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Dynamic testing of the twin circulating fluidized bed bioreactor (TCFBBR) for nutrient removal from municipal wastewater

Mehran Andalib[1], George Nakhla*, Jesse Zhu[2]

Chemical and Biochemical Engineering Department, The University of Western Ontario, London, ON, N6A 5B8 Canada

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ABSTRACT

The performance of a lab-scale twin circulating fluidized bed bioreactor (TCFBBR) for biological nutrient removal from synthetic and real municipal wastewater was studied with lava rock, 850–1125 μm in diameter, used as a biofilm carrier media. The work showed >90% COD, >85% nitrogen, and 20–55% phosphorus removal efficiencies at an synthetic influent (phase I) and real (phase II) municipal wastewater (MWW) flow rate of 260 L/d, with corresponding organic loading rates (OLR) of 2.7and 4.3 kg COD/(m^3 d) and nitrogen loading rates (NLRs) of 0.3 and 0.51 kg N/(m^3 d). The overall hydraulic retention time (HRT) was 2.3 h with empty bed contact times (EBCTs) of 0.22 and 0.71 h in the anoxic and aerobic columns, respectively. The TCFBBR effluent was characterized by <1.0 mg NH_4-N/L, <5.1 mg NO_3-N/L, <8 mg TN/L, and <11 mg SBOD/L throughout the experiment. Due to a long sludge retention time (SRT) of 40 days, 31–32 days in the anoxic column and 6.8–7.6 days in the aerobic column, very low sludge yields of 0.093 and 0.101 g VSS/g COD were observed. The sustainability of the system, in terms of nitrification–denitrification, tested at a hydraulic peaking factor of 4 for 3 h, demonstrated the high efficiency of the TCFBBR during wet weather conditions. Nitrification was found to be very sensitive to the SCOD concentration, with effluent ammonia concentration increasing from 1.8 to 14 mg/L in 10 h concomitant with a rise in effluent SCOD from 18 to 350 mg/L due to a carbon shock test.

1. Introduction

The need for nutrient removal from wastewater discharges to water sources has become evident in many countries through a generally perceived deterioration of surface water quality. Moreover, stringent provincial and federal regulations for tertiary water quality discharge require nutrient removal from waste streams. Extensive research on the mechanisms of biological nutrient removal (BNR) in suspended growth systems during the last two decades has greatly expanded the integration of BNR into advanced wastewater treatment [1]. However, there have been few comprehensive studies to integrate BNR processes with particulate biofilm processes such as fluidized bed bioreactors [2–4] with and without intermittent feeding and aeration [5–7].

In general, the main reactor types applicable for the suspension of particulate biofilms in wastewater treatment processes are categorized into Anaerobic Upflow Sludge Blanket (UASB), Fluidized Bed Reactors (FBR), Expanded Granular Sludge Blanket (EGSB), Biofilm Airlift Suspension (BAS), and Internal Circulation (IC) reactors [8]. Fluidized bed bioreactors have been investigated for all of the basic secondary and tertiary processes and shown many advantages over other technologies such as conventional suspended growth [9] including: a large specific surface for attached biological growth of 800–1200 m^2/m^3, high biomass concentrations of 8000–12,000 mg/L for nitrification and 30,000–40,000 mg/L for denitrification [8,10,11], long sludge residence times (SRT) and low observed yields which reduce sludge management costs and may result in elimination of secondary clarification requirements [12].

The BNR capability of another form of particulate biofilm reactors (airlifts) has also been studied at the bench scale level for the treatment of municipal wastewater where high BOD and ammonia removal efficiencies were reported [13]. Research on Biofilm Airlift Suspension (BAS) reactors in the late eighties [14,15] led to the concept of CIRCOX® airlift reactor [8]. A CIRCOX® in combination with a denitrifying CIRCOX® reactor achieved effluent nitrogen (<6 mg N/L) in a pilot-plant scale treating municipal wastewater at Zaandam, The Netherlands [16].

The Circulating Fluidized Bed Bioreactor (CFBBR), introduced and developed by Nakhla and his coworkers [4,17–20], was tested for BNR from municipal wastewaters in both lab and pilot scales. The CFBBR consists of an anoxic riser and an aerobic downer with fast and conventional fluidization regimes, respectively. More than 90% organic, 70–80% total nitrogen and 50–70% phosphorous

* Corresponding author. Tel.: +1 519 661 2111x85470; fax: +1 519 850 2921.
E-mail addresses: mandalib@uwo.ca (M. Andalib), gnakhla@eng.uwo.ca (G. Nakhla), zhu@uwo.ca (J. Zhu).
[1] Tel.: +1 519 697 1533.
[2] Tel.: +1 519 661 3807; fax: +1 519 661 3498.

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Nomenclature

Abbreviations

S_0	concentration of initial SCOD in the SDNR batch test (mg/L)
U_d	superficial liquid velocity in the downer (m/s)
U_g	superficial gas velocity in the downer (m/s)
U_r	superficial liquid velocity in the riser (m/s)
X	biomass concentration in the offline SDNR test (mg/L)
X'_r	biomass concentration in the riser (kg VSS/m^3)
X'_d	biomass concentration in the downer (kg VSS/m^3)
X_r	mg VSS/g media in the riser
X_d	mg VSS/g media in the downer
d_m	particle average diameter (m)
f_d	fraction of biomass that remains as cell debris
k_d	biomass decay-rate coefficient (g VSS/g VSS d)
SRT	sludge retention time (d)
Y	biomass true yield (g VSS/g sCOD)
Y_{obs}	biomass observed yield (g VSS/g sCOD)

Greek letters

ρ_{mt}	true particle density (kg/m^3)
ρ_{md}	dry bulk density (kg/m^3)
ψ_t	total porosity of the bare particles (0.62)
ψ_i	internal porosity of the bare particles (0.18)

removal were reported at EBCTs of 0.5–1.5 h, HRTs of 2–3 h, with an observed biomass yield of 0.12–0.16 g VSS/g COD [4,18,19]. Circulation of the media with the biofilm between anaerobic/anoxic and aerobic columns was reported conducive to enhanced biological phosphorus removal (EBPR) in CFBBRs.

While the CFBBR has successfully incorporated fluidized bed systems with BNR, the required height of 5.5 m makes it difficult for retrofitting existing plants. Thus, a new twin fluidized bed system with rectangular cross-sectional area columns, and a height of 3.6 m to facilitate retrofits of existing plants, as well as an anoxic volume of 60% of the aerobic volume was designed, fabricated and tested with synthetic municipal wastewater for 65 days and real municipal wastewater (MWW) for 45 days. Due to the particle transfer through two sloped pipes between the columns, the system was called twin circulating fluidized bed bioreactor (TCFBBR). In contrast to the CFBBR which employs fast fluidization in the anoxic riser to affect particle recirculation, the fluidization regime in the TCFBBR is conventional in both the riser and downer columns. The responses of the system to the dynamic loading conditions and carbon shock tests were also examined to simulate wet weather condition and the effect of organic shock loads on nitrogen removal.

2. Materials and methods

The synthetic municipal wastewater (SMW) was prepared from tap water combined with concentrated stock solution of CH_3COONa (as carbon source), NH_4Cl (as nitrogen source), and KH_2PO_4 (as phosphorus source) as well as a mineral stock solution at a volumetric ratios of 1:0.0025, 1:0.001, 1:0.001 and 1:0.002, respectively. The concentrated stock solutions contained 125 g CH_3COONa/L, 100 g NH_4Cl/L, 20 g KH_2PO_4/L and the mineral salt stock solution contained 75 mg $NiCl{\cdot}6H_2O$/L, 75 mg $CoCl_2{\cdot}6H_2O$/L, 200 mg $CuCl{\cdot}2H_2O$/L, 125 mg Zn Cl_2/L, 1250 mg $MnCl_2{\cdot}4H_2O$/L, 750 mg $FeCl_3{\cdot}6H_2O$/L, 200 mg $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$/L, 125 mg H_3BO_3/L, 40 g Mg $SO_4{\cdot}H_2O$/L and 6 g $CaCl_2{\cdot}H_2O$/L.

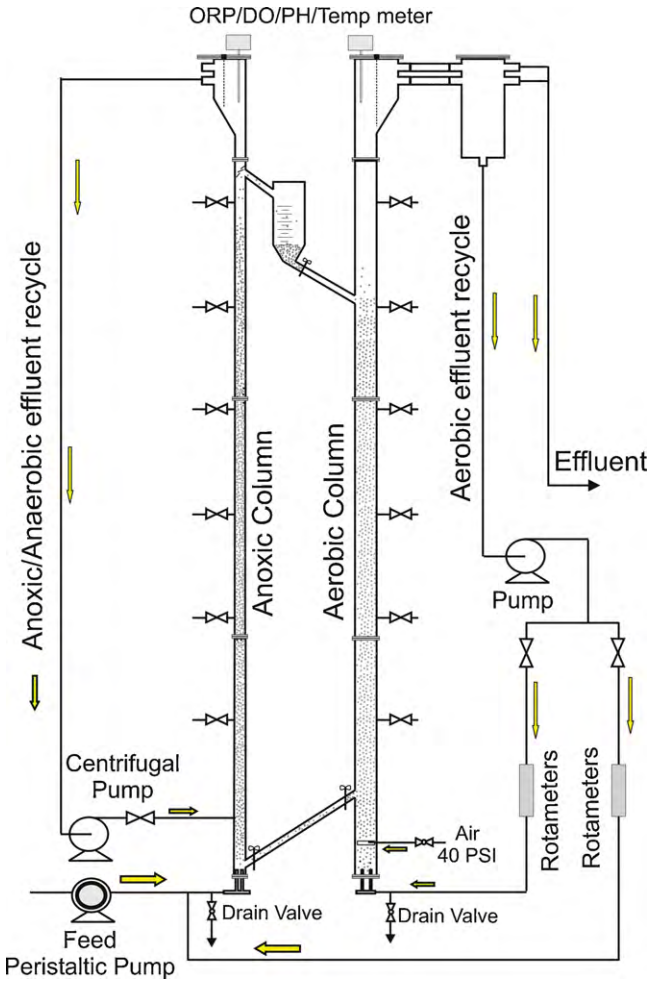

Fig. 1. Schematic of the twin circulating fluidized bed bioreactor.

2.1. System description

The TCFBBR (Fig. 1) is comprised of two plexi-glass columns operated as anoxic and aerobic FBRs with a height of 3.6 m each. The columns were made rectangular (aerobic: 5 cm × 8.5 cm, and anoxic: 5 cm × 5 cm) to investigate the system potential for retrofitting conventional wastewater treatment tanks. Lava rock particles were used in both columns with an average diameter (d_m) of 850–1125 μm, a total porosity (ψ_T) of 62% (44% external and 18% internal), a dry bulk particle density (ρ_{md}) of 1012 kg/m, a true particle density (ρ_{mt}) of 2628 kg/m^3 and a specific surface area determined by BET (Micromeritics ASAP 2010, Micromeritics Co., USA) of 0.48 m^2/g. The design EBCTs were 0.22 h in the anoxic column and 0.71 h in the aerobic column in phases I and II (Table 1a), corresponding to particle masses of 2.5 kg in the riser and 8 kg in the downer which were initially estimated based on the specific nitrification rates (SNRs), specific denitrification rates (SDNRs) and the attached biomass per g media, reported in the literature for the CFBBR, SNR of 0.09–0.14 g NH_4-N/(g VSS d) and SDNR of 0.033–0.243 g NO_x-N/(g VSS d), respectively [4]. In the riser, heterotrophic bacteria grow on the media and the biofilm becomes thicker. At a certain biofilm thickness, depending on the superficial liquid velocity, the biofilm coated particles reach the height where they can be transferred to the downer through the inclined pipe. However, an intermediate graduated container was placed between the two columns, as shown in Fig. 1, to monitor the particle transfer rate. After exposure to the high shear force in the gas–liquid–solid phase in the downer, the biofilm detaches and

Table 1a
Operating conditions.

		Phase I	Phase II
Influent flow, Q_{in} (L/d)		262 ± 8.2	260 ± 5
Organic loading (kg COD/(m^3 d))		2.7 ± 0.8	4.3 ± 0.5
Nitrogen loading (kg N/(m^3 d))		0.3 ± 0.1	0.51 ± 0.06
Phosphorus loading (kg P/m^3 d))		0.032	0.06
R–R recirculation ratio (Q_{r-r}/Q_{in})		10.7 ± 3	9.4 ± 3.1
D–R recirculation ratio (Q_{d-r}/Q_{in})		4.5 ± 2.1	6 ± 2
D–D recirculation ratio (Q_{d-d}/Q_{in})		16.2 ± 4	21 ± 5
EBCT (h) = $V_{compact}/Q_{in}$	Anoxic	0.22	0.22
	Aerobic	0.71	0.71
HRT (h)	Anoxic	0.86	0.87
	Aerobic	1.43	1.44
Air flow (mL min^{-1})	(40 psig)	2060	2150
DO (mg/L)	Aerobic	5.4 ± 0.7	4.3 ± 0.5
	Anoxic	0.2 ± 0.2	0.3 ± 0.1
X (mg VSS/g lava rock)	Anoxic	25.1	29.5
	Aerobic	3.5	4.7
Biomass (g VSS)	Anoxic	113	145
	Aerobic	22.3	28.3
F/*M* ratio (g COD/(g VSS d))		0.58	0.48
Detachment rates (1/d)	Anoxic	0.061	0.086
	Aerobic	0.18	0.2
Superficial liquid velocity, u_l (cm/s)	Anoxic	1.3–1.9	1.3–1.9
	Aerobic	1.1–1.5	1.1–1.5
Estimated SRT (d)	Anoxic	32[a]	31
	Aerobic	7.6	6.8
	Overall	39.6[b]	37.4
Run time (d)		65	45

[a] Based on Eq. (1).
[b] Based on Eq. (2).

leaves the system along with the effluent. Particles from the bottom dense phase of the downer with a thin biofilm (<40 μm) are transferred back to the riser manually to make up the particles in the riser. Particle transfer cycles were observed to occur every 17 days.

Table 1a displays the detailed design parameters and operational conditions of the TCFBBR. The feed solution was pumped into the bottom of the anoxic column by a peristaltic pump (Masterflex I/P, Masterflex AG, Germany). To ensure fluidization, riser to riser recirculation flows to feed ratios of 9.4:1–10.7:1 and downer to downer recirculation flows to feed ratios of 16:1–21:1 were provided. Biomass was wasted at the equivalent of 1.2 g VSS/d and 2.1 g VSS/d in phases I and II, respectively (Table 1b). All recirculation flows were maintained using two centrifugal pumps (IWAKI MD-40RT-115NL, IWAKI CO., Ltd. Japan) and monitored by rotameters (OMEGA FL-812 and OMEGA FL-5331G, Omega Engi-

Table 1b
Influent and effluent characteristics in phases I and II.

	Phase I (synthetic)			Phase II (municipal)		
	Feed	Riser	Eff.	Feed	Riser	Eff.
PH		7.5 ± 0.3	7.4 ± 0.2		8 ± 0.1	7.7 ± 0.3
ORP (mV)		−88 ± 38	38 ± 41		−21 ± 60	81 ± 37
TCOD (mg/L)	278 ± 31	60 ± 18	31 ± 16	398 ± 52	101 ± 40	50 ± 21
SCOD (mg/L)	252 ± 35	27 ± 14	14 ± 4	118 ± 24	31 ± 8	22 ± 5
SBOD(mg/L)	189 ± 26	20 ± 10	9 ± 5	72 ± 14	18 ± 4	11 ± 3
TN (mg/L)	31 ± 3.1	6.7 ± 1.2	5.4 ± 1.3	48 ± 5.8	11.4 ± 4	8 ± 1.6
STN (mg/L)	29.6 ± 3	4.6 ± 1.2	3.9 ± 0.8	31 ± 5	7.6 ± 2.3	6.1 ± 2.1
NH_3-N (mg/L)	29.1 ± 3	4.1 ± 1.1	0.7 ± 0.4	30 ± 4.5	4.1 ± 0.4	0.9 ± 0.4
NO_3-N (mg/L)	0.5 ± 0.2	0.5 ± 0.2	2.6 ± 0.5	0.8 ± 0.3	3.2 ± 1.9	5.1 ± 1.6
NO_2-N (mg/L)	0.01	0.01	0.6 ± 0.5	0.03	0.3 ± 0.2	0.1 ± 0.1
Alkalinity[a]				250 ± 10	160 ± 15	135 ± 20
TP (mg/L)	3.1 ± 0.3		2.4 ± 0.4	6.5 ± 1.4		3.2 ± 0.6
PO_4-P (mg/L)	2.9 ± 0.3	2.4 ± 0.3	2.3 ± 0.3	3.4 ± 0.7	3 ± 0.5	3 ± 0.5
TSS (mg/L)	18 ± 6	35 ± 17	26 ± 14	214 ± 41	62 ± 30	33 ± 14
VSS (mg/L)	13 ± 5	28 ± 12	16 ± 10	183 ± 30	50 ± 27	24 ± 10
Biomass Wastage (g VSS/d)			1.2			2.1
C:N:P	9:1:0.1			8:1:0.12		

[a] As mg $CaCO_3$ equivalent/L.

neering, Inc., Canada). Superficial liquid velocities of 1.3–1.9 cm/s and 1.1–1.5 cm/s were maintained in the anoxic and the aerobic columns. Eight manometers (four along each column) were employed to observe the pressure drop along each column. Air, at 40 psi, was injected at the bottom of the aerobic column using a perforated tube. Air flow was monitored around 2.1 L/min by an air flow meter, OMEGA, FL-3696 ST.

2.2. Acclimatization and start-up

A similar approach to CFBBR [4] start-up was undertaken to seed the TCFBBR with enriched nitrifiers, acclimatized in the lab using 15 L of returned activated sludge (RAS) from the Adelaide Pollution Control Plant, London, Canada, with TSS and VSS concentrations of approximately 3500 and 2800 mg/L, respectively. Meanwhile, the clean media was fluidized in the columns at $U_d = U_r = 1.1$ cm/s. The seed was pumped into the system and recirculated between the two columns for 2 days to transport and trap the bacteria from the bulk liquid on the media surface and the pores. Thereafter, the continuous synthetic feed was initiated at a flow rate of 260 L/d corresponding to OLR and NLR of 2.7 kg COD/(m^3 d) and 0.3 kg N/(m^3 d). Within a period of 2 weeks, most of the particles in both columns were coated with biomass with average concentrations of 5 and 28 mg VSS/g media in the aerobic and anoxic columns, respectively.

2.3. Analytical methods

Samples from the feed tank, top of the anoxic column, and the final effluent were collected and refrigerated at 4 °C prior to analysis. Total suspended solids (TSS), volatile suspended solids (VSS) and 5-day biochemical oxygen demand (BOD) were analyzed in accordance with Standard Methods 2540D, 2540E and 5210, respectively [21]. HACH methods and testing kits (HACH Odyssey DR/2800) were used to analyze total and soluble chemical oxygen demand (TCOD and SCOD), total and soluble nitrogen (TN and STN), total phosphorus (TP), NH_4-N, NO_2-N, NO_3-N, and PO_4. Alkalinity was measured by titration with 0.01N H_2SO_4 in accordance with the Standard Method no 2320 [21]. DO and ORP were measured onsite using an Oakton DO 6 meter, and an Oakton ORPTestr 10 (Oakton, Singapore). The size of the bare and biofilm coated particles was measured using Visiongauge (Flexbar Machine Co, New York, USA) synchronized to a microscope (Mitutoya, Sakada, Japan) coupled with a camera (Leica DC 300, Germany), at a magnification of 50×. Based on Standard Method no 2540G (APHA, 1998), the attached biomass on the carrier media was measured and expressed as mg VSS/g clean particles. Approximately 10–20 g bio-particles were taken from each of the two columns, suspended in a 100 mL vials, and sonicated for 3 h at 30 °C in an Aquasonic sonicator (SK 1200H Kupos, China) with a rated power of 45 watts. After sonication, the TSS and VSS content of the detached biomass was determined following Standard Methods no 2540D and 2540E [21].

2.4. Batch tests

Batch tests were carried out to test the maximum SNR and SDNR of the attached biomass in the system following the methods previously used for the CFBBR. Batch reactors (0.5 L working volume) equipped with magnetic stirrers were used for nitrification by injecting air and alkalinity or for denitrification by avoiding intrusion of air and injecting SCOD. To reduce the effect of substrate mass transfer limitation into the biofilm, the biofilm was removed from 10 to 40 g media using sonication and then placed into the reactors. The biomass in the SDNR and the SNR tests were in the range of 1500–4000 mg VSS/L and 240–500 mg VSS/L, respectively, considering the amounts of biofilm in the anoxic and aerobic column of 25–50 mg VSS/g media and 4–6 mg VSS/g media, respectively. The initial acetate COD in the denitrification batch tests was set at 350–450 mg/L while the initial alkalinity used in the nitrification test was 250–350 mg/L as $CaCO_3$. For the SNR tests, the initial ammonia concentrations were 35–55 mg/L, added as ammonium chloride.

2.5. Dynamic hydraulic and carbon shock tests

The impact of dynamic loadings on nutrient removal efficiency of the TCFBBR was tested at different influent flows. While maintaining the same organic and nitrogen loading rates of 4.1 kg COD/m^3 d and 0.39 kg N/m^3 d, respectively, the hydraulic loading was gradually increased by adding clean tap water from 260 to 520 L/d and eventually to 1040 L/d at 3 h intervals, corresponding to hydraulic retention times (HRTs) of 2.3, 1.16, and 0.58 h, respectively, in the hydraulic loading test, and subsequently decreased to 520 and 260 L/d at the same intervals. All of the operational conditions were maintained the same during the test.

In order to also test the sensitivity of system nitrification and carbon removal capabilities to organic shock loads, the influent COD was increased from 420 to 740 mg/L and 1200 mg/L in intervals of 3 h while maintaining overall HRT of 2.3 h. Samples from the effluent top of the riser were taken every 0.5 h for measurement of water quality parameters.

3. Results and discussions

3.1. Nutrient removal

In order to ensure attainment of the steady-state conditions in the system, the suspended and attached biomass in the aerobic and anoxic columns are measured and depicted in Fig. 2a and b , respectively. As noticed from the data in the figure, the coefficient of variation (COV) for attached biomass in the aerobic and anoxic columns in phase II are 8.9% and 4.8%, respectively. Although it is arguable that suspended VSS concentrations varied more widely, as reflected by COV of 26.4% and 20.7%, this process is indeed a fixed-film system and 99.99% of the biomass inventory in the system is in the form of attached biomass. Moreover the nitrification and denitrification activity per gram media depicted in Fig. 2a and b, respectively, demonstrates that the SNR and SDNR coefficients of variation in Phase II are 5.7% and 7.3%. Therefore, the attached biomass and biomass activity reached steady-state.

The system was tested at an average flow rate of 260 L/d with synthetic and real municipal wastewater for 65 and 45 days denoted henceforth as phases I and II, respectively. Figs. 3a–c and 4a–c show the performance of the TCFBBR with respect to chemical oxygen demand (COD), suspended solids (SS), nitrogen (N) and phosphorus (P) removal efficiencies. Fig. 3c also shows the VSS to TSS ratio of 0.847 for the detached biomass which is slightly higher than the conventional suspended biomass [1].

As illustrated in Table 1b and Fig. 3a and b, TCOD removal efficiencies of 90% and 87% at a total empty bed contact time (EBCTs) of 0.93 h and organic loading rates (OLR) of 2.7 ± 0.8 and 4.3 ± 0.5 kg COD/(m^3 d) were observed in phases I and II, respectively. Based on the soluble effluent organic matter, COD removal efficiency would be >96% in phases I and II. The effluent SBOD during both phases was <11 mg/L despite operation at an HRT of 2.3 h. Even though the influent TSS was relatively high in phase II at 214 ± 40 mg/L, an average effluent TSS of 33 mg/L was achieved, corresponding to suspended solids removal efficiency of 86%, without using a clarifier or filter (Fig. 3c). As shown in Table 1a, biomass first-order detachment rate coefficients calculated, based on Patel et al. equation, were 0.06–0.08 1/d in the anoxic column and 0.18–0.2 1/d in

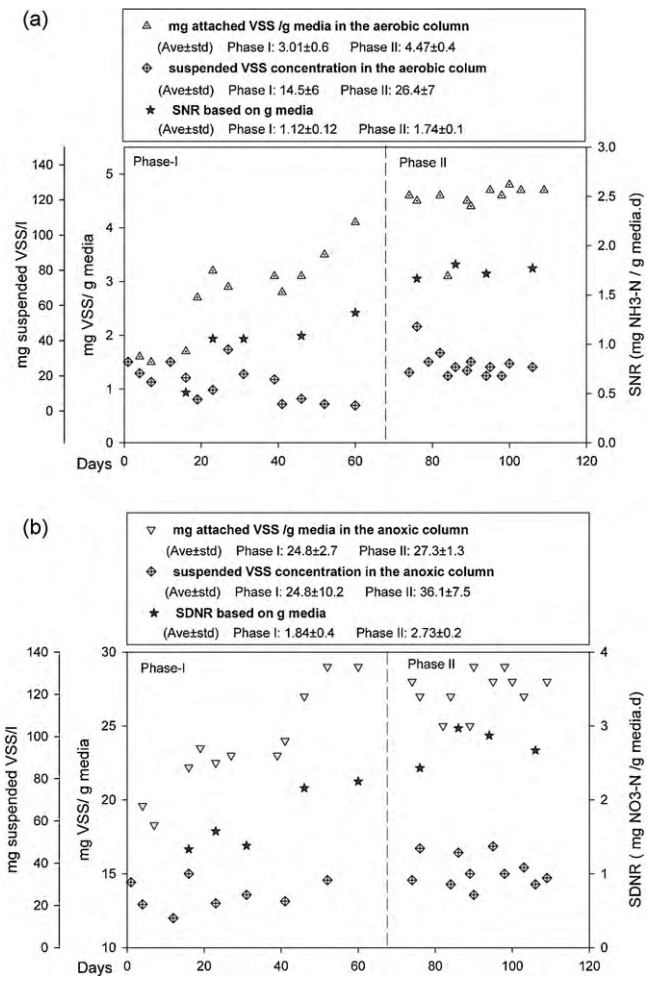

Fig. 2. (a) Trend of attached and suspended biomass and specific nitrification rate in the aerobic column. (b) Trend of attached and suspended biomass and specific denitrification rate in the anoxic column.

the aerobic column [22]. The observed biomass detachment rate for the anoxic column was lower than the CFBBR of 0.13–0.17 1/d whereas the detachment rates of CFBBR and TFBBR aerobic columns were comparable [22,24].

As shown in Table 1b and Fig. 4a, at nitrogen loading rates (NLR) of 0.3 and 0.51 kg N/(m^3 d) in phases I and II, respectively, the system achieved 84.5 ± 1.3% TN removal in phases I and II with STN <4 mg/L in phase I and STN<6.1 mg/L in phase II, which met the tertiary standard limit of 10 mg/L [23]. Effluent TN during phases I and II averaged 5.4 and 8 mg/L, respectively. Nitrification predominantly occurred in the downer with dissolved oxygen (DO) in the range of 4.3–5.2 mg/L. Fig. 4b depicts the influent and effluent NH_3-N, effluent NO_3-N and effluent NO_2-N. As illustrated in Fig. 4b, the effluent NH_3-N was <0.9 mg/L throughout phases I and II with average influent NH_3-N concentration of 30 mg/L. The nitrification rate based on the weight of the media were calculated 1.03 mg NH_3-N/(g media d) and 1.51 mg NH_3-N/(g media d) in phases I and II, respectively. To measure the maximum nitrification rate of the biomass, batch tests were conducted which resulted in SNR based on the media weight of 1.12 and 1.74 mg NH_3-N/(g media d) in phases I and II (Fig. 2a). It is noteworthy that the aerobic biofilm thickness of <50 μm did not hinder diffusion significantly, thus rationalizing the relative agreement (6–11% discrepancy) between in-line and offline SNRs in phases I and II. The produced nitrate in the downer was recycled to the riser (anoxic column) with a recirculation flow to the feed flow ratio of 4.5–6. At an empty bed contact time of 0.22 h, effluent NO_x-N concentrations of 3.2 and 5.2 mg/L were observed in phases I and II with nitrite concentrations of 0.1–0.6 mg/L. The denitrified-nitrogen loading rate based on the anoxic column volume was 0.70 kg N/(m^3 d) in phase I and 1.19 kg N/(m^3 d) in phase II, corresponding to the biomass specific denitrification rate based on media weight of 1.47 and 2.27 mg NO_3-N/(g media d) in phases I and II, respectively. The aforementioned SDNR of TCFBBR are within 20% of offline biomass maximum denitrification rates of 1.84 and 2.73 mg NO_3-N/(g media d) in phases I and II (Fig. 2b), at S_0/X ratio of 0.3–0.4 g COD/g VSS. It must be asserted that the batch test results show the maximum nitrification and denitrification capacity of the system and may not reflect exactly the TCFBBR rates due to mass transfer limitation. As a result, the 20% difference in the online and

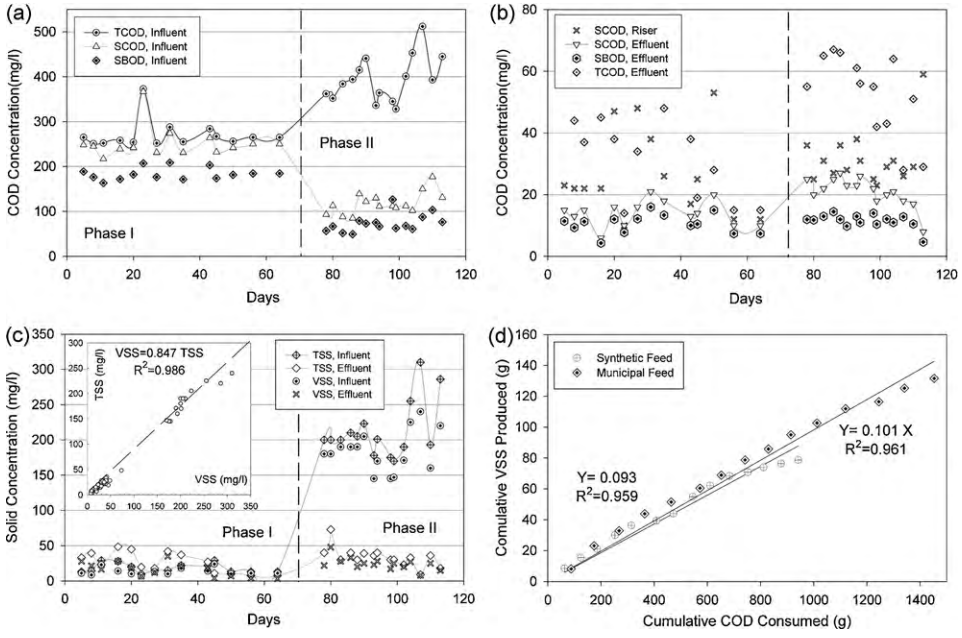

Fig. 3. (a) TCOD, SCOD and BOD in the influent. (b) COD and BOD concentrations in the riser and downer. (c) Suspended solids removal in the system. (d) Sludge yield during phases I and II.

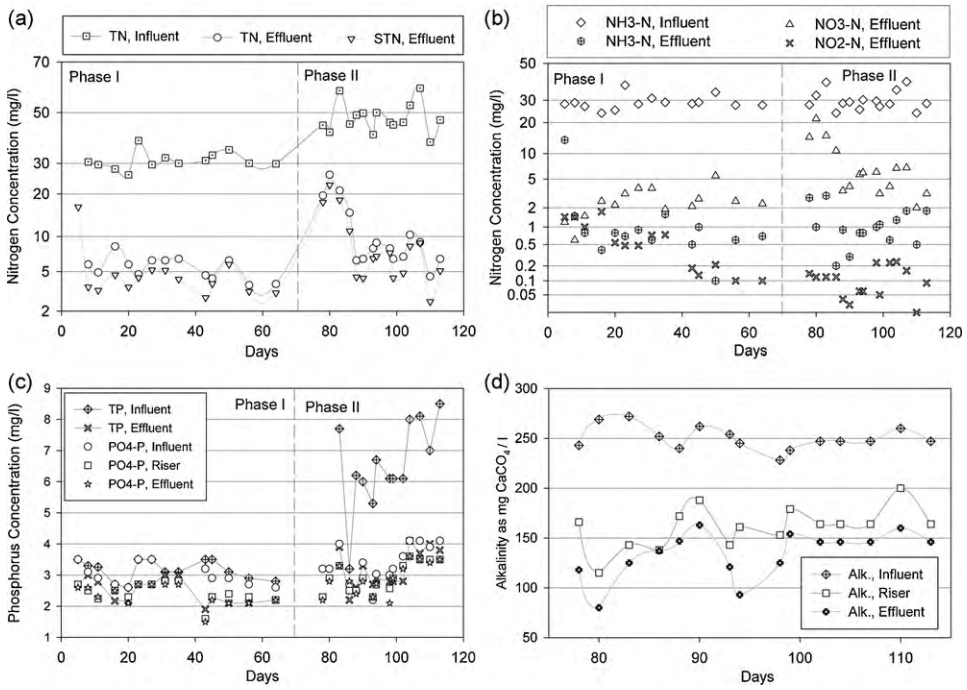

Fig. 4. (a) Total nitrogen removal during the two phases. (b) Ammonia, nitrate and nitrite concentrations in the influent and effluent. (c) Phosphorus removal (total and ortho-phosphate). (d) Alkalinity concentrations in the influent, riser and effluent.

offline denitrification rates is due to the nitrate diffusion limitation in the anoxic biofilm with up to 400 μm thickness.

Total and ortho-phosphorus (OP) removals in phases I and II are shown in Fig. 4c. Approximately, 18 ± 7% and 55 ± 8% phosphorus removal was observed in phases I and II at phosphorus loading rates of 0.032 and 0.06 kg P/(m^3 d), respectively. As apparent from Fig. 4c, OP release in the riser, as the phosphorus accumulating microorganisms (PAOs) activity indicator, was insignificant, at 0.1–0.13 g/d (as shown in Table 3) throughout the tests. Phosphorus content of the effluent biomass was measured as 1.8 ± 0.5% by weight of VSS, which is similar to the conventional sludge phosphorus content of 1–2%. In general, phosphorus removal in the TCFBBR occurred through biomass synthesis, and precipitation. Fig. 4d depicts the concentrations of alkalinity in phase II in the riser and effluent as mg $CaCO_3$/L which shows 100–120 mg $CaCO_3$/L overall consumption of alkalinity through two stages of nitrification denitrification.

3.2. Biomass yield

Fig. 3d illustrates the linear regression of cumulative VSS produced, based on the sum of the effluent biomass, the net change in attached biomass and biomass wasted, versus cumulative COD removed. A very low observed yield of 0.093 g VSS/g COD was observed in phase I with an average effluent VSS concentration of 15 mg/L. Although there was a 38% increase in the OLR in phase II to 4.3 kg COD/(m^3 d), the observed yield increased marginally to 0.101 g VSS/g COD, a 7.8% increase compared to phase I. As shown in Table 1a, overall sludge retention time (SRT) of 37.8–39.6 days were calculated based on equations (1) and (2), with anoxic SRTs of 31–32 days.

The long SRT and also up to 54.4–62.7% influent COD consumption in the anoxic column (as shown in Table 3) rationalize the reduced yield in the TCFBBR. The detailed calculations to justify the experimental observed yields are described in Appendix A. The observed sludge yield of 0.093–0.1 in the TCFBBR is 30% lower than the CFBBR.

$$\mathrm{SRT_{Total}} = \frac{M_d X_d + M_r X_r}{Q_{eff} VSS_{eff} + X_{wast}} \quad (1)$$

$$\mathrm{SRT_{ano}} = \mathrm{SRT_{Total}} \frac{M_r X_r}{M_d X_d + M_r X_r} \quad (2)$$

3.3. Loading tests

At the end of the experiment with the real municipal wastewater (phase II), the loading tests including the dynamic loading test as well as the organic shock tests were conducted.

3.3.1. Dynamic hydraulic test

The impact of the dynamic loading on the TCFBBR effluent quality and its nutrient removal efficiencies were monitored by simulating wet weather condition at a maximum peaking factor of 4 for 3 h. The hydraulic loading was gradually increased by the addition of clean tap water from 260 to 520 L/d for 3 h and reached a maximum of 1040 L/d while maintaining all initial recirculation flows at their steady-state rates, translating to overall hydraulic retention times (HRTs) of 2.3, 1.1, and 0.57 h, respectively. Although the nutrient loading during the hydraulic loading test was not increased, the overall hydraulic retention time decreased to 1.2 h and 0.6 h which is equivalent to 0.75 h and 0.37 h retention time in the aerobic zone. The main purpose of this dynamic test was to test whether nitrogen removal and specifically nitrification would be hindered at a very low retention time since the biological reaction rates are kinetically limited.

The characteristics of the riser effluent and final effluent are shown in Table 2. As shown in Table 2 and Fig. 5a–c, the effluent concentrations were <0.9 mg NH_3-N/L, <3 mg NO_3-N/L, <25 mg SCOD/L, <3 mg PO_4-P/L, <16 mg VSS/L and <20 mg TSS/L after 12 h of the dynamic loading which emphasizes the favourable response of the TCFBBR to the dynamic loadings and the sustainability of performance without loss of nutrient removal capacity and biomass. The system did not show any significant deterioration in terms of nitrification, and denitrification during the test, which was confirmed

Table 2
Influent and effluent characteristics during dynamic loading tests at different phases D_0 (260 L/d), D_1 (520 L/d) and D_2 (1040 L/d).

Parameter	Influent	Phase D_0		Phase D_1		Phase D_2	
		Riser	Effluent	Riser	Effluent	Riser	Effluent
DO (mg/L)		0.38	4.8	0.35 ± 0.1	5 ± 0.2	0.2 ± 0.0	5.5 ± 0.4
ORP (mV)		−95	4	−103 ± 12	17 ± 12	−85 ± 28	47 ± 10
Alkalinity[a]	270	243	161	213 ± 33	175 ± 14	165 ± 30	144 ± 30
TCOD (mg/L)	393	65	51	53 ± 7	44 ± 10	30 ± 6	31 ± 4
SCOD (mg/L)	177	29	18	25 ± 11	21 ± 3	11 ± 5	18 ± 4
NH_4-N (mg/L)	24.1	2.7	0.6	2.1 ± 0.5	0.6 ± 0.4	1.4 ± 0.2	0.2 ± 0.1
NO_3-N (mg/L)	0.2	0.4	2	0.3 ± 0.1	1.7 ± 0.3	0.2 ± 0.0	0.5 ± 0.1
NO_2-N (mg/L)	0.0	0.0	0.0	0.0	0.01 ± 0.0	0.0	0.01 ± 0.0
TN (mg/L)	37		4.8		3.7 ± 1.1		2.6 ± 0.2
PO_4-P (mg/L)	3.9	3.5	3.4	2.9 ± 0.3	3.0 ± 0.3	1.7 ± 0.6	2.0 ± 0.3
TP (mg/L)	7		3.9		3.3 ± 0.4		2.2 ± 0.3
TSS (mg/L)	193	38	36	35 ± 6	20 ± 3	19 ± 5	11 ± 2
VSS (mg/L)	160	30	25	26 ± 6	16 ± 6	17 ± 2	10 ± 2

[a] As mg $CaCO_3$ equivalent/L.

by the batch specific nitrification (SNR) and denitrification (SDNR) tests, shown in Fig. 5b. Table 3 shows the COD, nitrogen and phosphorus mass removal rates in phases D_1 (520 L/d) and D_2 (1040 L/d). TCOD removal of 78% and 71.6% as well as nitrogen removal of 75.8% and 70.8% were observed in phases D_1 and D_2, respectively which indicated a deterioration of COD and nitrogen removal rates by 10% and 11–15%, respectively, compared to the steady-state system operation prior to the loading test. After 10 h into the dynamic load, the SNR decreased from 0.31 g NH_3N/(g VSS d) to 0.26 g NH_3N/(g VSS d) while SDNR decreased from 0.05 g NO_3-N/(g VSS d) to 0.04 g NO_3-N/(g VSS d). The batch tests also indicated 13% and 20% reduction in the activity of the nitrifiers and denitrifiers, respectively, relative to the steady-state values before the dynamic tests. Table 3 also shows that phosphorus mass removal significantly decreased from 50% at the beginning of the test to 7% in phase D_1. Interestingly, the effluent soluble phosphorus in phase D_2 was higher than the influent which is attributed to the dissolution of the precipitated phosphorus on the media as a result of the high flow rate and relatively low alkalinity in the diluted wastewater, potentially decreasing pH and solubilising metal phosphates.

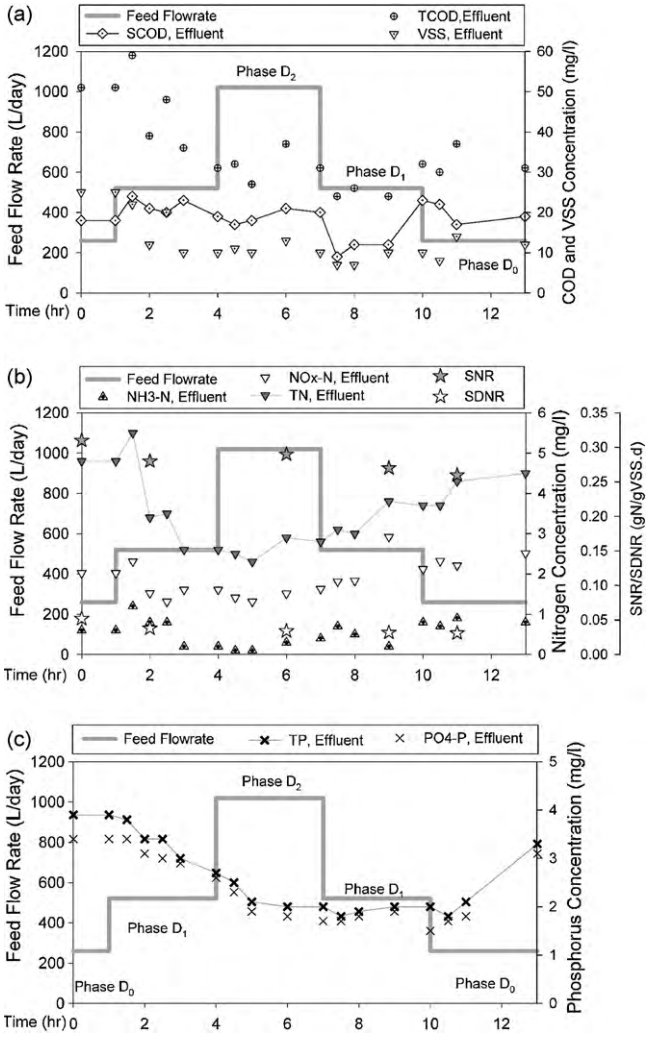

Fig. 5. (a) Dynamic test effect on the effluent COD and VSS. (b) Dynamic loading test effect on the effluent nitrogen. (c) Dynamic loading test effect on the effluent phosphorus.

3.3.2. Organic shock tests

The sensitivity of the system performance in general, and nitrification in particular to organic shocks was tested. Using sodium acetate, the COD of the influent was increased from 420 to 720 mg/L for 4.5 h and then to 1200 mg/L for 4 h corresponding to an ultimate OLR of 13.2 kg COD/(m^3 d). Theoretically, in attached growth systems used for nitrification, most of the BOD must be removed before nitrifying organisms can be established. The heterotrophic bacteria have a higher biomass yield and thus can dominate the surface area of fixed-film systems over nitrifying bacteria [1]. Since the duration of each of the two carbon shock tests was about 2 turnovers of the mean system HRT, it is estimated based on the completely mixed flow regime that about 87% of the reactor contents would have been displaced at every carbon shock loading. Therefore, it must be asserted that the observed impacts represent short-term effects. As shown in Fig. 6a and b, the COD removal efficiency dropped from 93.4% to 64.1% with the effluent SCOD increasing from 18 mg/L prior to the test to as high as 350 mg/L while effluent NH_3-N rose from 1.8 to 14 mg/L after 9 h. It is interesting to note from Fig. 6a and b that the jump in both effluent SCOD and ammonia concentrations started simultaneously at t = 1.8 h. As expected, nitrification efficiency in the downer was hindered to 49% from the initial 95% due to dominance of heterotrophs at the outside of the biofilm as well as DO limitations. The concentrations of DO in the riser and downer at the beginning of the test were 0.3 and 4.9 mg/L, respectively, but decreased to 0.0 and 2.5 mg/L after 9 h. Fig. 6b also depicts the results of offline SNRs test on the decanted aerobic biomass during the carbon shock test with DO of 8 mg/L and SCOD of 30 mg/L. The average SNR after 10 h of carbon shock testing was 0.26 g NH_3-N/(g VSS d), 15% lower than prior to the test. Since the SNR is reflective of nitrifiers activity, it is apparent that 15% of the nitrifying population prior test was evidently washed out during the dynamic carbon shock testing. Based on the nitrifying growth rate equation

Table 3
Nutrient balances in phases I, II, D_1 and D_2.

	Mass in influent (g d^{-1})	Mass consumed (g d^{-1})	Mass Utilized (g d^{-1})	Mass in effluent (g d^{-1})	Mass wastage (g d^{-1})	Percent closure (%)
		Anoxic	Aerobic			
Phase I-synthetic WW (260 L/d)						
TCOD	69.8 ± 2.5			6 ± 1.4	0.9[a] ± 0.01	98.0[b]
(sCOD)	64.6 ± 1.1	38.6 ± 3.5 (17.8)[c] (17.5)[d]	22.9 ± 3.1	2.7 ± 1.0		
TN	7.8 ± 0.6			1.45 ± 0.3	0.12[e] ± 0.0	90.6[f]
NH_4-N	7.6 ± 0.6	0.9 ± 0.6 (0.17)[g]	5.6 ± 0.9	0.15 ± 0.8		
NO_3-N	0.18 ± 0.03	5.4 ± 0.3	−5.5 ± 0.2	0.78 ± 0.3		
NO_2-N	0.0 ± 0.0	0.15 ± 0.02	−0.18 ± 0.03	0.04 ± 0.01		
TP	0.8 ± 0.1			0.66 ± 0.1	0.021[h] ± 0.01	92.7[i]
PO_4-P	0.74 ± 0.04	−0.1 ± 0.1	2.5 ± 0.7	0.5 ± 0.02		
Phase II-municipal WW (260 L/d)						
TCOD	108.8 ± 10			12.1 ± 3.4	2.48[a] ± 0.2	97.8[b]
(sCOD)	33.5 ± 6.9	50.1 ± 9.3 (26.6)[c] (18.5)[d]	42 ± 11.2	5.0 ± 0.5		
TN	11.4 ± 2.1			1.87 ± 0.5	0.21[e] ± 0.05	89.6[f]
NH_4-N	7.8 ± 1.4	1.2 ± 0.08 (0.26)[g]	8.0 ± 1.1	0.3 ± 0.1		
NO_3-N	0.15 ± 0.07	7.6 ± 0.4	−7.7 ± 0.35	1.1 ± 0.5		
NO_2-N	0.0 ± 0.0	−0.34 ± 0.3	0.3 ± 0.3	0.03 ± 0.02		
TP	1.89 ± 0.3			0.85 ± 0.1	0.034[h] ± 0.01	50.1[i]
PO_4-P	1.0 ± 0.04	−0.13 ± 0.03	2.4 ± 0.6	0.78 ± 0.03	0.8[j]	89.1[k]
Alkalinity	64 ± 1.4	−26.8[l] ± 1.3	55.4[m] ± 8	44 ± 3.7		93.3[n]
Phase D_1-dynamic loading test (520 L/d)						
TCOD	102.4			22.1 ± 3.3	2.2[a] ± 0.1	92.2[b]
(sCOD)	46.3	37.5 ± 2.2 (18.1)[c] (17.5)[d]	32.4 ± 4.4	10.4 ± 1.5		
TN	9.26			2.24 ± 0.4	0.22[e] ± 0.0	84.9[f]
NH_4-N	6.5	0.8 ± 0.08	5.6 ± 1	0.3 ± 16		
NO_3-N	0.05	5.4 ± 0.31	−5.5 ± 0.14	0.8 ± 0.07		
NO_2-N	0.0	0.018 ± 0.0	−0.02 ± 0.0	0.0 ± 0.0		
TP	1.82			1.7 ± 0.06	0.04[h] ± 0.0	95.6[i]
PO_4-P	1.04	−0.2 ± 0.2	0.57 ± 0.6	1.5 ± 0.1		
Phase D_2-dynamic loading test (1040 L/d)						
TCOD	102.4			29.3 ± 4.9	2.5[a] ± 0.1	97.9[b]
(sCOD)	46.3	27 ± 5.8 (14.8)[c] (13.1)[d]	26.9 ± 4.8			
TN	9.26			2.75 ± 0.2	0.3[e] ± 0.01	93.8[f]
NH_4-N	6.5	0.7 ± 0.4	5 ± 0.17	0.19 ± 0.13		
NO_3-N	0.05	4.7 ± 0.23	−4.8 ± 0.2	1.2 ± 0.15		
NO_2-N	0.0	0.0 ± 0.0	−0.02 ± 0.0	0.01 ± 0.01		
TP	1.82			2.4 ± 0.17	0.06[h] ± 0.0	75.8[i]
PO_4-P	1.04	−0.28 ± 0.8	1.0 ± 0.3	1.78 ± 0.38		

[a] COD equivalent content of 1 g biomass was measured at 1.48 ± 0.08 g. However, for the COD mass balance a value of 1.42 g COD/g VSS was used.
[b] COD % closure = $\frac{38.6+22.9+6+0.91}{69.8} \times 100$.
[c] SCOD consumption through denitrification based on [1]

$$\frac{\text{g SCOD}}{\text{g NO}_3\text{-N}} = \frac{2.86}{1 - 1.42Y_{obs}} \quad (5)$$

for example Phase I $= 5.4 \times \frac{2.86}{1 - 1.42 \times 0.093}$.

[d] Aerobic SCOD consumption in the riser;

for example Phase I $= \frac{\Delta O_2}{\Delta t} \times (1 - Y_H)^{-1} = 0.0018\,(\text{g O}_2/\text{L}) \times (10.7 + 1 + 4.5) \times 260\,(\text{L/d}) \times (1 - 0.4 \times 1.42)^{-1}$.

[e] Nitrogen (N) content of 1 g biomass was measured at 0.094 ± 0.01 g.
[f] Nitrogen % closure = $\frac{1.45+5.5+0.12}{7.8} \times 100$.
[g] Nitrogen assimilated for denitrification;

for example Phase I $= 5.4 \times \frac{2.86}{1 - 1.42 \times 0.093} \times 0.093\,(\text{g VSS/g SCOD}) \times 0.1\,(\text{g N/g VSS})$.

[h] Phosphorus (P) content of 1 g biomass was measured at 0.018 ± 0.05 g.
[i] Phosphorus % closure = $\frac{0.66+0.021}{0.8} \times 100$.
[j] The value of phosphorus precipitated based on MINTEQ.
[k] Phosphorus % closure with precipitation = $\frac{0.85+0.034+0.8}{1.89} \times 100$.
[l] Alkalinity generated in the anoxic column;

for example Phase II $= 7.7\,(\text{g N}_{\text{denitrified}}) \times 3.57\,(\text{g Alk}_{\text{generated}}/\text{g N})$.

[m] Alkalinity consumed in the aerobic column; for example Phase II;

for example Phase II $= 7.7\,\text{g N}_{\text{nitrified}} \times 7.14\,(\text{g Alk}_{\text{consumed}}/\text{g N})$.

[n] Alkalinity % closure = $\frac{64-(55.4-26.8)}{44} \times 100$.

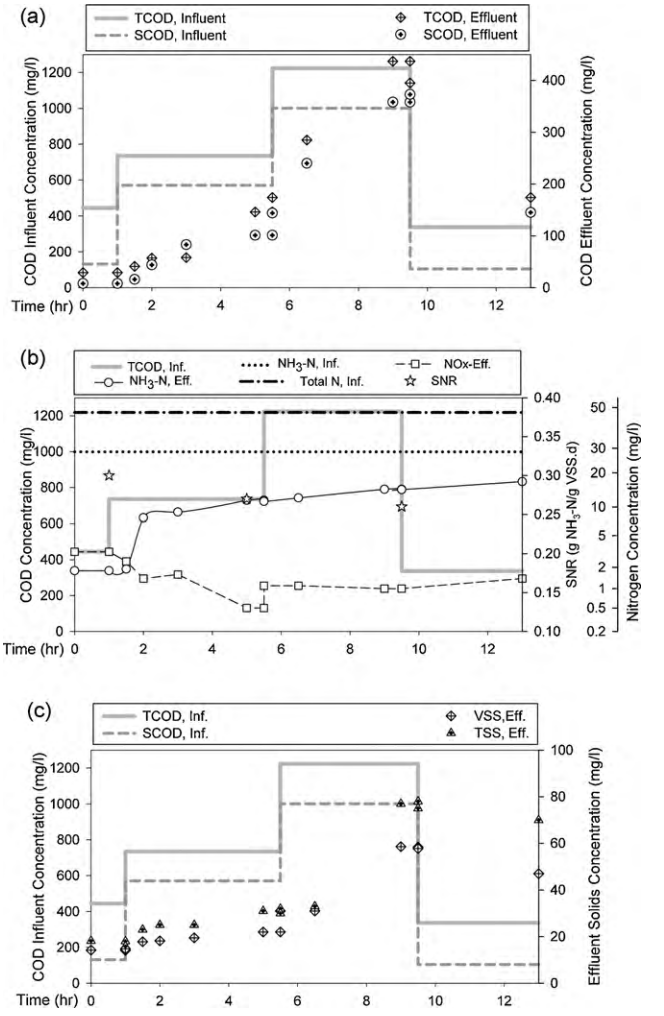

Fig. 6. (a) Effect of carbon shock test on the COD removal. (b) Effect of carbon shock test on the biological nitrogen removal. (c) Effect of carbon shock test on the effluent solids.

(3), adopted from ASM2, the aforementioned decrease in ambient DO concentration in the aerobic downer as the result of a very high oxygen demand reduces nitrification rate by 11%. It is estimated that the combination of oxygen limitation and nitrifier population reduction would reduce the overall nitrification rate by 25%, well below the observed 44% reduction, clearly emphasizing the sensitivity of nitrifiers to high ambient COD concentration.

$$r_{\mathrm{Nit}} = \mu_{\mathrm{aut}} \frac{S_{O_2}}{K_{O_2} + S_{O_2}} \cdot X_z, \qquad \text{where } K_{O_2} = 0.5\,\mathrm{g\,O_2/m^3} \tag{3}$$

Fig. 6c shows the effect of carbon shock test on the effluent suspended solids. The VSS in the effluent increased from 14 mg/L to an average value of 55 mg/L after 10 h which indicated a higher activity and detachment rate of the rapidly growing heterotrophs both in the downer and the riser.

3.4. Mass balances

Table 3 illustrates the steady-state mass balance for COD, TN, NH_3-N, NO_3-N, NO_2-N, TP, PO_4-P and alkalinity for phases I and II and dynamic loading tests at flow rates of 520 and 1040 L/d, where positive values indicate removal and negative values denote generation. The mass balances were based on experimental data of the influent, anoxic and final effluent characteristics, recirculation flows and the sludge wastage for each phase individually. As shown in Table 3, mass balance closures of 98.0% and 97.8% for COD, 90.6% and 89.6% for nitrogen, 92.7% and 97.8% for phosphorus and 93.3% for alkalinity were observed in phases I and II, respectively.

Anoxic COD consumption was observed to account for 53–58% of overall removal. The COD removal in the anoxic column was due to the denitrification process COD uptake (17.8, 26.6, 18.1 and 14.8 g/d in phases I, II, D_1 and D_2) as well as aerobic utilization as a result of DO recirculation from the aerobic column (17.5, 18.5, 17.5 and 13.1 g/d in phases I, II, D_1 and D_2) whereas the predominant COD removal in the aerobic zone was due to aerobic heterotrophic utilization (22.9, 42, 32.4 and 26.9 g/d in phases I, II, D_1 and D_2). The average liquid flow recirculation from the aerobic to anoxic column of 41.6 L/h with DO concentration of 5.5 mg/L mixes with the riser recirculation flow with 1 mg/L DO concentration. Therefore, the DO concentration at the bottom of the riser was 1.6 mg/L which may have contributed to aerobic COD removal in the riser. For instance in phase I, 14.1 mg/L COD was aerobically degraded in the anoxic zone as shown in the footer of Table 3. The measured COD consumption in the riser in phases I, II, D_1 and D_2 agree with the calculated COD consumption (d) and (e) within 90.1–94.5% accuracy.

Ammonia nitrogen was utilized by nitrification in the downer (5.6, 8.1, 5.6 and 5 g/d in phases I, II, D_1 and D_2) as well as ammonia nitrogen assimilation through denitrification process in the anoxic zone. There might be an insignificant nitrification zone in the anoxic column, since differences between the experimental ammonia nitrogen consumption and calculated through assimilation were observed (0.9 g NH_3-N/d versus 0.17 g NH_3-N/d in phase I and 1.2 g NH_3-N/d versus 0.26 g NH_3-N/d in phase II). As apparent from Table 3 in phase I, nitrification mass rates in the riser and downer were, respectively, 0.73 (0.8–0.12) and 5.6 g NH_4-N/d. Nitrification in the riser accounted for 11% of the overall nitrification, similarly in phase II, riser nitrification of 0.94 g NH_4-N/d accounted for only 10.6% of the overall system nitrification. As shown in Table 3, there was nitrite generation in the aerobic column which was not converted to nitrate, −0.18 g/d in phase I, and also nitrate conversion to nitrogen gas in the anoxic column. Alkalinity was produced in the anoxic column as due to denitrification at 26.8 g/d in phase II, and consumed in the aerobic column as carbon source for autotrophic nitrifiers at 55.4 g/d in phase II.

Phosphorus removal was found to be due mainly to the biomass assimilation. However additional phosphorus removal was observed while treating the municipal wastewater. As a result, the phosphorus mass balance closure in phase II as shown in Table 3 dropped to 50.1%. The additional phosphorus removal was as a result of precipitation by predominantly calcium existing in the wastewater in accordance with Eq. (4) [1,24].

$$10\mathrm{Ca} + 6{PO_4}^{3-} + 2\mathrm{OH^-} \leftrightarrow \mathrm{Ca_{10}(PO_4)_6(OH)_2} \tag{4}$$

Worth mentioning, no significant changes in total solids and attached biomass of the TCFBBR were noticed in this study. The precipitation of the inorganic metal phosphates and its strong adherence to media resulted in an accumulation of P in the system, unaccounted for in the mass balance. Assuming the entire unaccounted soluble phosphorus (approximately 1.7 mg/L) was removed by the calcium, based on Eq. (4) it would have generated around 2.3 g of $Ca_{10}(PO_4)_6(OH)_2$ per day, translating to approximately 270 g of solids over the study period or <2.5% of the media mass. The average concentrations of calcium, magnesium and aluminum in the municipal wastewater were measured 59.8, 12.9 and 0.76 mg/L, respectively. Considering the aforementioned metal concentrations and ortho-phosphate concentration in the influent with the effluent pH of 7.7 ± 0.3 and temperature of 22 °C (Table 1b), the amount of phosphorus removed by precipitation was calculated as 3.1 mg/L using MINTEQ ver. 2.61 [25], thus improving the phos-

phorus mass balance closure in phase II to 89.1% from the 50.1% reported above.

4. Conclusions

The lab-scale TCFBBR was operated at loading rates of 2.7–4.3 kg COD/m^3 d, 0.3–0.51 kg N/m^3 d, and 0.032–0.06 kg P/m^3 d to study nutrient removal efficiencies of the system at a very short HRT of 2.3 h. The principal findings of this study are:

(i) Approximately >90% organic, >85% nitrogen, and 20–51% phosphorus removal were experienced using the TCFBBR at nutrient loading rates of 4.3 kg COD/(m^3 d), 0.51 kg N/(m^3 d), and 0.06 kg P/(m^3 d), and an EBCT as low as 1.0 h.
(ii) Effluent TN of <8 mg/L indicates the system efficiently removed nitrogen by nitrification–denitrification.
(iii) Due to precipitation and assimilation 17–51% of the influent phosphorus was removed without addition of any chemicals.
(iv) As a result of a long SRT of up to 40 days, very low observed yield of 0.093–0.101 g VSS/g COD were observed.
(v) The system did not show any considerable deterioration in nutrient removal efficiency during dynamic testing at a hydraulic peaking factor of 4 for 3 h.
(vi) A 50% loss of nitrification efficiency was observed during a carbon shock test due to DO limitations, washout of nitrifiers, and high COD concentrations in the aerobic downer.

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Appendix A.

Based on Eq. (A.1) [1] and the COD consumption in the riser and downer in each phase, shown in Table 3, the observed yield can be calculated.

$$Y_{obs} = \frac{Y}{1+(k_d)\mathrm{SRT}} + \frac{(f_d)(k_d)(Y)\mathrm{SRT}}{1+(k_d)\mathrm{SRT}} \quad (A.1)$$

where Y = 0.4 g VSS/(g sCOD), k_d = 0.15 g VSS/(g VSS d) and f_d = 0.15 g VSS/g VSS [1].

Phase I:

In the riser:

$$Y_{obs} = \frac{0.85\times0.4\times38.6}{1+0.15\times32} + \frac{0.15\times0.15\times0.85\times0.4\times32}{1+0.15\times32} = 2.302\,\mathrm{g\,VSS/d}$$

In the downer:

$$Y_{obs} = \frac{0.4\times22.9}{1+0.15\times7} + \frac{0.15\times0.15\times0.4\times7}{1+0.15\times7} = 4.498\,\mathrm{g\,VSS/d}$$

Overall yield:

$$Y_{obs} = \frac{2.302+4.498}{69.8} = 0.097\,\mathrm{g\,VSS/g\,COD}$$

The experimental observed yield in phase I is 0.093 g VSS/g COD. For phase II also the same precision can be achieved.

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