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Biogas Recovery from Anaerobic Digestion of Selected Industrial Wastes

Pengchong Zhang

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Abstract

Treating industrial wastes requires large amount of capital investment and also creates environmental concerns from several aspects. One of the techniques to reduce these concerns is anaerobic digestion. By applying anaerobic digestion technique, the organic waste from various industries could be removed and recovered to renewable energy, mostly in the form of biogas (methane); therefore, waste treatment process shifted from a cash negative process to an economic beneficial process. In this chapter, various kinds of industrial wastes were selected and descripted, followed by a gradually progressive order. The selected waste streams include paper mill wastes, brown grease, and corn ethanol thin stillage. Due to their dissimilar properties, the motivations of treating these wastes are also different. Paper mill effluents and solid wastes contain large portion of refractory or toxic chemicals and fibers; their bio-treatability, organic removal efficiency, and substrate utilization rate have been investigated and the results showed good anaerobic treatability. Brown grease is already well-known as a treatable substrate; therefore, the economic effort by using a high-rate anaerobic digester will be more important. For thin stillage, a systematic design of incorporated anaerobic digestion process was analyzed; the cost analysis was also conducted; and the possibility of using this technique as an add-on system was discussed.

Keywords: anaerobic digestion, biogas, brown grease, corn-to-ethanol, paper mill waste

1. Introduction

Among the increasing energy consumption, natural gas (mainly methane) demand is increasing. Methane (CH_4) is created both in the natural environment and through various human activities. Derived from the decay of organic material, CH_4 is easily produced and abundant. Although in most cases CH_4 created from human activity cannot completely replace significant energy needs, it could lower the costs and decrease a facility's reliance on the electrical grid [1].

Therefore, biogas as a sort of renewable energy is gaining more attraction throughout several nations of the world [2]. Biogas is the gaseous emission produced by the breakdown of organic matter in the absence of oxygen. It is a mixture of CH_4 and carbon dioxide (CO_2) along with other trace gases (H_2S, H_2, SO_2, etc.). CH_4, the primary component of natural gas (98%), makes up 55–90% by volume of biogas (depending on the source of organic matter and conditions of degradation). CH_4 is the only constituent of biogas with significant energy value. The inert diluents of CO_2 and nitrogen lowers the calorific content of the gas, while the corrosive nature of hydrogen sulfide (H_2S) wears down the anaerobic digester and pipes involved in the gas distribution. Biogas has a very wide industrial application range, includes heat combustion systems, motors, turbines, and fuel cells, and it can also be sold as a by-product separately. Biogas can be generated by anaerobic treatment of organic wastes. In the past decades, researchers have been conducting massive experiments on evaluating the conversion of miscellaneous wastes such as animal manure, municipal solid waste, energy crops, municipal biosolids, and food waste to biogas [3, 4]. In this chapter, three kinds of wastes were selected for investigation: bleaching and pulping effluent from paper mill, brown grease from food waste, and corn stillage from the bio-ethanol plant.

1.1. Introduction of anaerobic digestion (AD) process

Anaerobic digestion (AD) is the consequence of a series of metabolic interactions among various microorganisms. It occurs in three stages: hydrolysis/liquefaction, acidogenesis, and methanogenesis [3, 5]. In these three stages, complex organic materials are converted to CH_4 and CO_2 in the absence of O_2 *via* activity of several groups of anaerobic microorganisms. Firstly, fresh organic matter was hydrolyzed to soluble particles. Afterwards, soluble organic matter was biodegraded to volatile fatty acids (VFAs) and alcohols by a heterogeneous microbial population called acidogens. Finally, a limited number of organic compounds were used as carbon and energy sources and to be transferred to CH_4 by microbes called methanogens. AD process is an effective proven technology for handling and treating municipal or industrial wastes and effluents, for the generation of district heating and electricity supplies, as well as for clean environment.

1.2. Important operating parameters in AD process

During the AD process, many operating parameters must be controlled to optimize the microbial activity and keep the system efficiency stable and superior. Ideally, the performance of an AD process should be evaluated by observing those important parameters.

pH and temperature are important factors for keeping functional AD process. Generally, anaerobic process happens in neutral pH range (pH 6.5–7.6) [6], because anaerobic bacteria, especially the methanogens, are sensitive to the acid concentration within the digester and their growth can be inhibited by acidic conditions. Based on pH level, two temperature ranges, named mesophilic (30–45°C) and thermophilic (45–65°C), were commonly applied in industrial fields [7]. Redox potential (ORP) is a parameter to reflect changes in oxidizing or reducing agents; it represents the oxygen inhibition situation during the AD process. At the same time, the concentration of dissolved oxygen (DO) could also be monitored as an indicator of oxygen inhibition.

The concentration of specific volatile fatty acids (VFAs) and total alkalinity (ALK) can give vital information on the status of AD processes. VFA is an important intermediate product in the AD process, which should be converted to CH_4 finally, and proper amount of ALK is used to offset the excess VFA to keep the pH value at the stable level. System retention time, including hydraulic retention time (HRT) and solid retention time [(SRT), in solid digesters] may influence the system performance. A longer retention time comes with a higher organic mass removal, but it can also lead to possible VFA accumulation and decrease of system treatment efficiency. The required retention time for the completion of AD reactions varies with different reactor types, temperature, and waste composition.

Organic loading rate (OLR) is the measurement of the biological conversion capacity of the AD system. Feeding the system above, its sustainable OLR will result in low biogas yield due to the accumulation of inhibiting substances such as VFA [8]. Generally, OLR was calculated based on the concentration of chemical oxygen demand (COD) of volatile solids (VSs). The composition of OLR may contain biodegradable organic loading and refractory organic loading; this composition will affect the biogas yield and quality and the organic removal efficiency as well.

Biogas production is one of the main purposes of AD process. Tracking the biogas production is a widespread online measurement in AD control systems. A low biogas production may indicate accumulation of some inhibitive intermediate compounds. The measurement of CH_4 is important because it is the major energy output of the AD system. The concentration of tract gases such as H_2S in produced biogas reflects the current presence and degradation of sulfide-containing compounds. H_2S has a certain amount of toxicity; thus, its concentration needs to be cautious to not reach inhibiting levels when treating rich H_2S substrates.

1.3. Current research background

The technology of AD has developed in many aspects [8]. There are a lot of studies that use AD to treat different kinds of municipal, agricultural, and industrial wastes. Gunaseelan [9] has summarized the application of AD to over 100 kinds of wasted biomass to recover CH_4. Appels et al. [10] have applied AD technology in wastewater treatment plant (WWTP) to treat the waste-activated sludge and successfully recovered CH_4 from those discarded organic matters to save over 50% of the WWTP cost. Hansen et al. [11] have used a continuous stirred tank reactor to treat high-ammonia swine manure and obtained a CH_4 yield of 0.022–0.188 m^3-CH_4 kg-VS-1. Bouallagui et al. [12] and Zhang et al. [13] also applied a batch AD reactor to food waste. Angelidaki et al. [14] have defined the measurement protocol for biomethane potential (BMP). Furthermore, in the study of De Baere [15], the anaerobic treatment capacity of solid waste in Europe was over 1 million tons in the year 2000.

To develop and extend the application of AD technique to large-scale fabrication plants and industries, three kinds of industrial wastes were selected to be treated in a pilot-scale AD process. The waste substrates include paper mill effluents (comes from different paper making processes), brown grease (a kind of common food waste), and thin stillage (a kind of intermediate from corn grain-to-ethanol process). All the selected waste substrates come from real industries and practical plants. The reason for choosing these industrial wastes included, firstly, for these selected wastes, the traditional treatment technique seems inefficient. For example, for paper mill effluents, the traditional treatment technique is activated sludge process, which could

remove up to 90% of biochemical oxygen demand (BOD) but the chemical oxygen demand (COD) removal efficiency is just in the range of 20–50% [16–19]. Secondly, the United States Environmental Protection Agency (US EPA) have strict and specific policies about these industrial wastes, such as the US EPA CMOM (capacity, management, operation, and maintenance program, including grease control program), the US EPA final pulp and paper cluster rule and amendments, the US EPA CWA (Clean Water Act), the FOG ordinance/FOG management policy, and so on. That could be considered as the driving force to push industries to treat these wastes before discarding. Finally, these materials from industrial waste contain high organic content, which means they have the potential to be treated anaerobically as the energy feedstock.

2. Substrates

2.1. Paper mill effluents

Pulp and paper industry produces a large quantity of wastewater of high organic strength [20, 21]. Even with the most modern operations, about 60 m^3 of wastewater is generated for every ton of paper produced [22]. In the paper manufacturing processes, pulping and bleaching processes creates most of the wastewater streams [23, 24]. These wastewaters typically have high organic content (COD 800–4400 mg^{-1}) [24–26], high biological content (BOD 300–2800 mg^{-1}) [24–26], and high dye content (1200–6500 color unit) [24–26]. Several steps of treatment process were generally involved, including a primary clarification process to remove the suspended solids, a secondary treatment process to remove most of the air lagoons, and a final biological treatment process (aerobic) to remove the biological content (BOD5) [25, 26]. However, due to the recalcitrant chemical properties, the final effluent always still contains large amount of high molecular weight organic compounds [25].

Anaerobic treatment technique has not been widely used in the pulp and paper industry yet [27, 28]. One major advantage of anaerobic treatment is that the process is capable of treating high-organic strength streams that are not suitable for aerobic processes [30, 31]. Furthermore, it has the added benefit of lower treatment cost because the produced biogas can be diverted to energy generation [32]. Traditional treatment technique of energy-rich wastes should be avoided as far as possible mainly because of their low energy recovery efficiency [33], but the recovered biogas from anaerobic digestion process has a high methane content (60–80%) and can be directly used as fuel [34]. One of the current research issues is most of the evaluations for pulp and paper wastewater are only focused on synthetic waste stream in the lab-scale environment (reactor size 5–50 L) [29, 35–40], which makes the results less representative to large-scale industrial fabrications, a research utilizing pilot-scale system, and practical waste streams directly from the paper mill would be more helpful and relevant.

2.2. Brown grease

Using biogas as an alternative source of energy is gaining more attention globally in recent decades [41, 42]. There have been an increasing number of studies performed to evaluate the

conversion of waste streams such as animal manure, municipal solid wastes, energy crops, municipal biosolids, and food wastes to biogas [43–46]. In Europe, there are over 50 waste treatment plants using these materials to produce biogas [16, 45, 46]. For instance, ~15% of organic wastes are being converted annually in Germany [47]. The practice of converting wastes to energy provides a two-fold benefit of environmental protection and energy recovery.

Brown grease (BG) is a mixture consisting of trapped grease, sewage grease, and black grease collected in grease interceptors (traps) of restaurants and food industries [48]. In the United States, there are 1.84 million tons of BG produced every year [49]. Most collected BG eventually ends up in landfills. The landfill cost for BG is ~5 cents per pound [50]. This results in a very high direct disposal cost. In addition, the moisture content in BG can lead to soil and water pollution, making the soil sterile and unable to support plant life [51]. Because of these drawbacks, the European Union enacted a general ban on landfilling organic waste in 2005 [52]. An earlier study suggested that 14×10^6 m^3 of CH_4 could be produced in the United States annually by converting the generated BG into biogas [53]. This is a substantial amount of renewable bioenergy. Recovering the energy and eliminating the waste input to landfills yields both economic and environmental benefits [54, 55].

AD is a treatment process capable of producing biogas from organic wastes. The benefits of anaerobic digestion include smaller reactor size in terms of organic loading, lower air emissions, and a smaller amount of generated sludge compared to aerobic biological treatment [55]. Greasy wastes such as BG have been added as a lipid-rich cosubstrate in earlier AD studies for sewage sludge [56–58], municipal wastewater [59–61], and the digestible fraction of municipal solid wastes [62]. Typically, it is blended at 2–50% of the primary substrate's organic loading to improve the biogas yield and methane content [56–62]. However, higher lipid loading (>50% of the substrate) can cause long-chain fatty acid (LCFA) inhibitions [55, 61, 62], scum and foam formation, and fat clogging problems [56]. To our knowledge, there are few studies devoted to investigating the degradability and biogas production using BG alone.

2.3. Stillage from corn-to-ethanol process

Based on the increased demand of renewable energy, bio-ethanol as an alternative energy source was considered and has enormous economic and strategic advantages. In the past decade, the national total annual fuel-grade ethanol production has increased from 1.77 billion gallons (in 2001) to 13.95 billion gallons (in 2011). In 2005, 67% of this ethanol was produced from dry mill corn [63], and this percentage has kept on increasing because of the low cost of this technology [64].

In a typical bio-ethanol production process, corn mash has been fermented and distilled to produce high purity ethanol, and the fermentation residue is called whole stillage, which is centrifuged to produce wet cake (precipitate) and thin stillage (supernatant). About 50% of the thin stillage is recycled as backset. The remainder is further concentrated by evaporation to produce syrup and blended with dried wet cake to create a feed product known as distiller's dry grain with soluble (DDGS). The effluent of evaporation process was purified and recycled as water reuse.

Stillage handling is the most energy consuming process in the life cycle of corn to ethanol process. The drying and evaporation of stillage will take more than 35% of the total energy consumption [65], which makes the stillage treatment technique a main limitation of bio-ethanol making process [66]. Except for energy consumption, thin stillage is also a kind of high strength wastewater, which exhibits a considerable pollution potential [67]. Up to 20 l of stillage will be produced for each l of corn ethanol [68, 69], and the pollution potential of generated stillage can reach to a chemical oxygen demand (COD) of over 100 g L^{-1} [69].

The problems described in the previous paragraph have a significant negative impact on the industrial cost of the corn to ethanol process. Thus, a gate-to-gate life cycle assessment for thin stillage treatment was needed to provide a synergistic effect for energy recovery and cost saving. AD technique could be used to remove COD from thin stillage and also to convert the organic fraction of thin stillage into methane, which is a readily in-plant-usable energy source for ethanol industries [69]. Once this AD process was linked as a gate-to-gate life cycle to the ethanol production chain, the efficiency of the complete cradle-to-gate evaluation will be improved and the total cost will be reduced.

3. Biogas production for different substrates

3.1. Anaerobic treatability of paper mill effluents

As mentioned in Section 2.1, most of the current researches related to paper mill effluent treatment are focused on lab-scale experiments; therefore, an upscaling research is necessary to predict more comprehensive and representative results. In this study, a pilot scale sequential reactor system was introduced to evaluate the biotreatability of paper mill waste stream. Various waste streams from different paper making process were used, including liquid waste from bleaching process (DO), liquid discharge from alkaline extraction operating process (EOP), foul condensate from chemical pulping process (FC), and screw press liquor from dewatering operation process (SPL). For pH adjustment purpose, as well as improve the biodegradability, a small volume of wasted sugar water (SW) from a food processing plant was also blended in, as a co-digestion substrate used in this study.

The entire pilot system was established on a property outside of a pulp and paper mill, waste streams were obtained from the paper on a daily basis. The whole system consists of an equalization tank with a volume of 2.1 m³ to blend all substrates equalized. Before sending to the packed-bed AD column, a 0.95 m³ continuous stirred tank reactor was used for predigestion. The AD column is a cylindrical column with 1.07 m in diameter and 2.60 m in height, 85% of the AD column was packed with commercial ceramic bio-packing media. The discharge of the AD column will be fed to a 0.95 m³ aerobic tank for final aeration, and the sample was taken on each tank on a daily basis.

The evaluation lasted for 156 days and was divided into six periods according to different feeds and operating conditions. Initially, the packed-bed column was operated as a downflow digester. From the 80th day, the flow direction was changed, and the AD column is operating

as an upflow flooded-bed reactor, the HRT is about 1.7–2.4 days. The feeding and operational characteristics are summarized in **Table 1**.

The evaluation lasted for 156 days, and was divided into six periods according to different feeds and operating conditions. Initially, the packed-bed column was operated as a down-flow digester, with a recirculation ratio of 5.0. Note for this stage, there is no water retention. Beginning with the 80th day, the AD column was operated upflow direction; the HRT was kept at 1.7–2.4 days. The entire operation is built on neutral pH range (6.92–7.60, see **Table 1**) and slightly mesophilic condition (T = 31.5–34.5°C, measured for effluent, see **Table 1**).

Table 2 listed the initial characteristics of each kind of substrates. The COD concentrations for each type of substrate ranged from 2800 to 4500 mg L^{-1}. In this study, the waste streams from paper mill are mostly in liquid phase and have relatively very low solid content (TS < 1 wt%, see **Table 2**). As mentioned above, a sugar water substrate was used to adjust the pH of the mixed substrate. The sugar water (SW) is a high organic content and slightly acidic substrate (COD = 408,000 mg L^{-1}, pH = 3.99). In this study, the sugar water was blended for about 0.5 wt%.

Figure 1 shows the plots between cumulative CH_4 production and the cumulative COD digested (mass basis) against the time axis. Note the system start-up and recovery during substrate changes were not included in the figure. There are totally six linear stages (Stage I–Stage VI, see **Figure 1**) that the system has a stable and consistent CH_4 production rate; these six periods were considered as steady state periods. The CH_4 yield was calculated as the ratio of the slopes of the two curves in **Figure 1**. The values range from 0.22 to 0.34 m^3-CH_4 kg-COD-1 for the substrates evaluated.

Based on the treatability study listed above, all waste streams are readily treatable. The anaerobic treatment removed 50–65% of substrate COD. Coupled with the aerobic treatment using a CSTR ASP, the overall COD removal efficiency was 55–70%. The application of anaerobic treatment has the potential of significantly improving the energy footprints of the pulp and paper industry.

Operating periods	1	2	3	4	5	6
Duration (days)	1–36	45–81	82–135	136–142	143–148	149–156
Substrate	FC + SW	EOP + SW	EOP + SW	EOP + DO + SW	EOP + DO	EOP + DO + SPL
Flow scheme	Downflow	Downflow	Upflow	Upflow	Upflow	Upflow
OLR (kg-COD/m³d)	2.96 ± 0.70[1]	3.02 ± 0.38[1]	2.25 ± 0.81[2]	2.75 ± 0.70[2]	1.59 ± 0.48[2]	1.44 ± 0.48[2]
HRT (d)	—	—	2.44 ± 0.83	1.72 ± 0.51	2.12 ± 0.91	1.82 ± 0.55
pH in digester	6.92 ± 0.39	7.23 ± 0.11	7.42 ± 0.10	7.60 ± 0.48	7.25 ± 0.02	7.26 ± 0.09
Temperature of effluent (°C)	32.7 ± 2.6	34.3 ± 1.6	31.5 ± 3.1	34.5 ± 1.6	32.2 ± 1.8	33.3 ± 0.9

[1]Based on the volume of packing media.

[2]Based on total volume of the packed-bed digester.

Note: Day 37–44 was in maintenance and recovery mode.

**Table 1.** Pilot-scale feeding activities and conditions during six operating periods.

Parameters	Foul condensate (n = 11)*	DO filtrate (n = 8)	EOP filtrate (n = 4)	Screw press liquor (n = 13)
COD (mg L^{-1})	2973 ± 142	2886 ± 381	3901 ± 1940	4498 ± 2020
dCOD (mg L^{-1})$^{\#}$	2740	2445 ± 151	2890	609 ± 189
TS (mg L^{-1})	406 ± 104	4718 ± 522	4744 ± 532	8768 ± 7957
VS (mg L^{-1})	210 ± 14	2497 ± 346	1903 ± 136	3742 ± 1666
VS/TS ratio	0.53 ± 0.1	0.53 ± 0.02	0.4 ± 0.02	0.5 ± 0.1
TSS (mg L^{-1})	357 ± 577	868 ± 365	388 ± 127	4048 ± 1750
VSS (mg L^{-1})	339 ± 461	758 ± 339	296 ± 204	1997 ± 875
TSS/VSS ratio	0.83 ± 0.25	0.86 ± 0.04	0.79 ± 0.23	0.49 ± 0.06
Alkalinity (mg L^{-1} as CaCO$_3$)	205 ± 50	–	915 ± 263	–
pH	9.28 ± 0.18	5.19 ± 1.04	9.29 ± 0.29	8.44 ± 0.83
TN (mg L^{-1})	52.2 ± 4	4 ± 1.3	27 ± 43.3	2.3 ± 0.1
TP (mg L^{-1})	0.24 ± 0.09	6.33 ± 0.18	3.98 ± 5.22	0.41 ± 0.04
Conductivity (ms cm^{-1})	5 ± 5.7	–	4.6 ± 0.4	–
Sulfide (mg L^{-1})	52.2 ± 18.1	<0.5	<0.5	–
Sulfate (mg L^{-1})	<40	–	106 ± 23	–
Chloride (mg L^{-1})	–	–	335 ± 39	–

*n stands for sample size, i.e. testing times for raw industrial waste streams.
$^{\#}$dCOD stands for dissolved COD concentration.

Table 2. Initial characteristics of the evaluated substrates.
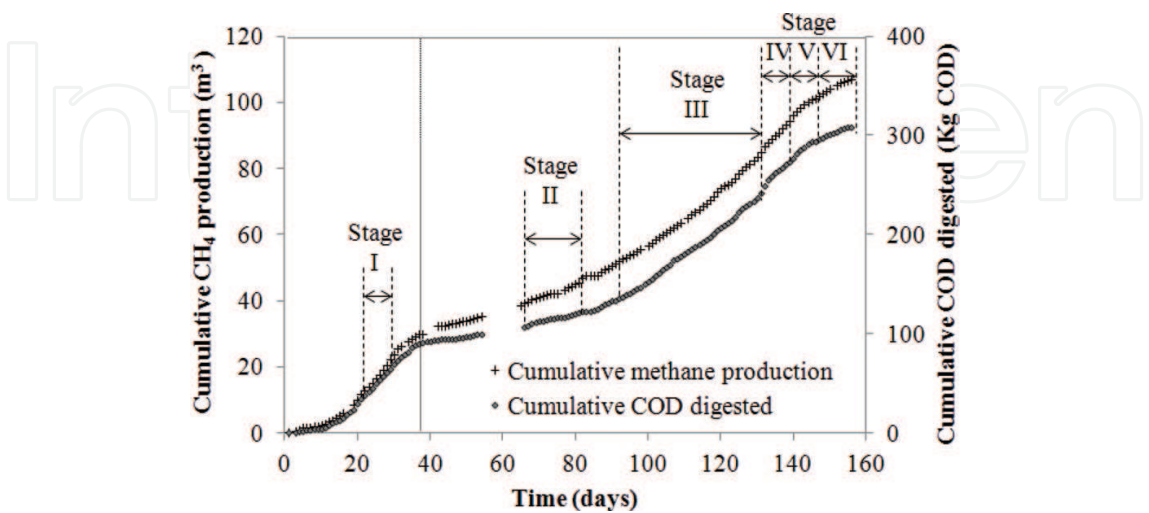
Figure 1. Cumulative CH$_4$ production at STP and cumulative COD mass digested during the evaluation period. There are six linear stages (I–VI) during which the data were used for calculating the CH$_4$ yield.

3.2. High-rate anaerobic digester to treat brown grease

The high-rate anaerobic digestion system employed in this work comprises three CSTRs and a clarifier: a balance tank (BAL), a facultative tank (FAC), an anaerobic digester (AD), and a final sedimentation tank (ST). The BAL and FAC are rectangular shaped tanks having an adjustable volume of 0.2–1.0 m³. AD is a cylindrical tank with a total volume of 7.6 m³ (1.6 m in diameter and 3.8 m in height) with adjustable reaction volumes of 4.3, 5.8, and 7.6 m³. It has a Plexiglas window at the top for observing the mixed liquor in the digester. Various liquid waste streams from paper mill wastewater including foul condensate (FC) and screw press liquor (SPL) were blended as an effort to minimize the water use in the feed. The sedimentation tank (1.5 m³) has a cylindrical shape with a conical bottom at 1:1 slope.

The evaluation period lasted for 343 days. Excluding the system start-up, maintenance, and feeding transition periods, process data were collected for 238 days. The evaluation was divided into five intensive evaluation periods (I–V). During each operating period, a steady stage (S1–S5) defined as a state with relatively consistent biogas production and organic removal) was selected for intensive measurement and data analysis. **Table 3** summarizes the evaluation schedule and the corresponding operating parameters in each stage.

	System start-up	I	II	III	IV	V
Date	4/13/11–7/26/11	7/27/11–8/7/11	8/8/11–10/24/11	10/25/11–12/7/11	12/8/11–2/29/12	3/1/12–3/21/12
Days of operation	/	1–12	13–90	91–135	136–217	218–238
Days of intensive evaluation[a]	/	1–12 (S1)	34–45 (S2)	107–133 (S3)	184–217 (S4)	218–238 (S5)
Sedimentation tank	No	No	Yes	Yes	Yes	Yes
Feeding	BG	BG	BG	BG + FC	BG	BG + SPL
Influent COD (mg L⁻¹)	/	34,510 ± 2557	56,570 ± 3894	26,570 ± 6264	33,881 ± 9176	30,200 ± 1503
Influent VS (mg L⁻¹)	/	13,965 ± 1262	23,937 ± 1625	10,139 ± 754	13,224 ± 3236	13,225 ± 1891
OLR[b]	/	2.0 ± 0.2	2.7 ± 0.3	0.8 ± 0.2	0.6 ± 0.2	0.9 ± 0.3
HRT[b]	/	7.3 ± 0.6	8.9 ± 0.9	15.2 ± 1.1	15.8 ± 1.9	11.0 ± 0.1
Activity	Seeding and initiating	Establish BG steady state	Add ST	Establish BG + FC steady state	Back to BG steady state	Establish BG + SPL steady state

[a]S1–S5 stands for five selected stages with intensive evaluation and stable data consistency.
[b]OLR and HRT in S1 and S2 were calculated based on AD only, while in S3–S5 were calculated based on AD + ST. Data were collected in five different periods for analysis.

Table 3. Feeding characteristics and reactor configuration during the evaluation.

The characteristics of BG feedstock, FC, and SPL are shown in **Table 4**. Since the BG has an extremely high organic content (~1 kg-COD kg-BG-1, **Table 4**), the feeding stream was diluted to the range of 25,000–50,000 mg L^{-1} COD. FC and SPL have a relatively low COD concentration and solid content compared with BG (**Table 4**). In addition, their mild alkalinity (**Table 4**) effectively offset the mild acidity in BG. FC is a liquid substrate with relatively low solid content (TS = 400 mg L^{-1}), its major organic content is in the dissolved phase (dCOD is >90% of total COD, **Table 4**). SPL has a TS content less than 1.0 wt%. Its dCOD concentration is <20% of total COD concentration (**Table 4**), which indicated the major organic content is in the solid phase.

The daily biogas production during the evaluation is summarized in **Figure 2**. In S1, the biogas production is 5–6 m^3 d^{-1}. The biogas production (~7 m^3 d^{-1}) was higher in S2 because of the higher organic removal. During S3–S5, the average daily biogas production was lower than S1 and S2 since the system OLR was reduced. In S3, the COD removal efficiency was higher than S4 and S5, leading to higher biogas production (~5.6 m^3 d^{-1}) compared to that in S4 and S5 (~3.5 m^3 d^{-1}) (**Table 5**). The easily digested dCOD in FC may account for this increase. Another reason for the lower biogas production was the lower OLR applied in S4 and the slightly lower organic removal in S5. Generally, the biogas production trend in S3–S5 was

Parameters	Brown grease (BG)[a]	Foul condensate (FC)	Screw press liquor (SPL)
	($\mu \pm \sigma$, n = 17)	($\mu \pm \sigma$, n = 11)	($\mu \pm \sigma$, n = 13)
COD (mg L^{-1})	910,634 ± 229,993	2973 ± 142	4498 ± 2020
dCOD (mg L^{-1})	/	2740 ± 125	609 ± 189
TS (mg L^{-1})	437,778 ± 91,348	406 ± 104	8768 ± 7957
VS (mg L^{-1})	372,111 ± 77,646	210 ± 14	3742 ± 1666
VS/TS ratio	0.85 ± 0.06	0.53 ± 0.1	0.5 ± 0.1
TSS (mg L^{-1})	/	357 ± 577	4048 ± 1750
VSS (mg L^{-1})	/	339 ± 461	1997 ± 875
VSS/TSS ratio	/	0.83 ± 0.25	0.49 ± 0.06
Alkalinity (mg L^{-1} as CaCO$_3$)	/	205 ± 50	/
pH[b]	6.51 ± 0.77	9.28 ± 0.18	8.44 ± 0.83
TN (mg L^{-1})	/	52.2 ± 4	2.3 ± 0.1
TP (mg L^{-1})	/	0.24 ± 0.09	0.41 ± 0.04
Sulfide (mg L^{-1})	/	52.2 ± 20.5	/
Sulfate (mg L^{-1})	/	<40	/
Moisture content (wt%)	56 ± 9	/	/

[a]Here BG stands for pretreated brown grease in solid phase, thus the unit of COD, TS, and VS is mg kg^{-1}.

[b]pH of brown grease was measured by suspending 100 g brown grease in 1 L tap water. Tap water has pH of 8.05 and alkalinity of 55 mg L^{-1} as CaCO$_3$.

Brown grease was used as the primary substrate and the other two liquid wastes were used as co-substrates in part of the evaluation.
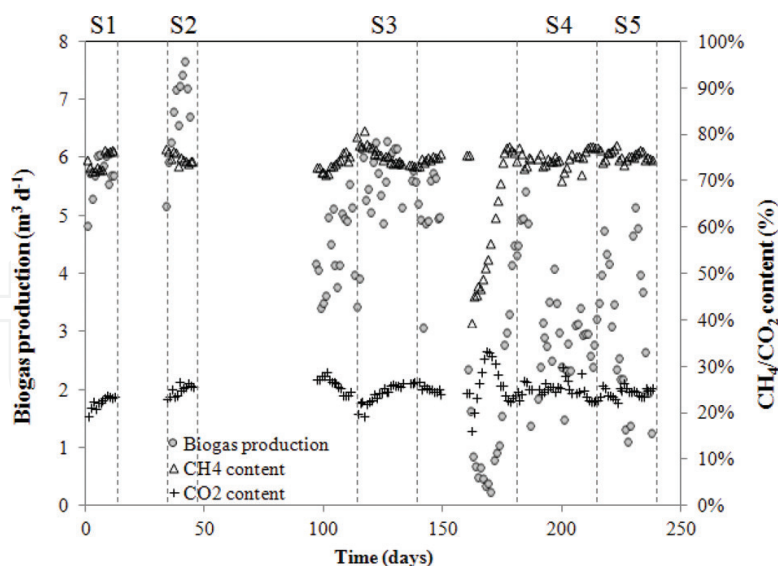
Table 4. Substrate characteristics.

Figure 2. Measured daily biogas production and CH_4/CO_2 content.

consistent with the organic removal (**Table 5**), suggesting that the biogas production was not significantly affected by the addition of co-substrate.

The pilot-scale system produced biogas of excellent quality (75% CH_4 content), with a CH_4 yield in the range of 0.40–0.77 m^3-CH_4 kg-VS^{-1}. The addition of paper mill waste streams (FC and SPL) as co-substrate did not adversely affect the CH_4 yield. BG has the industrial potential to be anaerobically treated as a biofuel feedstock and there has been an ongoing commercial effort to build large-scale digesters using BG as the primary substrate. Using BG for biofuel recovery could serve as a profitable model for converting waste to renewable energy.

3.3. AD addendum unit to improve corn-to-ethanol process

The integrated anaerobic-aerobic system employed in this work contains three CSTRs, two transfer tanks, two clarifiers, and one serious CSTR aeration basin. The receiving tank (REC) is a rectangular CSTR with total volume of 4.5 m^3 (1.2 m width × 2.5 m length ×1.5 m height). Facultative tank (FAC) is a cylindrical CSTR with total volume of 0.35 m^3 (0.6 m diameter and 1.5 m height), the operating level is adjustable from 0.15 to 0.30 m^3. Anaerobic digester (AD) is a cylindrical CSTR whose volume is 10.4 m^3 (2.1 m diameter and 3 m height) and the operating level is 7.2–9 m^3. Two transfer tanks were respectively set between FAC and AD (0.04 m^3) and between AD clarifier and aerobic basin (0.15 m^3). The aerobic basin is rectangular whose volume is 2.5 m^3 (0.7 m width × 3 m length × 1.2 m height), three baffle plates were placed inside to divide the whole basin into four equal-sized serious tanks (0.6 m^3 for each). Two clarifiers were set after AD (anaerobic clarifier) and after aerobic basin (aerobic clarifier), respectively, both of them have a volume of 0.7 m^3.

Stillage feedstock was obtained daily from the ethanol plant. Generally, the raw stillage has a COD concentration of ~100 g L^{-1} [66]. In this study, to make the experimental results more comprehensive, the feeding concentration was adjusted to different ranges, which will be discussed later. Homogenized feeding substrate from REC was pumped to FAC for predigestion.

Stages	1	2	3	4	5	Typical range [a]
pH	7.34 ± 0.05	/	7.12 ± 0.08	7.10 ± 0.07	7.01 ± 0.17	6.5–8.5 [33][b]
T (°C)	36.0 ± 0.7	36.3 ± 0.7	34.3 ± 1.8	34.3 ± 2.1	37.9 ± 1.0	35–40 [33][b]
DO (mg L^{-1})	0.01 ± 0.00	/	0.06 ± 0.04	0.15 ± 0.05	0.10 ± 0.03	/
ORP (mV)	−209 ± 14	−228 ± 24	−243 ± 40	−247 ± 37	−263 ± 23	−400—−150 [31]
TN (mg L^{-1})	591 ± 83	409 ± 37	237 ± 74	314 ± 50	306 ± 46	60–1000 [32]
TP (mg L^{-1})	3.4 ± 2.4	1.5 ± 0.4	0.9 ± 0.4	2.3 ± 1.1	2.2 ± 0.4	6–50 [32]
Alkalinity (mg L^{-1} as CaCO$_3$)	3087 ± 282	/	1455 ± 457	2478 ± 291	2204 ± 222	1500–5000 [33][a]
VFA (mg L^{-1} as HAc)	274 ± 97	/	199 ± 76	394 ± 84	469 ± 378	<1800 [31]
COD removal efficiency (%)	42.1 ± 6.7	50.6 ± 5.8	73.8 ± 11.0	61.7 ± 12.3	53.5 ± 8.7	/
VS removal efficiency (%)	26.8 ± 7.9[b]	37.1 ± 4.3[b]	72.7 ± 7.4	57.9 ± 13.2	56.4 ± 9.9	/
CH$_4$ content (%)	74.3 ± 2.0	74.6 ± 1.0	75.9 ± 1.9	74.6 ± 1.8	75.4 ± 1.0	/
CO$_2$ content (%)	22.3 ± 1.3	/	23.9 ± 1.9	25.2 ± 1.8	24.2 ± 1.0	/
H$_2$S content (ppm)	38.2 ± 4.1	/	147.2 ± 34.8	185.2 ± 28.1	371.7 ± 127.6	/
CH$_4$ yield (m^3-CH$_4$ kg-VS^{-1})	0.40–0.49	0.58–0.77	0.49	0.48	0.45	0.11–0.42 [52][a], [72]

[a]Typical value of operating parameters including pH, T, ORP, TN, TP, VFA and alkalinity were based on the description of typical anaerobic digestion systems. Typical values of CH$_4$ yield were based on earlier literature.
[b]For comparison purpose, VS removal efficiency in S1 and S2 has not been corrected by biomass calculation.

Table 5. Anaerobic digestion operating parameters and system performance in five selected stages.

This predigestion process will initially introduce a series of microbial strains, and to eliminate potential process inhibitors as reported earlier [55–57]. Afterwards, the predigested substrates were pumped continuously to AD for digestion. The effluent of AD was transferred via gravity to clarifier for sedimentation. The upper flow of clarifier was pumped to aerobic basin for aerobic treatment.

The evaluation period lasted for 171 days. Excluding the system set-up period, process data were obtained mainly from day 100 to day 171. Two intensive evaluation periods (day 100–116, period I, and day 161–171, period II) were applied in the study. These two intensive periods were corresponding to two different scenarios of anaerobic treatment for stillage in this study. In period I (day 100–116), the system organic loading rate was 8.54 kg COD m^{-3} day^{-1}, the raw thin stillage (~120,000 mg L^{-1} as COD) was directly fed into the REC without dilution, and no aeration process was added. The purpose of this period was to maximize the methane production by anaerobic treatment. In period II (day 161–171), the system organic loading rate was reduced to 40% of the period I, which is 3.50 kg COD m^{-3} day^{-1}. The thin stillage was diluted to ~50,000 mg L^{-1} as COD before fed to the REC, and the aeration process was operated to further polish the AD effluent and to produce another economic product, single cell protein (SCP). These two scenarios will be discussed later.

The cumulative CH$_4$ production and digested VS in stage I and II are shown in **Figure 3**. The CH$_4$ yield was calculated as the ratio of the two slopes. The CH$_4$ yield was reported based on VS
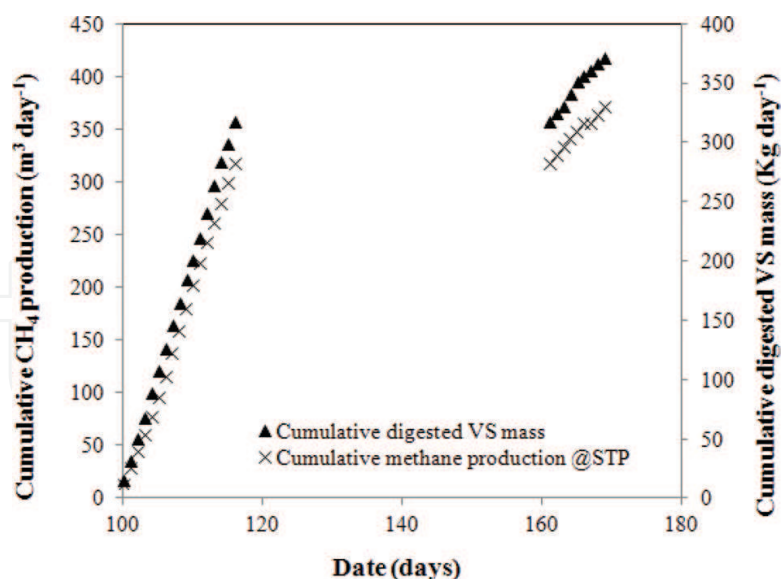
Figure 3. Cumulative CH$_4$ production at STP and cumulative VS digested during two intensive evaluation periods (I and II). CH$_4$ production yield was calculated based on the ratio of the two slopes.

removal because the organic loading of the stillage was mainly in the solid phase (VS 67,224 mg L^{-1}, COD 122,743 mg L^{-1}). The calculated CH$_4$ yield was 0.790 m^3-CH$_4$ kg-VS digested^{-1} (at standard temperature and pressure, STP). All the gas volumes mentioned hereafter have been normalized to STP) in period I, and 0.824 m^3-CH$_4$ kg-VS digested^{-1} in period II. Based on the measured VS concentration in thin stillage and the mean VS removal efficiency, the CH$_4$ yield could be converted to 0.507 m^3-CH$_4$ kg-VS fed^{-1} in period I and 0.414 m^3-CH$_4$ kg-VS fed^{-1} in period II.

AD process could be integrated to traditional corn-to-ethanol process as a treatment technique to the thin stillage product. The generated methane will partially replace the nonrenewable fuels and a large amount of energy could be saved from the removed evaporation process. Based on the lower heating value (LHV) of CH$_4$ (50.00 MJ kg^{-1}) and our CH$_4$ yield, the total energy output in this anaerobic system is 16.8 MJ kg-VS fed^{-1} in period I and 13.7 MJ kg-VS fed^{-1} in period II.

The energy saving is calculated based on several areas. In traditional process, the stillage treatment process including evaporation and syrup flash drying will take 38 MJ for each gallon of 95% ethanol produced [65], and DDGS treatment process will take 8.4 MJ [70]. In periods I and II, the evaporation process was removed to save 38 MJ, and DDGS productivity was decreased by 45.2 and 39.8% (SCP was considered as the same quality animal feed with DDGS); thus, the saved energy from these two processes was calculated and listed in **Table 6**. Energy recovered from produced methane was calculated based on the CH$_4$ yield and it is LHV. The consumed energy of applied anaerobic system was mainly focused on three mixing pumps in REC, FAC, and AD, respectively and the aeration activity in the aerobic system during period II. The energy cost of transfer pumps is negligible.

The power of the mixing pump was calculated based on the Camp-Stein equation for mixing with an impeller:

$$P = G^2 \mu V \tag{1}$$

Scenario		I	II	III
Description		Traditional EtOH process	High thin stillage feeding to maximum methane production, all the generated thin stillage will be treated	Low thin stillage feeding to produce methane and single cell protein, 40% of the generated thin stillage will be treated
Raw material		Corn grain 0.357 Bushel (12.6 L)		
Main product		95% ethanol 1 gallon (3.785 L)		
By-products	Biosolids (pound)	DDGS 6.43	DDGS 3.52	DDGS 3.52 and SCP 0.35
	Methane (m³@STP)	N/A	0.568	0.464
Energy saved (Megajoule)	From stillage treatment process	N/A	38	38
	From operation of applied AD system	N/A	−18.3 from three mixing pumps	−4.5 from three mixing pumps, and −0.02 from air diffuser
	From DDGS treatment process	N/A	3.8	3.4
	From methane recovered	N/A	18.7	15.3
	Total	N/A	42.2	52.2
Industrial cost saving (US cents)	From power saved by stillage treatment process	N/A	68.7	68.7
	From power saved by DDGS treatment process	N/A	6.9	6.1
	From operation of AD system	N/A	−33.1	−8.2
	From methane produced	N/A	9.9	8.2
	From biosolids produced	N/A	−40.2	−35.2
	Total	N/A	12.2	39.6

Three scenarios (traditional, high thin stillage feeding, and low thin stillage feeding) were applied.

Table 6. Summary of energy and industrial cost saving in traditional ethanol making process and integrated processes for producing one gallon of 95% ethanol.

where P is the power requirement (W), G is the average velocity gradient (S⁻¹), μ is the dynamic viscosity (N S m⁻²), and V is the reactor volume (m³). In this study, the applied G was a typical value in rapid mixing operations reported by Metcalf and Eddy [72], which is 1000 S⁻¹. μ was water dynamic viscosity at 60°C, 4.66 × 10⁻⁴ N S m⁻². V was calculated based on the flow rate (1.9 × 10⁵ L h⁻¹ thin stillage in period I and 7.6 × 10⁴ L h⁻¹ thin stillage in period II) and the applied HRT. HRT in the system is 5 days for REC, 1.5 days for FAC, and 12.6 days for AD in period I. In period II, the HRT for REC and FAC was kept the same, the HRT for aerobic basin is 1 day. To

save the dilution water usage and the operation cost, generally the system will use less flow rate rather than dilution in real industries. Thus, the applied HRT in period II should be the calculated result, which is mentioned in kinetic analysis section (5.34 days). The calculated energy consumption in mixing pumps is listed in **Table 6**. For each pump, a 70% pump efficiency was assumed.

The power of the aerator (mostly an air diffuser) is the main operation cost of the aerobic section. The power requirement was estimated based on the reported typical energy requirement form Metcalf and Eddy [72], which is 30 kW/10^3 m^3, the estimated result is shown in **Table 6**. To sum up, compared with the traditional ethanol making process, the anaerobic integrated process could save 42.2 MJ (period I) or 52.2 MJ (period II) for each gallon of 95% ethanol produced.

The calculation of industrial cost saving was similar to the energy saving. The power cost saved by thin stillage treatment process was calculated based on electricity (price based on US EIA report 2013). The operation cost of anaerobic system was calculated based on the energy consumption. The price of CH_4 comes from US EIA report 2013, and the price of DDGS comes from USDA livestock and grain market report (2013). The price of the SCP was assumed to be the same with DDGS. Since the anaerobic system cost and DDGS productivity reduction is the capital of the integrated system, in **Table 6**, they were shown in cash-negative format. After the calculation, the integrated system saved 12.2¢ (period I) or 39.6¢ (period II) for each gallon of 95% ethanol produced. Period II has a higher cost saving because the system applied in period II has just treated 40% of the generated stillage; thus, the energy consumption was lower. For a typical ethanol plant with 100 million gallon 95% ethanol yr^{-1} productivity, by applying this AD integrated system, the cost saving of the plant could reach $ 12.2 million by completely treating thin stillage with AD, or $ 15.8 million by partially (40%) treated. This amount is higher than the reported amount ($ 7–17 million, most likely 10 million) in the study of Schaefer and Sung [71] because the gate-to-gate life cycle assessment was more comprehensive in this study.

By changing the influent condition, two different scenarios of anaerobic digestion were studied in this research. For each gallon of 95% ethanol produced, when thin stillage was fed directly to the anaerobic digester without dilution, the produced CH_4 will be 0.568 m^3 at STP, system energy saving was 42.2 MJ, and industrial saving will be 12.2 cents compared with traditional dry mill process, which means a typical ethanol plant could save 12.2 million dollars per year. When thin stillage was partially (40%) fed to a smaller sequential anaerobic-aerobic system, the produced CH_4 will be 0.464 m^3 at STP, and system energy saving was 52.2 MJ. The industrial saving will be 39.6 cents, which means a typical ethanol plant could save 15.8 million dollars with this 40% of thin stillage. This study shows that thermophilic AD is a better use of thin stillage and is applicable to practical dry mill ethanol plants.

4. Benefit of anaerobic digestion process in biofuel recovery

In this chapter, three kinds of waste streams from real industries were selected to investigate their anaerobic treatability, economic feasibility, and applicability to the practical plants. Generally, these selected waste streams were applied to a pilot-scale anaerobic-aerobic biological treatment system to convert their organic fraction into renewable energy in the form of CH_4.

For paper mill effluents, the improved COD removal efficiency (55–70%) and the substrate utilization rate (0.28–0.46 d^{-1}) indicated that it is anaerobically treatable. The CH_4 yield (0.22–0.34 m^3-CH_4 kg-COD^{-1}) showed that the application of anaerobic technique has the potential to improve the energy footprints of the pulp and paper industry. For brown grease, the COD removal efficiency had somewhat been sacrificed (58%) to the maximum methane yield (0.40–0.77 m^3-CH_4 kg-VS^{-1}). The obtained high CH_4 yield showed that using brown grease for biogas production could serve as a profitable model for converting waste to renewable energy. For thin stillage, based on the high CH_4 yield (0.464–0.568 m^3 CH_4 @ STP per gallon 95% ethanol produced) and reducing energy consumption, a typical ethanol plant (producing 100 million gallon 95% ethanol per year) could save 12.2–15.8 million dollars per year, which indicated anaerobic digestion is a better use of thin stillage and is applicable to practical dry mill ethanol plants.

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