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High solid temperature phased anaerobic digestion from agricultural wastes: Putting several reactors in sequence

M. Dooms^{a,b}, H. Benbelkacem^a, P. Buffière^{a,∗}

^a Univ Lyon, INSA-Lyon, DEEP Laboratory – Wastes Water Environment Pollutions, EA 7429, 9 rue de la physique, F-69621 Villeurbanne Cedex, France ^b Arkolia Energies, 16 rue des vergers, F-34130 Mudaison, France

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A B S T R A C T

High solid anaerobic digestion of agricultural wastes (manure and grass silage) was tested on temperature phased anaerobic digester (TPAD) at lab-scale. A 4 days hyperthermophilic hydrolysis was followed by a 15 days methanogenesis. The TPAD was divided into 4 or 5 tanks in series without recycling: 2 sectors for hydrolysis (2 days HRT each) and 2 or 3 sectors for methanogenesis (5+10 or 3×5 days HRT). Two different combinations of temperatures (65 °C−37 °C, 65 °C−55 °C) were investigated to assess the process efficiency.

For all tested conditions, the TPAD operated steadily with a methane productivity of 235 +/−3 mLCH4 $_{\rm STP}$ g_{VS}⁻¹. Using four or five reactors in series enabled to identify that during the two first days of hydrolysis, the hydrolysis yield and acidification yield were much higher (22% and 13%, respectively) than during the next two days (2% and 2% respectively). Moreover, concerning methanogenesis it appeared that the retention time may be shortened by 5 days with little loss in methane production (11%). In the methanogenic sectors, the uptake of volatile fatty acids and the methane production were higher at 55 ◦C than 37 ◦C.

The results suggest that the hydrolytic sector could be limited at 2 days HRT, while the methanogenic sector would support a 10 days HRT with the feedstocks used.

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1. Introduction

Anaerobic digestion (AD) of agricultural wastes is a sustainable process allowing not only to lower the emissions of greenhouse gases and odors from livestock wastes, but also to turn part of it into a valuable energy source, namely biogas. Though used for decades, research is still required for further process improvement. Among these improvements, high-solids AD (also called dry AD) and temperature phased AD (TPAD) are promising ways [\[1\].](#page--1-0)

TPAD usually relies on two sectors, of which the first one is dedicated to hydrolysis with a short retention time and the second one to methanogenesis with a longer retention time. It is called temperature phased since hydrolysis is favored by higher temperatures, while methanogenesis requires lower temperatures [\[2,3\].](#page--1-0)

Han et al.[\[4\]](#page--1-0) showed on primary sludge that TPAD (thermophilic stage followed by amesophilic stage) enabled similar performances as one-step reactors while cutting by half the retention time. Since then, several works on sludge confirmed the advantages of such

∗ Corresponding author. E-mail address: pierre.buffiere@insa-lyon.fr (P. Buffière).

<https://doi.org/10.1016/j.bej.2017.11.011> 1369-703X/© 2017 Elsevier B.V. All rights reserved. a technology on these substrates. For digesting waste activated sludge, the advantage of using a rather short Hydraulic Retention Time (HRT) in the first (thermophilic) stage is commonly recognized. A 3 days HRT in the thermophilic stage, followed by a 12 days HRT in the mesophilic stage was found optimal by Riau et al. [\[5\]](#page--1-0) and Wu et al. $[6]$, while other authors found that a 2-days HRT was sufficient to provide optimal performance [\[7\].](#page--1-0) Yu et al. [\[8\]](#page--1-0) found that a 4 days thermophilic stage at 45° C followed by a 16 days mesophilic stage was the most efficient among the tested conditions (2–6 days in the 45 ◦C stage, 16 and 20 days in the mesophilic stage). In many cases, an important production of methane (and vs removal) is observed in the thermophilic stage when operating over 3 days HRT [\[2,9–11\].](#page--1-0)

Other substrate than municipal sludge were also investigated. Orozco et al. [\[12\]](#page--1-0) have tested the effect of thermophilic hydrolysis (55 \degree C) in continuous mode ad various HRT on the methane potential of grass silage. The highest methane production was obtained after 4 days hydrolysis. Fernandez-Rodriguez et al. [\[13\]](#page--1-0) had similar results on organic fraction of solid municipal wastes, with a 35–45% increase in methane productivity with a 4 days HRT thermophilic stage followed by 10 days mesophilic stage compared to a singlereactor systems with similar retention time.Aspecific attention has to be paid to feedstocks that are subject to a strong acidification. Wu et al. [\[6\]](#page--1-0) found no improvement in TPAD systems fed with oily food wastes due to pH instability, while the thermophilic stage of a codigestion system treating sewage sludge and sugar beet residues was partially acidified after 6 days HRT [\[14\].](#page--1-0)

These studies show that TPAD brings improvements with short retention times. The application of this technology for agricultural wastes is however less documented than sludge. Lv et al. [\[11,15\]](#page--1-0) showed the advantages of using a TPAD for treating manure and the crucial role of microorganisms selection by chemo-physical conditions.

Concerning high solid AD, agricultural substrates may have relatively high total solid (TS), thus being adapted for such treatments. High solids reactors operate with higher TS content and require less water addition than conventional reactors. The volume of these reactors is lower than conventional wet systems while keeping good performances. Sung and Santha [\[16\]](#page--1-0) showed that TPAD system was applicable to dairy cattle wastes and pointed out that the TS content may inhibit the reaction when over 11%, but hardly does below. The combination used was 55 ◦C in hydrolysis (4 d HRT) and 35 ◦C in methanogenesis (10 d HRT), and with 10% TS the methane recovery was 215 mLg_{VS}⁻¹.

The compartmentation of the methanogenic sector has been studied by several authors $[17,18]$ at 55 °C. Several partitions of the retention time were tested, splitting methanogenesis in two tanks in series. The ratios tested were: 90/10, 80/20,70/30, 50/50, 30/70, 13/87, indicating the percentage of retention time spent in each methanogenic sector, respectively. It was found that splitting brings improvement. The setups 90/10 and 80/20 brought 11% increase in the methane production compared with singlestep reactor, while 70/30 and 50/50 brought between 13 and 18% increase. However, the HRT ratio 30/70 induced instability, while 13/87 brought no more improvement in biogas production and a lower methane content. Using several tanks in series for the methanogenic stage of AD could thus bring improvements as long as the retention time in the first methanogenic sector is long enough.

Interestingly, and due to the pasty consistency of the medium and the limited diffusion [\[19\],](#page--1-0) a large reactor can be considered as several smaller reactors (or sectors) working in series with limited back mixing. This can be turned out into an advantage, since triggering stirring parameters may enable to adjust the substrate to micro-organisms ratio of each sub-reactor. That, while promising perspective for industrial plants management, requires a good understanding of the behavior of each sub-sector.

Together with retention time, temperature is a key parameter. Ge et al. [\[20\]](#page--1-0) showed that a thermophilic hydrolytic stage prior to mesophilic digestion of primary sludge increased vs destruction from 44% to 54% compared to a similar system operating at 37° C. This was explained by a higher hydrolysis rate. Similar observations were made on waste activated sludge [\[21\],](#page--1-0) where highest temperatures (up to $65 °C$) brought the highest solubilization extent, while keeping methane production low. Beyond the raw hydrolytic rate, the VFA profile is also impacted. Indeed, Verrier et al. [\[22\]](#page--1-0) showed that higher temperatures could favor the production of acetate and reduce the amount of propionate, thus improving the overall performance of the system.

Other works have studied the specific effect of temperature above 55 °C in this sector, from 55 °C [\[23\],](#page--1-0) to 65 °C [\[24,25\],](#page--1-0) and even higher $[26,27]$. The benefits induced by this 10 °C difference were confirmed to be far beyond only thermodynamic improvement of kinetics. Indeed, the authors found that a strong biological shift appeared in the microbial populations as suggested by the lower methane production and the VFA profile modification. A 65 ℃ temperature was found to bring the highest solubilized COD while keeping the methane production low.

In the methanogenic stage, 37 ◦C and 55 ◦C are the more commonly used temperatures. A 55 ◦C hydrolysis is followed by a 37 ◦C methanogenesis, while 55 ◦C methanogenesis is rather for one step AD or TPAD with hyperthermophilic hydrolysis.

Relying on this information and its industrial background, the company Arkolia Energies patented a TPAD reactor (ArkoMetha TM) that is designed as a succession of four to five sectors. It is a highsolids technology dedicated to organic solid wastes. In the common design proposed by the company, the hydrolytic stage is divided in two sectors, while the methanogenic stage can be divided in two or three sectors. In the industrial setup, walls physically separate the sectors, and openings are provided to enable the digesting medium to flow through. The temperature of the hydrolytic sectors is 65 ◦C since it brings better hydrolysis, while the methanogenic part can be operated from 37 °C to 55 °C.

This study simulates the behavior of such a plant design by using several tanks in series at the bench scale, each tank being assimilated to a "sector" of the industrial plant. The objective is to investigate the effect of the retention time in each stage and subsectors on the hydrolytic and methanogenic activity, and to find the optimal combination for temperature.

2. Materials and methods

2.1. Experimental setup

Three different runs were performed using 4 or 5 tanks in series. In the following, each tank is named "sector", and the terms "reactor" or "line" are associated to the combination of the 4 or 5 sectors in series. The total retention time in each line was 19 days for all runs. The two first sectors of each line were dedicated to hydrolysis, with a temperature of 65 \degree C and a shorter retention time (2 days per sector). The methanogenic sectors operated at a lower temperature (37 \degree C or 55 \degree C depending on the runs) and longer retention time (5–10 days depending on the sectors). This configuration simulated the operation of a TPAD. The experimental setup of the tree runs is displayed on [Fig.](#page--1-0) 1.

Each sector was a 1 L glass vessel with a water jacket for temperature control. A Ritter Milli-gas counters (MGC®) was used to measure the biogas produced by each sector. A septum on the top of each reactor enabled gas sampling in order to assess the gas composition using a microGC 3000 (Agilent). A Pt100 temperature probe was placed in each reactor to monitor the temperature at $\pm 2^{\circ}$ C of the desired value.

The reactor content ranged between 400 g and 800 g depending on the sectors. The feed loads were calculated according to the desired retention time in each sector. Each sector was fed, stirred and purged with N_2 once a day. This was made necessary to prevent oxygen from disturbing the operation of the anaerobic consortia during the feeding/withdrawal process. In addition, the biogas composition of each sector was measured before and after each opening. Consequently, the daily methane production can be accurately calculated as the sum of the produced methane flowing from each sector (knowing biogas flow rate and composition) and of the methane accumulating in the headspace of each sector.

The first sector of each series was fed with the feedstock described hereafter ([Table](#page--1-0) 1). Each sector but the first one was fed with the digestate from the previous sector. Logically, the daily feeding operations began with the last sector of each line, thus avoiding back mixing.

2.2. Feedstock and operations

Runs were fed with a mixture including cattle slurry, manure and grass silage, as presented on [Table](#page--1-0) 1. The proportions are given Download English Version:

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