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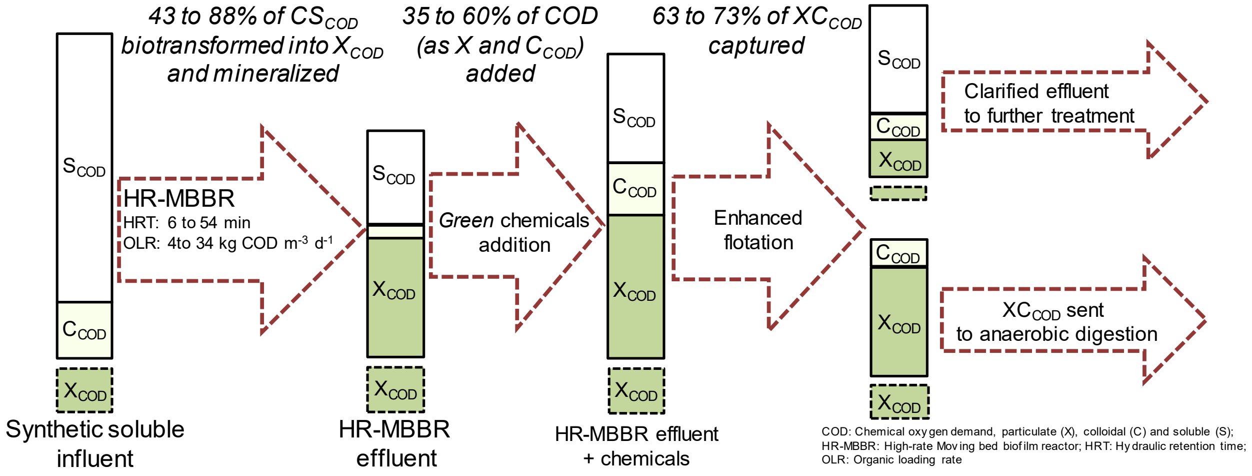

Highlights

- A treatment train combining an HR-MBBR with an enhanced flotation process was studied
- CS_{COD} removal efficiency increased with the MBBR HRT (inversely with OLR) up to 88%
- An MBBR OLR of 6 to 10 kg COD m^{-3} reactor d^{-1} resulted in the best X_{COD} recovery
- MBBR effluent solids were characterized by a bimodal volume particle size distribution
- Enhanced flotation with *green* chemicals allowed to reach 10 ± 3 mg TSS/L in the MBBR effluent

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Abstract

High-rate wastewater processes are receiving a renewed interest to obtain energy positive/efficient water resource recovery facilities. An innovative treatment train combining a high-rate moving bed biofilm reactor (HR-MBBR) with an enhanced flotation process was studied. The two objectives of this work were 1) to maximize the conversion of soluble organics to particulate matter in an HR-MBBR and 2) to maximize the particulate matter recovery from the HR-MBBR effluent by *green* chemicals to enhance biogas production by anaerobic digestion. To achieve these objectives, lab-scale MBBRs fed with synthetic soluble wastewater were operated at organic loading rates (OLRs) between 4 and 34 kg COD m^{-3} reactor d^{-1} corresponding to hydraulic retention times (HRTs) between 6 and 54 minutes.

Colloidal and soluble chemical oxygen demand (COD) removal efficiency in the HR-MBBR increased with HRT to reach a plateau of 85% at an HRT longer than 27 minutes. Carrier clogging observed at an OLR higher than 16 kg COD m^{-3} d^{-1} (HRT $<$ 13 minutes) resulted in about 23% loss in colloidal and soluble COD removal efficiency. Thus, the recommended parameters were between 22 and 37 minutes and between 6 to 10 kg COD m^{-3} d^{-1} for the HRT and the OLR, respectively, to maximize the conversion of soluble organics to particulate matter.

Total suspended solids (TSS) recovery of 58 to 85% and 90 to 97% were achieved by enhanced flotation using *green* and unbiodegradable chemicals, respectively, corresponding to a TSS effluent concentration below 14 and 7 mg TSS/L. Among the synthetic polymers tested, a high molecular weight and low charge density cationic polyacrylamide was found to give the best results with less than 2 mg TSS/L in the clarified effluent (97% TSS recovery). *Green* chemicals, although performing slightly less for solids separation than unbiodegradable chemicals, achieved a mean TSS concentration of 10 ± 3 mg/L in the clarified effluent.

Keywords

Moving bed biofilm reactor; innovative separation process; enhanced flotation; green coagulant; green polymer; high-rate process

Abbreviations

AD: anaerobic digestion
AS: activated sludge
BOD: biochemical oxygen demand
C: clarified effluent after separation process
CAS: conventional AS
C_B: colloidal biodegradable COD
C_{COD}: colloidal COD
COD: chemical oxygen demand
CS_{COD}: colloidal and soluble COD
DAF: dissolved air flotation
E: MBBR effluent before separation process
EF: enhanced flotation
f_{COD}: filtered COD
f_{CV}: particulate COD to VSS fraction
ff_{COD}: flocculated-filtered COD
f_{VT}: VSS to TSS fraction
HR-MBBR: high-rate MBBR
HRT: hydraulic retention time
MBBR: moving bed biofilm reactor
Me: metal
MW: molecular weight
NH_3: free ammonia
NH_4^+: ammonium ion
NO_2: nitrite (HNO_2 + NO_2^-)

NO_3: nitrate (HNO_3 + NO_3^-)
OLR: organic loading rate
PAM: polyacrylamide
PSD: particle size distribution
R: removal efficiency
S_B: soluble biodegradable COD
S_{COD}: soluble COD
S_{NH4}: total ammonia (free ammonia plus ammonium)
S_{NOx}: oxidized nitrogen (nitrite plus nitrate)
S_{PO4}: woluble phosphate
SR: wpecific removal
TKN: total Kjeldahl nitrogen
TP: total phosphorus
TSS: total suspended solids
VSS: volatile suspended solids
WRRF: water resource recovery facility
WWTP: wastewater treatment plant
X_B: particulate biodegradable COD
X_{COD}: particulate COD
X_{OHO}: heterotrophic biomass
X_{STO}: stored particulate matter

1 Introduction

Wastewater treatment plants (WWTPs) are now renamed water resource recovery facilities (WRRFs) to reflect the new processes requirements to recover not only water quality but also organic matter, nutrients, other compounds and energy. High-rate (HR) wastewater processes are receiving a renewed interest for their potential to obtain energy positive/efficient WRRFs. They are most often associated with the activated sludge (AS) process operated at a high food to microorganism ratio, a short hydraulic retention time (HRT) and a short aeration time (Tilley, 2011; Nogaj *et al.*, 2015).

The advantages of the moving bed biofilm reactor (MBBR) over the conventional activated sludge process (CAS) are to reduce footprint and eliminate sludge recirculation (Ødegaard, 2000). A relatively high sludge age and consequently biomass specialization can be achieved in biofilm reactors even in the absence of sludge recirculation. The MBBR can be used as primary, secondary or tertiary treatment, under aerobic, anoxic and anaerobic conditions, depending on the treatment objectives (Ødegaard, 2000). The MBBR can easily be retrofitted in existing WRRFs due to its compactness when compared to conventional technologies (McQuarrie and Boltz, 2011).

Biomass grown in HR processes transforms the rapidly biodegradable chemical oxygen demand (COD) that is soluble (S_B) and colloidal (C_B) into stored particulate matter (X_{STO}) and heterotrophic biomass (X_{OHO}), leaving the particulate fraction (X_B) practically unchanged.

$$X_B + C_B + S_B + S_{O2} \rightarrow X_{STO} + X_{OHO} + X_B \qquad (1)$$

The MBBR HRT typically ranges from 30 to 90 minutes and is kept low partly to prevent X_B hydrolysis, which would complicate its downstream recovery. Hence, under low HRT and high-rate conditions, the influent particulate fraction of COD (X_B) can be neglected for design purposes (Ødegaard, 1999; Ødegaard *et al.*, 2000). The capture of the rapidly biodegradable COD combined with the minimization of X_B hydrolysis and its capture allows to maximize biogas production by anaerobic digestion (AD).

Biomass grown under high-rate conditions is known for its poor settleability due the generation of un-flocculated biomass and pin flocs, which is often reported for MBBR effluent solids using the method of wide volume particle size distributions (PSDs) (Ødegaard *et al.*, 2000, 2010; Melin *et al.*, 2005; Åhl *et al.*, 2006; McQuarrie and Boltz, 2011; Karizmeh, 2012). Thus, when the MBBR is in a secondary treatment configuration, an efficient downstream solids separation process combined with appropriate coagulating agents is required to recover the effluent solids (Ødegaard *et al.*, 2010; Ivanovic and Leiknes, 2012). Dissolved air flotation (DAF) and conventional settling are among the most frequently used separation processes for MBBR effluents. They are often combined with metallic based coagulants (Ødegaard, 2000; Ødegaard *et al.*, 2010; Ivanovic and Leiknes, 2012).

Innovative separation processes are needed to reduce energy costs, footprint, unbiodegradable chemicals addition and to promote the use of natural-based or *green* chemicals, such as tannins and starches. In this perspective, there is an increasing interest in organic *green* chemicals and previous experiments have revealed their high performance in municipal and industrial wastewater treatment along with their biodegradability potential (Özacar and Şengil, 2003; Omar *et al.*, 2008; Beltrán-Heredia and Sánchez-Martín, 2009; Beltrán-Heredia *et al.*, 2010; Sánchez-Martín *et al.*, 2010a; Beltrán-Heredia *et al.*, 2011; Fatehah *et al.*, 2013, Lapointe and Barbeau, 2015). More specifically, the potential of capturing the COD in the effluent of a high-rate MBBR (HR-MBBR) with a *green* coagulant was demonstrated (Schubert *et al.*, 2013).

The enhanced flotation process is an alternative to the conventional DAF process. This process uses low-density beads instead of air as flotation medium and works without the energy consuming air pressurization system of a conventional DAF. The beads may be recovered from sludge by hydrocyclones, centrifuges or vibratory screens and recycled for multiple utilisations (Eades and Penno, 2005; Jarvis *et al., 2009*). The high natural organic matter and total suspended solids (TSS) recovery efficiency of a similar enhanced flotation process treating water from a drinking water treatment plant and sewer overflows was demonstrated by Jarvis *et al.* (2011) and Stanley and Evans (1977). The efficiency of enhanced flotation for the recovery of particulate matter in the effluent of an HR-MBBR remains has not yet been reported.

The aim of this paper was to determine the efficiency of an innovative secondary treatment process train for achieving high-energy efficiency and low carbon footprint. This process train can be used for designing or retrofitting existing WRRFs. An MBBR operated at high-rate conditions for the biotransformation of colloidal and soluble chemical oxygen demand (CS_{COD}) into particulate organics is followed by a chemically enhanced solids separation process for maximum recovery of biodegradable organics that can then be converted into biogas by anaerobic digestion.

The two specific objectives of this project were to:
1. determine the optimal MBBR HRT and organic loading rate (OLR) to maximize CS_{COD} biotransformation, and
2. determine the efficiency of the innovative enhanced flotation process combined with *green* chemicals to achieve a strict target TSS concentration of 10 mg/L or less.

2 Methods

2.1 Synthetic feed

Two 1 L lab-scale MBBRs operated in parallel were fed with a synthetic soluble influent. This was done to ensure constant influent characteristics to understand and describe the underlying phenomena of biotransformation and capture of organic matter throughout the treatment train (Figure 1). A concentrated feed of 10 g COD/L was prepared every four days, autoclaved (121°C, 2 bar for 15 minutes) and kept at 4°C. The concentrated solution was based on a recipe adapted from OECD (1976) to obtain a typical C/N/P ratio of 100/12/2 for a medium to high strength domestic wastewater (Metcalf and Eddy AECOM, 2014) (Table 1). The synthetic influent provided C, N, P and minerals to favor biofilm growth.

The solution was pumped and diluted with tap water before entering the reactors to obtain a COD concentration of about 160 mg/L and an alkalinity of 150 mg/L as $CaCO_3$, to be representative of the soluble fraction (without TSS) of a medium strength wastewater (Metcalf and Eddy AECOM, 2014). Tap water was used as dilution water to provide additional minerals (Mg, Ca, etc.) and alkalinity to the influent. The characteristics of the synthetic influent, after dilution of the concentrated solution, are presented in Table 2.

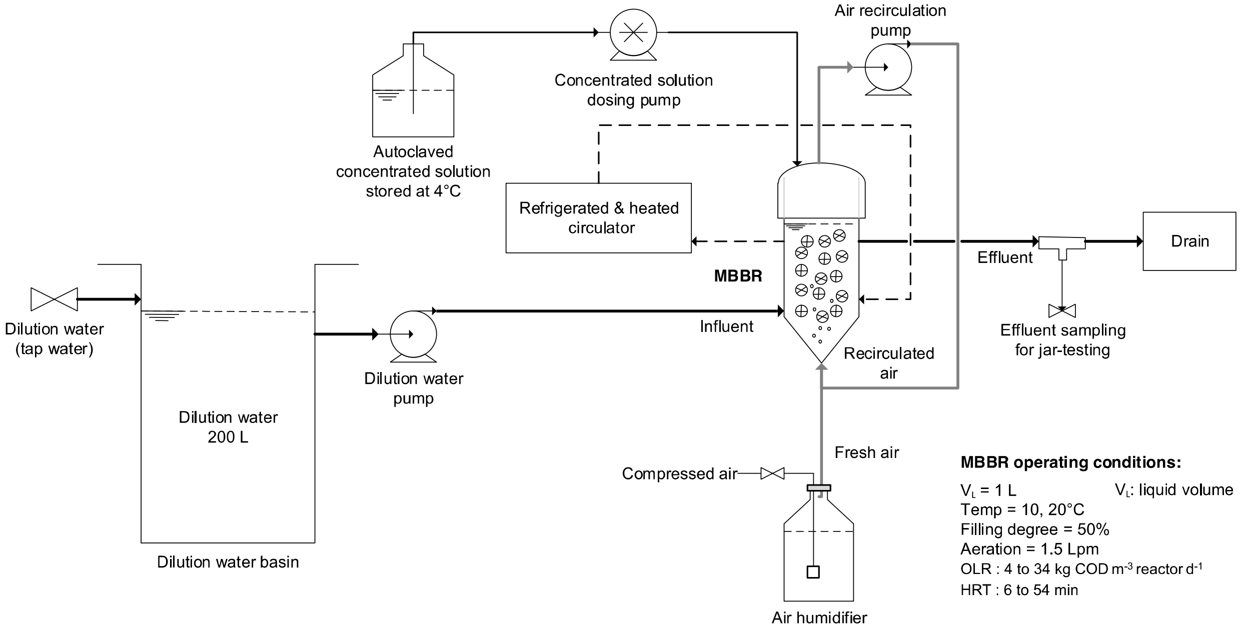

Figure 1. Schematic of the experimental setup.

Table 1. Concentrated feed composition and COD concentration used for the synthetic influent

Compounds	Chemical formula	COD concentration (g/L)
Sodium acetate trihydrate	$CH_3COONa{\cdot}3H_2O$	3.7
Sodium propionate	CH_3CH_2COONa	1.6
Soy peptone	-	5.7
Nutrient broth	-	1.2
Dipotassium phosphate	K_2HPO_4	1.1
Ammonium chloride	NH_4Cl	2.1
Sodium chloride	NaCl	0.25

Table 2. Average characteristics of the lab-scale MBBR synthetic influent after dilution with tap water

Parameters	Symbol	Units	Value
Total COD	COD	mg/L	168 ± 17
Particulate COD	X_{COD}	mg/L	1 ± 6
Colloidal COD	C_{COD}	mg/L	29 ± 3
Soluble COD	S_{COD}	mg/L	138 ± 8
Total 5-d carbonaceous BOD	BOD	mg/L	95 ± 5
Biodegradability COD/BOD ratio	f_{COD_BOD}	-	2.1 ± n.a.*
Total Kjeldahl nitrogen	TKN	mg N/L	19 ± 1
Total ammonia (NH_3 + NH_4^+)	S_{NH4}	mg N/L	9.9 ± 0.6
Oxidized nitrogen (nitrite plus nitrate)	S_{NOx}	mg N/L	0.08 ± 0.06
S_{NH4}/TKN ratio	f_{SNH4_TKN}	-	0.53 ± 0.04
Total phosphorus	TP	mg P/L	3.9 ± 0.2
Soluble phosphate	S_{PO4}	mg P/L	2.2 ± 0.6
S_{PO4}/TP ratio	f_{SPO4_TP}	-	0.6 ± 0.2

*n.a.: not available

2.2 Lab-scale MBBRs

The biofilm carriers were AnoxKaldnes™ K5 (Veolia Water Technologies Canada) at a filling degree of 50% and were kept in suspension by aeration. The aeration flow rate was maintained at 1.5 Lpm. To minimize evaporation, the aeration consisted of 0.5 Lpm of fresh pre-humidified air and of 1.0 Lpm of recycled air from the airtight headspace. The temperature was controlled in the double-jacketed MBBRs by a refrigerated and heated circulator (Programmable Circulator 9712, PolyScience, USA).

The MBBRs were inoculated with previously colonized K3 carriers sampled from the full-scale MBBR of Terrebonne/Mascouche WRRF to favor biofilm growth and the development of a microbial ecosystem resembling that of a WRRF. The full-scale MBBR is located downstream of the first aerated lagoon and is followed by three lagoons for sludge storage and phosphorus removal (with a coagulant added just prior to the last lagoon). The K3 carriers were added to the lab-scale MBBRs containing the un-colonized K5 carriers during five days and were removed prior to the experiments.

2.3 Operating conditions

Seven operating conditions were tested under pseudo steady state conditions with MBBR volumetric OLR and HRT varying concomitantly from 4 to 34 kg COD m^{-3} reactor d^{-1} and from 6 to 54 minutes (Table 3). The MBBR stability was determined by the treatment efficiency and effluent characteristics. Data was rejected when clogging of the carriers was observed. Each operating condition was conducted during 3 to 5 weeks including a growth and stabilisation period of 1 to 2 weeks followed by a characterization period of 2 to 3 weeks. The MBBR liquid volume was corrected to account for the volume taken by the carriers and air bubbles.

Experiments were conducted at 20°C except for two additional experiments at 10°C to evaluate the effect of temperature (Table 3).

Table 3. Expected and measured HRT, OLR, temperature and dissolved oxygen (DO) throughout the lab-scale MBBR seven operating conditions (OC)

OC	HRT		OLR		Temperature	DO
	Expected	Measured	Expected	Measured	Measured	Measured
	min	min	kg COD m^{-3} d^{-1}	kg COD m^{-3} d^{-1}	°C	mg O_2/L
OC1	7	5.8 ± 0.2	34	34 ± 2	20	5.1 ± 0.4
OC2	10	9 ± 1	24	24 ± 1	20	4.4 ± 0.9
OC3_20°C	15	13 ± 1	16	15.6 ± 0.5	20	2.8 ± 0.3
OC3_10°C	15	13.5 ± 0.1	16	13.1 ± 0.01	10	n.a.*
OC4_20°C	25	22 ± 1	10	9.6 ± 0.3	20	4.0 ± 0.1
OC4_10°C	25	23 ± 1	10	7.9 ± 0.02	10	n.a.
OC5	30	27 ± 3	8	7.2 ± 0.1	20	4.2 ± 0.5
OC6	40	37 ± 1	6	6.1 ± 0.1	20	4.4 ± 0.4
OC7	60	54 ± 0.8	4	4.1 ± 0.1	20	5.1 ± 0.4

*n.a.: not available

CS_{COD} removal efficiency ($R_{CS_{COD}}$) was evaluated by comparing CS_{COD} content (in mg/L) in the MBBR influent and effluent:

$$R_{CS_{COD}}(\%) = \frac{\left(CS_{COD_{influent}} - CS_{COD_{effluent}}\right)}{CS_{COD_{influent}}} \times 100\% \tag{2}$$

which can be expressed per unit reactor volume as the volumetric CS_{COD} specific removal ($SR_{CS_{COD}}$).

The observed yield ($Y_{obs,TSS}$ in g TSS produced/g CS_{COD} removed) and the X_{COD} yield ($Y_{obs,X_{COD}}$ in g X_{COD} produced/g CS_{COD} removed) were determined using the following equations:

$$Y_{obs,TSS} = \frac{X_{TSS_{effluent}}}{CS_{COD_{influent}} - CS_{COD_{effluent}}} \tag{3}$$

$$Y_{obs,X_{COD}} = \frac{COD_{effluent} - CS_{COD_{effluent}}}{CS_{COD_{influent}} - CS_{COD_{effluent}}} \tag{4}$$

The theoretical yield for heterotrophic biomass was assumed to be of 0.66 g X_{COD} produced/g COD consumed (Ekama and Marais, 1984) corresponding to:

$$0.66\ \frac{g\ X_{COD}}{g\ COD} \times \frac{1}{1.42}\ \frac{g\ VSS}{g\ X_{COD}} = 0.46\ \frac{g\ VSS\ produced}{g\ COD\ consumed} \tag{5}$$

and, assuming 10% of inorganic matter (f_{VT} = 0.90 g VSS/g TSS):

$$0.46\ \frac{g\ VSS}{g\ COD} \times \frac{1}{0.9}\ \frac{g\ TSS}{g\ VSS} = 0.52\ \frac{g\ TSS\ produced}{g\ COD\ consumed} \tag{6}$$

2.4 Jar-testing

2.4.1 Operating conditions

Jar-tests were conducted using a six-paddle stirrer in 2 L beakers B-KER^2 (Phipps & Bird, USA) with a valve at 1/3 of their height (42 mm above bottom level and 105 mm below water level) for clarified water sampling. All jar-tests were conducted at room temperature, corresponding to 22 ± 1°C.

2.4.2 Lab-scale MBBR effluent samples

Lab-scale MBBR effluent samples were collected over a 2 to 10 hour period depending on the required effluent volume for the jar-test experiments, which varied from 2 to 28 L, and were stored at 4°C if not used immediately for analysis.

First, the TSS recovery efficiency of unbiodegradable chemicals, alum as coagulant and charged/uncharged synthetic polymers, was compared to that of *green* chemicals. Second, the influence of the MBBR HRT, and resulting OLR, on the TSS recovery efficiency by *green* chemicals was studied. The MBBRs were operated as continuous flow systems and were used to feed the jar-test experiments which were conducted in batches.

Regarding the unbiodegradable chemicals, the efficiency of high molecular weight (MW) cationic or anionic polyacrylamide (PAM) polymers was compared during the enhanced flotation process. Lab-scale enhanced flotation and conventional settling process efficiencies were compared at given chemical dosages using adapted jar-test procedures.

Regarding the *green* chemicals, a tannin coagulant, Hydrex 3818, and a potato starch polymer, Hydrex 3842, were used. The effect of MBBR HRT and OLR on enhanced flotation efficiency was evaluated using the *green* chemicals at given dosages. The chemicals tested with their associated dosage ranges are presented in Table 4.

Table 4. Jar-test operating conditions: type and dosage of chemicals used to assess TSS recovery efficiency of conventional settling and enhanced flotation

Chemical	Name	Description	Dosage range
Unbiodegradable coagulant	ALS	Aluminum sulphate (alum)	0.0 - 0.2 mmol Me*/L
Green coagulant	Hydrex 3818	Tannin-based	0 - 33 mg/L
Synthetic polymer	A-100 HMW A-110 A-130 C-492 N-300 Hydrex 3551	Charged/Uncharged	0 - 1 mg/L
Green polymer	Hydrex 3842	Potato starch-based	0 - 20 mg/L

*Me: Metal

2.4.3 Jar-test procedures

The enhanced flotation jar-test procedure included three periods:
- period 1. addition of coagulant and mixing at 150 rpm (G of 170 s^{-1}; adapted from graphic of Cornwell and Bishop, 1983) during 2 minutes - coagulation period,
- period 2. addition of polymer and beads and mixing at 300 rpm (390 s^{-1}) during 2 minutes - flocculation & beads-solids adhesion period, and
- period 3. mixing stopped during 2 minutes - quiescent flotation period.

Mixing energy had to be increased during the flocculation & beads-solids adhesion period to allow the low-density medium to be thoroughly homogenized in the sample. The enhanced flotation medium consisted of low density (d = 24 kg/m^3; median diameter = 40 µm) expanded thermoplastic microspheres and was dosed at 9 mg/L.

The conventional settling jar-test procedure included three periods:
- period 1. addition of coagulant and mixing at 160 rpm (180 s^{-1}) during 2 minutes - coagulation period,
- period 2. addition of polymer and mixing at 45 rpm (35 s^{-1}) during 15 minutes - flocculation period, and
- period 3. mixing stopped during 15 minutes - quiescent settling period.

2.5 Analytical methods

The characterization of the MBBR influent and effluent was done 2 to 5 times per week. Chemical oxygen demand (COD), total suspended solids (TSS) and volatile suspended solids (VSS) were measured according to APHA *et al.* (2012). Glass microfiber 1.2 µm filters (Whatman® 934-AH™, GE Healthcare Life Sciences, GBR) and 0.45 µm cellulose membrane filters (MF-Millipore™, EMD Millipore, USA) were used for TSS and COD, respectively.

Flocculated-filtered COD (ff_{COD}) was measured according to Mamais *et al.* (1993). All nitrogen and phosphorus species were measured according to APHA *et al.* (2012) using a flow injection analysis instrument (Quickchem® 8500, Lachat Instruments, USA). Alkalinity (S_{Alk}) was measured with an automated titrator (DL28 Titrator, Mettler Toledo, USA). Dissolved oxygen (DO) concentration was measured with a portable DO-meter (HQ40d, Hach Company, USA) and a LDO® probe (Hach Company, USA).

The following definitions were used for COD size fractionation:

soluble COD: S_{COD} (or flocculated-filtered COD, ff_{COD}): < 0.04 µm
colloidal COD: C_{COD}: 0.04 to 0.45 µm, and
particulate COD: X_{COD}: > 0.45 µm.

Particle size distribution was measured using a Mastersizer 3000 (Malvern Instruments Ltd., U.K.). Particles were considered non spherical and their distribution was expressed in volume equivalent. Visual observations were made using an Axioskop 40 (Carl Zeiss, DEU) microscope and a binocular (Portable Luminaire model 52343, Underwriters Laboratories, USA).

2.6 Statistical analysis

Student's t-test analyses were conducted on the MBBR efficiency and sludge production sets of data. T-TEST function in EXCEL was used with bilateral and heteroscedastic distributions and the least significant difference was set at $p < 0.05$.

3 Results and discussion

3.1 MBBR results

3.1.1 Biotransformation of CS_{COD} into X_{COD}

The determination of the MBBR optimal operating condition(s) for maximizing the production of biodegradable sludge was based on three criteria:

- maximization of the specific removal efficiency of CS_{COD},
- maximization of the observed yield and
- non-clogging of the carriers.

A summary of the lab-scale MBBRs performance and effluent characteristics for the seven operating conditions is presented in Table 5. Effluent nitrate concentration remained around 0.1 mg N/L thus showing no significant nitrification occurring in the MBBRs as expected at such high rate conditions (results not shown).

Table 5. Summary of the lab-scale MBBR effluent average characteristics and performance for the seven operating conditions (OC) at 20°C and 50% v/v K5 carriers

Parameters	Symbol	Units	OC1	OC2	OC3	OC4	OC5	OC6	OC7
HRT	-	min	5.8 ± 0.2	9 ± 1	13 ± 1	22 ± 1	27 ± 3	37 ± 1	53.7 ± 0.8
OLR	-	kg COD m^{-3} d^{-1}	34 ± 2	24 ± 1	15.6 ± 0.5	9.6 ± 0.3	7.2 ± 0.1	6.1 ± 0.1	4.1 ± 0.1
Flow	-	mL/min	151 ± 6	99 ± 9	66 ± 6	40 ± 2	32 ± 4	23 ± 1	16.2 ± 0.3
Total COD	COD	mg/L	129 ± 7	136 ± 15	119 ± 11	118 ± 10	113 ± 22	116 ± 12	83 ± 17
Particulate COD	X_{COD}	mg/L	31 ± 9	36 ± 5	43 ± 11	63 ± 12	86 ± 23	88 ± 7	62 ± 15
Colloidal COD	C_{COD}	mg/L	12 ± 1.3	10 ± n.a.*	12 ± 2	20 ± 3	n.a.	4 ± n.a.	6 ± 1.2
Soluble COD	S_{COD}	mg/L	84 ± 6	83 ± n.a.	67 ± 6	35 ± 5	n.a.	43 ± n.a.	13.8 ± 0.7
TSS	X_{TSS}	mg/L	27 ± 4	29 ± 2	37 ± 5	47 ± 12	59 ± 13	61 ± 12	48 ± 13
VSS	X_{VSS}	mg/L	23 ± 3	26 ± 3	34 ± 4	41 ± 11	59 ± 14	53 ± 11	40 ± 10
Alkalinity	S_{Alk}	mg $CaCO_3$/L	n.a.	n.a.	141 ± 9	141.9 ± 0.5	138 ± n.a.	151 ± 3	147 ± n.a.
pH	-	-	n.a.	n.a.	7.6 ± 0.3	7.8 ± 0.1	7.7 ± n.a.	7.7 ± 0.2	7.6 ± n.a.
Volume PSD median	Dv50	µm	376 ± 26	n.a.	182 ± 1	n.a.	n.a.	117 ± 6	215 ± 3
HRT	-	min	5.8 ± 0.2	9 ± 1	13 ± 1	22 ± 1	27 ± 3	37 ± 1	53.7 ± 0.8
OLR	-	kg COD m^{-3} d^{-1}	34 ± 2	24 ± 1	15.6 ± 0.5	9.6 ± 0.3	7.2 ± 0.1	6.1 ± 0.1	4.1 ± 0.1
TSS production	-	g/d	5.7 ± 0.8	4.1 ± 0.6	3.5 ± 0.3	2.6 ± 0.3	2.4 ± 0.6	2.1 ± 0.4	1.4 ± 0.2
VSS production	-	g/d	5.0 ± 0.5	3.7 ± 0.6	3.1 ± 0.3	2.2 ± 0.3	2.5 ± 0.4	1.8 ± 0.4	1.0 ± 0.2
X_{COD}/VSS ratio	$f_{CV} = f_{XCOD\_VSS}$	g X_{COD}/g VSS	1.46 ± 0.16	1.39 ± 0.10	1.24 ± 0.16	1.43 ± 0.09	1.56 ± 0.09	1.60 ± 0.13	1.54 ± 0.17
VSS/TSS ratio	$f_{VT} = f_{VSS\_TSS}$	g VSS/g TSS	0.87 ± 0.06	0.89 ± 0.03	0.89 ± 0.05	0.86 ± 0.03	0.91 ± 0.03	0.88 ± 0.05	0.81 ± 0.18
CS_{COD} removal efficiency	R_{CSCOD}	%	43 ± 1	45 ± 2	56 ± 3	66 ± 3	83 ± 2	87 ± 2	88 ± 2
CS_{COD} specific removal rate	SR_{CSCOD}	kg CS_{COD} m^{-3} d^{-1}	15.0 ± 0.4	11 ± 1	8.9 ± 0.3	6.3 ± 0.3	6.0 ± 0.1	5.2 ± 0.2	3.6 ± 0.1
TSS observed yield	$Y_{obs,TSS}$	g TSS/ g $CS_{COD,removed}$	0.42 ± 0.07	0.42 ± 0.04	0.42 ± 0.02	0.41 ± 0.04	0.40 ± 0.05	0.39 ± 0.04	0.31 ± 0.07
VSS observed yield	$Y_{obs,VSS}$	g VSS/ g $CS_{COD,removed}$	0.37 ± 0.06	0.38 ± 0.05	0.37 ± 0.03	0.35 ± 0.04	0.39 ± 0.04	0.34 ± 0.02	0.28 ± 0.06
X_{COD} observed yield	$Y_{obs,XCOD}$	g X_{COD}/ g $CS_{COD,removed}$	0.48 ± 0.05	0.49 ± 0.05	0.50 ± 0.05	0.54 ± 0.07	0.58 ± 0.08	0.54 ± 0.02	0.42 ± 0.08

*n.a.: not available

CS_{COD} removal efficiency and specific removal rate are shown in Figure 2. CS_{COD} removal efficiency increased with HRT (or inversely for OLR) as expected. For an HRT longer than 27 minutes (below an OLR of 7 kg COD m^{-3} d^{-1}), the efficiency reached a plateau at 85% (Figure 2A & B). A similar tendency between OLR and total COD removal efficiency was obtained by Aygun *et al.* (2008) although their efficiencies were systematically higher due to higher available surface area for biofilm growth, influent total COD concentration and HRT in their experiments. Moreover, their COD removal efficiencies were calculated by measuring the difference in COD concentration in MBBR influent and in settler clarified effluent, located downstream of the MBBR (Aygun *et al.*, 2008).

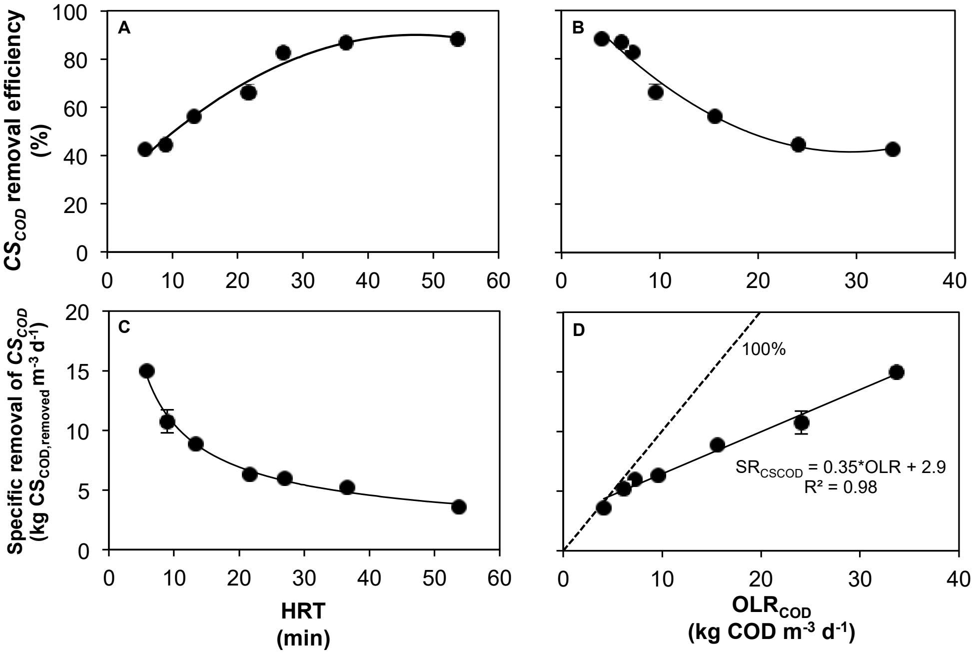

Figure 2. Lab-scale MBBR CS_{COD} removal efficiency (A, B) and specific removal rate (C, D) as a function of HRT (A, C) and OLR (B, D) at 20°C and 50% v/v K5 carriers. The errors bars indicate the standard deviation and are not shown if smaller than symbol size.

A maximum specific removal rate of 15 kg $CS_{COD,removed}$ m^{-3} d^{-1} was reached at the highest OLR applied of 34 kg COD m^{-3} d^{-1} (Figure 2D). The linear increase of specific removal rate with OLR was also observed by previous lab- and pilot-scale experiments with HR-MBBRs (Ødegaard *et al.*, 2000; Helness *et al.*, 2005). The slope of 0.35 obtained indicated that around 65% (difference between an ideal slope of 100%) of the colloidal and soluble COD in the influent was not consumed even at the highest HRT tested (54 minutes) (Ødegaard *et al.*, 2000). An asymptotic relationship between the mass of attached biomass as dry solids and OLR was observed by Orantes and González-Martínez (2003). At OLRs over 4.8 kg COD m^{-3} d^{-1}, they observed no supplementary biomass attached. Hence, at high loading rates the COD biodegradation does not depend on OLR, but is limited by the short HRT used (Ødegaard *et al.*, 2000; Orantes and González-Martínez, 2003; Helness *et al.*, 2005).

The observed yields (Y_{obs}) expressed as the production of TSS and of X_{COD} over the substrate consumption are shown in Figure 3 for the seven operating conditions along with the theoretical yield values. $Y_{obs,TSS}$ increased with OLR and inversely with HRT to reach a constant value of 0.42 g TSS/g $CS_{COD,removed}$ (or 0.37 g VSS/g $CS_{COD,removed}$; Table 5) from 16 to 34 kg COD m^{-3} d^{-1}. A maximum $Y_{obs,XCOD}$ of 0.58 g X_{COD}/g $CS_{COD,removed}$ was determined at an HRT of 27 minutes and an OLR of 7 kg COD m^{-3} d^{-1} close to the theoretical value of 0.66 g X_{COD}/g $COD_{removed}$ (Figure 3C & D).

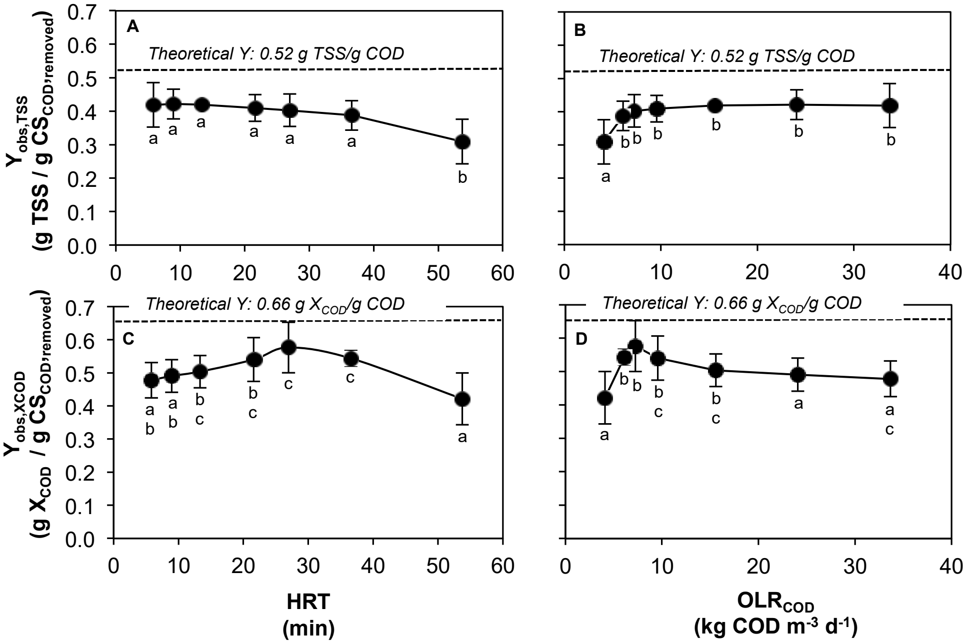

Figure 3. Lab-scale MBBR observed yields expressed in TSS (A, B) and in X_{COD} production (C, D) as a function of HRT (A, C) and OLR (B, D) at 20°C and 50% v/v K5 carriers. The theoretical yields (Y) are shown at the top of each panel. The errors bars indicate the standard deviation and are not shown if smaller than symbol size. Student's t-test parameters: bilateral distribution; heteroscedastic t-test; least significant difference: $p < 0.05$; significantly different results are identified with different letters on each panel.

Based on these results, the calculated Y_{obs} are consistent with other experiments as Orantes and González-Martínez (2003) found a maximum Y_{obs} of 0.40 g TSS/g $COD_{removed}$ at 2.8 kg COD m^{-3} d^{-1}. By calculating the ratio of the mass of TSS contained in the extracted sludge from a settler located after an MBBR to the COD removal between MBBR influent and settler clarified effluent, Aygun *et al.* (2008) obtained sludge productions ranging from 0.12 to 0.56 g TSS/g $COD_{removed}$ for an OLR from 1.5 to 24 kg COD m^{-3} d^{-1}. At similar OLR, sludge productions reported by Aygun *et al.* (2008) were in the same range than the Y_{obs} presented in Figure 3B except for their highest OLR condition at 24 kg COD m^{-3} d^{-1} for which Aygun *et al.* (2008) found 0.56 g TSS/g $COD_{removed}$ compared to this study at 0.42 g TSS/g $CS_{COD,removed}$.

3.1.2 MBBR effluent

The VSS to TSS ratio (f_{VT}) in the effluent was between 0.81 to 0.91 g VSS/g TSS for all operating conditions, indicating the predominant volatile fraction due to detached biofilm (Table 5). The particulate COD to VSS ratio (f_{CV}) in the effluent varied between 1.24 to 1.60 g X_{COD}/g VSS. The effluent alkalinity and pH varied between 138 and 151 mg $CaCO_3$/L and between 7.6 and 7.8, respectively.

The DO concentration in the bioreactors was at its lowest for an HRT of 13 minutes and an OLR of 16 kg COD m^{-3} d^{-1} and may have indicated a maximum biofilm activity at this operating condition as the DO concentration is related to the biofilm growth and activity (Schubert *et al.*, 2013) (Table 3). However, this is incoherent with the Y_{obs} results as a maximum Y_{obs} was found for another operating condition at an HRT of 27 minutes and an OLR of 7 kg COD m^{-3} d^{-1} (Figure 3C & D).

It was found that stability was easier to reach for operating conditions with high HRT and low OLR (> 13 minutes and < 16 kg COD m^{-3} d^{-1}). At high OLR stability was hard to maintain and was associated with clogging issues.

3.1.3 MBBR effluent solids

MBBR effluent PSD as a function of HRT is presented in Figure 4A. Results showed bimodal volume PSD from around 5 to 1000 μm with a higher fraction of larger particles. In comparison, the PSD of AS measured by a similar laser diffraction particle size analyzer (MastersizerS, Malvern Instruments Ltd., GBR) revealed a wide and bimodal distribution ranging from around 0.2 to 200 μm (Govoreanu *et al.*, 2004). No clear shift in distribution was observed in PSD with varying HRT (and thus with varying OLR) as confirmed by the volume particle size median *Dv50* (Figure 4B & Table 5).

Previous experiments have shown that particles agglomeration is occurring by increasing HRT between 0.75 to 4 hours (Melin *et al.*, 2005; Åhl *et al.*, 2006; Ødegaard *et al.*, 2010; Karizmeh, 2012). It was later demonstrated that a shift towards smaller particles could occur by independently decreasing HRT and OLR (Karizmeh *et al.* 2014). In the present experiment, HRT was kept low and a contact time lower than 37 minutes did not seem to be enough to allow agglomeration. The shift towards larger particles seemed to begin between 37 and 54 minute HRT. However, agglomeration cannot be confirmed in the absence of intermediate operating conditions.

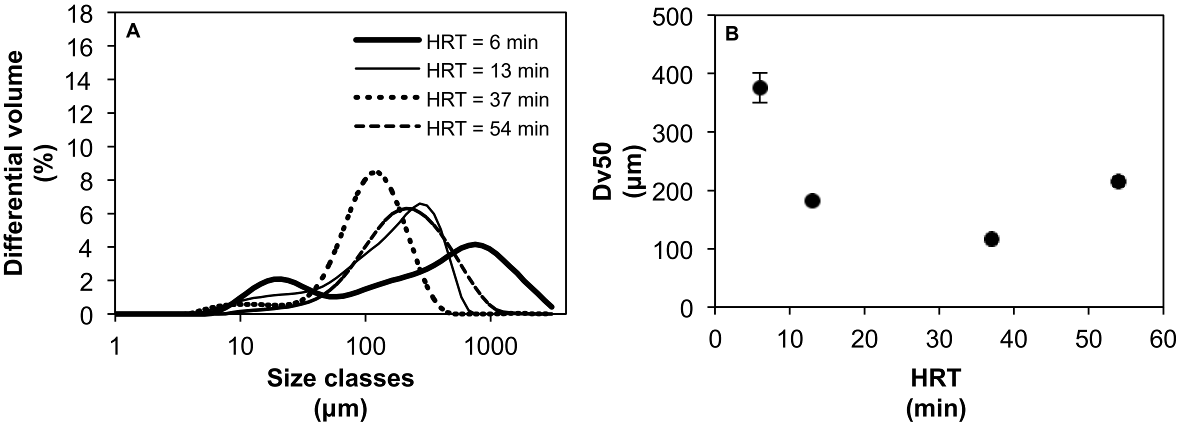

Figure 4. Lab-scale MBBR effluent A) volume differential PSD and B) volume PSD median *Dv50* at 20°C and 50% v/v K5 carriers. The errors bars in B) indicate the standard deviation and are not shown if smaller than symbol size.

Stability was difficult to reach for an HRT < 13 minutes (OLR > 16 kg COD m^{-3} d^{-1}) and these operating conditions were associated with carrier clogging problems. It is plausible that effluent PSD would have been difficult to analyze accurately at these unstable operating conditions. For further PSD analyses it is suggested to first separate components smaller and greater than 1.2 µm to properly study their behaviour. It is also recommended to analyze MBBR effluent PSD for a wider range of HRTs, i.e. between 6 minutes and 4 hours to possibly find the minimum contact time for agglomeration and to compare with previous experiments.

The predominant COD fractions in the MBBR effluent were particulate (> 0.45 µm) and soluble (< 0.04 µm) COD, whereas the colloidal COD fraction (0.04-0.45 µm) was relatively small (Figure 5). This is in accordance with the PSD results showing that 99.9% of the particle volume distribution was above 5 µm. The removal efficiency of total COD, C_{COD} and S_{COD} varied respectively from 19 to 51%, 32 to 86% and 39 to 90%.

The solids settleability using static settling (no chemicals added during jar-tests) was greatly fluctuating and ranged from 35 to 78% (treated effluent TSS concentration from 10 to 23 mg/L) at 37 minute HRT (results not shown). Thus, chemicals addition for coagulation and flocculation appeared necessary to ensure sufficient TSS recovery and reduce the impact of HR-MBBR effluent poor settleability (Ødegaard *et al.*, 2000).

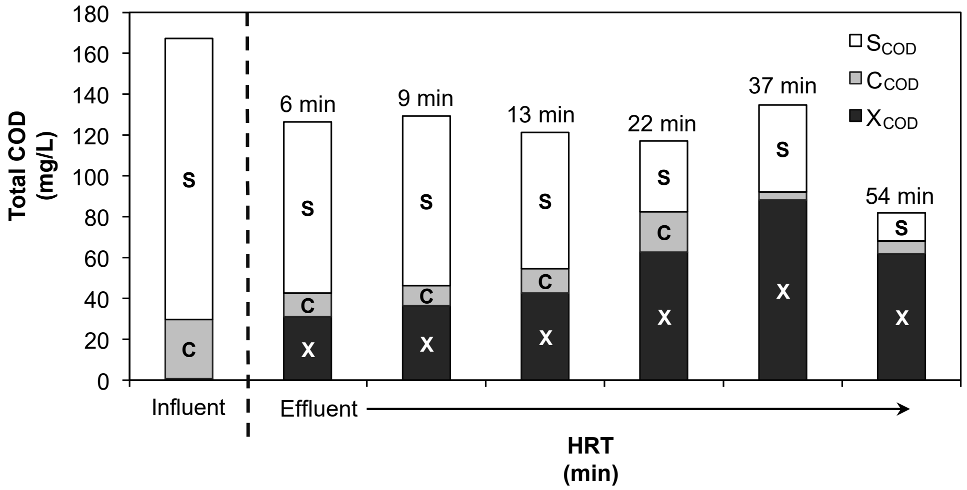

Figure 5. Lab-scale MBBR influent and effluent mean COD fractionation as a function of HRT at 20°C and 50% v/v K5 carriers.

3.1.4 Carrier clogging

High OLR and low HRT conditions (> 16 kg COD m^{-3} d^{-1} and < 13 minutes) were associated with carrier clogging issues. Clogging caused a significant decrease in CS_{COD} removal efficiency of 23% (Table 6). This result is in agreement with the effect of biofilm thickness on substrate diffusion: thinner biofilms facilitate the diffusion of substrate (Ødegaard, 2006). Carrier clogging did not significantly affect the observed yields.

Microscopic observations of the biofilm accumulated on clogged carriers revealed the predominance of filamentous bacteria and rotifers. In addition, monitored parameters in the effluent showed high variations at that time, perhaps due to the instability of the reactors. Airflow rate was maintained constant throughout the different operating conditions and was possibly not sufficient to provide enough biofilm detachment at low HRT. For further lab-scale MBBR experiments at high-rate conditions, it is suggested to control the biofilm thickness by increasing the shear force applied on the biofilm by increasing the airflow rate.

Table 6. Effect of carrier clogging on the performance of the lab-scale MBBR at 20°C and 50% v/v K5 carriers using Student’s t-test

Parameters	Symbol	Units	Unclogged		Clogged		p-value
			Value	n*	Value	n	
HRT	-	min	5.8 ± 0.2	9	6.6 ± 0.9	8	**0.03**
OLR	-	kg COD m^{-3} d^{-1}	34 ± 2	9	30.6 ± 0.6	8	**0.001**
TSS production	-	g/d	5.7 ± 0.8	8	4.8 ± 1.1	7	0.10
VSS production	-	g/d	5.0 ± 0.5	8	4.3 ± 1.0	7	0.14
CS_{COD} removal efficiency	R_{CSCOD}	%	43 ± 1	4	33 ± 5	7	**0.002**
CS_{COD} specific removal rate	SR_{CSCOD}	kg CS_{COD} m^{-3} d^{-1}	15.0 ± 0.4	4	10 ± 2	7	**6E-5**
TSS observed yield	$Y_{obs,TSS}$	g TSS/g $COD_{CS,removed}$	0.42 ± 0.07	6	0.48 ± 0.08	7	0.13
VSS observed yield	$Y_{obs,VSS}$	g VSS/g $COD_{CS,removed}$	0.37 ± 0.06	6	0.43 ± 0.06	7	0.11
X_{COD} observed yield	$Y_{obs,XCOD}$	g X_{COD}/g $COD_{CS,removed}$	0.48 ± 0.05	4	0.59 ± 0.13	7	0.06

*n: number of values

Student’s t-test parameters: bilateral distribution, heteroscedastic t-test, least significant difference: $p < 0.05$. A bold p-value indicates a significant difference.

3.1.5 Temperature effect

Two operating conditions were repeated at 10 and 20°C to study the effect of temperature on the MBBR performance (Table 7).

The temperature coefficient Θ was evaluated by comparing the mean CS_{COD} specific removal rates at 10 and 20°C and using the van't Hoff-Arrhenius law (Rusten *et al.*, 1995):

$$k_{T2} = k_{T1}\,\theta^{(T2-T1)} \tag{7}$$

in this case,

$$SR_{CS_{COD}\,20} = SR_{CS_{COD}\,10}\,\theta^{(20-10)} \tag{8}$$

The temperature coefficients obtained, 1.08 and 1.04 are within the typical range for organic matter removal systems of 1.00 to 1.08 (1.04 for AS) (Metcalf and Eddy Inc., 1991; Kadlec and Reddy, 2001).

The temperature decrease of 10°C was found to cause a significant reduction of 15 to 41% of CS_{COD} removal efficiency and 30 to 52% of specific removal rate.

Table 7. Effect of lab-scale MBBR temperature on their performance at 50% v/v K5 carriers using Student's t-test

Parameters	Symbol	Units	Temperature				p-value	Temperature				p-value
			10°C		20°C			10°C		20°C		
			Value	n*	Value	n		Value	n	Value	n	
HRT		min	13.5 ± 0.1	5	13.3 ± 1.2	9	0.62	23.4 ± 1.2	5	21.6 ± 1.0	7	**0.03**
OLR		kg COD m^{-3} d^{-1}	13.11 ± 0.01	5	15.6 ± 0.5	9	**5E-7**	7.85 ± 0.02	5	9.6 ± 0.3	7	**1E-5**
TSS production	-	g/d	3.2 ± 0.2	3	3.5 ± 0.3	9	0.09	2.3 ± 0.4	5	2.6 ± 0.3	6	0.24
VSS production	-	g/d	2.8 ± 0.2	3	3.1 ± 0.3	7	0.08	2.0 ± 0.4	5	2.2 ± 0.3	6	0.29
CS_{COD} removal efficiency	R_{CSCOD}	%	33 ± 2	5	56 ± 3	6	**5E-8**	56 ± 1	5	66 ± 3	7	**2E-5**
CS_{COD} specific removal rate	SR_{CSCOD}	kg CS_{COD} m^{-3} d^{-1}	4.3 ± 0.3	5	8.9 ± 0.3	6	**1E-9**	4.4 ± 0.1	5	6.3 ± 0.3	7	**4E-7**
TSS observed yield	$Y_{obs,TSS}$	g TSS/ g $COD_{CS,removed}$	0.70 ± 0.04	3	0.42 ± 0.02	7	**0.005**	0.49 ± 0.03	4	0.41 ± 0.04	6	**0.01**
VSS observed yield	$Y_{obs,VSS}$	g VSS/ g $COD_{CS,removed}$	0.61 ± 0.03	3	0.37 ± 0.03	5	**0.0004**	0.41 ± 0.02	4	0.35 ± 0.04	6	**0.02**
X_{COD} observed yield	$Y_{obs,XCOD}$	g X_{COD}/ g $COD_{CS,removed}$	0.80 ± 0.07	3	0.50 ± 0.05	7	**0.01**	0.53 ± 0.03	4	0.54 ± 0.07	6	0.82
Temperature coefficient of SR_{CSCOD}	$\Theta_{SRCSCOD}$	-	1.08				-	1.04				-

*n: number of values

Student's t-test parameters: bilateral distribution, heteroscedastic t-test, least significant difference: $p < 0.05$. A bold p-value indicates a significant difference

3.2 Physico-chemical capture

3.2.1 Unbiodegradable chemicals

When treating the lab-scale MBBR effluent with enhanced flotation, dosages used for alum and PAM polymer were respectively 0.2 mmol Al/L and 1 mg polymer/L.

At 0.2 mmol Al/L and 1 mg polymer/L, the efficiency of several high MW anionic, non-ionic and cationic polymers were compared (Figure 6). All polymers allowed good TSS recoveries, ranging from 90 to 97%, with less than 7 mg TSS/L in the clarified effluent. When using a non-ionic polymer, TSS recovery efficiency was reduced by 7%. Charged polymers resulted in similar efficiencies, i.e. 96-97%. A cationic polymer gave a more constant clarified effluent quality: less than 17% variation in TSS final concentrations compared to 26 to 39% variations for other charged polymers.

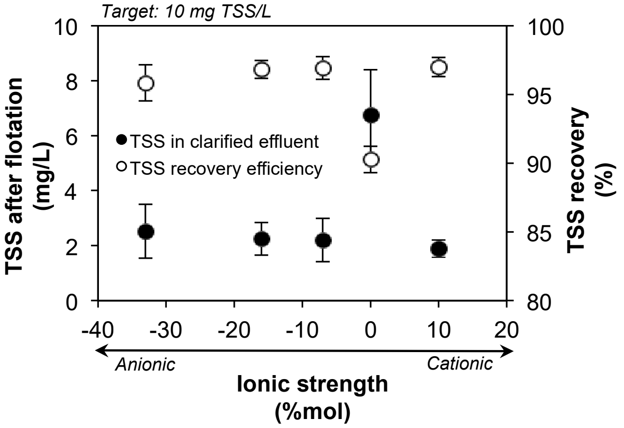

Figure 6. Effect of synthetic polymers ionic strength on TSS recovery efficiency and concentration after enhanced flotation. MBBR: 37 minute HRT, 20°C and 50% v/v K5 carriers. Mean TSS concentration in MBBR effluent: 64 ± 10 mg/L. The errors bars indicate the standard deviation.

These results are in agreement with the ones presented by Stanley and Evans (1977), when treating domestic wastewater with a similar batch flotation process using low-density beads, as they observed higher TSS recoveries for cationic than anionic polymers.

The high efficiency of cationic polymers to treat MBBR effluent with settling or DAF and to reduce coagulant dosage demand and consequently sludge production was demonstrated in previous experiments (Melin *et al.*, 2002; Melin *et al.*, 2004; Ødegaard *et al.*, 2010). It was recommended to use high cationic strength and low to medium MW PAM or polyDADMAC polymers in addition of a metal coagulant (Melin *et al.*, 2002; Ødegaard *et al.*, 2010). In one of these experiments, a multivariate analysis model was compared to experimental data to study the effect of chemical dosage, polymer charge density and MW on TSS recovery efficiency (Melin *et al.*, 2002). It appeared that TSS recovery was mainly influenced by effluent characteristics and chemical dosage and less affected by the polymer nature in the work of Melin *et al.* (2002).

Using alum and a cationic polymer at 0.2 mmol Al/L and 1 mg polymer/L, conventional settling and enhanced flotation achieved similar efficiencies and effluent quality, 96% TSS recovery (3 mg TSS/L) and 97% (2 mg TSS/L), respectively. Alkalinity consumption was higher at a given alum dosage for enhanced flotation than settling (9% and a final alkalinity of 138 mg $CaCO_3$/L versus 4% and a final alkalinity of 149 mg $CaCO_3$/L).

3.2.2 *Green* chemicals

Dosages used for *green* coagulant, Hydrex 3818, and polymer, Hydrex 3842, were respectively 33 mg tannin/L and 20 mg Hydrex 3842/L. Alkalinity consumption was below 5% (final alkalinity above 129 mg $CaCO_3$/L).

When *green* chemicals were added during the enhanced flotation process, flocs and bonds between solids and beads appeared to be more fragile than with the unbiodegradable chemicals. Thus, the enhanced flotation jar-test procedure was modified to allow a complete dispersion of coagulant during the 2 minute coagulation period at 300 rpm and of beads and half of the polymer during 15 seconds before the flocculation period at 300 rpm. The mixing speed was lowered to 50 rpm during the 2 minute flocculation period to allow sufficient bonding. The remaining of the polymer was added at the beginning of the flocculation period at 50 rpm. The duration of the flotation period was set at 2 minutes, but it was found that 95% of the total TSS recovery efficiency was reached after only 30 seconds of flotation (results not shown).

The effect of HRT on enhanced flotation efficiency in terms of TSS concentration and COD fractionation is presented in Figures 7 & 8. The mean TSS concentrations measured in MBBR effluent throughout the seven operating conditions increased with HRT (Figure 7A & Table 5). The mean TSS concentrations measured in MBBR effluent on the days of enhanced flotation experiments are also shown in Figure 7.

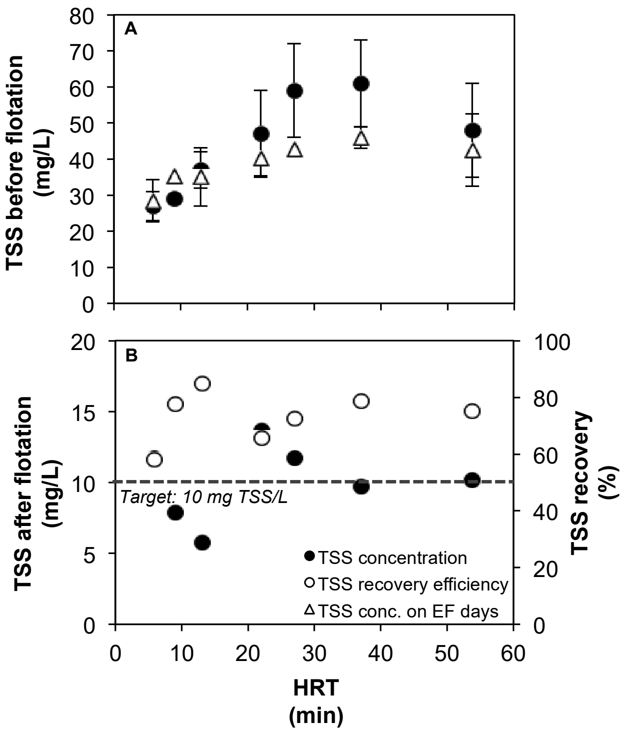

Figure 7. Effect of MBBR HRT on TSS concentration A) in MBBR effluent and B) in clarified effluent after enhanced flotation with *green* chemicals. Mean TSS concentrations in MBBR effluent on enhanced flotation experiment days (EF days) are shown in A). MBBR: 20°C and 50% v/v K5 carriers. The errors bars in A) indicate the standard deviation and are not shown if smaller than symbol size.

Throughout the seven operating conditions, *green* chemicals achieved a mean TSS concentration of 10 ± 3 mg/L in the clarified effluent (Figure 7B). However, the target TSS concentration of 10 mg/L was met for half of the operating conditions. TSS recovery efficiency did not seem to be related to TSS concentration before flotation, as also observed by Melin *et al.* (2004) for a DAF separation process. No tendency was observed between OLR (or, inversely, HRT) and TSS recovery efficiency. The negative effect of high OLR on MBBR effluent solids settleability is caused by an increase of small sized unsettleable particles with increasing OLR. The effect is reduced by chemicals addition, which facilitates the agglomeration of these particles during flocculation (Ødegaard *et al.*, 2000).

The evolution of COD fractionation throughout the treatment train, beginning by MBBR influent and effluent, COD contribution of *green* chemicals and COD remaining in the clarified effluent is presented in Figure 8A. The enhanced flotation process mainly captured X_{COD} fraction (from 68 to 87%) and C_{COD} fraction (from 37 to 56%) when comparing the MBBR effluent after chemicals addition and the clarified effluent.

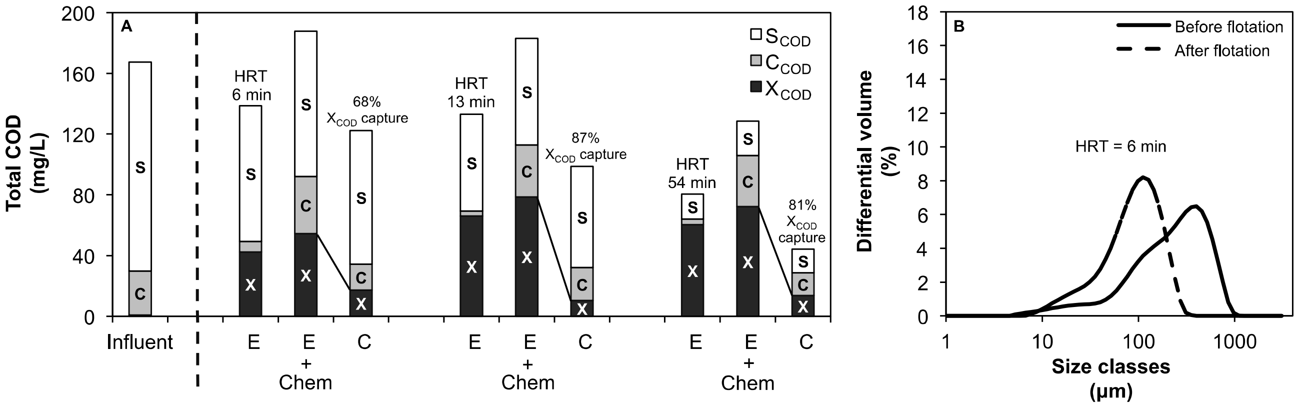

Figure 8. Effect of enhanced flotation combined with *green* chemicals on A) MBBR effluent COD fractionation and B) PSD (HRT = 6 minutes). MBBR: 20°C and 50% v/v K5 carriers. Legend: E: MBBR effluent before enhanced flotation; Chem: COD addition due to *green* chemicals; C: clarified effluent.

The increase of C_{COD} fraction between MBBR effluent (before chemicals addition) and clarified effluent may be due to the chemicals added and remaining in the clarified effluent. However, in previous experiments using a similar tannin-based chemical, no or very few residual tannin concentrations were measured after treatment (Özacar and Şengil, 2003; Sánchez-Martín *et al.*, 2010b). It is therefore recommended for further tests to measure the residual tannin and starch in the clarified effluent to verify if the increase in C_{COD} fraction can be attributed to the addition of these *green* chemicals. In accordance with the present experimentation, Melin *et al.* (2002) demonstrated that DAF combined with an iron coagulant and a polyDADMAC polymer can capture mainly particles larger than 11 µm and that COD fraction < 0.1 µm remains almost unaltered throughout the process and dominates in the clarified effluent.

Volume PSD of MBBR effluent at 6 minute HRT indicated a major decline in particles larger than 200 µm after enhanced flotation process (Figure 8B). However, the effect on smaller sized particles could not be determined with these PSD results as higher volume was associated to larger size particles.

A summary of the jar-test performance for the seven MBBR operating conditions is presented in Table 8.

Table 8. Summary of jar-test performance in terms of TSS concentration and TSS recovery efficiency after enhanced flotation with unbiodegradable or *green* chemicals. MBBR: 20°C and 50% v/v K5 carriers

Parameters	Symbol	Units	OC1	OC2	OC3	OC4	OC5	OC6	OC7
HRT	-	min	5.8 ± 0.2	9 ± 1	13 ± 1	22 ± 1	27 ± 3	37 ± 1	53.7 ± 0.8
OLR	-	kg COD m^{-3} d^{-1}	34 ± 2	24 ± 1	15.6 ± 0.5	9.6 ± 0.3	7.2 ± 0.1	6.1 ± 0.1	4.1 ± 0.1
TSS effluent	X_{TSS_E}	mg/L	27 ± 4	29 ± 2	37 ± 5	47 ± 12	59 ± 13	61 ± 12	48 ± 13
Unbiodegradable chemicals (alum + charged/uncharged PAM polymers)									
TSS after flotation	X_{TSS_C}	mg/L	n.a.*	n.a.	n.a.	n.a.	n.a.	3 ± 2	n.a.
TSS recovery efficiency	R_{TSS}	%	n.a.	n.a.	n.a.	n.a.	n.a.	95 ± 3	n.a.
Green chemicals (Hydrex 3818 + Hydrex 3842)									
TSS after flotation	X_{TSS_C}	mg/L	12 ± 3	8 ± n.a.	6 ± 2	13.7 ± 0.3	12 ± n.a.	9.73 ± 0.05	10.2 ± 0.5
TSS recovery efficiency	R_{TSS}	%	58 ± 10	78 ± n.a.	85 ± 3	66 ± 5	73 ± n.a.	79 ± 1	75 ± 7

*n.a.: not available

Legend: E: MBBR effluent before enhanced flotation; C: clarified effluent.

4 Conclusions

The objectives of this experiment were 1) to optimize MBBR HRT and OLR to maximize biodegradable sludge production and 2) to determine the potential of an innovative enhanced flotation separation process combined with natural-based *green* chemicals. A target TSS concentration of 10 mg/L was set in the separation process clarified effluent. An MBBR with a synthetic soluble feed was operated at seven operating conditions with OLR and HRT varied simultaneously from 4 to 34 kg COD m^{-3} reactor d^{-1} and from 6 to 54 minutes, respectively. The MBBR effluent was used afterwards for jar-test experiments and PSD analyses. The following conclusions were drawn:

- CS_{COD} removal efficiency increased with HRT (and inversely with OLR) up to 27 minutes (OLR of 7 kg COD m^{-3} d^{-1}), from which efficiency was maintained constant at 85%. Efficiency was significantly reduced by clogging issues occurring at high OLR and low HRT conditions (> 16 kg COD m^{-3} d^{-1} and < 13 minutes) and by a 10ºC temperature decrease;
- An MBBR HRT of 22 to 37 minutes (OLR between 6 and 10 kg COD m^{-3} d^{-1}) for a temperature of 20ºC was found best to maximize the biotransformation of CS_{COD} into X_{COD} without MBBR clogging;
- Bimodal PSDs were observed in the MBBR effluent without evidence of agglomeration taking place for HRTs < 37 minutes;
- The innovative enhanced flotation process combined with alum and charged or uncharged polymers allowed to reach a target TSS concentration of 10 mg/L in the MBBR clarified effluent. When *green* chemicals were used, bonds between solids and beads appeared to be more fragile, although they could reach a mean TSS concentration of 10 ± 3 mg/L in the MBBR clarified effluent.

An MBBR followed by an enhanced flotation process is a promising treatment combination that can result in a compact and sustainable process train for wastewater treatment and resource recovery. Further work using real wastewater containing solids with industrial size pilot units would allow to better consider mass transfer and media clogging phenomena and obtain more accurate design criteria to evaluate the sludge production of *green* chemicals and energy consumption of the proposed treatment train.

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