Kinetic evaluation and process performance of an upflow anaerobic filter reactor degrading terephthalic acid

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ABSTRACT

The anaerobic degradation of terephthalic acid (TA) as the sole organic carbon source was studied in an upflow anaerobic filter (UAF) reactor. The reactor was seeded with biomass obtained from a full-scale upflow anaerobic sludge bed (UASB) reactor and was used to treat wastewater from a petrochemical facility producing dimethyl terephthalate. The UAF reactor was operated for 252 d with a constant hydraulic retention time of 24 h, and the organic loading rate (OLR) was gradually increased from 1 to 10 g-chemical oxygen demand (COD)/L d. After a lag period of approximately 40 d, the COD removal efficiency increased exponentially and high removal rate values (≈90%) were obtained, except for at highest OLR (10 g-COD/L d). The high removal rates and the robustness of the reactor performance could be attributed to the formation of biofilm as well as granular sludge. The methane production rates (0.22 to 2.15 L/d) correlated well with the removed OLRs (0.3 to 6.8 g-COD/L d) during the various phases of treatment, indicating that the main mechanism of TA degradation occurs via methanogenic reactions. The average methane content of the produced biogas was 70.3%. The modified Stover–Kincannon model was found to be applicable for the anaerobic degradation of TA in UAFs ($U_{max} = 64.5$, $K_B = 69.1$ g-COD/L d and $Y_{max} = 0.27$ L-CH_4/g-$COD_{removed}$). These results suggest that UAF reactors are among the most effective reactor configurations for the anaerobic degradation of TA.

Key words | petrochemical wastewater, terephthalic acid, upflow anaerobic filter reactor

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INTRODUCTION

Terephthalic acid (TA) is widely used as a raw material for various petrochemical products such as polyethylene terephthalate bottles, polyester films and textile fibres (Savostianoff 1990). TA is among the top 50 chemicals manufactured in the world, with an annual production of 24.9 million tons in 2002 (Zhang *et al.* 2006). The polymer-grade TA manufacturing process generates approximately 3–10 m^3 of high strength wastewater per ton of TA produced. The wastewater (TA wastewater) contains TA and acetic acid (AA) as the main pollutants as well as aromatic intermediates of *p*-xylene oxidation (*p*-toluic acid, and benzoic acid (BA)) and 5–20 kg-chemical oxygen demand (COD)/m^3 (Macarie *et al.* 1992; Kleerebezem *et al.* 1997).

TA and its esters are suspected to cause cancer, renal damage and endocrine disruption (Dai *et al.* 2005). The US EPA has added this class of chemicals to the list of priority pollutants (US EPA 1992). Therefore, wastewater that contains these chemicals must to be treated before being discharged into the environment.

The anaerobic treatment of TA wastewater faced challenges including long start-up period, achieving a stable operation of full-scale reactors and complex biodegradation mechanisms for the degradation of the pollutants present in TA wastewater (Fajardo *et al.* 1997; Kleerebezem *et al.* 1999a; Joung *et al.* 2009). When considering the anaerobic degradation of compounds in TA wastewater, AA and BA are classified as readily biodegradable, whereas TA and *p*-toluic acid can only be degraded after longer periods of time or at very low degradation rates (Kleerebezem *et al.* 1999a). Kleerebezem *et al.* (2005) suggested that *p*-toluic acid should be removed during the aerobic post-treatment of anaerobic effluent due to the poor degradation of *p*-toluic acid under anaerobic conditions. Therefore, terephthalate degradation represents the main bottleneck in the anaerobic treatment of TA wastewater.

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To overcome the drawbacks associated with anaerobic TA degradation, a number of reactor configurations and operational patterns have been studied. Guyot *et al.* (1990) operated two upflow anaerobic sludge bed (UASB) reactors (A and B) seeded with different inocula (A, anaerobic stabilization ponds; B, anaerobically adapted activated sludge) to evaluate the anaerobic digestion of TA wastewater. They reported low COD removal values of 46.6 and 43.9% for reactors A and B, respectively, at loading rates of 2.6 (reactor A) and 2.2 kg-COD/L d (reactor B). Macarie *et al.* (1992) presented a comparative study of anaerobic systems using UASB and a down-flown tubular fixed bed reactor for the treatment of TA wastewater, reporting that the highest COD_{total} removal efficiency (84%) was obtained using the fixed-film reactor at a hydraulic retention time (HRT) of 3.4 d when combined with pre-settling. Cheng *et al.* (1997) found that a UASB reactor can achieve up to a 62% COD removal efficiency at a volumetric loading rate of 2.93 kg-COD/L d. Kleerebezem *et al.* (1997) found that the maximum removal capacity of a laboratory-scale UASB reactor was 3.9 g-COD/L d at a loading rate of 4.5 g-COD/L d and an HRT of 24 h. Young *et al.* (2000) investigated a two-stage anaerobic treatment system consisting of hybrid and contact processes and reported high removal rates of 83% and 88%, respectively. Kleerebezem *et al.* (1999b) reported high removal rates of 10–17 g-COD/L d at HRTs between 5 and 8 h in anaerobic hybrid reactors. Tsuno & Kawamura (2009) developed an expanded-bed granular activated carbon anaerobic reactor to treat wastewater containing terephthalate and reported high COD removal efficiencies (≥90%) at a loading rate of 4 kg-COD/m^3 d and a terephthalate loading rate of 1 kg-TA/m^3 d. Joung *et al.* (2009) determined a COD removal efficiency of 79% under a the volumetric loading rate of 5.05 g-COD/m^3 d and an HRT of 50 h in an anaerobic filter process for TA wastewater treatment.

Determining the kinetic parameters of a biological process is useful for describing and predicting the performance of a system. However, limited information is available for the kinetic evaluation of TA degradation in anaerobic filter reactors (AFRs). Based on the results of previously published studies, it is not feasible, from an engineering perspective, to determine whether it is practicable to treat TA anaerobically using AFRs. Therefore, in this work, a laboratory-scale upflow anaerobic filter (UAF) reactor was continuously operated to: (i) determine the length of the lag period; (ii) evaluate the performance of the reactor under varying organic loading rate (OLRs); and (iii) determine the kinetic parameters of substrate removal.

MATERIAL AND METHODS

Anaerobic filter reactor and seed sludge

Continuous flow experiments were performed in a UAF constructed from PVC tubes with a liquid volume of 1,500 mL. The reactor was equipped with a water jacket and a thermostat-controlled water circulation pump to operate the system in mesophilic conditions ($35 \pm 2\,^{\circ}C$). Ceramic raschig rings (1.2 cm diameter × 1.2 cm height) were used as the supporting material and the working volume of the reactor after packing was 1,150 mL. The specific surface area of the supporting material was calculated to be 223 m^2/m^3, corresponding to a total surface area of 0.44 m^2 in the reactor. The biogas produced in the reactor was collected by the downward displacement of acidified water (0.05 M H_2SO_4), and the volume was measured at standard temperature and pressure. The effluent tube was 'U' shaped and a liquid seal was maintained to prevent gas from escaping through the line.

The UAF was seeded with methanogenically active granular anaerobic sludge obtained from a full-scale UASB reactor treating dimethyl terephthalate (DMT) wastewater in mesophilic conditions. The reactor was inoculated with 365 mL of wet seed sludge containing 95 g/L volatile suspended solids (VSS), resulting in a total of 30 g-VSS/L of reactor volume.

Medium and substrate preparation

The basal nutrient medium solution used in this study was described by Ahn & Forster (2000). The substrate solution was prepared by dissolving crystalline TA in distilled water amended with excess NaOH. The pH was then adjusted to between 7.0 and 7.2 with the addition of concentrated HCl. The substrate and nutrient medium were prepared separately and mixed (1/1; v/v) immediately before being fed into the reactor. The feed was adjusted to the desired loading rate. The influent was stored at $4\,^{\circ}C$ to prevent deterioration during the study.

Analytical procedures

The pH, total alkalinity and soluble COD concentrations of the influent and effluent were measured daily using standard methods (APHA 2005). Additionally, the total solids and total suspended solids were determined to characterize the seed sludge (methods SM 2540-B and SM 2540-E, respectively).

The TA concentration was determined by high pressure liquid chromatography (Perkin Elmer, Series 200 model) using a Spheri-5 C18 column (220 × 4.6 mm). The mobile phase was a mixture of methanol and 1% AA in water at a ratio of 40:60, at a flow rate was 1 mL/min. TA measurements were performed at ambient temperatures using spectrophotometric UV detection at a wavelength of 230 nm. The retention time of TA was 4.53 min.

The composition of biogas produced in the reactor was determined by gas chromatography (GC) (Perkin Elmer, GLX model). The biogas was sampled and injected into the gas chromatograph using an airtight syringe. The GC system used a stainless-steel column (2 m × 2 mm) with a Propack Q support (mesh size 80–100), which was connected to a thermal conductivity detector. Nitrogen gas was used as the carrier gas (10 mL/min). The temperatures of the column and the detector were 35 and 140 °C, respectively.

All sample analyses were performed in duplicate. The deviation of the results from the mean was less than 5%.

Experimental procedures

The UAF reactor was operated continuously for 252 days under varying OLRs and the loading rate was gradually increased from 1 to 10 g-COD/L d. During the study, the HRT was held constant (24 h) to evaluate the TA degradation performance at different OLRs. The operation phases and parameters are summarized in Table 1.

The modified Stover–Kincannon model

Kinetic modeling is an accepted approach for describing the specific parameters used to monitor the overall performance of biological reactors. The Stover–Kincannon kinetic model is the most widely used mathematical model for determining the kinetic constants in immobilized systems. In this model, the substrate utilization rate is expressed as a function of the organic loading rate using monomolecular kinetics for biofilm reactors (Işık & Sponza 2005). The modified version of the model was established based on the effective volume of the reactor instead of the active surface area. This model has been applied to various types of wastewater systems, including soy bean (Yu *et al.* 1998), simulated starch (Ahn & Forster 2000), paper mill (Yilmaz *et al.* 2008), milk permeate (Wang *et al.* 2009), and food-processing (Şentürk *et al.* 2010) wastewater. However, it has not been applied for the determination of kinetic constants of anaerobic TA degradation systems. The modified Stover–Kincannon model is described as

$$\frac{dS}{dt} = \frac{Q(S_i - S_e)}{V} = \frac{U_{max}(QS_i/V)}{K_B + (QS_i/V)} \quad (1)$$

Equation (1) can be linearized as

$$\left(\frac{dS}{dt}\right)^{-1} = \frac{V}{Q(S_i - S_e)} = \frac{K_B}{U_{max}}\left[\frac{V}{QS_i}\right] + \frac{1}{U_{max}} \quad (2)$$

where dS/dt is the substrate removal rate (g/L d), U_{max} is the maximum utilization rate constant (g/L d), and K_B is a saturation value constant (g/L d). If the inverse of the loading removal rate, $V/Q(S_i–S_e)$, is plotted against the inverse of the total loading rate, $V/(QS_i)$, K_B/U_{max} is the slope and $1/U_{max}$ is the intercept point of a straight line.

Methane production is an important parameter for anaerobic treatment systems and, thus, the methane production kinetics should also be addressed. Yu *et al.* (1998) expressed that methane production is also a function of the OLR. Therefore, they used an expression similar to Equation (2) to derive kinetic constants for the determination of methane production in the reactor and used the specific methane production (m^3CH_4/m^3 reactor volume) as the biogas component in the equation. However, Ahn & Forster (2000) used the specific methane yield parameter (SMY, $m^3CH_4/kg\text{-}COD_{removed}$) instead of the specific methane production for practical applications by using a similar method as previously described in Equation (2)

$$\left(\frac{1}{SMY}\right) = \frac{A}{Y_{max}}\left[\frac{V}{QS_i}\right] + \frac{1}{Y_{max}} \quad (3)$$

where Y_{max} is the maximum specific methane yield ($m^3CH_4/kg\text{-}COD_{removed}$) and A is a constant (g/L d).

Table 1 | Experimental phases and operational parameters in the UAF reactor

Phase	Time (days)	HRT (hours)	TA (g/L-mM)	OLR (gCOD/L.d)
I	1–56	24	0.69–4.17	1
II	57–118		1.38–8.33	2
III	119–140		2.07–12.5	3
IV	141–161		3.46–20.83	5
V	162–214		5.19–31.24	7.5
VI	215–252		6.92–41.69	10

RESULTS AND DISCUSSION

Reactor start-up

The UAF reactor was started using an OLR of 1.0 g-COD/L d (690 mg-TA/L d) with TA fed as the sole carbon source. The start-up period for TA degradation was considered to be 56 d and period was assessed during the lag, exponential and stationary states (Figure 1). The lag phase lasted for 40 d and COD removal was insignificant (18%) during the first 35 days of the lag phase. Between days of 35 and 46, the COD removal rate increased exponentially to 90% and remained at this level throughout the stationary phase. During the start-up period, the methane production showed an inverse relationship with the effluent COD, indicating that the main mechanism of TA removal was achieved via methanogenic activity. The mean methane content of the biogas was 61% during the start-up period and reached 70% in the stationary phase.

Although a start-up time of up to 2 years is required for full-scale bioreactors used in TA wastewater treatment (Macarie *et al.* 1992), only a very limited amount of information is available on reactor start-up for anaerobic TA degradation. Kleerebezem *et al.* (1999a) determined that the phase of three different types of seed material obtained from anaerobic bioreactors treating sewage, starch processing and paper mill wastewater, lasted between 44 ± 4 and 61 ± 7 d at half of the TA concentration (2.1 mmol, 348.6 mg-TA/L) used in a batch experiment. Kleerebezem *et al.* (1999b) inoculated hybrid reactors (HR1, HR2 and HR3) using a seed sludge from the treatment of PTA (purified terephthalic acid) wastewater and determined that the lag phase duration was approximately 80 d at an OLR and HRT of 1.3 mmol-TA/L d and 15 h, respectively.

In this study, the lag phase was slightly shorter than these published values. This difference can be attributed to the origin of the seed sludge and reactor configuration. The UAF was inoculated with sludge obtained from a UASB reactor used to treat DMT wastewater, which contains similar aromatic constituents but at significantly different concentrations compared to TA wastewater. Kleerebezem *et al.* (1999a) concluded that the time needed for the start-up of anaerobic bioreactors treating TA is largely independent of the microbial composition of the seed material, but may require several months. The duration of the lag phase obtained in this study supports this suggestion, despite the fact that the UAF was inoculated with sludge obtained from a full-scale reactor treating DMT wastewater. The shorter length of the lag phase may be attributed to the filter reactor configuration, which allows high amounts of VSS (30 g/L) to be maintained in the reactor.

Performance of the UAF reactor

After the UAF start-up period, the OLR was increased by increasing the TA concentration while the HRT was kept constant. During phase II, the reactor feed OLR was increased to 2 g-COD/L d (1.38 g-TA/L d) and was operated between days 57 and 118 until steady state results were obtained. On the initial days, the COD removal rate decreased sharply to 55%, resulting in an increase in the effluent COD to 0.9 g/L. However, high removal efficiencies (≈90%) were maintained after day 77, indicating that the

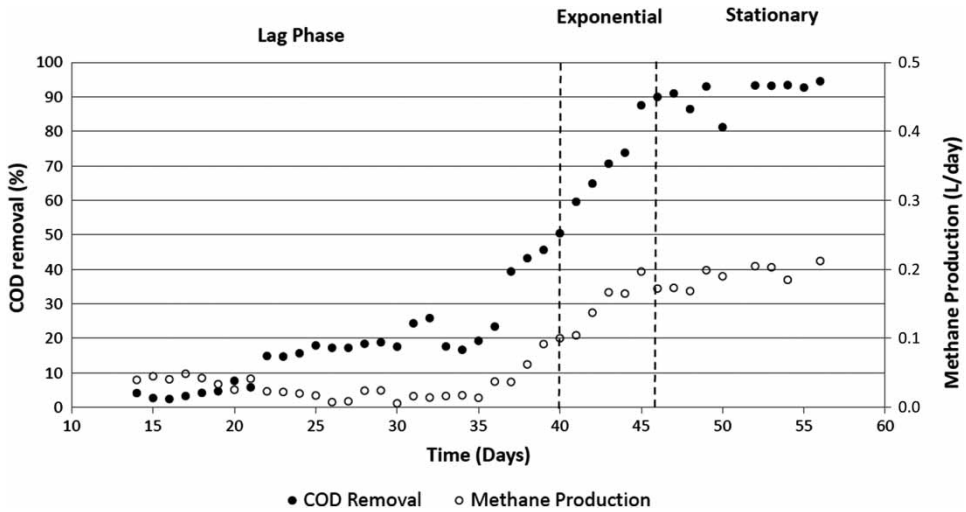

Figure 1 | Reactor start-up period of TA degradation.

reactor responded well to the elevated TA concentration (see Figure 2). A similar COD removal was observed at an OLR of 3 g-COD/L d (2.07 g-TA/L d), between days 119 and 140. The COD removal was slightly lower than at the previous loading rate (76%), indicating that the biomass in the reactor was completely enriched, and COD removal efficiency was restored to greater than 90% within only 12 d. After the steady state condition was obtained, the reactor was operated at an OLR of 5 g-COD/L d (3.46 g-TA/L d) between days 141 and 161. However, the COD removal rates were not affected by increasing the OLR to 5 g-COD/L d, suggesting that the formation of biofilm on the supporting material of the filter reactor was completed, and maturated biofilm played an active role in TA degradation. Thus, robust reactor performance was afforded by the adapted biofilm as well as the granular biomass.

As shown in Figure 2, the reactor performance began to fluctuate in phase V at an OLR of 7.5 g-COD/L d (5.19 g-TA/L d). Similar COD removal efficiencies (≈95%) were observed during the initial loading period (≈10 d) compared to phase IV and then decreased to 77% (mean of 85%). However, the amount of COD removed remained higher than or equal to that of the previous phase. In this phase, the maximum TA removal capacity and maximum specific TA degradation activity of the biomass grown in the UAF were 4.63 g-TA/L d (27.9 mmol/L d or 6.8 g-COD/L d) and 0.15 g-TA/g-VSS d, respectively. Kleerebezem *et al.* (1997) reported a maximum removal capacity of 3.9 g-COD/L d at an HRT of 24 h in a laboratory-scale UASB reactor in which TA was the sole carbon source. Cheng *et al.* (1997) reported a removal capacity of 62% at an OLR of 2.93 g-COD/L d and an HRT of 2.1 d in a pilot scale UASB process for PTA wastewater treatment. Kleerebezem *et al.* (1999b) reported a removal capacity of 0.91 g-COD/L d at an OLR of 1.44 g-COD/L d and an HRT of 20 h in a UASB reactor with TA as the sole carbon source. Based on these results, UAF reactors appear to be a more effective reactor configuration compared to UASB systems.

Kleerebezem *et al.* (1999b) obtained a high TA degradation rate of 71 mmol TA/L d in hybrid anaerobic reactors at a volumetric loading rate of 84 mmolTA/L d at an HRT 10 h. They attributed the high removal capacities to enhanced biomass retention due to the development of biofilms on the carrier material as well as formation of granular biomass. The specific surface area of the carrier material used in the hybrid reactor (800 m^2/m^3) was three-times higher than that of the raschig rings (approximately 225 m^2/m^3) which allows for a greater extent of biofilm formation and a higher sludge age, which resulted in a high removal efficiency compared to the UAF reactor.

During the final phase, at an OLR of 10 g-COD/L d, the COD removal efficiency decreased linearly to 65% (Figure 2). First, in phase VI, difficulties occurred during the reactor feeding at a constant OLR, which varied between 8.4 and 10.7 g-COD/L d (mean ± standard deviation: 9.75 ± 0.53) due to operational irregularities such as the influent pumping and substrate preparation; Joung *et al.* (2009) reported that the removal performance of an anaerobic filter reactor treating TA wastewater was negatively affected by organic loading disturbances. Second, a

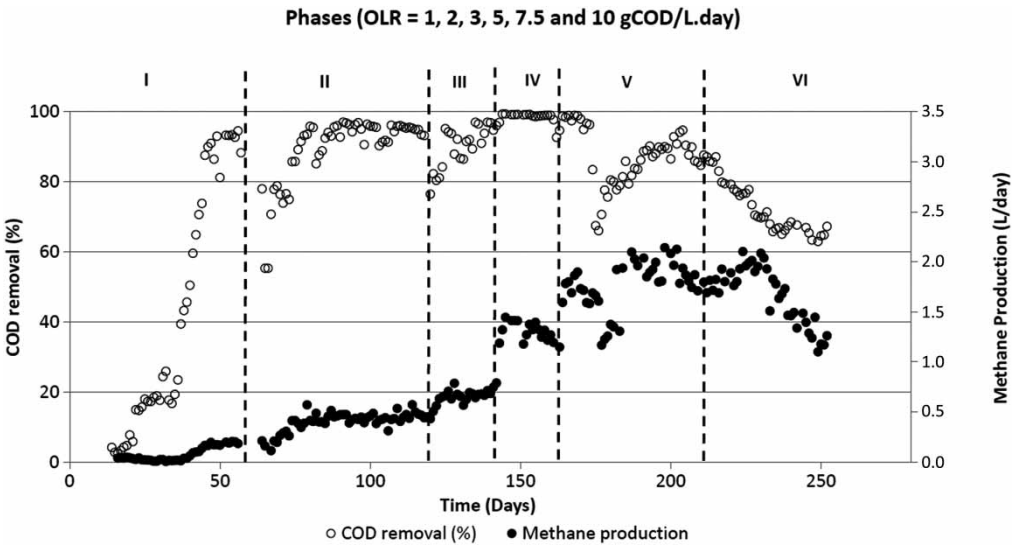

Figure 2 | COD removal efficiency and methane production profiles in the UAF reactor.

decrease in the reactor performance in this study may correspond to the high ionic strength of the influent. The electrical conductivity of the influent increased to 18 mS/cm due to the high amounts of chemicals used to prepare the influent (mainly, NaOH and HCl). Rittmann & McCarty (2001) reported that the high ionic strength of wastewater is a reasonable source of inhibition. Furthermore, many studies have reported an inverse relationship between reactor performance and sludge loading rate (g-COD/(g-MLSS L d)) for anaerobic treatment systems (Yu *et al.* 1998; Ahn & Forster 2000; Şentürk *et al.* 2010).

The maximum biogas production rate was 2.6 L/d during phase V. The specific methane yield varied from 0.22 to 0.33 $LCH_4/COD_{removed}$ and the maximum value was obtained at an OLR of 7.5 g-COD/L d. This level was close to the theoretical methane yield of 0.35 $LCH_4/COD_{removed}$ and comparable to previously reported results (Ahn & Forster 2000; Yilmaz *et al.* 2008). Throughout the study, the percentage of methane in the biogas was largely constant under steady-state conditions (67 to 73%), indicating that TA does not have toxic effects on the methanogens and that the intermediate products from the initial steps of anaerobic TA degradation were effectively converted to methane (Figure 2). Fajardo *et al.* (1997) determined that TA concentrations of greater than 100 mmol (24 g-COD/L) are required to inhibit the hydrogenotrophic methanogenic activity by 50%, and 10 mmol TA (2.4 g-COD/L) was shown to inhibit acetoclastic methanogenic activity by less than 12%. According to the results of Fajardo *et al.* (1997), a decrease in methane production may be attributed to a high concentration of TA (>40 mmol, 9.6 g-COD/L). However, at a higher OLR (>7.5 g-COD/L d), a decrease in COD removal efficiency may be associated with the saturation of the substrate concentration, where the removal rate shifts from first order to zero order, as defined by Monod (1949). Efficient methanogenic activity during the anaerobic degradation of TA indicates that one of the initial steps is the rate-limiting step. Kleerebezem *et al.* (2005) proposed that the rate of TA hydrolysis is the rate-limiting step in anaerobic TA degradation.

Modeling

The plot developed using the modified Stover–Kincannon model, based on the COD for the UAF reactor is shown in Figure 3(a). In this plot, the points fit a straight line with a small intercept; the regression coefficient (0.992) strongly supports the validity of the model. Thus, it can be concluded

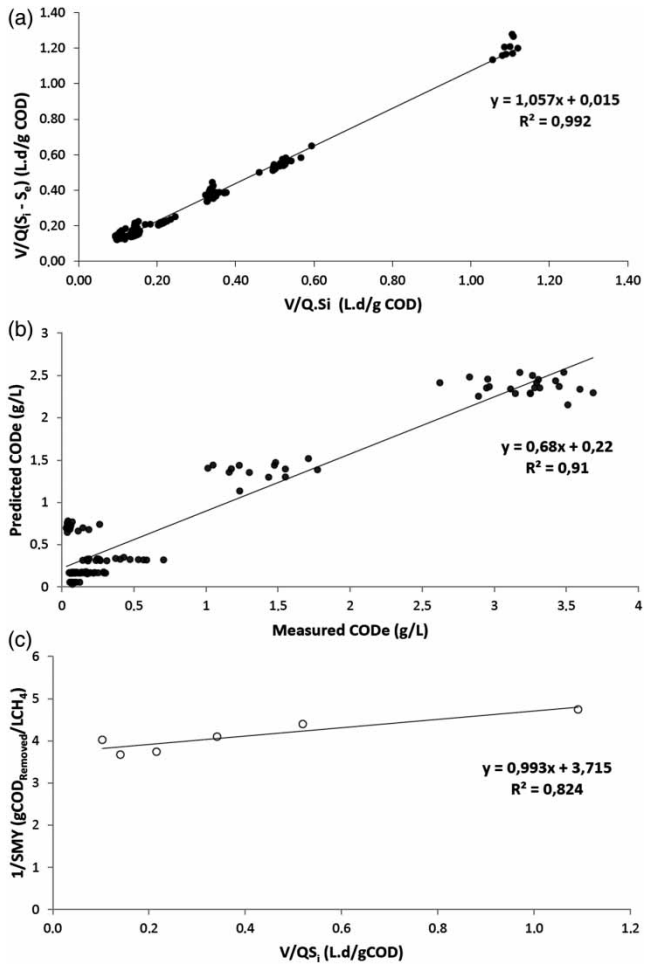

Figure 3 | (a) The modified Stover–Kincannon model plot for the UAF, (b) comparison of the measured effluent concentrations with the predicted effluent concentrations for UAF, (c) determination of the maximum specific methane yield (Y_{max}) of anaerobic TA degradation.

that the modified Stover–Kincannon model is capable of describing the performance of the UAF for treating TA in this study. The calculated values of U_{max} and K_B were 64.5 and 69.1 g-COD/L d, respectively. A comparison of the kinetic values obtained by the model for various substrates and wastewater is shown in Table 2.

The U_{max} was significantly higher than the maximum OLR (10 mg/L d) used during this study. Wang *et al.* (2009) and Şentürk *et al.* (2010) attributed this to the fact that their reactors have a higher potential of coping with high-strength wastewater. Ahn & Forster (2000) state that the difference between the U_{max} and the maximum OLR is due to an appreciable deterioration in performance, which occurred at the highest OLR.

A prediction of substrate effluent concentration (S_e) can be obtained based on the mass balance of the substrate in

Table 2 | A comparison of Stover–Kincannon kinetics obtained for different wastewater and substrates

Reference	Source of substrate	OLR_{max} (g/L.day)	U_{max} (g/L.day)	K_B (g/L.day)
This study[a]	Terephthalic acid	10	64.5	69.1
Şentürk *et al.* (2010)	Food processing wastewater	5	22.95	23.58
Wang *et al.* (2009)	Milk permeate	28.0	89.3	102.3
Yilmaz *et al.* (2008)	Paper mill wastewater	12	86.21	104.15
Ahn & Forster (2000)	Starch wastewater	3.39	49.8	50.6
Yu *et al.* (1998)	Soy bean wastewater	13.5	83.33	85.5

[a]U_{max} and K_B represented as COD equivalence.

the reactor by substituting kinetic constants U_{max} and K_B, resulting as

$$S_e = S_i - \frac{U_{max} S_i}{K_B + (QS_i/V)} \quad (4)$$

A comparison of the measured and predicted effluent data is presented in Figure 3(b). The high correlation ($R^2 = 0.91$) between the two parameters indicates that the kinetic constants can be used to estimate the treatment efficiency of full-scale reactors with the same operational conditions.

Figure 3(c) shows the TA loadings plotted as a function of the mean values of the specific methane yield. The kinetic constant for methane production was determined to be $Y_{max} = 0.27$ L-CH_4/g-$COD_{removed}$. These results show that the obtained methane production constants were applicable for an anaerobic filter when it is not overloaded ($\leq$10 g-COD/L d).

CONCLUSIONS

The duration of the lag phase for TA degradation in the UAF reactor was determined as 40 d. After the reactor start-up, the COD removal efficiencies ranged from 89% to 98%, in steady-state conditions, except for the final OLR of 10 g-COD/L d (67%). The maximum TA removal capacity of UAF and maximum specific TA degradation activity of biomass grown in the UAF were 4.63 g-TA/L d (27.9 mmol-TA/L d) and 0.15 g-TA/g-VSS d, respectively. Throughout the study, the percentage of methane gas in the total biogas was approximately 70.3%. Based on these results, the use of a UAF reactor for TA degradation appears to be a more effective approach compared to the use of UASB systems.

The kinetic analysis of the UAF for COD removal using the modified Stover–Kincannon model showed that the U_{max}, K_B (64.5 and 69.1 g-COD/L d, respectively), and Y_{max} (0.27 L-CH_4/g-$COD_{removed}$) were highly correlated ($R^2 > 0.99$). There was good agreement between the observed and predicted COD values, indicating that the kinetic parameters can be used to design an anaerobic filter reactor for anaerobic TA degradation.

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