input (slpm)	Gas/Water flow ratio				
Pressure inside the cone	3	10	3.43	40%	2.20	1.17	2.1%				
				30%	1.65	0.88	1.6%				
				20%	1.10	0.58	1.1%				
				10%	0.55	0.29	0.5%				
				5%	0.28	0.15	0.3%				
	3	40	3.43	40%	4.86	2.58	4.7%				
				30%	3.64	1.94	3.5%				
				20%	2.43	1.29	2.4%				
				10%	1.23	0.65	1.2%				
				5%	0.60	0.32	0.6%				
Inlet velocity	6	10	3.40	40%	4.39	2.33	2.1%				
				30%	3.30	1.75	1.6%				
				20%	2.20	1.17	1.1%				
				10%	1.10	0.58	0.5%				
				5%	0.55	0.29	0.3%				
	6	10	1.20	55%	6.07	3.22	3.0%				
				36%	3.94	2.09	1.9%				
				28%	3.03	1.61	1.5%				
				22%	2.43	1.29	1.2%				
				17%	1.88	1.00	0.9%				
				7%	0.75	0.40	0.4%				
				5%	0.56	0.30	0.3%				
				Recirculation flowrate	3	10	3.43	40%	2.20	1.17	2.1%
								30%	1.65	0.88	1.6%
								20%	1.10	0.58	1.1%
10%	0.55	0.29	0.5%								
5%	0.28	0.15	0.3%								
6	10	3.40	40%		4.39	2.33	2.1%				
			30%		3.30	1.75	1.6%				
			20%		2.20	1.17	1.1%				
			10%		1.10	0.58	0.5%				
			5%		0.55	0.29	0.3%				

Table 2 Experimental design for the evaluation of the operational parameters effects on the Speece cone oxygen transfer performance in mixed liquor (MLSS~5 g L⁻¹)

Evaluated parameter	Recirculation flowrate (m ³ h ⁻¹)	Pressure (psig)	Inlet velocity (m s ⁻¹)	% of max HPO flow	Theoretical max oxygen output (kg d ⁻¹)	Oxygen input (slpm)	Gas/Water flow ratio
Pressure inside the cone	3	10	3.43	40%	2.20	1.17	2.1%
				30%	1.65	0.88	1.6%
				20%	1.10	0.58	1.1%
				10%	0.55	0.29	0.5%
				5%	0.28	0.15	0.3%
	3	40	3.43	40%	4.86	2.58	4.7%
				30%	3.64	1.94	3.5%
				20%	2.43	1.29	2.4%
				10%	1.23	0.65	1.2%
				5%	0.60	0.32	0.6%

3. Results and discussion

3.1. Overall oxygen mass transfer rate coefficient $k_{La_transference}$

The individual effects of the pressure, inlet velocity, and recirculation flowrates on the $k_{La_transference}$ were evaluated and standardized at 20 °C. The standardized $k_{La_transference}$ are reported as $k_{La20_transference}$.

The effects of the pressure on the $k_{La20_transference}$ are presented in Figure 46(a). The effects were not that significant, and only noticeable above HPO flowrates of approximately 20%. Beyond that point, the $k_{La20_transference}$ was slightly lower at the highest evaluated pressure. These results agree with those reported both by Barber *et al.*, (2015) who evaluated a pressurized column fed with pure oxygen, and by Versteeg *et al.* (1987) who experimented with gas-liquid mass transfer at elevated pressures. The authors proposed that the gas phase mass transfer coefficient (k_G) was negatively impacted as the overall pressure was increased in the system due a reduction in the renewal rate of oxygen

molecules at the gas-liquid interphase; that is, the gas diffusivity decreases with increasing pressure failing to keep the boundary layer under saturated conditions causing the mass transfer to diminish. Another approach for explaining the observed reduction in the $k_{La20_transference}$ at high pressures considers a potential reduction of the bubbles' sizes as the pressure increases. Under high pressure, the sizes of the gas bubbles may be significantly reduced; consequently, depending on their final sizes, the small bubbles can be pushed out of the system by the influent water flow reducing the $k_{La20_transference}$. This particular observation was also reported by *McGinnis et al., (1998) and McGinnis et al., (2002)* for different aeration systems including the Speece cone technology.

The inlet velocity influenced the $k_{La20_transference}$ slightly more than the pressure did at the evaluated conditions (Figure 46 (b)). Higher inlet velocities may promote more intense mixing conditions enhancing the gas-liquid contact, not only at the interphase, but especially along the Speece cone's height. The liquid downward velocity breaks and pushes the gas bubbles down as the bubbles try to bounce back up due to their own buoyancy; both the bubble size and the buoyant force progressively decrease until the gas get dissolved into the liquid phase. Low inlet velocities, below certain optimal value, result in gas bubbles accumulation at the cone's top section; whereas extremely high inlet velocities, above the optimal value, cause the gas to be washed out of the system without getting dissolved. This is one of the most important Speece cone's design principles that allows having a bubble contact time higher than the hydraulic retention time when the system is operated at the optimal inlet velocity conditions. For the evaluated range in this study, operating the Speece cone at higher inlet velocities seemed to positively affect the cone's performance regarding the $k_{La20_transference}$. However, in another study conducted by *Ashley et al. (2014)*, the inlet velocity was reported to have a negative effect over the k_{La} . Nevertheless, that evaluation was carried out at inlet velocities much higher than the optimal recommended design velocity of 3 m s^{-1} . Therefore, the role of velocity needs to be further investigated and considered for maximizing the $k_{La20_transference}$.

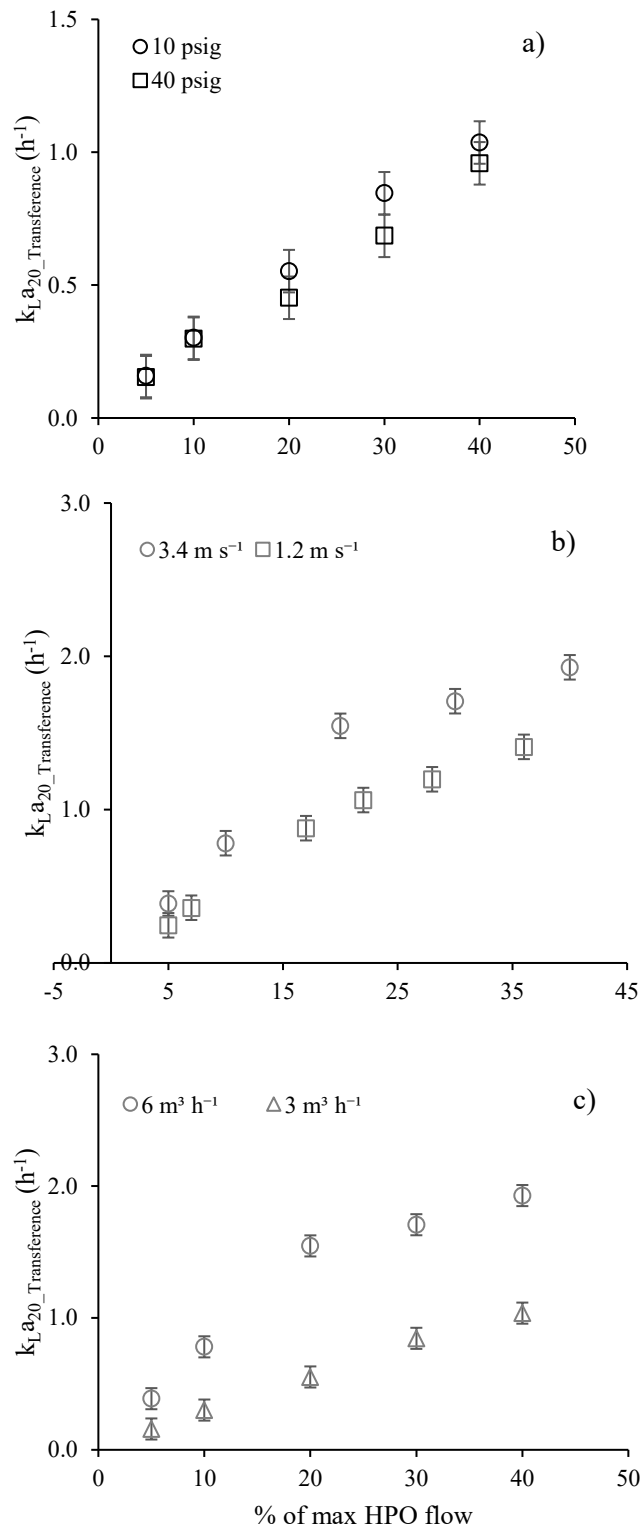


Figure 46 Influence of operational parameters on the mass transfer coefficient $k_L a$ (h^{-1}) in clean water. (a) Pressure, (b) Inlet velocity, (c) Recirculation flowrate.

Higher recirculation flowrates led to higher $k_L a_{20_transference}$ values (Figure 46 (c)). The differences between low and high recirculation flowrates (3 and 6 $m^3 h^{-1}$) were evident

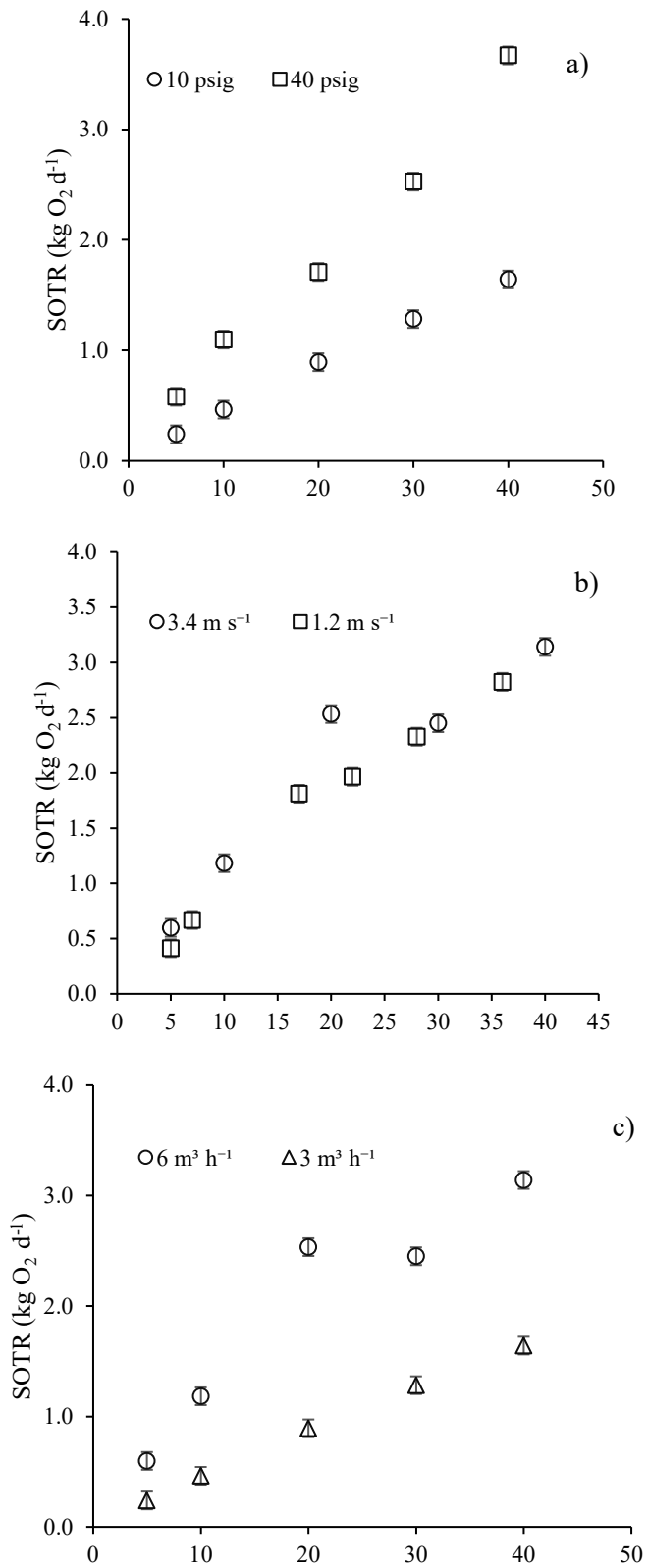
for the entire HPO flowrate range; this difference could be attributed to an increase in the turbulence and mixing intensity imposed by the high recirculation flowrates; in addition, these could cause both the rupture of the bubbles into smaller size bubble (*García-Ochoa et al., 2009*), as well as the increase of the shear effect (*Durán et al., 2016*) contributing to a better distribution and interaction between the liquid and the gas phases. Higher mixing intensities are generated by the larger water flowrates discharge into the cone which enhances the interaction between phases; therefore, increasing the mass transfer rate at the interfacial region. That is, operating at higher recirculation flowrates may have a positive impact on the performance of the Speece cone regarding the $k_{LA20_transference}$.

The HPO flowrate was found to be the dominant parameter affecting the $k_{LA20_transference}$ at the evaluated HPO flowrates and operational conditions (pressure, inlet velocity, and recirculation flowrate). The main reason for having a high k_{LA} under increasing HPO flowrates include the following: i) high HPO flowrates means more oxygen molecules coming inside the cone; this increases the collision frequency with the liquid molecules resulting in a large ratio of interfacial area to the volume of the liquid in the cone; and ii) a higher HPO flowrate provide additional turbulence (mixing intensity) in the cone enhancing the gas exchange transfer rate at the interfacial area. Therefore, without taking into account any efficiency considerations in the discussion, the higher the HPO flowrate into the cone, the better the cone performance in terms of the $k_{LA20_transference}$ at all the evaluated pressures, inlet velocities, and recirculation flowrates.

3.2. Observed standard oxygen transfer rate SOTR and max SOTR

The individual effects of the pressure, inlet velocity, and recirculation flow rates on the observed and maximum SOTRs were evaluated and reported in this section.

The effects of the pressure on the observed SOTR as a function of the HPO flowrate were noticeable for the entire HPO flowrate range. The pressure positively influenced the SOTR, and the effects were even more noticeable at high HPO flowrates. The differences in the observed SOTRs between high and low pressure were up to 2.5 kg O₂ d⁻¹ and up to 4.1 kg O₂ d⁻¹ for the low (5-20%) and high (30-40%) HPO flowrates, respectively (Figure 47 (a)). As discussed in the previous section (Section 3.1), the pressure did not exhibit a strong direct impact on the $k_{LA20_transference}$; in fact, the opposite trend was observed in accordance with the work by Barber et al. (2015). Nevertheless, the pressure did affect the overall oxygen output by raising the oxygen saturation concentration (C_{sHPO}) inside the Speece cone. The higher the C_{sHPO} , the higher the magnitude of the concentration gradient (ΔC); that is, the oxygen concentration difference between the cone (C_{sHPO}) and the receiving basin (C_{MBR}) as described by the Equation (51). The concentration gradient directly influences the driving force for the oxygen transfer. Therefore, the higher the pressure, the higher the oxygen transfer rate.



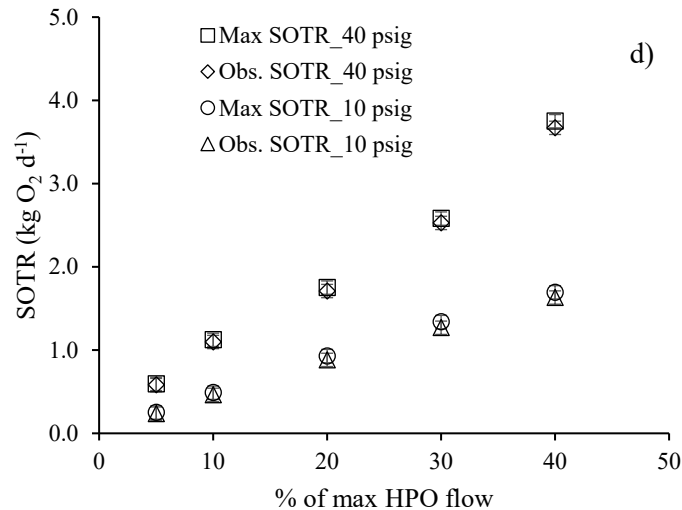


Figure 47 Influence of operational parameters on the SOTR (kg O₂ d⁻¹) in clean water. (a) Pressure, (b) Inlet velocity, (c) Recirculation flowrate, (d) Max. vs. Observed SOTR.

Figure 47 (b) presents the observed SOTR as a function of the HPO flowrates at the evaluated inlet velocities. As described when discussing the effects of the inlet velocity on the $k_{LA20_transference}$, a higher inlet velocity produces more turbulence and mixing intensity which normally leads towards an enhanced oxygen transfer. In addition, increasing the inlet velocity may extend the oxygen gas bubbles retention time with a positive effect on the oxygen transfer. However, for the evaluated range of inlet velocities minor changes were noticed on the observed SOTR. These findings are consistent with the little differences found and reported on the $k_{LA20_transference}$ as a function of the inlet velocity (Section 3.1). Considering the strong dependence of the k_{LA} on the SOTR (Equation 55), the same conditions previously described in Section 3.1 (Figure 46 (b)) affecting the k_{LA} would impact on the SOTR. That is, the additional turbulence and the extended bubble residence time can explain the effects of the inlet velocity on the SOTR. Ashley *et al.*, (2014) reported a much-pronounced effect of the inlet velocity on the SOTR. However, their experiments were performed at a much higher inlet velocity range (from 5.7 to 8.5 m s⁻¹) compared to those used in this work. Higher inlet velocities may probably result in higher $k_{LA20_transference}$ coefficients. Therefore, higher SOTR values may be expected at higher inlet velocities. Consequently, operating at a higher inlet velocity range may have a positive effect on the cone's performance regarding the mass transfer rate.

The effects of the recirculation flowrate on the SOTR as a function of the HPO is presented in Figure 47 (c). The observed SOTR significantly increased at higher recirculation flowrates. Considering the strong dependence of the k_{LA} on the SOTR, the SOTR follows the same behavior of the k_{LA} when the same conditions are applied. Higher recirculation flowrates introduce higher mixing intensities; therefore, yielding higher $k_{LA20_transference}$ coefficients. An increase on the overall $k_{LA20_transference}$ results in a higher SOTR. Therefore, operating at higher recirculation flowrates enhances the performance of the Speece cone regarding the observed SOTR.

The higher the HPO flowrate, the higher the observed SOTR at the three evaluated operational parameters (pressure, inlet velocity, and recirculation flow rate) as shown in Figure 47 (a, b, c). This observation is in accordance with the results obtained for the $k_{LA20_transference}$ discussed in Section 3.1. That is, the increase on the observed SOTR is due to the increase of the $k_{LA20_transference}$ at higher HPO flowrates. Therefore, and without considering any efficiency transfer, the higher the HPO flowrate into the cone, the better the cone performance in terms of the net oxygen transfer rate at all the evaluated pressure, inlet velocity, and recirculation flowrate conditions.

The SOTRs observed in this research indicated that the Speece cone can deliver similar amounts of oxygen under different operational conditions. For instance, an oxygen demand of approximately $3.5 \text{ kg O}_2 \text{ d}^{-1}$ can be supplied either working at a recirculation flowrate of $6 \text{ m}^3 \text{ h}^{-1}$, pressure 10 psig, and HPO flowrate of 55%, or alternatively at a recirculation flowrate of $3 \text{ m}^3 \text{ h}^{-1}$, pressure of 40 psig, and HPO flowrate of 40%. This gives the system operational flexibility to be used under different influent loadings or flowrate conditions. In addition, this operational flexibility could be useful for standardization purposes since one size Speece cone unit would cover a wide range of oxygen demands with little modifications.

The maximum SOTR represents the extent of the oxygen transfer rate when the DO concentration in the receiving basin is equal to zero. Figure 47 (d) compares the observed and maximum SOTR as a function of the HPO flowrate for the two evaluated pressures (10 and 40 psig). The values for the observed and maximum SOTRs were almost identical, meaning the actual transfer rate is almost as high as it can possibly be.

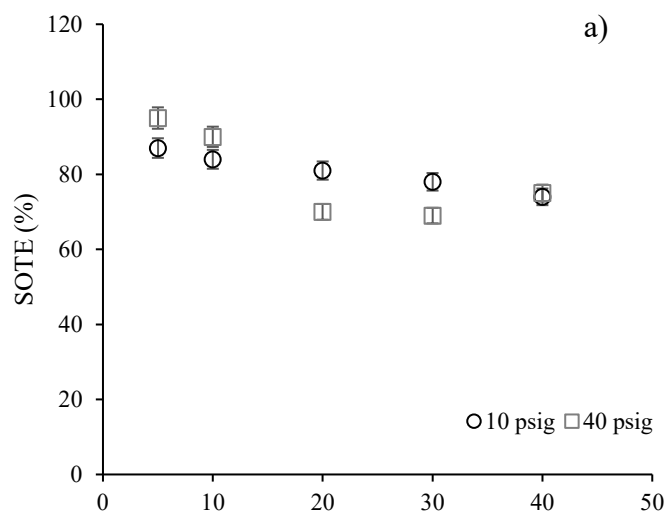
3.3. Standard oxygen transfer efficiency

The effects of the pressure, inlet velocity, and recirculation flow rates on the SOTE were evaluated and reported in this section.

The SOTE did not significantly change at the evaluated pressures. As shown in Figure 48 (a) very similar SOTEs were reported at 10 and 40 psig. Regarding the inlet velocity, as shown in Figure 48 (b), similar SOTEs were observed at HPO flowrates higher than 20%; however, at HPO flowrates lower than 20%, the highest inlet velocity of 3.4 m s^{-1} reported higher SOTEs compared to the lowest inlet velocity of 1.2 m s^{-1} . With respect to the recirculation flowrates, as shown in Figure 48 (c), same trends as with the inlet velocity were observed. The SOTE is strongly related to the $k_{LA20_transference}$ coefficient. Therefore, the higher the $k_{LA20_transference}$ at the evaluated operational parameters, the higher the expected SOTEs. As reported in Section 3.1, when evaluating the effects of the pressure, inlet velocity, and recirculation flowrates on the $k_{LA20_transference}$, it was observed that the pressure did not affect that much the $k_{LA20_transference}$; however, the inlet velocity and the recirculation flowrate showed a more pronounced effect on the $k_{LA20_transference}$.

A strong dependence of the SOTEs on the HPO flowrates was noticed. As shown in Figures 48 (a, b, c), at all the evaluated operational parameters, the SOTEs decreased at higher HPO flowrates. SOTEs of approximately 100% were reported at the lowest HPO flowrate of approximately 5%, while SOTEs of approximately 70% were reported at the highest HPO flowrate of approximately 40%. Similar trends were observed for all the evaluated operational conditions, except for the experiments conducted at an inlet velocity of 1.2 m s^{-1} where an SOTE as low as 70% was reported at the lowest HPO flowrate of 5% (Figure 48 (b)).

The higher the HPO flowrates, the higher both the interfacial oxygen transfer area per volume of liquid phase, as well as the mixing intensity (as reported in Section 3.1); therefore, the higher the $k_{LA20_transference}$ coefficient. However, as can be observed in Figures 46 (a, b, c), the relationship between the $k_{LA20_transference}$ and the HPO flowrate was not completely linear; that is, the $k_{LA20_transference}$ coefficients levelled off as the HPO flowrate increased. For instance, when doubling the amount of the HPO flowrate, the $k_{LA20_transference}$ did not double. That is, beyond a certain HPO flowrate, the rate at which the $k_{LA20_transference}$ increases is not fast enough to cope with the increase of the HPO flowrate. When the $k_{LA20_transference}$ transfer capacity is exceeded, usually at high HPO flowrates, there are more oxygen bubbles which cannot be fully dissolved into the liquid phase; these additional oxygen mass either accumulates inside the cone or leaves the system without getting dissolved into the solution. That extra undissolved oxygen causes a reduction on the SOTE (Figure 48 (a, b, c)). The Speer cone's capacity for dissolving the additional oxygen gas supply depends on its geometry and on the specific operational conditions. Similar observations were reported by (Ashley et al., 2014); in addition, simulations were performed by (McGinnis et al., 1998) confirming these findings.



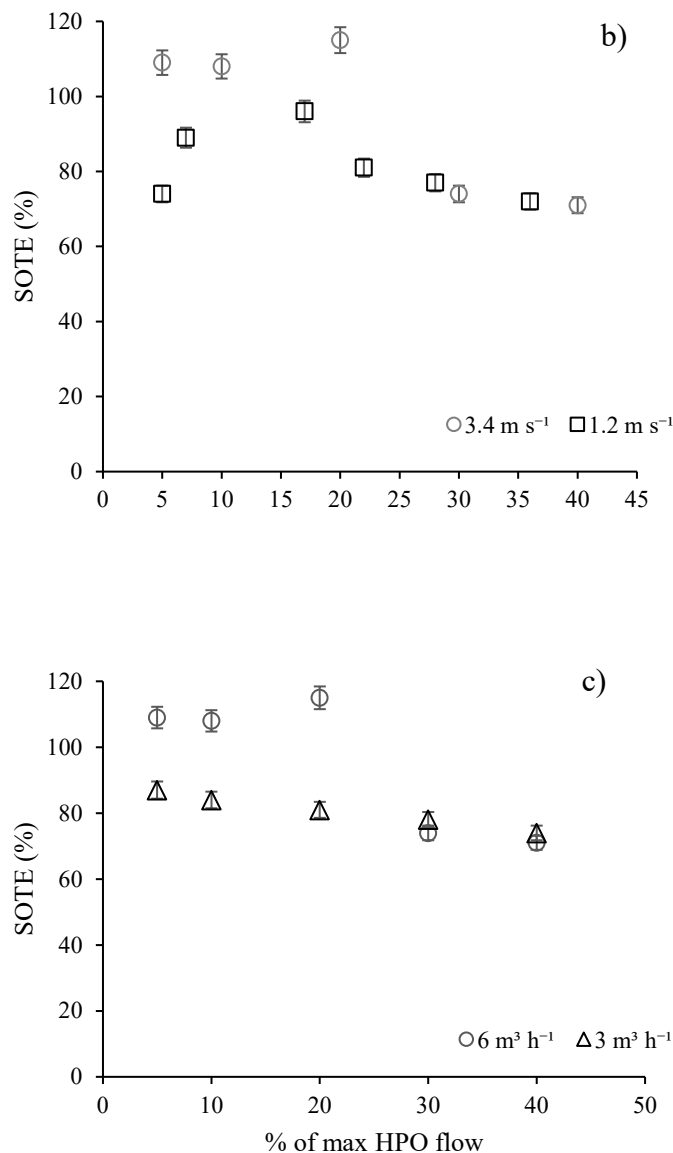


Figure 48 Influence of operational parameters on the SOTE (%) in clean water. (a) Pressure, (b) Inlet velocity, (c) Recirculation flowrate.

Depending on the Speece cone's design conditions, there is a specific set of HPO flowrate and operational conditions (pressure, inlet velocity and recirculation flowrate) at which the oxygen dissolution rate (given primarily by the $k_{L,a_{20_transference}}$) matches the amount of oxygen introduced by that specific HPO flowrate; at such point, 100% SOTE should be achieved. This research was carried out with a pilot-scale Speece cone system provided with a specific geometry and with a specific set of operational conditions (pressures, inlet velocities, and recirculation flowrates). For this specific system, SOTEs close to 100% were achieved almost at all the evaluated operational conditions but working at the low range of HPO flowrates. However, adjusting some of the Speece cone system's design parameters such as the geometry and the possibilities for working at higher inlet velocities and recirculation rates, higher SOTEs than the reported in this research may be eventually

obtained at high HPO flowrates; therefore, increasing the transfer capacity of the system. However, there will be eventually a maximum HPO flowrate at which the oxygen introduced to the system cannot be fully dissolved into the cone. That is, beyond certain HPO flowrate, the linearity between the $k_{La20_transference}$ coefficient and the HPO flowrate may be lost. The experiments conducted at an inlet velocity of 1.2 m s^{-1} reported a lower-than-expected SOTE of approximately 70%. When working at such low HPO flowrates both the interfacial oxygen transfer area per volume of liquid phase, as well as the mixing intensity are considerably affected. Presumably, the combined effects of such low inlet velocity together with such low HPO flowrates, set operational conditions for the Speece cone way distant from the ideal situation; therefore, lower SOTEs than expected were observed at that particular set of operational conditions.

One of the most important implications of these results refers to the selection of the values for the operational parameters (pressure, inlet velocity and recirculation flowrate). The selection can be done either maximizing the SOTRs regardless of the SOTEs, or maximizing the SOTEs at the expense of lower SOTRs. Therefore, if a higher SOTR is the desired output, the Speece cone system should be operated at the maximum pressure, inlet velocity, recirculation flow rate, and HPO gas flowrate. However, despite higher HPO gas flowrates will result in higher SOTRs, lower SOTEs are expected due to the mass transfer limitations and excess oxygen going to wastage. So the decision as to which HPO flowrate the cone should operate must be based on both the oxygen needs of the system, and on financial considerations regarding the system's capital versus operational costs.

Moreover, it is important to highlight that all the reported SOTEs in this study (even the lowest SOTE) were much higher than those observed in conventional diffused aeration systems (Mueller et al., 2002; Henze et al., 2008; Henkel et al., 2011; Xu et al., 2017). Compared to fine bubble diffused aeration, the Speece cone is much more efficient; depending on the operational conditions, nearly 100% of the supplied oxygen can be effectively delivered into the final receiving solution. The high efficiency aeration concept, or aeration on demand (dosing only what the process requires), has been held back by the misconception of using pure oxygen as being expensive. Currently in the wastewater treatment field very large amounts of air (with only 21% of oxygen) are applied to biological reactors through bubble diffusers at very low SOTEs of approximately 3% SOTE per meter of submergence - provided by brand new fine bubble diffusers (Henze et al., 2008). This conventional aeration concept disregards the vast amount of energy necessary to compress tremendous amounts of air which mostly end up back in the atmosphere carrying odors and other volatile compounds.

3.4. Oxygen transfer in mixed liquor

During the mixed liquor evaluations, the MLSS concentration ranged between 3.9 ± 0.4 to $4.9 \pm 0.8 \text{ g L}^{-1}$. The activated sludge was taken from a wastewater treatment plant operating

at stable conditions. Therefore, little variations on the activity of the sludge were observed during the entire experimental period. An average active fraction of 0.88 ± 0.03 gVSS gTSS⁻¹ was reported.

3.4.1. $k_{La_transference}$ and SOTR in mixed liquor and clean water

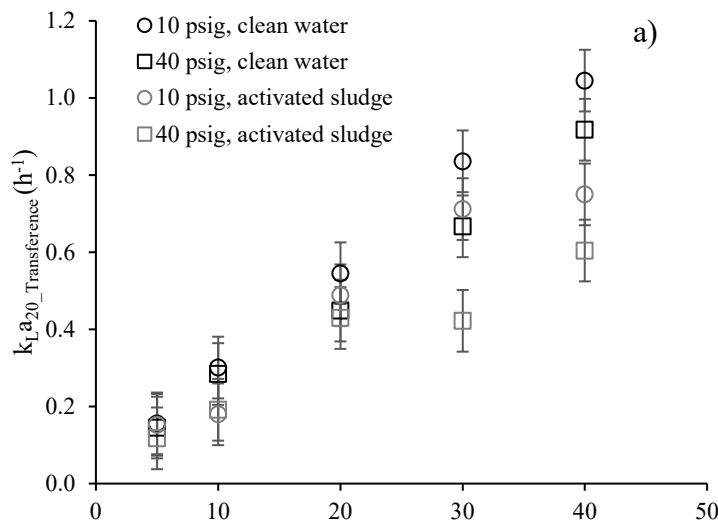
The impact of the MLSS concentration on the $k_{La20_transference}$ is presented in Figure 49 (a) as a function of the HPO flowrate for the two evaluated operational pressures. The $k_{La20_transference}$ increased with the HPO flowrate for the entire evaluated range following similar trends as observed in clean water. As depicted in Figure 49 (a), slightly higher $k_{La20_transference}$ coefficients were reported when working at the lowest pressure as also observed in the clean water experiments (Section 3.1). In addition, higher $k_{La20_transference}$ coefficients were observed at higher HPO flowrates, as it was also observed and reported in Section 3.1.

Lower $k_{La20_transference}$ values were obtained in mixed liquor compared to clean water due primarily to the influence of the total suspended solids from the mixed liquor matrix on the oxygen transfer. This trend is even more noticeable at high HPO flowrates as observed in Figure 49 (a). The $k_{La20_transference}$ both in clean water and in mixed liquor increased with the HPO flowrate until a HPO flowrate of approximately 20%; beyond that point, the increase of the $k_{La20_transference}$ in clean water was significantly higher compared to the increase of the $k_{La20_transference}$ in mixed liquor. The effects of the suspended solids on the oxygen transfer were more noticeable at HPO flowrates higher than 20% mainly due to the hindering effect that the suspended solids exert on the mass diffusion process. This effect has been extensively reported by several authors (Stephenson, 2000; Mueller et al., 2002; Krampe et al., 2003; Trussell et al., 2007; Gillot et al., 2008; Judd, 2008; Henkel et al., 2009; Racault et al., 2010; Henkel et al., 2011; Durán et al., 2016).

Figure 49 (b) shows the impact of the MLSS concentration on the SOTR at the evaluated pressures as a function of the HPO flowrates. Similar trends as reported in the clean water experiments described in Section 3.2 were observed. That is, the higher the pressure, the higher the SOTR. The pressure did not exhibit a strong direct impact on the $k_{La20_transference}$, although it did affect the SOTR by raising the saturation concentration inside the cone CsHPO as explained in Section 3.2. The effects of the suspended solids on the SOTR were also noticed at HPO flowrates higher than 20%. The same observations as for the k_{La} discussed in the previous paragraph can be considered to explain the effects of the suspended solids on the SOTR. The clean water experiments exhibited higher SOTRs compared to the mixed liquor experiments at the two evaluated operational pressures. However, both in clean water and in mixed liquor, the higher the HPO flowrate, the higher the SOTR. Therefore, when working with mixed liquor, the performance of the cone regarding the SOTR can be maximized by working at the maximum attainable pressure.

Figure 49 (c) shows the calculated alpha factor as a function of the HPO flowrate for the two evaluated operational pressures. The alpha factor was calculated as the ratio of the $k_{La20_transference}$ in mixed liquor and in clean water. The alpha factor indicates the impact of the mixed liquor (or process water) on the oxygen transfer efficiencies of the system. The $k_{La20_transference}$ coefficients both in clean water and in the mixed liquor matrices did not change significantly at the two evaluated pressures as indicated in Section 3.1 and in Section 3.4.1. Therefore, similar values were observed for the alpha factor at the two evaluated pressures.

A strong dependence of the SOTEs on the HPO flowrate was also noticed in the mixed liquor experiments (not shown in this study) as observed in the clean water matrix experiments (Section 3.3). SOTEs in mixed liquor of approximately 100% were observed at the lowest HPO flowrate of approximately 5%, while SOTEs in mixed liquor of approximately 50% were observed at the highest HPO flowrate of approximately 40%. That is, a decrease on the SOTEs at higher HPO flowrates was also observed in the mixed liquor experiments. However, lower SOTEs were reported at the same HPO flowrates on the mixed liquor matrix compared to the clean water matrix; mostly at high HPO flowrates. Therefore, alpha factors below 1.0 were obtained (Figure 49 (c)) at large HPO flowrates. Most of the experiments conducted in the mixed liquor matrix reported SOTEs which fitted well on the SOTE range previously described, except the experiments conducted at an HPO flowrate of 10% where the SOTEs dropped below the expected values. That is, lower alpha factors than expected were obtained at an HPO flowrate of 10% (Figure 49 (c)).



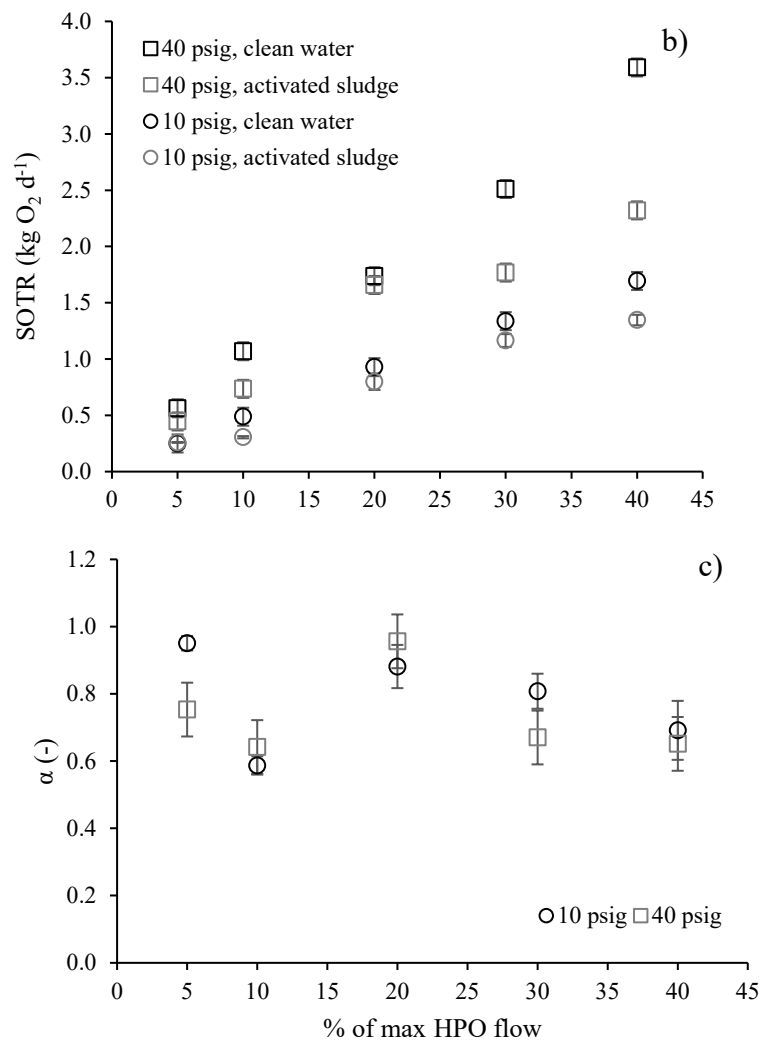


Figure 49 Influence of pressure and suspended solids on the process parameters in clean water and mixed liquor. (a) Mass transfer coefficient k_{La} (h^{-1}), (b) SOTR ($kg\ O_2\ d^{-1}$), (c) alpha factor (-)

3.4.2. Alpha factor

The higher the HPO flowrates, the higher the $k_{La20_transference}$ coefficient. However, the relationship between the $k_{La20_transference}$ and the HPO flowrate was not totally linear, and as observed in Figure 49 (a) the $k_{La20_transference}$ coefficients levelled off at high HPO flowrates. Similar trends were reported on the non-linear relationship between the $k_{La20_transference}$ and the HPO flowrate on the clean water experiments (Section 3.3). Nevertheless, the presence of the suspended solids in the mixed liquor matrix exerted a stronger negative effect on the oxygen transfer, and this effect was more pronounced at high HPO flowrates. Therefore, not only lower $k_{La20_transference}$ coefficients were obtained in the mixed liquor matrix compared to the clean water matrix, but also a more pronounced reduction of the $k_{La20_transference}$ was observed as the HPO flowrate increased. Therefore, the non-linearity between the $k_{La20_transference}$ and the HPO flowrates is further

exacerbated in the mixed liquor experiments compared to the clean water experiments. As explained on the clean water experiments (Section 3.3), as the HPO flow rates increases (that is more oxygen is introduced into the system), the $k_{La20_transference}$ coefficient also increases but at a lower rate. That is, the increase of the $k_{La20_transference}$ (that is, the oxygen dissolution capacity of the system) cannot cope with the amount of oxygen introduced into the system. This effect is even more pronounced in the mixed liquor matrix than on the clean water matrix; therefore, the reported SOTEs at a given HPO flowrates were lower in the mixed liquor matrix compared to the clean water matrix, and alpha factors lower than one were reported; particularly, at high HPO flowrates.

However, as the HPO flowrate is reduced, depending on the Speece cone's design, there is a specific set of HPO flowrate and operational conditions at which the oxygen dissolution rate (given by the $k_{La20_transference}$) matches the amount of oxygen introduced by that specific HPO flowrate; at such point 100% SOTEs and alpha factors of one can be achieved. SOTEs of approximately 100% and alpha factors of approximately one in the mixed liquor matrix were achieved and reported in this research. However, to achieve such high SOTEs and alpha factors the system needed to be operated at extremely low HPO flowrates. Therefore, it is possible to reach 100% SOTEs and alpha factors of approximately one in the mixed liquor matrix, but at expenses of working at low HPO flowrate; that is, working at a low oxygen transfer capacity.

This research was carried out on a pilot-scale Speece cone system provided with a given design to operate at a given set of operational conditions. Minor changes on the pilot-scale system design can be introduced such as the possibility to operate at larger inlet velocities and/or recirculation flowrates that may increase the oxygen dissolution capacity (that is the oxygen transfer capacity) of the system. Therefore, higher efficiencies and alpha factors than the reported in this research at high HPO flowrates may be eventually obtained enhancing the treatment capacity of the system. The experiments conducted at an HPO flowrate of 10% reported lower alpha factors than expected. When working at such low HPO flowrates both the interfacial oxygen transfer area per volume of liquid phase, as well as the mixing intensity in the Speece cone are considerably affected. That is, the operational conditions set for the Speece cone were far away from the optimal scenario; and therefore, lower than expected alpha factor were observed.

As previously discussed, the changes in the operational pressures in the cone when working with mixed liquor did not significantly affect the performance of the oxygen transfer in the cone regarding the alpha factor. One of the most important implications of this result, is that the cone should be operated at the highest possible pressure for maximizing the SOTR, considering that the alpha factor (and the SOTE) would be approximately identical regardless the selected pressure. However, the HPO flowrate did strongly affect the alpha factor and the SOTE in the mixed liquor. At higher HPO flowrates in mixed liquor, higher SOTRs are expected, but the SOTEs (and the alpha factors) will be negatively affected. That is, choosing the operational HPO flowrate set

point should be based on both the oxygen needs of the system and on financial considerations. However, one additional advantage of the Speece cone system compared to bubble aeration is that by fine tuning the HPO gas flowrate set point, SOTEs of approximately 100% and alpha factors of approximately one can be eventually reached.

Conventional diffused aeration systems (coarse and fine bubble) rely heavily on submergence to compensate for their low oxygen transfer efficiencies. Usually, alpha factors range from approximately 0.3 to 0.9 in aerobic reactors working at a water depth of 4 meters. For conventional activated sludge systems working at an MLSS concentration of 5 g L⁻¹ *Muller et al., (1995) and Krampe et al., (2003)* reported alpha factors of 0.8 and 0.6, respectively. In comparison, the maximum alpha factor obtained in this research was approximately 0.96 indicating promising result considering that the Speece cone pilot-scale system was working at a water depth of only one meter.

3.5. Applications in wastewater treatment

Superoxygenation systems have several advantages compared to conventional diffused aeration systems. These advantages include: i) SOTE of approximately 100%, ii) reduced impact of the suspended solids effects on the oxygen transfer, and most importantly iii) the elimination of the high submergence and large surface area requirements. The SOTE in conventional systems can be increased by having a deeper reactor, allowing the bubbles to travel longer distances increasing their contact time with the liquid phase; however, as the height of the reactor is increased, the available surface area is reduced, and the remaining surface area might not be large enough to accommodate a sufficient number of diffusers to deliver the required air flowrate. Most of the time the depth needs to be reduced to enlarge the surface area. A larger surface area provides enough space for placing additional diffusers at the expense of increasing the capital expenses and reducing the transfer efficiency. That is, larger volumes of air need to be compressed and pumped in order to meet the biological oxygen demand at a much higher and unnecessary operational cost.

The required oxygen needs for a hypothetical wastewater treatment plant with a treatment capacity for 100,000 population equivalent (P.E.) is presented in Figure 50. The theoretical required oxygen flowrate (dotted lines, Ton O₂ h⁻¹) was determined for a Speece cone and for a conventional fine bubble diffuser considering two alpha factors (0.5 and 0.8) and assuming a fixed aerobic reactor volume. The theoretical required oxygen flowrate includes both the biological oxygen demand of the 100,000 P.E. wastewater treatment system, as well as the additional oxygen needed to be supplied to overcome the oxygen transfer inefficiencies. That is, the theoretical required oxygen flowrate represents the gross amount of oxygen that needs to be supplied through the fine diffusers and/or Speece cone to fully satisfy the biological needs of the wastewater treatment system. Since different reactor depths were evaluated, the surface area available for placing the diffusers (and the corresponding maximum oxygen flow that could be

delivered through them) was reduced with increasing depths (that is, deeper tanks offer higher SOTEs). The gross oxygen needs calculations considered both the positive effect of water depth for fine bubble diffusers, as well as the negative effect of the alpha factor on the SOTEs. That is, the higher the submergence, the higher the SOTEs for the fine bubble diffusers; hence, the required oxygen flowrate is lower. Two typical alpha factors for MBR and CAS systems were evaluated of 0.5 and 0.8, respectively. Assuming an alpha factor of 0.8, the fine bubble diffuser system would require a gross oxygen flowrate demand of 1,100 and 170 Ton O₂ h⁻¹ at depths of 2 and 12 m, respectively. Assuming an alpha factor of 0.5, the fine bubble diffuser system would require a gross oxygen flowrate demand of 2,800 to 430 Ton O₂ h⁻¹ at depths of 2 and 12 m, respectively. These two series (dashed lines) are shown in Figure 50 together with the maximum oxygen discharge delivered by a fine bubble system covering 25% of the available reactor surface area (continuous line). That is, the dash lines represent the amount of oxygen required considering both the biological demand of the wastewater treatment system and the inefficiencies of the fine bubble diffusers in terms of alpha factors and SOTEs. On the other hand, the continuous line represents the amount of oxygen actually delivered (not dissolved) by a fine bubble diffuser systems covering 25% of the total available surface area. It was also assumed that no more than 25% of the available surface area would accommodate the fine bubble diffusers, and based on a standard fine bubble diffuser diameter, the maximum number of diffusers that could be placed on the available surface area was calculated.

The calculated maximum diffusers discharge is sufficient to supply enough oxygen for a system with the alpha factor of 0.8; however, for the systems with an alpha factor of 0.5, as it would be the case for MBRs, the fine bubble diffusers fail to supply the required oxygen flowrate. On the contrary, the Speece cone (dotted line) can deliver an oxygen flowrate which is fully independent on both the water depth and available surface area. The required oxygen flow for the Speece cone was calculated at 1.24 Ton O₂ h⁻¹; this value is 130 times lower than the best scenario with fine bubble diffusers at an alpha factor of 0.8 and at a water depth of 16 m. The Speece cone's profile was calculated using an alpha factor of 0.77 which yields an overall SOTE in mixed liquor of 60% at an MLSS concentration of 5.1 g L⁻¹. This SOTE corresponds to an experimental value observed in this research carried out at a pressure of 10 psig, an inlet velocity of 3.2 m s⁻¹, a recirculation rate of 6 m³ h⁻¹, and an HPO gas flowrate of 20%.

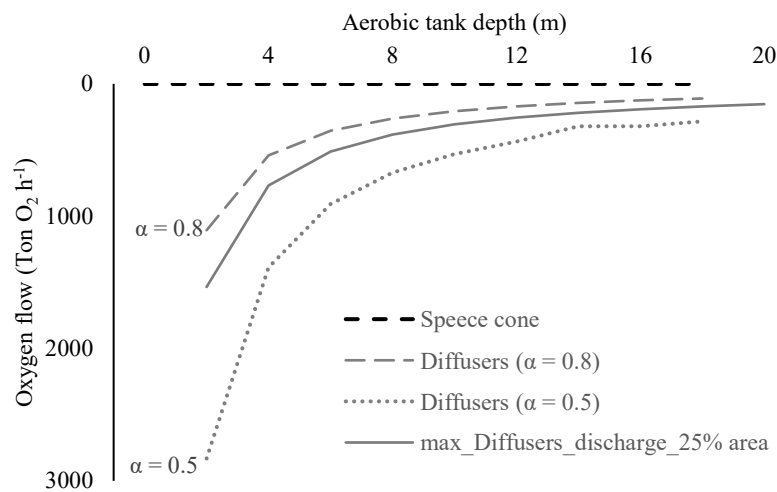


Figure 50 Comparison of the required oxygen input for 100,000 P.E. using a Speece cone system and fine bubble diffusers at different depths.

Superoxygenation methods such as the Speece cone may outperform the SOTE of current conventional diffuse aeration methods without most of their disadvantages. In this study, a maximum SOTE of 96% was observed in comparison to a maximum SOTE of approximately 25% for a fine bubble diffuser system operated at four meters depth in a clean water matrix. Moreover, the Speece cone may also be maintenance free since it does not require diffusers cleaning and replacement. Even though the oxygen transfer was affected by the suspended solids concentration in the mixed liquor, higher alpha factors were found for the Speece cone compared to conventional bubble diffuse aeration systems at the same MLSS concentrations. Further research is needed to evaluate the effects on the cone's oxygen transfer performance at a higher MLSS concentration range. If the Speece cone application in wastewater is successful, compact-portable, and more efficient wastewater treatment systems such as MBR operated at high MLSS concentrations can be designed. Some of the potential advantages of the Speece cone high MLSS MBR may include reduced footprint requirements, reduced generation of waste sludge, better portability, enhanced operational flexibility, and reduced operational costs, among others. Furthermore, and unlike conventional aeration system, the Speece cone system does not depend on the available surface area, nor on the system submergence. The oxygen dissolution process occurs in the Speece cone, not on the aerobic basin, eliminating the need for bubble diffusers. The findings of this research may introduce innovations in the wastewater treatment field such as the design of highly efficient low submergence portable sanitation systems for dealing with emergency sanitation applications.

4. Conclusions

This research demonstrated that the Speece cone technology can be implemented as an effective aeration alternative for supplying large amounts of dissolved oxygen at high oxygen transfer efficiencies in wastewater treatment applications. The evaluated experimental conditions suggest that high HPO flowrates lead to high SOTRs at the expense of low SOTEs. When working on a mixed liquor lower SOTEs can be observed compared to a clean water matrix due to the MLSS effect on the oxygen transfer. However, by selecting the proper HPO flowrate, alpha factors of approximately one (1) can be eventually obtained.

5. References

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4

OXYGEN TRANSFER IN ACTIVATED SLUDGE USING SIDESTREAM SUPEROXYGENATION IN A HIGH MLSS MEMBRANE BIOREACTOR

This Chapter is based on Barreto, C. M., Kolekar, P., García, H. A., Hooijmans, C. M., Herrera, A., & Brdjanovic, D. Oxygen transfer in activated sludge using sidestream superoxygenation in a high MLSS membrane bioreactor. Submitter to the Journal of Environmental Management, 2023.

Abstract

A pilot scale sidestream superoxygenation system known as the Speece cone was evaluated at delivering dissolved oxygen into activated sludge (AS) at mixed liquor suspended solids (MLSS) concentrations ranging from approximately 5 to 30 g L⁻¹. The liquid phase mass transfer coefficient k_{La} , Standard oxygen transfer efficiency (SOTE), Standard aeration efficiency (SAE), alpha factor, endogenous respiration, sludge viscosity and particle size distribution were determined. The evaluated variables influencing the process included the high purity oxygen (HPO) flow to the cone, pressure and inlet velocity. Results showed alpha factor values very similar to conventional aeration of 0.6 and 0.2 for MLSS concentrations 5 and 20 respectively, when operating at 5% HPO flow; in contrast, a higher HPO flow of 30% yielded alpha factors of 0.9 and 0.7 for the same MLSS concentrations. In addition to the HPO flow, the system performance was strongly influenced by the inlet velocity and the suspended solids concentration. The Speece cone exhibits very high performance in terms of SOTE and alpha factors even at high MLSS as compared to conventional aeration methods, presenting an alternative for operation at high biomass concentrations.

1. Introduction

Reducing the footprint requirements of wastewater treatment (WWT) systems is highly desirable when either there is not enough land available, or the WWT capacity of the system needs to be expanded. The required space for WWT systems can be significantly reduced, but mostly at the expense of increasing the mechanical complexity of the system. Meaning, adding additional equipment such as pumps, air blowers, and membranes, among others, with a direct impact on the capital expenditures (*Guender, 2000; Judd, 2008, 2010; Le-Clech et al., 2003; Stephenson, 2000*). Choosing a particular technology, amid a myriad of alternatives, always requires finding the best possible cost-benefit relation for the site-specific conditions.

For decades, the MBR technology has been recognized as the best option for achieving footprint reduction. The footprint restrictions happen more frequently in industrial scenarios, where the space for wastewater treatment competes with the space for industrial production. The conventional activate sludge (CAS) process and its variations such as the sequencing batch reactors (SBR)s or the moving bed bio-reactors (MBBR) are all viable alternatives for aerobic treatment; however, the MBR can achieve higher footprint reduction compared to the other alternatives (*Barreto et al., 2017; Barreto et al., 2018; Brookes et al., 2006; Durán et al., 2016; Germain et al., 2007; Gil et al., 2012; Karkare & Murthy, 2012; Kim et al., 2020; Krampe & Krauth, 2003; Lousada-Ferreira et al., 2010; Lousada-Ferreira et al., 2015; Muller et al., 1995; Racault et al., 2010*). MBR systems usually operate at a mixed liquor suspended solids (MLSS) concentration of 10 g L^{-1} .

The MBR treatment capacity can be increased by allowing higher biomass concentration in the bioreactor, meaning that operating at higher MLSS allows for a lower system footprint. However, MBR systems are usually provided with conventional fine bubble diffused aeration which limits the operation of MBRs at a maximum MLSS concentration of approximately 20 g L^{-1} (*Kim et al., 2015*). This is due to the poor oxygen transfer performance of fine bubble diffusers even at MLSS concentrations as low as 4 g L^{-1} (i.e., typical MLSS concentrations used in the CAS processes). The oxygen transfer efficiency for conventional fine bubble aeration ranges from 3 to 7 % per meter depth in clean water; such values are even further reduced when applying the alfa factor (α) or ratio between process-water to clean-water oxygen mass transfer coefficient. As such, most of the applied oxygen mass passes through the WWT system without being dissolved, introducing energy losses, and the unnecessary addition of installed power. Thus, fine bubble diffused aeration limits the footprint reduction that could be achieved in MBRs (*Barreto et al., 2017; Barreto et al., 2018; Kim et al., 2021*).

Bubble diffusers are very sensitive and negatively affected by the influence of suspended solids in the mixed liquor; their apparent good performance indicators only hold true for

MLSS concentrations far below 10 g L^{-1} . Alpha factors larger than 0.8 are commonly observed at MLSS concentrations lower than 10 g L^{-1} , while they rapidly fall to values lower than 0.2 and 0.1 before reaching MLSS concentration of 15 and 20 g L^{-1} , respectively (Germain *et al.*, 2007; Germain & Stephenson, 2005; Guender, 2000; Kim *et al.*, 2020; Muller *et al.*, 1995; Racault *et al.*, 2010; Zhichao Wu *et al.*, 2007). Larger alpha factors can be obtained by increasing the solid retention time (SRT) and optimizing process operation control (Muller *et al.*, 1995). The pressurized dissolution of pure oxygen into a liquid stream, also known as superoxygenation, is one among other alternative aeration methods for coping with the diminished oxygen mass transfer capacity exhibited by fine bubble diffused aeration (Abbas *et al.*, 2014; Adachi, 2015; Ashley *et al.*, 2014; Barber *et al.*, 2015; Barreto *et al.*, 2017; Barreto *et al.*, 2018; Khdhiri *et al.*, 2014; Kim *et al.*, 2020; Kothiyal *et al.*, 2016; Kumar *et al.*, 2017; Lazić *et al.*, 2012; Mitra *et al.*, 2016; Pi *et al.*, 2014; Tian *et al.*, 2015; Yang & Park, 2012; Zhuang *et al.*, 2016). Particularly, when operating in a side-stream configuration and in continuous operation mode, such as is the case for the Speece cone technology (Ashley *et al.*, 2014; Ashley *et al.*, 2008; Barreto *et al.*, 2018; Kowsari, 2008; McGinnis & Little, 1998; Singleton & Little, 2006; Speece, 1975; Speece, 2007; Tan *et al.*, 2014). Superoxygenation can deliver more oxygen under harsh conditions, meaning in those cases where the mass transfer is limited or hindered by external factors such as the suspended solids in a biological reactor. The main reason for sidestream superoxygenation to deliver higher oxygen transfer rates compared to diffused aeration is that the actual mass transfer takes place inside a pressurized vessel where the elevated pressure and consequently enlarged concentration gradient drives oxygen into the solution. On the contrary, conventional diffused aeration relies only on the oxygen concentration gradient between the gas and liquid phase which is itself almost five times lower compared to sidestream superoxygenation; conventional aeration uses air with 21% oxygen content rather than approximately 100% as used in superoxygenation. In addition, almost all the oxygen delivered to the pressurized vessel in sidestream superoxygenation will end up being dissolved into the solution; thus, higher oxygen transfer efficiencies can also be achieved in superoxygenation systems than in diffused aerations.

The oxygen transfer performance of the Speece cone in clean water and activated sludge (AS) at a MLSS concentration of approximately 5 g L^{-1} was previously evaluated by (Barreto *et al.*, 2017; Barreto *et al.*, 2018). Currently, there are no published studies reporting on the Speece cone oxygen transfer performance when operating a WWT system at mixed liquor concentrations higher than 5 g L^{-1} ; that is, the performance of the Speece cone sidestream superoxygenation technology has not been evaluated in the context of conventional MBRs operating at higher MLSS concentrations than CAS systems. Moreover, the higher the concentration of MLSS in the MBR the lower the footprint requirements. Thus, evaluating the performance of the Speece cone at MLSS

concentrations even higher than the MLSS of conventional MBR systems is highly desirable. Preliminary results show the potential of implementing the Speece cone as a novel method for precision aeration in the wastewater treatment field (*Barreto et al., 2017; Barreto et al., 2018; Kim et al., 2021*). The introduction of this technology may allow the design of compact systems and in some cases could serve as an alternative for upgrading existing wastewater treatment plants (WWTP) from CAS to MBR eliminating the need for diffusers, and more importantly, reducing the specific power demand.

This study aimed at evaluating the oxygen transfer performance of a Speece cone system in activated sludge at MLSS concentrations ranging from 5 to 30 g L⁻¹. The oxygen transfer performance was assessed by determining the following parameters: the liquid phase mass transfer coefficient (k_{LA} ; h⁻¹); the standard oxygen transfer efficiency (SOTE; %); the specific oxygen uptake rate (SOUR; mgO gVSS⁻¹); and the alpha factor (α). Different operational conditions (process variables) in the Speece cone were modified to evaluate the oxygen transfer performance, including the inlet velocity to the Speece cone, the high purity oxygen (HPO; kg d⁻¹) flowrates to the Speece cone, the pressure inside the Speece cone system, and the biomass concentration in the biological WWT system expressed as the MLSS concentration. The activated sludge apparent viscosity (μ_{app} , mPa s) and the particle size distribution (PSD; %) were also determined.

2. Materials and methods

a. Experimental setup description

A Speece cone system (*Speece, 1975*) was built for the aeration of a pilot scale membrane bioreactor (MBR) in a side-stream configuration. The Speece cone system consists of a cone-shaped pressure vessel known as the Speece cone (ECO2 Oxygen Technologies LLC, USA), an oxygen mass flow controller (Alicat Scientific, MC-5SLPM-D, USA), a progressive cavity sludge pump with a variable frequency drive (Netzsch, NM045BY02512B, Germany), an electromagnetic flow transmitter, and a control panel. The pilot MBR had an effective volume of approximately one cubic meter equipped with submerged tubular membranes (MEMOS, Germany). The system components are depicted in a process flow diagram in Figure 51. The oxygen transfer performance of the Speece cone was evaluated using the same pilot scale membrane bioreactor (MBR) described in (*Barreto et al., 2017; Barreto et al., 2018*), consisting of an aerobic bioreactor (volume 1.1 m³) provided with an ultrafiltration tubular membrane module (area 20 m²) and a pure oxygen fed Speece cone system for sidestream superoxygenation with an installed oxygen delivery capacity of 5 kg O₂ d⁻¹. The experimental work was carried out at the Delft Blue Innovations Research Hall at the Harnaschpolder wastewater treatment plant in Delft, The Netherlands (www.delftblueinnovations.nl).

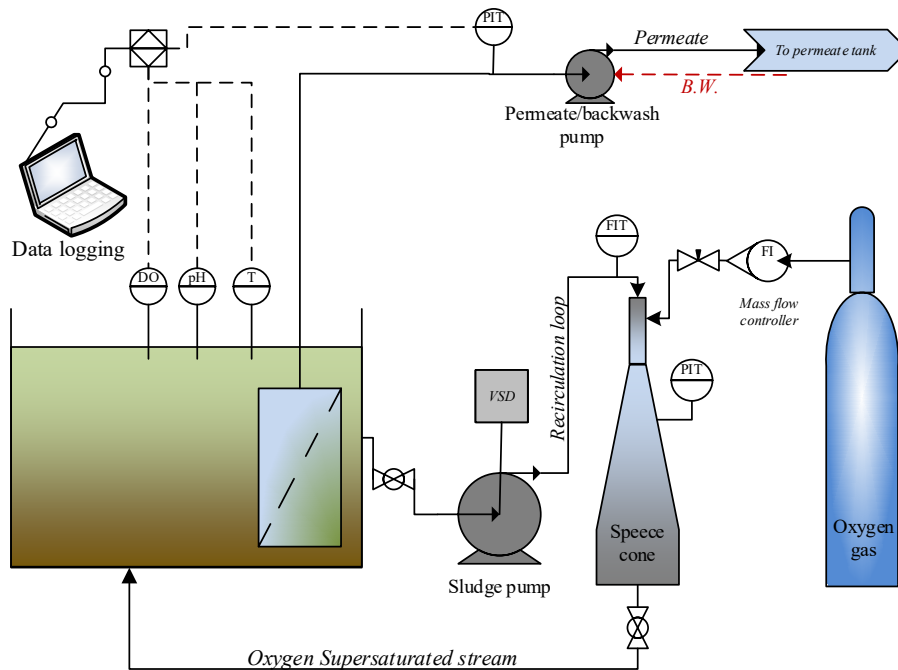


Figure 51 Process flow diagram (PFD) of the sidestream superoxygenation setup.

b. Experimental procedure

The oxygen mass transfer performance of the Speece cone system was determined as described by (Barreto *et al.*, 2018) following the reaeration method as defined by the Standards of the American Society of Civil Engineers (ASCE, 1997). The addition of an oxygen scavenger was not necessary in this case due to the presence of active biomass depleting the dissolved oxygen (DO) in the system by aerobic respiration. The oxygen intrusion due to the atmospheric oxygen was measured and considered when carrying out the oxygen transfer performance determinations. Activated sludge was fed from the MBR to the cone in a recirculation loop. The sludge entered the cone at the top section where it is the narrowest (the cone's throat or neck), and so at this point the inlet velocity is the highest. The mixed liquor inlet velocity to the cone was set using PVC inserts of different diameters depending on the applied flowrate ranging from 3 to 6 m³ h⁻¹. After setting the flowrate, the working pressure in the cone was controlled using a valve at the discharge line. The oxygen mass flow rate was set on the mass flow controller prior to starting the DO data loggers. DO was logged for a couple minutes before the test in order to confirm all the previously mentioned variables were properly set. Then, the oxygen flow to the cone was started and recorded until the DO reached saturation concentration. After each experiment was finished, the data logging was kept recording the activated sludge respiration under the very same MLSS concentration and temperature. Specific OUR (SOUR) determinations allowed to check for the activated sludge activity. Activated sludge taken from the Harnaspolder wastewater treatment plant (Delft, The Netherlands) was used in these experiments. In order to increase the MLSS concentration,

it was necessary to concentrate the mixed liquor using the membrane module to extract permeate while adding fresh activated sludge until the desired MLSS was achieved. The MBR was operated under a non-continuous flow regime meaning that sludge was added until the target MLSS concentration was reached.

c. Experimental design

Batch experiments were carried out aimed at establishing the oxygen mass transfer capabilities of the aeration device under increasing MLSS concentrations or levels by conducting mass balances. The dissolved oxygen data series were processed in triplicates to yield a mean value for the mass transfer rate coefficient (k_{La} ; h^{-1}), the standard oxygen transfer efficiency (SOTE; %) and the alpha factor (α). Each experiment was performed three times at every MLSS level in what was called a run. High purity oxygen (HPO) was fed to the cone at increasing oxygen flowrates from 5 to 40% of its maximum theoretical saturation capacity in the cone at the evaluated experimental conditions.

The mixed liquor recirculation flow and pressure through the system were kept at $6 \text{ m}^3 \text{ h}^{-1}$ for all the evaluated MLSS levels with the exception of the last one (28 g L^{-1}) for which such flowrate could not be fully sustained. The pressure in the cane ranged from 10 to 60 psig. The inlet velocity was prioritized and kept within the optimal range between 3 to 3.4 m s^{-1} for MLSS 5, 10, 12 and 28 g L^{-1} ; for MLSS concentrations of 14, 18 and 20 g L^{-1} , a higher inlet velocity of 4.81 m s^{-1} was applied. The detailed experimental conditions for the different MLSS levels are presented in Table 3. The focus of the research was on exploring the effect of the MLSS concentrations in the HPO range from 5% to 40% of the theoretical maximum saturation capacity.

Table 3 Superoxygenation in activated sludge under different testing conditions

MLSS (g L^{-1})	Recirculation flow ($\text{m}^3 \text{ h}^{-1}$)	Pressure (psig)	Inlet velocity (m s^{-1})	HPO (%) (kgO d^{-1})	
5	6	25	3.4	5%	0.83
				10%	1.66
				20%	3.32
				30%	4.98
				40%	5.39
10	6	25	3.4	5%	0.83
				10%	1.66
				20%	3.32
				30%	4.98
				40%	5.39
12	6	25	3.4	5%	0.83

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				10%	1.66
				20%	3.32
				30%	4.98
	6	48	3.4	5%	1.31
				10%	2.51
				20%	5.02
				30%	5.66
				40%	8.28
14	6	60	4.81	5%	1.56
				10%	3.12
				20%	6.25
	6	60	1.2	30%	9.37
				33%	10.31
18	6	60	4.81	5%	1.56
				10%	3.12
				20%	6.25
				30%	9.37
				33%	10.31
	6	60	1.2	10%	3.12
				20%	6.25
20	6	60	4.81	5%	1.56
				10%	3.12
				20%	6.25
				30%	9.37
				33%	10.31
	5	10	2.83	5%	0.43
			3.34	10%	0.86
				19%	1.64
				30%	2.58
				39%	3.36
28	4.5	25	2.55	5%	0.62
			3.01	10%	1.24
				20%	2.49
				30%	3.73
				40%	4.98
				50%	6.64

d. Data analysis

Raw data series were processed in triplicates to yield a mean value and the standard deviation represented by a single data point for each one of the aeration runs. The net oxygen mass transfer was determined using the mass balance presented in Equation 58.

$$V \left(\frac{dC_{MBR}}{dt} \right) = \text{Net OTR}_{(MBR)} + \text{Intrusion} - \text{OUR}_{en} \quad (58)$$

The atmospheric oxygen intrusion to the MBR was accounted for, performing identical tests in activated sludge without the addition of oxygen gas. The intrusion mass transfer rate coefficient $k_{LA_intrusion}$ was calculated at each MLSS concentration using Equation 59.

$$V \left(\frac{dC_{intrusion}}{dt} \right) = K_{L}a_{-Intrusion} (C_s - C_{MBR}) \times V - \text{OUR}_{en} \quad (59)$$

e. Analytical procedures

The dissolved oxygen (DO) concentration was measured using DO probes (CellOx325, Germany) mounted on portable data loggers (WTW3310, Germany) with a measuring range between 0 to 50 mg L⁻¹ ± 0.5%. The total and volatile suspended solids in the sludge were measured according to the standard methods for the examination of water and wastewater (AWWA, 2014). The oxygen uptake rate (OUR) in the activated sludge was determined following the U.S. Environmental Protection Agency (EPA) method 1683 Specific OUR in biosolids (EPA, 2001). In addition, sludge particle size distribution and viscosity were determined using a laser particle counter (Microtrac, Japan; 10⁻³ to 10³ μm) and a rheometer (Anton Paar, Switzerland; 10⁻² to 10³ s⁻¹), respectively.

i. Determination of the mass transfer coefficient k_{La} , Oxygen transfer rate OTR, and Oxygen transfer efficiency OTE.

The parameter estimation in this study followed the same approach as described in (Barreto *et al.*, 2018). The process k_{La} was determined using Equation 60 resulting from the overall mass balance applied to the system and the results were standardized to 20°C (Equation 614).

$$V \frac{dC_{MBR}}{dt} = [k_{La}(C_{s\text{HPO}} - C_{MBR})] \times V + [k_{La\text{Intrusion}}(C_s - C_{MBR})] \times V - \text{OUR}_{en} \quad (60)$$

$$k_{La20} = k_{LaT} \theta^{(20-T)} \quad (61)$$

The oxygen transfer rate (OTR; kg O₂ d⁻¹), was calculated as depicted in the first term on the right-hand side of Equation 58 and in Equation 59 as the total change in concentration

minus the sludge respiration. The OTR was standardized to 20°C by applying the expression described in Equation 62.

$$\text{SOTR} = \text{OTR} \times \theta^{(20-T)} \quad (62)$$

The standard oxygen transfer efficiency (SOTE %), was determined as the fraction of the supplied oxygen gas that has been measured as dissolved oxygen in the system (Equation 63), meaning the gas to liquid transfer process efficiency.

$$\text{SOTE} = \frac{\text{Oxygen}_{\text{measured (dissolved)}}}{O_{\text{supplied (gas)}}} \times 100 \quad (63)$$

ii. *Alpha factor*

Alpha, expressed as the ratio between the clean-water to activated sludge k_{La} (h^{-1}), was determined using Equation 64. The k_{La} corresponding to clean-water conditions was obtained in identical experiments performed in advance before the addition of activated sludge to the bioreactor.

$$\alpha = \frac{k_{La_process\ water}}{k_{La_clean\ water}} \quad (64)$$

iii. *Apparent Viscosity*

The activated sludge apparent viscosity occurring inside the cone was determined following the approach presented by (Lopez *et al.*, 2015) as a function of the solids concentration (TSS; g L^{-1}), the fluid velocity (v ; m s^{-1}), and the pipe diameter (d ; m). The fluid consistency index (k ; mPa s^n) and the flow index (n) were also determined using the coefficients a_1 to a_5 presented in the research by (Lopez *et al.*, 2015) as depicted in Equations 65 to 67.

$$\mu_{app} = k \left(\frac{3n+1}{4n} \right)^n \left(\frac{8u}{d} \right)^{n-1} \quad (65)$$

$$k = a_1 e^{a_2 TSS^3} \quad (66)$$

$$n = 1 - a_4 TSS^{a_5} \quad (67)$$

3. Results and discussion

This section presents the major findings regarding the most commonly used indicators for the assessment of the oxygen transfer performance within the wastewater field, namely the liquid phase mass transfer coefficient (k_{LA} , h^{-1}), the standard oxygen transfer efficiency (SOTE; %), and the alpha factor (α). The activated sludge respiration (SOUR), viscosity, and particle size distribution are also reported in this section. The standard aeration efficiency (SAE; kgO kW h^{-1}) is also presented at the end of the section.

a. Liquid phase mass transfer coefficient (k_{LA})

Following similar trends as observed for other aeration methods, the oxygen mass transfer performance was negatively affected by increasing the solids concentration (*Barreto et al., 2017; Barreto et al., 2018; Cornel et al., 2003a, 2003b; Durán et al., 2016; Germain et al., 2007; Henkel et al., 2009; Jiang et al., 2017; Kim et al., 2020; Krampe & Krauth, 2003; Krause et al., 2003; Le-Clech et al., 2003; Lousada-Ferreira et al., 2010; Lousada-Ferreira et al., 2015; Muller et al., 1995; Poyatos et al., 2009; Racault et al., 2010, 2011; Rodriguez et al., 2011; Trussell et al., 2001; Trussell et al., 2007; Wagner et al., 2002; Zhichao Wu et al., 2007; Xu et al., 2017*). Such a reduction in the mass transfer capacity can be observed as a decreasing k_{LA} as a function of the MLSS concentration in Figure 52a. There was an inverse relationship between the MLSS concentrations and the mass transfer performance. The higher the MLSS concentration, the lower the mass transfer coefficient k_{LA} due to physical hindering of the mass transfer process. Nevertheless, the solids concentration is only one among other variables affecting the mass transfer from the gas to the liquid phase. Solids retention time (SRT), turbulence, depth, viscosity, and air flowrate can all have a strong influence on the k_{LA} ; therefore, all these factors must be considered together when evaluating and comparing the performance of different aeration methods.

The k_{LA} values obtained under the experimental conditions described in Table 3 are presented in Figure 52 as follows. Figure 52a and 52b show the changes on the k_{LA} as a function of the MLSS concentrations and different HPO flowrates. While Figures 52c and 52d show the changes of the k_{LA} as a function of the pressure. The effect of the inlet velocity over k_{LA} is presented in Figure 52e. In addition, the SOUR as a function of the MLSS is presented in Figure 52f.

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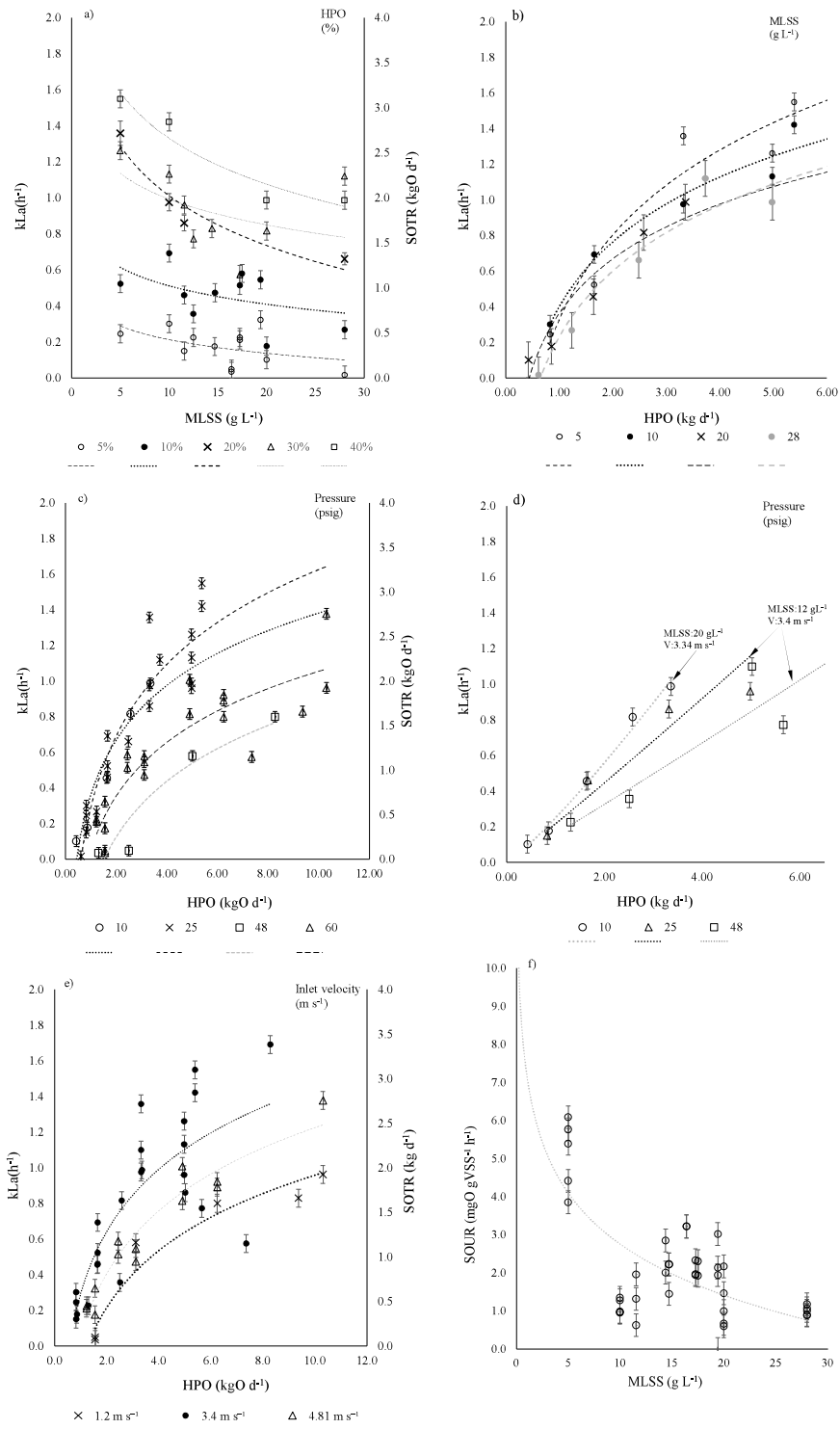


Figure 52 Oxygen mass transfer coefficient kLa for different a) HPO flows, b) MLSS concentrations, c, d) Pressure, and e) Inlet velocities; f) indicates the standard oxygen uptake rate (SOUR) as a function of the MLSS concentrations.

It is important to clarify that k_{LA} data points for each curve in Figure 52 for a given MLSS level were obtained by fine tuning all variables involved in the system as described before (Table 3), meaning the plotted markers represent the resulting combination of all factors as reported, being MLSS just one of them. Other variables can have a stronger influence over the process, therefore the MLSS concentration negative impact can be overcome by optimizing for instance the inlet velocity or the HPO flowrate as discussed further below.

From the experimental conditions evaluated in this study, as depicted in Table 3, k_{LA} coefficients of up to 3.24 h^{-1} and 1.69 h^{-1} were determined in clean water (CW) and activated sludge (AS), respectively in an MLSS concentration range from approximately 5 to 30 g L^{-1} . The decreasing k_{LA} trend as a function of the MLSS concentration followed a similar path for the different evaluated HPO flowrates (Figure 52a). The negative influence of the MLSS concentration on the k_{LA} seemed to be amplified when increasing the HPO flowrate. That is, the range between $k_{LA_{min}}$ and $k_{LA_{max}}$ is larger for the 40% HPO flowrate than it is for the 5% HPO flowrate. The largest values for k_{LA} in terms of gas flowrate was observed between 20% and 40% HPO (Figure 52a). On the contrary, the variation observed on the k_{LA} as a function of the different MLSS concentrations was lower than as function of the HPO flowrates as Figure 52b depicts. That is, the k_{LA} was not that negatively affected by the MLSS concentrations as much as it was positively affected by the HPO flowrate under similar operational conditions.

The negative impact of the solids on the mass transfer can be overcome by applying larger HPO flowrates (Figure 52a, 52b). Meaning the system's output can be improved on demand by adjusting the HPO flow to meet the demand fluctuations allowing precise dosing and minimizing wastage. However, as it was pointed out previously by (Barreto *et al.*, 2018), a HPO flowrate higher than 40% is not recommended because it causes the breakage of the gas pocket at the cone's neck. This latter condition may favour gas wastage since a cone that is fed gas in excess lowers its ability to effectively deliver it into the liquid phase via enhanced contact because the gas-water interphase is disrupted or even broken. Without a dynamic interphase the cone loses its geometrical advantage and relies only on the pressure gradient as the driving force for the gas dissolution, like a regular saturation vessel.

Adjusting the HPO flowrate determines the process intensity by contributing to: a) an enhanced gas-liquid contact; b) higher turbulence under higher HPO flowrates; and c) via concentration gradient by providing an oxygen rich gas-liquid interphase, as long as it is kept within the recommended range below 40% HPO.

Pressure has a major role in the gas-liquid equilibria since it defines the dissolved oxygen saturation concentration. When looking at the pressure effects over the k_{LA} in Figures 52c and d, similar k_{LA} were obtained under different pressures at comparable HPO rates. However, an increment in pressure did not necessarily lead to higher k_{LA} values, as observed in Figure 52c, where higher k_{LA} values were obtained at low pressure values of

10 and 25 psig compared to at high pressure values of 48 and 60 psig. This is further illustrated in Figure 52d; still the highest k_{La} values were obtained at the lowest pressure (10 psig), despite the latter corresponding to a higher MLSS concentration of 20 g L⁻¹. These results are in accordance with previous studies conducted by (Barreto *et al.*, 2018). Such studies attributed the effect of higher pressure on lower k_{La} to: i) bubble size reduction inside the cone which disrupts the size-buoyancy balance leading to bubble washout and reduced contact time (Barber *et al.*, 2015; Barreto *et al.*, 2018; McGinnis & Little, 1998, 2002), and ii) reduction in the mass exchange rate due to stagnation at the boundary layer via reduced gas diffusivity coefficient (k_G) (Versteeg *et al.*, 1987).

As presented in Figure 52c, 52d and 52e, the positive effect of the evaluated variables (pressure and velocity in particular) on the k_{La} showed an optimum value between the highest and lowest condition being tested. Meaning that at least for these two variables, at the evaluated experimental conditions, a higher pressure and/or velocity did not necessarily result in a larger process intensity or in an improved performance. Pressure raises the maximum DO concentration delivered by the cone by setting the maximum saturation concentration; however, larger pressures do not lead to higher k_{La} values alone.

Despite the direct relation between pressure and saturation concentration, the overall effect of the pressure on the k_{La} was in some cases overpowered by the combined effect of the many other variables involved. That is, higher operating pressures alone may not lead to enhanced performance if other variables are not also optimized. The k_{La} values corresponding to a higher-pressure series falling below those k_{La} values obtained at lower pressures could be seen as the result of a sub-optimal setting of the other variables; this finding can also be used as a main indicator for process optimization.

Inlet velocity controls the process efficiency by enhancing the contact between the liquid and the gas phase, and by temporarily modifying the sludge apparent viscosity. The inlet velocity in conjunction with the cone's distinctive geometry contributes to maximizing the contact time between the gas bubbles and the bulk liquid, taking advantage of the relation between bubble size and buoyancy. The mass transfer rate or net mass output is strongly influenced by the cone's dimensions meaning its inlet diameter, as well as by the target DO concentration and dilution factor of the supersaturated stream. The specific role of the inlet velocity is further discussed in the viscosity section.

The mass transfer coefficient k_{La} is usually determined on-site at the facility level, meaning that different facilities using the same aeration method can have significant variations in their aeration performance. Such variations are derived from the differences in the process configuration, mixing conditions, specific air flow, temperature, viscosity, MLSS concentration, among others. For this reason, the k_{La} cannot be compared without taking into account the aforementioned parameters; for instance, it is expected to observe a viscosity influence on the k_{La} as the MLSS concentration increases (Ashley *et al.*, 2014; Barreto *et al.*, 2018; Durán *et al.*, 2016; Germain *et al.*, 2007; Germain & Stephenson,

2005; Kim *et al.*, 2020; Rodriguez *et al.*, 2012; Terashima *et al.*, 2016; Trussell *et al.*, 2007; Z. Wu *et al.*, 2007). The k_{LA} coefficient would always be extremely useful for comparing the performance of different aeration methods, although the comparison must be carried out under the same mixing conditions and for the same sludge characteristics. That is, the k_{LA} coefficient can be substantially improved by enhancing the mixing intensity, which increases the oxygen transfer performance of a system. However, increasing the mixing intensity would require an extra power input with an impact on the capital and operational costs. In addition, depending on the process conditions in the WWT system, different sludge matrices may result in diverse rheological properties which also can impact the oxygen transfer. Activated sludge can exhibit different properties under different process configurations, operation regimes, feed composition and environmental conditions (Lousada-Ferreira, 2011; Lousada-Ferreira *et al.*, 2010; Lousada-Ferreira *et al.*, 2014; Lousada-Ferreira *et al.*, 2015; Moreau *et al.*, 2009a; Yang *et al.*, 2016). Therefore, comparing the k_{LA} coefficients from different aeration methods may be deceiving; particularly, when selecting the most appropriate technology for full scale applications.

Being aeration the most energy demanding process in WWTPs, the use of specific process indexes which are objective and indicate the overall impact over the process are highly needed. Examples of such potential indexes include: (i) the net mass of oxygen ending in the liquid phase able to take part in the biological organic matter degradation process (i.e., the specific oxygen transfer rate (SOTR); kgO d^{-1}) instead of just the applied airflow; (ii) the energy expenditure expressed as the standard aeration efficiency (SAE, kgO kW h^{-1}) considering all the aeration-mixing equipment taking up power besides the air blowers; and (iii) the specific energy demand (SED; $\text{kW h}^{-1} \text{m}^{-3}$).

b. Specific oxygen uptake rate (SOUR)

The highest SOUR value of $6 \text{ mgO gVSS}^{-1} \text{ h}^{-1}$ was obtained when operating the system at an MLSS concentration of 5 g L^{-1} (Figure 52f), which corresponded to a virtually unchanged recirculating sludge from the CAS source used to seed the MBR. All the other evaluated MLSS concentrations up to 30 g L^{-1} exhibited lower SOUR as compared to the CAS sludge; such lower SOUR values fall well within the typical range for endogenous respiration in full scale MBRs (Figure 52f). SOUR values were also determined on the same pilot setup, including exogenous respiration determination, in a previous evaluation carried out by the authors assessing the performance of such system in a continuous operation regime (Barreto *et al.*, 2017). Having a high shear being applied on the sludge is known for affecting the cell viability causing cell damage and diminishing the conversion rates and/or synthesis of bioproducts (Esperança *et al.*, 2019). The SOUR measured in this study indicates that beyond 5 g L^{-1} there was a decrease in the biological activity after exposing the sludge to a very large endogenous respiration period at a high mechanical shear; however the SOUR values fall well within the typical range for endogenous respiration in full scale MBRs (Figure 52f). Though, when the system was

operated in continuous operation regime, it was able to successfully treat domestic wastewater at MLSS up to 28 g L^{-1} as reported by (Barreto et al., 2017).

c. Standard oxygen transfer efficiency (SOTE)

The suspended solids influence on the SOTE can be compensated by applying a larger HPO flowrate, up to a certain HPO flowrate – the breaking point. Beyond that point bubble wash-out begins to be observed, wasting undissolved gas escaping at the outlet of the cone; thus, reducing the SOTE (Figure 53a). It is worth mentioning that very similar results regarding the SOTE were obtained for both the lowest and highest MLSS concentrations, meaning that the SOTE is not affected by the solids concentration as much as conventional aeration methods are (Figure 53a). Applying different HPO flowrates influenced the process efficiency more strongly at lower HPO flow rates. That is, when HPO is 40%, the SOTE remains virtually unchanged for all MLSS, however, for HPO 5%, the transfer efficiency can differ in a range of 80% at 10 g L^{-1} and 10% at 28 g L^{-1} . Figure 53a and 53b show that under increasing MLSS concentrations, the influence of the HPO flowrate on the SOTE appears to diminish.

It can be stated that all values converge in a band between 40 and 60% efficiency at the evaluated experimental conditions, some process conditions favour more efficient operation while others can be optimized. This process is mainly governed by the extent of gas wastage caused by excessive gas being fed, ultimately breaking, or at least disrupting the gas-liquid interphase or gas pocket inside the cone. This indicates that a process efficiency of at least 50% can be maintained regardless of the MLSS when HPO is 40%. A much higher SOTE close to 100% can be obtained with HPO below 40%. Higher HPO flowrates lead towards oxygen gas wastage at the discharge point when the gas input surpassed the system's gas dissolution rate.

When the gas to liquid ratio is exceeded, more gas is present at the interphase, then the process mass transfer rate is negatively affected. Excessive gas or gas applied in excess accumulates at the cone's neck expanding the gaseous phase volume; thus, disturbing the enhanced contact and reduced viscosity effect on the oxygen transfer.

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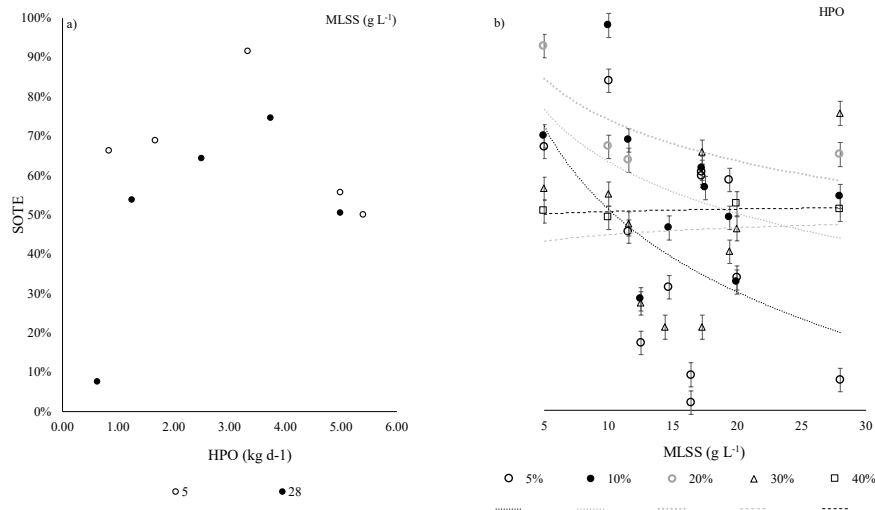


Figure 53 Standard oxygen transfer efficiency SOTE for different MLSS concentration and HPO flowrates.

The standard oxygen transfer efficiency (SOTE) yielded a variety of results, showing the wide range of operation and ability to swap features such as efficiency and mass transfer rate. The operational flexibility of the Speece cone is evident and remarkable. By adjusting the operational parameters, the operation that favours can be chosen transfer efficiency over transfer rate or vice versa. In addition, it can adapt to fluctuating oxygen demands related to daily patterns related to increased oxygen demand, meaning one size can serve a range of WWTP sizes, particularly, the decentralized small-scale ones.

d. Alpha factor, viscosity, and particle size distribution (PSD).

Figure 54a shows that the MLSS influence on reducing alpha factor is compensated by the HPO flowrate, since higher alpha values were obtained at higher HPO flowrates both at low and high MLSS concentration levels. For instance, in Figure 54a higher alphas observed on the top left corner corresponded mostly to low MLSS concentrations namely 5, 10, 12 g L⁻¹; at similar HPO, the alpha factors decreased as the MLSS increased up to 28 g L⁻¹ meaning that there was indeed a negative impact of the suspended solids over the oxygen transfer. However, this effect was overpowered for instance by the inlet velocity; that is, when the inlet velocity was not optimal, the obtained alpha factors were significantly low even at the lowest evaluated MLSS concentrations (at which the mass transfer is less hindered) as elaborated below. When the HPO flowrate was approximately 3 kg d⁻¹, the 20 and 28 g L⁻¹ curves yield alpha factors close to 0.5 (Figure 54a), while the 18 and 20 g L⁻¹ resulted in $\alpha < 0.3$; i.e., when the inlet velocity was not optimal lower alpha factors are observed even at lower solids MLSS concentrations (Figure 54a). As indicated in Figure 54b, the higher alpha factor values were reported when operating at an optimal inlet velocity of approximately 3 m s⁻¹, while the lower alpha factor values were reported when operating at other (sub-optimal) inlet velocities of either 1.2 or 4.81

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m s^{-1} . This demonstrates the cone super oxygenation flexibility, in this case, the 18 and 20' g L^{-1} series were obtained under sub-optimal conditions when the velocity was too high causing bubbles to wash out. Two different groups can be observed when the alpha factors are classified based on the inlet velocity as shown in Figure 54b. The first group included the higher alpha values of above 0.5 for all the evaluated MLSS concentration levels. Such higher alpha factor values were observed when the inlet velocity was set between 3 and 3.4 m s^{-1} . Lower alpha values were obtained for higher and lower velocities namely 4.8 and 1.2 m s^{-1} , indicating an optimum operational value for the inlet velocity range between 3 - 3.4 m s^{-1} (Figure 54b). These observations are in accordance with the k_{LA} results observed for different inlet velocities presented in Figure 52e, indicating the role of the inlet velocity as the driving force that allows an improved performance through a momentary change in the apparent viscosity as it will be discussed below.

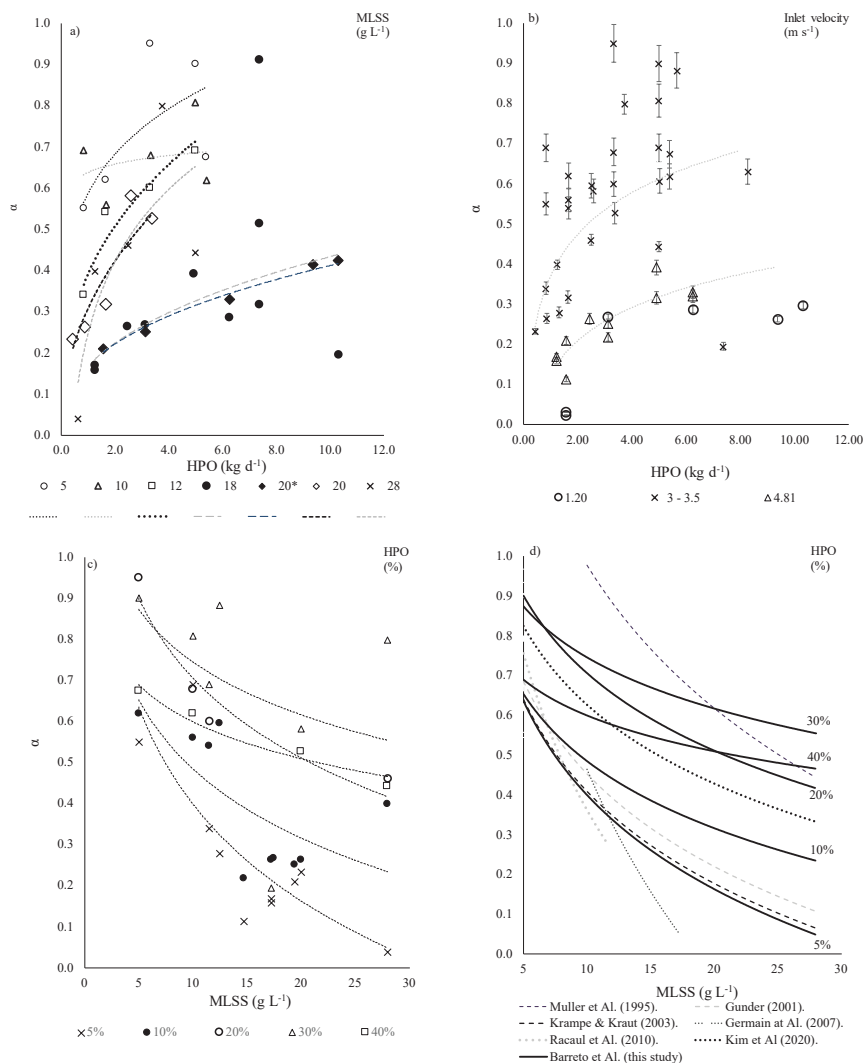


Figure 54 Alpha factor for different a) MLSS concentration b) Inlet velocity c) HPO flowrate and d) alpha factors by other researchers.

At the cone's neck, the velocity is the highest it can be along the system; therefore, a higher shear is exerted on the fluid precisely at the neck where the mixed liquor comes into contact with the oxygen gas. The apparent viscosity is then momentarily modified favouring mass transfer due to the very vigorous mixing and enhanced contact. This advantageous condition is amplified by the combined effect of both the increased pressure, and the oxygen concentration gradient making the partial pressure of oxygen nearly ten times higher as compared to an air fed system. All previous factors converge at the cone's neck for ultimately improving the mass transfer from the gas to the liquid phase.

Sidestream superoxygenation showed similar alpha values when operating at the lowest HPO flowrates of 5 and 10% as compared to fine bubble aeration (Figure 54c, 54d). For such low HPO flowrates (5 and 10%), the alpha values followed a trend almost identical as previously reported by other authors on fine bubble aeration (*Guender, 2000; Krampe & Krauth, 2003*). However, at higher HPO flowrates the Speece cone exhibited higher alpha factors, even at the highest evaluated MLSS concentrations. Similar systems such as the Supersaturated dissolved oxygen (SDOX) system (*Kim et al., 2020; Kim et al., 2021*) operated a laboratory scale setup in a sidestream configuration for delivering supersaturated streams to a bioreactor, finding alpha factors similar to those in this research, confirming the advantages of sidestream supersaturation.

The rheogram presented in Figure 55a shows the relation between the applied shear rate and the corresponding measured apparent viscosity of the activated sludge exposed to the high shear condition inside the pilot scale Speece cone. Ranging from 3.64 to 245 mPa s (at 50 s^{-1}), and from 3.07 to 160 mPa s (at 100 s^{-1}) for 5 and 40 g L^{-1} respectively, the apparent viscosity was higher for the activated sludge with higher solids concentrations. For the highest MLSS of 40 g L^{-1} , the apparent viscosity was approximately 160 mPa s at a shear rate of 100 s^{-1} , similar to the shear rate in typical aerated tanks with conventional fine bubble aeration which ranges from approximately 40 to 220 s^{-1} at MLSS concentrations from 3 to 10 g L^{-1} (*Durán et al., 2016*). However, unlike conventional aeration methods, the mass transfer process in the Speece cone takes place inside the pressurized vessel, where the shear rate is much higher.

Figure 55b depicts the difference in magnitude of the apparent viscosity in the same activated sludge (MLSS 5 to 40 g L^{-1}) subject to shear stress typically observed in two scenarios: i) conventional aeration (top) and ii) sidestream superoxygenation (bottom); the top part in Figure 55b displays the laboratory measured viscosity observed for the AS exposed to the Speece cone high shear conditions. The measurements were carried out at (low) shear rates (from 0.1 to 100 s^{-1}) similar to those of aerated tanks and in all cases yielding viscosity with values higher than 1 ($\mu_{\text{app}} > 1$) even for additional tests (not shown) performed under a higher shear rate of 1000 s^{-1} which can occur in more vigorously mixed tanks. The bottom section in Figure 55b presents the calculated viscosity for the same sludge at the conditions that occur at the cone's neck, for the three inlet velocities evaluated in this research, namely 1.2, 3.4 and 4.8 m s^{-1} . These calculated viscosity series

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were obtained as described in the methods section, based on the work published by (Lopez et al., 2015; Rosenberger et al., 2002) who proposed a mathematical expression for calculating the apparent viscosity of activated sludge in a flowing pipeline, meaning the actual in-process apparent viscosity for the sidestream superoxygenation.

Depicted at the bottom part in Figure 55b, the calculated in-process viscosity showed differences along two orders of magnitude in the range of 10^{-3} to 10^{-1} mPa s. Such low viscosity resulted from the high shear the sludge is subject to inside the cone. This condition gives dynamic sidestream superoxygenation a viscosity 3 to 5 orders of magnitude lower than the viscosity in aerobic tanks under ideal conventional aeration conditions. Making use of this condition allows for the actual gas-liquid mass transfer process to take place under more favourable conditions, meaning a lower viscosity added to a higher concentration gradient in a pressure vessel unlocking mass transfer benefits that come with lower viscosity, which can be translated to energy savings.

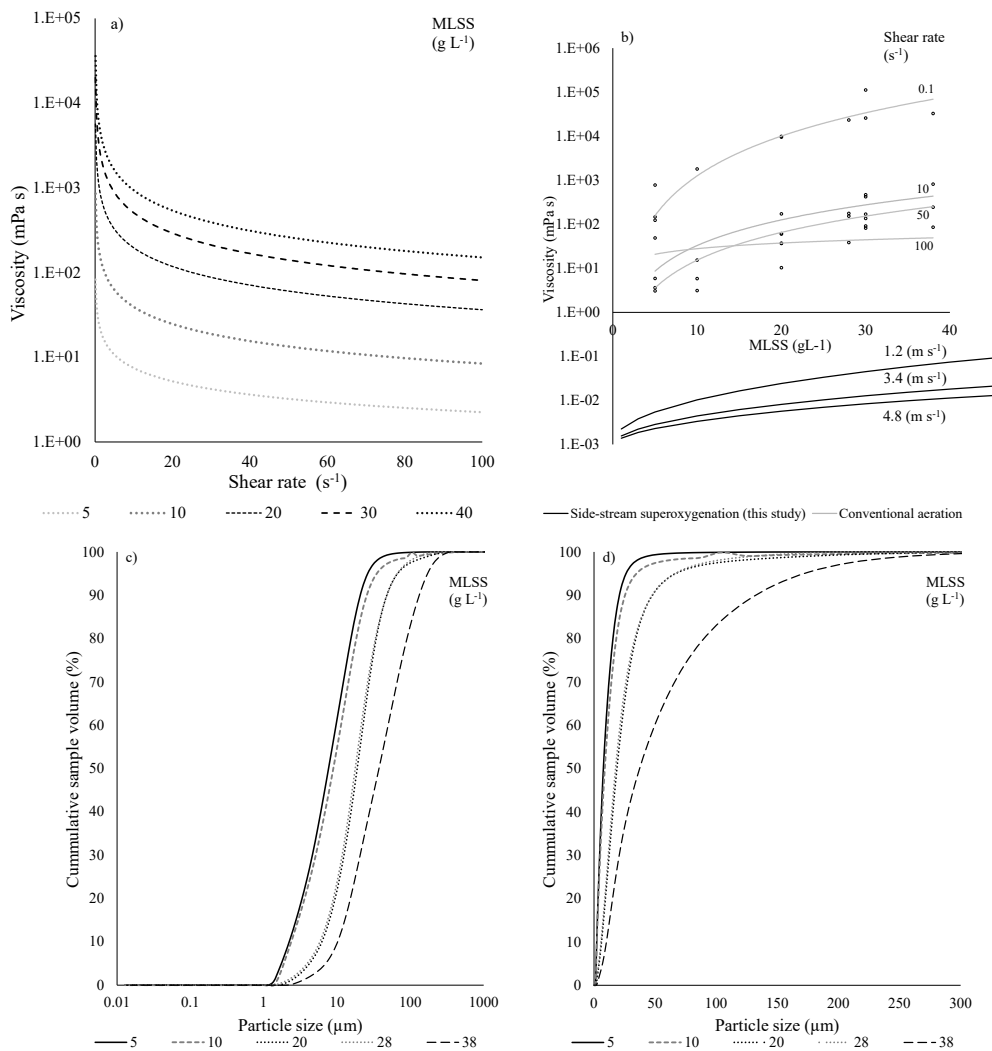


Figure 55 AS viscosity: a) AS rheogram, b) Top: Measured viscosity for shear typical of conventional aeration; bottom: Calculated viscosity for shear inside the cone. PSD for: increasing MLSS concentrations c) abscissa log scale, d) linear scale.

The Speece cone exhibits higher mass transfer efficiencies and alpha factors compared with conventional aeration. The role of viscosity is underestimated when discussing the performance of MBRs. Both the permeate production performance considering the filtration theory, as well as the aeration performance are strongly impacted by the viscosity. Viscosity has been reported to influence both key processes, as well as other processes related to the operation of MBRs (*Durán et al., 2016; Hong et al., 2018; Lopez et al., 2015; Lousada-Ferreira et al., 2010; Moreau et al., 2009b; Rodríguez et al., 2010; Rodríguez et al., 2012; Rosenberger et al., 2002; Z. Wu et al., 2007; Yang et al., 2016*). However, its mention is merely informative and so far, it has not played a relevant role in the wastewater treatment plant process design; particularly, in aeration design considering that aeration is responsible for the largest fraction of the energy expenses in a WWT plant there is a big potential for optimization. Viscosity is an objective and precisely measurable parameter that should be elevated at the same relevance as the SRT, MLSS and biological conversion rates; particularly, in regards of aeration and membrane filtration. In addition, energy efficiency and financial benefits can be obtained from a better knowledge of the relation between viscosity-MLSS- α and the power consumption of the equipment at the facility level. In-line viscosity measurements can be implemented in any installation by following a similar approach as described by (*Lopez et al., 2015*) who used a tubular membrane element for establishing the AS viscosity under controlled conditions. Similar arrangements can be made at full scale installations for more precise operation with submerged membranes for the determination of the actual in-process viscosity.

Figure 55c and 55d show the changes in PSD as the MLSS concentration was increased in the MBR. Initially, the PSD of the WAS from the WWTP at 5 g L^{-1} served as a baseline to assess the effect of high shear of the cone system on the sludge PSD. The mean particle size as well as the 80% cut off size increased with higher MLSS. Flocs seemed to aggregate instead of breaking apart, despite the periodic addition of WAS necessary to sustain the desired MLSS concentration, which might have affected the results in the measurements we report by shifting down the mean particle size towards the typical values found in WAS.

It is important to remark that the presence of particles with size below one micron or sub-micron particles (SMP) was very low as compared to other sludge types. Having lower SMP presents other benefits such as membrane fouling reduction at least the fraction associated with pore blocking mechanisms.

e. Energy usage compared to conventional aeration.

In order to obtain the most benefits out of a supersaturated system such as the Speece cone, the DO concentration at the cone's outlet should be maximized. Meaning that instead of targeting a low dissolved oxygen (DO) concentration of approximately 8 mg L^{-1} in the recirculation loop, a much lower by-passed volume can be supersaturated to DO

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concentrations up to 40 mg L^{-1} and then applied back to the bioreactor, improving the specific energy consumption. By diluting such supersaturated stream in a by-pass arrangement, the energy demand per cubic meter treated can be significantly reduced.

The SAE (kgO kW h^{-1}) was computed using the net oxygen mass transferred to the liquid divided by the total nominal power of the sludge pump used in the recirculation loop at a pilot scale, meaning there is room for energy savings that come with scaling up and process control-automation. The calculated SAE for both activated sludge and clean water are presented as a function of i) the HPO flowrate and ii) the MLSS concentrations in Figure 56a and 56b, respectively. The Speece cone SAE for the operational conditions, pump type and system size evaluated in this research exhibited lower values of approximately $0.1 \text{ kgO kW h}^{-1}$ (both in clean water and in sludge up to a MLSS concentration of 30 g L^{-1}) compared to conventional aeration such as fine bubble aeration or paddle aerators which can range from 0.6 to 1 kgO kW h^{-1} in clean water only.

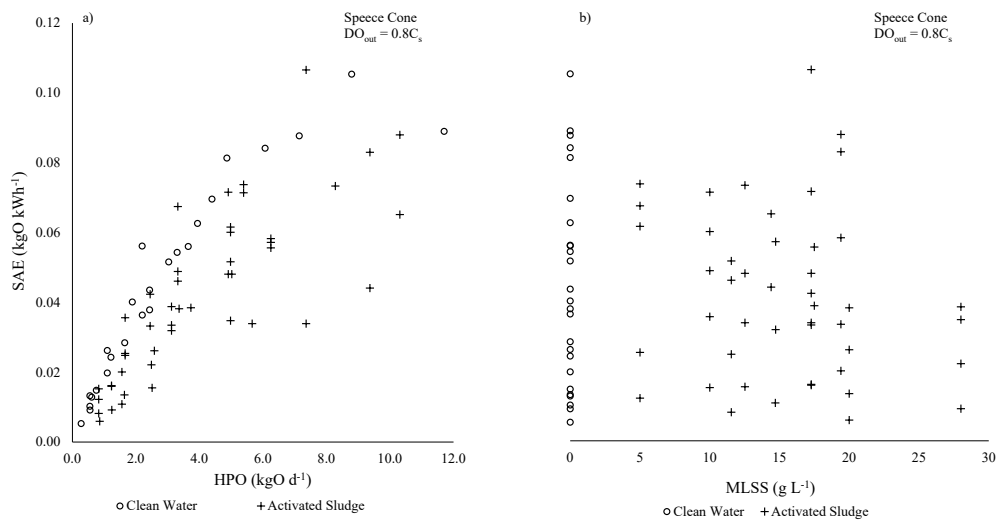


Figure 56 Sidestream superoxygenation specific aeration efficiency (SAE; kgO kW h^{-1}) for clean water and activated sludge for increasing a) HPO and b) MLSS.

Despite their low efficiency and their elevated power-maintenance demand, fine and coarse bubble aeration are the most widely applied methods for delivering oxygen to the AS process. Their SAE appears to be high, but this holds true for clean water only. The clean water SAE performance of bubble aeration diminishes after such values are adjusted considering the alpha factor. When considering the alpha factor, lower SAE values are obtained, increasing the demand not just for oxygen flux but also for the installed power in full scale MBRs for which alpha factors are even lower ($\alpha \leq 0.5$). When adjusted for local operation conditions including the effect of solids via alpha factor, the SAE strongly diminishes beyond the 15 g L^{-1} MLSS mark. The presented results are calculated based on the nominal power; therefore, the energy demand can be further reduced by implementing process control improvements such as a PID control loop for controlling the pump speed and the HPO flow simultaneously with a commanding DO signal as discussed in the next section.

*f. A proposed process configuration for the Speece cone application in full scale
wastewater treatment facilities*

Getting to know how versatile the Speece cone is in delivering oxygen can only lead towards applications where precision is valued. Not only for the wastewater treatment and sludge digestion fields, but also in other types of industrial biological processes which require dissolving oxygen or other gases for the production of target compounds or biomass or for process control.

An alternative configuration for wastewater treatment plant that includes a Speece cone for sidestream superoxygenation is presented in a process diagram in Figure 57c. The process diagram focuses on the treatment steps regarding the organic matter oxidation at the aerobic tank (AE-TK) and its corresponding process lines. Other treatment steps are supposed to remain the same according to their specific treatment configuration for biological nutrient removal.

In the proposed configuration, the Speece cone is operated using sieved influent instead of activated sludge for the saturation circuit, in this way improved mass transfer can be achieved since the effect of solids and the complex rheological behaviour of sludge are removed from the picture. Moreover, by using influent instead of sludge, the k_{La} effectively doubles as is shown in Figure 57a. Delivering supersaturated streams up to 300% saturation allows for a dilution ratio of 1:15 for the by-pass stream, having 2 mgO L⁻¹ for a target concentration at the AE-TK. High DO concentrations can be reached quickly depending mostly on the applied pressure and HPO flowrate; Figure 57b shows the supersaturation of AS with MLSS of 30 g L⁻¹ within minutes. Even higher supersaturation levels can be maintained for both clean water and sieved influent.

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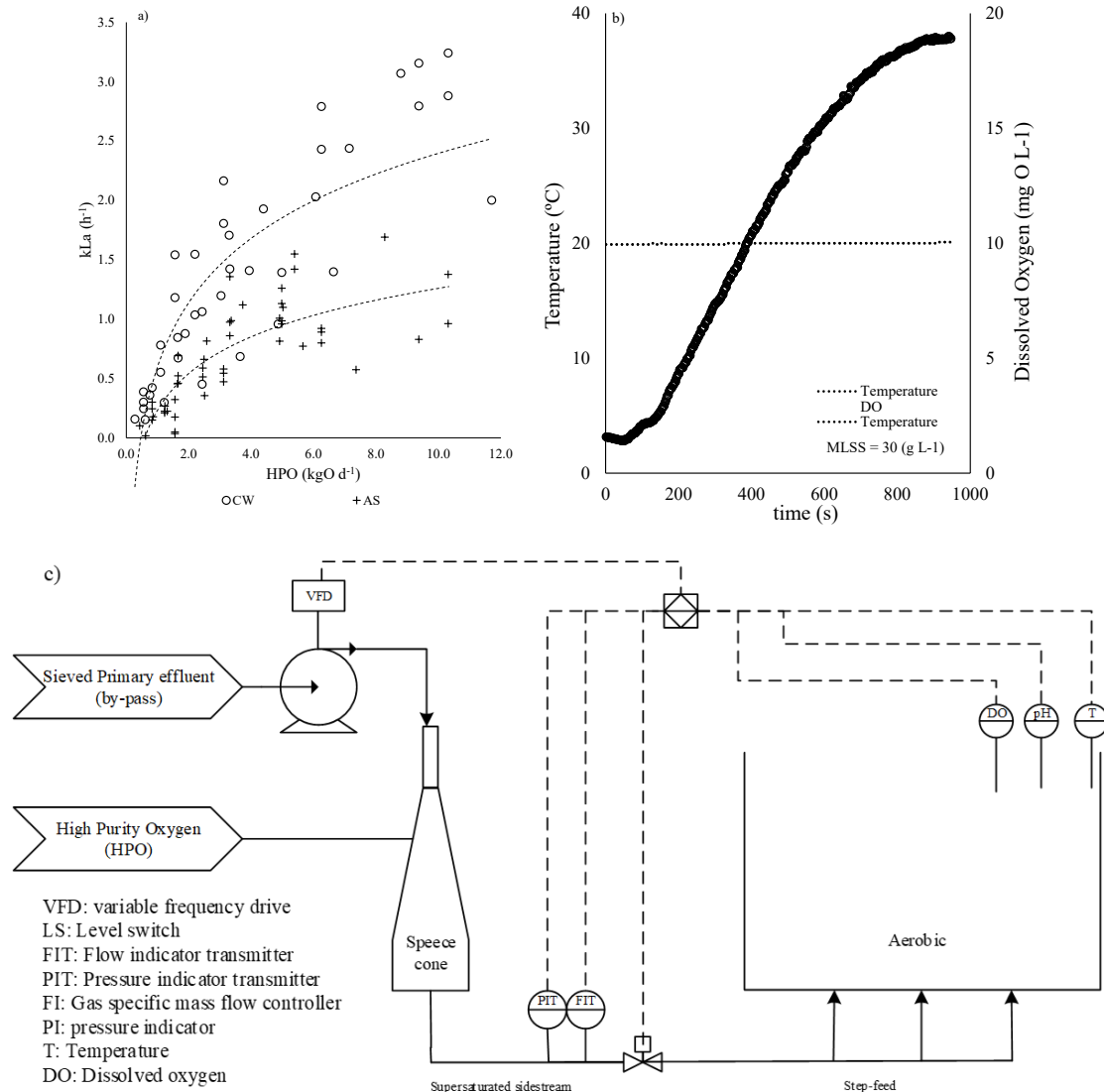


Figure 57 a) $k_L a$ comparison for clean water (CW) and Activated sludge (AS) b) time to supersaturate 1 m³ AS MLSS:30 g L⁻¹ c) proposed aerobic tank configuration using sidestream supersaturation.

4. Conclusions

The Speece cone performance was strongly affected by the inlet velocity and the HPO flowrate. On the contrary, the operational pressure did not have a major effect on the SOTE nor the alpha factor. The apparent efficiency played a major role in the gas-liquid mass transfer efficiency; having the process occurring inside the cone where conditions are ideal unlocks process benefits such as volume reduction and high load capacity under harsh process conditions. The advantages of supersaturation can be implemented in applications with high load and high biomass bioreactors, or in cases where space saving or volume reduction is needed.

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5

MEMBRANE FILTRATION PERFORMANCE IN ACTIVATED SLUDGE AT HIGH MIXED LIQUOR SUSPENDED SOLIDS CONCENTRATIONS: AN ALTERNATIVE PROCESS CONFIGURATION FOR FOULING MITIGATION

This Chapter is based on Barreto, C. M., García, H. A., Hooijmans, C. M., Herrera, A., & Brdjanovic, D. Membrane filtration performance in activated sludge at high mixed liquor suspended solids concentrations: An alternative process configuration for fouling mitigation. Submitted to Journal of Environmental Management, 2023.

Abstract

Three different types of submerged membrane were tested in a pilot scale Membrane bioreactor (MBR) coupled with a sidestream superoxygenation system known as a Speece cone. The MBR was operated at different mixed liquor suspended solids (MLSS) concentrations of 20 and 38 g L⁻¹, considered to be higher solids concentrations as compared to conventional MBR which are usually operated at approximately 10 g L⁻¹. The short-term membrane filtration testing was carried out following a modified version of the flux step method. Results showed that despite the adverse filtration conditions under such high MLSS concentrations, it is possible to perform short-term filtrations test, however, the permeate flow could not be sustained over longer periods of time as it is required for full scale installations. In order to cope with the excessive fouling at such high MLSS concentrations, a novel activated sludge pre-conditioning process was proposed as a centrifugation stage prior to the membrane filtration tank. Preliminary laboratory scale tests showed that by the application of such sludge pre-conditioning treatment the Total suspended solids (TSS; g L⁻¹) concentration and the viscosity can be reduced by one order of magnitude.

1. Introduction

Despite still being approximately 50% more expensive than conventional activated sludge systems, the implementation of membrane technology using a membrane bioreactor (MBR) is nowadays more attractive than in previous decades, mostly due to the significant price reduction which has been observed in the membrane market in recent years, driven by the competition among membrane manufacturers that can bring even lower implementation costs in the future *Krzeminski et al., (2017)*. New membranes manufacturers have emerged during the last decades, making available in the market a myriad of membranes of different materials, shapes, and operational features which allow for innovation in the process configuration for specific applications. The footprint reduction and portability of a wastewater treatment system are among the most relevant features to look when improving the design of such systems; particularly, when looking at alternatives for the provision of sanitation where the footprint of the system is of the utmost importance such in humanitarian response situations. The footprint reduction of wastewater treatment systems can be achieved for instance by operating an MBR at high mixed liquor suspended solids (MLSS) concentrations (higher than the conventional 10 g L⁻¹ values); a similar approach using the high MLSS MBR concept was reported by *Kim et al., (2020)*; *Kim et al., (2021)* as the high-loaded MBR system (HL-MBR). A bioreactor operated at a high MLSS concentration of for instance 30 g L⁻¹ would require up to one tenth the volume as that of a conventional activated sludge system (CAS) operated at a MLSS of 3 g L⁻¹ *Barreto et al., (2017)*. However, typical MBR operation is usually limited to a range between 10 to 15 g L⁻¹ *Lousada-Ferreira et al., (2010)*; *Lousada-Ferreira et al., (2015)*; *Barreto et al., (2017)*, *Barreto et al., (2018)*, *Kim et al., (2020)*; *Kim et al., (2021)* mainly due to i) the very limited oxygen transfer of conventional aeration methods such as fine bubble diffusion at such high MLSS concentrations, and ii) the exacerbated membrane fouling caused by the high-solids concentration.

The oxygen transfer efficiency (OTE) of conventional aeration methods (fine bubble) in clean water, i.e., without the presence of suspended solids, meaning under ideal conditions, is approximately 3% per meter depth *Henze et al., (2008)*; *Judd, (2010)*. On the other hand, under process conditions, meaning using activated sludge instead of clean water, the effects of the suspended solids are taken into account by multiplying the clean water OTE by a correction factor, the alpha factor. Alpha (α) represents the ratio between the mass transfer coefficient (k_{La}) of process water and clean water. For instance, for an MBR operating at MLSS concentration of 10 g L⁻¹, $\alpha = 0.5$, meaning that the OTE will be reduced by 50%, to a value as low as 1.5% per meter dept, leaving the remaining 98.5% of all the air volume that is introduced into the aerobic bioreactor (21% oxygen only) pass through the system without being dissolved into the solution. In order to cope with this, wastewater treatment designers have made the aeration basins as deep as possible in an attempt to give fine bubbles more distance (time) to deliver their oxygen content into the

mixed liquor; however, deeper tanks are way more expensive to build and demand more installed power in the blowers due to the additional required discharge pressures. This is energy that is being wasted, making the aeration process the second largest operational cost in wastewater treatment plants; there is a lot of room and interest in improving the aeration specific energy demand using more efficient aeration methods.

In contrast, superoxygenation methods can easily overcome the mass transfer limitations occurring at high biomass concentrations reaching OTE values higher than 90% even in activated sludge operating at higher than usual solids concentration. For instance, the supersaturated oxygen aeration system (SDOX) was reported to reach alpha factors between 0.4 and 0.5 at MLSS concentrations of approximately 20 g L^{-1} , as compared to negligible alpha factors reported for conventional fine bubble diffusers at similar MLSS concentrations *Kim et al., (2021)*. Another superoxygenation system is the Speece cone which can be used as an alternative aeration device in intensive bioreactors for portable and compact sanitation applications as reported by *Barreto et al. (2017) and Barreto et al. (2018)*. The authors reported alpha factors of approximately 0.6, 0.65 and 0.7 at MLSS concentrations of 38, 20 and 10 g L^{-1} respectively. At lower MLSS of 10 g L^{-1} , standard operational MLSS setting for MBR systems, alpha factors of approximately 0.8 were observed in contrast with conventional methods which can barely reach an alpha factor of 0.5 at such MLSS concentration. Such results indicated a high performance of the superoxygenation systems under harsh operational conditions, presenting superoxygenation methods as a feasible alternative for high rate and/or portable MBR systems; superoxygenation systems can offer an alternative aeration method for conventional systems and other precision dosing applications.

However, operational problems operating MBR systems at such high solids concentration conditions can be observed due to membrane filtration fouling issues. Membrane fouling can be an important limitation when operating at high MLSS concentrations. Several efforts have been made in coping with membrane fouling including exploring both alternative methods for membrane pre-coating *Deowan et al., (2016)*, the application of mechanical scouring methods *Chen et al., (2016)*, as well as innovative operational conditions and cleaning methods *Delrue et al., (2011)*, *Zsirai et al., (2012)*. Several studies have pointed at MLSS concentrations as one of the main operational parameters influencing membrane fouling, for instance, *Zhang et al., (2006)* described in detail the main parameters in terms of membrane properties, nature of the feed wastewater, environmental, operational and design characteristics, and presented a fouling roadmap and fouling mechanisms map in which the biomass plays a central role in the membrane fouling. However, little effort has been allocated on eliminating the actual cause of excessive fouling, which is the high solids concentration in the biological systems. The low OTE of fine bubble diffusers observed in HL-MBR systems can be eventually overcome by using superoxygenation systems for supplying dissolved oxygen. However, the membrane fouling still could limit the development and implementation of HL-MBR

systems aiming at reducing the footprint of wastewater treatment systems. Thus, there is a need to advance the membrane filtration knowledge in the context of MBR systems operated at higher than usual MLSS concentration including: i) the determination of the effects of high solids concentrations on the short-term membrane filtrations performance; ii) the evaluation of the performance of different membrane types; and iii) the establishment of the optimal operational conditions of the high MLSS MBR systems considering the membrane filtration performance of the system, among others.

This article addressed those needs directly by evaluating the membrane filtration performance of a pilot scale MBR coupled with a sidestream superoxygenation system, the Speece cone, operated under high MLSS conditions. This paper aimed at determining: (i) the short-term membrane filtration performance of three different types of submerged membranes in activated sludge along MLSS concentrations ranging from 20 to approximately 40 g L⁻¹; and (ii) the sludge rheological properties of activated sludge exposed to high shear effects that occur in the Speece cone superoxygenation method. Parameters such as membrane permeability, sludge resistance to filtration, fouling rates, dynamic viscosity, and particle size distribution (PSD) were assessed. In addition, a sludge pre-conditioning alternative for membrane fouling mitigation was proposed and evaluated at the laboratory-scale by centrifuging activated sludge samples to mimic the effects of a hydrocyclone introduced in between the aerobic bioreactor and the membrane tanks in a full-scale MBR installation.

2. Materials and methods

2.1. Experimental design

Membrane filtration tests following a modified flux step method were performed using three different submerged membranes namely polymeric flat sheet (FS), polymeric Tubular (T) and ceramic flat sheet (K) in activated sludge concentrations of 20 and 38 g L⁻¹ and in clean water (CW) as a reference; each filtration test was carried out at least three times for all the evaluated MLSS concentrations. The raw data series were processed in triplicates to yield a mean value of membrane permeability (Perm; L m⁻² h⁻¹) and total fouling rate (F_T; m⁻¹ s⁻¹) for each one of the flux step tests. Subsequently, a strategy for mitigating the negative effects of the high solids concentration on the membrane filtration performance on an MBR-Speece cone system operated at high MLSS concentration was proposed and simulated. The strategy consisted of adding a preliminary solid-liquid separation step (sludge pre-conditioning) before the sludge reaches the membrane filtration system of the MBR-Speece cone system by means of a hydrocyclone. The strategy was evaluated by simulating the effects of the hydrocyclone via centrifugal forces. That is, different sludge samples at varied high MLSS concentrations (15, 20, 30, 38g L⁻¹) were centrifuged to mimic the effects of a hydrocyclone on sludge pre-conditioning in an MBR system. The rheological properties of the activated sludge exposed to high shear typical of the Speece cone superoxygenation method were assessed before and after

centrifugation and their potential effects on the membrane filtration process were discussed. Parameters including the dynamic viscosity, and particle size distribution (PSD) were measured.

2.2. Experimental setup description

The membrane filtration evaluations were carried out in a bench scale setup comprised of two identical polypropylene (PP) cylindrical tanks (d: 0.45m; h: 0.9 m) of 100 L capacity each; the Ultrafiltration (UF) membrane modules were submerged in the cylindrical tanks. One tank was filled with clean water (control), while the other tank was filled with activated sludge at the evaluated MLSS concentrations. A custom-made membrane module, able to fit three different membrane types at the same time was built for these tests. The membrane module provided air scouring and the geometry necessary to keep both spacing and velocity within the typical ranges observed in full scale applications for each membrane type.

Permeate was extracted by means of a peristaltic pump (Cole-Palmer, USA) with an automatic variable speed control loop set for fixed flux operation. A high precision flow meter (OMEGA, UK) and a pressure transmitter (Endress+Hauser, Switzerland) were installed in the permeate line for continuous data logging. A control panel with a programmable logic controller (PLC, SIEMENS, Germany) and a custom-made software were designed and built for this research. The program controlled the filtration cycles, steps, backwash and signal processing-logging for three membranes operating at the same time and under the same conditions in a parallel configuration. Air for membrane scouring was provided with a diaphragm air blower (SECOH, UK). A schematic depicting the experimental setup is presented in Figure 58 and the system is presented in Figure 59.

During the filtration tests, three different type of bench scale UF membrane elements were evaluated as follows: Flat Sheet (FS) (Kubota, Japan), Tubular (T) (Memos, Germany) and Ceramic (K) (Mycrodin, Germany); each element with a filtration area of 0.1 m² was connected to an independent set of tubing and sensors.

Membrane filtration performance in activated sludge at high mixed liquor suspended solids concentrations: An alternative process configuration for fouling mitigation

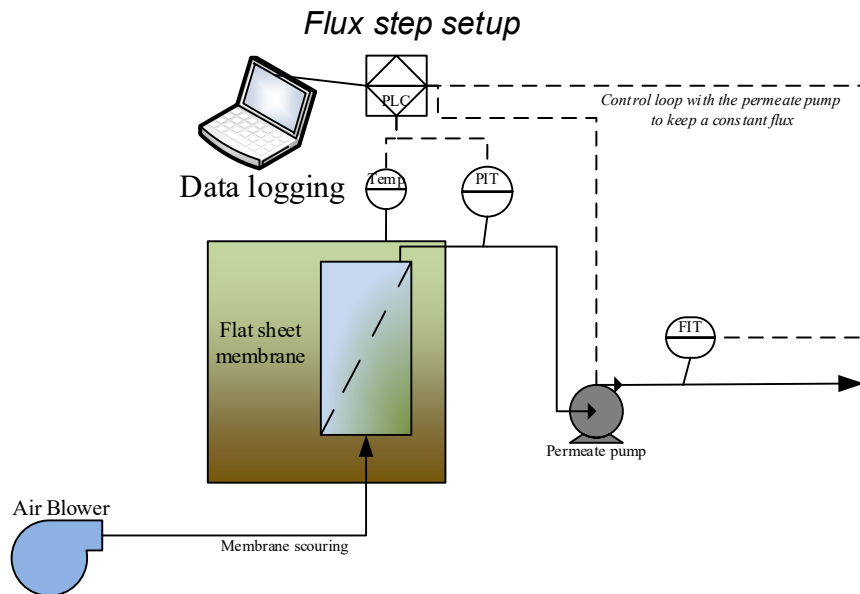


Figure 58 Process flow diagram for the modified flux step method experimental setup.

Figure 59 displays pictures taken around the experimental setup; the top left image shows the control box to the top right corner and below one of the peristaltic pumps can be observed, on the left side there are two laptops for data logging and equipment controlling. The top right picture displays the back view of the system with the two cylindrical tanks for the clean water and activated sludge aligned to the left-hand side and some field probes for oxygen, pH and conductivity recording at the centre of the image. The bottom left image shows a close-up of the tank containing sludge during a filtration test. The bottom right image shows the two tanks and the membrane modules inside the open tank.



Figure 59 Experimental setup for testing the submerged membranes in high MLSS activated sludge.

2.3. Analytical procedures

The total suspended solids (TSS) and volatile suspended solids (VSS) were determined following the standard methods for the examination of water and wastewater (AWWA, 2014). The activated sludge particle size distribution and viscosity were measured with a laser particle counter (Microtrac, Japan; 10^{-3} to 10^3 μm) and a rheometer (Anton Paar, Switzerland; 10^{-2} to 10^3 s^{-1}) respectively. The laboratory analyses were performed at the IHE-Delft's laboratory and at the Civil Engineering Department's Water Laboratory of the Delft University of technology (TUD) in Delft, The Netherlands.

2.4. Experimental procedure

The research activities took place at the Delft Blue Innovations Research Hall in the Harnaschpolder wastewater treatment plant (www.delftblueinnovations.nl). Activated sludge from the Harnaschpolder wastewater treatment plant (Delft, The Netherlands) was used for the experiments. The sludge retention time (SRT) was kept at 20 days by the continuous addition of fresh sludge from the treatment plant to a pilot scale membrane bioreactor (MBR) with a total volume of one cubic meter. In order to increase the MLSS concentration while having a relatively low SRT, it was necessary to increase the concentration of the mixed liquor using the pilot MBR's membrane module for extracting water from the activated sludge while adding fresh activated sludge until the desired MLSS concentration. Once the solids concentration reached the desired level, the sludge was transferred by gravity to the 100 Liters cylindrical filtration tank, and the filtration evaluations were performed immediately.

The filtration evaluations were based on the flux step method initially proposed by *Le-Clech et al. (2006)*, and then modified or improved by *van der Marel et al. (2009)*. In this research, an additional modification to the testing protocol was added in order to cope with the high solids concentration. A backwash step was introduced between flux steps along the filtration testing sequence in order to remove the fouling background. The backwash was performed in between flux steps at the same flux as the previous step before the cycle would initiate the next flux step.

The filtration cycle started at a flux of $10 \text{ L m}^2 \text{ h}^{-1}$ during a five-minute period (step duration), followed by a backwash step of 3 minutes with 30 seconds pause in between the two. Once the filtration and backwash time expired, the flux was incremented by $10 \text{ L m}^2 \text{ h}^{-1}$ (step height) before moving to the next value or flux step, up until a maximum flux of $80 \text{ L m}^2 \text{ h}^{-1}$ was reached. The measurements also took place in the descending part of the sequence by decreasing the flux in the same fashion from $80 \text{ L m}^2 \text{ h}^{-1}$ until the initial flux of $10 \text{ L m}^2 \text{ h}^{-1}$ was reached again to complete one full test. The results obtained from the activated sludge filtration tests were compared to the baseline results obtained with the exact same protocol in clean water filtration tests. During the filtration tests, the pressure and permeate flow were recorded every second for the determination of the

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membrane permeability (Perm; $L m^{-2} h^{-1} bar^{-1}$) and total fouling rate (F_T ; $m^{-1} s^{-1}$). After the filtration tests were finished, activated sludge samples were taken to the laboratory immediately and processed within one hour for determining viscosity, PSD, TSS and VSS.

In order to simulate the conditions in a hydrocyclone for solid-liquid separation, the activated sludge samples were subject to centrifugation treatment at 10,000 rotations per minute (rpm) equivalent to 12,000 g-force over a period of three minutes. After this, the centrate (supernatant) was extracted from the top part of the test tubes for further TSS, VSS, PSD and viscosity analysis. The centrifugation tests were carried out using a laboratory centrifuge (Thermo Fisher Scientific, USA) at the IHE Delft's water Laboratory.

2.5.Data analysis and parameter estimation

The membrane permeability (Perm; $L m^{-2} h^{-1} bar^{-1}$) was calculated using Equation 59 based on the transmembrane pressure (TMP; bar) and permeate flow (Q; $L h^{-1}$) measured during the filtration tests. The membrane area (A; m^2) was 0.1 m^2 for all membranes.

$$Perm = \frac{J}{TMP} = \frac{Q}{A \cdot TMP} \quad (59)$$

The total fouling rate (F_T ; $m^{-1} s^{-1}$) was calculated using Equation 60, where P_{end} and $P_{initial}$ (P; mbar) correspond to the membrane pressure registered at the beginning and end of a flux step; η is the permeate dynamic viscosity (η ; Pa s) and J_H the applied flux (J ; $L m^{-2} h^{-1}$) at each flux step.

$$F_T = \left(\frac{dR_{total}}{dt} \right) = \frac{P_{end} - P_{initial}}{\eta J_H} \frac{1}{dt} \quad (60)$$

3. Results and discussion

3.1. Modified flux step method (output overview)

The data in Figure 60 and Figure 61 present the output plots from our modified flux step method (rows 1, 2 and 3) and the filtration tests pressure profiles (row 4). All plots in Figure 60 correspond to an MLSS concentration of 20 g L^{-1} , while the plots in Figure 61 correspond to an MLSS of 38 g L^{-1} . The filtration tests were performed on three different types of submerged membranes, namely polymeric Flat sheet (FS; left hand-side column), Polymeric Tubular (T; central column), and flat panel ceramic (K, right hand-side column). The target flux or set point (SP) for the permeate flow at each one of the flux steps is depicted in the background on each of the first and second row plots as a continuous grey line forming a stair-like shape. The increasing series of flux steps starts with $10 \text{ L m}^2 \text{ h}^{-1}$, goes up to $80 \text{ L m}^2 \text{ h}^{-1}$ and descends back to $10 \text{ L m}^2 \text{ h}^{-1}$ again; reaching towards the setpoint series goes along the actual process flux value (PV_lmh) depicted as a continuous black line following the SP on each flux step. The membrane pressure (P_mbar) is depicted with black circular markers at the bottom of each figure in the first and second row, its value can be read at the right hand-side axis. A zoom out to these graphs is presented in the third row, in which the difference in the amplitude of pressure between the three different membranes can be appreciated better. The total fouling rate (F_Total) is presented on the top part with grey circular markers forming data clusters above each one of the flow steps. The fourth row in each Figure 60 and Figure 61 depicts the pressure profiles for the same tests and conditions. The pressure profile plot shows two data groups, corresponding to i) clean water or baseline in grey and ii) activated sludge at the given MLSS concentration in black.

A first look into Figure 60 shows that for MLSS equal to 20 g L^{-1} , the FS membrane required lower pressure to produce as much permeate as the other two membranes, staying below 200 mbar for all flux steps. The tubular membrane (T) was the one with the highest-pressure demand as the pressure profile displays in the fourth row, reaching approximately 700 mbar for flux 70 and $80 \text{ L m}^2 \text{ h}^{-1}$. The permeate flows, particularly at fluxes of 10 and $20 \text{ L m}^2 \text{ h}^{-1}$ were more unstable for all three membranes compared to the flows at higher fluxes; this is possibly due to the fact that every test begins with a clean membrane, meaning there is no cake layer by the time the filtration process starts when it is suddenly set to $10 \text{ L m}^2 \text{ h}^{-1}$. However, these are short term filtration tests; additional extensive testing using both short and long-term tests, in addition to complete membrane filtration characterization using other assessing methods is still to be done to better understand such trends on the membrane flows.

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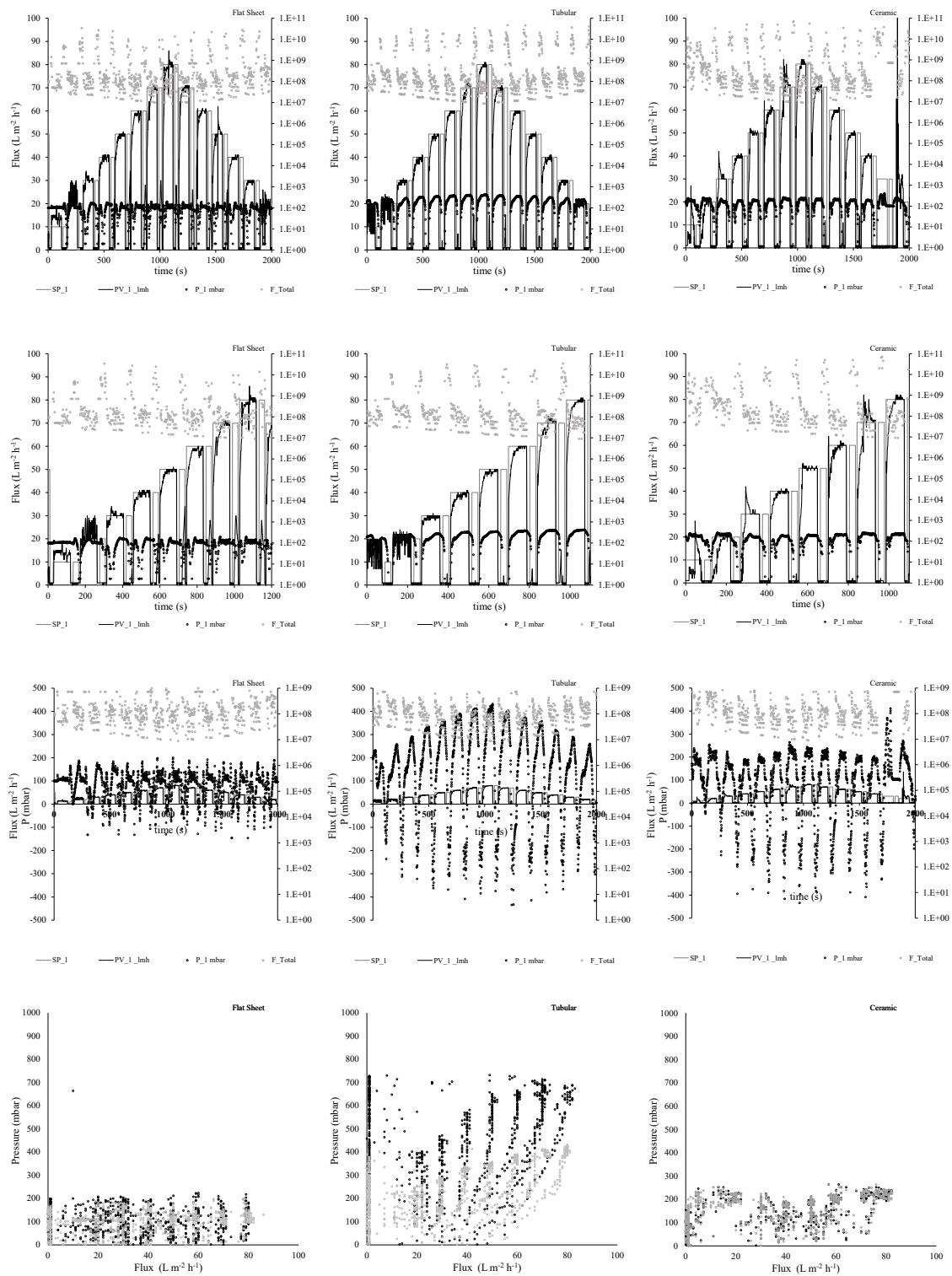


Figure 60 Membrane filtration characterization at 20 g L⁻¹. Column 1: Flat sheet, column 2: Tubular, column 3: Ceramic

Similar studies have been reported on the performance of the membrane filtration at high solids concentration exhibiting similar results *Gil et al., (2011)*; *Lousada-Ferreira et al., (2014)*. However, such studies were carried out in crossflow membrane configuration rather than submerged. Membranes arranged in crossflow filtration configuration demand more energy due to the higher crossflow velocity that is required for membrane scouring. This additional energy cost is a technological driver for implementing submerged membranes instead of crossflow in high biomass bioreactors.

Both the FS and K membranes presented pressure profiles very similar to their baselines or to the tests carried out in clean water. The negative effects of the solids on the pressure demanded to achieve each flux step was not so much noticeable in such short-term evaluations and MLSS concentrations; similar results as in clean water were obtained. On the contrary, membrane T displays a significant difference when compared to its baseline. This can be observed in the fourth-row plots in Figure 60 where the clean water pressure is depicted in grey dots. This indicates that the Tubular membrane T has the highest initial resistance among the three or in other words the membrane-only related resistance. In the same way, the FS membrane has the lowest initial resistance, it is important to highlight the very little variation of pressure demanded even at the highest flux steps as compared to the other two membranes, this can be observed at the fourth-row plots in Figures 60 and Figure 61. This makes the FS membrane type a better candidate for a membrane with clearly lower energy demand.

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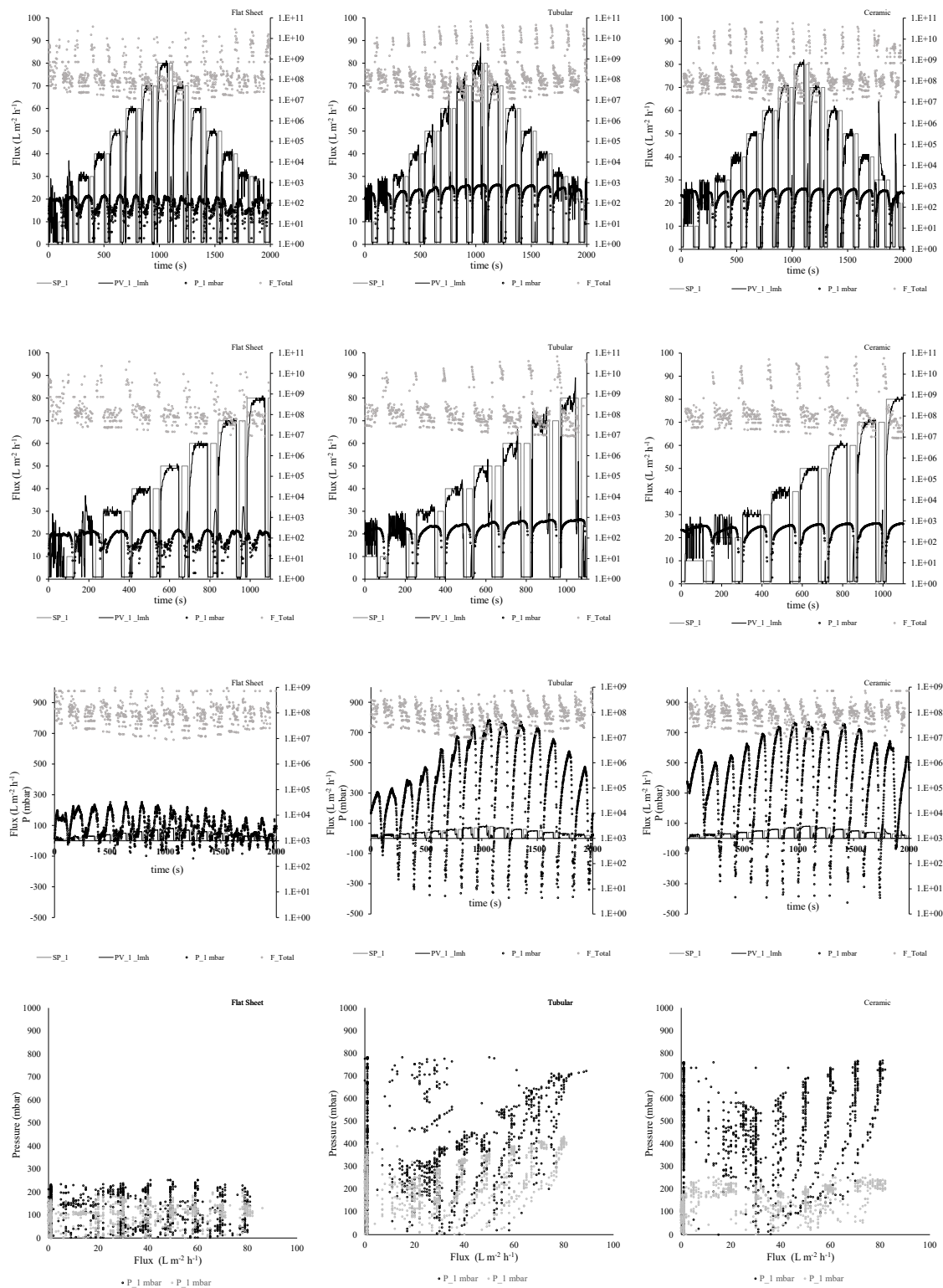


Figure 61 Membrane filtration characterization at 38 g L⁻¹. Column 1: Flat sheet, column 2: Tubular, column 3: Ceramic

The results when evaluating the membranes at an MLSS concentration of 38 g L^{-1} are shown in Figure 61. The FS membrane remained demanding the lowest pressure from all the three evaluated membranes, staying always below 250 mbar for all the evaluated flux steps. This is remarkable, considering the very hectic filtration conditions for a submerged membrane at such MLSS concentration. On the other hand, both the other membranes T and K required more pressure for producing the same flux as the FS membrane. Nevertheless, it is important to remark that membrane T started showing signs of reduced permeate flow at flux step $30 \text{ L m}^2 \text{ h}^{-1}$, and it failed to reach the flux setpoint at flux 50, 60 and $80 \text{ L m}^2 \text{ h}^{-1}$ for most of the step duration as displayed in the centre plot at the second row in Figure 61 (MLSS: 38 g L^{-1}) as compared to the same plot in Figure 60 (MLSS: 20 g L^{-1}) for which the flux was reached at the lower MLSS concentration

3.2. Permeability and Fouling rate.

Figure 62 presents the membrane permeability obtained during the same modified flux step method evaluations described in the previous section. Results are arranged per membrane type in columns, having the first-row displaying results for clean water (CW), and the second and third row for activated sludge at MLSS 20 g L^{-1} and MLSS 38 g L^{-1} respectively.

By looking at the first row in Figure 62, which corresponds to the membrane permeability in clean water, the difference in performance between the different membrane types is evident. The flat sheet polymeric membrane exhibited the highest permeability values of 1117.5 and $2351.1 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ at fluxes of 30 and $40 \text{ L m}^{-2} \text{ h}^{-1}$ respectively, whereas the remaining tubular polymeric (T) and flat sheet ceramic (K) membranes exhibited lower permeability values for identical operational conditions. The tubular membrane exhibited permeability values in clean water of 215 and $381 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$, at fluxes of 30 and $40 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ respectively; while the ceramic membrane (K) presented 158 and $389 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ respectively. That is, the permeability obtained without the presence of suspended solids and serves as a baseline. A reduced permeability was obtained when evaluating the membranes in activated sludge due to the presence of suspended solids. As expected, the decreased in the permeability values worsened with increasing MLSS concentrations; however, it was possible to obtain steady permeate production at most of the flux steps with a few exceptions at the very high end of the MLSS concentration and flux ranges being evaluated. This means that the filtration process using submerged membranes in such high solids conditions is an alternative and that process improvements can remove operational barriers for achieving a more compact membrane bioreactor.

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All three membranes display their own signature pattern in which a permeability peak was observed followed by a substantial reduction in the flow; such reduction in the permeability was observed as the MLSS concentration increased; thus, the filtration process was negatively affected due to the added resistance and viscosity of the sludge at a higher solids concentration.

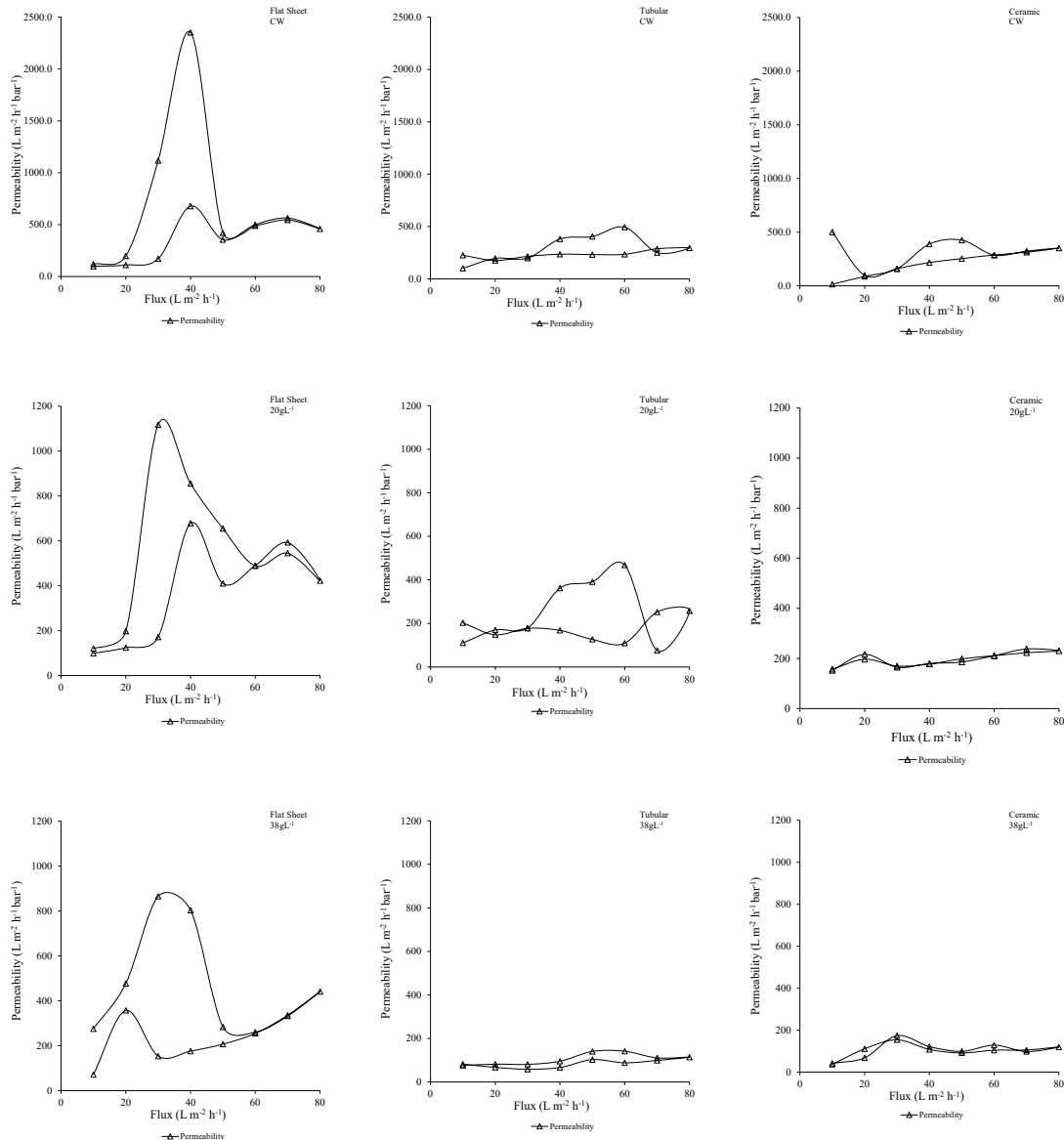


Figure 62 Pressure profiles, Permeability for three submerged membranes in clean water (CW) and activated sludge with MLSS 20 and 38 gL⁻¹.

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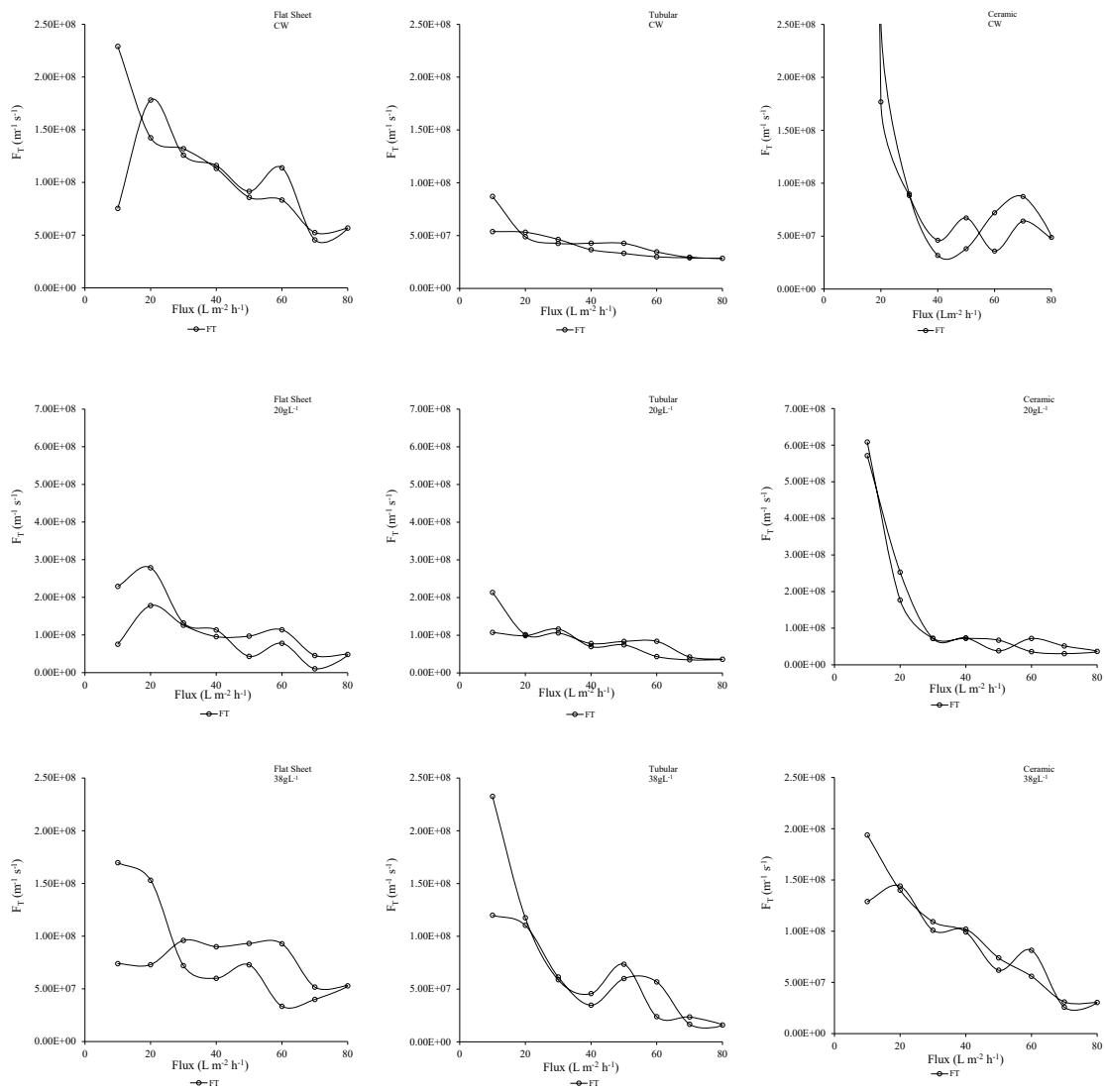


Figure 63 Total fouling rate for three submerged membranes in clean water (CW) and activated sludge with MLSS 20 and 38 gL⁻¹.

For the total fouling rate (F_T), as it was the case with the membrane permeability, each membrane type displayed its own and distinctive signature pattern which changed with the MLSS concentrations. Important to note in the first-row plots in Figure 63, the clean water evaluations showed the membrane resistance only. It is important to note that the total fouling rate (F_T) showed a decreasing trend with increasing flux values, which is unusual. This could be attributed to the positive effect of forming a sludge cake which contributed to improving the filtration performance by pre-coating the membrane with a layer that retains particles which otherwise would block the membrane. In addition, the introduction of backwash in between flux steps allows the removal of pore blocking particles while keeping the benefits of a cake layer. From the first-row plots, the T membrane exhibited the lowest intrinsic resistance; this is the resistance associated to the

membrane only operating in clean water. The resistance results for all the evaluated membranes stayed within one order of magnitude from the initial baseline of the corresponding clean water test meaning that even at such high MLSS concentrations the filtration process is still technically feasible.

3.3. *Strategy for mitigating the negative effects of high solids concentration on the operation of a high MLSS MBR*

The original driver for developing this research was to develop a sanitation/wastewater treatment technology able to cope with large amount of organic biodegradable waste demanding the smallest possible footprint. The development of such compact system with improved filtration performance would promote the implementation of portable/movable wastewater treatment systems for on-site sanitation provision including the application of such systems in emergency scenarios, refugee camps, and open-air events among others.

Even though previous results (based on short-term testing) demonstrated that is possible to operate a system at such high MLSS concentrations, it is a well-known fact that operating a MBR at low solids concentrations is more convenient from the membrane filtration process perspective. That is, in the long term the membrane performance and durability can be significantly better and more financially attractive (when intended for industrial use) to operate at lower solids concentrations as compared to the ones evaluated and mentioned above in this study. This is the main driver for introducing process and technology changes that contribute to achieving such goal of improving the filtration conditions for submerged membrane operation in cases where performance is prioritized over durability as in the case of emergency sanitation.

Therefore, a strategy for mitigating the negative effects of the high-solids concentration is proposed and presented in Figure 64. The scheme in Figure 64 shows an alternative process configuration for a compact wastewater treatment system (high-MLSS MBR) with biological nutrient removal via anaerobic, anoxic and aerobic stages. The aeration is provided with superoxygenation device, the Speece cone in this case, which allows for high efficiency oxygen transfer in such MLSS conditions. The Speece cone can be operated using screened influent instead of activated sludge (as depicted on Figure 64) in order to improve its performance and compensate for the negative effects that solids have on the oxygen transfer as presented in a previous study by *Barreto et al., (2017); Barreto et al., (2018)* in which the Speece cone was used for superoxygenation of high solids activated sludge with significant improvement when feeding the cone clean water or water with a lower solids concentration.

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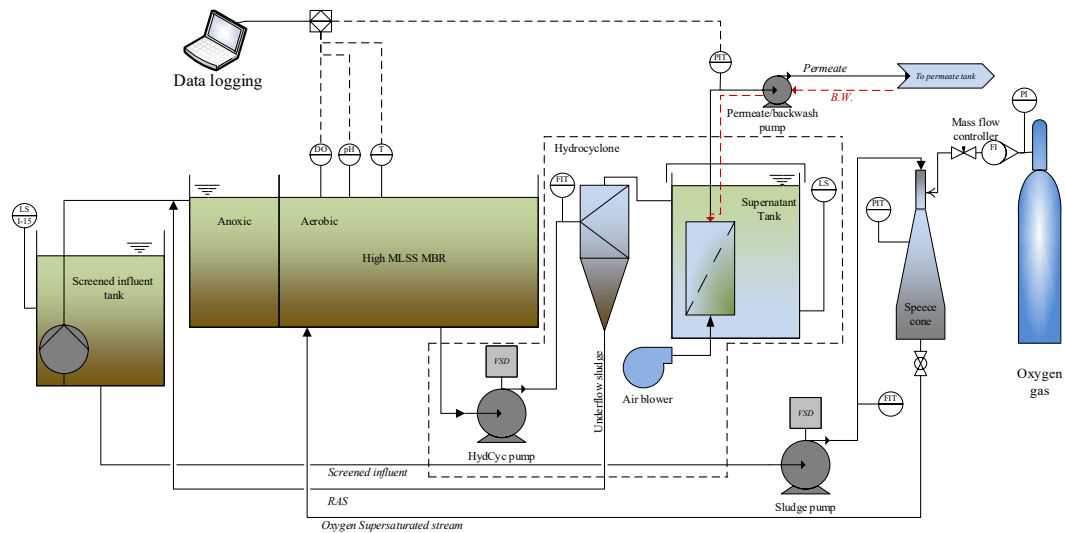


Figure 64 proposed WWTP configuration using sidestream supersaturation.

The hydrocyclone placed in between the biological reactor and the filtration tanks can separate the sludge into an even more concentrated underflow to be returned to the biological process, and a clarified overflow or centrate with significantly lower solids concentrations with potential benefits in terms of improving the membrane filtration performance. To assess the effects of a hydrocyclone on treating the sludge mixture coming from the high MLSS MBR reactor a series of centrifugation experiments were carried out. That is, the effects of the hydrocyclone were mimicked using a centrifugation process. The centrate resulting from the centrifugation tests is an analogue that represents the stream leaving the hydrocyclone from the top part, which is the lower solids concentration fraction or pre-conditioned sludge.

Figure 65 shows the processed high MLSS sludge samples subject to a pre-conditioning treatment in a centrifuge running at 10,000 r.p.m. equivalent to over 12,000 times g-force simulating the conditions easily attained in a hydrocyclone. The treatment was applied for three minutes and then the centrate separated manually for analysis. In this case, being a laboratory scale test in a test tube with nowhere to go, the sludge was smashed against the tube's bottom forming a highly compacted paste. This is not the case with hydrocyclones from which the underflow can be continuously recirculated back to the process with no physical damage whatsoever. Moreover, hydrocyclones can be designed for a specific cut-off size, meaning it is possible to yield a particle size which is ideal for obtaining a supernatant or centrate with improved conditions for the membrane filtration process in terms of reduced fouling and higher operational flux.



Figure 65 Resulting centrate from activated sludge samples pre-conditioning tests. From left to right, initial MLSS: 38, 30.4, 22.8, 15.2 g L⁻¹. Final MLSS: 1.14, 0.8, 0.67, 0.37 g L⁻¹.

The supernatant or centrate resulting after the centrifugation was collected and analysed for total suspended and volatile solids, viscosity, and particle size distribution. The results from the analytical tests are summarized in Table 4 and Table 5.

3.4. Sludge solids concentration, viscosity, and particle size distribution

The centrifugal treatment applied on activated sludge for pre-conditioning showed that it is possible to obtain a centrate or supernatant with physical characteristics that could significantly reduce the membrane fouling in MBR installations operating at high solids concentrations with limited space or interested in compact systems with portability features. The most noticeable improvement was in the total suspended solids concentration, with removal efficiencies ranging from 97% and 97.6% for 38 g L⁻¹ and 15.2 g L⁻¹ respectively.

Table 4 Activated sludge characteristics before (TSS) and after (TSSf) the centrifugal pre-conditioning.

TSS (g L ⁻¹)	TSSf (g L ⁻¹)	% Removal (%)	VSS (g L ⁻¹)
15.2	0.37	0.98	0.29
22.8	0.67	0.971	0.445
30.4	0.80	0.974	0.595
38.0	1.14	0.970	0.925

There is an enormous operational advantage when comparing centrifugal methods to conventional solid-liquid gravity separation (settling) in terms of footprint and most importantly the solids separation efficiency; however, other parameters such as the particle size distribution need to be monitored since they might still severely affect the membrane filtration negatively. Nevertheless, the mean particle cut-off size in hydrocyclone can be selected at the design level and fine-tuned onsite for optimal results. This in combination with membrane start-up protocols that allow the formation of a cake layer as pre-coating can contribute towards reduced membrane fouling and improved performance.

One of the main parameters for assessing both the membrane filtration and the oxygen transfer in activated sludge is the viscosity. In Figure 64, activated sludge samples were taken at the end of the membrane filtration tests and centrifuged to mimic the hydrocyclone effect. It is very important to note that activated sludge behaves as a non-Newtonian fluid, meaning its viscosity reduces or thins under shear. Such shear is the result of all hydraulic conditions in the filtration tank and depends on the mixing conditions and aeration intensity mainly which can be very different from installation to installation and therefore is very difficult to standardize for comparison. Typical shear in aerated bioreactors falls between a large range from approximately 40 to 220 s⁻¹ according to *Durán et al., (2016)* but the shear depends greatly on the specific mixing and aeration conditions at each bioreactor or treatment installation, in addition to the resulting shear, the sludge rheological characteristics also depend on many other factors, namely the pH, solids concentration as *Hong et al., (2018)* discussed in relation to sludge in different treatment stages in a wastewater treatment plant.

Figure 64 shows a difference of one order of magnitude in viscosity before and after the sludge pre-conditioning centrifugal treatment. In the first row, the activated sludge viscosity for MLSS concentrations of 15.2, 22.8, 30.4 and 38 g L⁻¹ ranges between 20 and 200 mPa s at a shear rate of approximately 100 s⁻¹ while the same sludge samples that were subject to centrifugation treatment displayed a significant reduction in viscosity with a much narrower range of approximately 5 to 10 mPa s as depicted in more detail in the second-row plots in Figure 64.

By comparing the two plots on the left column, it can be noted that after the sample was subject to centrifugation treatment, the solids reduction had an impact so high on the fluid that the viscosity profile no longer follows the typical shear-thinning trend typical of non-Newtonian fluid for such type of sludge but instead, depicts a typical water viscosity profile in which the initial thinning phase at lower shear is followed by a thickening phase at higher shear rates.

Table 5 Particle size distribution summary for mixed liquor and centrate

TSS (g L ⁻¹)	mean size (Volume) (µm)	mean size (Area) (µm)	Cut-off (50%) (µm)	Cut-off (80%) (µm)	Cut-off (95%) (µm)	SMP (<1 µm) (%)
15.2	345.1	27.92	50.61	982.9	1504	0
22.8	314.7	27.55	47.73	807.6	1497	0
30.4	305.3	26.28	47.23	739.6	1500	0
38	255.9	24.44	43.54	166.8	1406	0
C_15.2	108.1	1.278	24.49	269.3	351.5	26.2
C_22.8	97.48	1.118	29.58	242.4	333.2	29.84
C_30.4	80.07	0.674	22.58	188.9	286.2	34.59
C_38.0	51.2	0.816	2.34	72.97	281.6	39.29

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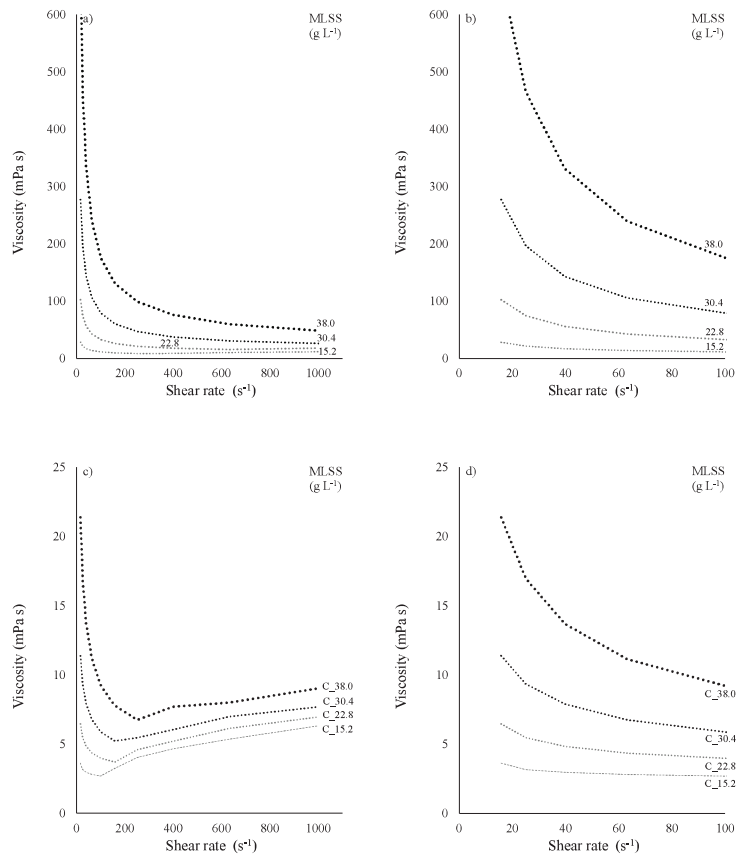


Figure 66 Activated sludge viscosity before and after centrifugation treatment. Top row: activated sludge MLSS 15.2, 22.8, 30.4, 38 g L⁻¹; Bottom row: processed activated sludge centrate MLSS: 0.37, 0.67, 0.8, 1.14 g L⁻¹.

In addition to the viscosity, both activated sludge and centrate samples were analysed for particle size distribution (PSD). Results showed that the centrate is holding a larger fraction of the smaller particles including the sub-micron particles (SMP), which in principle are detrimental for the filtration performance, meaning the SMP could eventually block the membrane faster via pore-blocking mechanisms; however, this could be mitigated by implementing operational protocols for membrane start-up that considers this condition. It is possible to form a sludge cake on the membrane surface at low flux in order to pre-coat it at the beginning of a membrane module start-up or restart after maintenance or cleaning procedures. In such way, a sludge cake can be formed on top of the membrane with a sludge that has not been conditioned (temporarily by-passing the hydrocyclone) in order to obtain the cake layer filtration benefits on fouling mitigation. Once the cake layer has been formed the hydrocyclone system can be started-up for initiation of the centrifugal sludge conditioning and low-solids membrane filtration mode.

In order to make the high solids portable MBR a feasible concept, the oxygen transfer and the membrane filtration issues have to be addressed. The aeration limitations of conventional fine bubble methods can be tackled by using sidestream supersaturation methods such as the SDOX *Kim et al., (2020); Kim et al., (2021)* or the Speece cone *Barreto et al., (2017); Barreto et al., (2018)*. As it is proposed in this study, the filtration performance can be substantially improved by implementing a low-solids filtration stage using a hydrocyclone for sludge pre-conditioning in between the aerobic bioreactor and the membrane tanks. The pre-conditioning process allows for reducing the solids concentration and the sludge viscosity by one order of magnitude in activated sludge of up to 38 g L^{-1} , therefore, it is expected to observe a better filtration performance presenting potential benefits for membrane operation in the pre-conditioned sludge or centrate with lower solids concentration. The results presented in this study suggest that such benefits can be easily obtained in terms of better permeability and reduced membrane fouling. More research is needed in order to establish the long-term effects on the membrane filtration performance and for testing the hydrocyclone concept in a pilot scale and continuous mode. Nevertheless, the particle size distribution analyses performed on the treated samples showed a large increase in sub-micron particles (SMP) (Table 5 and Figure 67) which may cause pore blocking if not addressed properly. It is recommended to run additional tests on novel operational protocols for pre-coating the membrane surface with a cake layer that allows for retention of such smaller particles. Such pre-coating stage should be carried out at a much lower flux and the specific conditions for it to contribute improving the fouling mitigation needs to be determined in further research.

The results in Table 5 show the effect of centrifugation (sludge pre-conditioning) on the particle size distribution (PSD) in activated sludge with MLSS concentrations from 15.2 to 38 g L^{-1} and for the centrate extracted after the treatment. The PSD is presented in terms of: i) mean particle size in micrometres measured per volume unit and per area (columns 2 and 3), the cut-off mean size which represents the mean particle size accounting for 50%, 80% and 95% of all particles in the sample (columns 4, 5 and 6), and in the last column to the right, the percentage of particles with a size smaller than 1 micrometre or sub-micron particles (SMP).

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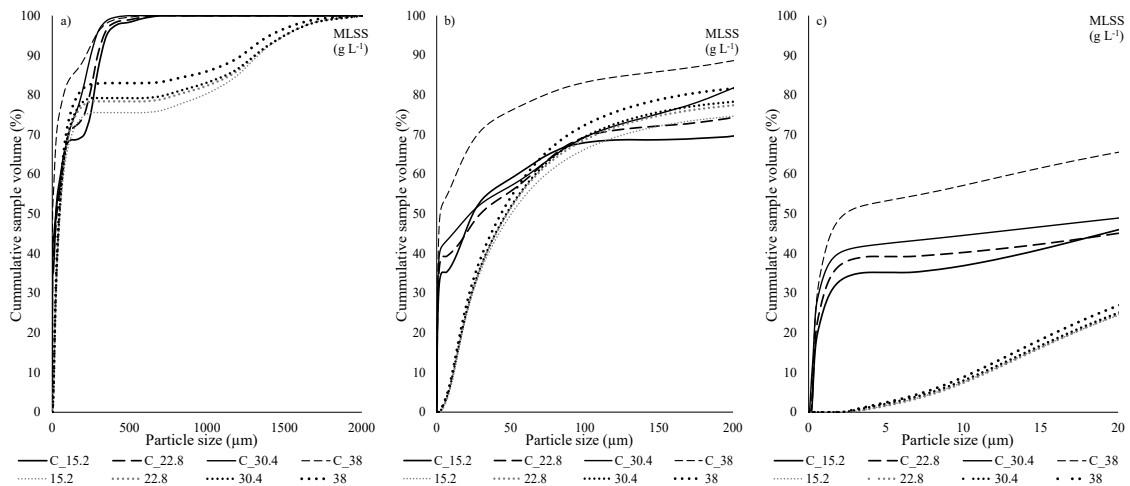


Figure 67 Particle size distribution (PSD) for activated sludge before (MLSS: 15.2; 22.8; 30.4; 38 g L⁻¹) and after centrifugation treatment (C_15.2; C_22.8; C_30.4; 38 g L⁻¹) for solid liquid separation (sludge pre-conditioning).

4. Conclusions

The concept of a high MLSS MBR coupled with a superoxygenation setup was evaluated in terms of the membrane filtration with positive results in regards of the viability of using submerged membranes in high solids concentrations from 5 to 38 g L⁻¹. The results confirmed the anticipated complications in terms of fouling; however, the filtration process was still possible to perform even at MLSS of 38 g L⁻¹ which is beyond expectations for submerged membranes. Nevertheless, more research is needed in the form of long term filtration tests for the determination of the membrane performance in more realistic operational conditions. The sludge pre-conditioning strategy proposed in this study presents an alternative for reducing the membrane fouling in a process configuration that includes a hydrocyclone. The preliminary tests showed very promising results in reducing the activated sludge MLSS concentration and viscosity by one order of magnitude. More experimentation and pilot testing is required in this regard in order to improve and develop a method for introducing a low-solids stage for the membrane filtration process.

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6

REFLECTION AND OUTLOOK

Evaluating a novel approach for overcoming process limitations

The first part of this research was aimed at evaluating the performance of a pilot scale high MLSS MBR coupled with a superoxygenation setup, the Speece cone (SC) as a novel concept for ultra compact wastewater treatment under emergency conditions, this concept is called the High MLSS-SC MBR; Such evaluation focused firstly on the organic load removal and the ability to treat real municipal wastewater under increasing solids concentrations until process limitations could be noticed. During this phase there were operational problems directly related to the solids concentration above 15 g L^{-1} , for instance, the interference of activated sludge with the capacitive level sensors, leading to failure in controlling the MBR level and therefore the complete emptying of the bioreactor contents resulting in massive spilling episodes. After such biomass losses, the MBR had to undergo inoculation and start-up process several times until the decision to change the level sensing method for one that could cope with the new condition could be implemented; In this case, an ultrasonic level sensor was installed, and it fully served its purpose by eliminating such undesired events. Another relevant drawback was the risk of permanent permeability loss (via irreversible fouling) when a moderate flux was applied on new membranes; Meaning the relevance of membrane pre-conditioning by the application of progressively increasing flux when the MLSS changed from low to medium and high concentrations is paramount. After five years of operating such experimental pilot membrane bioreactor, it is fair to say that despite the evident volume and footprint reduction and other benefits such as superior effluent quality, reliability, and portability that can be achieved by increasing the biomass concentration in a membrane bioreactor, running a high MLSS MBR is very challenging from the operational point of view; it requires additional attention, knowledge and specially maintenance; in addition, the equipment wear and tear is accelerated, particularly in the pumps and membranes.. The high solids concentration and high viscosity require more sturdy equipment to cope with exacerbated wear and tear which can be an important factor to consider when choosing a technological alternative for wastewater treatment in terms of operational expenses. Although, in some specific water treatment applications where space is a constraint or portability is desired, the treatment process proposed in this research might present a way to achieve very high effluent quality while keeping the system's footprint minimal. Such applications may include heavy industry with biodegradable effluents, shipping vessels, temporary human settlements such as refugee camps, emergency camps, construction sites, open air public events among many others in which a large human population settles in a short period of time.

The high effluent quality which is typical of MBRs can be maintained under the harsh operational conditions tested in this research. Nevertheless, the additional treatment capacity comes with a price in terms of energy consumption; In other words, the introduction of a superoxygenation method such as the Speece cone, on the one hand enables the effective and efficient transfer of oxygen under very high solids conditions which is by no means possible when using conventional aeration methods such as fine

bubble diffusers; however, on the other hand, and as tested so far in the pilot scale, the energy demand is still high as compared to the mechanical aerators that are currently used in full scale wastewater treatment facilities. In this regard, the system's energy demand can still be optimized via economy of scale, meaning the cost per kilogram of oxygen can be reduced once the system is implemented beyond the pilot scale in small and medium scale applications and further minimized in full scale water treatment systems; In addition, by using more efficient pumps and by including this novel aeration alternative from the design stage will allow for additional savings in terms of its energy demand since it can operate with a very low pressure demand if the pumping equipment and cone are placed in a lower ground where a hydraulic advantage can be obtained.

From the perspective of today, the application of this technology for ultracompact wastewater treatment systems may be more expensive in terms of the operational expenses (OPEX) mainly due to the energy demand in the pilot scale and in municipal wastewater applications; In compensation, it displays other interesting attributes such as the ability to treat high load wastewater from industrial effluents is a reduced footprint, water reuse quality at the discharge, ease to deploy and quick start-up that other conventional wastewater treatment methods could provide with much larger capital expenditures (CAPEX) and complex infrastructure.

Aerobic MBRs are typically used for treating municipal wastewater and in some cases industrial wastewater with COD concentrations above 1500 mg L^{-1} and higher in the organic load spectrum in terms of water pollution. In other industrial applications with higher COD load and thus higher treatment demands, the introduction of anaerobic stages or in some cases anaerobic MBR presents a better option not so much in terms of effluent quality but mostly in the energy demand and the net energy balance when coupled with a co-generation stage using the biogas that can be produced in the anaerobic bioreactor. The anaerobic MBR can be a good alternative for higher load effluents or decentralized municipal wastewater treatment where co-generation associated to the onsite biogas production is possible.

Speece cone characterization in wastewater treatment applications

The Speece cone oxygen transfer capabilities were tested in activated sludge with different solids concentrations ranging from 3 to 40 g L⁻¹, and also in clean water as the baseline. The experimental evaluation considered different combinations of the three main variables governing the mass transfer process, namely the high purity oxygen gas flow to the cone (HPO flow), the mixed liquor recirculation flowrate through the cone, and the operational pressure inside the cone; The results showed that the system's process variables can be adjusted or fine tuned for reaching transfer efficiencies higher than 90% in all cases in MLSS concentrations as high as 36 g L⁻¹. The extensive testing performed on this experimental setup presents very interesting findings in terms of what a Speece cone can do and how far it can go in delivering dissolved oxygen to mixed liquor with solids concentrations up to 40 g L⁻¹. This research showed the operational flexibility of such a system and the capacity to allow for precision dosing minimizing wastage. However, more research is still required in order to improve the energy demand via economy of scale, meaning using bigger and more efficient equipment beyond the pilot scale.

Membrane filtration test (short term)

The modified flux step tests provided many interesting insights regarding the limits for membrane operation under high solids concentrations. The performance was better than expected for most of the evaluated MLSS range, meaning it was possible to reach high fluxes for a submerged type of membrane. However, filtration in concentrations higher than 40 g L⁻¹ seems to mark a boundary for the current process conditions. Beyond this point, it is recommended to develop a method for improving the filtration conditions by pre-conditioning the bulk sludge and in addition the introduction of sidestream (dead-end) membranes which allow for higher fluxes and therefore lower operational expenses in terms of the required energy for permeate production.

Reducing fouling impact via sludge pre-conditioning

Another key part of the process improvement proposed in this research which is yet to be tested in the pilot scale is the preliminary solid-liquid separation of the mixed liquor (or sludge pre-conditioning) prior to the membrane filtration stage using a hydrocyclone; This technological addition to the wastewater treatment process goes hand in hand with the initially proposed High MLSS-Sc MBR and together they form the High MLSS-Sc-Hydrocyclone MBR. So far, laboratory experiments we carried out in order to validate the proposed idea as a proof of concept. The results of laboratory tests showed very interesting results in terms of what can be achieved when the activated sludge is centrifuged under different conditions and solids concentrations. Continuing this research in a pilot scale study focusing on the filtration characterization of pre-conditioned sludge could find very positive results regarding maximum operational permeate flux and fouling reduction that can be achieved with the resulting centrate or mixed liquor supernatant.

The sludge pre-conditioning tests showed that centrifugation can substantially reduce the solids concentration, removing a large part of the suspended particles. However, in the treated samples the Particle size distribution (PSD) was shifted towards smaller particle sizes which can have a negative effect on membrane filtration if not addressed adequately, meaning without performing membrane pre-conditioning by building up a sludge cake layer that can act as an additional barrier for pore blocking particles. More research in this direction in the form of a pilot scale study is of interest for establishing operational protocols of membranes to compensate and mitigate any other negative effect and for measuring the possible membrane performance improvement that can be achieved when a lower-solids stage is introduced. Additional long-term filtration experiments using the low-solids centrate are necessary in order to verify the net operational cost of such configuration considering additional pumping demand but also the ability to process higher volumes of water via the application of much higher fluxes, and also the membrane durability in the long term.

Another interesting addition to this research would be the evaluation of the system using a sidestream membrane filtration arrangement (cross-flow filtration) which can further contribute to an even more compact treatment layout and reduce the membrane cost due to the much higher fluxes that can be applied in such membranes.

The development and operation of a pilot scale system that includes both sidestream additions proposed in this research, i.e the Speece cone for enhanced aeration and a hydrocyclone for enhanced membrane filtration, will produce the necessary data for establishing the extent of the process benefits that can be obtained as well as the long-term effects on membranes and the associated operational costs per meter cube.

The results presented in this study provide sufficient evidence for considering the superoxygenation arrangement as a technically feasible way to overcome the aeration limitations that occur at high MLSS concentrations. Could it be implemented in a context with the impact it was intended for? Yes, and not only in emergency camps but especially in water reclamation and reuse in regions suffering from water scarcity.

An alternative for upgrading small-scale and decentralized wastewater treatment plants.

In addition to the portable sanitation for emergencies and the high-load wastewaters from industry with space limitations, this system could be implemented as an upgrade in small scale municipal wastewater treatment plants with growing populations and limited budgets too. The interventions can be achieved with minimal impact on the current operation if the solution is applied in the form of plug and play skids containing sidestream aeration and sidestream membrane filtration modules for upgrading existing treatment systems by turning conventional activated sludge facilities into adapted MBRs. Making use of existing infrastructure but in higher MLSS operational regime allows for significantly higher effluent quality even to the point of reuse and reclamation, which can be interesting in locations with limited available water particularly in agricultural sector.

As usual, the benefits of implementing such methods can be amplified if it is implemented in combination with other technological advances, for instance, it would be better to couple this technology to renewable energies in order to minimize the demand for resources or furthermore in conjunction biogas-based co-generation methods and for resource recovery in terms of energy and nutrients. Or even further, implementation of a novel WWTP with hydrogen-based energy co-generation which can at the same time provide the residual oxygen that is generated during the hydrolysis process when producing hydrogen onsite, this oxygen gas can be used to feed the superoxygenation system as well. Moreover, in combination with a strong primary treatment process such as electrocoagulation, the concept can grow to an ultra compact alternative for intensive wastewater treatment able to deliver water with high quality at the effluent suitable for reuse in many other applications besides municipal wastewater treatment.

This research thesis contributed in the scientific evaluation of a Speece cone superoxygenation system in wastewater treatment more specifically in membrane bioreactors; The findings regarding the operational capabilities of the Speece cone applied in a high MLSS MBR allow to confidently proceed to the design of new water treatment devices that are more compact and able to provide an alternative for portable sanitation equipment which can be used in emergency settings such as refugee and emergency camps. The results on sludge pre-conditioning and membrane fouling indicate the potential to substantially improve the filtration performance if a low-solids stage is introduced prior to the membrane filtration step.

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His research topic was the assessment and implementation of a pilot superoxygenation system known as the Speece cone, for the supersaturation of activated sludge in a membrane bioreactor (MBR) in order to validate the concept and evaluate the benefits and drawbacks of introducing compact and mobile wastewater treatment systems in the context of sanitation in emergency conditions.

After completion of his PhD studies and research work, he moved back to Colombia where he continued the development and commercialization of compact MBR and other membrane technology equipment for the provision of drinking water in remote areas and industrial clients in his own OEM water company based in Colombia, "*Water treatment engineering S.A.S*". In parallel, he opened a start-up company *NuAgua-Colombia S.A.S* and *NuAgua B.V.* in The Netherlands, focused on the application of water treatment and advanced aeration in the field of aquaculture adopting biotechnology and circular economy principles for the sustainable production of food with a more efficient use of waste and resources.

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Journals publications

- Barreto, C. M., García, H. A., Hooijmans, C. M., Herrera, A., & Brdjanovic, D. (2017). Assessing the performance of an MBR operated at high biomass concentrations. *International Biodeterioration & Biodegradation*, 119, 528-537.
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- o *Enhanced Oxygen transfer at high MLSS concentrations in a membrane bioreactor.* IHE Delft Annual PhD Symposium 1-3 October, 2017, Delft, The Netherlands
- o *High MLSS MBR with mixed liquor preconditioning for fouling mitigation.* IHE Delft Annual PhD Symposium 1-3 October, 2017, Delft, The Netherlands

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This research work tackles the operational limitations that arise when an aerobic membrane bioreactor (MBR) operates at higher than conventional mixed liquor suspended solids concentrations (MLSS). Under high MLSS conditions, the oxygen transfer capabilities of conventional fine bubble diffusion methods are reduced so drastically that it creates a process barrier, preventing the application of more capable wastewater treatment systems that are smaller and more portable, particularly in the emergency sanitation context where these characteristics are of great value. By implementing an alternative sidestream superoxygenation method known as the Speece cone, it is possible to push such

process limitations and increase the MLSS concentration in the bioreactor up to ten times as compared to conventional aeration methods and still have oxygen transfer efficiency (OTE) rates higher than 80%. In addition to improved aeration performance, an alternative MBR process configuration is proposed to include not only the Speece cone but also a hydrocyclone that can reduce the fouling potential of high solids activated sludge and minimize its negative impact on membrane fouling via sludge pre-conditioning using the solid-liquid separation capabilities of hydrocyclones to create a low-solids stage in between the aerobic bioreactor and the membrane tank.