Effect of hydraulic retention time on pretreatment of blended municipal sludge

S. Koyunluoglu-Aynur, R. Riffat and S. Murthy

ABSTRACT

The objective of the present work was to evaluate the effect of hydraulic retention time (HRT) on hydrolysis and acidogenesis for the pretreatment processes: acid phase digestion (APD) and autothermal thermophilic aerobic digestion (ATAD) using blended municipal sludge. The effect of the different pretreatment steps on mesophilic anaerobic digestion (MAD) was evaluated in terms of methane yield, keeping the operating conditions of the MAD the same for all systems. Best operating conditions for both APD and ATAD were observed for 2.5 d HRT with high total volatile fatty acids (tVFA), and the highest methane yield observed for MAD. No significant difference was observed between the two processes in terms of overall volatile solids (VS) reduction with same total HRT. The autothermal process produced heat of 14,300 J/g VS removed from hydrolytic and acetogenic reactions without compromising overall methane yields when the HRT was 2.5 d or lower and the total O_2 used was 0.10 m^3 O_2/g VS added or lower. However, the process needs the input of oxygen and engineering analysis should balance these differences when considering the relative merits of the two pretreatment processes. This is the first study of its kind directly comparing these two viable pretreatment processes with the same sludge.

Key words | acid/gas phase digestion, acidogenesis, ATAD, hydraulic retention time, VFA production

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INTRODUCTION

The main objectives of municipal sludge treatment are to stabilize organic matter together with volume reduction and pathogen destruction, in order to produce biosolids with a quality suitable for meeting the requirements of agricultural land application. Anaerobic digestion is the traditional method to reduce solids volume but it requires long retention times with most digesters operated at mesophilic temperatures. Anaerobic digestion can be enhanced using various pretreatment options, resulting in higher volatile solids (VS) reduction, and increased pathogen destruction using shorter hydraulic retention time (HRT) (Borowski & Szopa 2007).

Anaerobic digestion is composed of three major steps: hydrolysis of insoluble particulate matter; acidogenesis where complex organic substances are converted to volatile fatty acids (VFAs) and other low molecular weight soluble carbon compounds, and methanogenesis as the final step at which waste stabilization occurs. In single-stage anaerobic digestion of municipal sludge, all three steps take place in the same reactor, hydrolysis of complex organic compounds being the rate-limiting step. On the other hand, two stage processes separate VFA production (hydrolysis and acidogenesis stages) and utilization (methane forming stage) by optimizing the operational conditions for acidogenic and methanogenic bacteria (Banerjee *et al.* 1998; Ponsa *et al.* 2008).

Two-phase systems provide the separation of hydrolysis/acidogenesis and methanogenesis steps, increasing the process stability. In general, thermophilic temperatures are used for the pretreatment step, having the advantage of higher destruction rates of organic solids and increased destruction of pathogens. Hydraulic retention time has a considerable effect on the population levels of methanogens and composition of fermentative products like VFAs (de la Rubia *et al.* 2006). In addition to thermophilic temperatures, short retention times are adopted for the pretreatment step, in order to inhibit methanogenic population and increase acid production.

One option for two-phase systems is employing thermophilic anaerobic digestion as the pre-treatment step followed

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by mesophilic anaerobic digestion. This process is called acid/gas phase digestion.

Autothermal thermophilic aerobic digestion (ATAD) is a sludge treatment process where heat is released by the aerobic microbial degradation of organic matter (Layden 2007). In ATAD, the heat released by the digestion process is the major heat source used to achieve the desired operating temperature (EPA 1990).

An ATAD process with a relatively short residence time can be used as a pretreatment step to the mesophilic anaerobic digestion, and that is termed dual digestion (DD) (Ward *et al.* 1998; Zabranska *et al.* 2003). If ATAD is operated under oxygen limiting conditions in conjunction with short HRT, this results in the formation of VFAs through the fermentative metabolism of thermophilic bacteria (Ward *et al.* 1998; Borowski & Szopa 2007). In the ATAD step, sludge is pretreated by solubilization and partial acidification resulting in enhanced digestion, together with improved pathogen destruction (Borowski & Szopa 2007; Nosrati *et al.* 2007) while at the same time producing autothermal heat.

The pretreatment step in both dual digestion and acid/gas phase digestion provides consistent pH buffered feed with ammonification and high VFA concentration to the methanogenic stage, which improves volatile solids destruction in the downstream anaerobic digester (McIntosh & Oleszkiewicz 1997; Warakomski *et al.* 2007).

There are many facilities in the world today that are operating either of these two acid producing pretreatment processes. However, it is difficult to determine the merits of the individual processes without a side-by-side study. The objective of the present work was to evaluate the effect of HRT on hydrolysis and acidogenesis for two pretreatment processes: acid phase digestion (APD) and ATAD, using blended municipal sludge from Blue Plains Advanced Wastewater Treatment Plant. In addition, the effect of the different pretreatment steps on mesophilic anaerobic digestion (MAD) were evaluated in terms of methane yield, keeping the operating conditions of the MAD the same for all systems. Two advanced digestion processes were operated for this purpose: (i) Acid phase digester at 55 °C followed by gas phase digester at 35 °C (APD-MAD); and (ii) Dual Digestion process consisting of ATAD at 55 °C followed by mesophilic digester at 35 °C (ATAD-MAD). The pretreatment reactors were operated at HRTs of 1.5, 2.5 and 3.5 d. This is the first reported side-by-side study of these two pre-treatment processes.

MATERIALS AND METHODS

Experimental set-up

The feed for both processes was collected from Blue Plains Advanced Wastewater Treatment Plant in Washington, DC. Blended primary and waste activated sludge (1:1 by volume) were thickened to 6.5% total solids (TS) using a laboratory centrifuge. The raw sludge used for this study had a TS level of 6.56 ± 0.16%, VS of 5.22 ± 0.14%, total COD of 84,792 ± 7,500 mg/L, soluble COD of 9,800 ± 1,800 mg/L, alkalinity of 2,500 ± 220 mg/L as $CaCO_3$, pH of 6.31 ± 0.28 and NH_3-N of 310 ± 63 mg/L as N. The digesters were fed once per day.

Batch fermentation reactors with 25 L total volume from Hobby Beverage Equipment Company (Temecula, California) were used for the study. The reactors were heated using Thermolyne Heating Tape (Columbia, Maryland). Mixing was achieved by recirculating the headspace gas using peristaltic pumps (6–600 rpm modular drive). Biogas was collected in Restek tedlar bags (Bellefonte, Pennsylvania). For ATAD reactor, pure oxygen (99%) was added to the gas recirculation system. The experimental set-up for the digesters is illustrated in Figure 1. Flow diagrams for the two systems, APD-MAD and ATAD-MAD are illustrated in Figure 2. As can be observed from Figure 2, HRT for MAD was kept constant at 7.5 d and the HRT of the pretreatments, APD and ATAD were varied at 1.5 d, 2.5 d, and 3.5 d.

The 7.5 d MAD SRT was deliberately selected to be large enough to get stable methanogenesis, but small enough to see the impact of variations in the pretreatment steps in terms of gas production. The TS of the raw sludge was chosen to be 6.5% (resulting in raw sludge VS of 5.2%) as envisioned for the DC Water Biosolids Program in order

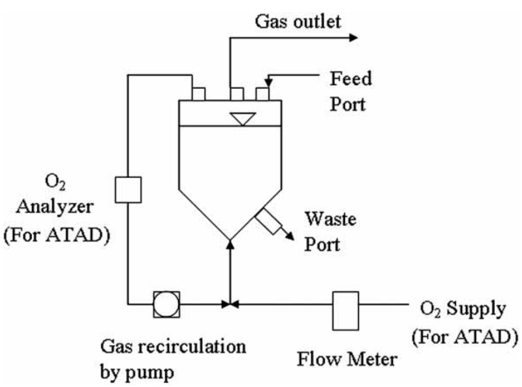

Figure 1 | Set-up for the ATAD reactor.

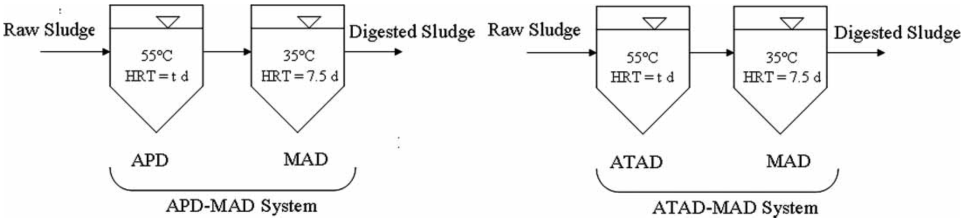

Figure 2 | Flow diagrams of APD-MAD and ATAD-MAD systems.

to reduce the reactor volumes. The choices of short HRT for the MAD and TS for the raw sludge as explained above resulted in higher VS loadings for this study compared to generally accepted range.

In addition to these two processes a conventional MAD as a control process was operated at 10 d HRT, 5.2 g VS/L/d OLR and at 35 °C using the same raw sludge.

Table 1 gives the operating conditions for APD-MAD and ATAD-MAD processes operated at different pretreatment HRTs.

Methods

Total solids (TS), volatile solids (VS), chemical oxygen demand (COD), soluble COD (sCOD), ammonia and alkalinity of the feed and effluents were measured for all the reactors according to Standard Methods (APHA 1998). pH was measured daily using Denver Instrument Model 250 pH meter. Oxidation Reduction Potential (ORP) of the ATAD effluent was measured using Accumet pH/mV/Temperature Meter (Pittsburgh, PA). O_2 content of the recirculation gas was measured for the ATAD reactor by using a Maxtec Oxygen Analyzer OM-25 AE (USA).

VFAs were measured in the solution phase of each reactor. Samples were filtered through a 0.45 μm filter and frozen prior to analysis. VFAs were measured using a Shimadzu Gas Chromatograph (Model GC-2010) with flame ionization detector (FID) together with Restek Stabilwaxfi-DA capillary column (USA). Headspace methane (CH_4) and carbon dioxide (CO_2) were analyzed on an SRI 8610C Gas Chromatograph with a thermal conductivity detector (TCD). Helium was used as the carrier gas at a flow rate of 8 ml/min.

RESULTS

In order to select proper oxygen input for the ATAD reactor, two different O_2 flow rates at 17.5 and 35 ml/min were first evaluated. These O_2 flow rates were used to facilitate solubilisation, fermentation and create autothermal conditions. Pure oxygen was added to the headspace gas recirculation system. The corresponding oxygen inputs as volume of O_2 per volume of reactor per hour were 0.105 and 0.210 v/v/h, respectively. These two different flow rates resulted in two different oxygenation states; anaerobic aerated condition and anoxic condition (McIntosh & Oleszkiewicz 1997). The oxygen input of 0.105 v/v/h corresponds to anaerobic aerated conditions (ORP levels less than −300 mV). On the other hand, the oxygen input of 0.210 v/v/h corresponds to anoxic conditions (ORP levels 0 to −250 mV).

Table 1 | Operating conditions for the APD-MAD and ATAD-MAD systems

	Acid/gas phase digestion			Dual digestion		
Pre-treatment	**Run 1**	**Run 2**	**Run 3**	**Run 4**	**Run 5**	**Run 6**
Temperature (°C)	55	55	55	55	55	55
HRT (d)	1.5	2.5	3.5	1.5	2.5	3.5
OLR (g VS/L/d)	34.8	20.9	14.9	34.8	20.9	14.9
O_2 input (m^3/kg VS added)	–	–	–	0.073	0.122	0.171
O_2 input (v/v/)h	–	–	–	0.105	0.105	0.105
MAD						
Temperature (°C)	35	35	35	35	35	35
HRT (d)	7.5	7.5	7.5	7.5	7.5	7.5
OLR (g VS/L/d)	5.9	5.5	5.3	5.4	5.3	5.2

It was observed that an oxygen supply of 0.210 v/v/h produced corresponding VFA concentration of 7,300 mg/L as acetic acid (HAc). Under the same conditions, the ammonia concentration increased by 132%. For the lower oxygen supply (0.105 v/v/h), both VFA and ammonia levels were lower. The biological heat of oxidations were calculated to be 14,300 J/g VS removed and 15,900 J/g VS removed for the oxygen flow rates of 0.105 v/v/h and 0.210 v/v/h, respectively.

When comparing the two oxygen flow rates for the overall dual digestion process, the higher oxygen input did not have a significant effect on overall VS removal, and biogas yield. It would appear that while it would be favourable to add more oxygen to attain required autothermal conditions to meet Class A requirements, a lower supply of oxygen would help produce a more favorable net energy balance (since the overall VS removal is similar), with similar methane produced in the downstream anaerobic digestion process and less energy used to supply the oxygen (Aynur *et al.* 2009). Therefore, for Run 4, Run 5 and Run 6, O_2 input of 0.105 v/v/h was chosen and applied to the ATAD reactor for this study.

VFA production is a key parameter in determining the efficiency of acidogenic phase, showing the extent of hydrolysis as well. As observed in Figure 3, both total VFA (tVFA) concentration (reported as mg/L acetic acid) and sCOD concentration increased when the HRT was increased from 1.5 d to 2.5 d for acid-phase digestion. On the other hand, there was a drop in tVFA and sCOD levels when the HRT was further increased to 3.5 d. ATAD follows the similar trend. When the HRT is increased from 1.5 d to 2.5 d for ATAD, both tVFA and sCOD levels increased. A drop in tVFA was observed when the HRT was further increased to 3.5 d. On the other hand, there was no difference between Run 5 and Run 6 statistically in terms of sCOD concentration. The data points in the figures are an average of 7 to 10 observations and the bars show the standard deviation for each data point.

Table 2 presents the VFA composition for all six runs. It would appear that the acetogenesis reactions for the ATAD process was more complete at the respective HRTs compared to the acid-phase process. For acid-phase digestion, the acetic acid content of the VFAs decreased from 53 to 32% of tVFA with propionic acid content being in the range of 20 to 29%. For the ATAD reactor, Run 4 and Run 5 had similar acetic acid content with 71 and 70%, respectively and the same propionic acid content of 8%.

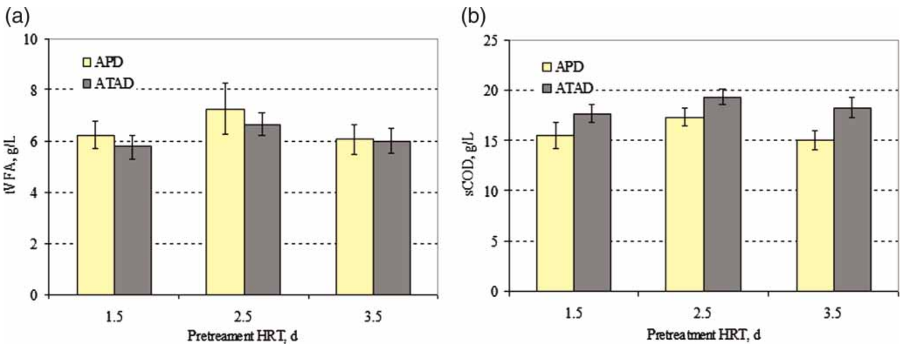

Figure 3 | (a and b) Total VFA and sCOD levels for APD and ATAD at different HRTs.

Table 2 | Volatile fatty acid composition of raw sludge and pretreatment effluents

VFAs mg/L acetic acid	Raw Sludge	APD			ATAD		
		Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
Acetic	1,529 ± 50	3,306 ± 113	3,465 ± 158	1,951 ± 100	4,206 ± 298	4,644 ± 256	3,762 ± 194
Propionic	570 ± 42	1,559 ± 71	1,486 ± 105	1,768 ± 110	446 ± 59	535 ± 47	658 ± 65
Butyric	58 ± 8	306 ± 31	362 ± 22	458 ± 28	296 ± 25	350 ± 25	430 ± 28
Iso-butyric	121 ± 15	460 ± 20	1,059 ± 81	1,082 ± 106	444 ± 42	484 ± 32	501 ± 22
Valeric	8 ± 9	491 ± 28	589 ± 32	693 ± 46	414 ± 29	520 ± 28	650 ± 42
Iso-valeric	49 ± 6	114 ± 27	304 ± 24	121 ± 9	100 ± 17	117 ± 22	–
Total VFAs	2,335 ± 78	6,236 ± 138	7,265 ± 259	6,073 ± 194	5,906 ± 275	6,650 ± 264	6,001 ± 245

For Run 6, there was a significant decrease in acetic acid content to 63% and an increase in propionic acid content to 11%. These results show that propionic acid production was increased when the HRT was increased to 3.5 d for Run 6 for ATAD. In addition propionic acid accumulation was much higher in the acid-phase digestions when compared to the ATAD. Products of acidogenesis stage are important for the performance of the methanogenic stage and the whole system. It has been shown in the literature that, propionic acid accumulation would have an inhibitory effect on methanogens. According to Yeole *et al.* (1996), propionic acid concentration of 5,000 mg/L at pH 7 caused a decrease in methane yield in the range of 22–38%. It is reported in the literature that propionic acid values in the range of 0.8 g/L to 3.0 g/L are tolerable for anaerobic reactors (Speece 1996; Ma *et al.* 2009). Although the propionic acid concentration for APD was much higher than that of ATAD, the levels observed in this study were still not high enough to cause inhibition in the downstream MAD.

It is expected to see a decrease in methane content of biogas together with decrease in methane yield when the HRT is decreased for acid-phase digestion, since methanogenic population becomes growth limited at shorter retention times. As can be seen from Figure 4, there is a significant increase in methane yield when HRT is increased from 2.5 to 3.5 d. This seems to be the critical point at which methanogens start not to be growth limiting. It can be observed from high CO_2 content of the biogas (Figure 4), with HRTs of 1.5 and 2.5 d, that methanogenic bacteria were being washed out of the system. These results are in compliance with the higher levels of tVFAs and sCOD observed for Run 1 and Run 2 when compared to Run 3. Therefore, it can be said that best working conditions for acid-phase digestion was observed for Run 2 with 2.5 d HRT, 67% CO_2 content in biogas, methane yield of 0.12 L CH_4/g VS removed, and tVFA levels of 7,264 mg/L as acetic acid.

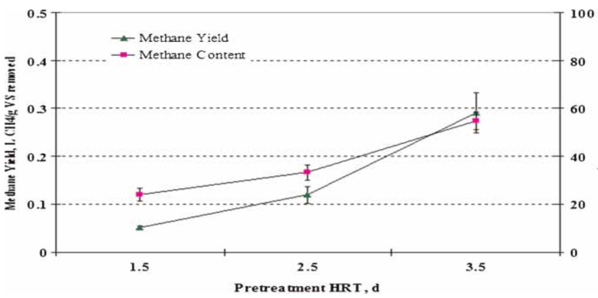

Figure 4 | Methane yield and methane content for APD for different HRTs.

In addition to comparison of the pretreatment steps, a comparison of the effects of these pre-treatment conditions on downstream anaerobic digestion in terms of methane yield would be helpful. For this purpose, the effluents from the six runs were fed to a mesophilic anaerobic digester operated at 35 °C with fixed HRT of 7.5 d.

As illustrated in Figure 5, when the comparison is made in terms of L CH_4/g VS added, there was no statistical difference in gas production between Run 1 and Run 2. On the other hand, there was a drop in methane yield when the HRT was increased to 3.5 d for the APD-MAD process. For the ATAD-MAD process, methane yield increased when the HRT was increased from 1.5 to 2.5 d and there was a drop in methane yield when the HRT was further increased to 3.5 d. The drop in methane yield for 3.5 d HRT was also observed when the comparison is made in terms of L CH_4/g VS removed, demonstrating higher efficiency for dual digestion in terms of biogas production for HRTs of 1.5 and 2.5 d. This drop in acid (Table 2) and methane yield for the DD would suggest that the total oxygen input (0.171 m^3 O_2/kg VS added) in the 3.5 d HRT is counterproductive to optimizing acid and

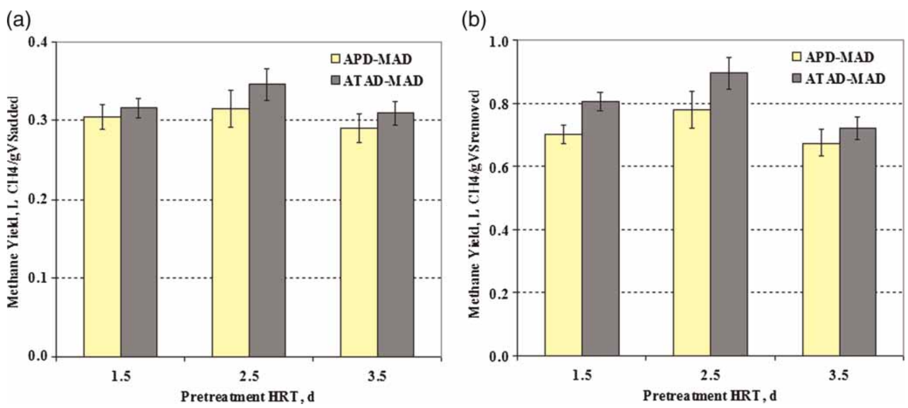

Figure 5 | (a and b) Methane yield for the MAD reactors.

subsequent methane formation, with the volatile fatty acids being used in non-methanogenic pathways. Therefore, the drop in VFAs in ATAD reactor and the subsequent drop in methane yield in MAD reactor are a consequence of the high amount of O_2 input to the ATAD reactor rather than the result of increased HRT. Oxygen input of 0.171 m^3 O_2/kg VS added resulted in acetic acid being consumed rather than being produced.

For acid/gas phase digestion, methane yield increased in terms of L CH_4/g VS removed when the HRT increased from 1.5 to 2.5 d, and then decreased for HRT of 3.5 d. Similar trend was observed with VFA production for this system. This is expected, since improved gas production is a result of the VFAs produced in the pretreatment step and as mentioned before highest VFA production is observed at 2.5 d HRT for APD. Furthermore as shown in Figure 4(b) the separation of acids and methane steps for both pretreatment processes were optimized at the 2.5 d SRT with the highest methane yields being produced in the 7.5 d MAD. For the conventional MAD process, the methane yields were 0.45 ± 0.043 L CH_4/g VS removed and 0.22 ± 0.021 L CH_4/g VS added with much lower methane content of the biogas, 60 ± 1.6%.

A comparison between APD-MAD and ATAD-MAD systems in terms of overall VSr and COD removal (CODr) was made having the same overall HRTs. As can be observed from Table 3, there was no considerable difference in VSr and CODr for APD-MAD and ATAD-MAD systems within the same overall HRTs. The VS removals were higher for the APD-MAD and ATAD-MAD processes when compared to conventional MAD which had a VS removal of 48%. The selection of the processes should be based on relative merits of using an autothermal process to produce heat from oxygen versus anaerobic acidogenesis.

During the period of one feed and the next, the digesters can be considered a closed system and the mass balance for each digester can be performed for a balanced system. Mass balance analysis was conducted taking into account the mass of COD in the influent and effluent of the digester and the volume and composition of biogas produced for anaerobic digesters or oxygen consumed for aerobic digesters. The stoichiometric theoretical value of 0.35 L CH_4/g COD at STP conditions was used for the mass balance calculations.

Table 4 | COD mass balance for the APD-MAD process Run 1

	COD_{inf} (g/d)	COD_{eff} (g/d)	COD in CH_4 (g/d)	COD Error (g/d)	% Error
APD	169.6	164.2	1.9	3.5	2.1
MAD	131.4	66.7	61.7	3.4	2.6

The COD mass balances for APD-MAD process Run 1 (Table 4) as well as all the other processes studied were found to be within a 10% error band.

CONCLUSIONS

The following are the conclusions from this research:

- The optimum separation of acid and methane bacteria for both pretreatment processes occurred at the 2.5 d HRT.
- The optimum operating conditions for separation of acid and methane bacteria for the APD-MAD system was observed at 2.5 d HRT of pretreatment step. APD pretreatment had 67% CO_2 content in biogas, methane yield of 0.12 L CH_4/g VS removed, and total VFA levels of 7,264 mg/L as acetic acid. When the comparison was made in terms of biogas production of the downstream mesophilic anaerobic digester, the highest methane yield in terms of L CH_4/g VS removed was observed when the HRT for the pretreatment step was 2.5 d as well.
- For the dual digestion (ATAD-MAD) process, methane yield increased when the HRT of ATAD increased from 1.5 d to 2.5 d. There was a significant drop in methane yield when the HRT of ATAD was increased further to 3.5 d. The drop in methane yield would suggest that the overall oxygen input in the 3.5 d HRT is counterproductive to optimized methane yields. This drop was

Table 3 | Overall VS reduction and COD reduction for APD-MAD and ATAD-MAD systems

	Total HRT = 1.5 + 7.5 d		Total HRT = 2.5 + 7.5 d		Total HRT = 3.5 + 7.5 d		HRT = 10 d
	APD-MAD	ATAD-MAD	APD-MAD	ATAD-MAD	APD-MAD	ATAD-MAD	Conv. MAD
VSr (%)	51.5 ± 2.0	53.1 ± 1.8	52.3 ± 1.6	53.8 ± 1.2	57.9 ± 1.6	57.1 ± 1.8	48.1 ± 1.1
CODr (%)	50.8 ± 1.7	50.9 ± 2.9	52.7 ± 1.4	53.9 ± 3.4	54.1 ± 3.6	54.1 ± 3.9	42.3 ± 1.2

observed when the comparison was made in terms of L CH_4/g VS removed as well demonstrating higher efficiency for dual digestion in terms of biogas production for HRTs of 1.5 and 2.5 d. These results are in agreement with the higher levels of total VFAs and sCOD observed for the two HRTs. The drop in acid and methane yield observed at 3.5 d HRT for the DD would suggest that for the total oxygen input (0.171 m^3 O_2/kg VS added) for this condition, acetic acid started to be consumed rather than produced, with the volatile fatty acids being used in non-methanogenic pathways. Therefore, the drop in VFAs in ATAD reactor is a consequence of high amount of O_2 input to the ATAD reactor rather than the result of increased HRT.

- When the VFA composition for APD and ATAD were compared, acetogenesis reactions appear to be more complete for the ATAD step compared to the APD step for similar HRTs.
- Both processes at all pretreatment HRTs investigated were able to produce higher overall VSr when compared to conventional digestion process.
- When deciding upon merits of the two processes, site-specific engineering analysis should be performed to evaluate whether it is sufficient to compensate the cost of aeration with relative autothermal heat produced for ATAD, compared to operating an APD process at 2.5 d HRT or lower.

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